PRESIDENT'S LETTER

Dear Members and Friends,

By the time you receive this issue of the Bulletin, I hope that you have already made your reservations and travel arrangements for the Thirty-eighth General Assembly of T.I.C. in Xian, China.

Further on in this Bulletin, you will be informed about the meeting arrangements and technical programme in detail.

As you can see from the titles and short abstracts of the technical presentations, great emphasis is placed on the tantalum and niobium activities in China. These will be complemented with the annual overview of tantalum and niobium activities world wide in the last year, two important presentations from Japan and one from Israel.

The social activities promise to be very exciting and a visit to Ningxia Non-ferrous Metals Smelter will offer an unusual opportunity to get acquainted with this important metallurgical producer in China.

In the name of the T.I.C., I would like to express our appreciation to the Ningxia Non-ferrous Metals Smelter and to the Chinese Non-ferrous Metals Society for their invaluable help in preparing the Thirty-eighth General Assembly in Xian.

It is a pleasure to report that tantalum and niobium had a good first half of the year 1997 and there is every indication that this positive development will continue into the second half.

Looking forward to seeing you in Xian,

yours,

S.S. Yeap
President

1998 ASSEMBLY

The meeting in 1998 will be held in Prague, Czech Republic, from October 11th to 13th, including a visit to the capacitor plant of AVX Ltd.

TECHNICAL PROGRAMME

DEVELOPMENTS IN TANTALUM AND NIOBIUM DURING THE LAST YEAR
by George J. Korinek, T.I.C.

Overview of the technical and commercial developments which occurred in tantalum and niobium since our last General Meeting in Greenville, South Carolina, will be given.

RESOURCES AND DEMAND/SUPPLY SITUATION OF TANTALUM RAW MATERIALS IN CHINA
by Wu Rui Rong, Qian Guo Wen, Nie Ming Lian, Zhang Xu Qing, et al.

Presentation will give an overview of China's tantalum resources, their locations and the characteristics of some deposits. The production of tantalum concentrate at the Yichun Mine will be discussed in detail. Forecast for the trend of the tantalum industry in China will be given.

HYDROMETALLURGICAL PROCESSES IN CHINA FOR EXTRACTION AND SEPARATION OF TANTALUM AND NIOBIUM
by Hu Ji Lin, Zhan Zong Guo, Xu Zhong Ting, et al.

Major processors will be introduced, the outstanding features of their processes and facilities and the quality level of their products will be discussed.
TANTALUM AND NIOBium carbide production and applications in china
by Zhou Ju Qiu, Tan Ri Shan, Huang Lie Ru, Zeng Fang Ming, et al.

The major producers of TaC/NbC, the main processes and the quality level and applications of their products will be discussed. The trends in development in China's carbide industry will be presented.

CHINA'S NIOBium industry and its products
by Yin Wei Hong, Chen Qin Yuan, Fu Jun Yan, Chu You Yi, et al.

The presentation will be an overview of the five major factories that are producing thermo-reduced niobium products including niobium bars and ferro-niobium. Nine manufacturers of niobium wrought products, their capacity and individual products will be presented.

An introduction to China's tantalum capacitor industry
by Lu Yi Sheng, Leng Shi Ming, Liu Zi Wen, Li Yun An, et al.

A short history of the tantalum capacitor in China will be given. The main manufacturers, their estimated capacity and the main products will be discussed, as well as the demand situation for powder and wire.

The negotiations, start-up, and operation of a high technology manufacturing joint-venture in China
by Arthur J. Yarzumbeck, Shenzhen GKI Electronics Company (IBM China and Great Wall Computer Group)

Many multi-national corporations have recognized the enormous potential of conducting operations in China. Balancing the benefits are the well (and sometimes not so well) publicized pitfalls of doing business in an economy emerging from approximately 50 years of fundamentalist communist rule as well as displaying cultural traditions enigmatic to typical Western management practices. This short presentation will highlight the personal experience of an American expatriate general manager from a large multi-national corporation (IBM) in the negotiations, initiation and continuing operations of a technology based manufacturing joint venture successfully conducting international business from a factory in the Shenzhen SCZ.

EVOLUTION OF HITACHI'S TANTALUM CAPACITORS
FROM T.I.C. MEETING IN AIZU TO T.I.C. MEETING IN XIAN
by Susumu Wada, Hitachi AIC

Map of various capacitors; evolution of chip tantalum capacitors; second generation chip tantalum capacitors of Hitachi; application of just-in-time concept to Hitachi's capacitors will be discussed.

NEW STRUCTURE OF SURFACE MOUNT TYPE TANTALUM CAPACITOR
by Takeshi Obara, Capacitor Division, Matsushita Electronic Components Co.

Our surface mount type tantalum capacitor with the new structure, which has plating terminals, makes it possible to achieve drastic miniaturization and large capacitance. Tantalum capacitors will be able to satisfy market needs in the multi-media age with their new structure and highly developed tantalum powder technology.

COMPARISON OF TANTALUM AND NIOBium SOLID ELECTROLYTIC CAPACITORS
by Yuri Pozdeev, VIEC

The presentation deals with the comparative investigation of the performance and life test results for the tantalum and niobium solid electrolyte capacitors. The attraction of niobium is its lower density and price. Whereas the two metals have much in common the electrical properties of tantalum and niobium capacitors are different. In particular niobium capacitors are characterized by a monotone increase of the current leakage during life test. This causes the parametric failure of the niobium capacitors. On the contrary the current leakage of the majority of the tantalum capacitors does not change significantly for a long time, but then increases sharply in some samples, hence the catastrophic failure is typical of some tantalum capacitors. The physical nature of these phenomena will be discussed.

MEMBER COMPANY NEWS

CBMM raising ferro-niobium capacity
Brazilian niobium producer Cia Brasileira de Metalurgia e Mineracao (CBMM), the parent company of our member Reference Metals Inc., has announced that it will be raising its ferro-niobium production capacity from the current 23,000 tpy to 30,000 tpy by the end of this year. The announced expansion is the first since 1980 and is aiming towards maintaining stability in world supply and pricing of ferro-niobium. Together with the capacity of Canada's Cambior and Brazil's Catalao the world capacity should grow to 57,000 tpy.

According to CBMM's statement, this capacity expansion will be connected with a relatively small investment as it will be achieved mainly through modifications of the production flow. The actual output in 1997 should be around 25,000 tons.

The production levels of niobium metal and niobium oxide should remain at the present level of 50 tpy of metal and 2,000 tpy of niobium pentoxide.

The company is also planning to start a production of around 150 tpy of high purity niobium pentoxide mainly for the optical glass industry.
**NIOBIUM-BASED SUPERCONDUCTORS: DEVELOPMENTS AND APPLICATIONS IN JAPAN**

This is a summary of the paper presented by Professor Kyoji Tachikawa of the Faculty of Engineering, Tokai University, Hiratsuka, Kanagawa 259-12, Japan, at the T.I.C. meeting in Greenville, 1996.

- **Filament**: Nb
- **Bronze**: Cu-13.0 wt% Sn-0.25 wt% Ti
- **Wire size**: 1.37 x 3.44 mm
- **Filament diam.**: 6.5 μm
- **No. of Filament**: ~30,000
- **Cu ratio**: 0.25
- **Non-Cu Jc (191)**: $1.20 \times 10^{10} \text{A/cm}^2$

![Cross-section of Nb$_3$Sn wire](image1)

**Figure 1**: Bronze-processed (Nb, Ti)$_3$Sn NRM 21T 60mm bore magnet

![Enlarged view of filaments](image2)

**Figure 2**: Nb$_3$Sn wire doped with Ta

Niobium-titanium superconducting wire containing 46.5 wt% Ti is being widely used for MRI, magnetically levitated (Maglev) trains, superconducting generators, accelerators, etc. A recent topic for Nb-Ti wire is the development of new wires for AC use, which are composed of a large number of very thin filaments of about 0.1 μm in diameter. A coil wound with Nb-Ti wire with Cu-2.5 wt% Sn alloy matrix has been stably operated in the frequency range 50-60 Hz. Nb$_3$Sn wires with n-Ti-doped bronze matrix are being used for generating fields over 15 T, and have generated a field of 21.5 T in operation at 1.8 K (fig. 1). A new Nb$_3$Sn wire doped with Ta has been fabricated, starting from Nb$_3$Sn intermediate compound powder (fig. 2), which exhibits enough critical current even at 23 T and 4.2 K. Meanwhile, Nb$_3$Sn-Al wires with large current-carrying capacity have been fabricated, with better strain tolerance than Nb$_3$Sn wires (fig. 3). Quenching from high temperatures and subsequent annealing provides excellent high-field performance in Nb$_3$Sn wires (fig. 4).

![Cu/Nb$_3$Sn multilayered wire by jelly-roll process](image3)

**Figure 3**: Cu/Nb$_3$Sn multilayered wire by jelly-roll process

![Nb tube process](image4)

**Figure 4**: Nb tube process
The test run of the practical vehicle of the superconducting Maglev train started in April 1997 using a new 18.4km long track located north of Mount Fuji. After a few years of testing, the speed of the vehicle is expected to reach 550km/h (figs. 5 and 6). A 70MVA superconducting generator has recently been completed at the Super-GM project, using three different types of superconducting rotors (fig. 7). The test site is located near the new Kansai International Airport. The winding of a Large Helical Device for fusion has been completed at NIFS using a 35km long Nb-Ti conductor with a large current-carrying capacity. The major radius of the device is 8m, and the total fusion system will be operated from fiscal year 1998. Japan is participating in the R&D of the huge ITER device, as well as that of the Large Hadron Collider at CERN. Recently refrigerator-cooled superconducting magnets have been realized which make the utilization of high magnetic fields more convenient. One example of the magnets generates 10T in a 10cm room temperature bore, using Ti-doped Nb3Sn (fig. 8). High-Tc oxide current leads, which have much smaller thermal conductivity than conventional high-purity Cu current leads, play a key role in this type of superconducting magnet. Meanwhile, Nb SQUID devices are applied for precise measurements in geophysics, magnetoencephalography (fig. 9), etc.

As for high-Tc oxide superconductors, tapes are being applied for power transmission cables operating at 77K, a refrigerator-cooled superconducting magnet operating at 20K, and a high-field insert coil operating at 4.2K. 50m long 2kArms high-Tc power transmission cables have been tested in Japan (fig. 10). An 800kVA superconducting transformer operating at 66K has also been developed. Bulk high-Tc superconductors act like a strong permanent magnet, and are being applied to non-contact transportation systems, magnetic bearings and small motors. A small car for golf course service using a high-Tc superconducting motor has been produced (fig. 11). High-Tc SQUID has been successfully applied for magnetoencephalography (fig. 12). High-Tc film devices, e.g., filters, antennas and low-noise amplifiers, have gained a great deal of interest in microwave applications of mobile telecommunication systems.
EXPERIENCE IN THE MANUFACTURE OF NIOBium-TITANIUM ALLOYS FOR SUPERCONDUCTORS

by Tadeu Carneiro, Harry Stuart, Reference Metals Company, Inc.

INTRODUCTION

As part of its continuous efforts to supply those niobium products required by the market, CBMM - Companhia Brasileira de Metalurgia e Mineração and its subsidiaries Reference Metals Company in the USA and Niobiun Products Company in Germany [1] successfully developed manufacturing processes to produce niobium-titanium alloys for superconducting applications. This specific program started in the late 80s. It coincided with the installation of CBMM's electron-beam furnace in Araxá, Brazil. The production of the first niobium ingot was in May of 1989. Since then, CBMM became a fully integrated niobium company - from pyrochlore ore to pure niobium metal.

Attempts to develop niobium-titanium finished rods in Europe were based on vacuum-arc remelting technology to melt the alloy. Following these attempts, RMC - Reference Metals Company - developed in the U.S. a complete manufacturing process for niobium-titanium based on a plasma-arc melting technology.

This paper presents the experience accumulated over the years in the successful manufacture of niobium-titanium products for superconducting applications via plasma-arc melting technology.

NIOBium-TITANIUM MANUFACTURE

Starting materials

The niobium used in the process is produced by CBMM in Brazil. Figure 1 shows CBMM's electron-beam furnace used to obtain ingots approximately 10" in diameter by 60" long used as the niobium feedstock by RMC in the U.S. The parameters used in refining niobium metal at CBMM have been presented elsewhere [2,3]. Table 1 shows RMC's specification with regard to required niobium and titanium chemistry used in the process. The level of impurities specified in the starting materials is dictated by the chemistry specification of the final niobium-titanium alloy, which is also presented in Table 1.
Melting and Solidification

It is well known that the three major problems associated with manufacturing of niobium-titanium alloys are macroheterogeneities (titanium freckles), microsegregation (sometimes referred to as coring) and niobium-rich inclusions (4,5). As the final filaments in the multifilamentary superconducting wires are drawn to smaller sizes, elimination of these defects is of crucial importance. The equilibrium diagram of the niobium-titanium system shown in Figure 2 reveals the potential for the occurrence of these defects when melting these alloys. The much higher melting point of pure niobium (2,469° C) as compared to that of pure titanium (1,670° C) and the large temperature interval of solidification in the system are the basic causes of the occurrence of these defects if wrong melting parameters are chosen.

![Figure 2: Section of the phase diagram for the niobium-titanium system](image)

RMC’s alloy manufacture scheme uses plasma-arc melting technology to ensure a final alloy with minimized microsegregation and free from titanium freckles and niobium-rich inclusions. Although it is possible to use vacuum-arc remelting technology to obtain similar results, the intrinsic characteristic of having melting and solidification controlled by the same liquid metal pool makes the task a much more difficult one to achieve. The first problem arises because on the one hand the liquid metal pool in the VAR must be as deep as possible to obtain complete melting and homogenization, but on the other hand it should be as shallow as possible to allow a solidification pattern without segregation. In addition, any unmelted niobium particles will settle to the bottom of the pool, remaining included in the ingot. In the past the electron-beam technology to manufacture this alloy was successfully used as an alternative to the VAR process (6). Although this technique can be used even in conjunction with VAR technology to allow better homogeneity in the final alloy, it also has its own limitations such as the need to control the evaporation rate of titanium to assure consistent homogeneity along the length of the ingot.

The process developed by RMC uses Ritech’s plasma-arc melting technology specifically configured for refractory metals cold-hearth melting. Figure 3 (7) shows a schematic of the Ritech furnace used in this development. The process combines separate alloying and casting steps within the furnace to ensure consistently good homogeneity, freedom from inclusions and fine microstructure. The initial stage of the process includes vigorous stirring of the molten metal in the cold-hearth during melting. This activity maximizes dissolution and/or melting of pure niobium in the niobium-titanium liquid pool. In addition, any high-density inclusions that remain, such as unmelted niobium particles, will tend to settle down to the bottom of the hearth. This benefit sharply reduces the leading cause of wire breaks during cable manufacturing.

After this melting stage, the liquid metal is poured into the casting crucible at low superheat, producing an homogeneous ingot with a fine grain structure. A second plasma torch plays on top of the crucible to control the solidification rate as the ingot is withdrawn down from the furnace. This process provides for high homogeneity in the alloy, with the assurance of a lackle and niobium-rich-particle free material.

![Figure 3: Plasma-arc melting furnace used to develop RMC’s process for melting of niobium-titanium alloys](image)

RMC started industrial production of niobium-titanium alloys for superconductivity in 1993, using the melting equipment installed at H.C. Starck, Inc., in Newton, MA, USA. Figure 4 shows the furnace utilized by RMC in this operation.
Mechanical Working

The niobium-titanium ingot produced in the plasma-arc melting furnace is subsequently processed into large diameter bars (3" to 8" in diameter) and small diameter rods (diameter smaller than 1.5"). Figure 5 shows niobium-titanium finished products. Extrusion allows a much more uniform deformation over the entire cross section of the product as compared to processes based on forging. As a consequence, during Cu-NbTi wire drawing the individual filaments display a smoother interface between the copper and the NbTi, which is important when niobium barriers are used in the multi-filamentary wire. The homogeneous deformation also ensures that the filaments will not go "out-of-round" during the final wire drawing stage.

EXPLORING THE POTENTIAL OF PLASMA TECHNOLOGY

RMC has used the plasma-arc melting technology to melt niobium-titanium alloys for superconducting applications with the restriction that the material must pass, and respond to heat treatment in a similar way to vacuum-arc remelted material. This limitation does not allow the full exploitation of the benefits of plasma melted material. The solidification mechanism in the plasma furnace results in a much finer grain size in the "as-cast" material as compared to ingots obtained via VAR melting or electron-beam melting. It would be possible to optimize both casting parameters during solidification and final steps of superconducting Cu-NbTi wire manufacture to avoid all prior mechanical deformation of the niobium-titanium ingot.

Table 2: Chemistry Results. Niobium - titanium - tantalum alloys

Alloying Elements (wt%)

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4. CONCLUSION

Plasma-arc melting technology associated with optimized thermomechanical processing can be used to obtain niobium-titanium products which match the highest quality product available in the market produced via vacuum-arc remelting.
technology allows highly consistent quality, manufacturing flexibility and potential for cost saving for the superconducting industry.

5. REFERENCES


COMPANY PROFILES

We are pleased to publish short profiles of some of the new members recently elected to the T.I.C.

4 3/4 miles, Lahat Road, 31500 Lahat, Perak, W. Malaysia

BEH Minerals Pty. Ltd. had its inception in December 1969 as a company to process and concentrate the various heavy minerals, such as ilmenite, zircon, monazite, xenotime, cassiterite, rutile and struvite (a tantalum ore) from the heavy ore rejects of the Malaysian tin industry. It is also involved in mining and processing for production of kaolin for the paper filter and the fibre glass industries.

In as far as struvite is concerned, the company had done much R&D work, and in 1972 it introduced the first commercial parcel of struvite as a raw material for the tantalum industry. Since then it has progressively produced from 50MT to 300MT annually of struvite. Over the last four years production capacities ranged from 230 to 300MT of struvite with 10-11% Ta2O5 content.

Specialty Metals Company
Rue Tenbosch 42A, B-1050 Brussels, Belgium.

Our company has been operating for the last twenty years and used to be called Sasson Metals and Chemicals. The change of our company name was made three years ago following a change in management.

We produce, through tolling arrangements, and market a number of metals used in high temperature alloys such as cobalt, tungsten, tantalum, niobium, molybdenum, vanadium, as well as light metals such as titanium, magnesium and calcium.

We source mainly in Africa, China and CIS and have offices in China, Russia and Kazakhstan.

King Metallurgical Industry Co., Ltd
69 Jiefang Road, (E), Changsha, Hunan, China.

King Metallurgical Industry Co. was established on the mainland in 1990, specializing in the processing of rare metals such as tungsten, molybdenum, tantalum and niobium, and the preparation of cemented carbides.

Since that time, such products as metallic tantalum powder, TaC, (Ta,Nb)C, Ta₂O₅, WC, NbC and molybdenum have been successfully turned out by this company, selling well in Europe, U.S.A., Japan and other countries.

The company constantly incorporates domestic and foreign advanced equipment and up-to-date technology to achieve high quality in the products.

MEMBER COMPANY NEWS

H.C. Starck V Tech
Mr Hiroo Naito has succeeded Mr Hiroshi Tashiro as the company's delegate to the T.I.C. Mr Naito is now President of the company, and Mr Tashiro is Chairman of the Board.

Matsushita Electronic Components, Capacitor Division
Mr Umio Nishiyama, Manager, Engineering Department, has taken over from Mr Kimura as T.I.C. delegate.

NEC
Mr Yoshihiko Saiki, Senior Manager Engineering, Circuit Components Division, has become the company's T.I.C. delegate, succeeding Mr Morimoto.

King Metallurgical Industry
Please note the following modified numbers:
Telephone: +86 731 411 4910
Fax: +86 731 412 1367

Aprobase
Mr Polak (associate member)
Please note the following modified numbers:
Telephone: +33 4 60 39 91 52
Fax: +33 4 60 39 92 27

Gwalia
Gwalla Consolidated reported an increase in tantalum production to 671,122 lb for the financial year ending June 1997.

As a result of modifications to the milling circuit at the Wodgina tantalum mine, production economics have improved to allow profitable processing of lower grade ore which significantly increased the resource base and extended the life of the mine. The production levels at the Wodgina mine will be maintained between 150,000 and 175,000 lb of tantalum in concentrate for the foreseeable future.

Tantalum-Niobium International Study Center,
40 rue Washington,
1050 Brussels, Belgium
Tel.: (02) 649 51 58
Fax: (02) 649 64 47
Please note that there is no longer a telex line to 40 rue Washington

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