

## Activities of the T.I.C. - Sixth General Assembly

The 6th. General Assembly of the T.I.C. took place in Brussels in the conference room of Cie Geomines, on Tuesday 12th. October 1976.

Of the 20 members, 18 were present in person and two represented by proxy. Additionally there were 6 prospective new members from Japan, USA and Europe, two of whom were elected to join. The other four are expected to join shortly. There were also guests including a representative from the US Bureau of Mines in Washington.

The General Meeting reaffirmed its charter as a scientific organization with the objective of promoting information and development of tantalum and related products such as columbium. It will continue to make public any news of interest through its quarterly Bulletin and it has entered into discussions with the US Bureau of Mines and the Commission of the Euro-

pean Communities to cooperate in the collection and administration of statistics.

The 7th. General Assembly of the T.I.C. will be held in Winnipeg Manitoba Canada, on May 3rd 1977, the first of the rotating meetings agreed upon during the last Assembly. This meeting will include a visit to the mining operation of Tantalum Mining Cooperation of Canada at Bernic Lake Manitoba. It was furthermore decided to sponsor a General Symposium on tantalum in Europe during the spring of 1978.

The meeting received and discussed the detailed report prepared by outside consultants during the last 12 months on tantalum markets with specific data and forecasts of production and consumption through 1980. The Assembly decided to further improve the understanding of the industry through continuation of such research.

## Approved by-law amendments

During the T.I.C. Fourth General Assembly on September 18, 1975, an extraordinary General Meeting of the members approved changes in the by-laws of the T.I.C.

- Changes in the membership requirements to include other segments of the tantalum industry than just the tantalum producers.
- Provision to increase the Executive Committee from three to five members.

These amendments were drafted and submitted to King Baudouin of Belgium in accordance with the Belgian law under which the Tantalum Producers International Study Center is chartered.

By Royal Ordinance of June 11, 1976 the amendments of Articles 5, 7, and 13 of the Charter were approved. They now become a part of the basic Charter of the T.I.C.

## T.I.C. study - Phase II

As a part of its continuing effort to develop an overall organizational strategy and an implementation program, the T.I.C. engaged the services of Emory Ayers Associates, the New York consulting group who made the T.I.C. Study- Phase I, to study the worldwide tantalum outlook from 1976 through 1980. The study has been completed and provided to both T.I.C. members and non-members who subscribed to support it.

The report is the first comprehensive attempt to define the tantalum material demand and supply. As such, it is an important step for the T.I.C. toward its educative objective and should serve to heighten awareness of the market and the important issues surrounding it.

The study was conducted by a team of consultants in the United States and Europe, supplemented by interviews conducted during the Phase I study in Africa and South America. More recent interviews were held with end users, equipment fabricators, merchants, tantalite miners, and smelter operators. Additional discussions took place with government officials, World Bank personnel, and others with insight into the tantalum market. All available data on tantalum demand and product shipments were reviewed, as well as end product useage and forecasts. Supply information and forecasts were provided by producing companies.

The report is divided into four chapters, of which

### T.I.C. SIXTH GENERAL ASSEMBLY

On October 12, 1976 the Sixth General Assembly of the T.I.C. convened in Brussels. Twenty members were in attendance. Others attending included two new member applicants and four guests who are considering membership. In addition to discussion of the general business on hand, a presentation was made by Mr. Emory Ayers and Mr. Tom Barron, both of Emory Ayers Associates, of the T.I.C. Study - Phase II.

The next General Assembly will be held in Winnipeg, Manitoba, Canada on Tuesday, May 3, 1977.

- Chapter 1 assesses demand for tantalum products.
- Chapter 2 develops the worldwide raw material outlook.
- Chapter 3 describes the role of scrap in the tantalum industry.
- Chapter 4 presents the combined supply and demand outlook and draws conclusions.

The conclusions of the report show the demand for each tantalum product segment of the market adjusted for economic cycles. Capacitor and cemented carbide products will continue through 1980 as the major uses of tantalum. Product design and product mix effects will, however, have significant impact on the actual requirements. Improvement in the quality of tantalum powder and reduction in the average size of capacitors will tend to flatten somewhat the demand even though the number of capacitor units will greatly increase. Some substitution in the cemented carbide products will effect the demand in this segment of the market. Nevertheless, there will be a continuing increase in the tantalum demand through the five year period.

The sensitivity of demand as a function of the tantalite price has been assessed. The reaction time after price increases has been evaluated so as to determine the impact on demand before 1980 if near future material price increases should happen.

Supply data shows some growth and the growth potential has been evaluated at various possible tantalite price levels. The possible use of low-grade tin slags (those containing less than 2.5 % Ta<sub>2</sub>O<sub>5</sub>) has



been evaluated as a potential source of material in the event that the supply of tantalite, higher grade tin slags, and tantalum containing columbites should be inadequate to meet the needs of the market. The effects of the lag in scrap availability exaggerating the extremes of both material shortage periods and material surplus periods have been considered. Current excess inventories of raw material will also contribute to the material supply.

The results of this study are recommendations to the T.I.C. to:

- Continue and enhance efforts to educate companies and other organizations interested in the tantalum business to the nature of the market.
- Organize an approach to study the tantalum reserve situation worldwide to result in an implementable long-term development plan for the industry.

## The production of solid tantalum capacitors

On March 20, 1975 at the Third General Assembly of the T.I.C., Mr. Peter Fenwick of Union carbide U.K. Limited, Electronics Division, made a presentation to the members concerning the production of solid tantalum capacitors. The presentation is being printed for the benefit of the readers of the "Bulletin" who were not in attendance at that time. Union Carbide has requested that this paper should not be reprinted from the "Bulletin" without the expressed permission of Union Carbide U.K. Limited.

Tantalum has been used in the manufacture of capacitors since the late 1950's and it is this application which accounts for 60 % of its current useage.

There are several features of the metal which favour its use in capacitor manufacture. It is a chemically inert material possessing a non-crystalline oxide of exceptional stability and high dielectric constant which can be formed electro-chemically in a very thin layer.

Tantalum can be produced in powder form, allowing the manufacture of a porous slug which offers a very large surface area in a small volume.

These factors considered in the context of the classical formula for a simple parallel plate capacitor, namely

$$C = \frac{K}{4\pi} \cdot \frac{A}{D}$$

where C = Capacitance.

A = Common surface area of plates.

D = Distance between plates.

and K = Dielectric constant of the material separating the plates.

show tantalum to be an ideal capacitor material in terms of volumetric efficiency.

Add to this the long term reliability of the solid tantalum capacitor and the end product is made even more attractive.

There is now a multitude of tantalum powders on the market for use in capacitor production, however all are produced by either electron-beam production or by a sodium reduction process.

Powders produced by electron beam processing are inherently of higher purity than those produced by sodium reduction alone. Consequently, they have even higher breakdown voltages than the latter and they are used mainly for the production of higher working voltage devices or

those devices with extreme reliability requirements.

The two production methods give rise to particles of two distinctly different physical forms. Electron beams powders are typically of a chunky grain structure whereas sodium reduced powders are almost "coral-like" in form and offer a much larger available surface area per unit weight of powder. Consequently less sodium reduced powder is needed to manufacture a given capacitor than would be the case if an electron beam powder were employed. Since sodium reduced powders are also less expensive than electron beam materials, the drive toward the use of the former material is self-explanatory.

No matter which tantalum powder is used, the production techniques employed are essentially the same. Tantalum powder is mixed with a suitable binderizing agent and pressed into cylindrical or rectangular slugs; a length of tantalum wire being simultaneously inserted into the slug. The binder facilitates bulk handling. However, since it is a source of impurities, it must be removed. This is achieved by the use of a dewaxing furnace which may or may not operate under a vacuum, depending on the type of binder used.

An alternative approach is to press slugs without binder which means that the subsequent binder removal is unnecessary.

The tantalum slugs are then sintered under a high vacuum at temperatures in the region of 2000 °C depending on the type of powder being used and the type

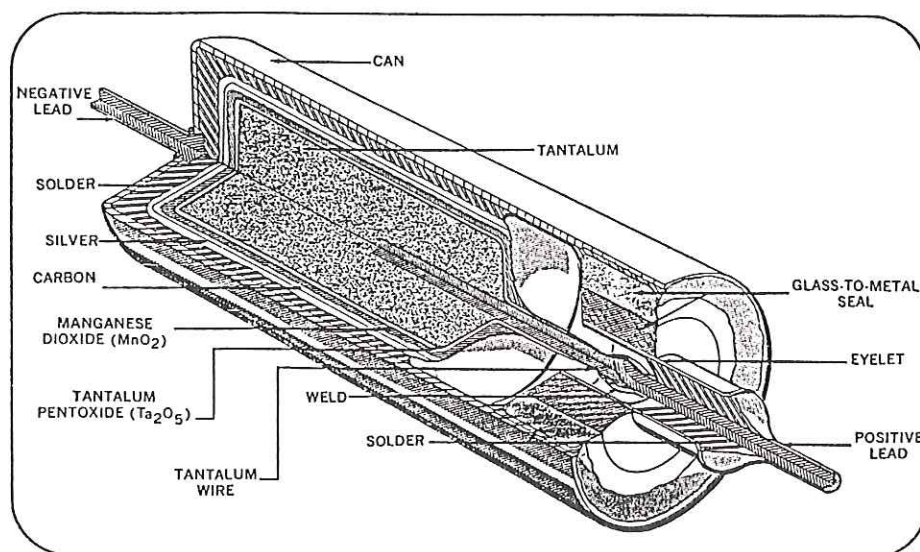
of capacitor to be produced. The sintering temperature and the times are critical since they determine the size of the slug, its density, the size of the pores and its purity. The resultant coherent, porous structure is known as the anode as it forms the positive electrode of the finished capacitor.

The next stage in the manufacturing process is to electro-chemically form a layer of tantalum pentoxide over the entire surface of the anode. This is achieved by anodic oxidation of the anode in a dilute acid solution. The thickness of the pentoxide film is approximately  $25 \times 10^{-10}$  meters per applied volt and it controls both the capacitance and the working voltage of the finished device. This film forms the dielectric of the capacitor.

Impregnation of the solid electrolyte, manganese dioxide (a semiconductor), is achieved by immersing the formed anodes in a solution of manganese nitrate, which soaks into the pores, and subsequently converting the nitrate to manganese dioxide by pyrolysis in a furnace regulated to a temperature of approximately 300 °C. The immersion/decomposition cycles are repeated several times to achieve a uniform build-up of electrolyte. However, the repeated exposure to elevated temperatures can cause minor defects in the dielectric and it is usually necessary to repair these by utilizing a variation of the formation process to reform the failure sites.

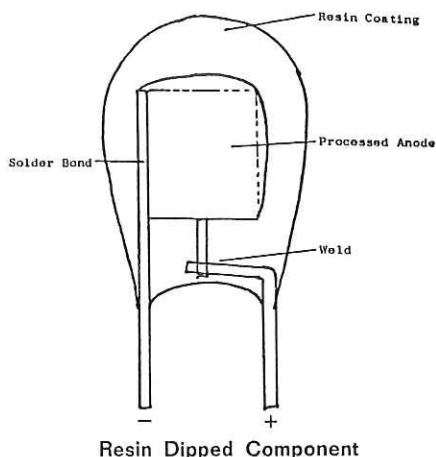
The counterelectrode consists of a layer of carbon and a layer of silver. The carbon coating is deposited by soaking the anode in an aqueous solution of graphite and drying it in an oven. The coating of silver is achieved by dipping the anode in a suspension of silver in thermoplastic resins and curing this into it. The cured silver provides a surface which will solder either to a metal can in the case of a hermetically sealed device or to a lead wire in the case of a plastic encapsulated device.

A cross section of a hermetically sealed device and a schematic diagram of a resin encapsulated device are shown in the illustrations. In both cases the positive lead wire is welded to the tantalum wire.



Cross Section of Metal Case Capacitor (Hermetically Sealed)





The hermeticity of the metal can capacitor is obtained by using a glass to metal seal. In a resin encapsulated device, the resin coating is applied either by immersing the component in a liquid resin system followed by a curing of the resin or by employing fluidized bed techniques. Low cost capacitors with precise dimensions for automatic insertion into printed circuit boards are obtained by molding with plastic resin. Finished parts are generally "burnt-in" to repair defect sites. Heat generated at the defect site causes conversion of the semi-conducting manganese oxide to an oxide of higher resistivity

thereby isolating the fault and reducing leakage current across the dielectric. This self healing mechanism makes the solid tantalum capacitor an extremely reliable device.

Metal case devices typically operate over the temperature range  $-65^{\circ}\text{C}$  to  $+125^{\circ}\text{C}$  although devices are produced which can operate over temperatures in excess of this range. These devices are used mainly in applications requiring extreme reliability such as heart pacemaker units.

Resin encapsulated devices operate over the temperature range  $-55^{\circ}\text{C}$  to  $85^{\circ}\text{C}$  and they are used mainly in consumer applications such as home entertainment products, electronic watches, and automotive products. Rapid growth of both sections of the market is assured.

The use of tantalum capacitors in many consumer applications has been achieved by a combination of small size, reliability, and low prices due to high volume production techniques. Rising tantalum prices have prompted producers to develop materials with higher CV (Capacitance  $\times$  voltage) per gram. Capacitor producers are also developing techniques to utilize these materials so that costs can be controlled and tantalum supplies conserved. In this way the position of the solid tantalum capacitor in the market place will be consolidated and the future of the tantalum industry will be assured.

## OCCURRENCE OF COLUMBITE AND STRUVERITE

In peninsular Malaysia, the tantalum/nio-bium minerals are mined at Semiling Bakri, occurring as ferro-columbite as the major mineral or second to columbite. The Nb:Ta ratio is approximately 4:1 with a specific gravity of about 5.5. Higher tantalum contents have been recorded. Small amounts of columbite are also in dredge concentrates, tin dressing tailings, and in final cassiterite concentrates ready for smelting.

The occurrence of struverite is widespread throughout the country in association with cassiterite and ilmenite in heavy mineral concentrates. The content is seldom of a significant percentage. The Nb:Ta ratio is approximately 1:1 and the specific gravity about 5.5.

## GENERAL DRESSING METHODS

The current methods employed in dressing the tantalum and niobium minerals are gravity concentration, electrostatic and high tension separation. Separation by froth flotation has not been successful. With the Malaysian assemblage, gravity concentration alone cannot make a separation but it is used to obtain a heavy-mineral concentrate free from the lighter minerals of tourmaline, topaz, and quartz. Electrostatic and high tension separation can usually effect an excellent separation between the columbite and struverite as conductors and the zircon and monazite as non-conductors. The main problem encountered in the dressing of these minerals is the overlap of magnetic susceptibility between the tantalum minerals and the altered ilmenite.

Columbite is recovered as a by-product of tin mining. Presently, it is produced mainly in the Semiling and Bakai areas. Estimates cannot be made for the quantities of struverite available as a properly conducted survey has yet to be made.

## BEH Minerals Sdn. Bhd.

BEH Minerals Sdn. Bhd. of Lahat, Perak, Malaysia was established as a local minerals processor in 1970. The company operates plants in Selangor and Perak. During the last five years, they have become the largest Malaysian processor and exporter of tin-associated minerals: ilmenite, monazite, zircon, xenotime, and struverite. A joint venture, Malaysian Rare Earth Corp., has been formed with Mitsubishi Chemicals of Japan. This company chemically upgrades locally processed xenotime.

The processing business begins where the mining ends. BEH is not directly involved in tin-mining and so acquires as its feed materials the discards from tin-sheds and the tin mines. These discards are methodically stripped of the mineral content through the employment of wet gravitational, pneumatic, and magnetic/electrostatic methods of ore separation.

The tantalum containing ores handled by BEH Minerals are columbite and struverite. Columbite is more of a trading material and the quantity handled is in the region of 40-50 tons per year, almost 50 % of the total annual Malaysian export. The struverite (tantalum rich rutile) is processed entirely in the BEH plants. The first commercial lot of struverite was produced in 1972. Early setbacks in production methods and quality control, however, prevented regular production until 1975. Actual exports to date have been as follows:

Year	Struverite Export
1973	30 tons
1974	25 tons
1975	92 tons
1976	100 tons (planned)

The typical analysis of struverite is:

Ta <sub>2</sub> O <sub>5</sub>	12-13 %
Cb <sub>2</sub> O <sub>5</sub>	12-13 %
TiO <sub>2</sub>	56-57 %
SnO <sub>2</sub>	4-5 %

## Japanese tantalum industry and market

*The following information has been derived from an article published in the "Rare Metal News", Tokyo, dated March 24, 1976.*

### HISTORY

Tantalum metal processing was started in Japan in 1960 by Komatsu Seisakusho, Showa Denko, Shinetsu Chemical, and Nihon Soda. In 1961, Tokyo Denkai started production to join the race with other four companies. However, this did not last long and Komatsu Seisakusho gave up production in May 1965 and then Nihon Soda in March 1966. Tokyo Denkai also withdrew from basic refining to concentrate only on metal processing using scrap as source material. It was found that tantalum refining was not profitable.

By 1967 the number of producers of tantalum metal powder had been reduced from five to two; namely, Shinetsu Chemical and Showa Denko. With the dramatic increase in ore prices in 1967-1968, both of the remaining companies also gave up basic refining and began to import relatively cheaper potassium tantalum fluoride from West Germany and U.S.A. In these years the Japanese demand for tantalum powder was only 16-17 tons of which 4-5 tons were imported. At this low volume, basic refining was not economic.

During 1971, Showa Denko formed a joint venture on a 50/50 share basis with Kawecki Berylco Industries. The new company was named Showa KBI. Potassium tantalum fluoride and other tantalum pro-



ducts for processing in Japan are imported from Kawecki Berylco in the U.S.

Shinetsu Chemical was left as the only Japanese metal producer up to the present. But they have now decided to discontinue refining and sales operations during 1976 as their outworn facilities would require a further investment of hundreds of million yen in order to continue production. Due to a tight capital budget and foreign price pressure, Shinetsu has no choice but to close the plant down rather than run the risk of accidents caused by the deteriorated facilities.

For many years Mitsui Metal Mining has operated a basic refining plant using imported ores to produce tantalum oxide, tantalum carbide, and columbium oxide. They remain the only direct user of tantalum ores, purchasing most of the material direct from the mines rather than going through foreign traders.

Whereas purchases were formerly made from as many as ten different source countries (1972-1974), 1975 purchases of 62 tons were 68 % from Australia, 16 % from Malaysia, and 8 % each from Mozambique and a U.S. trading source.

## CURRENT MARKET CONDITIONS

Shinetsu's withdrawal has left the market entirely free for the large U.S. producers to make inroads. The market is being serviced by Showa-KBI, NRC Inc. through their agent — Shinku Yakin, and Fansteel through a joint venture with Greg Gary International. Although to date the Japanese market has been supplied on about an equal share by KBI and NRC, Fansteel is trying to get into the Japanese market. The Fansteel products have been handled by Mitsui Buson and it is expected that such will continue with Fansteel/GGI providing technical services.

Sinku Yakin, while representing NRC in the supply of capacitor powder, also produces mill products in the form of sheet, wire, rod, etc., made from scrap imported from the U.S. In view of the scrap price increase to Y 18,000 per kilogram, almost double the 1974 price, they are now considering the use of imported metallurgical grade powder.

Tantalum product prices in the Japanese market are higher than those in foreign markets by 15-20 % due to custom duties, freight, etc. Higher prices of ores and slags as well as basic refining costs are expected to further increase product prices during 1976. It is feared by the tantalum consumers in Japan, such as producers of capacitors and end-use mill products, that they will no longer be competitive in the international market. Mr. H. Okuda, Managing Director of Nihon Denki is quoted expressing his concern: "Although our monthly production of tantalum capacitors has increased to 25,000,000, our product prices are even now 20 % higher

compared with those of imported products and it is getting more difficult for us to defend our share in the domestic market from foreign producers."

The difficulty of Japanese product producers dependent on foreign refined materials is evidenced by the fact that potassium tantalum fluoride prices have increased from Y 3,580 per kg. in 1973 to Y 5,970 in 1975, an increase of 67 %. Tantalum metal powder prices, however, have increased by only 22-25 % from Y 27,000-28,000 per kg. to 33,000-35,000.

## FORECAST AND DEVELOPMENTS

The increased demand in 1976 is forecast as requiring 48 tons of capacitor grade powder and 4 tons of metallurgical grade powder for a total of 52 tons, an increase of about 47 % over 1975. It is expected that 21 tons of this will be imported (mostly from NRC) and the balance will be produced by Showa KBI. Since Showa KBI states that they have the production capacity of 80 tons per year, they can supply this quantity without difficulty. Wire, foil, and other tantalum products used by the electronics industry is forecast to reach 10.7 tons, an increase of 20 % over 1975.

## New tantalite prospect in Brazil

Companhia de Estanho Sao Joao del Rei is carrying on a Brazilian-wide prospecting program for tantalite-bearing pegmatites while continuing their routine mining operations at their current Nazareno site. A promising prospect has been located and claimed for an area of about 50 square kilometers located between Belo Horizonte and Juiz de Fora, about two hundred kilometers directly north of Rio de Janeiro in the state of Minas Gerais. The claims have been duly registered some time ago by the D.N.P.M., the Brazilian Department of Mines. The claims cover the whole of an important series of pegmatite outcrops

General industrial products are forecast at 9 tons, 184 % over 1975, and the combined tantalum carbide and oxide consumption will be 22 tons, slightly under the 1975 level of 23.6 tons.

Mitsui Metal Mining developed a plan to produce tantalum metal at their Miike plant after their merger with Sankin Rare Earth. Since very advanced technology is required to meet the specifications of the capacitor producers; it is possible that they might acquire Shinetsu's know-how. There have been no discussions in this direction, however, between the two companies.

Tohoku Kogyo Research and Development Laboratory, on a joint basis with the Thai government, has been developing processes to obtain tantalite, columbite, zircon, etc. from the Amang dumps in Thailand. According to Mr. T. Okada of the Resources Development Department, the Thai Amang dumps contain 2-3 % niobium, 0.2-0.3 % tantalum. An upgrading process has produced a columbite containing 36 % niobium and 10 % tantalum. Since Thailand consumes no columbite directly, the production will be for export. It is expected that this processing will be a supply source for the Japanese tantalum processing industry in the future.

within a gneissic country rock, lying in a similar fashion to the Nazareno deposit. A classic survey is being developed by drilling and cutting to obtain samples for assaying. It is still too early in the exploration to draw any conclusions, but all signs seem to point to the possibility of a workable deposit. In fact, several of the bodies surveyed have indicated promising mineral concentrations of decomposed stuff. The proposition and further development, however, is dependent upon the evolution of the tantalite market and the recognition in that market of the increasingly difficult conditions of tantalite mining operations.

### T.I.C. MEMBERSHIP

During the Sixth General Assembly on October 12, 1976  
the following companies were elected to membership in the T.I.C. :

#### REFINEMET INTERNATIONAL COMPANY

One Main Street  
Mapleville, R.I. 02839 - U.S.A.

#### TENNANT TRADING LTD.

9 Harp Lane  
Lower Thames Street  
London EC3R 6DR, England