

T I C

TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

PRESIDENT'S LETTER

The Thirty-third General Assembly of the T.I.C. is now complete. The exotic venue of Phuket, an interesting technical program, and the opportunity to meet and interact with the members of the tantalum/niobium supply and user communities combined to produce a high level of attendance.

Passing a few days at a distant tropical resort provides welcome respite from the realities of struggling world economies and the slow growth/no growth environment of the tantalum business. Thailand, however, is a country rather unique to our business, being host to three phases of tantalum activity : ore extraction and treatment, being represented by tin mining and the Thaisarco smelter ; end product manufacturing at the capacitor plant of NEC ; and metal production which is now beginning at the new plant of Thai Tantalum. This very modern and integrated facility represents the completion of a project stretching over about 10 years. It will permit Thailand to retain a more significant share of the value added in the use of a valuable natural resource.

At a time when our industry is pressured by poor world economic conditions and slow or no growth, it is encouraging to see that despite the consolidations and departures which have been common in the tantalum/niobium business during the past several years, others have sufficient confidence to invest in the future of our business.

Finally, I would like to take this opportunity to invite you to join with the members of the Executive Committee at our informal business meeting. The meeting will be held at the T.I.C. offices in Brussels on April 26-27, 1993.

Best Wishes for the New Year

Peter Maden
PRESIDENT

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T.I.C. IN THAILAND

Le Meridien Hotel on the island of Phuket was a superb setting for the meeting organised by the T.I.C. from November 16th to 19th, with association members Thailand Smelting and Refining, Thai Tantalum and S.A. Minerals as hosts. The white sand of the palm-fringed beach, the warm blue waters of the swimming pool and the numerous sporting activities were much appreciated by the delegates as they relaxed between technical presentations and plant tours.

The conference was opened with a reception and barbecue buffet on the terrace by the swimming pool, and the social programme enjoyed by those accompanying the delegates included sightseeing tours, a boat trip to Phang-Nga, and a visit to the Baan Thai cultural village.

A splendid gala dinner was generously hosted by Thaisarco, Thai Tantalum and S.A. Minerals : Mr Yeap Soon Sit welcomed everyone on behalf of the hosts, and the Deputy Minister of Industry, Mr Pornthep Techapaibul, gave the keynote speech; the Phuket Governor Dr Yuwat Vuthimedhi addressed the guests, Mr Peter Adams replied to the Governor, and Dr George Korinek expressed the gratitude of the delegates and guests.

GENERAL ASSEMBLY

The Thirty-third General Assembly approved the applications of four new members, including the first Chinese company to be elected, and the second Russian member (see last page) : the membership tally is now 55.

Mr Yoichiro Takekuro, Vacuum Metallurgical Company, completed a successful year as President; for 1992-93 Mr Peter Maden of Vishay Sprague assumes the Presidency. In an amendment to the association's Charter the possible number of members of the Executive Committee was increased to twelve. Mr Mark Hague (Cabot Performance Materials), Mr Hubert Hutton (Sogem-Afrimet) and Mr Yeap Soon Sit (Thai Tantalum) were elected to the Committee, and Mr Hans-Jürgen Heinrich (GfE) resigned. Mr David Ratcliffe will take the place of Mr Peter Adams on the Executive Committee when he succeeds Mr Adams shortly as Managing Director of Thaisarco. The other members of the Committee were re-elected to a further term of office.

Mr Rod Tolley reported an active year in his post as Technical Adviser, including among his promotional activities a T.I.C. display stand for the exhibition at the 1992 Powder Metallurgy World Congress in San Francisco.

VIENNA, OCTOBER 1993

The Thirty-fourth General Assembly will be held in Vienna as part of a conference from October 4th to 6th 1993 which will include a field trip to see the plant of Treibacher Chemische Werke.

T.I.C. STATISTICS

TANTALUM

PRIMARY PRODUCTION

<i>(quoted in lb Ta₂O₅ contained)</i>	<i>3rd quarter 1992</i>
Tin slag (2 % Ta ₂ O ₅ and over)	118 663
Tantalite (all grades), other	177 016
Total	295 679

Note : 13 companies were asked to report, 12 replied.
The companies which reported included the following, whose reports are essential before the data may be released :
Datuk Keramat Smelting, Gwalia/Greenbushes, Malaysia Smelting, Mamoré Mineração e Metalurgia, Metallurg group, Pan West Tantalum (Wodgina Mine production), Tantalum Mining Corporation of Canada, Thailand Smelting and Refining

QUARTERLY PRODUCTION ESTIMATES

<i>(quoted in lb Ta₂O₅ contained)</i>			
LMB quotation :	US \$ 30	US \$ 40	US \$ 50
4th quarter 1992	299 400	388 600	404 000
1st quarter 1993	307 400	396 600	412 000
2nd quarter 1993	307 400	446 600	462 000
3rd quarter 1993	307 400	446 600	512 000
4th quarter 1993	307 400	446 600	512 000

Note : The quarterly production estimates are based on information available, and do not necessarily reflect total world production.

PROCESSORS' RECEIPTS

<i>(quoted in lb Ta contained)</i>	<i>3rd quarter 1992</i>
Primary raw materials (e.g. tantalite, columbite, struverite, tin slag, synthetic concentrates)	251 304
Secondary materials (e.g. Ta ₂ O ₅ , K ₂ TaF ₇ , scrap)	64 776
Total	316 080

Note : 17 companies were asked to report, 16 replied.

PROCESSORS' SHIPMENTS

<i>(quoted in lb Ta contained)</i>	<i>3rd quarter 1992</i>
Product category	
Ta ₂ O ₅ , K ₂ TaF ₇	22 502
Carbides	65 904
Powder/anodes	228 496
Mill products	108 543
Ingot, unworked metal, other, scrap, alloy additive	62 200
Total	487 645

equivalent to 658 321 lb Ta₂O₅.

Notes :

In accordance with the rules of confidentiality, categories have been combined as shown.

17 companies were asked to report, 15 replied in July, 16 in August and September. For both receipts and shipments by processors, reports by the following companies are essential before the data may be released :

Cabot Performance Materials, W.C. Heraeus, Kennametal, Metallurg Group, Mitsui Mining and Smelting, NRC Inc., Showa Cabot Supermetals, H.C. Starck, Thai Tantalum, Treibacher Chemische Werke, Vacuum Metallurgical Company, V Tech

NIOBIUM

PRIMARY PRODUCTION

<i>(quoted in lb Nb₂O₅ contained)</i>	<i>3rd quarter 1992</i>
Concentrates : columbite, pyrochlore	11 620 804
Occurring with tantalum : tin slag (over 2 % Ta ₂ O ₅), tantalite, other	136 693
Total	11 757 497

Note :

15 companies were asked to report, 14 replied. The companies which reported included the following, whose reports are essential before the data may be released : Cambior, Mineração Catalao de Goiás, Niobium Products Co. (CBMM)

PROCESSORS' SHIPMENTS

<i>(quoted in lb Nb contained)</i>	<i>3rd quarter 1992</i>
Compounds and alloy additive : chemical and unwrought forms (e.g. NbCl ₅ , Nb ₂ O ₅ , NiNb, FeNb [excluding HSLA grades])	627 895
Wrought niobium and its alloys in the form of mill products, powder, ingot and scrap	
(i) Pure niobium	30 294
(ii) Niobium alloys (such as NbZr, NbTi and NbCu)	55 524
HSLA grade FeNb	6 241 503
Total	6 955 216

Note :

18 companies were asked to report, all 18 replied. Reports by the following companies are essential before the data may be released : Cabot Performance Materials, W.C. Heraeus, Kennametal, Metallurg Group, Mitsui Mining and Smelting, Niobium Products Co. (CBMM), NRC Inc., H.C. Starck, Teledyne Wah Chang Albany, Thai Tantalum, Treibacher Chemische Werke, Vacuum Metallurgical Company

CAPACITOR STATISTICS

CONSUMPTION BY AREA

	<i>(figures in millions of units)</i>			
	Average per quarter			
	1988	1989	1990	1991
North America	340	302	337	356
Europe	220	206	232	230
Japan	635	681	808	965
Rest of world	207	304	361	492
World	1402	1493	1738	2043

	1992			
Quarter :	1st	2nd	3rd	4th
North America	392	403	448	
Europe	245	259	216	
Japan	847	769	735	
Rest of World	541	637	592	
World	2025	2068	1991	

Source : Members' estimates

TECHNICAL PROGRAMME

The technical sessions opened with a talk by the retiring T.I.C. President, Yoichiro Takekuro of Vacuum Metallurgical Company, on the support given by the Japanese Government through MITI (the Ministry of International Trade and Industry) to technological development. Government funding for completed large-scale projects (those which should reach practical application within 15 years) has been about 1.2 billion dollars for 22 programmes over 25 years. Fourteen other completed projects on basic technologies for future industries (generally more basic research) have been funded by some 300 million dollars over 10 years. Private industry has contributed a similar amount to these programmes to give a grand total of about 3 billion dollars. A typical project in the large category is for the development of advanced robot technology, while one for a future industry investigated three-dimensional integrated circuits. Priority use of the technology developed in a project is given to the private industry partner for a short time only. Thereafter the technical information is released and the patents and know-how are made available to all (including foreign companies at a maximum 50 % ownership).

Mr Ishimaru (also of VMC) followed with a talk on some new developments in the use of metallic tantalum. His company specialises in tantalum heat exchangers, particularly for the chemical industry, and has an increasing market for tantalum, tantalum silicide and tantalum pentoxide sputtering targets for film deposition. Of particular interest was his description of the ultrafine tantalum powder which, in an inert gas stream, can be used for "jet printing" onto a moving substrate. This can be applied to making a patterned conducting film, to making cone-shaped spots to serve as micro electrodes, or to repairing a damaged pattern. It has the advantage over current methods of being dry, and of requiring much lower temperatures.

Professor Krivan of the University of Ulm then spoke on the methods of analysis available for testing high purity niobium and tantalum. As specific impurity levels reach down to low parts per billion, the chemist is directed towards capital-expensive modern techniques such as nuclear activation.

Mr Zeng Fang Ping from the Ningxia Non-ferrous Smelting Company (elected to T.I.C. membership earlier that morning) then gave a description of their tantalum and niobium plant prepared by the Managing Director, Mr Wu Rui Rong. That paper is printed later in this Bulletin.

Also printed below is a resume of the paper given by Dr Eckert of H.C. Starck which followed a lunch break. His subject was new developments in the non-steelmaking uses of niobium.

Mr Hillbeck of Gwalia Consolidated Ltd, the owners of the Greenbushes tantalum mine (already the world's largest) in Western Australia, reported on the likely expansion of their tantalite output when they start mining the hardrock deposit. The current annual production levels of 400 000 pounds Ta_2O_5 will for the time being be maintained, but any improvement in the market can be met by the new facilities with a rise to 600 000 pounds Ta_2O_5 . The open cast workings will be up to 300 metres deep, and recoverable Ta_2O_5 is estimated as 10.5 million pounds, over a 13 year life.

Mr Sangat Piyasin, Thaisarco's geology adviser, described the three bands of granite which run north-south down the length of Thailand, each with distinctive mixtures of niobium-, tantalum-, yttrium- and titanium-bearing minerals. That to the west running through Phuket contains the greatest proportion of tantalum minerals, including tantaliferous cassiterite (tin ore).

Professor Paschen of Montan University Leoben gave a paper on the total energy consumption in the extraction of tantalum and niobium. His charts highlighted the large heat use in reduction of the oxides or fluorides by electrolytically produced metals such as aluminium or sodium, and also the high power consumption, per ton of pure metal produced, of electron-beam refining (much of the energy then lost being to the cooling water).

Mr Shunichi Yamamoto of the Crystals Division, Mitsui Mining and Smelting, then told how lithium niobate and tantalate crystals were serving a vital purpose in many types of electrical equipment in the average household. There they may be found in television sets and VCRs as waveguides and modulators.

Mr David Maguire of Kemet, in a series of charts, showed the dramatic growth in the use of tantalum capacitors over the past twenty years, and the consequent overall consumption by weight of tantalum powder. The latter had flattened off in recent years as a result of technical improvements both in the powder and in the fabrication of the capacitor, so that the 8 billion or so capacitors currently made worldwide needed very little more powder in their manufacture than a quarter of that number required only five years ago. That progression was only disrupted in 1980 when high tantalum prices drove away many of the mass market applications of capacitors to others (ceramic, aluminium); his chart showed that it took some



Plant tour group

four years of stable tantalum prices to bring his "learning curve" back on slope.

The programme ended with a stirring call from our (twice) Past President, Dr George Korinek, to put all our efforts into publicity and research for broadening the applications and improving the marketing of tantalum which seemed to have reached a hiatus in its growth. Perhaps more countries should emulate Japan in sponsoring the type of research that Mr Takekuro described at the beginning of the assembly?

PLANT VISITS

On the following morning, some eighty delegates visited the dressing sheds of S.A. Minerals (a T.I.C. member). It operates what is known in S.E. Asia as an amang plant, treating with highly-specialised and diverse equipment the mineral "middlings" produced by tin mines (both dredge and open cast). These still carry a significant percentage of tin as the heavy mineral cassiterite, which the mine itself has been unable to separate from other heavy minerals with the relatively simple equipment at its disposal. In the past, the amang dumps were sold merely on the basis of their tin content, and any other valuable minerals recovered helped cover the buyers' costs, but the present very depressed price of tin means that the other valuable minerals present (e.g. columbite/tantalite and struverite for tantalum and niobium, monazite for rare earths) assume greater importance than hitherto. The S.A. Minerals plant (owned by the family of our new Executive Committee member, Mr Yeap Soon Sit) is a very good example of its kind; it is very versatile and flexible so that the products can have the greatest overall market value at any given time.

We stopped briefly at the abandoned tantalum recovery plant tragically destroyed by fire in 1986, on our way to the Thaisarco tin smelter. Not so very long ago, the latter was the biggest single source of tantalum in the world through its tin slags. It is still of course a significant producer, but the combination of very depressed tin prices and the drawn-out end of the monsoon made for a particularly low level of activity at the time of our visit. However, the plant was immaculate, and it was clear how great an effort had been put into employee motivation and safety. The lunch (given by Thaisarco) afterwards provided a good opportunity for those delegates from tin smelters to get together with their counterparts from Thaisarco to "cry in their beer" over the state of their industry!

The final day covered a visit to the new tantalum plant of Thai Tantalum to the east of Bangkok, of which Mr Yeap Soon Sit is the Managing Director. He had arranged (and most generously agreed to underwrite) the charter of an aircraft from Thai International to fly delegates from Phuket to Utapao (the Naval Air Station, once a major staging-post for the Vietnam war) and after the visit on to Bangkok to connect with their onward travel. A most elegant arrangement and amply justified in the end by the enrolment of some eighty delegates for the flight and visit.

The plant is most generously laid out and has the benefit of a Laporte hydrofluoric acid plant alongside which will be able to pump in the principal reagent. Capital expenditure to date has been some 740 million baht (\$ 30 million U.S.) and the equipment is clearly of a high standard (both in the plant and the support sections e.g. the laboratory). Know-how also would seem to be well taken care of as the engineers on the staff were well known to several delegates. We wish the new plant (which should be commissioned shortly) every success.

R.J. Tolley
Technical Adviser

STATISTICS : COMMENT

I would like to draw readers' attention to three matters concerning interpretation of our T.I.C. statistics which were raised at the Phuket meeting :

1. Our processors' statistics for tantalum are probably the most comprehensive of those we publish, and so should reflect accurately worldwide consumption of our two metals. They have, however, been influenced in 1992, and will continue to be so through March 1995, by the activities of the Defense Logistics Agency in the United States. This includes the DLA purchase for their stockpile of tantalum-bearing material from O'Dell Mining, and which they are taking as tantalum oxide. These transactions are showing as processors' purchases of concentrate, and shipments of oxide. The recently (September 1992) agreed contracts for upgrading DLA stockpile material to ingot tantalum and niobium will have a similar effect.

The DLA is also, however, currently making purchases of ingot tantalum and vacuum grade ferroniobium, and these will merely show as increases in shipments by processors. For the convenience of those whose job it is to interpret the statistics, shipments to the DLA as declared to date [December 1992] will total :

Processors' receipts	Processors' shipments
1. Ore ex O'Dell 200 000 pounds Ta ₂ O ₅ contained	200 000 pounds Ta ₂ O ₅ in 8 lots of 25 000 each Jan 92/Nov 94
2. 185 700 pounds Ta contained in powder and ore	185 700 pounds Ta ingot Oct 92/Mar 95
3. 159 653 pounds Nb contained in powder and ore	159 653 pounds Nb ingot Oct 92/Mar 95
4.	32 000 pounds Mar 93/Feb 94 16 000 pounds Jul 93/Oct 94 15 000 pounds Nov 93/Feb 95 63 000 pounds Ta ingot
5.	100 000 pounds Jan 93/Jun 93 75 000 pounds Mar 93/Aug 93 75 000 pounds Apr 93/Sep 93 250 000 pounds Nb in vacuum grade ferroniobium

2. We have conducted a survey of our members' knowledge or opinions of the amount of supplier material shipped (tantalum and niobium) which is returned either to processors as scrap, or to another consumer for use in his (presumably less quality or form conscious) process.

The consensus on overall tantalum return to processor is 20 % which agrees well with our processors' receipts statistics. In the case of powder 21 % is returned to processors, but only 7 % of ingot metal purchases. However, the general advice is that most of in house scrap (in one case 95 %) of tantalum ingot purchasers is sold on to superalloy or carbide makers.

In the case of TaC 23 % of in house and customer scrap is currently recycled. Makers of tantalum salts (e.g. lithium tantalate of very high purity) return 20 % of the Ta₂O₅ contained to processors.

There is little or no recovery of the niobium used in steelmaking. Overall 12 % of "pure" niobium deliveries are returned to processors as scrap.

The great majority of niobium metal scrap is sold for use in alloy or carbide making. The exception is niobium in fabricated NbTi superconducting cables which are embedded in copper, and so too complex to recover. Less than 15 % of NbC scrap is recycled in-house or by customers. Makers of salts, e.g. lithium niobate, return 5 % scrap to processors.

3. We have had considerable trouble in recent times in obtaining meaningful data on the world consumption of tantalum capacitors. From this issue of the Bulletin we plan to publish our members' estimates of consumption in North America, Europe, Japan and the rest of the world. We will be showing the current year's quarterly figures, with the average per quarter for each of the four previous years for comparison. (See p. 2)

NIOBIMUM AND TANTALUM : NEW APPLICATIONS AND TECHNOLOGY FROM RECENT LITERATURE

NIOBIMUM

1. Niobium may be recovered from uranium-niobium alloys (machining scrap etc) by reacting the metal with hydrochloric acid and a fluoboric acid catalyst to give a solution containing the bulk of the uranium, and a residue of mixed oxides of niobium and uranium. A nitric acid leach frees the rest of the uranium, leaving a hydrated niobium oxide which can be calcined to the anhydrous oxide.

Nuclear Metals Inc U.S.P. 5 084 253 (28.1.92)

2. The yttrium and niobium content of Chinese porcelain has been found to be useful in the detection of fakes. The Nanking Cargo, which was recovered in 1985 from a ship sunk in about 1750 consisted of over 100 000 pieces of porcelain made in Jingdezhan (Nanking was the transshipment port, not the city of manufacture as many traders believed). Many art historians were worried about the origins of some of the pieces now being traded, and non-destructive testing by X-ray fluorescence has confirmed their fears. The modern fakes have a very high concentration of yttrium, and more than twice the niobium content of the genuine pieces.

C.T. rap. Applied Spectroscopy 45, 4, 584-587 (1991)

3. The Baotou iron ore of China has a high niobium content, and much research has been conducted in the past four years (principally at the University of Science and Technology at Beijing) on recovering the niobium from converter slags resulting from the smelting of that ore. In this the latest paper on the subject, the authors describe the slow-cooling accelerated crystallization of the slag, with niobium-bearing crystals up to 48 μ from the 3,7 μ in the normal production slag. At this size the crystals which are of niobium pyrophanite (25-57 % Nb₂O₅) may be mechanically separated.

X. Zhang and Z. Yang, Nonferrous Metals (China) 43 (2) 35-38, 43 (May 1991)

4. A catalyst for the reduction of nitrogen oxides (e.g. from exhaust gases) which is highly resistant to poisoning may be made from copper, nickel, chromium or cobalt powder

(10-60 mesh) by coating it with niobium or chromium carbide powder. The latter is preferably of 200 mesh grain size, and the coating of from 5 μ to 60 μ thickness is obtained by milling in a pot-type ball mill. The composite is then sintered in an inert atmosphere or vacuum to produce the catalyst.

Sumitomo Metal Ind. Ltd., Japanese Patent 03-232532 (16.10.91)

5. In the preparation of Nb₃Al alloy for superconducting wire it has been found that the addition of between 10 and 2000 ppm beryllium to the aluminium used greatly improves later processability and homogeneity of the filament.

Sumitomo Electric Ind. Ltd., Japanese Patent 03-230421 (14.10.91)

6. A titanium alloy containing 20-55 % niobium and 0.05-20 % ruthenium is claimed to have excellent corrosion resistance in a nonoxidising environment particularly in high temperature, high concentration hydrochloric or sulphuric acids. The alloy has good workability.

Nippon Mining Co Ltd, Japanese Patent 03-193836 (23.8.91)

7. In an earlier paper (see Bulletin No. 69), the authors described their experiments on the reduction of niobium oxide and pyrochlore by carbon (in order to save on the expensive aluminium currently used in the preparation of ferroniobium). The main solid product was niobium carbide and reaction rates were not high. Further trials here reported had an addition of iron, and this had the effect of speeding up the process - thought to result from reactions between carbon dissolved in the iron and the niobium pentoxide.

Munz and Chin, Canadian Met. Quarterly 31 (1) 17-24 (1992)

8. In two articles the author described how commercial ferroniobium may be used as a source of niobium metal and niobium compounds. The ferroalloy is made friable by reacting it with hydrogen : it is then broken up (it is fragile enough to be broken up with the fingers !) and ground to -140 mesh. To produce the metal or the nitride the powder is then heated to 1200 degC under nitrogen to produce a mixture of iron and niobium nitrides from which the iron may be removed by leaching with 2N hydrochloric acid at 50 degC. If the metal is now required, it may be obtained by denitriding with arc- or plasma-melting after the addition of some aluminium to assist compaction and electrical conductivity.

The "ferroniobium hydride" will also react with methane to produce mixed iron niobium carbides, and with aluminium to produce Al₃Nb. In all these cases the niobium-bearing product may be freed of iron and other impurities by leaching with 6N hydrochloric acid + 1 % hydrofluoric acid.

J.A. Sommers et al, Light Metals 1992 (TMS), pp 1303-1307.

TANTALUM

1. Powder metallurgy has been made even more versatile in recent times by the introduction of hot isostatic pressing (HIP). If specially shaped charges are to be made, the mould must be lined with a material of sufficient hot strength, ductility and fine grain size. This article considers the relative merits of an iron-nickel-tungsten alloy, a copper-tungsten alloy, and tantalum for the application, and concludes that tantalum gives the best overall performance.

2. A tin-tantalum alloy with 0.3-6.0 % Ta is used in the production of commercial Nb₃Sn superconducting wires. It may be made by melting the tin in a high-frequency furnace and bringing it to 700-1800 degC before adding the required amount of Ta powder under an inert gas atmosphere. The mixture is agitated and skimmed and the alloy is cast at 650-1800 degC.
Mitsubishi Electric Corp. Japanese Patent 03-177523 (1.8.91)
3. Tantalum and niobium are difficult to recover during the treatment of wolframite for extraction of tungsten, because they tend to be dispersed amongst various products. The tungsten is recovered as ammonium paratungstate crystals, and the mother liquor from which it is obtained contains most of the tantalum and niobium together with much recycled tungsten. The authors report that by adding caustic soda solution to the mother liquor while stirring it at 100 degC. Ta and Nb are precipitated as their sodium salts. If these are digested with 20 % hydrochloric acid, washed with 3 % HCl, dried and calcined, the commercial mixed oxides are obtained.

*Tao Zhengji, Zhuzhou Cemented Carbide Ind. Co.,
Refr. Metals & Hard Materials 10, 143-146 (1991)*

ELECTRONICS

1. Many newly-developed high frequency dielectric ceramics are based on barium salts with a perovskite structure. A recently patented composition may be represented as Ba(MgZn)_{1/3}(NbTa)_{2/3}O₃ and there is a particular claim in respect of one containing between 46.7 % and 48.3 % Ta₂O₅. The crystal grain size is controlled at 5-20μ, and porosity is less than 7 % without the need for lengthy sintering or heat treatment.
*Matsushita Electric ind. Co. Ltd.
European Patent 452 945 (23.10.91)*
2. Silver tantalum sulphides, either Ag₇TaS₆ or AgTaS₃ have useful properties as semiconductors. They may be formed by making a compact of silver, tantalum and tantalum sulphide (or elemental sulphur) in the correct proportions and heating it to 450-600 degC in an alumina crucible set in an evacuated and sealed quartz tube.
*Nat. Inst. Res. in Inorganic Materials
Japanese Patent 03-257022/3 (15.11.91)*
3. Magnetic films of iron-tantalum or iron-niobium nitrides are said to have excellent soft magnetic characteristics, and superior corrosion resistance and hardness to those made with the equivalent carbides. The film is formed by sputtering or vacuum deposition, and is then heat treated to produce a fine crystal grain texture in vacuum or under an inert gas.
N.E.C. Corporation, Japanese Patent 03-263306 (22.11.91)

R.J. Tolley
Technical Adviser

NEW DEVELOPMENTS OF NIOBIUM COMPOUNDS AND ALLOYS

(A resume of a talk given by Dr. J. Eckert from H.C. Starck to the T.I.C. Assembly in Phuket)

Niobium is obtained as the oxide from tin slags or columbite and other concentrates by acid solution (HF) and solvent extraction to separate it from tantalum. Ammonia precipitates it from solution as niobic acid, which is then calcined to Nb₂O₅. An increasingly used alternative is to chlorinate a ferroalloy containing niobium and tantalum, and then fractionally distil the mixed chlorides. The niobium chloride recovered is simply hydrolysed with steam to the oxide.

The choice of process may be decided by the final grade of oxide required: so-called pure grade Nb₂O₅, for use in the preparation of niobium metal or alloys has an oxide content of 99.8 % — the metal produced is amenable to refining in an electron beam or plasma furnace.

A slightly higher quality with not less than 99.8 % Nb₂O₅ is required for preparing perovskite based electroceramics — hence the name "ceramic grade". These complex niobates, of which lead magnesium niobate (PMN) is typical, have considerable use in electronic circuits with very good temperature characteristics.

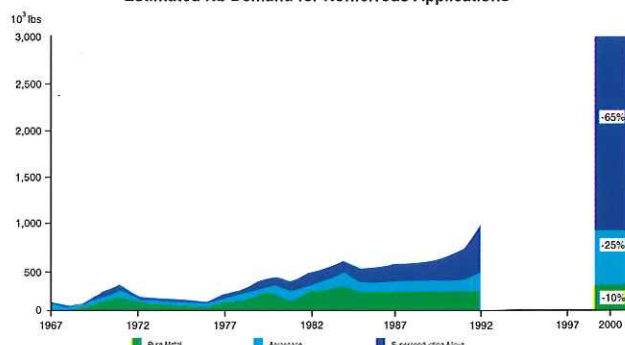
The two highest grades of the oxides are "optical" at 99.95 % purity (it is used in the formulation of glass with a very high refractive index — powerful lenses with much less weight) and single crystal grade, 99.99 % pure, used in the manufacture of lithium niobate for surface wave filters.

Niobic acid is coming into its own as a catalyst in petrochemical and similar process reactors which require acid catalysis. It has the advantage over liquid acids in that (i) it is easily separated from the product (ii) it is not corrosive to the reactor material (iii) there is no environmental problem in discarding the spent catalyst. An example is the alkylation of aniline which can be effected over niobic acid at 250 °C with a conversion efficiency greater than 95 %.

In 1991, 34 million pounds of niobium products were consumed worldwide of which 88.5 % was in the form of commercial ferroniobium. A mere 2 % of the total is used as metal. Of the 650 000 pounds of pure niobium metal used annually, some 20 % is used as such, much of it for chemical process plant, for nuclear fuel processing, and for some electrical applications. This group has seen little growth in the past ten years and little more is expected by the end of the decade.

More promising is aerospace, at present accounting for 30 % of consumption for such things as engine and turbine components and fasteners. Much higher operating temperatures (and so efficiency and speed) will be available as

Estimated Nb Demand for Nonferrous Applications



niobium based aluminium titanium alloys are increasingly developed.

Finally the biggest part of consumption, and probably the fastest grower is in superconducting alloys such as are used for the powerful magnets for MRI, in medical diagnosis, and for the Maglev train. This category's growth is such that a 65 % proportion of all metallic uses of niobium can be seen in the year 2000, by which time we forecast annual worldwide consumption will exceed 3 million pounds of Nb.

INNOVATION AT THE NINGXIA SMELTING PLANT AND RESEARCH INSTITUTE FOR NON-FERROUS METALS

by Mr. Wu Rui Rong, Managing Director

The Ningxia Smelting Plant and Research Institute for Non-Ferrous Metals has been in the tantalum-niobium industry for nearly three decades. After years developing from a small former laboratory of the Beijing Non-Ferrous Metals Research Academy, it has grown up in Ningxia, an autonomous region 1200 kilometers west of Beijing, into an enterprise with more than 1000 employees working in its tantalum/niobium division. Compared with its early years of operation in the tantalum/niobium division, the capital investment has increased ten-fold. The main product, tantalum powder, has a 12-fold increase in output and the sales value has increased 15-fold. But it is still a small company. In response to the increasing demands from both the domestic and international markets, it is now undertaking an innovation project including improvements and expansion in the following fields.

1. EXPANSION OF DIGESTION CAPACITY

A two-chamber vibratory ball mill with double the old capacity was recently installed in place of the old single chamber machine. A further mill of the same design has been ordered and will be installed next year after which the ore grinding capacity will be four times greater than in 1991.

The Ningxia Smelting Plant (NSP) uses hydrofluoric acid and sulphuric acid to dissolve ores in lead-lined tanks. The solution goes through a series of mixer-settler boxes, counter-currently contacted with MIBK, when tantalum and niobium are extracted, refined and separated from each other, and then converted to potassium tantalum double fluoride and niobium pentoxide.

New digestion tanks of better construction and bigger size have already made a contribution to this year's production and a further one is under construction and will go into service next year. Some improvements in the mixer-settler boxes, including an expansion of capacity and upgrading of efficiency have been effected or will be soon. A rotary vacuum dryer will soon replace the old stationary shell dryer for drying the double fluoride salt, and a rotary kiln for niobium oxide calcination is now at the blueprint stage; it is scheduled to be installed by the end of 1993.

After finishing all these improvements, NSP will have enough capacity in ore digestion to meet the expected needs of the next three to five years. In addition, combining these improvements with the experience gained from both production line and laboratory research, NSP will be more efficient to digest tantalum-bearing raw materials from diversified sources.

2. IMPROVEMENTS IN TANTALUM POWDER OPERATIONS

NSP began its sodium reduction in the middle 60's replacing the electrolytic process which produced low CV tantalum powders. After about ten years of development, powders with more than 8000 CV values were developed and introduced to the market. Currently, NSP produces a wide range of sodium reduced powders from 3500 to 23 000 CV/g. The present improvement project for sodium reduction was initiated last year with emphasis on equipment replacement. A series of items of new equipment is in the process of being installed, or will be installed in the near future. Among these, the most important are : three new retorts, whose construction is finished; three heating pits, whose blueprints are completed; two agglomeration furnaces which will be commissioned in March next year. A large blender for blending 300 kg of powders has been in service for the past few months and an even bigger blender is scheduled to be completed by the end of this year.

NSP has a 150 KW electron beam furnace melting tantalum ingots for EBM powders and alloyed or unalloyed tantalum or niobium ingots for mill products. A more powerful EBM furnace is under consideration. Larger ingots are needed for the EBM powder process and mill products manufactured inside and outside the plant. NSP established EBM powder production ten years ago and made it work, but the scale is very small. Under the present innovation project, a series of facilities including pulverization and classification equipment is going to be installed in the second half year of next year. With these improvements and expansions finished, it is expected that the EBM powders will be as attractive as sodium-reduced powders in both the domestic and international markets.

3. INNOVATION PROJECTS IN TANTALUM WIRE

In NSP, capacitor lead tantalum wire production was started more than ten years ago with iso-statically pressed round rod as starting material, vacuum sintered and rotating swaged to 2.5 to 2 mm diameter line, and then drawn into wire with diameter down to 0.5 mm to supply the domestic market. In order to keep pace with the changing market, the management of NSP determined to replace some equipment and to make process improvements. Those to be purchased include a drawing machine, an advanced die maintaining machine and a fine spooling machine. It is planned to finish the project within one year. After the innovation, the wire products of NSP will be more capable of meeting domestic and international customers' requirements in quantity, size, mechanical properties and physical performance.

4. INCREASED OUTPUT OF NIOBIUM PRODUCTS

NSP has several well-established production lines for niobium oxides including crystal grade and optical grade niobium oxides, niobium carbides, niobium powders, powder sintered products, niobium EBM ingots and niobium mill products. These products sell well in China, and sometimes overseas.

These production lines employ very common processes and are capable of expansion to balance the raw materials consumption. However, in order to upgrade the production efficiency, some advanced facilities and accessories will also be considered in the present innovation project.

5. STRENGTHEN RESEARCH WORK

Laboratory research has played a very important role in NSP. Most of its process technology such as liquid-liquid extraction, sodium reduction, high-purity niobium oxide and

tantalum oxide, lithium niobate and lithium tantalate, single crystal growth, tantalum wire, niobium tube, etc., have been developed by the engineers working in our laboratories. The management of NSP has realized that, in response to the challenge to the tantalum industry, maximum efforts should be exerted in technology improvement, product development, and equipment replacement. The laboratory research has to be further strengthened. In addition to equipment and instruments, NSP decided to invest more in personnel training and information collection and dissemination. A new library is under construction and a closer connection to the national information network is under consideration.

Promoting cooperation over a wide range, such as the development of its technology, the manufacturing, marketing and use of its products and equipment is the longterm policy in the Ningxia Smelting Plant and Research Institute.

I hope that this short presentation will be of some help to those who are interested in understanding and/or cooperating with Ningxia Smelting Plant and Research Institute for Non-Ferrous Metals.

NEWS ITEM

Our long-standing member H.C. Starck has strengthened its position in the market for aluminium oxide by acquiring from Gesellschaft für Elektrometallurgie (GfE) the 50 % of the Rhina Schmelzwerk GmbH of Laufenberg, Germany, that it does not already own. Rhina, in addition to being a leading supplier of fused alumina in Europe and abroad, also operates facilities for the production of atomized ferromanganese and ferrosilicon powders as well as a number of other welding additives which are at present being marketed by HCST. Starck, an affiliate of Bayer AG, is one of the world's largest producers of metal powders and intermediates for advanced ceramics.

TANTALUM AND NIOBIUM AGAINST GLOBAL WARMING

A recent European Patent (No 483 938) granted to Sorbios GmbH described an ozone producer which uses tantalum or niobium electrodes. If restraint on the use of CFC's doesn't succeed in halting the decline of the ozone layer, could we put some of it back ?

MEMBERSHIP

The four companies elected to membership by the Thirty-third General Assembly are :

Ningxia Smelting Plant for Non-Ferrous Metals

P.O. Box 105, Shizuishan City,
Ningxia Province 753000, P.R. China.

Solikamsk Magnesium Works

St Pravda 9, 618500 Solikamsk,
Permskay region, Russia.

Technologies International

P.O. Box 528, Washington Crossing,
PA 18977, U.S.A.

Thai Pioneer Enterprise Co., Ltd.

102 Tiwanon, Tambol Banmai,
Amphur Muang, Patumtanee 12000, Thailand.

The following companies have resigned from membership :
Ashton Mining, Bayer, Concord, Liarte Metalquimica, Minex, Nava Bharat, Nigerian Mining, North American Capacitor (Mallory), Sassoon Metals and Chemicals, Climax Special Metals Fabrication, Tansitor/Waycom, Toho Titanium.