# TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

# PRESIDENT'S LETTER

Dear Friends,

It's hard for me to believe we are already heading into July. It seems as if we just issued the spring newsletter.

Your Executive Committee has been reviewing the abstracts submitted for the technical sessions at the forthcoming Fifty-fourth General Assembly in York, England, this coming October. I want to thank all the authors for their excellent submissions. Among all these very interesting talks, we are planning one session to focus on recent activities and developments in the area of Conflict Minerals. This session will include presentations and a panel discussion with a number of global experts covering the various aspects of CFS protocol, regulations and harmonization as well as the latest on the ground information in the affected countries. I expect this to be a highlight of the General Assembly and look forward to hearing from the experts.

In advance I wish to thank Metalysis for hosting this General Assembly and welcoming us to their plant. Ian Margerison and the team at Metalysis have been very busy working with Emma to make this visit to York a memorable one. Keep your fingers crossed for great autumn weather during the meeting.

This issue of the Bulletin contains two of the papers presented in Cape Town. Ulric's paper on transport of Class 7 materials, a current topic of discussion within the Executive Committee, as well as the paper presented on the Dodd-Frank act. I think both of these papers will be pertinent to the discussions in York.

At the York meeting, as an organization, we will discuss and vote on a few items relating to member compliance in the area of the CFS protocol, which the T.I.C. as an association fully supports, as well as an issue related to Class 7 compliance. The full content of the discussions is still being formulated; however, the debate and outcome will further prove to the global community that the T.I.C. is fully vested in supporting and advancing compliance and regulations in all areas pertaining to our global business and as an organization we will act to make certain our membership remains in compliance. I believe these discussions will be of the utmost interest to the General Assembly as a whole.

Lastly, I am pleased to report that we have already received six applications for admission to the T.I.C. at the upcoming General Assembly. This is excellent news and as I view it confirmation that the T.I.C. continues to be seen as an organization bringing value to its membership. I hope every member feels the same.

With that, please enjoy your summer or winter, depending on your hemisphere and as always please keep in contact with your Executive Committee should you have issues or questions of interest to the membership. You will find the names and contact details of all the Committee members in the last section of this Bulletin. We look forward to hearing from you.

PS: Congratulations to David and Jessica Gussack on their recent marriage.

Regards,

Dr Daniel F. Persico (Dan) President

CONTENTS	
President's Letter	18th Plansee Seminar14Member company news18Executive Committee18

BULLETIN Nº 154 ISSN 1019-2026

# FIFTY-FOURTH GENERAL ASSEMBLY

The Fifty-fourth General Assembly and associated technical meeting will be held in York, England, from October 13th to 16th 2013. The conference will take place at the Royal York Hotel & Events Centre, where a block booking of bedrooms has also been secured.

On Sunday October 13th, the registration desk will be open from 10a.m. to 1p.m. and 2p.m. to 5p.m. All participants are invited to a Welcome Reception that evening, from 6p.m. to 8p.m.

The formal General Assembly of the association will be held from 8.30a.m. till 9.30a.m. on Monday October 14th, this session is for current members only. It will be followed by technical presentations until mid-afternoon, with a break for a buffet lunch.

Companies wishing to apply for membership at this General Assembly are reminded that their completed application form should be returned to the T.I.C. by September 14th at the very latest. For any further information on becoming a member, please refer to the following page of our website: <a href="http://tanb.org/applmemb">http://tanb.org/applmemb</a>

On Monday evening, all participants are invited to a Gala Dinner to be held in the very special setting of the National Railway Museum, just a few steps away from the hotel.

A second technical session will be held on Tuesday October 15th, breaking for a buffet lunch and ending mid-afternoon.

The full technical programme is published herebelow. Papers on subjects related to the Supply Chain will be grouped together and followed by a panel discussion.

On the morning of Wednesday October 16th, delegates will be given the opportunity to visit the facility of Metalysis, located a little over an hour away from York. Lunch will be taken at the Monk Fryston Hall Hotel, a stately manor house dating back to the 12th century. The busses will then continue to the small market town of Masham, where a guided tour of the Theakston Brewery has been arranged.

Tours for accompanying persons are also being arranged for Monday, Tuesday and Wednesday. The first day will focus on the city of York, with its Medieval streets and Georgian avenues, the famous Minster and city walls. Participants will also discover the Jorvik Viking Centre and York's Sweet Story. The second day will take participants a little further afield, aboard a magnificent steam train, to the picturesque village of Goathland. The afternoon will be spent visiting Castle Howard, a beautiful stately home set in spectacular grounds. On Wednesday, accompanying persons will leave York at the end of the morning for Monk Fryston Hall Hotel, where they will take lunch with the delegates who visited Metalysis. They will then continue to Masham for the guided tour of the Theakston Brewery.



Views of Theakston Brewery

An invitation will be sent to the nominated delegate of each member company in the second half of July. Others who would like to attend should contact the T.I.C. as soon as possible.

# TECHNICAL PROGRAMME

The following papers are expected. The announced presenter is the first author listed, unless otherwise specified. The papers are shown in alphabetical order of first author (not in running order).

#### Taboca - moving forward and getting into upper gears

by Ian Gordon Hall Dun and Jorge Diaz, Mineração Taboca S.A.

#### Modeling the limitations to CV/g in tantalum capacitors

by James Allen Fife, KEMET Blue Powder Corp.

#### New high productive electron beam melting system for bulky refractory metal materials

by Jochen Flinspach, Arno Niebling and Dieter Kaufhold, ALD Vacuumtechnologies GmbH (engineering company of the AMG group)

#### Conflict free mineral supply chains: from planning to action

by Tyler Gillard, OECD

#### Ferro-niobium in cored wire

by Rainer Hackstein, Global Metwire Injection S.L.

#### Novell high voltage tantalum powder for new applications

by Marcel Hagymási, Helmut Haas, Christoph Schnitter and Holger Brumm, H.C. Starck GmbH

#### The volumetric efficiency of competing capacitance solutions

by Randy Hahn and Jonathan Paulsen, KEMET Electronics

#### Field update on the iTSCi system

by Karen Hayes, Pact

### Study of the morphology and electrical properties of tantalum powders prepared by electro-deoxidation method

by Li Jun-Yi, Zheng Ai-Guo, Wang Dong-Xin and Luo Wen, Ningxia Orient Tantalum Industry Co. Ltd (presented by Guo Hong)

# The Metalysis flexible manufacturing process - transforming the world of tantalum

by Ian Margerison, Metalysis

#### A unique Metalysis tantalum product offering for capacitor manufacture

by Ian Mellor, Ian Margerison, Greg Doughty and Lucy Grainger, Metalysis

#### Developing a basis of safety for the manufacture and processing of metal powders by Michael Merritt and David Firth, Chilworth Technology Ltd (a DEKRA Company)

High voltage tantalum polymer capacitors with small form factor and low ESR, supporting leading-edge consumer equipment

by Tak Ohashi, SANYO Electric Co. Ltd

Dodd-Frank expectations and Conflict-Free Smelter Program delivery for tantalum, tin, tungsten and gold supply chains by Michael Rohwer, EICC

#### Stimulating statistics: the goal of rapid reporting of dependable data

by Ulric Schwela, Tantalum-Niobium International Study Center

#### The tantalum supply chain: strengthening transparent traceability

by Ulric Schwela, Tantalum-Niobium International Study Center

#### The effect of additives to the conductive polymer solution for low-ESR electrolytic capacitors by Yasuhiro Tomioka, Yasuhisa Sugawara and Koji Sakata, NEC TOKIN Corporation

(presented by Takasi Kono)

#### How tantalum competes in global markets

by Dennis Zogbi, Paumanok Publications, Inc.

# TRANSPORT PROBLEMS AND SOLUTIONS

Paper presented on October 8th 2012 by Ulric Schwela, Technical Promotion Officer of the T.I.C., as part of the Fifty-third General Assembly & Symposium 2012 on Tantalum and Niobium, held in Cape Town, South Africa.

# **ABSTRACT**

Ten years ago, the T.I.C. took its first steps into tackling the difficulty of transporting tantalum raw materials, which are Naturally Occurring Radioactive Materials (NORM). Back in 2002 there was an ecdotal information from a few members about new regulations which were classifying tantalum raw materials as radioactive and this made it difficult to obtain transport. At the prompting of A&M Minerals and Metals, a T.I.C. delegation of four people from A&M, A.H. Knight (AHK) and H.C. Starck (HCST) attended a small meeting in Paris organised by the International Atomic Energy Agency (IAEA). It was the T.I.C.'s first exposure to how the IAEA functions and a lot of useful information was exchanged. In July 2003 another T.I.C. delegation from AHK and HCST plus the Secretary General attended a major transport conference held at the IAEA headquarters in Vienna. In October of the same year, at the T.I.C. General Assembly in Lisbon, the President Dr Gerblinger called for a 'Working Group on Transport of Tantalum-bearing Materials with Naturally Occurring Low-level Radioactivity'; this was later shortened to the 'Transport Committee'! The T.I.C. had taken the first step down a long and winding path which was to see preliminary data collected from members, a major study into the risk posed by transport of tantalum raw materials, close co-operation with the IAEA in determining the appropriate regulatory level for transport of NORM, being a founding member of the International Steering Committee on Denial of Shipment (ISC) and later being Chair of the ISC, as well as developing a number of information papers and tools which could assist members in their task of arranging transport of their NORM tantalum raw materials. The above review will lead us to 2012 and the current status of the transport regulations, the IAEA Coordinated Research Project on NORM, the work of the ISC and other stakeholder bodies of industry and regulators. The presentation will conclude with a time line for the work anticipated for 2013 onwards.

# **RADIOACTIVE MATERIALS TRANSPORT REGULATIONS**

### WHEN TANTALUM MET RADIOACTIVITY

Back in 2002, a number of companies in the tantalum industry began experiencing difficulties in transporting their raw materials, the tantalum minerals and tin slags which provide the bulk of the supply of tantalum, and they brought this issue to the attention of the T.I.C. These companies were being told that their hitherto ordinary minerals were now being classed as radioactive during transport due to the small but measurable levels of naturally occurring thorium and uranium present in those raw materials.

Thorium and uranium? These natural elements are present in all the soils and rocks of the world, sometimes at insignificant and sometimes at significant levels, most famously in the beach sands of Guarapari in Brazil and in parts of the Kerala and Tamil Nadu states of southern India, the bedrock at Ramsar in northern Iran, or almost anywhere where people live on granite such as Cornwall in the United Kingdom or Colorado in the U.S.A. In order not to consider everything in the world as radioactive and instead focus on regulating materials where there is a positive safety vs. cost balance, authorities set an 'exemption level' below which material is considered normal.

As we later found out, up until 1996 this exemption level had been above the typical radioactivity of tantalum raw materials. In 1996 the IAEA <sup>1</sup> issued a new BSS <sup>2</sup> and, at the same time, new transport regulations based on the values in the new BSS. This resulted in a lower exemption level which brought almost all tantalum raw materials into the scope of radioactive materials transport.

It took a few years for these high level regulations to trickle down and become legally binding. First they were taken up in the UN <sup>3</sup> model regulations, often known as the 'orange book' due to its distinctive appearance. Somewhat later the revised values in the UN orange book were then incorporated into modal regulations such as the ICAO <sup>4</sup> Technical Instructions which regulate international air transport, and more importantly the IMO <sup>5</sup> Dangerous Goods code, known simply as the IMDG code, which regulates international sea transport. Later still, the new exemption level was applied in national legislation and regulations of individual countries, which then became legally binding on any transport within that country. This was all very confusing but one thing was certain, they all agreed 'our' material was radioactive. It also resulted in a number of questions:

- What was the consequence of this change?
- Why was this change brought about? Was it justified?
- Was the new exemption level correct? Could it be changed again?
- How could we determine what the correct exemption level should be?

And perhaps most importantly:

Who could answer all our questions?

All these questions and their answers still lay in the future. First, the T.I.C. set about learning more about the issue.



Figure 1: Overview of the regulatory flow from the IAEA BSS, through the IAEA transport regulations, the UN model regulations, to various modal regulations *e.g.* IMDG

<sup>4</sup> International Civil Aviation Organization

<sup>&</sup>lt;sup>1</sup> International Atomic Energy Agency

<sup>&</sup>lt;sup>2</sup> Basic Safety Standards

<sup>&</sup>lt;sup>3</sup> United Nations

<sup>&</sup>lt;sup>5</sup> International Maritime Organization

### **OUR FIRST BABY STEPS – AN INTRODUCTION TO TERMINOLOGY**

In October 2002, at the prompting of A&M Minerals and Metals, a delegation from the T.I.C. members A&M, A.H. Knight and H.C. Starck attended a small technical meeting in Paris that was part of an IAEA research project of great relevance to the transport of tantalum raw materials.

The tantalum problem was presented to the other participants which had quite varied backgrounds including someone from the zircon industry in South Africa, and the Brazilian regulator looking at uranium ores. The delegation established useful contacts for the future and learnt the correct interpretation and application of the transport regulations. These regulations involved much new terminology for tantalum raw materials:

- the main radionuclides of interest were Th(nat) and U(nat);
- the exemption level for Th(nat) and U(nat) was 1  $Bq/g^6$ , however:
  - o para. 107 (e) applied to this material, increasing the exemption level to 10 Bq/g;
- the so-called "A2" value for Th(nat) and U(nat) was "Unlimited", and consequently:
  - o the material was LSA-I 7;
  - o the UN number was 2912, except if the surface dose rate was below 5  $\mu$ Sv/h then:
    - the UN number was 2910;
      - the packaged material was an "Excepted Package".

This wasn't the whole story and we understood we had so far only learnt the basics. Apparently, the material was also NORM <sup>8</sup>, it had a lot of daughters, sometimes ten and sometimes fourteen, which some people included in their calculations and others excluded. Which was correct? We decided we needed to learn some more.

# EXTENDING OUR LEARNING OF THE REGULATIONS

### TAKING A WALK - MEETING THE REGULATORS

The following year, in July 2003, delegates from A.H. Knight, H.C. Starck and the T.I.C. itself attended a major transport conference at the IAEA headquarters. By sheer good fortune, very early in its learning curve the T.I.C. had the opportunity to be exposed to the full gamut of radioactive materials transport: this was the first time the IAEA had organised such a large conference dedicated to transport.



**Ulric Schwela** 

Figure 2: IAEA headquarters, Vienna

Instead of a small group of regulators such as were present at the Paris meeting, here under one roof the T.I.C. could introduce itself to almost every regulator in the world in order to gauge their opinion and interpretation of the transport regulations and foster useful contacts.

It became clear that we did not have to include any daughters in calculations, even though a few solitary regulators insist on doing this differently to everybody else and including them.

### **GETTING OFF TO A TROT – CLARIFYING THE EXEMPTION LEVEL**

Along the way we came across more and more references that would apply to tantalum raw materials in various situations, including the IAEA Safety Glossary, and the radiation safety guidance document RS-G-1.7, both of which referred to **four** natural radionuclides that should be considered, not just the two Th(nat) and U(nat) we first had been told about: K-40, Th-232, U-235 and U-238. This was resolved by learning that K-40 and U-235 are never a limiting factor in tantalum raw materials, therefore it is sufficient to consider Th(nat) and U(nat) only.

The document RS-G-1.7 also referred to exemption levels of 1 Bq/g for Th(nat) and U(nat) and with no 'para. 107(e)' factor of 10; however this was also resolved in that the document applied to exempting materials being stored and handled and did not apply to transport.

<sup>6</sup> Becquerels per gramme

<sup>&</sup>lt;sup>7</sup> Low Specific Activity, category I

<sup>&</sup>lt;sup>8</sup> Naturally Occurring Radioactive Material

So despite these apparent distractions and the riot of alphanumeric radionuclide codes that we were being threatened with having to deal with, it all came back to the same IAEA transport regulations. The IAEA shorthand code for these regulations has changed over time being variously ST-1, ST-1 (Revised), TS-R-1 and now, as of 2012, they are known as SSR-6. The content however has remained essentially the same since 1996, with none of the changes affecting tantalum raw materials. The exemption level for tantalum raw materials is still 10 Bq/g for the combined thorium and uranium parents, or Th(nat) and U(nat).

**IAEA Safety Glossary** 

Terminology Used in Nuclear Safety and Radiation Protection 2007 Edition



Figure 3: IAEA Safety Glossary

### **UP AND RUNNING – ESTABLISHING THE TRANSPORT COMMITTEE**

After this year of learning, what did the T.I.C. decide to do? At the end of October 2003, at the Forty-fourth General Assembly in Lisbon, the "Working Group on Transport of Tantalum-bearing Materials with Naturally Occurring Low-level Radioactivity" was set up. Being an awfully long name for a working group, this was soon shortened to a much simpler "Transport Committee".

Over the next few years the Transport Committee set about determining what the correct exemption level for tantalum raw materials should be, in order that it could present an informed opinion to the IAEA and other stakeholders as appropriate.

# PREPARING AND DEFENDING A THESIS

### **PERFORMING A TRANSPORT STUDY**

Having established a working group to study the subject in more depth, the first task was to prepare a tender document for a risk assessment into the transport of tantalum raw materials. After reviewing a number of project proposals, the company chosen to conduct this work was SENES <sup>9</sup>, based near Toronto, Canada. The transport study commissioned by the T.I.C. ran from 2005 to 2007 and included significant co-operation by a number of T.I.C. members whether in providing analytical services or mineral samples.



Figure 4: T.I.C. transport study

The study looked at the characteristics of the tantalum raw materials during transport, mainly in normal and also in accident conditions. A great number of radiation surveys were conducted on loaded containers, including background readings. Various exposure scenarios were developed according to each type of transport worker, with the frequency and length of time exposed carrying these materials. Doses were also modelled and compared with the measured doses. From this we found out a number of things:

- the uranium and thorium distribution is fairly uniform in tantalite, whereas in tin slag it is in distinct groups according to its provenance;
- the concentration of the non-radioactive elements, the mineral grain size and density have no significant effect on the radiation properties;
- 95% of the 64 shipments of tantalum raw materials surveyed exceeded the 10 Bq/g exemption level;
- modelled doses were very close to measured doses, modelled exceeding measured slightly due to containers often not being fully loaded;
- doses to the public were much lower than the 10 μSv/y upper limit for trivial doses, by at least two orders of magnitude.

From the large amounts of data generated, a table comparing dose factors was drawn up (Table 1) which showed us that the most exposed workers were those at facilities, however as they are not within the scope of transport regulations this meant that the most exposed transport worker was the truck driver. Focusing on the truck driver, we drew up a table comparing the exposure that would result from various possible exemption levels.

<sup>9</sup> Specialists in Energy, Nuclear and Environmental Sciences

This showed us that:

- the most exposed transport worker is the truck driver <sup>10</sup>;
- if the exemption level was ten times higher *i.e.* 100 Bq/g and all material transported was at that level, then the doses to truck drivers would be 0.91 mSv/y, below the 1 mSv/y limit for uncontrolled exposure to members of the public;
- if a dose criterion of 0.3 mSv/y was taken into account as recommended by ICRP <sup>11</sup>, this would suggest an exemption level of 30 Bq/g to be the most appropriate level for tantalum raw materials.

This figure of 30 Bq/g is the level which our transport study tells us is the appropriate safe exemption level for tantalum raw materials and which the T.I.C. continues to aim to achieve via its on-going involvement with TRANSSC <sup>12</sup>, the standing body of the IAEA which reviews and revises the transport regulations.

	Potential	Material	% of Shipments That Exceed Potential Exemption Value	Combined Annual Dose (mSv/y) at Potential Exemption Value			
Table 1: T.I.C. transport study, potential annual doses to transport workers	Exemption Value (Bq/g)			Truck Driver	Dock Worker	Seaman	Trainman
	10	Tantalite	78	0.12	0.016	0.0021	0.0097
		Slag	45	0.12	0.016	0.0021	0.0098
	30	Tantalite	16	0.31	0.040	0.0052	0.024
	30	Slag	27	0.37	0.049	0.0062	0.029
	50	Tantalite	2	0.48	0.062	0.0080	0.038
		Slag	23	0.54	0.071	0.0091	0.043
	70	Tantalite	0	0.65	0.084	0.011	0.051
		Slag	9	0.71	0.093	0.012	0.056
	90	Tantalite	0	0.82	0.11	0.014	0.064
		Slag	9	0.89	0.12	0.015	0.070
	100	Tantalite	0	0.91	0.12	0.015	0.071
		Slag	0	0.97	0.13	0.016	0.077

# WORKING WORLD – DISCUSSION (AND MORE DISCUSSION)

# WHAT IS A DELAY/DENIAL OF SHIPMENT?

Having obtained a result from the transport study, this now needed to be applied in the real working world. Going back to first principles, the initial reason for performing the transport study was due to denial of shipment. The information gained from the transport study could then be applied in three ways:

- In the long term, by seeking to refine the application of the radioactive materials transport regulations through the use of the most appropriate level of regulation according to the radiological risk posed by the transport of these materials. This would reduce the burden on all stakeholders, including regulators, by exempting materials whose regulation is not warranted.
- In the medium term, by identifying and addressing the root causes of the denial of shipment experienced by industry, and subsequently developing and disseminating tools for industry to use in communication with other stakeholders.
- In the short term, by providing the information and knowledge needed to confidently discuss and resolve the immediate issues and concerns of other stakeholders, be they carriers, local authorities or national regulators, whenever a specific instance of denial of shipment should arise.

To be clear about what we mean by denial of shipment, it is useful to consider a definition for this:

The refusal (whether explicit or implicit) to carry a shipment of radioactive material even though it conforms to all the applicable radioactive materials transport regulations. It does NOT include where the physical capability to handle the radioactive material is not in place.

What then are the regulations that should be applied? And why is transport denied even if regulations are being met?

<sup>&</sup>lt;sup>10</sup> Workers involved in loading and unloading containers are exposed to higher doses than truck drivers, however as per the IAEA transport regulations, they are not considered transport workers if the loading/unloading facility is a licensed radiation facility with a radiation protection programme.

<sup>&</sup>lt;sup>11</sup> International Commission on Radiological Protection

<sup>&</sup>lt;sup>12</sup> Transport Safety Standards Committee

## WHY IS THERE DENIAL OF SHIPMENT?

Referring again to the diagram of regulatory hierarchy in Figure 1, we found out that national regulations are derived directly from the IAEA transport regulations and not from the UN 'orange book' or modal regulations. In some countries this does not pose a problem as they apply the IAEA transport regulations directly, however many countries exercise the option to amend parts of the regulations or to place additional requirements. These additional regulations or national variations all contribute to a great variety of differences between countries. Further, at the national, regional and local level there may be overlapping interests between various regulatory bodies, be they transport, environmental, maritime safety, radiation safety or others still. This cocktail of jurisdictions can result in contradictory requirements which are consequently impossible to meet.

The solution to this dilemma is covered by one word: **harmonisation**. Harmonisation is variously used to describe the alignment of national regulations with the common body of IAEA transport regulations, or the alignment of the latter with modal regulations and the UN orange book for here too is fertile ground for seeds of discord to grow. The end goal is the same: fewer differences, fewer contradictions, fewer problems. As a result radioactive materials transport regulations could be applied more uniformly and more consistently, with a net benefit to safety.

# **COLLABORATION AND COORDINATION**

### **COMMITTEES & NETWORKS**

To resolve these differences it was necessary to engage with the many diverse stakeholders, to which end the ISC <sup>13</sup> was set up in 2006, with members drawn from UN agencies, national regulators and various industries. The T.I.C. was one of the founding bodies of the ISC and also chaired the ISC in 2011-2012.



Figure 5: Structure of the International Steering Committee on Denial of Shipment of Radioactive Material (ISC) Over time the ISC developed into a structure at three levels, each with its respective co-ordinator:

- the ISC, led by the Management Team;
- a Regional Network 14, led by its Regional Co-ordinator;
- a National Committee, led by its National Focal Point <sup>15</sup>.

Over the years this Committee worked along six parallel lines:

- awareness: raising the profile of the denial of shipment issue;
- communication: preparing a common message and providing tools;
- economic: assessing the impact of denial of shipment;
- harmonisation: identifying and reducing the differences in regulation;
- lobbying: explaining the benefits of radioactive materials and the need for transport;
- training: providing improved capacity in handling transport of radioactive materials.

# **TECHNICAL COOPERATION – JORDAN**

Following the lines of awareness, harmonisation and training, in 2012 the IAEA has begun rolling out a training programme coordinated by its Technical Cooperation Department, with a seven-figure budget for the first four years and a rolling two-year programme renewal. The programme is intended for national regulators and, significantly, it is the first time that the element of denial of shipment is being included in training for regulators.

The participants get funding for attending the training course on the condition that they supply the IAEA with details of their own national regulatory infrastructure and the nature of Class 7 transport in their country, including which materials and which industries are involved. This 'questionnaire' approach encourages regulators to think about what exactly radioactive materials transport involves in their country and who is affected by its transport, or indeed, who is affected by denial of shipment.

The programme's first training workshop was held in Jordan and included a number of regulators from western Asia / the Middle East.

<sup>&</sup>lt;sup>13</sup> International Steering Committee on Delay and Denial of Shipment of Radioactive Materials

<sup>&</sup>lt;sup>14</sup> Regional Networks were set up for Africa, Americas, Asia and Europe.

<sup>&</sup>lt;sup>15</sup> Each IAEA Member State has been asked to appoint a National Focal Point (NFP). Currently 82 countries have appointed a NFP. A list can be found at:

http://www-ns.iaea.org/downloads/rw/transport-safety/denial-shipments/national-focal-points-list.pdf

## **TECHNICAL COOPERATION – ZIMBABWE**

The initial workshop was followed soon after in July with one in Zimbabwe for African countries, with participation by Algeria, Burkina Faso, Democratic Republic of the Congo, Egypt, Ethiopia, Ghana, Cote d'Ivoire, Kenya, Mauritius, Morocco, Nigeria, Sierra Leone, Sudan, United Republic of Tanzania, Zambia and Zimbabwe. Most of these countries are of relevance to the T.I.C. and transport of tantalum raw materials.

Following a SWOT (Strengths, Weaknesses, Threats & Opportunities) analysis conducted at the initial meeting, a more tailored training will be prepared for the individual national regulators.

# TECHNICAL COOPERATION – FLORIDA, U.S.A.

A third workshop for countries from both North and South America was scheduled for December, to be held in Florida, U.S.A.<sup>16</sup>

# **PRODUCTION – THE HOLY GRAIL**

## **COMMUNICATION TOOLS**

As part of the communication line of work of the ISC, a number of tools have been prepared over the years, including simple but practical fact sheets. For tantalum these explain what the material is, what it is used for, that the metal isn't radioactive but that the mineral is Class 7 and is affected by denial of shipment.



Figure 7: Brochure on Denial of Shipment of Radioactive Material, 2nd edition, 2012

Various information videos are available for materials that have similar Class 7 transport issues as tantalum raw materials, such as uranium ore concentrates or uranium yellowcake. After all, the radioactivity in tantalum raw materials is due in large part to the presence of uranium, as well as the thorium also present, all of natural origin. Those radioactive materials which are produced artificially also have much in common with the transport of all radioactive materials, e.g. cobalt-60 or medical radioisotopes.

The IAEA set up a website for further information, with an introduction to the issue and a number of supporting documents:



Figure 6: Brochure on Denial of Shipment of Radioactive Material, 1st edition, 2011

#### http://goto.iaea.org/denialofshipment/

Furthermore a brochure was prepared by a joint IAEA and cobalt-60, radiopharmaceutical and tantalum industry collaboration. Its goal is to demystify denial of shipment and explain the need for Class 7 transport, first issued in 2011 and updated in 2012.

### NOT JUST MORE MEETINGS!

Looking to the future, a reduced number of meetings presents itself as the T.I.C. focuses on more effective participation in a core schedule:

- TRANSSC being the permanent body with responsibility for deciding on transport regulations, it is important to remain engaged in order to have advance warning of developments on the horizon.
- Denial of shipment is likely to remain an issue for some time, however the ISC is scheduled to dissolve after its eighth and final meeting to be held June 11th to 14th 2013, as this year was set by the IAEA as a target for resolving denial of shipment. It is currently unclear who will take over its workload. It may either be incorporated into TRANSSC where there is

<sup>16</sup> It was later understood that the Florida workshop has been postponed.

already some common ground in TRANSSC's own work plan, or an alternative proposal of expanding an existing body of UN agencies may gain traction.

In addition to the above two core transport activities, the T.I.C. is sometimes also involved with other on-going activities which have the potential to impact on the tantalum industry:

- The EU's focus on NORM, likely to continue with yearly meetings held every November and sometimes also in June.
- The series of PATRAM <sup>17</sup> conferences held every three years, alternating between the U.S.A. and another host country. The next one is scheduled for August 2013 in San Francisco, followed by 2016 in Japan.
- The series of major NORM conferences now also established every three years, with the next one (NORM VII) scheduled for Beijing from April 22nd to 26th 2013. <sup>18</sup>

### A FUTURE PROTOCOL

A future focus for the T.I.C. will be the preparation of a protocol for the transport of Class 7, as requested by the T.I.C. Executive Committee. This may be based on the IAEA document TS-G-1.6 which is a collection of schedules arranged according to UN number, e.g. UN 2912 which applies to tantalum raw materials.

All known regulatory additions and variations should also be compiled as much as possible, including differences at the national, regional and local/port level.

There should also be background information on what NORM and LSA are, with clarification and a clear definition, as some regulators have a different interpretation to that which has broad consensus and it is important to record this so that users are aware of it.

Reference can also be made to posters prepared for previous conferences and included in an information package.

#### IAEA Safety Standards

Schedules of Provisions of the IAEA Regulations for the Safe Transport of Radioactive Material (2005 Edition)

Safety Guide No. TS-G-1.6

Figure 8: IAEA guidance document TS-G-1.6

### **S**YMPOSIUM SOUVENIR – TRANSPORT TOOLS AND REGULATORY RESOURCES

The Symposium participants were offered a selection of items to take away, including copies of the denial of shipment brochure, USB memory sticks pre-loaded with the relevant regulations and supporting documents and guidelines, various denial of shipment reporting forms, information posters <sup>19</sup> and videos, a copy of the T.I.C. transport study report, plus a number of stickers prepared by another Class 7 transport stakeholder with the words "Life Saving", which is true of much of the work that is being done to resolve denial of shipment. Symposium participants were encouraged to display these stickers prominently to help spread the message of the beneficial transport of Class 7.

As shown by the materials available, over the years the T.I.C. has contributed to the production of a number of disparate communication tools and publications in order to assist with resolving the problem of transport of Class 7 tantalum raw materials. Now a more comprehensive and collected work in this area is planned, that should help put all the information into context and make it more accessible and usable.

<sup>&</sup>lt;sup>17</sup> Packaging and Transport of Radioactive Materials

<sup>&</sup>lt;sup>18</sup> All the T.I.C. Symposium participants were invited to contribute abstracts for NORM VII and/or PATRAM by the end of October 2012.

<sup>&</sup>lt;sup>19</sup> For the full size posters you may access the following links:

<sup>-</sup> Figure 9: <u>http://tanb.org/webfm\_send/222</u> (from 2010)

<sup>-</sup> Figure 10: <u>http://tanb.org/webfm\_send/223</u> (from 2012)



Figure 9: Poster from 2010 on the need for transport of tantalum raw materials and the results of the transport risk assessment, presented at NORM VI, Marrakech, Morocco



# **CONCLUSION**

Looking back at the initial list of questions, we can see that we have some answers and that these, in turn, have led to new avenues to pursue. The changes to the transport regulations that came about as a result of the new exemption level introduced in1996, have had a major impact on the tantalum industry, more so than any other as its raw materials are the only ones whose radioactivity concentration straddle the current exemption level. While the changes brought about in 1996 were no doubt justified overall, the appropriateness of the current exemption level to specific contexts such as the tantalum industry is very much open to question given the findings obtained by the T.I.C.'s transport study. Just as the exemption level changed in 1996, in theory it could change again, however justifying another change and ensuring that any new change takes cognisance of its potential impact on other industries will require a great deal of skill. Encouragingly there exists today a much better overview and awareness of the various industries utilising low level radioactive materials, particularly NORM.

The preparation of a T.I.C. protocol for Class 7 transport and continued engagement and co-operation with the IAEA, as well as key national regulators where appropriate, is the current way forward to address the difficulties experienced by the tantalum industry in transporting its raw materials in a safe and sustainable manner.

# SEC FINAL RULE ON DODD-FRANK CONFLICT MINERALS SECTION 1502

Paper presented on October 8th 2012 by Dr Daniel F. Persico of KEMET Electronics, as part of the Fifty-third General Assembly & Symposium 2012 on Tantalum and Niobium, held in Cape Town, South Africa.

# **INTRODUCTION**

This review of the Dodd-Frank Wall Street Reform and Consumer Protection Act, Conflict Minerals Section 1502 final rule is meant to serve as a broad overview and is in no way comprehensive. The final document spans 356 pages and covers not only the original proposed rule but also includes a significant number of the comments as received during the comment period as well as the details of the final rule. Individuals should read the entire document to fully understand the responsibilities this rule places on them and/or their companies.

# DISCUSSION

The Dodd-Frank Wall Street Reform and Consumer Protection Act (Pub.L. 111-203, H.R. 4173) was signed into U.S. federal law on July 21st 2010. Title XV of this legislation contains several specialized disclosure provisions of which Section 1502, the 'Conflict Minerals Statutory Provision' is one. The intent of Section 1502 is to support regional and international efforts to prevent the sale of 'conflict minerals' (cassiterite, columbotantalite <sup>20</sup>, wolframite and gold) that directly or indirectly finance or benefit armed groups in the covered countries (the DRC and the nine adjoining countries).

Most simply stated, Section 1502 proposed to require that a 'person described' (someone who is required to file a report under the Exchange Act Section 13 (p)2) disclose annually whether any conflict minerals that are 'necessary to the functionality or production of a product manufactured by such a person', originated in the covered countries. If this is the case, the person would be required to file a report describing, among other matters, the measures taken to exercise due diligence on the source and chain of custody of those minerals, including an independent private sector audit of the report, to be certified by the person filing the report.

On December 15th 2010, the SEC proposed rules necessary to implement Section 1502 and opened up an initial comment period to end January 31st 2011. This initial comment period was extended to March 2nd 2011. In addition to the comment period, a public roundtable was held on October 18th 2011, in Washington D.C., at which time further comments were requested.

The SEC received 420 individual comment letters in response to the proposed rules, including one from the T.I.C. and others from T.I.C. member companies (current and past). The contents of these comment letters were considered in formulating the final rule. On August 22nd 2012, by a 3-2 vote, the SEC issued a final rule requiring issuers to disclose their use of conflict minerals and whether those minerals originated from the covered countries.

An issuer (domestic or foreign) is subject to the rule if (1) it files Exchange Act reports with the SEC and (2) conflict minerals are necessary to the functionality or production of a product that it either manufactures or contracts to be manufactured. The first reporting period is calendar year 2013, with the first report due by May 31st 2014.

When considering if the conflict mineral is 'necessary to the functionality of the product', the final rule eliminates the reporting of conflict minerals that are contained in, but unintentionally added to products, e.g.:

• trace or naturally occurring by-products do not require reporting.

Another consideration is whether the mineral is necessary to the product's generally expected function, use or purpose, e.g.:

when the primary purpose of a product containing a conflict mineral is ornamental or for decoration, the conflict
mineral is unrelated to the product's functionality - therefore a report is not required.

When determining if the conflict mineral is 'necessary to the production' of a product, the issuer should consider whether the conflict mineral is intentionally added in the production process, is included in the product, or necessary to produce the product. In a change from the original proposal, the minerals must be contained in the product and necessary to its production, e.g.:

• reporting is not required when a conflict mineral is present in a catalyst used in the production of the product.

Using the same logic, a machine tool made of a conflict mineral may be necessary to the production of the product. However, as the conflict mineral is not contained in the final article reporting is not required.

If the issuer is not directly manufacturing the product, yet has some influence over the manufacturing of the product, an issuer is considered to be 'contracting to manufacture'. In a change to the proposed rule, a company is not deemed to have influence over the manufacturing if it only:

- attaches its brand or logo to a generic product manufactured by a third party (e.g. private label);
- services, maintains or repairs a product manufactured by a third party;
- specifies or negotiates contractual terms with a manufacturer that do not directly relate to the manufacturing of the product.

The final rule is divided into a three-step compliance process:

- 1) A company must determine whether it is subject to the requirements of the Conflict Minerals Statutory Provision. As discussed above, this determination is based on whether the conflict minerals in question are necessary to the functionality or production of a product manufactured or contracted by the issuer to be manufactured.
- 2) If yes, the company must conduct a reasonable country of origin inquiry (RCOI) to determine if the necessary conflict minerals used originated in the covered countries or are from recycled or scrap sources.
- 3) If a company determines, or has reason to believe, that the conflict minerals originated in the covered countries and are not, or may not be, from recycled or scrap sources, it must exercise due diligence on the source and chain of custody of the conflict minerals and may need to provide a Conflict Minerals Report (CMR) describing the due diligence.

The RCOI will result in a conflict mineral source designation, which must fall into one of the following categories:

- DRC conflict free;
- not DRC conflict free;
- DRC conflict undeterminable.

<sup>20</sup> Referred to in the SEC Rules as columbite-tantalite

The source designation determines the appropriate next steps such as:

- due diligence on the source and chain of custody;
- filing of a CMR;
- an independent private sector audit of the CMR.

If due diligence is necessary, it must conform to a nationally or internationally recognized due diligence framework such as the guidance issued by the Organization for Economic Co-operation and Development (OECD).

Regardless of the final source designation, all issuers must file the Form SD providing a brief description of the RCOI and the results.

Companies that find they are not able to determine the exact source of their conflict minerals ('DRC conflict free' or 'not DRC conflict free') are able to report their sources as 'DRC conflict undeterminable' giving them additional time (up to 2 additional years for large companies or up to 4 additional years for smaller reporting companies) to determine their source. The SEC defines 'smaller reporting companies' as companies that have less than \$75 million in public float, or if the company cannot calculate public float, less than \$50 million in revenue.

If an issuer determines its products fall under the 'DRC conflict undeterminable' category, no independent private sector audit of the CMR is required, however, the report must still include:

- a listing of the issuer's products that fall under this designation;
- country of origin and a listing of the facilities used to process the minerals, if known;
- the efforts taken to determine the mine or location of origin;
- steps taken or to be taken to improve the due diligence and to mitigate any potential that the conflict minerals are benefiting armed groups.

The 'DRC conflict undeterminable' designation is most likely the result of the extensive lobbying by groups such as the National Association of Manufacturers and the United States Chamber of Commerce so as to allow companies more time to define their supply chain. The 'DRC conflict undeterminable' designation is a major point of contention for some NGOs which believe this is just another method to allow those companies that have been on the fence regarding compliance to further delay their efforts to eliminate 'not DRC conflict free' minerals from their supply chain.

In a departure from the original proposal, excluded from reporting are (1) issuers that mine conflict minerals but do not manufacture anything and (2) conflict minerals 'outside the supply chain' prior to January 31st 2013. This includes conflict minerals that have been smelted prior to this time or stockpiles that have been moved 'outside the supply chain' (that is, are outside of covered countries) by this time. This second exclusion addresses the concerns related to stockpiles of conflict minerals that were mined prior to the final rule and as such grandfathers them under a specific set of conditions; therefore allowing everyone to look forward rather than backward. This exclusion is a point of concern for certain NGOs.

If a company's conflict minerals are determined to be derived from recycled or scrap sources, products containing such minerals are considered 'DRC conflict free'.

Report issuers will be required to provide disclosure on their use of conflict minerals on the new specialized disclosure form, Form SD. Issuers required to provide a CMR will provide this report as an exhibit to the Form SD. The specialized disclosure for the specific calendar year must be provided by May 31st of the following year and filed under the Exchange Act.

In another change from the proposal, a grace period is allowed for an issuer that acquires or obtains control over a company that manufactures or contracts to manufacture products containing conflict minerals that previously had not been obligated to provide a conflict minerals disclosure. In this case, the issuer can delay reporting until the end of the first reporting calendar year that begins no sooner than eight months after the effective date of the acquisition.

The final rule clarifies that an officer of the company need not sign the audit certification as was initially proposed. Instead, this certification takes the form of a statement in the CMR that the issuer obtained an independent private sector audit. The CMR must be posted on the issuer's website for a period of one year.

# CONCLUSION

The final SEC rule requires publically traded companies to disclose information related to their use of conflict minerals on an annual basis. Gathering this information will, in some cases, require considerable effort and time. While at first pass only publically traded companies are affected, it is expected that the requirements for transparency will flow through the entire supply chain.

The final SEC rule can be found here:

http://www.sec.gov/rules/final/2012/34-67716.pdf

The entire Dodd-Frank document can be found here:

http://www.sec.gov/about/laws/wallstreetreform-cpa.pdf

The OECD (2013) Due Diligence Guidance for Responsible Supply Chains of Minerals from Conflict-Affected and High-Risk Areas: Second Edition, OECD Publishing is available at: <u>http://dx.doi.org/10.1787/9789264185050-en</u>. (This reference has been updated since the paper was presented in October 2012).

This overview is not intended to be comprehensive. Issuers should refer to the rule text and the preamble's more complete narrative description for the requirements of the rule.

Some comments in this presentation represent the opinion of the author.

# **18TH PLANSEE SEMINAR**

The 18th Plansee Seminar, an international conference on refractory metals and hard materials, was held on the premises of the Plansee Group in Reutte, Austria from June 3rd to 7th, 2013. It is held every four years; the previous 17th Plansee seminar took place from May 25th to 29th, 2009.

Since its beginnings in 1952, the Plansee Seminar has been regarded as a global forum to exchange ideas and keep up with the latest innovations and market trends in refractory metals and hard materials.

It provides the opportunity to gain insights into all aspects of this exciting class of materials, covering materials science, manufacturing technology and applications as well as to display the contributors' latest achievements in these areas. Practitioners and scientists, both experienced and novice, are cordially invited to participate.

The published programme listed a number of papers that specifically included a reference to niobium and/or tantalum in the title. A list of these papers is provided below, with kind permission from Plansee.

The full technical programme together with all the abstracts can be obtained from the following link: <u>https://www.plansee-seminar.com/?action=PublicSeminarProgram&pagestructid=62</u>

# Proceedings of the conference are planned to be published in September this year and interested parties are invited to consult the Plansee website <u>https://www.plansee-seminar.com/</u> for further details.

#### Thermal Analysis, Transformations and Properties of Pt- and Pd-Alloyed Zr-Nb Alloys

Gasik M.1, Nomura N.2, Kondo R.2, Hanawa T.2

<sup>1</sup>Aalto University School of Chemical Technology, Finland

<sup>2</sup>Tokyo Medical and Dental University, Japan

#### Abstract

Zirconium-niobium alloys are being favored for surgical applications where different temporary fixations should have the least osteointegration. By virtue of MRI analysis, materials should also have minimal magnetic susceptibility to avoid halo formation during examination. Pure Zr-Nb alloys however have complex transformations and insufficient mechanical and corrosion-resistant properties to be used directly for such purposes. In this work alloying of binary Zr-Nb system with by Pt and Pd has been studied. Thermal analysis (DSC and DIL) has indicated several phase transformations in these systems. Mechanical, corrosive and magnetic properties of these alloys have been in parallel investigated and the optimal alloy composition has been developed.

#### **Recent Advances in Ta Thin Film Applications**

<u>Abouaf M.</u><sup>1</sup>, Kumar P.<sup>1</sup> <sup>1</sup>H.C. Starck Inc., USA

#### Abstract

Conventional uses of Tantalum products are based on their chemical, physical and electrical properties. These include capacitors, ballistics, thin film diffusion barriers, and chemical processing equipment. One such application which has seen significant developments in recent years is thin films for the semiconductor industry.

The use of tantalum as a barrier layer under copper interconnects in integrated circuits is well established. H.C. Starck Inc. has worked since 2000 to establish itself as the technology leader in the supply of sputtering target materials for this application. Recent developments in the processing to improve and control the microstructure of Ta plates are reviewed. Modeling tools have proven an effective way to improve the processing routes, and in particular to control the evolution of crystallographic texture along the process steps. Sputtering data on full run targets as well as thin film characteristics are compared for different grades of Ta targets, reinforcing the importance of microstructure control.

#### Co-sputtered Mo-W and Mo-Nb Thin Films

Sun S.<sup>1</sup>, Zhang Q.<sup>1</sup>, Rozak G.<sup>1</sup> <sup>1</sup>H.C. Starck, USA

#### Abstract

Co-sputtered Mo-W and Mo-Nb thin films were created at various compositions and evaluated for their diffusion resistance and barrier functions, as well as the effect of composition on film resistivity and adhesion, corrosion and etching behaviors. The resistivity of MoW and MoNb films is always higher than the resistivity of its matrix metal films. The resistivity maximum with respect to composition is shifted towards the higher resistivity element side from the 50/50 ratio. Film adhesion is good on a-Si coated glass but poor on bare Eagle glass substrate. The environment corrosion behavior was tested at 60°C and 80% humidity for 63 days. MoNb films have better corrosion resistance than MoW films and both are better than pure Mo films. Etching rate and photolithography pattern etching behaviors of MoW and MoNb films are reported. Co-sputtered films were also compared with films sputtered from MoW and MoNb targets and their advantages and shortcomings demonstrated.

#### Characteristics of a Multicomponent and Low-Density Nb-Ti-Al Alloy

Feng W.<sup>1</sup>, Xin Z.<sup>1</sup>, Run B.<sup>1</sup>, Xiaomei C.<sup>1</sup>, Xiaoming Z.<sup>1</sup>, Zhongkui L.<sup>1</sup> <sup>1</sup>Northwest Institute for Non-ferrous Metal Research, China

### Abstract

This article investigated the microstructure and mechanical properties of the multicomponent and low-density Nb-35Ti-6AI-8V-1Zr alloy. The alloy with 6.0 g/cm<sup>3</sup> density are successfully prepared using arc melting and pressworking method, and characterized by optical microscopy (OPM), scanning electron microscope (SEM) and transmission electron microscope (TEM). The specimens were tensile tested in argon atmosphere from room temperature to 1100°C. Experiments results show that the average ultimate tensile strength (UTS) and elongation reach 990 MPa and 16% at room temperature and 80MPa, 44% at 1100°C respectively. This new alloy combines TiC nano-particles dispersion strengthening with solution strengthening to increase the UTS, but retain enough plasticity for hot and cold processing. The investigations prove it be a promising alloy in aerospace field.

#### Structure and Mechanical Properties of Nb-Al System Alloys with Intermetallide Hardening

Karpov M.<sup>1</sup>, Vnukov V.<sup>1</sup>, Korzhov V.<sup>1</sup>, Zheltyakva I.<sup>1</sup>, Prokhorov D.<sup>1</sup>, Gnesin I.<sup>1</sup>, Kiyko V.<sup>1</sup>, Kolobov Y.<sup>2</sup>, Golosov E.<sup>2</sup>, Nekrasov A.<sup>3</sup> <sup>1</sup>Institute of Solid State Physics RAS, Russia

<sup>2</sup>Belgorod State University, Russia

<sup>3</sup>Institute of Experimental Mineralogy RAS, Russia

#### Abstract

Alloys of 91.5wt%Nb-8.5wt%Al composition corresponding to the Nb3Al intermetallide and those of 88 wt%Nb-12 wt%Al composition corresponding to the Nb2Al intermetallide were produced by vacuum melting in suspension and further grinding. 10wt% niobium was added to the intermetallide powders obtained and the mixture was milled in a planetary-type mill. The samples were compacted by two methods. By the first one, the pre-pressed pieces were sintered in a vacuum by equalizing at 1600°C for 0.5h. By the other method, mixture of the composition required was filled into a high-strength graphite mold and sintered in a vacuum at 10.6 MPa and 1700°C for 30min. Irrespective of the compaction method, the samples showed a structure consisting of intermetallide blocks, niobium-based solid solution layers sandwiched between them and pores located inside the intermetallide blocks. The total pore volume in the samples obtained by the first method was approximately 10 times larger than that in the case of sintering by the second method. The strength of the pressure-sintered samples varied from 320 MPa at 1200°C to 135 MPa at 1350°C.

#### Phase Equilibria on the Nb-Cr-B System

<u>Coelho G.C.</u><sup>1</sup>, Rego C.E.<sup>2</sup>, Gigolotti J.C.J.<sup>1</sup>, Silva A.A.A.P.<sup>3</sup>, Nunes C.A.<sup>4</sup>, David N.<sup>5</sup>, Fiorani J.M.<sup>5</sup>, Vilasi M.<sup>5</sup> <sup>1</sup>Universidade de Sao Paulo (USP) and Centro Universitario de Volta Redonda (UniFOA), Brazil <sup>2</sup>Centro Universitario de Volta Redonda (UniFOA), Brazil <sup>3</sup>Universidade de Sao Paulo (USP) and Universite de Lorraine, Insitut Jean Lamour, Brazil

<sup>4</sup>Universidade de Sao Paulo (USP), Brazil

<sup>5</sup>Universite de Lorraine, Insitut Jean Lamour, France

#### Abstract

The engine efficiency is one of the most important aspects in the development of new turbines used for energy generation and in the aerospace industry. The fuel consumption of such machines diminishes with increasing operation temperature. Refractory metal alloys are potential candidate materials to substitute nickel based superalloys in such applications, among which those of niobium and molybdenum stand out. Phase equilibria data is fundamental for understanding microstructural stability and phase transformations that a structural material may undergo when submitted to high temperatures. The present work reports results of experimental investigation of phase equilibrium relations on the Nb-Cr-B ternary system. Samples were prepared by arc-melting mixtures of pure Nb, Cr and B under Ti-gettered argon atmosphere using a water-cooled copper crucible. Some samples were annealed at temperatures between 1200 and 1500 Celsius for time periods ranging from 120 to 600 hours. The microstructures of as-cast and annealed samples were characterized using X-ray diffractometry, Scanning Electron Microscopy and Electron Microanalysis. Partial liquidus projection and 1200 Celsius isothermal section are proposed.

#### Analysis of WC with Increased Ta Doping

Weidow J.1, Schubert W.-D.2, Halwax E.2

<sup>1</sup>Chalmers University of Technology, Sweden

<sup>2</sup>Vienna University of Technology, Austria

#### Abstract

Tungsten and tantalum metal powders were co-carburized to yield a mixture of cubic and hexagonal carbide. The carburization was made through a two-step carburization process with (W,Ta)2C powder as an intermediate product. X-ray diffraction analysis showed that the lattice parameters of the hexagonal phase in the fully carburized powder were larger than those of pure WC indicating the formation of a mixed crystal carbide, (W,Ta)C. The powder with the largest lattice parameters was investigated in detail. A method to produce atom probe tomography specimens of this powder was developed. The atom probe tomography measurement showed the Ta solubility expressed as Ta/(Ta+W) to be as high as 0.086 (i.e. about 8.6 at%). In addition, it was found with electron backscatter diffraction that the (W,Ta)C grains had a large fraction of  $\Sigma 2$  grain boundaries as well as a small fraction of what was suggested as  $\Sigma 4$  grain boundaries.

#### On the Oxidation Behavior and Protection of Niobium Silicide in Situ Composites

Portebois L.1, Knittel S.2, Drawin S.3, Vilasi M.1, <u>Mathieu S.1</u> <sup>1</sup>Universite de Lorraine, France <sup>2</sup>SNECMA, France <sup>3</sup>ONERA, France

#### Abstract

Progresses in the field of gas-turbine engines for aircrafts are controlled by the availability of structural materials able to withstand the higher- temperature hostile environments (high flow gas conditions containing oxygen, water vapor and CMAS, at more than 1150°C). It is one of the objectives of the European project HYSOP to develop the niobium silicide in situ composites solution. The effect of main constitutive elements (Ti, Al or Si) and also of Sn on the microstructure and oxidation resistance was investigated and the oxidation mechanism deduced. None of these alloys presented the required oxidation resistance for their application. Consequently, they can not be used for long term at temperatures around 1200°C without any protective coatings. Thus, a pack-cementation process was developed allowing the deposition of Ti, Fe/Co/Ni, Cr and Si in one step. Oxidation tests were performed under isothermal conditions up to 1300°C and cyclic conditions at 1100°C. Weight gains lower than 8mg.cm-2 were recorded for an exposure of 50h at 1300°C. Coated samples exhibit a high lifetime going up 3000 1h-cycle at 1100°C in air.

#### Tribological Profile of Binderless Niobium Carbide

Mathias W.<sup>1</sup>, Hardy M.<sup>2</sup> <sup>1</sup>BAM Federal Institute for Material Research and Testing, Germany <sup>2</sup>Niobelcon BV, Belgium

#### Abstract

The unlubricated (dry) friction and wear behavior of alumina (99,7%) mated against binderless niobium carbide (NbC) rotating disks under the type of motions of unidirectional sliding (0,03 m/s to 7 m/s) and oscillation (f= 20 Hz, dx= 200 mm, 2/50/98% rel. humidity, n= 105/106 cycles) will be shown. The microstructure and mechanical properties are also presented. Thanks to the tribological database TRIBOCOLLECT of BAM; the obtained tribological data will be benchmarked with different ceramics, cermets and thermally sprayed coatings. The established tribological profile revealed a strong position of NbC under tribological considerations and for closed tribosystems against traditional references, like WC, Cr3C2, (Ti,Mo)(C,N), etc.

#### Microstructure Analysis and Mechanical Properties of NbC-Co/Ni/Cu/Fe3Al Cemented Carbides

Huang S.<sup>1</sup>, Vanmeensel K.<sup>1</sup>, Vleugels J.<sup>1</sup>

<sup>1</sup>Katholieke Universiteit Leuven, Belgium

### Abstract

NbC has a high hardness (19.6 GPa) and melting temperature (3600°C), and is used as a grain growth inhibitor in WC-Co cemented carbides. In the present study, the influence of different binders on the sintering ability, microstructure and mechanical properties of NbC-matrix cemented carbides was investigated. The binders are Co, Ni, Fe3Al or Cu. The powder mixtures were sintered in the solid state by spark plasma sintering and in the liquid state by conventional vacuum sintering. The binders not only affected the densification but also the microstructure and mechanical properties. A limited NbC grain growth was found in the NbC-Cu and NbC-Fe3Al materials, whereas rapid grain growth occurred in the Co and Ni bonded NbC. A detailed microstructural analysis was conducted by electron probe microanalysis. Mechanical properties, such as Vickers hardness and indentation toughness were compared.

#### On the Formation Mechanism of TaC/TaNbC Aggregation in WC-Co Cemented Carbides

<u>Qu J.</u><sup>1</sup>, Xie W.<sup>1</sup>, Huang W.<sup>1</sup>, Chen X.<sup>1</sup>, Wen G.<sup>1</sup>, Wang S.<sup>1</sup>, Xiao C.<sup>2</sup>, Xiong W.<sup>2</sup> <sup>1</sup>Zhuzhou Cemented Carbide Cutting Tools Co., Ltd., China <sup>2</sup>Huazhong University of Science and Technology, China

Abstract

The formation of TaNbC aggregation in WC-Co hardmetal when the content of TaNbC is about less than 0.5wt% can be easily found, resulting in nonuniform distribution of Co phase and aggregation of TaNbC phase in local zone. However, the formation mechanism of TaNbC aggregation is still uncertain. To further investigate the mechanism, temperature sintering experiments of WC-Co-TaNbC alloys (1200-1450°C) were conducted, and the microstructure evolution of TaNbC were exhibited by SEM/EDS. The results indicated that when the sintering temperature is above 1350°C, TaNbC started to be aggregated. With the increase of the sintering temperature, the aggregation area increased obviously. Besides, the results demonstrated that TaNbC aggregation were sensitively dependent on their content. The lowest additional content of TaNbC with disperse distributed in WC-Co alloy are their saturation solid solubility in Co phase. The formation of TaNbC aggregation is related to their nucleation and growths in cooling. Fast cooling can alleviate or even eliminate the TaNbC aggregation.

#### Fabrication of a Nanocrystalline Surface Layer on Bulk Tantalum by Means of a Sliding Friction Treatment

Zhang Y.1, Yu Z.1, Dong F.1, Yuan S.1, Yu S.1, Niu J.1, Ma X.1, Zhang Y.1, Liu C.1, Wen B.1

<sup>1</sup>Northwest Institute for Non-ferrous Metal Research, China

#### Abstract

A novel intense plastic straining process named sliding friction treatment (SFT) to produce a nanocrystalline surface layer on metals is presented. A nanocrystalline surface layer is formed on a coarse-grained pure tantalum sheet by means of SFT. The average size of the top layer nano- grains of the SFT-Ta is about 7 nm and with a range from 3 ~ 13 nm. Nano-grains in surface layer of SF-Cu are completely random orientation with average size about 10 nm. Nano-grains of sample subjected to SFT have a large angle of orientation and non-crystalline structures are found in grain boundary. Deformation twins are also found in nanostructured grains.

#### Densification Behavior of Pure Tantalum Powder by Conventional and Spark Plasma Sintering

Kim Y.1, Lee J.2, Hwang J.2, Hong S.H.2, Kim E.-P.3, Lee S.3

<sup>1</sup>Agency for Defense Development/ Korea Advanced Institute of Science and Technology, South Korea

<sup>2</sup>Korea Advanced Institute of Science and Technology, South Korea

<sup>3</sup>Agency for Defense Development, South Korea

#### Abstract

The densification behavior of pure tantalum powders with coarse (25µm) and fine (2µm) sizes were studied by spark plasma sintering (SPS) and conventional pressureless sintering processes. The SPSed components would reach full density, regardless of particle sizes; however the three types of carbides (Ta2C, Ta4C3 to TaC) are formed. In order to prevent such contamination, cold isostatic pressing (CIP) and high vacuum sintering were performed. The effects of CIPed pressure and sintering temperature on densification of tantalum powders were investigated. As the pressure and temperature increased, the sintered density is also increased; however, full densification would not be successful.

#### Impact of AI on Structure and Mechanical Properties of NbN and TaN

Zehua Z.1, Rachbauer R.1, Holec D.1, Mayrhofer P.2

<sup>1</sup>Montanuniversität Leoben, Austria

<sup>2</sup>Vienna University of Technology, Austria

#### Abstract

Transition metal nitrides (TMN) are widely used as wear-resistant hard coatings for e.g., machining, casting or hot-forming applications. However, TMN's rapidly oxidise and often form porous oxides allowing for rapid scale growth. Therefore, ternary TM(1-x)AlxN coatings are the focus of many research activities as Al-incorporation promotes the formation of dense oxides. But the superior thermal and mechanical properties of TM(1-x)AlxN's are mainly obtained for face-centred-cubic (fcc) structures, requiring the need for detailed information on phase stability ranges. Especially NbNy and TaNy coatings are highly complex due to the variety of crystallographic phases present. Stoichiometric NbN as well as TaN can crystallize in either hexagonal or cubic structure. Through the incorporation of Al, soon the fcc structure is stabilised for x=0.1 0.44(Nb(1- x)AlxN) and 0.1 0.36(Ta(1-x)AlxN). Highest hardnesses are obtained for Nb0.56Al0.44N with ~32GPa and Ta0.64Al0.36N with ~34GPa. Single phase hexagonal Nb(1-x)AlxN and Ta(1-x)Alx coatings are accessible for Al contents above x=0.61 and 0.65, exhibiting hardnesses of ~23 GPa, respectively. The results obtained are in excellent agreement to computational predictions.

#### The Microstructure of Mo-Nb Alloy Single Crystals

<u>Zhongwu H.</u><sup>1</sup>, Zhongkui L.<sup>1</sup>, Tao Y.<sup>1</sup>, Linjiang G.<sup>1</sup>, Jianping Z.<sup>2</sup> <sup>1</sup>Northwest Institute for Non-ferrous Metal Research, China <sup>2</sup>Chinese Institute of Atomic Energy, China

#### Abstract

The microstructure of Mo-Nb alloy single crystals grown by electron beam floating zone melting was studied with the optical microscopy, scanning electron microscopy, electron back scatter diffraction and transmission electron microscopy. The results showed that molybdenum- niobium alloy single crystals were composed of a mass of sub-grains and growth striations. The size of the sub-grains decreased with an increasing of the growth rate of the single crystals. Moreover, the segregation degree of the alloying element niobium increased with an increasing of the growth rate. The increasing in the concentration of Nb also decreased the size of the sub-grains. The dimension of sub-grains changed after high temperature annealing in vacuum.

#### Diffusion of Oxygen in Cell Structure Strengthening Ta-W-Hf Alloy

Xiaomei C.<sup>1</sup>, Xiaoming Z.<sup>1</sup>, Yusheng Z.<sup>1</sup>, Feng W.<sup>1</sup>, Hui W.<sup>1</sup>

<sup>1</sup>Northwest Institute for Non-ferrous Metal Research, China

### Abstract

The diffusion behavior of oxygen in cell structure strengthening Ta-W-Hf alloy fabricated by adapting HIP method was studied. The results show that an oxygen concentration gradient exists within a grain in the sintered sample, which can be reflected by the hardness difference between the grain boundary and grain center. After annealing at 2000°C, oxygen element diffuses completely in the grain, results in the disappearance of the hardness gradient in the grain, and HfO2 disappear accordingly, which may be attributed to the decomposition of the oxide particles under high temperature.

#### Corrosion Properties of Tantalum Sheet Exposed to Air at Elevated Temperatures

<u>Aimone P.</u><sup>1</sup> <sup>1</sup>HC Starck, USA

#### Abstract

The results of a study on the corrosion performance of tantalum sheet exposed to air elevated temperatures is presented. Previous research had shown the color of tantalum oxide can be correlated with the duration and the maximum temperature of the exposure in air. The effects of prolonged exposure on the corrosion properties of the metal is discussed along with possible implications to various CPI applications.

#### P/M Manufacturing of Niobium Silicide Based Materials

Drawin S.<sup>1</sup> <sup>1</sup>ONERA, France Abstract

#### Abstract

Niobium silicide based structural materials are being developed for aeronautical engines because of their high temperature capability and/or their low density in comparison with nickel-base superalloys. Applications can hence be found in a wide range of temperatures, e.g. from 850°C to over 1200°C.

One of the primary objectives of the HYSOP project supported by the European Commission in the frame of the FP7 programme is to develop P/M manufacturing processes for these (ductile) Nb / (brittle) Nb-silicide based materials that could yield near net shaped components, thus avoiding costly shaping and machining steps. The selected routes to be investigated are near net shape hot isostatic pressing, selective laser melting (rapid manufacturing using a powder bed) and powder injection moulding. The aimed components are turbine components like blades and vanes, as well as seal segments. The main achievements at mid-project will be presented. This will range from the manufacture and characterisation of a powder stock with adequate particle sizes, to the manufacture and characterisation, both microstructural and mechanical, of bulk samples with various sizes and shapes.

#### Phase Stability, Thermal Stability and Oxidation Resistance of Arc-Evaporated Ti-Al-Ta-N Coatings

Hollerweger R.1, Arndt M.2, Rachbauer R.2, Polcik P.3, Paulitsch J.4, Mayrhofer P.H.4

<sup>1</sup>Christian Doppler Laboratory for Application Oriented Coating Development at the Institute of Materials Science and Technology, Vienna University of Technology, Austria

<sup>2</sup>OC Oerlikon Balzers AG, Liechtenstein

<sup>3</sup>PLANSEE Composite Materials GmbH, Germany

<sup>4</sup>Institute of Materials Science and Technology, Vienna University of Technology, Austria

### Abstract

Alloying Ti-Al-N with Ta has proven to enhance the hardness, thermal stability and oxidation resistance of sputter deposited coatings. To meet a balance between all these requirements for protecting tools during drilling and cutting applications, an optimized chemical composition of Ti, Ta and Al within the cubic stability range is necessary. However, only limited information is available on arc-evaporated Ti-Al-Ta-N coatings. Therefore, coating developments with an industrial scaled INNOVA Oerlikon Balzers plant, using powder metallurgical (Ti0.50Al0.50)0.95Ta0.05, (Ti0.50Al0.50)0.90Ta0.10, (Ti0.34Al0.66)0.95Ta0.05 and (Ti0.34Al0.66)0.90Ta0.10 targets, were carried out and investigated with respect on their phase stability, mechanical properties as well as thermal stability and oxidation resistance. Vacuum annealing treatments exhibit retarded film decomposition by the addition of Ta. Consequently, the formation of the stable wurzite AlN phase is shifted to higher annealing temperatures of ~1200°C, accompanied by the formation of hexagonal Ta2N. Furthermore, alloying Ta to Ti-Al-N promotes the formation of a dense oxide scale. Therefore, the Ti-Al-Ta-N coating is still intact under a protective oxide, even when treated at 950°C for 20h in ambient air.

#### Thermal Compression Treatment of WC-Co and TiC-VC-NbC-WC-Ni-Cr Hard Alloys

Kramar H.<sup>1</sup>, Bodrova L.<sup>2</sup>, Prokopiv M.<sup>3</sup>, Marynenko S.<sup>2</sup>

<sup>1</sup>Ternopil Ivan Pul'uj National Technical University, Ukraine

<sup>2</sup>Ternopil Ivan Pul'uj Nation, Ukraine

<sup>3</sup>2V. Bakul Institute for Superhard Materials, Ukraine

#### Abstract

Hard alloys on the basis of tungsten and titanium carbides are still the most effective instrumental materials. Their properties can be improved by using new technological approaches - thermal compression treatment (TCT), in particular. The aim of the work was to research TCT influence on microstructure and mechanical properties of WC - 10Co and TiC - 5VC-5NbC-5WC-18(Ni - Cr) hard alloys. The alloys sintered by standard technology were subject to TCT at the temperature  $1370^{\circ}$ C, argon pressure 3,0 MPa during 75 minutes. Decreasing of porosity, increasing of medium size carbide grains in 30% and appearance of separate recrystallized grains of 12...15 µm sizes were noticed for ultrafine WC - Co alloy after TCT. Thus, the transverse rupture strength increases in 20% and fracture toughness decreases in 6,5% comparing with untreated alloys. Considerable increase of carbide grains in TiC based alloys was not available. Unlike the WC based alloys, fracture toughness increased in 11,5%, that is explained by the change of size descriptions of carbide rim and redistribution of carbides and binder metals.

#### Incorporation of Titanium, Tantalum, and Vanadium into the Hexagonal WC Lattice

Pasquazzi A.<sup>1</sup>, Schubert W.D.<sup>1</sup>, Halwax E.<sup>1</sup>, Kremser G.<sup>2</sup>

<sup>1</sup>Vienna University of Technology, Austria

<sup>2</sup>Wolfram Bergbau und Hütten AG, Austria

### Abstract

Based on our earlier work on the formation of a hexagonal (W,Cr)C mixed crystal carbide, the formation of (W,Me)C (Me=Ti, Ta, V, V+Cr) solid solutions was studied in the temperature range of 1450°C to 1950°C. A two step carburization procedure was used for their preparation: In a first step, (W,Me)2C was formed, which in a second carburization step was then transformed into the (W,Me)C carbide.

#### Pore-Boundary Interaction in Sintered Niobium

Zilnyk K.<sup>1</sup>, Leite G.S.<sup>1</sup>, <u>Sandim H.<sup>1</sup></u> <sup>1</sup>EEL - USP, Brazil

### Abstract

High-vacuum sintering is a suitable technique for the obtainment of commercial-purity niobium with uniform fine-grained microstructure. Green compacts were prepared using two distinct compaction pressures (200 and 1500 MPa). High-vacuum sintering was performed in temperatures up to 2000°C to follow the evolution of both porosity and grain size. At sintering temperatures above 1800°C, grain growth becomes noticeable and the pore-boundary interaction plays an important role, in particular for green compacts pressed at 1500 MPa. At this high and unusual compaction pressure, grain growth may be explained by lower green porosity and work-hardening effects. Pore-boundary interaction was investigated in terms of size and type of pores (intergranular and intragranular). Results show that the volume fraction of intragranular pores increases when grain growth occurs during sintering of niobium (pore breakaway).

# MEMBER COMPANY NEWS

We would like to remind you that articles concerning T.I.C. members or the industry in general are posted regularly on the T.I.C. website in the section entitled '<u>News</u>'.

# **CHANGES IN MEMBER CONTACT DETAILS**

#### Duoluoshan Sapphire Rare Metal Co

The e-mail address of Duoluoshan Sapphire Rare Metal Co has been changed to dlsnsy@aliyun.com.

#### KEMET Electronics

The e-mail address of Dr Daniel Persico, delegate to the T.I.C. for KEMET Electronics and President of the T.I.C., has been changed to <u>danielpersico@rc.jp.nec.com</u>.

#### Zimmer – Trabecular Metal Technology

Mr Steven Seelman has been designated as new delegate to the T.I.C. for the company Zimmer – Trabecular Metal Technology. He can be reached on <u>steve seelman@zimmer.com</u>.

# **EXECUTIVE COMMITTEE**

According to the Charter of the T.I.C., the Executive Committee must consist of between two and eleven people, plus the President. 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 composition is approved by the T.I.C. members at each General Assembly, and it currently consists of (in alphabetical order):

- John Crawley.....h<u>jcrawley@rmmc.com.hk</u>
- José Isildo de Vargas.....<u>isildo@cbmm.com.br</u>
- Alan Ewart ......
- Alexander Gagarin ......<u>ulba.gagarin@gmail.com</u>
- David Henderson.....<u>dhenderson@rittenhouseir.com</u>
- Jiang Bin.....<u>jiangb\_nniec@otic.com.cn</u>
- Ian Margerison .....ian.margerison@metalysis.com
- William Millman ......<u>william.millman@eur.avx.com</u>
- Yasukazu Muto.....<u>yasukazu.muto@hcstarck.com</u>
- Hiroya Nishimoto......<u>nishimoto.hiroya@jp.panasonic.com</u>
- Daniel Persico (President) ......<u>danielpersico@rc.jp.nec.com</u>
- Itamar Resende ......