

T I C

TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

PRESIDENT'S LETTER

Dear friends and members of the T.I.C.,

This past April, the T.I.C. Executive Committee met in Brussels, along with our Supply Chain Officer Richard Burt, the Secretary General Emma Wickens, and the Technical Promotion Officer Ulric Schwela. Among other subjects, we discussed the plans for the Fifty-third General Assembly to be held in Cape Town, South Africa from October 7th to 10th 2012. Those plans are well underway and this meeting promises to be very engaging and informative, particularly as it will take the form of a Symposium, with an extended technical programme which is presented in this Bulletin.

For this first-ever T.I.C. event in Africa, we will not be the guests of a producer's or processor's plant, but we will be seeing a different type of plant, as we will take an interesting trip to the South African wine lands in the afternoon of October 10th.

We warmly encourage all of you to take the opportunity of coming to Cape Town. If you need any information on how to proceed with your visa applications and bookings do not hesitate to contact our Secretary General Emma Wickens. Complete details and invitations will be sent to all members around mid-July.

Furthermore, this release of the quarterly Bulletin marks a special event as it is the 150th issue. We take this opportunity to look back over the history of the association, as you will see in the following pages...

We hope you enjoy reading this issue and look forward to seeing you all in Cape Town in October.

José Isildo de Vargas
President

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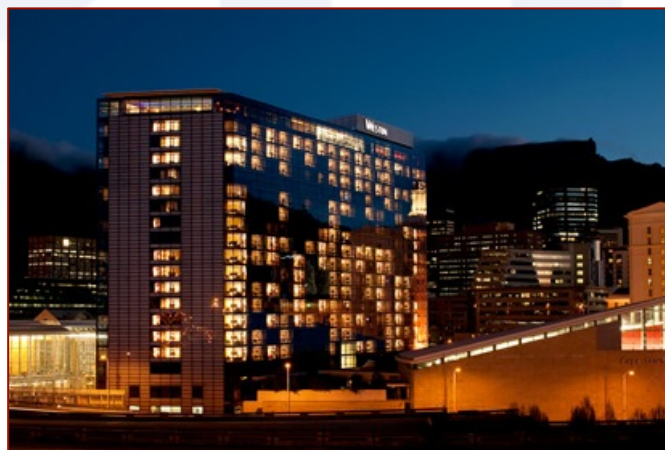
FIFTY-THIRD GENERAL ASSEMBLY & SYMPOSIUM 2012 ON TANTALUM AND NIOBIUM

The Fifty-third General Assembly and Symposium 2012 on Tantalum and Niobium will be held in Cape Town, South Africa, from October 7th to 10th 2012. Technical and social events will be held in the Cape Town International Convention Centre (CTICC) while a block booking of bedrooms has been secured at the Westin Hotel just across the street.

On Sunday October 7th, 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 at the CTICC from 6p.m. to 8p.m.

The formal General Assembly of the association will be held on Monday October 8th and will be followed by technical presentations until around 4p.m., with a break for a buffet lunch.

Companies wishing to apply for membership at this General Assembly are reminded that their completed application forms should be returned to the T.I.C. by September 8th at the very latest. For any further information on becoming a member, please refer to the following page of our website: <http://tanb.org/applmemb>



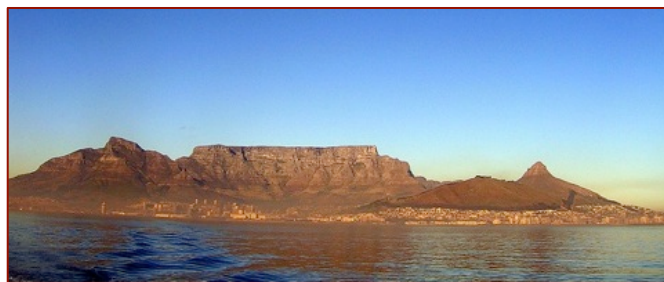
The Westin Hotel and the Cape Town International Convention Centre

© Westin

On Monday evening, all participants are invited to take part in a sunset cruise, which will offer stunning views of Cape Town, seen from the water. Transfer by bus to the harbour will take place at 5.30p.m. Drinks and canapés will be served on the boat and the activity will end by 7.30p.m. to enable guests to make their own plans for the evening.

A second technical session will be held on Tuesday October 9th, breaking for a buffet lunch and ending around 4p.m.

On Tuesday evening, all participants are invited to a Gala Dinner to be held in the ballroom of the Convention Centre.



View of Cape Town and Table Mountain from the sea

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View of Constantia Valley

© mikkelz @ Flickr

The third part of the extended technical programme will take place on the morning of Wednesday October 10th, ending around 11a.m.

The full technical programme is published herebelow.

On Wednesday afternoon, all delegates and accompanying persons are invited to take part in a trip to the nearby Constantia wine lands, including a cellar tour, wine tasting and lunch.

Sightseeing tours for accompanying persons are also being arranged for Monday and Tuesday. On one day, participants will discover the majestic Table Mountain and the famous Kirstenbosch Botanical Garden. The other day will take participants a little further afield, along the spectacular coastline of the Cape Peninsula, through quaint and charming historical villages, to visit the mythical meeting place of the two great oceans.

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

FIFTY-THIRD GENERAL ASSEMBLY: 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).

Niobium sheet for the synthetic diamond industry

by Paul Aimone, H.C. Starck, Inc.

Production of niobium and tantalum products from tin slag

by Luis Anderson⁽¹⁾, Maria de Campos⁽¹⁾, Brian Wiggins⁽¹⁾, Lucio Pareja⁽²⁾ and Julio Quiroz⁽³⁾
(1) Hatch Ltd, (2) Minsur, (3) Mineração Taboca

Comparison of pyrometallurgical processes for the production of ferroniobium

by Anthony Buragina, Maria de Campos and Brett van Groenewoud, Hatch Ltd

The eternal struggle in mining – mineralogy, metallurgy and money

by Richard Burt, GraviTa Inc.

The tantalum industry - how did it get to here, and where is it going?

by Richard Burt and Ulric Schwela, T.I.C.
(both presenting)

The conflict-free supply chain - an update on T.I.C. activities

by Richard Burt, T.I.C.

Niobium - market, capacity availability, latest trends and developments

by José Isildo de Vargas and Marcos Stuart, CBMM

Scrap – a critical part of the tantalum supply chain

by David Gussack and Mark Gussack, Exotech

Mineral traceability in the Great Lakes region: an update on iTSCI from the field

by Karen Hayes, Pact

Pitfalls and issues associated with proving transport exemption in low-level NORM materials

by Paul Hinrichsen, National Nuclear Regulator (NNR)

Pre-polymerized conductive polymer dispersions: capability and product overview

by Philip Lessner, Randy Hahn, Jayson Young and Yuri Freeman, KEMET Electronics Corporation

Solid tantalum capacitors with low (no) de-rating

by Philip Lessner and Yuri Freeman, KEMET Electronics Corporation

Processing technology research into large size niobium coating of target material

by Li Zhaobo, Zhang Guojun, Ren Xiao, Ningxia Orient Tantalum Industry Co. Ltd
(presented by Jiang Bin and Zheng Aiguo)

Responsible mineral supply chains from conflict-affected and high risk areas - the 'closed pipe' model

by William Millman, AVX

Due diligence regulation and implementation

by Gregory Mthembu-Salter, Phuzumoya Consulting

The Advanced Metals Initiative of South Africa

by Johann Nel, South African Nuclear Energy Corporation (Necsa)

Addressing delays and denials of radioactive shipments in Africa

by Mamdouh Yassin Osman, Sudan Atomic Energy Commission (SAEC)

Conflict-free and socially sustainable: a comprehensive vertically integrated tantalum supply chain

by Daniel Persico, KEMET Electronics Corporation

Next generation of high voltage, low ESR tantalum conductive polymer capacitors

by J. Petržílek, T. Zedníček, M. Uher, I. Horáček, S. Zedníček, M. Bárta, AVX
(presented by William Millman)

The Maboumine project

by Xavier Revest and Antoine Greco, Eramet

Statistics: the why, the how and the what

by Ulric Schwela, T.I.C.

Transport problems and solutions

by Ulric Schwela, T.I.C.

A historical analysis of T.I.C. membership

by Ulric Schwela, T.I.C.

Tantalum polymer capacitors that support leading-edge consumer equipment

by Yasushi Takeda, SANYO Electric Co., Ltd
(presented by Tak Ohashi)

Understanding tin slag: an important source of tantalum and niobium

by Ricardo Torrente and Egberto Silva, White Solder Group

THE QUARTERLY BULLETIN CELEBRATES ITS 150TH ISSUE – FLASHBACK OVER THIRTY-EIGHT YEARS OF EXISTENCE OF THE T.I.C.

This article is an updated version of the one first published in the souvenir booklet '1974-2009: thirty-five years of existence, fifty General Assemblies'. It has been written by Emma Wickens, based on information published in the quarterly Bulletins of the T.I.C. and on discussions held with William Millman and Axel Hoppe.

During a business discussion in 1973, Mr Paul Leynen of Geomines in Brussels, Mr Herman Becker-Fluegel of National Resources Trading in New York and Dr Cornelius Herkströter of Billiton Handelsgesellschaft in Lucerne found that they shared concerns about the lack of useful information on tantalum source materials. Following this conversation, the concept of an international organisation of tantalum raw material producers was born. The purpose of the association would be to spread information about tantalum and to promote the common interest and welfare of the producers, especially with public and private authorities, organisations and agencies. They gathered together several representatives of producers to work out the principles and charter of such an organisation.

From their effort, the Tantalum Producers International Study Center was chartered as a non-profit Association under Belgian law on October 24th 1974, with the main objectives 'to promote and further cooperation between members on matters of research in and studies of all aspects of the tantalum source materials industry and of collecting, disseminating and maintaining statistics and information on any matters affecting that industry'. By the time of the Third General Assembly in Brussels in March 1975, there were twelve members, all raw material producers, representing mining and smelting operations on five continents.

These founder members were: Cobelmin-Zaire, Companhia de Estanho São João Del Rei, Datuk Keramat Smelting, Greenbushes Tin, Hochmetals Africa, Cia. Industrial Fluminense, Makeri Smelting Company, Sociedade Mineira de Marropino, Symetain, Tantalum Mining Corporation of Canada, Thailand Smelting & Refining Company and Zairetain. With 'estanho' meaning 'tin' in Portuguese and 'étain' meaning 'tin' in French, the strong connection to the tin industry was evident.

Not long thereafter, the General Assembly in September 1975 amended the by-laws of the T.I.C. to include in the membership 'companies or manufacturers engaged in treating tantalum or columbium-tantalite raw materials, industrial consumers of the products supplied by the former (producers and processors) and commercial intermediaries who provide technical or financial assistance to mining operators of these primary materials, who are not members of the Association'. As a result, the membership grew rapidly, reaching 33 companies by the end of the fifth year and 77 companies in June 1985. These member companies represented all facets of the tantalum industry including source material producers, processors, a major portion of the end-product producers and the principal international companies engaged in the trading of tantalite. In June 1984, at the Twenty-first General Assembly, the name was changed to Tantalum International Study Center, to reflect the wider membership interests of the expanding organisation.

Around 1983, producers and processors of niobium started joining the association. In May 1986, at the Twenty-fifth General Assembly, the Charter was changed to extend the same membership rights to niobium producers, processors and consumers as

companies involved in tantalum-related activities. The name of the association was changed to Tantalum-Niobium International Study Center.

In 1994, twenty years after its foundation, the association was encountering some financial difficulties. Consolidation and integration, combined with several years of difficult economic circumstances for non-ferrous metals in general, had brought the number of members down to 50. That year, Cabot, H.C. Starck, Gwalia and Thai Tantalum donated 40000 US Dollars to the T.I.C. 'Prom Fund' to finance the promotion of tantalum and niobium. Associate membership was also introduced, in an effort to widen the membership base. Finally, the Third International Symposium was organised in Goslar, Germany in 1995. Its enormous success helped to improve the financial situation of the association.

By 1997, business was looking up, with the 'age of internet' leading to huge investments world-wide. The T.I.C. followed suit: in June 1998, the association had its own e-mail address and in March 1999, the website was born.

In 2000, all five major applications of tantalum reached a peak in demand simultaneously:

- gas turbines for power generation
- cutting tools containing carbides
- automotive applications
- corrosion-resistant tanks and other applications in the chemical industry
- mobile phones and high-speed internet devices in the electronics industry

Demand was high but speculation made the market look even bigger than it was, and prices escalated. This context of high demand also brought to light the issue of 'conflict coltan' from central Africa.

Shortly afterwards, the 'bubble' burst. Reacting to the increased prices and apparent shortage of supply, the electronics industry designed out tantalum capacitors wherever possible.

To date, the tantalum industry is still reconsolidating its position. The challenge is now to look for new opportunities, to increase demand.

In recent years, the tantalum industry has also been faced with several other challenges, related to the transport of raw materials, the enforcement of the REACH regulations and the on-going issues related to sourcing of minerals from the 'conflict regions' of central Africa.

In 2003, a Working Group on the transport of raw materials with naturally occurring low levels of radioactivity was formed under the aegis of the T.I.C. In 2005, a consultant, Dr Doug Chambers of SENES, was employed by the T.I.C. to write a risk assessment report for submission to the IAEA, with the goal of changing the current regulations for the transport of tantalum raw materials.

In January 2009, the T.I.C. established a Working Group on Tantalum and Niobium Mining. Its objectives were to promote the tantalum and niobium industries, to explore the issues related to the mining of their minerals on a world-wide scale and to help to improve the standards of artisanal and small-scale mining (ASM) operations.

The Group developed a T.I.C. policy of due diligence in the form of transparency and traceability of raw materials along the supply chain. The Artisanal and Small Scale Mining Policy was approved by the membership of the association during the Fiftieth General Assembly held in Tallinn, Estonia, in October 2009.

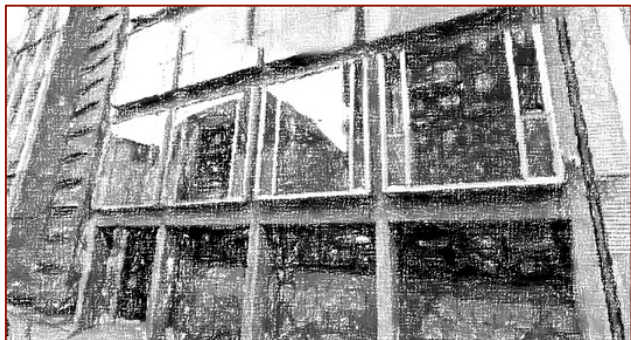
The T.I.C. then joined forces with the International Tin Research Institute (ITRI) in creating a transparent and traceable supply chain, providing seed funding in 2010 for the pilot implementation of iTSCi (ITRI Tin Supply Chain Initiative), encouraging T.I.C. members to join the scheme and representing the tantalum industry on the Steering Committee of the project.



The T.I.C. is actively involved in other initiatives that have arisen from the issue of conflict minerals, such as helping draft the Due Diligence Guidelines issued by the Organisation for Economic Cooperation and Development (OECD), which have subsequently become the common reference point for the recommendations by the United Nations Group of Experts, for the Mineral Tracking & Certification Scheme being developed by the International Conference on the Great Lakes Region (ICGLR) and, it is hoped, for the future regulations to be issued by the Securities Exchange Commission (SEC) in the USA to implement Section 1502 of the Dodd-Frank Act. The T.I.C. is also represented on the Interim Governance Group that has been established to oversee the on-going implementation of the OECD Due Diligence Guidelines, and continues to work together with the Electronic Industry Citizenship Coalition (EICC), Global e-Sustainability Initiative (GeSI) and Public-Private Alliance for Responsible Minerals Trade (PPA).

Mr Richard Burt has been following very closely all these issues, first during his two years as President of the T.I.C. and then, since October 2011, in the position of Supply Chain Officer.

During these same recent years the niobium industry has created its own market by promoting the use of ferro-niobium in steel. This is by far the major application. Other important applications include powder for capacitors, pure metal and alloys for superconducting applications, alloys for turbine blades... The niobium industry is characterised by a long-term perspective, with stable pricing and stable supply.



40 Rue Washington, 1050 Brussels

The first Secretary General of the T.I.C. was Mrs Jan Goodyear. She left in 1977, when she moved abroad. The affairs of the T.I.C. were managed by Mrs Judy Wickens for the following 30 years, until her retirement. Since 2007, Ms Emma Wickens has taken over the position.

In 1985, the position of Technical Promotion Officer was created. It has been occupied successively by:

Mr Andrew Jones from 1985 to 1989
 Mr Rod Tolley from 1989 to 1993
 Dr George Korinek from 1993 to 1998
 Mr C. Edward (Ed) Mosheim from 1999 to 2003
 Mr William Serjak from 2003 to 2005
 Mr Ulric Schwela since 2005

The T.I.C. 'Bulletin' has been published quarterly since February 1975 starting as a simple four-page issue which gradually expanded into a regular eight-page publication. The first 71 issues (until September 1992) were in black and white, then colour was introduced. After 146 issues published in hard copy, the Bulletin embraced the electronic era in September 2011. There have been 150 issues to date. In addition to providing in print some of the presentations made at the T.I.C. meetings and reporting on the activities of the T.I.C., articles have been published covering many facets of the tantalum and niobium businesses from mining to the use in end-products, as well as periodic analyses of the supply and demand conditions of the market.

The Bulletin was edited by Mr Graham Brown from 1975 to 1985. After that date, the Technical Promotion Officers took over the role. In recent years, the Bulletin has been edited by the Secretary General, with the help of the Technical Promotion Officer for the layout since the Bulletin has become electronic.

The statistics collected and disseminated from the very beginning of the association's existence also evolved over time, with categories changing to reflect the informational needs of the industry. In 2006 the collection of statistics also went electronic and in 2009 the reporting frequency for all statistics changed back from bi-annually to quarterly.

At first, two General Assemblies were held in each year. The first six took place in Brussels, where precious help with the organisation was provided by Mr Paul Leynen. Then, in 1977, a meeting was held in Winnipeg, Canada, where the T.I.C. was invited by Mr Herman Becker-Fluegel to tour the mine of the Tantalum Mining Corporation of Canada (Tanco) at Bernic Lake. Thereafter until 1987 there continued to be two Assemblies in each year, one in Brussels and one in another part of the world so that a visit to a mine or plant could be included in the conference programme, alongside the administrative business and the presentation of technical and scientific papers by distinguished and knowledgeable speakers.

In 1987, the Charter was modified to allow the association to choose to hold only one General Assembly per calendar year, while also permitting a greater number of meetings if these were required. Since then, one Assembly has been held each year, but the tradition of seeing an appropriate facility is maintained and the venue has moved around the world, thanks to our many hosts!

In total, over 37 years, 52 General Assemblies have taken place. Attendance at these meetings has grown from a mere 46 at Tanco in 1977 to around or even over 200 in recent years, demonstrating the rapid development of the T.I.C.

Five of the General Assemblies have been held in conjunction with an International Symposium, a longer event with an increased number of technical papers. The sixth International Symposium is scheduled for October 2012 in Cape Town, South Africa, marking the first-ever meeting in Africa.

The association is run by its Executive Committee. This Committee has always reflected the range of activities of the members, and it covers the geographic spread of the membership, too. Presidents have been drawn from all sectors of the industry and from many parts of the world. The term of office is normally one year.

Since its inception, the Tantalum-Niobium International Study Center has grown and developed to encompass the changing nature of the tantalum and niobium industries and will continue in the same spirit in facing future challenges.

The business of the T.I.C. has been carried out by the Secretariat located in or near Brussels, first in a small attic sub-let from a larger organisation at 1, Rue aux Laines, 1000 Brussels, then, from 1983 until 2008, in its own office in the International Association Centre, 40 Rue Washington, 1050 Brussels, then, since 2008, at Chaussée de Louvain 490, 1380 Lasne.



Chaussée de Louvain 490, 1380 Lasne

PRESIDENTS OF THE ASSOCIATION

| | |
|------|--|
| 1975 | Cornelius Herkströter, Thaisarco |
| 1976 | Herman Becker-Fluegel, National Resources Trading / Tanco |
| 1977 | Paul Leynen, Zairetain |
| 1978 | Reinhard Deil, GfE |
| 1979 | Joseph Abeles, Kawecki Berylco Industries |
| 1980 | Brian Reynolds, Thaisarco |
| 1981 | George Korinek, Hermann C. Starck Inc. |
| 1982 | Conrad Brown, Fansteel |
| 1983 | John Linden, Greenbushes Tin |
| 1984 | Robert Franklin, STC Components |
| 1985 | Carroll Killen, Sprague Electric Company |
| 1986 | Chikara Hayashi, Vacuum Metallurgical Company |
| 1987 | Rod Tolley, Datuk Keramat Smelting |
| 1988 | Hans-Jürgen Heinrich, GfE |
| 1989 | Harry Stuart, Niobium Products Company |
| 1990 | George Korinek, NRC |
| 1991 | Peter Adams, Thaisarco |
| 1992 | Yoichiro Takekuro, Vacuum Metallurgical Company |
| 1993 | Peter Maden, Vishay Sprague |
| 1994 | Hubert Hutton, Sogem-Afrimet |
| 1995 | Peter Köhlert, H.C. Starck GmbH |
| 1996 | Robert Barron, Cabot Performance Materials, David Maguire, Kemet |
| 1997 | Yeap Soon Sit, S.A. Minerals |
| 1998 | William Millman, AVX |
| 1999 | John Linden, Sons of Gwalia |
| 2000 | Charles Culbertson II, Kemet |
| 2001 | Thomas Odle, Cabot Performance Materials |
| 2002 | Axel Hoppe, H.C. Starck GmbH |
| 2003 | Josef Gerblinger, Epcos |
| 2004 | David Reynolds, Kemet |
| 2005 | William Millman, AVX |
| 2006 | William Millman, AVX |
| 2007 | Axel Hoppe, H.C. Starck GmbH |
| 2008 | William Young, Cabot |
| 2009 | José Isildo de Vargas, CBMM |
| 2010 | Richard Burt, GraviTa Inc |
| 2011 | Richard Burt, GraviTa Inc |
| 2012 | José Isildo de Vargas, CBMM |

RESEARCH ON PROCESSING LARGE GRAIN NIOBIUM SHEET USED IN SUPERCONDUCTING ACCELERATING CAVITIES

This paper was written by Xie Weiping, Chen Lin, Li Mingyang, He Jilin and Li Bin from Ningxia Orient Tantalum Industry Co., Ltd. It was presented by Guo Hong on October 17th 2011, as part of the Fifty-second General Assembly held in Almaty, Kazakhstan.

This article studies the processing of large grain niobium sheet used in producing superconducting RF cavities. The studied production process for large grain niobium sheet is simple and easy to control, does not stain the niobium material and so makes it easier to meet the technical requirements of the superconducting RF cavity. The results show that the RRR value and physical properties of the large grain niobium sheet are similar to ingot and meet the technical requirements of the superconducting RF cavity, because the production process does not need forging, annealing, and rolling processes that can increase interstitial element (C, N, H, O) contents and would reduce the RRR value of the niobium material.

1. FOREWORD

Superconducting RF cavities are now basically made with pure niobium or niobium film. For more than 20 years, international research has been using niobium plate of a uniform grain size of more than 5 to manufacture superconducting cavities, thus setting a standard. With the progress of science and technology, a set of higher demands has been introduced for RF superconducting accelerators, such as higher acceleration gradients and lower energy loss. Some research laboratories are actively producing large-grain niobium superconducting cavities, such as DESY, JLab, Cornell, KEK^[1], Heavy Particle Physics Institute in Beijing University, and the High Energy Physics Institute of the Chinese Academy of Sciences. At present, the large grain niobium sheet can meet the requirements of the current manufacture of superconducting cavities. To simplify the production process, the pollution of material needs to be eliminated from the large grain niobium sheet preparation process, such as during forging and rolling. At the same time, we removed the electrochemical polishing step which is an expensive part of the superconducting cavity processing technology, as the acceleration gradient of the superconducting cavity can still meet the requirements. The researchers tested the RF superconducting cavity which can meet the grain size of more than 5, and encountered some non-normal loss such as high acceleration gradient Q-slope phenomenon; most explanations for these

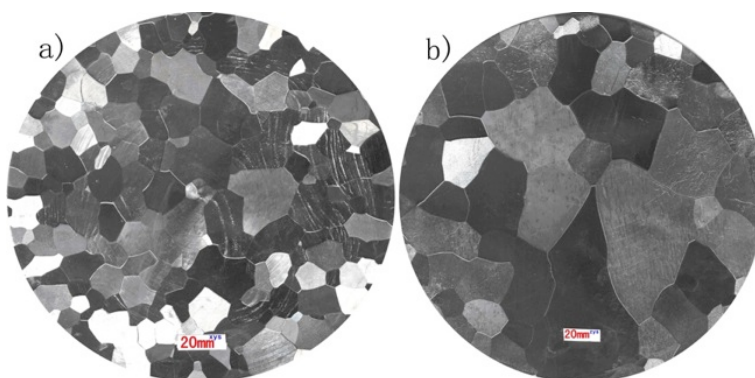
phenomena are related to the niobium grain interface. Large grain superconducting cavity research has been an international hotspot in recent years, using only standard BCP. The smelting process of niobium plate can also be simplified^[2]. The International Conference of SRF2007 held in Beijing specifically discussed the production of large grain superconducting niobium cavities. The single cell superconducting cavity of Cornell University could achieve acceleration gradients of up to 59 MV/m in March 2007.

Hellas in Germany, CBMM in Brazil, Huachang Company in the U.S.A. and Ningxia Orient Tantalum Industry carried out research into large grain niobium sheet with the support of the TTC (TESLA Technology Collaboration). This paper describes the development process of large grain niobium sheet, product performance and superconducting cavity test results at Ningxia Orient Tantalum Industry.

2. TEST PROCESS

The processing technology of TESLA superconducting cavities can be summarised as: niobium raw materials → multiple electron beam melting → high-purity niobium ingot (RRR300) → forging → annealing → rolling → surface treatment → pickling → annealing → analysis and detection → high-purity niobium sheet of superconducting cavity (RRR300). It is a long process. Niobium ingot is usually produced in an electron beam furnace. The macro-grain size is shown in Figure 1 a), with a large number of small sized grains distributed over the entire plane, suitable for processing plates, rods, tubes and other products, with RRR values between 40 and 50 which can not meet the requirements of RF superconducting cavities.

Currently a large number of high-purity niobium ingots (with RRR values of 300 or more) are used for the RF superconducting cavities. The grain size is shown in Figure 1 b), with larger sized grains compared to ordinary niobium ingot, however, significantly the same section has more grains and can not meet the requirements of large grain niobium material, i.e.: fewer grains and fewer grain boundaries. Consequently we optimised the electron beam furnace melting process parameters based on the production of high-purity niobium ingot, including the number of melts, melting rate and the electron beam energy distribution.



**Figure 1: Photos of grain size for
a) commercial grade Nb ingot; b) high purity Nb ingot**

2.1. PREPARATION PROCESS FOR HIGH-PURITY LARGE GRAIN NIOBIUM INGOT (RRR300)

Using Nb₂O₅ as raw material, produced by a re-crystallization process giving few high-melting-point metal impurities, Nb-Al alloy was produced by aluminothermic reduction. The RRR value of large grain niobium ingots of high purity can reach 300 or more after multiple electron beam furnace melts.

The melting process of the large grain niobium sheet is based on the production of high purity niobium ingot. The focus is on improving some electron beam furnace parameters such as melting power, melting rate, melting vacuum, electron beam running track and cooling intensity, and where determined appropriate, new smelting process parameters. The electron beam furnace produced superconducting large grain high purity niobium ingot (Ø 300 mm, RRR300), with chemical composition fully meeting the DESY standards, and grain structure meeting the processing requirements of the RF superconducting cavities.

2.2. THE PRODUCTION OF LARGE GRAIN NIOBIUM SHEET

After the process of sawing, mechanical polishing and chemical polishing, the large grain niobium ingot was turned into high-purity niobium sheet which was used to prepare the RF superconducting cavities.

2.2.1. MECHANICAL PROCESSING OF HIGH-PURITY LARGE GRAIN NIOBIUM SHEET

The processing of large grain niobium sheet should be controlled strictly to avoid influencing the RRR. The correct cutting size, cutting speed and processing methods should be determined, and the niobium sheet should be refrigerated effectively to reduce its oxygen content and RRR value. Currently, multi-wire saw technology is the research hotspot for large grain niobium sheet.



Figure 2: Large grain niobium sheet multi-wire saw technology

2.2.2. ACID PICKLING OF HIGH-PURITY LARGE GRAIN NIOBIUM SHEET

Acid pickling could determine the final performance of the niobium sheet. The composition of the acid solution and pickling time were very important in this process. The correct proportion of hydrofluoric acid, nitric acid and phosphoric acid, in addition with accurate pickling time, could reduce the hydrogen content in the sheet and lead to superior superconductivity.

3. RESULTS

3.1. ANALYSIS OF RESULTS OF HIGH-PURITY LARGE GRAIN NIOBIUM INGOT

The chemical composition and crystal phase of high-purity large grain niobium ingot are shown in Table 1 and Figure 3, respectively. The RRR sampling and RRR value are shown in Figure 4 and Table 2.

Table 1: Composition of high purity Nb ingot

| Impurities (ppm) | | | | | | | |
|------------------|---------------|-----------|-----------|----------|---------------|-----------|-----------|
| Elements | DESY Standard | Results 1 | Results 2 | Elements | DESY Standard | Results 1 | Results 2 |
| Ta | ≤500 | 100 | <100 | Si | ≤30 | <10 | <10 |
| W | ≤70 | 28 | 10 | H | ≤2 | 2 | 2 |
| Ti | ≤50 | <5 | <5 | N | ≤10 | 8 | 10 |
| Fe | ≤30 | <5 | 5 | O | ≤10 | 7 | 5 |
| Mo | ≤50 | 10 | 10 | C | ≤10 | 6 | 8 |

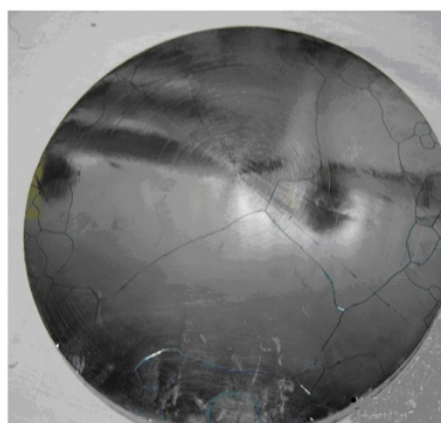


Figure 3: Photo of large grain in Nb ingot

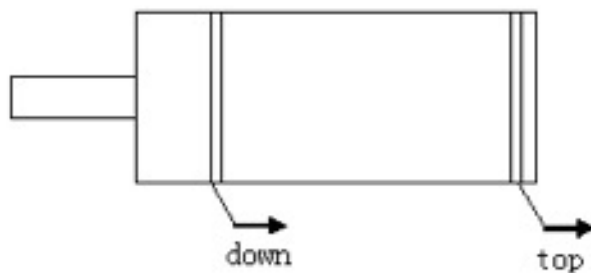


Figure 4: Sampling position of large grain ingot for RRR values and mechanical properties

Table 2: RRR values of ingots

| | DESY standard | Ingot 1 | | Ingot 2 | |
|-----|---------------|---------|------|---------|------|
| | | top | down | top | down |
| RRR | ≥300 | 442 | 478 | 414 | 418 |

3.2. THE PERFORMANCE OF HIGH-PURITY LARGE GRAIN NIOBIUM SHEET

The RRR value and performance of large grain niobium sheet are shown in Table 3.

Table 3: Performance test results of large grain Nb sheet

| | Tensile strength (MPa) | Yield strength (MPa) | Elongation (%) | HV (10N) | RRR |
|------------|------------------------|----------------------|----------------|------------------|-----|
| Sample 1-1 | 90.8, 89.5 | 50.6 | 80.4 | 57.6, 56.4, 55.6 | 418 |
| Sample 1-2 | 97.7 | 52.7 | 53.2 | 59.2, 57.8, 58.6 | 460 |
| Sample 2-1 | 89.2 | 50.2 | 46.0 | 48.2, 49.2, 49.5 | 370 |
| Sample 2-2 | 86.7, 88.6 | 76.9, 78.1 | 59.6, 62.4 | 53.2, 52.1, 51.5 | 426 |

We can see from Table 2 and Table 3 that RRR values of the niobium sheet are approximately equal to those of the niobium ingot (with an error range of ±5%). The large grain niobium sheet with good mechanical properties can completely meet the requirements for the preparation of superconducting cavities.

3.3. THE APPLICATION OF HIGH-PURITY LARGE GRAIN NIOBIUM SHEET

The high-purity large grain niobium sheet was used to prepare single cell superconducting cavities at JLab in America and met the design requirements (shown in Figure 5). Meanwhile, the large grain niobium sheet made by Ningxia Orient Tantalum Industry had been used in the experiments on superconducting cavity preparation which were carried out at the Institute of Heavy-ion Physics of Beijing University (shown in Figure 6), the High Energy Physics Institute of the Chinese Academy of Sciences, Cornell University and DESY in Germany, and all obtained good results.

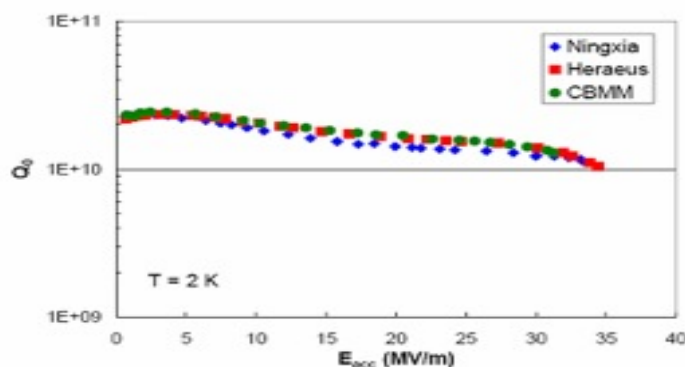


Figure 5: Performance testing by JLab of large grain superconducting Nb sheet produced by different manufacturers

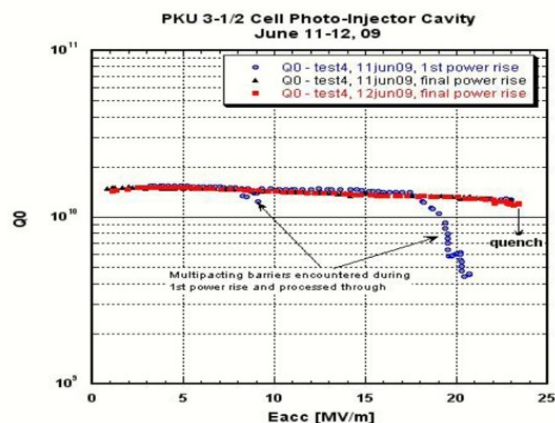


Figure 6: Large grain niobium sheet made by OTIC used to produce superconducting cavities at Beijing University and its performance

4. CONCLUSION

Compared with the processing for traditional superconducting niobium sheet, the new large grain niobium sheet does not reduce RRR value, does not increase the content of interstitial elements (C, N, O and H) and the processes of hammer cogging, annealing and rolling are no longer necessary. The RRR values of the niobium sheet and niobium ingot are basically identical.

The high-purity large grain niobium sheet can meet the requirements for the preparation of RF superconducting cavities.

REFERENCES

- [1] Singer W., Brinkmann A., Iversen J. *et al.*: Large Grain Superconducting RF Cavities at DESY, in Proceedings of LINAC 2006, Knoxville, Tennessee, USA, p. 327.
- [2] Hao Jiankui, Zhao Kui, Zhang Bao-Cheng *et al.*: Researches on Large Grain SC Cavities at Peking University, Chinese Physics C.V01. 32-2008, p. 181-183.

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 '[News](#)'.

CHANGES IN MEMBER CONTACT DETAILS

Advanced Material Japan Corporation

The offices of Advanced Material Japan Corporation have moved.

New contact details are:

Sanno Park Tower 12F, 2-11-1 Nagata-cho, Chiyoda-ku, Tokyo, 100-6112, Japan.

Tel.: +81 3 3507 2304

Fax: +81 3 3507 2302

Firadec

The delegate to the T.I.C. for Firadec, Mr Claude Boudet, has changed e-mail address. His new address is:

claude.boudet@exxelia.com

Refractory Metals Mining Co Ltd

The company Refractory Metals Mining Co Ltd has nominated a new delegate to the T.I.C.: Mr John Crawley. His e-mail address

is: jcrawley@mmc.com.hk

www.tanb.org
e-mail to
info@tanb.org