

T I C

TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

PRESIDENT'S LETTER

Friends,

If you are an average member of the general public, you don't expect much to happen in mid-summer and if you are like the writer you are quite content with that state of affairs.

One interesting and possibly far reaching development to report is the "Prom Fund": as far as I can recall, we have talked among the membership and in the Executive Committee about the desirability of promoting our metals. The applications in electronics, carbide and chemical equipment have been around for a long time, but the newer ones such as ballistics and (single crystal) superalloys came to us from the *outside*! Shouldn't we take the initiative now and try to spread some information which would encourage the search for new uses by potential consumers??? The snag in the obvious answer was in the budget: there are more immediate services requiring the existing funds and an increase in fees would peg these too high. This eternally vexing problem has now been solved by a joint undertaking of the "Big Four" (Cabot, Gwalia, Starck and Thai Tantalum) who are providing forty thousand U.S. dollars for such promotion. The executing officer is none other than our Technical Adviser, George Korinek, who is doing this as an additional duty. The T.I.C. personnel provides the clerical management of the fund which is kept and accounted for entirely separately by dedicated bank account etc. Among the numerous projects are participations in and spreading the word at the International Minerals Conference in September, the Tungsten and Refractory Metals Conference in Washington in October, writing articles for the special section of the American market devoted to niobium and tantalum, and contact with the U.S. stockpile authorities so our voice will be heard when new plans are made and decisions taken.

The enthusiasm for this kind of action has spread to the regular T.I.C. Without spending any money we now expect our General Assembly in Japan to be shown under "Calendar of events" in the monthly London Metal Bulletin and we also expect to get some notice for our Goslar **Symposium** into the bi-weekly LMB. This is something that many members can pitch in and help: if you can

arrange for any free publicity go to it!! Just have a word with the Secretary General beforehand to avoid interference and duplications.

So much for this hot summer afternoon. See you in Japan!

Kindest regards,

Hubert Hutton,
for the T.I.C.

JAPAN, OCTOBER 1994

By the time that you read this issue of the Bulletin, final preparations for the meeting in Aizu-Wakamatsu, Japan, from October 24th to 26th, 1994 will have been made and we shall have reached the deadline for reservations and pre-registration. Anyone who wishes to attend and has not yet sent in forms should contact the secretariat without delay to see if it is still possible to make a booking.

In addition to the General Assembly of the association and an impressive technical session (see below), there will be a complementary social and sightseeing programme, as well as a plant tour of Showa Cabot Supermetals, with an alternative tour of the Shotic plant, manufacturing aluminium precision components.

Technical programme

The policy for non-ferrous metal industries in Japan,

by Mr Toshio Ibi, Director, Non-ferrous Metals Division, Basic Industries Bureau, Japanese Ministry of International Trade and Industry (MITI).

Volumetric efficiency of tantalum powders,

by Mr J.A. Fife, Mr R.W. Steele and Dr H.J. Chang, Cabot Performance Materials.

Development of high performance powders by HCST,

by Dr T.B. Tripp, Dr K. Andersson and Mr H. Naito, H.C. Starck, to be presented by Mr Naito.

Some characteristics of high CV tantalum powder,

by Mr Tomoo Izumi, Manager, Research and Development, Showa Cabot Supermetals.

Fracturing phenomenon and countermeasure of tantalum-box to sinter pellet at sintering process for high-CV powder,

by Mr Kageyoshi Sakamoto and Mr Kouichi Takina (presented by Mr Sakamoto), Tantalum Technology team, Pure Material Laboratory Ltd.

Tantalum capacitor overview: general trends and future,

by Mr Susumi Wada, Executive Managing Director, Hitachi AIC.

SUMMARY

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New technology and application of tantalum mill products in Japan,

by Mr Yasuhiro Higashiguchi, General Manager, Development Division, Vacuum Metallurgical Company.

Improving the properties of PMN ceramics,

by Dr K. Reichert, Dr F. Schlenkrich, Mr L. Seffner and Dr A. Hoppe, H.C. Starck, to be presented by Dr Hoppe.

The tantalum and niobium industry - today and tomorrow,

by Dr George J. Korinek, Technical Adviser to the T.I.C.

The Basel Convention and its effect on the tantalum and niobium industry,

panel discussion to be led by Dr George J. Korinek, Technical Adviser to the T.I.C.

ULBA METALLURGICAL PLANT (PO "UMZ")

PO "UMZ" recently became a member of T.I.C. Here is a short profile of this new member.

The production association "Ulba Metallurgical Plant" (PO "UMZ") was founded nearly 50 years ago in Ust-Kamenogorsk, East-Kazakhstan region, Kazakhstan Republic. PO "UMZ" was the leading producer of tantalum products in the former Soviet Union. Starting with the production of potassium fluotantalate, using the classical Marignac process, it has developed into a great facility, integrated from processing of tantalum concentrates through to the production of various products, including electron beam melted ingots, plates, sheets, foil, wire, discs, capacitor powders - including those of high voltage - and tantalum alloys.

Tantalum production was developed based on the newest technologies employing ion exchange processes and vacuum arc and electron beam refining. Scientific research and design departments of the association and the leading scientific and engineering institutes of the former Soviet Union contributed much to the creation of tantalum production at PO "UMZ". The developed technology is characterized by the high efficiency of extraction from raw materials, flexibility of using different concentrates and production of metal of high purity with not less than 99.8% of tantalum.

Niobium which usually occurs with tantalum in mineral concentrates is separated and converted into niobium hydroxide or oxide. Stringent quality control guarantees high quality of the manufactured products.

Tantalum products include:

Tantalum ingots: ingots with 100-120 mm diameter and length of up to 1500 mm are produced, and can be supplied in machined or non-machined form. A minimum purity of 99.8% Ta by weight is guaranteed.

Tantalum capacitor powders: five different types of capacitor powders are produced, both the sodium reduced and EB melted variety. Specific capacitance of sodium reduced powders is up to 22 000 $\mu\text{F/g}$ and of EB melted 5600 $\mu\text{F/g}$.

Wire and foil: PO "UMZ" supplies the capacitor industry also with tantalum wire and tantalum foil. Depending on the diameter, the actual length of the wire is from 150 to 2000 m. The finest wire has a diameter of 0.05 mm.

Mill products: PO "UMZ" produces tantalum sheets up to a width of 150 mm, and tantalum strip 60 mm wide. The thickness of

both is 0.25 mm. Tantalum rods with a diameter of 3-90 mm are also available.

In addition to tantalum products, PO "UMZ" is involved in the development and production of superconducting materials based on niobium-titanium alloy and niobium-tin. Wire and filaments of both these alloys are also produced.

The Director General of PO "UMZ", Vitali Leonidovich Mette, is the official delegate of PO "UMZ" at T.I.C.

ABSTRACTS FROM RECENT LITERATURE : NIOBIUM AND TANTALUM

1. Niobium and niobium compounds: this paper first outlines the physical and chemical properties of niobium metal and then goes on to describe occurrence and resource processing.

The commercially most important deposits in Brazil, Canada, Nigeria and Zaire are described. World reserves are estimated to be 4.1×10^6 t of niobium, of which 78% is in Brazil.

Processing of niobium ores is described in detail. The common niobium compounds are described as to their physical properties and preparation with the main emphasis on oxides.

The industrial production of niobium metal by reduction of the pentoxide or halides is treated in detail.

A good overall review paper on niobium and its compounds.

J. Eckert (HCST)
High Temperature Material Processes 11 (1-4) 97-118 (1993)

2. The pyrochlore deposit of Araxá, Brazil, is widely recognized as the world's longest known niobium reserve. This deposit has been mined by CBMM (Companhia Brasileira de Metalurgia e Mineração) since the 1960's, and has supplied over 60% of world niobium consumption. The history, geology and mineralogy of the deposit are described in detail.

The specific name of the niobium bearing mineral in the Araxá ore is pandait, a pyrochlore in which Na^{+1} and Ca^{+2} are virtually absent and have been replaced by Ba^{+2} . The main constituents of pandait are Nb_2O_5 63.4% and BaO 16.5%.

Total ore reserves are given as 461.7 million tons with an average content of 2.50% Nb_2O_5 .

Concentration of the niobium mineral is by selective froth flotation of the pyrochlore. Flotation concentrate having 55-60% Nb_2O_5 has to be treated in a calcining and leaching plant to reduce the relatively high level of phosphorus, sulfur and lead. The majority of the leached concentrate is reduced batch-wise aluminothermically to ferroniobium. Ore reduction charge will produce approximately 11 tons of 66% ferroniobium. Recovery of niobium is in the order of 96 to 97%. Since 1980 CBMM also produces niobium oxide, which is mainly used as a raw material for the production of vacuum grade ferroniobium, nickel-niobium and niobium metal.

Mining, ore preparation and niobium-based materials
production at Araxá, Brazil
O. Fo (CBMM)
High Temperature Material Processes 11 (1-4) 119-137 (1993)

3. An improved flaked tantalum powder and process for making the flaked powder is disclosed. The powder is characterized by a mean particle size in the range of ± 2 to ± 55 micrometers and a BET surface area in the range of ± 0.5 to ± 5.0 m²/g. For preference the mean particle size and BET surface area are selected so that the flakes have an aspect ratio (D/T) in the range of ± 2 to 50. Agglomerates of the flaked tantalum powder provide improved flowability, green strength and pressing characteristics compared to conventional flaked powders. The improved tantalum flake powder can be made by preparing a flaked powder and then reducing the flake size until it is in the range of about 5 to 20 micrometers and BET surface area is in the range of 0.5 to ± 1.5 m²/g.

James Fife

World Patent 93003191 (18.2.93)
assigned to Cabot Corp.

4. Production of very fine metal powders including niobium and tantalum with a particle size of between 1.0 nm and 3 μ m. These powders are produced in a gas phase reaction of e.g. halides of the metal and hydrogen. By controlling the heating up of the reactants, their mixing and cooling of the product mixture in a special apparatus, powders with a very narrow particle size distribution and low amount of impurities can be produced.

T. Koenig

European Patent 0568863A1
assigned to H.C. Starck, Goslar

5. Production of very fine oxide powder: equivalent patent to 0568863A1

T. Koenig

European Patent 4214724A1
assigned to H.C. Starck, Goslar

6. Treatment of industrial wastes containing tungsten and other refractory metals, such as tantalum and niobium, by digesting the waste material with a mixture of sulfuric acid and hydrogen peroxide. The digestion residue containing major portions of tungsten and other refractory metals is reacted with NaOH to put into solution the major part of the tungsten as a liquid that is separated from the insoluble solid that contains the major part of the other refractory metals, such as tantalum and niobium. The solid is treated with HCl to separate the iron from the refractory metals.

Wen-Chao Lai, W.J. Rourke

US Patent 5180563
assigned to GTE Products Corp., Danvers, Conn.

7. The physical and material properties of tantalum and niobium depend on the residual metallic and non-metallic impurities. The concentration of interstitially dissolved elements like carbon, oxygen, nitrogen and hydrogen have strong detrimental effects on material properties. Especially for the superconducting application of niobium and its alloys, the content of these elements must be kept at the lowest possible level. The electron beam drip melting furnace is the most suitable instrument for the production of superclean refractory metals. Depending on the desired ingot purity two melting cycles are usually required for tantalum and niobium of commercial purity and up to six cycles for superconducting niobium with high RRR-value. The total value of interstitials can be brought down to less than 40 ppm.

Refining of tantalum and niobium

A. Choudhury (Leybold Duferrit)

High Temperature Material Processes 11 (1-4) 49-60 (1993)

8. Metallic tantalum was electrochemically dissolved in an organic electrolyte containing n-propylamine. This resulted in the formation of a viscous fluid. Heating of the solution resulted in the formation of a glassy amorphous solid. Calcination of this precursor in an atmosphere of argon or ammonia resulted

in the formation of tantalum carbide, with nitrogen content in the range of 0.6-2.5 wt %. Calcination at comparably low temperatures, 800-1000°C, led to products with extremely broadened X-ray diffraction patterns; this is supposedly due to crystallite sizes in the nanometric range.

P. Zahneisen (University Jena)

Journal Materials Science Letters 13 (1) 20-22

9. Ultra high purity refractory metals and some selected compounds for microelectronic and microtechnology. In today's very large scale integrated ICs, chemically and thermally stable elements and their compounds have to be used. For a failureless function α -emitters like U and Th have to be diminished to 1 ppb, in the case of the 64 MBit-DRAM down to 0.1 ppb. Mobile alkali metals and transition metals which deteriorate the semiconductor properties of the silicon have to be removed. This leads finally to refractory metals of a purity of 6N (= 99.9999%). A similar development can be observed in the case of tantalum powder for microcapacitors. Also in this case impurities had to be reduced systematically to achieve today's high level of reliability.

G. Winter et al (H.C. Starck, Goslar)

Berg.-Huettenmaen. Monatsh. 138(1) 12-16 (in German)

10. Mineral processing of tantalum and niobium ores is examined in detail, mainly in terms of gravity concentration and flotation. Flow sheets of the main producing plants are given. These include: Wodgina in W. Australia; Tantalum Mining Corp. of Canada, Manitoba; Greenbushes, W. Australia; Araxá, Brazil; Niobec, Canada.

R. Burt (Tantalum Mining Corp. of Canada)

High Temperature Material Processes 11 (1-4) 35-48 (1993)

TWENTIETH ANNIVERSARY

1994 sees the twentieth anniversary of the foundation of the T.I.C., established by royal decree published on October 24th 1974. The nominated founders of the Tantalum Producers International Study Center were Mr Cornelius Herkströter, Mr Herman Becker-Fluegel and Mr Paul Leynen, who created the T.I.C. as an association of raw material producers - both miners and tin smelters. Its headquarters was registered at 1 rue aux Laines, where the office was in a tiny attic, before it moved to more spacious surroundings at 40 rue Washington in 1983 when the International Association Centre was set up in that building.

The aims of the association are to collect, and to disseminate, statistics and information on the industry, and to promote knowledge and use of tantalum and niobium. This is carried out by holding meetings and conferences, and by means of publications such as this Bulletin, and in any other way that presents itself.

Initial membership of 12 companies grew rapidly to 20 in 1976 when the by-laws on eligibility were broadened to allow the admission of those engaged in processing or offering technical or financial assistance to producers. The name was modified to Tantalum International Study Center in 1984. Membership was opened to companies chiefly involved with niobium in 1986, when the name was changed to its present form and the charter extended accordingly, although a number of members had always combined activities in niobium with their work on tantalum. Membership reached a peak in 1985 when the roster included 77 firms, although consolidation and integration, combined with difficult economic circumstances for non-ferrous metals in general, have now brought the total to 47.

The Executive 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:

1974	Cornelius Herkströter, Thaisarco
1975	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./NRC
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, Sprague Electric/Vishay Sprague
1994	Hubert Hutton, Sogem-Afrimet

The first Secretary of the association was Jan Goodyear; she was succeeded by Judy Wickens who has been Secretary General since 1977. The first forty issues of the Bulletin were edited by Graham Brown; Andrew Jones became editor in 1985 when he was appointed Technical Officer. Rod Tolley took over as Technical Adviser in 1989, and now George Korinek is the Bulletin editor and Technical Adviser.

At first two General Assemblies were held in each year: the first six took place in Brussels, then in 1977 a meeting was held in Winnipeg, where the T.I.C. was invited to tour the mine of Tanco at Bernic Lake. Thereafter until 1987 there were two Assemblies 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. Now there is only one Assembly each year, but the tradition of seeing an appropriate facility is maintained and the venue is different each year, while contact between delegates is promoted by an informal gathering in the spring, in Brussels. In this way delegates have visited tantalum and niobium mines, tin smelters producing tantalum as a valued by-product, processors and capacitor manufacturers, in Winnipeg, Rothenburg-ob-der-Tauber, Perth, Torquay, Bad Harzburg (Goslar), Tulsa, Penang, Stockholm, Boston, Kobe, Rio de Janeiro, Orlando, Frankfurt, Perth (again), Philadelphia, Phuket, Vienna - a remarkably international record and a tribute to our many hosts!

In 1978 the first Tantalum Symposium was held in Rothenburg-ob-der-Tauber in Germany. Building on the success of this event, tantalum and niobium were both covered in the Symposium and exhibition organised in Orlando, Florida, in 1988, which attracted over 180 participants and more than fifty accompanying people. A programme even more wide-ranging and ambitious is being composed for the next Symposium, in Goslar in 1995, an event not to be missed in the history of niobium and tantalum.

SYMPOSIUM

International Symposium on Tantalum and Niobium

Goslar, Germany

September 25th to 27th, 1995

PM '94 IN PARIS

T.I.C. participated with an exhibit at the Powder Metallurgy World Congress and Exhibition in Paris from June 6th to 9th 1994. A total of about 2000 people attended this congress, which took place at the CNIT Conference Center located in the La Défense area of the city.

Over 100 companies exhibited at the event: displays of powder producers, presses and other equipment, PM parts and products of both American and European companies were included.

We distributed over 50 fliers about the upcoming Symposium in Goslar and a similar number of tantalum/niobium pamphlets. About 20 qualified requests were received for the T.I.C. Bulletin and several companies inquired about membership of the T.I.C. Pictures of our exhibit are shown below.



PM '94 : T.I.C. stand

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