TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

PRESIDENT'S LETTER

The meeting of the T.I.C. to be held in Japan in October of this year will give the industry an opportunity to review the previous twelve months. As we all know, the electronics industry of Japan is very important to that country and, indeed, to the world. The T.I.C. counts among its members many of the Japanese firms which manufacture or use electronic components, so it is appropriate for our association to meet on their home ground. The situation of the Japanese electronics industry will be a topic specifically addressed by a speaker from MITI, as part of our technical programme spanning both metals and their uses.

We are especially pleased that a number of our Japanese member companies will support us by sponsoring the gala dinner, always a highlight of our conference, and that Nichicon will host a field trip to its capacitor plant in Adogawa.

The city of Kyoto will be a highly attractive setting for our annual meeting, providing memorable places for our delegates to visit in their free time. And Nara, an ancient city with delightful surroundings, is only a short journey away. We are fortunate that our industry has a presence in so many different parts of the world. This enables us to arrange our series of Assemblies in fine and varied places yet maintain the tradition of visiting an appropriate company facility.

The statistics collected from the member companies of this association have shown that shipments of products other than capacitor powder and mill products held up well in 2001. The mill products category includes capacitor wire, of course, so as the market recovers both mill products and the powder category should show increased activity. The niobium processors' shipments continue to show a steady increase.

In order to be able to release the totals in a timely fashion, the T.I.C. needs all reporting members to make their returns promptly. Forms are sent out in June and December, and to the capacitor manufacturers also in April and October: please give them your close attention.

I encourage you to sign up for the General Assembly, and I hope to see you in October.

Axel Hoppe President

KYOTO IN OCTOBER 2002

The T.I.C. will hold its Forty-third General Assembly in Kyoto, Japan, on Monday October 7th 2002, as part of a meeting from October 6th to 8th at Kyoto Hotel Okura.

The T.I.C. registration desk will be open on Sunday October 6th. All participants must pre-register in advance with the T.I.C. secretariat, delegates who have not pre-registered will not be accepted.

A reception will welcome all delegates, guests and accompanying persons on the evening of Sunday October 6th from 6 to 8p.m.



Kinkaju-ji (Golden Temple)

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The General Assembly will be held at 8.30a.m. on Monday October 7th, for delegates of member companies.

After a coffee break, guests will join the group for the opening of the session of technical presentations. Papers will cover recent events in the industry, new developments in tantalum and niobium and applications of both metals, with a focus on the industry in Japan (see below for Technical Programme). With a break for lunch, the technical presentations will continue until about 5p.m.

In the evening of Monday, all the delegates, guests and accompanying persons are cordially invited to the gala dinner by Japanese member companies of the T.I.C. The Japanese companies which have kindly offered to sponsor and host the dinner are:

Nichicon
Cabot Supermetals
Fujitsu Media Devices
H.C.Starck-V Tech
Hitachi AIC
Matsushita Electronic
Mitsui Mining & Smelting
NEC Tokin
Nippon Chemi-con
Sanyo Electronic Components

On Tuesday **Nichicon Corporation** will offer a guided tour of its capacitor plant at Adogawa, followed by lunch. This will be an excellent opportunity for those not familiar with the tantalum capacitor manufacturing process to see an up-to-theminute factory.



Kiyomizu-dera



Five-storey pagoda, Tō-ji (Photos by JW)

Sightseeing tours and a social programme will be arranged for those accompanying the meeting delegates. A tour of the historic sites of Kyoto is planned for Monday, with a trip to Nara on Tuesday.

Invitations will be sent to the nominated delegate of each member company three months before the meeting. Others interested in attending, if they have not yet been in touch with the secretariat, should contact the T.I.C. as soon as possible: 40 rue Washington, 1050 Brussels, Belgium, telephone +32 2 649 51 58, fax +32 2 649 64 47, e-mail info@tanb.org.

All participants must pre-register, and the last date for booking is September 6th.

TECHNICAL PROGRAMME

Japanese government support of the new metal business in Japan; update on recovery of the IT business in Japan a senior executive of METI

Tantalum and niobium – a year in reviewMr C. Edward Mosheim, Technical Promotions Officer, T.I.C.

Topics on anti-oxidation coating technology of niobium-based superalloys

Professor Toshio Narita, Hokkaido University

Status of niobium-based superalloys developmentDr Akio Kasama, JUTEM – Japan Ultra-High Temperature
Materials Research Center

Alternative reduced tantalum powdersDr Karlheinz Reichert, H.C. Starck GmbH

The effect of metallurgical properties of tantalum target on sputtering performance

Mr Ichiroh Sawamura, Innovative Materials Development Center, Nikko Materials Co., Ltd.

The development of solid organic polymer niobium electrolytic capacitor

Mr K. Mitsui, Director of Engineering, Nichicon Corporation

Tantalum supply chain and problems of management Mr David Paull, Sons of Gwalia

Tantalum capacitors – applications review Mr John Prymak, Kemet

The global tantalum market - what the future holds Dr Andrew Cole, Metal Bulletin Research

TANTALUM AVAILABILITY - 2000 AND BEYOND

by William A. Serjak, H.C. Starck Inc., Hady Seyeda and Christian G. Cymorek, H.C. Starck GmbH

This article has been condensed from a paper by H.C. Starck, and was not written by the T.I.C.

In 1999 and 2000 many end-users were unable to obtain all the tantalum capacitors they wanted. Long after the problems of availability and pricing have gone, there are still companies that believe tantalum is in short supply and is being allocated. Some believe it will again become a significant supply problem for them. This paper will explain the actions taken by the tantalum supply chain to ensure that problems with tantalum availability are a thing of the past.

MARKETS FOR TANTALUM

Tantalum capacitors are pervasive because they add functions to circuits that other dielectrics cannot. Tantalum capacitors are used in laptop computers and cell phones because they have very high volumetric efficiency and are very reliable. Tantalum capacitors, also, make laptops and cell phones lighter in weight and make the batteries last longer. Tantalum capacitors have a stable oxide film at temperatures from -55°C to +125°C. This makes them ideal for automotive applications where "underhood" temperatures can cause other capacitors to fail.

Tantalum metal is used in turbine engine alloys to allow aircraft and land-based turbines to operate at higher temperatures and thus give higher efficiencies. Tantalum is also used in corrosion-resistant applications such as heat exchangers, reaction vessels and vessel liners for the chemical process industry.

Tantalum oxide is used to change the refractive index of lenses to make them thinner, and to make x-ray imaging brighter with less dosage for the patient. The oxide is used in sputtering targets for chemical vapor deposition. Tantalum oxide is a major ingredient in fiber optics for making dense wavelength divisional multiplexing possible.

Starck estimates that 68% of all tantalum demand is tantalum

powder, wire and furnace hardware for tantalum capacitors. 2% goes into sputtering targets for electronics, and 9% goes into other electronic applications. Nearly 80% of the tantalum material is used in electronic end markets. The chemical process industry uses about 2% because tantalum has the same excellent corrosion-resistant properties as glass, but can be used at much higher temperatures.

The electronic industry is very important to tantalum businesses. Its influence is great on the entire tantalum supply chain because when electronics grow by 10%, the entire tantalum supply chain must grow by about 8%. Few other supply chains are so greatly affected by a single segment of their business.

Tantalum shipments in the year 2000 were 2267-metric tons, or more than 5 million pounds. This was a record-breaking performance for the tantalum supply chain. From 1992 to 2000, production of tantalum grew 242%, or 17% per year. Tantalum is an element; it can neither be divided into sub-parts, nor be made from other elements.

TANTALUM SUPPLY CHAIN

The tantalum supply chain is more than tantalum capacitors. It begins with tantalum ore in a mine. Mines may take from one to two years to respond to changes in the end market. The response time depends on the type of deposit, the resources of the miner and the magnitude of the change.

In 1999 and 2000, communications within the supply chain were typical of most supply chains: vendors and customers talked to each other. Normally this is a reasonable method of communication. But the tantalum supply chain is long and the demand was changing very rapidly in response to the growth in electronics.

Original Equipment Manufacturers (OEMs) and Contract Service Manufacturers (CSMs) who make cell phones, computers, and automotive electronics see the supply chain as it involves their customers, their suppliers, and tantalum capacitors. This is because the entire supply chain normally works very well, and the processors and miners do not make the news. But the supply chain also includes the tantalum metal processors and tantalum miners.

Processors buy the ore, concentrate it, refine it into an oxide, convert the oxide into a chemical compound (K₂TaF₇), and then reduce the 'K2' to the pure metal form. Processors sell powder and wire based on the needs of the capacitor makers.

Processors translate the end user needs into long-term take-orpay contracts with the miners. Contracts are needed because mining requires large capital expenditures. The processors are responsible for tying the two ends of the chain together. They assume the majority of financial responsibility for the continuity of the supply chain. A realistic view of the supply chain is shown in Figure 1. Ore and scrap are sometimes handled through brokers.



Figure 1: Tantalum supply chain

SHORTAGE - REAL OR PERCEIVED?

Was the shortage for tantalum capacitors real or perceived? To the end-users who could not buy all the capacitors needed to meet their demands, it did not make any difference if the shortage was real or perceived. But to understand the performance of the tantalum supply chain and return its credibility, the question is extremely important. Was the supply of tantalum capacitors and tantalum powder sufficient to meet the real demand of the electronic equipment being sold?

In 1997, prices for tantalum capacitors were low. Capacitor manufacturers were not expanding because profits were low. Low prices encouraged OEMs to design tantalum capacitors into new products. Tantalum capacitors have outstanding performance for the price. During the recovery of 1998, inventories of tantalum capacitors (and nearly all passive components) fell to very low levels.

At the same time, cell phones, hardwired communications, Internet capital spending and computer markets all began double-digit expansion. The NASDAQ boom brought capital to end markets for dot.com expansion. At the same time OEMs were moving significant amounts of their business to CSMs. These events had a significant impact on the tantalum and other electronic supply chains.

In 1999 and 2000, the supply chain became more complex than usual. The electronics press highlighted the tight supplies and increasing prices. This precipitated a scramble for capacitors and tantalum across the chain. Double ordering started and speculators entered the supply chain. Speculators disrupt the normal flow of material by buying at prices higher than existing supply chain prices, waiting for the prices to go up, then returning the material to the supply chain at a much higher price. Speculators entered the supply chain at almost every level: they bought and sold out-dated tantalum capacitors; they bought and sold tantalum scrap; and they bought and sold tantalum ore.

SUPPLY CHAIN MODEL

To determine if there were enough tantalum capacitors to meet the true demand, we built a model of the tantalum capacitor supply chain. Worldwide shipments of tantalum capacitors are directly related to the dollars of electronic shipments. The data for U.S. shipments are readily available. Worldwide capacitor shipments are highly correlated with U.S. electronic shipments. Using U.S. data does not minimize the importance of shipments in Europe, Japan and Asia. The period from 1992 to 1997 was relatively stable so it will be used as the baseline. Once established, the relationship between U.S. electronic shipments and worldwide tantalum capacitor shipments will be used to obtain the real (calculated) demand for tantalum capacitors from 1998 to 2002.

The data show that from 1992 to 1997 there were between 33 and 40 tantalum capacitors used worldwide per \$1000 of U.S. Electronic Equipment Shipments. For our base calculation we will use 38. (The data for the U.S. electronic equipment shipments are available from the U.S. Department of Commerce. Capacitor shipments are the estimate of H. C. Starck Inc.) In Figure 2, the figure of 38 capacitors per \$1000 is used to calculate the worldwide demand for tantalum capacitors from 1998 through 2000. The calculated demand for worldwide shipments is 15, 17 and 19 billion in years 1998, 1999 and 2000, respectively.

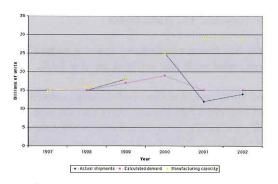


Figure 2: Tantalum capacitor shipments and manufacturing capacity

Figure 2 also shows the actual shipments were 15, 18 and 26 billion. Thus, the surplus was 1 billion in 1999, and perhaps as much as 6 billion in 2000. This is an accumulated surplus of over 7 billion in two years. The 7 billion excess capacitors represent a 6 to 9-month supply of tantalum capacitors. These 'surplus' capacitors are in OEM and EMS equipment, DSL boards, cell phones, internet equipment and other products that have not yet been 'shipped to the consumer'. Because of the shortage in 1999 and 2000, capacitor manufacturers (also Figure 2) added capacity (yellow triangles) that brought the industry capacity to 29 billion capacitors, by putting new plants on stream or by expanding. Some believe the present capacity could be 36 billion pieces. With the new capacity, there is sufficient capacity to meet a market growing at 20% average annual growth rate until the year 2005, or a 10% AAGR until the year 2010. The tantalum supply chain responded very well by shipping 6 to 7 billion more tantalum capacitors than were actually sold in the end market. There is plenty of capacity in the tantalum capacitor supply chain.

Having established that there were more than enough tantalum capacitors built during 1999 and 2000, it is then important to determine if this is also true for tantalum powder.

TANTALUM POWDER SHORTAGE?

The situation in the tantalum powder industry before 1998 was nearly the same as that for tantalum capacitors. Powder prices and profits were low and the processors were not adding capacity. When the end markets began to grow late in 1998, inventories of powder fell and demand began to increase. In 1999, the end markets increased their estimates of demand significantly. By 2000, the demand was only met because processors and miners liquidated their work-in-process (WIP) inventories. Immediately, new take-or-pay contracts were signed with the miners to ensure a continuation of supply.

The calculation for tantalum powder is based on its relationship to U.S. electronic equipment shipments. The period between 1992 and 1997 was, again, the standard and stable period and will be used as the baseline to determine the real demand for tantalum powder. The tantalum powder shipment data are provided by Tantalum-Niobium International Study Center. The data show that during this period about 1.8 grams of tantalum powder were shipped for every \$1000 of U.S. electronic equipment.

The demand for tantalum powder can be calculated using this relationship. During 1998, the calculated demand was 670 metric tons (1.47 million pounds). In 1999, the calculated demand was 820 metric tons (1.8 million pounds). In 2000 the calculated demand was 925 metric tons (2.04 million pounds).

The calculated demand for tantalum powder, the actual shipments of tantalum powder, the excess tantalum powder and the new capacity added by the tantalum powder processors are shown in Figure 3. The calculated demand for tantalum powder was much less than actual shipments. In 1999, there were 1000 metric tons (2.2 million pounds) of powder sold by the processors while the calculated demand of the end markets was only 820 metric tons (1.8 million pounds). In 2000, there were 1360 metric tons (3 million pounds) of powder shipments and the calculated demand was only 925 metric tons (2.04 million pounds).

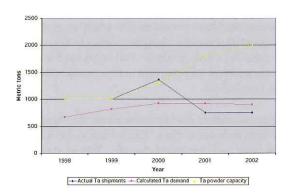


Figure 3: Tantalum powder calculated demand, shipments and production capacity

Surpluses of nearly 900 metric tons (2.0 million pounds) were produced in 1999 and in 2000. This powder was used in capacitors that are in inventories of routers, cell phones, computers, circuit boards, inventories at CSMs, and in inventories of powder within the supply chain. Two million pounds is nearly a one-year demand for tantalum powder at 2002 demand. With the demand for all electronic products

dropping in 2001, the surplus could last longer than one year.

In Figure 3, the yellow triangels show the capacity of the tantalum powder industry. It increased to about 1300 metric tons (2.9 million pounds) in 2000 and to 1800 metric tons (3.97 million pounds) in 2001. The industry will have capacity to ship 2000 metric tons (4.4 million pounds) in 2002. The increases in capacity are all part of long-term projects that had been planned and were initiated during 1999 and 2000. Even with no additional investment, there is enough capacity in place to meet the demands of electronics until 2006 or perhaps 2010.

The response of the tantalum powder processors was sufficient to meet the real demand. Shipments of tantalum powder were in excess of calculated demand. The excess shipments created a surplus of approximately one year. The tantalum processors added capacity to ensure the long-term viability of the tantalum industry.

The tantalum market in 1998, 1999 and 2000 was very near equilibrium. The inflated demand estimates, the double ordering and the bad press enticed speculators to enter the market. When this happened the supply chain failed to meet the needs of all of its members. There are real life situations that illustrate the same effect. If you live on the East Coast of the U.S. you are familiar with hurricanes. When the weatherman forecasts that a hurricane will arrive the next day, people flock to the stores and buy every flashlight battery, every loaf of bread and every bottle of milk. The entire stock of these products is exhausted. When things return to normal, no one ever questions the supply chain for batteries, bread or milk. Does anyone stop buying batteries, milk and bread? In 1999 and 2000, the tantalum supply chain received a hurricane warning.

TANTALUM RAW MATERIALS

There were sufficient quantities of tantalum capacitors and tantalum powder to meet the real demand. 7 billion surplus tantalum capacitors were made. There must have been enough ore, whether in process or in inventories. But the shortage warnings in 2000 caused the miners to take action. They took very positive action to ensure there were enough raw materials for the supply chain.

What types of tantalum raw materials are available? Where do they come from? How much is available? What is the future of the raw materials? What has been done to ensure that the future is stable?

Tin slags were a major source of tantalum for many years because tantalum is sometimes associated with tin ore. The decline in the demand for tin has greatly slowed tin mining, and the economic value of the tantalum in the tin ore is too low to justify mining of tin ore just for tantalum. It is doubtful that tin mining will be a significant source for new tantalum raw materials in the future.

Placer deposits are usually the cheapest to recover: they may be weathered, naturally concentrated and more easily mined.
Placer deposits can be brought to the market in about one year.

Hard rock mining requires high capital expenditure and is the most expensive. It usually involves deep hole or open pit mines where over-burden must be removed. Hard rock mines can be brought to market in two to three years. Most of the new

tantalum deposits are of the hard rock type. The largest active mines for tantalum are hard rock mines in Australia.

Carbonatite deposits are massive ore bodies that have a high content of complex mineralization which is expensive to develop. These resources are still untouched.

Tantalum ore is classified as either 'reserves' or 'resources' based on the degree of exploration and value of commercialization. A commercial deposit is one that can be exploited at or near 'today's' market value for the mineral, whatever that value may be. Commercialization changes as the value of the commodity in the market place changes. What is a 'non-commercial resource' today can become a 'commercial reserve' tomorrow. Market prices and availability are in control.

A 'reserve' is a deposit where the quantity and quality are defined as commercial. Usually, commercial deposits or reserves constitute less than a 30 years' supply. Exploring for reserves beyond 30 years does not make good economic sense because of market uncertainty and taxation practices.

Tantalum reserves, shown in Figure 4, contain 36 400 metric tons (79 million pounds) of tantalum. This is sufficient to supply the entire industry for 16 years. The two largest mines in the world are Greenbushes and Wodgina, both in Australia and owned by Sons of Gwalia. SOG is primarily a tantalum mining company and it has made major investments to create a secure, long term supply of tantalum.

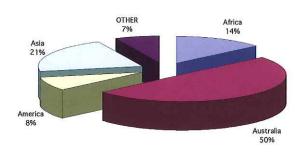


Figure 4: Worldwide tantalum ore reserves, in 2000

■ Africa ■ Australia □ America □ Asia ■ OTHER

'Resources' are raw material deposits that have been roughly defined, but may not be of commercial value at the present time. Tantalum resources are estimated at 287 000 metric tons (633 million pounds), sufficient for more than 125 years based on a consumption of 5 million pounds per year. The major resources are in Australia, Asia, Africa, and the Americas. The largest resources are in Australia: the political stability there will ensure the stability of tantalum raw materials for years to come. The size of the resource makes tantalum a more dependable raw material than oil or copper.

Although the resources of copper – estimated at 63 years - are significantly less than tantalum, copper remains a major raw material for the electronics industry. There are no concerns about shortages of copper. Tantalum compares very favorably with other raw materials.

Some resources have not proved to be commercially feasible and may not have infrastructure to produce any significant amounts

of material. But the resources have been identified and the estimated mineralization is available for the future growth of the industry. In 1997 and 1998 low ore prices and the low demand for tantalum products greatly reduced the exploration for tantalum ore. This changed greatly in 2000 and 2001.

The tantalum supply chain has another source of raw material: the U.S. Defense Logistics Agency (DLA) has been buying, storing and selling the government stockpile for many years. The original purpose of the DLA was to provide a stable supply of key materials in case of war. Tantalum is a key material because it is used in military electronics, cutting tools and the nuclear industry, and because there are no significant reserves within the U.S. The DLA may still have 580 metric tons in inventory.

ORE PRODUCTION

In 1998, the world's mines produced about 2 million pounds of tantalum per year. By the year 2000, the output had increased to over 3 million pounds. In 2003, the mines could be able to produce more than 5.5 million pounds, assuming the demand for enlarged capacity is there.

In 2000, the 3 million pounds of tantalum produced by the mines are much less than the 5 million pounds shipped to the tantalum capacitor manufacturers. During the year 2000, a significant amount of material came from the inventories of miners, processors, and the DLA. It is not unusual for miners and processors to carry an inventory that is greater than twelve months demand. In addition the recycle of scrap increased to 25% or 30%.

The shortages in the supply chain caused the major source of tantalum ore, Sons of Gwalia, to increase its production capability so now it alone can produce more than 2 million pounds of tantalum. In the year 2003, the production capacity of the mines could be more than 5.5 million pounds; the supply chain demand in 2003 would not require production at full capacity.

RECOMMENDATIONS

The tantalum supply chain needs better communications. Miners and processors need to communicate their plans for capacity, expansion, stockpiles and their long term policy to ensure that all understand that the supply is secure. Processors need to communicate technical improvements in their products that will benefit the supply chain.

Capacitor manufacturers need to communicate their long-range plans with both ends of the chain. OEMs need to share their long-range plans and anticipated changes. They must also communicate their requirements for information from the supply chain. All segments must find a way to communicate openly about the planning to ensure a stable supply chain.

Starck intends to establish links to the OEMs so they may understand the processing and mining part of the supply chain, and thus they may be reassured that the supply chain will be there to support their long term plans. Starck will communicate to OEMs any changes to production capacities, and to reserves and resources. Miners and processors will have an incentive to expand their capabilities to meet market demands, and capacitor makers will again utilize their production capacity fully. OEMs will design tantalum capacitors into applications that make

technical and economic sense. In this way Starck believes there need never be another shortage of tantalum.

TANTALUM - AN HISTORICAL PERSPECTIVE

by C. Edward Mosheim, Technical Promotions Officer, T.I.C.

The year 2002 marks the 200th anniversary of the discovery of the element tantalum. Anders Gustav Ekeberg is credited with finding the element in two different minerals, specifically, tantalite from Kimito, Finland and yttrotantalite from Ytterby, Sweden.

Anders Gustaf Ekeberg was born on January 16th 1767 in Stockholm, Sweden, the son of Joseph Erik Ekeberg, a shipbuilder for the King ¹. He attended boarding schools from the age of 10, developed a knowledge of Greek literature, and became a scholar in science, mathematics, and art at the age of 14. He attended and graduated from Uppsala University in 1788 upon the presentation of a thesis entitled, 'Oils Extracted from Seeds'. After graduation, he travelled throughout Germany, returning to Uppsala in 1790².

Ekeberg became a professor and began his teaching career at Uppsala University in 1794³. He was an early convert to the system of Antoine Lavoisier and introduced these concepts into Sweden. Lavoisier is credited with the classification of the elements and is considered an early architect of the Periodic Table⁴.



Ekeberg published his first paper on chemistry that same year and went on to distinguish himself as an analytical chemist. His publications were highly respected: two of the most notable were 'The Present State of Chemical Science' and 'The Advantages which Medicine Gains from the Most Recent Discoveries in Chemistry'.

The combination of his interest in analytical chemistry and fascination with minerals led him to perform analyses on a number of different specimens in 1802 from Ytterby, Sweden, and Kimoto, Åbo, Finland. The tantalite sample from Kimoto, Åbo, Finland was found in 1746 and believed to be a tin garnet at the time. [Comment - A study of the map of Finland suggests that the sample of tantalite from Kimoto was from the same general area as Kemiö Island, Finland, the site of the current Rosendal Project of Tertiary Minerals.] The mineral sample from Ytterby was yttrotantalite, a rare earth tantalate containing a high concentration of yttrium and tantalum. Experiments on these minerals resulted in the isolation of a 'new metal oxide' which Ekeberg called 'tantalum' due to the difficulty of decomposing the mineral in acids and isolation of the oxide of that element.

The name tantalum is derived from Tantalus, king of Phrygia in Greek mythology, and son of Jupiter, who was condemned to eternal frustration when he had to stand in water up to his neck, but the water receded as he attempted to drink.

Poor health in later years resulted in reduced activity concerning the publication of papers, except for those related to the analysis of gadolinite, topaz, and an ore of titanium. A young student named Jöns Jacob Berzelius assisted Ekeberg with the analysis of the waters of Medevi. Berzelius would later bring significant fame to the University of Uppsala, and it would be Berzelius in 1814 who would defend Ekeberg's claim to the identification of tantalum^{1,5}.

The identities of columbium and tantalum became confused when, in 1809, William Hyde Wollaston declared that columbium and tantalum were the same element, on the basis of limited experimental data.

Anders Gustaf Ekeberg died on February 11th 1813 at the age of 46.

A controversy erupted when one Thomas Thomson published an English translation of a memoir about Ekeberg written by Berzelius. The word tantalum was changed by Thomson to columbium. Berzelius wrote that 'Without wishing to depreciate the merits of the celebrated Hatchett, it is nevertheless necessary to observe that tantalum and its properties in the metallic as well as in its oxidized condition were not known at all before Mr Ekeberg'. And he continues 'The reason for the name tantalum (derived from the story of Tantalus) is still more valid if one adds that metallic tantalum, reduced to the finest powder, is not attacked by any acid, not even by aqua regia, concentrated and boiling'.

Berzelius and others agreed that the element discovered by Hatchett and Ekeberg were one and the same, but Berzelius believed that tantalum was a better name, possibly due to the influence of Ekeberg on his career.

It was not until 1840-1845, that Heinrich Rose^{1,6}, a pupil of Berzelius, was able to establish that there were two similar metallic elements in a variety of columbite and tantalite samples, and he named them tantalum and niobium. Niobe is the name of the mythical daughter of Tantalus. It was also apparent from the variety of density differences that this mineral series contained two elements with similar chemical behaviour. The work of Rose was not clearcut however as he appears to have mixed the order of the elements and assigned the name niobium to the heavier metal. During this same time frame, he believed that he had discovered a third metal which he called 'Pelopium', Pelops being the brother of Niobe. The confusion seems to be the result of his isolation of two different chlorides of niobium, namely, NbCl₅ and NbOCl₃.

These differences between niobium and tantalum were finally resolved through the efforts of Charles Bloomstrand and Jean Charles Galissard de Marignac by the determination of the differences in the vapour pressures of niobium and tantalum pentachlorides.

Between 1825 and 1900, numerous mineralogists and chemists identified various minerals containing niobium and tantalum. They studied the extraction and separation of niobium and tantalum and the preparation of many chemicals of these two elements.

The successful extraction and separation of niobium and tantalum is generally credited to J.C.G. de Marignac based upon the use of



his procedure on a commercial scale in the early days of industrial production of chemicals and metal of these two elements. It must be noted, however, that the so-called Marignac process was not successful in isolating a pure niobium product, the primary impurity of concern being titanium, a common impurity in many of the minerals.

Jean Charles Galissard de Marignac^{6,7,8} was born on April 24th 1817 in Geneva, Switzerland. He studied in Paris under Dumas, and in Giessen at the laboratory of Liebig. In 1841 he became professor at the University of Geneva. His scientific work is noteworthy because of his determination of the atomic weights of the rare earths and he is also credited with the discovery of ytterbium and gadolinium. He died on April 15th 1894.

Another of the accomplishments of this scientist was to develop a procedure for the determination of the niobium and tantalum contents of minerals containing these two elements. Various researchers attempted separations but were not successful primarily due to the similar chemical behavior of the same chemical form of these two elements. For example, the chloride compounds of niobium and tantalum are readily contaminated by oxygen. This results in a negative impact on the quantitive separation of the pure pentachlorides of these two elements.

J.C.G. de Marignac achieved success by his discovery that the complex fluorides of niobium and tantalum exhibited significantly different solubilities in dilute acid solution.

Marignac 'opened' the ore containing niobium and tantalum by first pulverizing it, following this with fusion in sodium hydroxide in an iron crucible. This was in turn followed by leaching the fusion mass in water. Filtration isolated the insoluble sodium tantalate and sodium niobate. The wet cake was digested in hydrochloric acid to convert the tantalates into the hydroxides of both tantalum and niobium, plus a sodium chloride waste stream.

The mixed hydroxides were dissolved in hydrofluoric acid to form a solution of H_2TaF_7 (heptafluorotantalic acid) and H_2NbOF_5 (oxypentafluoroniobic acid). The investigations by Marignac showed that the solubilities of the respective potassium salts of these two acids were quite different, with the tantalum salt being 12 to 13 times less soluble than the niobium salt at 15°C. The addition of potassium ion in the form of KF or KCl to the mixed acid solution of these fluoride complexes of tantalum and niobium led to the crystallization of potassium tantalum fluoride, K_2TaF_7 , with the niobium salt remaining in solution along with other impurities that were not removed during the hydrochloric acid leaching process. The potassium tantalum fluoride salt was dried and was then ready for conversion to tantalum metal.

The eventual development of this process on a commercial scale was the key to the successful production of 'pure' tantalum metal powder, from which ingots and then other fabricated shapes were developed. Modifications to this process provided pure tantalum oxide.

Marignac also studied the ammonium, zinc, and copper salts of the heptafluorotantalic acid.

Another scientist^{9,10,11} of the early 1900s who has had a great

impact on our understanding of the chemistry of tantalum compounds was Clarence William Balke (born March 29th 1880, died 1948), who obtained his PhD from the University of Pennsylvania in 1905. At least one chapter of his thesis was based on his work on preparation of complex fluoride salts of tantalum, i.e., (NH₄)₂TaF₇, Na₃TaF₈, K₂TaF₇, Rb₂TaF₇, CuTaF₇ · 4H₂O, LiTaF₆ · 2H₂O, Cs₂TaF₇, and C₅H₅N · HF · TaF₅. He remained at the University to serve as a research fellow and instructor through 1907. Balke's research centered on rare elements, one accomplishment being the determination of the atomic weight of niobium in 1907.

Later that year he moved to the Urbana-Champaign Campus of the University of Illinois as an associate professor in inorganic chemistry and while in residence there he determined the atomic weight of tantalum. His continued interest in 'rare metals' resulted in an organized research group of students. The proximity of the University of Illinois to Chicago, coupled with the knowledge base established by Dr Clarence Balke, eventually resulted in the development of a consulting relationship with Dr Carl Pfanstiehl, the founder of what became known later as the Fansteel Corporation. That story will be continued in a later issue.

The actual preparation of a very impure tantalum metal powder was first accomplished by Berzelius 12 in 1825 by the reduction of K₂TaF₇ with potassium metal under a salt layer in an iron crucible. Rose was able to produce a purer powder in 1866 by the sodium reduction of Na₂TaF₇. In both cases the reduction masses required leaching with water and acids to remove byproducts so that a powder could be isolated. Marignac believed the powders formed by this process were tantalum hydride, not the metal. One can speculate that these early attempts at powder preparation resulted in metal with not only numerous impurities, but also high oxygen content due to the high temperatures of reaction. There is no reference in these early studies to the use of a protective atmosphere that would inhibit the reaction of metal powder particles with oxygen. Any attempts to produce ingots would have resulted in brittle material that would have been difficult to fabricate into such articles as wire.

Sometime between 1900 and 1903, Drs Werner von Bolton and Otto Feuerlein¹³, chemists working for Siemens and Halske Company of Berlin, produced relatively pure powder by reducing tantalum oxide with carbon. The purity was sufficient to be worked into sheet and wire, and it was this technical achievement that led to the first commercial application for tantalum in the early 1900s - in lighting devices.

The incandescent lamp¹⁴ was developed by various researchers during the period from about 1870 to 1900 when individuals such as Edison began production of lighting devices for commercial sale. These early lamps contained 'carbonized' bamboo filaments in rectangular cross-section in a hairpin shape¹³. These bamboo filaments were used until about 1900, when they were replaced by the development of metal filaments.

An early attempt¹⁵ (about 1895) to use a high temperature metal for filaments in lighting was conducted by J.W. Aylsworth of Newark, NJ. He investigated and patented filaments of niobium, tantalum, molybdenum, titanium, and zirconium. The procedure involved the heating of a base material (carbon



core) and depositing one of these high temperature metals on the carbon substrate by the reduction of a volatile chloride of the metal with hydrogen.

Osmium, a member of the platinum group, was the first metal used commercially as a filament for incandescent lighting. This development was through the efforts of Dr Carl Auer von Welsbach¹⁶ (1858-1929) who is credited with the invention of the first metal filament light bulb. He took out two patents for the manufacturing of filaments, on January 15th 1890. He recognized that high melting metals were required. His process involved the mixing of osmium powder with either rubber or sugar and kneading the mixture into a paste. The paste is then 'stamped' through a small diameter nozzle to form filaments that were then dried and sintered.

The osmium filament resulted in a 57% reduction in the use of electricity, less blackening of the glass, a whiter light due to the higher filament temperature, and a longer life span.

Commercial production began in 1902 with the introduction of the 'Auer-Oslight', shown below. These lamps were not sold, but rented so that the lamp would eventually be returned to the manufacturer. The cost in 1903¹⁷ was \$1.25 and the user would recover \$0.19 if the burned out lamp was returned to the manufacturer within 18 months. The filaments were 'retreated' and reused. These lamps could only be used in the vertical position with the loop of the filament hanging down, until suitable filament support systems were developed. Widespread use was never achieved due to cost, but the successful use of high temperature metals was now proven.

The efficiency of carbon elements 18 was initially about 1 lumen per watt with improvements to a maximum of 3 lumens in 1898. Osmium filaments produced about 6 lumens and tantalum filaments about 7 lumens per watt.

Tantalum, with a melting point of 2996°C, was the replacement element metal for osmium^{11,17,18}. It was used as a drawn wire based on the work of von Bolton and Feuerlein at Siemens and Halske during the period from 1903 to 1911. The first tantalum filament lamp that withstood photometric and life testing was produced on December 28th 1902. The filament was of a loop design. By July 1903, the filament diameter was successfully reduced to 0.05mm diameter and by September of that same year, useable lamps for 110 volt use were produced.

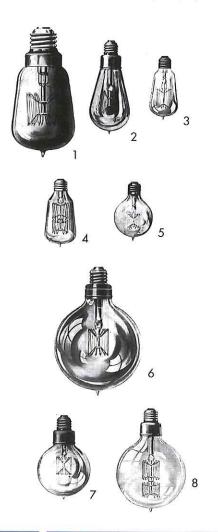
The work at Siemens and Halske resulted in numerous patents for these devices and negotiations with General Electric and the national Electric Lamp Company resulted in those two companies acquiring the exclusive rights to manufacture the lamp in the United States on February 10th 1906, at a cost to General Electric of \$300,000. Worldwide production of tantalum filament lamps began in about 1903 and continued through about 1911 when the improved technology of tungsten filaments caused tungsten to become the replacement for

tantalum. Worldwide production has been estimated at about 100 million units¹².

Various properties of tantalum made this metal a very good candidate for lamp filaments. In addition to its light output in comparison to the bamboo filaments, it exhibits a higher resistance than tungsten, making shorter lengths of wire comparable in output to tungsten. Alternatively, the use of a larger diameter wire can be used to achieve comparable resistance and added strength. Tantalum retains its 'softness' at high temperature, provided it is heated in a vacuum. This made it a better filament for automobile or railway lamps where vibration and impact resistance was a concern. Tantalum also becomes incandescent at 1700°C, more than 400 degrees lower than tungsten, making the lamp operation 20% cooler than tungsten. It is a 'getter' element in that it will adsorb gases at high temperature and retain them, a property which enhances its use in a vacuum, the condition in these lighting devices.

Bulletins describing the various lamps containing the tantalum filaments were available and a selection of those designs are shown through the courtesy of Edward J. Covington, retired engineer, General Electric Company, from his web site on lighting at the following address http://www.frognet.net/~eicov/tantalum.html.

The tantalum lamps contain very complicated filament designs and the intricacies of these designs for household, industrial, and locomotive lighting applications are represented in the illustrations, with descriptions following the photographs.







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The history of tantalum will be continued in the next issue of the Bulletin.

- 1. 20 candle power, 42 watts, 100-125 volts, bulb diameter = 2-3/8 inches, lamp length = 5 inches.
- 2. 40 candle power, 80 watts, 100-125 volts, bulb diameter = 3-1/16 inches, lamp length = 7 inches.
- 3. 20 candle power, 40 watts, 100 to 125 volts, bulb diameter = 2-5/16 inches, lamp length = 5-1/4 inches.
- 4. 50 watts, 200-250 volts, bulb diameter = 3-1/16 inches, lamp length = 5-1/8 inches.
- 5. 25 watts, 100-125 volts, bulb diameter = 2-3/8 inches, lamp length = 5 inches.
- 6. 80 watts, 100-125 volts, bulb diameter = 5 inches, lamp length = 7-5/8 inches.
- 7. 40 watts, 100-125 volts, bulb diameter = 3-3/4 inches, lamp length = 6-1/8 inches.
- 8. 80 watts, 200-250 volts, bulb diameter = 5 inches, lamp length = 7-5/8 inches.
- 9. Train lighting lamp, 28-34 volts, 8 or 12 candle power, 16 or 25 watts, bulb diameter = 2-5/16 inches, lamp length = 3-1/2 inches; 57-65 volts, 12 candle power, 25 watts, bulb diameter = 2-5/16 inches, lamp length = 3-1/2 inches.
- 10. Train lighting lamp, 28-34 volts, 8 or 12 candle power, 16 or 25 watts, bulb diameter = 2-1/8 inches, lamp length = 4-1/4 inches; 57-65 volts, 12 candle power, 25 watts, bulb diameter = 2-1/8 inches, lamp length = 4-1/4 inches.
- 11. Train lighting lamp, 57-65 volts, 20 candle power, bulb diameter = 3-3/4 inches, lamp length = 6-1/8 inches; 28-34 volts, 40 candle power, 80 watts, bulb diameter = 5 inches, lamp length = 7-5/8 inches; 57-65 volts, 40 candle power, 80 watts, bulb diameter = 5 inches, lamp length = 7-5/8 inches.
- 12. Headlight or side-light for electric vehicles, 28-34 volts, 16 watts, bulb diameter = 2-5/16 inches, lamp length = 3-1/2 inches; 28-34 or 57-65 volts, 25 watts, bulb diameter = 2-5/16 inches, lamp length = 3-1/2 inches.

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www.tanb.org e-mail to info@tanb.org

DLA

We thank the DLA for sending us news of its offers and awards.

A table of the Annual Materials Plan 2002 was printed in T.I.C. Bulletin 108

On April 2nd 2002, the Defense National Stockpile Center announced the award of approximately 19,500 pounds of tantalum metal vacuum grade under Solicitation for Offers, DLA-Tantalum metal vacuum grade-001. Offers were received on March 12th 2002 (see Bulletin 109). An award was made to ABS Alloys and Metals, Mexborough, England, for an approximate value of \$1.7 million. The award exhausted the amount of tantalum metal vacuum grade available for sale under the FY 2002 Annual Materials Plan.

On June 13th, the DLA announced Amendment no. 009 to its solicitation 001 for tantalum/columbium concentrates. Offers for the sale of 220 788.50 pounds of tantalum/niobium concentrates (80 437.40 pounds Ta₂O₅ contained) were requested for 10a.m. on Tuesday July 16th 2002. A section on Financial Exposure Limit was added to the solicitation, and a modification was made to the section on Responsibility Determination.

For more information about tantalum and columbium sales in Fiscal Year 2002, please contact the Directorate of Contract Sales, Defense National Stockpile Center at 8725 John J. Kingman Road, Fort Belvoir, VA 22060-6223. The telephone number is +001 7030 767 6500 and the web site is https://www.dnsc.dla.mil/.

UN REPORT

An interim report of the UN Panel of Experts on the Illegal Exploitation of Natural Resources and Other Forms of Wealth of the Democratic Republic of the Congo was submitted by the Secretary General of the UN to the Security Council on May 22nd 2002. The document is numbered S/2002/565, and an associated news release was dated May 24th.

MEMBER COMPANY NEWS

Angus and Ross

In May the international mining consultants Behre Dolbear confirmed the potential of the Motzfeldt deposit currently being explored by Angus and Ross. Angus and Ross reported that in the past three months it had not received a decision from Cabot on the options held by the latter. Angus and Ross was therefore seeking other industry partners to proceed with its development of the deposit.

AVX

AVX Corporation announced on April 17th its results for the quarter and the full fiscal year ended March 31st 2002. For the quarter net sales were \$275.1 million and a net loss of \$2.1 million was recorded. For the year, net sales were \$1249.9 million and a net loss of \$7.2 million resulted. The CEO and President stated that sales unit volumes increased in the quarter, but selling prices continued to be under heavy pressure as industry capacity remained in excess of demand. Indications were that sales would increase in the quarter to the end of June, but the company would continue to make cost reduction initiatives.

Cabot/Kemet/Vishay

On April 15th 2002 Kemet announced that it had been sued by Cabot Corporation with respect to existing supply agreements. Cabot alleged that Kemet was not accepting products offered by Cabot in accordance with a contract running from 2001 to 2003 which required Kemet to take a specified supply of powder and wire at contracted prices. Kemet denied liability and intended to defend itself vigorously.

On the same day Vishay Intertechnology also stated that it was being sued by Cabot for allegedly breaching an agreement to purchase tantalum products. Vishay said it had a sound defence and had complied fully with its obligations under the agreements. Cabot then declared, on June 6th, that its dispute with Vishay had been resolved. Amended supply agreements had been established, confirming 'Vishay's obligation to purchase tantalum products at regular intervals throughout the terms of the contracts, beginning immediately'. Subject to certain conditions, 'prices (starting in 2003) and volumes had been reduced, and the term of one of the supply contracts had been extended for one year'.

Sons of Gwalia said that these lawsuits would not affect its contracts with Cabot, and Cabot would be taking the raw materials from Sons of Gwalia which were agreed by long term contracts.

Cambior

In the first quarter of 2002 Cambior's share, 50%, of the production from the Niobec Mine was 429 tonnes of niobium. Production in the first quarter of 2001 was 364 tonnes. The company was budgeting for a production level in 2002 similar to that in 2001 (which was 1503 tonnes), with a target of 1555 tonnes for the year. The joint owners of the Niobec Mine, Cambior and Mazarin, were 'evaluating plans to increase production by 20% in order to maintain market share as world consumption of ferroniobium increases'.

Cluff Mining

In April it was reported by Metal Pages that Cluff Mining was in discussion with existing partners which might take over the Gabon niobium project from Cluff Mining. The company would then concentrate on its Blue Ridge platinum project in South Africa.

Sons of Gwalia

The General Manager of Business Development for the company, Mr David Paull, told the annual Prospectors and Developers Conference in Canada in March that a slight recovery in tantalum demand was foreseen for 2002. In 2003-2004 Mr Paull expected demand to return to its 'normal' growth rate of approximately 6-7% annually, saying this could be higher if third generation mobile phones began to sell well.

The expansion of both Greenbushes and Wodgina mines had led to the company producing a record 625 525 lb of tantalum in the quarter ended March 31st 2002, an increase of more than 30% over the preceding quarter.

Jiujianç

In April Metal Pages announced that Jiujiang had confirmed that it was planning to increase production of tantalum powder and wire 'massively'. However, it went on to say that difficult market conditions and lack of financial support had combined

to delay the company's plans, and there was no target date for the increase.

Kemet

Kemet reported net sales for the first quarter of 2002 of \$117.9 million, an increase of 0.5% over the December 2001 quarter, and a decrease of 65% compared with the March quarter 2001, in a news release dated April 22nd 2002. The company showed a net loss for the quarter of \$14.4 million, compared with net earnings of \$78.4 in the equivalent quarter of 2001.

Mr Maguire, Chairman, President and CEO, stated that total unit shipments had increased for two consecutive quarters, but average selling prices declined 9%. He anticipated that unit shipments would continue to increase, which would allow the company to reduce inventory. He observed that shipments would increase as customers' businesses recovered, and 'some electronics end markets, such as notebook computers and servers, appeared to have stabilised and begun growing again'.

Kemet's position on the leading edge of technology was featured in Digital Power Report, May 2002, which can be found on Kemet's web site. 'Tantalum is used to supply power. Tantalum capacitors store more power in less space than any other competitive alternative – which makes them essential to the delivery of highly order power to high-speed digital devices. The faster the digital logic, the more tantalum capacitors you need', says the article.

Metallurg

Metallurg reported a net loss of \$1.45 million for the fourth quarter of 2001, compared with net income of \$6.59 million in the fourth quarter of 2000. For the whole year Metallurg posted a net income of \$3.76 million, about one-third of the income for the previous year. Mr Alan Ewart, CEO, commented that operating results were good in the light of difficult conditions, and demand for niobium and tantalum alloys had helped.

For the first quarter of 2002, Metallurg reported net income of \$6.67 million, more than double that of the first quarter of 2001. Mr Ewart said that the difficult conditions persisted, and the company was responding by reducing costs and improving its management of working capital.

Mitsui Mining and Smelting

Mitsui Mining and Smelting posted a 'group net profit of 1.99 billion yen for the fiscal year ending March 31st 2002', announced Metal Pages on May 10th 2002, noting that this was a decline of nearly 90% from the previous year.

NEC

On April 1st 2002 the name of T.I.C. member company NEC was changed to NEC Tokin Corporation. The company's delegate to our association is Mr Hideo Oka.

New Millennium

In May New Millennium reported that it was continuing to work on its deposit in Safartoq, Greenland, which contained both niobium and tantalum at high grades. It was planning to use a unique and potentially low cost method of extraction, involving leaching out the pyrochlore with inexpensive acids and following this with a simple solvent extraction process.

Ningxia

On May 2nd it was reported in Metal Pages that Ningxia Orient Tantalum Industry, an associated company of Ningxia Non-ferrous Metals Smelter, had signed a joint venture with Lynas Corporation. The deal would provide funds for development of the Mount Weld deposit which contains tantalum and niobium together with rare earths.

Pacific Metals Ores and Chemicals

On January 28th 2002 the newspaper Nevada Appeal reported that Pacific Metals Ores and Chemicals/Niotan Inc. was applying for a special use permit to produce tantalum capacitor powder at Mound House in Lyon County. Niotan would not be involved in ore processing, but intended to produce 'tantalum powder from a tantalum salt, using a sodium reduction process followed by washing procedures using water and acids, followed by a heat treatment', the report continued. The firm would initially hire 62 employees, and future expansion to include production of wire and tube would require an additional 30 employees. If permits were approved, Niotan expected to be in production by the end of 2002. A decision due on February 7th was postponed until February 21st, and on that date approval was given by Lyon County Commissioners, said the newspaper.

Tantalum Australia

On May 7th Tantalum Australia announced that it had signed agreements to acquire the world rights to a patented process which it hoped would establish it as a low-cost producer of tantalum metal within 3-5 years. The process uses solid oxygen-ion-conducting membranes to convert oxides directly to metals, and was developed by Boston University's Manufacturing Engineering School. In work to date, yields approaching 100% had been achieved.

Also in May the company pursued its feasibility study on the Mount Deans deposit, and announced that its first shipment of concentrate had been delivered to an overseas customer.

Tertiary Minerals

This company is applying for membership of the T.I.C. at the Forty-third General Assembly.

It has announced that the latest drilling programme at its Rosendal project in Finland had shown tantalum grades much higher than a previous survey had indicated. These results which exceeded expectations supported Tertiary's strategy of fast tracking the Rosendal project into production. Further work on the company's Ghurayyah deposit in Saudi Arabia also continued. The company believed that tantalum inventories were being depleted and the outlook for tantalum demand was for continuing long term growth, but it would watch the market closely before deciding on its rate of production.

Symposium 2000 Proceedings available Hard cover book, 376 pages in colour Order from T.I.C. US\$200