# TANTALUM PRODUCERS INTERNATIONAL STUDY CENTER

QUARTERLY BULLETIN

ISSUE No. 33

**MARCH 1983** 

FIRST QUARTER

## T.I.C. Activities

#### **CHANGE OF ADDRESS**

The office of the T.I.C. has now moved to 40 rue Washington, 1050 Brussels, Belgium. The telephone number is (02) 649.51.58, and the telex number (for the International Associations Centre) is 65080 INAC B.

#### MEETING

A meeting including the Nineteenth General Assembly of the T.I.C. will be held in Penang, Malaysia, on May 23rd to 25th 1983. The formal business meeting of the T.I.C. members and the presentation of papers will be held in the Rasa Sayang Hotel in Penang. A plant visit will be made to the tin smelter of Datuk Keramat Smelting. This company, together with Malaysia Smelting Corporation and the Thailand Smelting and Refining Company, will be the joint hosts for the meeting.

The programme for the meeting will be:

Monday May 23rd — Rasa Sayang Hotel, Penang:

- Arrival of delegates and guests in Penang

Registration with the T.I.C.

Early evening welcoming Cocktail Party hosted by the T.I.C.

Tuesday May 24th — Rasa Sayang Hotel, Penang:

- At 9 a.m., the Nineteenth General Assembly, open only to delegates of member companies
- 10 a.m. Interval for coffee, during which other delegates and guests join the meeting
- 10.15 a.m. Presidential Address by Mr John Linden, President of the T.I.C., Managing Director of Greenbushes Tin Ltd.: "Tantalum and its cost of production"
- 11.00 a.m. Dr J. Rodney Lay, Managing Director, Thailand Smelting and Refining Company, will speak on "Production of tantalum-containing slags at Thaisarco"
   11.30 a.m. Mr Yeap Soon Sit, S.A. Minerals and Thailand Tantalum Industry Corporation Ltd., will speak on "The status of the TTIC project in Thailand"
- 12.00 p.m. Dr W. Rockenbauer, Hermann C. Starck Berlin, will speak on "Processing of tin slags of various grades"

12.30 p.m. Lunch adjournment

- 2.00 p.m. The programme of presentations will resume, including:
  - Dr G. Duderstadt, Gesellschaft für Elektrometallurgie, speaking on "Vertical integration in the tantalum industry", by the Metallurg Group
  - Mr Thomas C. Barron, Ayers Whitmore and Company, on "Update of March 1982 processor demand figures"
  - Dr Abdullah Hasbi Bin Haji Hassan, Director of Seatrad, speaking on "Malaysia as a source of tantalum"
  - Mr Sivavong Changkharisi, Director General, Mineral Resources Department, Thailand, has also been invited to address the meeting
- There will be a panel discussion, open to all delegates and guests, concerning current aspects of the tantalum industry and the prospects for the near future
- 8 p.m. Banquet: all the participants and their ladies are invited as the guests of the joint sponsors.

Wednesday May 25th - Penang:

There will be a conducted tour of the Datuk Keramat smelter in Penang.

Visits can also be arranged for delegates and guests, later in the week, to tin mines and processors in the vicinity of Ipoh, Malaysia, by courtesy of Malaysia Mining corporation, and to the Thaisarco tin smelter in Phuket, Thailand.

A touring programme will be available for the ladies in the party, visiting various points of interest in and around Penang.

Rooms have been reserved for delegates and guests at the Rasa Sayang Hotel. There is a principal airport in Penang with frequent flights from Singapore, Kuala Lumpur and Bangkok.

Invitations to attend the meeting are being sent to the representatives of all member companies by the Secretary of the T.I.C., from whom further information concerning the meeting may be obtained.

#### T.I.C. NINETEENTH GENERAL ASSEMBLY

The Nineteenth General Assembly of the T.I.C. will be held as part of a two day meeting from May 23rd through May 25th in Penang, Malaysia, hosted jointly by the south-east Asia tin smelters Datuk Keramat Smelting Corporation Sdn. Berhad, Malaysia Smelting Corporation Sdn. Berhad and Thailand Smelting and Refining Co. Ltd.

The General Assembly will be convened at 9 a.m. on Tuesday May 24th in the Rasa Sayang Hotel. The agenda will be:

- 1. Welcoming address by the President, Mr John Linden.
- Minutes of the Eighteenth General Assembly (held in Brussels on October 26th 1982).
- 3. New members : elections.
- 4. Accounts for 1982.
- Motion to amend the Charter of the T.I.C.
- 6. Report of the Executive Committee:
  - (a) legal representation;
  - (b) Ayers Whitmore study:
  - (c) rotation of Committee;
  - (d) new office.
- 7. Revision of membership fees.
- 8. Budget for 1983.
- Production and processor statistics.
- 10. Twentieth General Assembly.
- 11. Other business.

The formal business meeting will be followed by a series of presentations covering various aspects of tantalum materials production in Malaysia and Thailand. A panel discussion will be held on all aspects of the tantalum industry.

On Wednesday, a tour will be conducted to one of the local tin smelters.

# President's message

### Function of the T.I.C.

Our Secretary, Mrs Judy Wickens, has completed the move to new offices at 40 rue Washington and is busy establishing a functional work area for the T.I.C. and its members.

The idea is to build a library of information on all aspects of tantalum and the companies and people in the tantalum business.

T.I.C. members will benefit from this facility by being able to arrange with the secretary suitable times to visit the office and obtain copies of information required.

The collection of statistical information from member companies will be one of the main functions of the T.I.C. Naturally the service the T.I.C. provides to its members will be largely determined by the willingness of members to supply the information required.

We have seen a good response to producer statistical data over the past five years and now processors have commenced supplying the information on shipments of all forms of processed products. A start has been made to formulate a questionnaire for capacitor manufacturers so it should not be too long before the whole membership range is reporting in one or other category. Naturally the reporting of production and shipment data is a sensitive area to most companies and the T.I.C. is utilising the services of an independent accounting firm to receive and collate the responses. In this way strict confidentiality is maintained and the industry sees total industry statistics not identifiable by regions or individual company.

In an effort to include as many producers, processors, fabricators and capacitor manufacturers as possible in our data collection system a questionnaire is included with this Bulletin. The T.I.C. requests all members to complete this questionnaire and return it as soon as possible. In addition to completing this questionnaire we request you to advise the T.I.C. of other companies who are not currently members of the T.I.C. so that they may be approached to join and their statistical data be included with the T.I.C. returns. Alternatively copies of the questionnaire may be given direct to non member companies with the request that they complete the information and return it to the T.I.C. secretariat.

The T.I.C. Committee was able to meet during December and a number of important items were discussed and some decisions

The T.I.C. will have legal representation at all Committee meetings, General Assemblies and meetings of members officially called by the T.I.C. The function of the representative will be that of an observer at meetings and his role will be to stop any discussion among members from infringing on areas of "Anti Trust" or "Trade Practices" sensitivities.

Your Committee also commissioned Ayers Whitmore and Company Inc. to update the statistical "demand on processor" data contained in their report dated March 1982. Due to the continued depressing effects on commodities during 1982 your Committee felt it advisable to provide as accurate and up to date information as possible on tantalum demand and to a lesser extent on supply. The report has a low budget and is designed to update data rather than supply new information. The report will be distributed to members prior to the Penang meeting and an Ayers Whitmore representative will be available to answer questions.

Both the above decisions taken by the T.I.C. involve expenditure and your Committee has recommended certain changes to the T.I.C. charter to accommodate the additional funding required and also to provide a better timing and presentation of financial information to members.

Should the membership pass the proposed resolutions at the 19th General Assembly, the financial year of the T.I.C. will be changed to end on the 30th June each year. In this way annual accounts and budgets can be presented at the statutory Brussels October annual meeting of the T.I.C.

A further proposal to collect fees in US dollars rather than Belgian Francs will avoid erosion of the T.I.C. finances and guard against currency fluctuations as the majority of expenditure of the T.I.C. is in US dollars.

Implementation of the above recommendations will allow the T.I.C. to pay the additional costs incurred in legal representation, statistical data update and T.I.C. Bulletin expenditure without

imposing a levy on members during these economically difficult times.

I look forward to seeing you in Penang and having meaningful discussion on these and other topics of interest to our industry.

J. LINDEN President

# Penang, Malaysia

Penang is an island located about 2 km. off the northwest coast of Western Malaysia. It is about 25 km. long by 17 km. wide, situated  $5^{\circ}$  24' north of the equator.

The town of Penang, properly called the "City of George Town", is built on the promontory nearest to the mainland. The town itself, with a population of about 450,000 people, is distinguished by fine public buildings, tree-lined roads and the pulsating life of its Chinatown. Behind the town, Penang hill rises to a height of 740 m. Near Penang hill is the tiered Ayer Item (Buddhist) temple and at Sungei Kluang, about 12 km. away, is the famous Snake Temple. The Waterfall Botanic Gardens, completely surrounded by evergreen jungle-clad hills about 365 m. high, contain an unusual wealth of flowers, shrubs and trees. It is one of the finest botanic gardens in the east.

Penang was founded on July 17th 1786, the area having been ceded to the East India Company by the Malay sultan of Kedah in 1795 by an agreement with Captain Francis Light. It was then almost uninhabited and was made a penal settlement. In 1826 Singapore and Malacca were incorporated with it to become the so-called « Straits Settlements ». In 1867, the Straits Settlements were made a Crown Colony of the British Empire.

In December 1941, Penang was bombed and occupied by the Japanese. After the British return to Penang in 1945, its pre-war status was restored. As a result of the Federation of Malaya Agreement in 1948, steps were taken toward self-government in Malaysia which culminated in 1957 with the declaration of George Town as the first city of the Federation of Malaya.

As well as being a freeport — exempt from the usual import and export duties, which has created extensive industry and bustling trade — Penang is a vacation island. Europeans, Australians, Japanese and Singaporeans have made it a mecca for pleasure. The streets are lined with shops so brimful of goods that they spill out into the streets. Trishaws roll in all directions dodging in and out of the hordes of more conventional modern means of transport, the automobile. The Malacca Straits washes the western edge of the island and nowhere is the surf more joyfully received than on the beaches of Penang. The breeze swept beaches are a paradise, particularly on days when haze hangs in the sky and the humid heat is so oppressive.

Penang is the premier resort of southeast Asia.

But Penang is only a small part of the enchanting nation of Malaysia, a nation of two parts separated by 650 km. of the South China Sea. Western Malaysia, the lower end of the Malay Peninsula, was joined by the British colonies of Sabah and Sarawak on the island of Bornea (now Eastern Malaysia) and Singapore in 1963 to form the Federation of Malaysia. But two years later, the federal legislature voted Singapore out of the union. The entire country has an area of about 330,000 square km., about the size of East and West Germany combined. Forty per cent of the area is on the Malay Peninsula, but almost eighty per cent of the population of 12.5 million people live there.

The Federation today is made up of thirteen states with the capital at the city of Kuala Lumpur, about 275 km. south-southeast of Penang. All but four of the states are ruled by sultans who gather once every five years to elect from among their members a king of Malaysia. Although His Majesty is recognized as the supreme head of the country, the essentials of the governmental functions are left in the care of the Prime Minister and two houses of Parliament. The latest King was installed in February 1981 in a splendid ceremony in which he mounts the dais in the throne room and starts his reign by removing the Kris of State from its sheath and pressing it to his lips. All is done in the name of Allah as Islam prevails in Malaysia, brought by the Arak and Indian traders in the 15th century.

Situated just north of the equator and at no point more than 150 km. from the sea, Peninsular Malaysia has a climate characterized by high humidity and uniform temperature. The prevailing winds are controlled mainly by the southwest and northeast

monsoons. The effect of the former is relatively slight, but the northeast monsoon, blowing from October through February, brings high seas and much rain. There is, however, no dry season at any time of the year. Average rainfall over the whole peninsula is about 250 cm. per year. Day temperatures in Penang are generally between 25 and 32 °C and seldom exceed the latter. Night temperatures often fall below 20°.

# Datuk Keramat Smelting Sdn. Bhd. Tin Smelters Penang, Malaysia

Tin smelting on the island of Penang was first carried on by Mr. Lee Chin Hoe, a Chinese Towkay, at 73 Dato Kramat Road as far back as 1897, presumably as a private undertaking.

In 1907 Mr. Lee Chin Hoe sold the undertaking and assets of his business to a company named Eastern Smelting Company Limited Incorporated under the Companies Ordinance 1889 of the Straits Settlements. The capital of this company was Malayan \$ 1,500,000 (equivalent to £ 175,000) and it was managed by a Board of Directors comprised almost entirely of Chinese nationals including Mr. Lee Chin Hoe with a European Managing Director. The total purchase price paid to Mr. Lee Chin Hoe was Malayan \$ 300,000 of which \$ 150,000 represented the value of the works, premises, plant and machinery, the balance representing goodwill. At that time there were four small furnaces on the premises which were said to be capable of turning out 25 tons of refined tin per day.

The successor company of the same name was incorporated in the United Kingdom on July 20th 1911, largely through the instrumentality of the late Sir Cecil L. Budd, K.B.E., who, at that time, was senior partner in the well known metal broking firm of Vivian Younger & Bond, the firm which acted as metal brokers for Eastern Smelting Company Limited up to September 29th 1961. The original capital of the British company was £ 250,000 and in 1927 the capital was increased to £ 500,000.

In 1930 it was decided to redesign the works of Eastern Smelting Company Limited in Penang on modern and up to date lines. Further major alterations in 1956, 1965 and 1980 resulted in a plant which now comprises six large reverberatory oil-fired furnaces which together with the necessary ancillary plant is capable of dealing with 80,000 tons of high and medium grade concentrates per year. Continual research is carried on in an endeavour to improve the metallurgical processes.

The company smelts tin concentrates, the majority of which are produced in West Malaysia but supplies are also received from many other countries of which Australia is the most significant. The tin metal produced from these concentrates is sold throughout the world under the well-known ESCOY brand of "Straits Tin", the largest consumers of which are in the United States of America. In addition to the tin smelting plant, the company operates a small secondary lead smelting and refining plant supplying soft and antimonial lead to automobile battery manufacturers in Malaysia and type metal to the printing industry.

From December 28th 1929, on which date Consolidated Tin Smelters Limited (CTS) was incorporated, Eastern Smelting Company Limited was its wholly owned subsidiary. But in November 1961, 45 per cent of Eastern Smelting was sold on the Malayan market to afford to the public in that country the opportunity of acquiring an interest in the company and, to further this end, the residence and control of the company was transferred from the United Kingdom to the Federation of Malaya. Then, in September 1963, CTS sold a further 4.5 per cent of the equity of Eastern Smelting. By this time the Eastern Smelting shares were being traded on the Kuala Lumpur Stock Exchange.

In February 1971, the company's assets were sold to a Malaysian resident company, Syarikat Eastern Smelting Berhad. Then, in 1975, the company was again restructured with the formation of Datuk Keramat Holdings (DKH), Datuk Keramat Smelting (DKS), a wholly owned subsidiary of DKH, and Datuk Keramat Properties (DKP). CTS then held 50.5 per cent each of DKH and DKP with the former owning the investments and smelting interests and the latter the significant property interests of the former company.

In 1975, CTS itself became a wholly owned subsidiary of Amalgamated Metal Corporation (AMC) and in 1978 AMC, in its turn, became a subsidiary of Preussag A.G. (previously Patino N.V.

had held the controlling interest). Preussag is, therefore, now the ultimate holding company of the tin smelter. Datuk Keramat Smelting is controlled by a Board of Directors, the majority of whom are resident in Malaysia. The Chairman is Tan Sri Datuk Hamzah Sendut, a prominent Malaysian business man and the previous vice-chancellor of Universiti Sains Malaysia.

# Malaysia Smelting Corporation Sdn. Bhd.

The Company became operational on 1 April 1982 when Malaysia Mining Corporation took up a 42 % share in the tin ore buying and smelting business of The Straits Trading Company Limited.

This business was at that date hived off from the other interests of Straits Trading to form a separate company with a substantial Malaysian partner as part of the re-structuring of Straits Trading Company in line with the Malaysian Economic Policy.

#### HISTORY

The Straits Trading Company Limited was originally incorporated in Singapore in 1887 for the purpose of constructing and operating an efficient smelting plant, in comparison to the many small mine level smelting units then operating throughout the Malay peninsula.

The initial smelting operations were carried out during 1887 in Telok Anson but operations in the same year shifted to Singapore where the Company started construction of the Pulau Brani smelter on a small island off Singapore.

This smelter was to continue smelting tin concentrates and residues, except for a brief period during the Japanese Occupation, through until 1970 when it eventually closed down and the site reverted to the Singapore authorities.

In 1901 the Company operations were expanded with the opening of the Butterworth smelting plant. This plant was shut down during the recession in the 1930's and again during the Japanese Occupation but, after reconstruction of the furnaces and ancillary facilities, resumed smelting in 1955 and after the shutdown of Pulau Brani, now smelts all of the materials purchased or processed by the Company.

While the smelters were originally constructed to smelt concentrates primarily produced in peninsular Malaya, at the turn of the century ore from Thailand, Burma, Australia and North America was already being processed.

Today the plant continues to receive material from Burma, Australia, Europe, Africa, South America and elsewhere as well as domestic ores, and now, with the merging of Malaysia Mining and Straits Trading interests in a single smelting unit, the plant has consolidated its position as the largest tin metal producer in the world.

#### **TECHNOLOGY**

The original concept behind the formation of the Straits Trading Company was to introduce a higher level of technology into the smelting industry with consequent cost advantages.

The reverberatory furnace introduced showed specific and clear advantages over the various Chinese shaft furnaces then in use, while the metallurgical approach in terms of process theory and control was also at a markedly higher level.

The process at Butterworth today is, in principle however, little changed from that in use when the smelter first commenced operations in 1901.

The furnaces have grown from 2-tonne units up to 75-tonne units, heat exchangers have been incorporated, and fume collection systems added. Engineering and methods have been improved and the process modified and developed to a higher level of efficiency. In essence however, the process initially installed and subsequently developed has proven itself continuously superior to alternative systems in smelting high grade concentrates and Malaysia Smelting Corporation can now reasonably presume to operate the lowest cost high grade smelter currently existing.

#### CAPACITY

Installed smelting capacity is of the order of 70,000 tonnes concentrates though throughputs are now restricted by the

imposition of production quotas in April 1982 under the International Tin Agreement.

The quota presently runs at a 36.6% cutback on concentrate deliveries which affects not only tin supply to the market but also tantalum bearing slag production.

#### STRAITS TIN

The Straits Trading Company was instrumental in setting up and has subsequently operated the Daily Straits Tin market establishing a daily price for tin ex-smelters. Presently the Penang price is the reference price for buffer stock operations under the International Tin Agreement and this bears testimony to both the quality of the product and the integrity of the market.

Straits Refined Tin produced by The Straits Trading Company and now the Malaysia Smelting Corporation is high grade contract tin on the London Metal Exchange and is of guaranteed minimum 99.85 % tin content.

#### **TANTALUM**

Tantalum bearing minerals may occur to varying degree along with tin bearing ore and may then report in the tin concentrate as it is upgraded.

Where concentration of tantalum materials is sufficiently high a separation process may be introduced, but otherwise the tin concentrate received at the smelter may contain low variable levels of tantalum.

Tantalum is recovered as a by-product in residues from the smelting of tin concentrates containing minor or trace elements of this mineral. The tantalum content of Malaysian tin concentrates is generally much lower than in Thai concentrates, and concentration in slags usually run in the 1 % to  $2\frac{1}{2}$  % range. Some higher grade slags are derived from imported ores.

Depending on market conditions such slags are sold for further processing and upgrading of the tantalum content.

#### **FUTURE**

Malaysia Smelting Corporation is well placed to develop its smelting capacity and capability with its strong volume base in Malaysia.

With enhanced base feed materials from Malaysia, the Company is in a position to develop its competitive position for overseas materials and hopes to extend its capabilities in this direction.

Tantalum remains a potentially important secondary interest consequential upon the tin smelting operations, and improvement of recoveries in this direction will be sought.

The Company hopes to become a public listed company within approximately two years' time when a further stage of restructuring is scheduled to take place.

### Greenbushes Tin Ltd.

The information for the following article has been obtained from the recently issued Annual Report of Greenbushes Tin Ltd. covering its fiscal year from mid-1981 to mid-1982.

Although production at Greenbushes Tin was affected by the continuously changing market and price for tin and tantalum, the company established new production records in these commodities. 826,000 cubic meters of ore, containing 0.7 kg per cubic meter of cassiterite and 0.112 kg of tantalite, were treated. In addition 391,000 cubic meters of tailings were treated with an average content of 0.115 kg of cassiterite, 0.09 kg of tantalite and 0.08 kg of stibio-tantalite.

At the beginning of the year, in July 1981, the main ore treatment plant was producing from run-of-mine ore on a continuous shift basis. But by October this ore was of insufficient grade and mining was shifted to higher-grade alluvial ore bodies. Then, in February 1982, the main processing plant was reduced to one shift per day treating only the nearby high-grade ore. And the new Taillings Treatment Plant reached capacity operation by October 1981 after experiencing the usual range of start-up problems. With the slump in the market for tantalite, however, the plant was closed down in January of this year and placed on a stand-by basis.

Since the alluvial deposits, to which mining was shifted, were remote from the main ore treatment plant, the ore could not be economically transported to that plant. So, a new primary treatment plant was built, demountable for easy relocation. The capacity was set at 50 cubic meters per hour.

In view of the uneconomic nature, at current prices, of the company's average grade decomposed pegmatite reserves, exploration was confined to delineating high-grade alluvial deposits. It has been established that sufficient reserves exist of this type of ore to continue production for at least two years.

The development of the new underground mine and ore bodies was continued throughout the year but with operations being reduced from continuous 24 hour per day basis to one shift only. A summary of the development follows:

Surface diamond drilling 3,950 meters
Underground diamond drilling 1,856 meters
Development ore produced 19,300 m.t.
Development waste 54,100 m.t.
Development ore treated 14,900 m.t.

A ten m.t. per hour pilot treatment plant was put into operation in February. Production tests from underground ore showed gravity-circuit recoveries in excess of 75 % for tin and 70 % for tantalum. Overall recoveries will be increased to at least 85 % with the inclusion of a flotation circuit.

The facilities for further processing of concentrates at Greenbushes were also very active during the year.

- The tin smelter operated at 75 % of design capacity for the year, smelting 1,018 m.t. of concentrate to produce 750 m.t. of tin and 122 m.t. of tantalum bearing glassyslag. The production was about 50 % above the previous year and failed to reach full capacity only because of the introduction of export quotas in the spring of 1982.
- The tantalum solvent-extraction pilot plant was closed in September 1981 when plans to proceed with a new plant at Kwinana, a town about 30 km south of Perth, were developed. But the decline of the tantalum market dictated a deferment of the new processing plant and the pilot plant at the mine site was again put into operation in May. Production since then has averaged 1,500 kg of tantalum oxide per month from leached cassiterite and stibio-tantalite ores.

The company's production for the year was a record 240,000 lb. of  ${\rm Ta}_2{\rm O}_5$  contained in all products, the result of increased production capacity coming on stream during the year, the higher grade ore being mined and the processing of tailings. As the result of depressed sales of tantalum products during the year, however, inventories of tantalum products rose significantly and at the end of the fiscal-year had a value in excess of A \$7.5 million at current market values.

Even though the recession continues, plans for development of the new underground mine have not been abandoned. Discussions and negotiations with a number of major Australian and overseas mining companies are underway with a view to establishing joint ventures to bring the company's rich tantalum and lithium deposits into production. Such ventures will lead to a much earlier development of these prospects than could be achieved if the company was to rely on its own financial resources alone.

## NRC Inc.

Located in Newton, Massachusetts, U.S.A. in the heart of the renowned route 128 "high-technology belt" around Boston, NRC Inc. is one of the pioneer tantalum processors. The company was founded in 1940 as National Research Corporation. The original objectives of the company were to engage in the research and development of vacuum technology and its use to generate new processes and products. In its early stages, notable developments were frozen orange juice concentrate (a subsidiary was the forerunner of Minute Maid Corporation) and freeze-dried instant coffee. Once developed, new businesses were sold to provide added cash flow for the growth of the company.

During the 1950-decade, the company developed specialized expertise in vacuum melting of high temperature metals. Under partial funding from the U.S. Government, National Research applied their knowledge to the development of a process for

producing titanium involving research in the reduction of the metal compounds with sodium. This interest in titanium led to work on other reactive and refractory metals, particularly tantalum. Identifying a new market for the use of tantalum powder to make capacitors, the company focused its efforts in this direction as a commercial venture. The tantalum operation grew large enough that a separate Metals Division was formed in 1959.

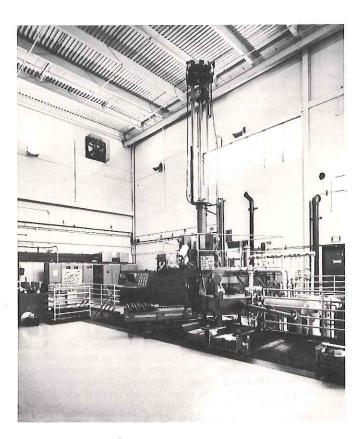
In 1963, National Research Corporation was merged into the Norton Company. The vacuum research activity became the central research laboratory for Norton and the Metals Division became an operating division specializing in refractory metals. During the ensuing years, the business grew and the Norton Metals Division became one of the three largest suppliers of tantalum products, particularly wire and powder for the capacitor industry.

The Norton Company decided during 1975, however, that the metals business no longer fitted their future business strategy and they negotiated the sale of the Metals Division. The purchasers were Hermann C. Starck and South American Consolidated Enterprises, both companies engaged in the tantalum business as a part of their over-all corporate activities. In order to retain continuity and its reputation in the marketplace, the name "NRC Inc." was chosen for the new company. The strong raw materials position and familiarity with the metals business of the two parent companies has been of great benefit to NRC.

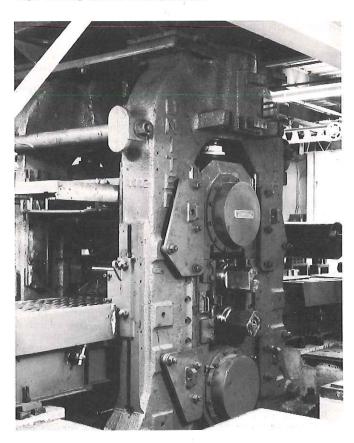
NRC is today one of the world's leading manufacturers of capacitor-grade tantalum for the electronics industry. It offers a complete line of both sodium-reduced and melt-purified, high surface area powder ranging from among the highest capacitance versions to the highest voltage-capability powders. But NRC is also a major supplier of mill products including ingot, rod, wire, tubing, foil and metallurgical-grade powder in all mesh sizes. These products provide an important source of high technology materials for the chemical processing, aerospace and nuclear industries.

During 1982, NRC has engaged in a major expansion of its manufacturing facilities, adding the most modern electron-beam melting furnace in the world, a complete rolling facility, laboratory facilities and additional office space. These complement other new facilities which include a scanning electron microscope, complete forming and fabricating systems and drawing equipment for all-tantalum capacitor cases.

The new 800 kilowatt electron-beam melting furnace was built by Leybold-Heraeus in West Germany. Delivery to NRC made news in that it was shipped from Germany to Boston by chartering a Boeing-747 and it constituted the largest single shipment ever to arrive at Boston's Logan Airport.



The new four-high United rolling mill will expand the capacity of NRC for rolled products and make it possible for the company to begin offering niobium sheet products.



With the new expansion, NRC's facilities will consist of two modern buildings with more than 100,000 square feet of manufacturing space and 20,000 square feet of administration space. The company employs about 215 people.

# The manufacture of tantalum capacitor powder

This article has been prepared entirely from patents and trade literature. It covers only the manufacture of tantalum capacitor powder. A future article will relate the characteristics of capacitor powder to the variations of processing.

The use of tantalum in electrolytic capacitors for electric circuits is greater than all other uses combined. Although there is some use of wire and foil in capacitors, powder used to make the capacitor anodes provides over 80 per cent of the electronic market consumption. In the United States, as an example, two-thirds of all tantalum use is the capacitor powder.

What is tantalum capacitor powder? It is simply basic tantalum powder which has been processed particularly to provide certain physical and chemical characteristics to make it possible to obtain desired electrical properties when incorporated into a porous anode for a capacitor.

The basic function of a capacitor in an electric circuit is to store energy so that it can be returned to the circuit when required for proper circuit operation. Its ability to store energy, the capacitance, is expressed by a simple relationship:

 $\mbox{Capacitance} = \frac{\mbox{Dielectric constant} \times \mbox{surface area of the anode}}{\mbox{Thickness of dielectric}}$ 

The advantages of tantalum in capacitors, in comparison to other materials, are due to its superiority in all three of the factors which affect capacitance: enormous surface area due to the fineness to which tantalum powder can be produced, the extreme thinness to

which a strong oxide film can be held to perform as the dielectric, and the inherent high dielectric constant of tantalum oxide.

#### PRODUCTION OF CAPACITOR POWDER

Any tantalum powder could probably be used to make a capacitor. But, to make a usable capacitor which is competitive with other types of capacitors, economically and quality-wise, the tantalum powder must be produced in such a way that the best capacitors will result.

Although rather simple, the capacitance equation demonstrates the objectives of the processing needed to obtain tantalum powder most suited to making the best capacitors. The principal objective, because of the inherent high cost of producing tantalum, must be to obtain the maximum amount of capacitance using the least amount of powder. Thus, the numerator of the equation should be as large as possible and the denominator as small as possible. The three elements of the equation can all be influenced by the manufacturing process.

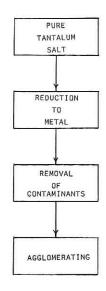
- Although the absolute value of the dielectric constant of tantalum is an inherent property of the metal, the ability to fully capitalize on it is influenced by other factors. Obviously, impurities in the tantalum, such as nickel, iron, carbon, etc., form alloys with the tantalum even if only in a very local area. Normal dispersion of these infinitesimal localities of alloy would result in some of them being on the surface of the tantalum particles. Since such alloys do not have as high a dielectric constant as the pure tantalum, the over-all dielectric constant will be reduced from what it would be if the tantalum were absolutely pure. So, it becomes evident that the processing must be such that the purest tantalum possible is obtained.
- Smaller particles have larger surface area per unit of mass than larger particles. And it is apparent that particles with irregular surfaces will have more surface area than ones with smooth surfaces. Therefore, to have the largest surface area per unit of mass, production processing must be designed to provide the smallest particles feasible with the most irregular surface.
- Any imperfection or contamination on the surface of the powder particles will prevent the formation of the thinnest film of dielectric. The film would have to be made thicker to cover up the irregularity for, if it is not, what essentially amounts to a hole in the film will exist and current will be able to leak through it. Thus, clean powder is essential and the processing must be such as to yield powder without, if possible, even a single infinitesimal speck of any contaminant on the surface of the powder particles.

The progressive improvement of capacitor powder through the past fifteen years has resulted from the intensive efforts of the processors to develop processing to maximize the purity of the tantalum, to obtain smaller sized particles with irregular shape and surface and to eliminate contaminants. Considering that the powder particles range in size from less than one micron to only a few microns, the processors are working in a microscopic world compared to that normal to the metals industry. It is no wonder that there have been times when the ultimate has been reached. But somehow, on each such occasion, the processors have evolved new methods of processing and have been able to move on to offer higher levels of capability for their powders than were ever dreamed to be possible only a few years ago.

#### **PROCESSING**

There are two types of tantalum capacitor powder. Most is produced directly from sodium reduced material but, for particular applications, some is produced by melting the sodium reduced tantalum into ingot in an electron-beam furnace and then producing powder from this purified material. EB powders are capable of forming a more perfect dielectric film because the powder particle surfaces are cleaner. Thus the film will withstand higher applied voltage before breaking down. These powders are used for high-voltage units. Sodium reduced powders are generally used when the application operating voltage is not over 20 volts whereas the EB powder is used at 35 volts or above.

For other than the melting and powdering sequences in the processing of EB-powder, the general procedure is the same for all capacitance powder.

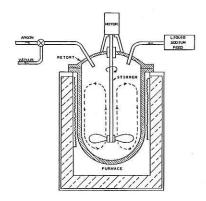


It all starts with the purest possible tantalum salt obtained by the chemical extraction of the tantalum from ore or slag. This salt must then be reduced to metal so that the purity is retained and no new contamination is added, a most difficult task. As a result, a series of steps follow reduction, washing and leaching to remove every bit of the unwanted reduction by-products. And the final step of the purification processing is usually distillation as only the high temperature in a very high vacuum will eliminate the last of the contaminants. Finally, because the super-fine particles, literally dust, are so difficult to handle in capacitor manufacture, larger particles must be generated without losing the fine particle size and shape identity, the step known as "agglomerating".

#### REDUCTION

All tantalum metal used today is processed by reducing potassium fluotantalate (k-salt) with sodium. Although the basics of the process were developed in the mid-nineteenth century, it was only in the late 1950-decade that sodium reduction reached use commercially. Originally, only a single type of sodium-reduced powder was produced and used for all tantalum products. But evolutionary development has resulted in the ability to control the process so precisely that the production of a powder needed for a particular capacitance design begins with special processing in the reduction stage.

Sodium reduction is carried out in a strong retort usually made of a nickel-base alloy to withstand the high temperature of the reaction without contamination of the tantalum due to corrosion or erosion. The retort is equipped to hold a vacuum but means are provided so that both inert gas and liquid sodium can be introduced during the processing. A means of agitation, usually a motor-driven paddle or propellor, is also provided. External heating is required to bring the temperature of the retort up to reaction temperature. In some cases, external cooling is also required to shorten the cycle time after the reaction has been completed.



In operation, a mixture of k-salt and diluent, such as NaCl or KCl, is loaded into the retort vessel. Then the cover of the vessel is put into place and secured. The vessel is then evacuated and generally back-filled with argon. By means of external heat, the salt mixture is melted. As soon as the molten condition is attained, agitation is started to assure homogenization. Molten liquid sodium at a temperature of about 100 °C to 160° is

introduced at a controlled rate through a line connected to the retort cover. Reaction occurs as the sodium contacts the k-salt:

$$K_2TaF_7 + 5 Na \rightarrow Ta + 2 KF + 5 NaF$$

Agitation is maintained throughout the reduction cycle. The reaction mass is allowed to cool, after completion of the reaction, to ambient temperature before the retort is opened.

In order to produce powder suitable for capacitors of different designs, particles of different sizes must be produced. There are several means of controlling:

- Considerable diluents, such as NaCl, are used in the charge mixture acting as an internal heat absorber or heat sink in the system. Ratios of diluent to k-salt in the range of 0.6 to 1.0 (by weight) tend to produce smaller particles, averaging less than 5 microns, while lower ratios from about 0.25 to 0.6 tend to produce somewhat larger particles averaging 5 to 8 microns.
- Controlling the rate of temperature rise during the nucleation period is accomplished by controlling the rate of sodium addition to the retort. Particles smaller than 5 microns result when the rate of temperature rise is higher, at least 20 °C per minute. But for larger particles, 5 to 8 microns, the rate is held down as low as 10 °C per minute.
- Since particle size is also inversely proportional to the time needed to complete the sodium addition, both the time span and the rate of temperature rise can be affected by the point in the cycle at which sodium injection is initiated. Starting when the molten bath is at a low temperature consumes proportionally larger amounts of sodium for any given rate of temperature increment and consequently reduces the overall process time.
- Another important factor is the temperature of reaction. In the range from 760 °C to 850° smaller particles are produced while at a temperature from 850 °C to 1,000°, somewhat larger particles are produced.

Thus the operator has opportunities to obtain any desired result, as far as particle size is concerned, if he is able to control all of these variables. In the search for technology to produce the ultimate capacitor powders, however, modification of the sodium reduction process has been developed and is practised by some of the processors.

- Precoating the salt particles with sodium before reaching reaction temperature provides better control by eliminating some of the variability inherent in the conventional method. After sealing and purging the reactor vessel, the salt is heated under an inert gas atmosphere to about 100 °C while stirring. Molten sodium is then added rapidly. The temperature is controlled high enough to maintain the sodium in a liquid state but sufficiently low to avoid initiating the reaction. Continued agitation assures homogeneous admixture of the charge. The molten sodium permeates through the charge and coats the salt particles. Then the heat is increased to initiate the reaction. It proceeds very rapidly and the temperature builds to the range of 600 °C to 800° but the reduction is essentially complete in 5 to 10 minutes compared to 8 to 10 hours for the conventional method.
- A similar process precoats the salt crystals with sodium but mixing is accomplished to produce a paste in a mixer separate from the retort. The paste is discharged from the mixer into tantalum boats on a conveyor belt. The belt carries the boats through a cooling zone entry to a furnace in order to bring the temperature from that needed for mixing down to 50 °C or below in order to keep the reaction temperature after ignition as low as possible. Then, under a protective atmosphere, the boats pass into the reaction zone where the mixture is ignited by a glowing tantalum wire. Reaction is very fast and the mass reaches a temperature above 700 °C. After completion of the reaction, the boats move on into a cooling zone to bring the temperature down to ambient before the mixture is exposed to air.

#### POST-REDUCTION PROCESSING

Irrespective of the reduction procedure used, the resultant product is a mixture of crystalline aggregate of tantalum metal embedded in solid potassium fluoride and sodium fluoride. This mixture of fluorides is not very soluble in water and is, consequently, very difficult to separate from the tantalum metal. In addition, there may be some remaining unreacted k-salt and other fluorides of tantalum even though in relatively small amounts. Since all of these salts, even if remaining in only tiny amounts in the final

tantalum powder, would be detrimental to proper electrical properties, they must be removed.

The mass from the reduction is crushed into fine powder and then washed in an elaborate leaching procedure. Generally, it takes as many as twenty steps of alternate washing with leaching reagents and rinsing to obtain desired purity. In order to increase solubility of the salts, as well as to remove any metallic contaminants picked up during reduction, many different reagents are used. The reagents most frequently described in the literature are aqua-regia, aluminium sulphate, aluminium chloride, sulphuric acid, hydrofluoric acid and hydrogen peroxide.

After washing, the powder is dried, often in a vacuum dryer. It is then classified, separating out fractions which are either too coarse or too fine to produce the desired end product. Often several lots are blended at this point to produce a specific particle size distribution.

The powder can now be used directly to produce capacitor anodes and this is sometimes the case particularly when the desired powder is coarse enough to flow easily. Since, however, it is desirable to enhance the surface area in relation to the volume in order to increase the capacitance, most processing today includes further treatment.

Such treatment consists of "agglomerating". Powders are prepared by heating them in a high vacuum, preventing oxidation and contamination, to a temperature ranging from 1,200 °C to 1,550°. This procedure results in a partial sintering action in which the powder particles are partially adhered to each other but without loss of individual identity. Care is taken, after agglomeration is completed, to assure that the mass remains in vacuum or in an atmosphere of inert gas (argon or helium) until it has cooled to room temperature. Unless this is done, considerable oxygen will be absorbed from the air and danger of catastrophic oxidation will exist.

The resulting particulate porous mass can then be crushed to form agglomerated polynodal powder granules of adhered particles with greater bulk volume and lower bulk density than unagglomerated powder of the same particle size. The powder should be processed with a single crushing action, however, rather than using a severe milling action in order to preserve the agglomerated condition.

The final powder is then only classified and blended, if necessary, to be ready for testing to evaluate electrical properties and for use in making porous sintered anodes for capacitors.

A following article in a future issue of the "Bulletin" will detail some alternative processing, particularly the production of EB-powder, and will relate the parameters of processing to the specific properties of the end-product, capacitors.

# Corrosion of tantalum by liquid metals

The information for this article has been obtained from the report, "Corrosion Data Survey on Tantalum", compiled by M. Schussler, Chief Scientist of Fansteel Inc.

Liquid metals, by virtue of their high heat capacity, excellent conductivity, low viscosity and relatively low vapor pressure, possess many of the requisites for heat-transfer media. The demand for compact, high-level power sources has given impetus to their utilization. The actual application of liquid metals for this purpose, however, has been limited by the corrosive properties of the metal.

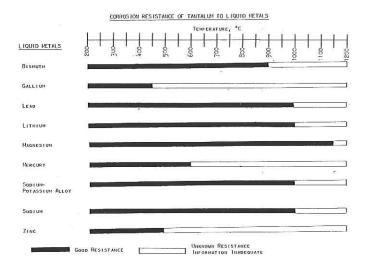
The mechanisms by which attack takes place are as follows:

- Simple solution alloying between liquid metal and solid metal,
- Intergranular penetration,
- Impurity reaction, and
- Temperature-gradient and concentration-gradient mass transfer.

The severity of attack by these mechanisms may be markedly increased by increasing temperature. Since operating temperatures in excess of 700 °C are desirable in most cases, refractory metals, including tantalum and niobium, are particularly useful materials of construction for containing liquid metals. The temperature range of usefulness begins at temperatures where

conventional superalloys still have nominal mechanical strength but just do not have the required resistance to corrosion by the liquid metals.

Tantalum generally shows good resistance to most of the low-melting liquid metals.



The excellent compatibility of tantalum with liquid metals can be degraded by impurity elements, but there are controls by which compatibility can be assured.

The presence of oxygen as an impurity in either the tantalum or the liquid metal has been responsible for most of the dramatic and catastrophic corrosion which has been observed in tantalum-alkali metal systems. In many cases the removal of oxygen from the tantalum by the alkali-metal is accompanied by intergranular and transgranular attack. The presence of oxygen as an impurity in sodium, potassium and cesium can also destroy the inherent corrosion resistance of the tantalum. Oxygen can react to form compounds which, if soluble in these alkali metals or removed mechanically by the flowing metal in a dynamic system, can lead to an accelerated rate of attack.

The effects of oxygen on the corrosion resistance of tantalum can be modified by alloying additions such as zirconium and hafnium. These elements form more stable oxides than the tantalum. As alloy constituents, zirconium and hafnium remove oxygen from solid solution by precipitating zirconium oxide and hafnium oxide, thereby increasing the threshold oxygen concentration associated with attack. Heat treatment is necessary to develop the corrosion resistant condition by enhancing the precipitation of the oxide phases.

# Thailand Smelting and Refining Company

During the early part of the 1960 decade, Union Carbide Corporation launched a programme to identify and secure a single source of tantalum which would secure their needs. At this time, Union Carbide was a major tantalum processor and expected to be the largest in the world. An extensive survey was made of the tin reserves in Thailand. Because of the high tantalum content in the tin concentrates Thailand was quickly recognised as potentially the largest undeveloped source of tantalum in the world. It was found, however, that only a limited amount of tantalite could be extracted from the tin concentrates and that at least 80 % of it remained in the concentrates.

The Thai tin concentrate production was shipped abroad for smelting, mostly to Penang. Since, at that time, the tantalum processing industry was not using tin slag extensively as a source of tantalum, the ores from Thailand with high tantalum content were, in general, smelted along with concentrates from Malaysia, Indonesia and other sources. The resultant slag did not have a high enough tantalum content to meet the needs of Union Carbide.

Union Carbide decided, therefore, to build a tin smelter in Thailand which would use only the Thai tin concentrate and which would be operated to maximize the tantalum content of the slag. Thaisarco was born as a Union Carbide subsidiary with significant ownership by Thai nationals.

The smelter was built on the island of Phuket, off the southwestern shore of Thailand in the area of the greatest tin mining activity. The plant, which began operations in 1967, consists of three reverberatory furnaces for smelting tin in the conventional fashion. For slag reprocessing, however, an electric-arc furnace was installed to recover the residual tin values from the slag. Since electric furnace processing did not require the addition of significant fluxing material, the tantalum content of the slag was actually enhanced. The resultant product, granulated by quenching in water, was a glassy, finely divided slag containing from 11 % to 14 % Ta $_2{\rm O}_5$ .

By 1970, Union Carbide corporate policy with respect to tantalum changed and their plans to be a tantalum processor were abandoned. Not having a major interest in tin production, per se, Union Carbide set out to find a partner who did have an interest. As a result, a joint-venture was formed in 1971 with Billiton International Metals. Finally, at the end of 1973 Union Carbide sold their remaining interests in Thaisarco to Billiton.

The smelter has a capacity to process 50,000 tons of tin concentrates. The tantalum produced in tin slags amounts to about 25,000 pounds  ${\rm Ta}_2{\rm O}_5$  per ton of tin metal produced. As a result, Thaisarco, with production of 500,000 pounds to 900,000 pounds  ${\rm Ta}_2{\rm O}_5$  content in slag per year, is the largest single source of tantalum materials in the world.

### Bulletin

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