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TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

PRESIDENT'S LETTER

I am glad to be able to tell you that plans for the forthcoming meeting in Lisbon in October of this year are progressing well.

The programme of technical presentations made during our prior meetings of the Tantalum-Niobium International Study Center is always highly informative. We are pleased to be able to present a rich and varied collection of papers full of information for those fortunate enough to be in the audience. The papers presented at the assembly in Kyoto in 2002 were of an exceptionally high quality, with good talks and excellent illustrations.

Now we are appealing to you to offer contributions to the technical session which will immediately follow our Forty-fourth General Assembly. We are looking to you, the membership, to your suppliers and customers and to the readers of this Bulletin to propose topics which will make the coming event a worthy successor to our Kyoto gathering. Subjects relating to the production of raw materials, to refining or processing and to applications of our two metals are all welcome, and I do hope that you will get in touch with me or another member of the Executive Committee with your suggestions. We have some proposals on the table, but we really do need more in order to present the quality of meeting the T.I.C. has come to expect, and we look forward to hearing from you.

As 2003 goes on, we become more hopeful of an improvement in the electronics industry and in the tantalum industry as a whole, and we are confident of the stability of the niobium business.

Josef Gerblinger, President

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1050 Brussels, Belgium.

The T.I.C. is an *association internationale*
under Belgian law.

LISBON, OCTOBER 2003



Lisbon is often described as one of the most attractive cities of Europe, so the T.I.C. is especially pleased that the Forty-fourth General Assembly meeting will be held there from October 12th to 14th 2003, centred on the Hotel Le Meridien Park Atlantic.

On Sunday October 12th the registration desk will be open during the day, and delegates will be welcomed with a cocktail reception from 6 to 8 p.m. The formal General Assembly to conduct the business of the association will take place early on Monday morning, and the rest of the day will be given over to the technical sessions with presentations covering many aspects of the tantalum and niobium industry.

In the evening all delegates and guests and those accompanying them will be invited to a gala dinner by EPCOS, the company of our current President Dr Gerblinger.

On Tuesday October 14th there will be a plant tour of the capacitor plant of Epcos at Evorá, and participants will also have a chance to see some of the historic and beautiful features of the town, which is classified as a World Heritage Site.

Sightseeing tours will be arranged for accompanying persons on Monday and Tuesday.

Invitations will be sent to the nominated delegate of each member company about three months before the event, for the delegate and all his member company colleagues. Anyone from a company which is not a current member of the association and who wishes to attend should contact the Secretary General, T.I.C.,
40 rue Washington, 1050 Brussels, Belgium;
telephone: +32 2 649 51 58,
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INDUSTRY POLICY OF THE JAPANESE GOVERNMENT

This is based on the report given to the Forty-third General Assembly meeting by Mr Hiroki Ishigaki, Deputy Director, Non-ferrous Metals Division, Manufacturing Industries Bureau of the Ministry of Economy, Trade and Industry of Japan, METI.

JAPAN'S ELECTRONICS AND INFORMATION TECHNOLOGY INDUSTRIES 2001

Although 2000 was a year in which domestic production by the electronics and information technology (IT) industries in Japan set new records (see Figure 1), the equivalent result in 2001 was 21.4 trillion yen, a drop of 16.3%, one of the highest year-on-year falls on record. The main factor in this decrease was the occurrence of the so-called 'global IT recession'. In particular, domestic production of electronic components and devices fell considerably in 2001, as demand for cellular telephones, personal computers (PCs) and other equipment was sluggish around the world. Exports from Japan of electronics and IT products, and components and devices in particular, also decreased after a record year in 2000. At the same time, the production of tantalum products in the world decreased 30%.

Year	Production (trillions of yen)	Exports (trillions of yen)
1992	22.2	11.4
1993	21.0	10.8
1994	21.6	11.1
1995	22.5	11.6
1996	24.0	12.0
1997	25.4	13.7
1998	23.0	13.2
1999	23.1	12.5
2000	25.6	14.3
2001	21.4	12.1
2002 forecast	22.3	

Figure 1: Production and exports of electronic equipment, 1992-2001
(Approximately 5% is consumer electronic equipment, 45% electronic components and devices, 50% industrial electronic equipment.)

FORECAST FOR 2002

In the industrial electronics segment, domestic production is anticipated to rise 0.1%, even though a decrease of 2.0% is forecast for production of personal computers. Production of electronic components and devices is expected to advance by 2.1%. As a result, overall domestic production by the Japanese electronics industry is forecast to increase 0.7% in 2002, to 22.3 trillion yen. (Figure 2)

As for short-term prospects, the impact of a large increase in exports and the incipient recovery in industrial production are expected to spread to the overall economy, and bring about the beginning of a recovery in that economy. On the other hand, developments in the world-wide decline in stock prices and the depreciation of the US dollar are further increasing uncertainty surrounding the future of the world economy, and there are concerns over the downward pressure on final demand that may be exerted by the development.

	2000 trillion yen	% of 1999	2001 trillion yen	% of 2000	2002 forecast* trillion yen	% of 2001
Consumer electronic equipment	2.2	108.0	1.9	85.8	1.9	100.5
Industrial electronic equipment	11.9	106.5	10.6	89.2	11.1	104.8
Electronic components and devices	11.6	117.1	9.0	77.6	9.4	104.4
Total	25.6	111.2	21.4	83.7	22.3	104.3

Figure 2: Forecast of Japan's electronics and IT industries in 2002
(*forecast for 2002 by JEITA, Japan Electronics and Information Technology Industries Association)

REVIVING INDUSTRIAL COMPETITIVENESS STRATEGY

For several years, the government has pursued 'structural economic reform'. From the point of view of boosting emerging manufacturing industry, one of the biggest problems is rapid shifting to foreign countries. The Minister of METI started an active discussion on structural economic reform in the 'Industrial Competitiveness Strategy Council', an advisory committee for the Minister, in November 2001, and this Council reported in May 2002. It was felt that Japanese industry had become less competitive because both Japanese industry and the Japanese government were slow to respond to changes in the world around them.

Six strategies were evolved with a view to reviving Japan's competitiveness and moving into an industrial structure which would foster world-class companies:

1. Promotion of technological innovation is one of the key strategies, and the government will therefore establish three to five year action plans in the following four areas in order to boost emerging markets and industries:
 - environment and energy;
 - broadband communications and IT;
 - medical, healthcare and biotechnology;
 - and nano and material technologies to support the first three of these areas.
2. Promotion of corporate restructuring with 'selection and concentration'.
3. Enhance labour mobility and create more employment in the services sector.
4. Attract foreign direct investment and talent from overseas.
5. Establish the 'East Asia Free Business Area'.
6. Create new markets for the 21st century.

METI's industrial policy and these six strategies were presented at the 41st OECD Council at Ministerial Level, on May 15th 2002, in Japan, by Mr Takeo Hiranuma, the Minister for Economy, Trade and Industry.

NANOTECHNOLOGY AND MATERIALS

The Non-ferrous Metals Division of METI takes part in research and development programmes for nanotechnology and for the nanotechnology and materials action plan. In fiscal year 2002 the Japanese government spent around \$650 million on nanotechnology. In fiscal year 2003 the government planned to launch a new R&D policy for revitalizing the Japanese economy, called 'Focus 21'. METI is working to promote actively the strategies and policies aimed at realising innovative

nanotech-based products. Features of Focus 21 include commercialisation within three to five years, the creation of new, large markets, and the commitment of private companies to invest.

The research and development projects in nanotechnology and related branches, with a value of \$1 billion for 2003, include:

- New generation semi-conductors (50nm processing rule)
- Micromachines
- New generation displays – organic EL display, carbon nanotube FED, PDP, etc.
- Nanocarbon applications
- Microchemical processing
- Artificial blood cell

The MIRAI Project – Millennium Research for Advanced Information Technology – is intended to establish unique technologies in Japan by mobilising the expertise of industry, the government and the academic community while remaining open, transparent and globally oriented. A feature of the Mirai Project is a Super Clean Room Facility, with government funding of 25.2 billion yen. On an extensive site, the various laboratories, offices and facilities total some 22000m². A Super Clean Room, 'clean level 1', of 3000m² and a Research Clean Room, 'clean level 1000', of 1500m² are provided.

HISTORY AND DEVELOPMENT OF THE TANTALUM INDUSTRY (CONTINUED)

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

EARLY PROCESS DEVELOPMENT

The final chapter will cover the time beginning just after World War II and continue into the early 1970s. It was during this period that the electronic applications for tantalum began to be examined more thoroughly for the advantages that tantalum could bring to circuitry in the form of capacitors. This fledgling industry also learned during the 1940s that the element provided a superior cutting tool when tantalum carbide was included in the cemented carbide compositions. Products in the form of sheet, wire, rod, ribbon, powder and various fabricated shapes and parts were produced for use in various applications ranging from electronics to corrosion resistant equipment to surgical appliances.

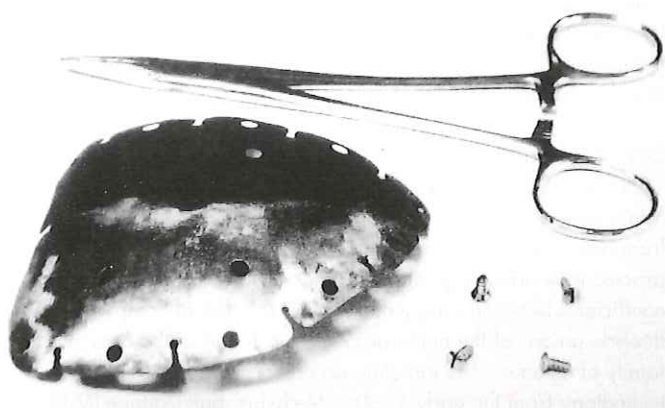


Figure 1: Skull plate, surgical tools, screws ⁽¹⁾

The Fansteel Corporation⁽¹⁾ learned that the successful marketing of its tantalum products in the peacetime era of the late 1940s was due to the ability of its research scientists to provide solutions to the technical problems that were faced during the early 1940s. These discoveries translated into applications involving chemical synthesis, electronics, radar, television, plastics, medicine, cutting tools, and X-rays.

Fansteel's production of tantalum was based on the Marignac process for the production of potassium tantalum fluoride. Electrolysis of molten salts⁽²⁾ was utilized for the production of metal powder. The electrolysis process was patented by C.W. Balke in 1931. Equal parts of potassium fluoride and sodium fluoride provided the salt bath medium for electrolysis. An amount of potassium tantalum fluoride, equal to as much as 50% of the weight of the fused salt mixture, was added to the molten salt bath. After it dissolved, a quantity of tantalum oxide, equal to about 10% of the K₂TaF₇ was also added and dissolved. The electrolysis vessel was a graphite crucible which functioned as the cathode while a carbon or nickel rod was generally employed as the anode. The operating temperatures of the molten salt bath during electrolysis ranged from 650° to 850°C. Voltage requirements were dictated by the distance between cathode and anode. Frequent additions of oxide were required since the potassium tantalum fluoride served as the dissolving agent for the tantalum oxide that was actually electrolyzed to tantalum powder.

The end of an electrolysis run involved the lifting of the anode covered with its deposit of tantalum powder out of the salt bath. A thin layer of salt adhered to the powder and prevented oxidation. The deposit of powder was then scraped off the anode.

The recovery of tantalum powder was achieved by leaching the powder to remove entrained salts and other impurities. Concentrated nitric and sulfuric acid mixtures were used during acid leaching to produce powders with carbon levels as low as 0.06%. Silicon was another major impurity in the powder, levels of 0.03 to 0.05% Si were common. The source of that element was the silicon content of the ore crystallizing as potassium silicofluoride.

Finished powders were screened into different sized fractions for use in various metallurgical product formulations, such as sinter bars (sheet products), rod bars (wire products) and powder for capacitors. Capacitance levels of 500 to 1000 CV/g were typical.

The Fansteel sodium reduction process was developed and patented in 1922, but was not brought into commercial use until the second half of the 1950s when competition from new processing companies caused considerable modification to the technical processing of the tantalum metal powder, especially for capacitor applications.

The analysis of the typical tantalum powder being produced by Fansteel at the end of the 1950s by electrolysis is shown in Table 1.

Fansteel was the only company in the United States that was engaged in the extraction and refining of tantalum in the 1940s through about 1955. There were at least three companies in Europe, namely Siemens and Halske, Gesellschaft für

Elektrometallurgie (GfE) in Germany, and Murex Limited in England, that were involved with the refining of tantalum chemicals and the production of the metal during this period. It appears that Siemens and Halske ceased operations in about 1945 while Murex Limited and GfE continued to extract and refine both niobium and tantalum. The Marignac process was utilized by these companies in the 1940s.

Impurity element	%	Impurity element	%
Nb	0.03	Zr	<0.05
Mo	0.003	Al	<0.02
W	0.01	Si	0.02
Fe	0.015	C	0.16
Ti	0.02	O ₂	0.17
Ni	<0.002	N ₂	0.01

Table 1: Typical analysis of impurities in Fansteel's tantalum powder, late 1950s

Siemens and Halske⁽²⁾ first used the sodium reduction of sodium tantalum fluoride in an iron crucible for preparation of the metal. The black metal powder was recovered after removal of salts and excess sodium by leaching with water. It became known that their preferred process involved the sodium reduction of potassium tantalum fluoride using diluent salts, such as sodium chloride, in the reaction mixture. The reduction was carried out in a 4-inch diameter steel tube covered with a weighted steel cover. This device was heated with a gas-fired ring burner until the reaction became self sustaining. Heating continued to insure complete reaction. Occasional explosions were not unexpected. The cooled reaction mass was 'infused' with methyl alcohol to react with excess sodium. The by-product fluoride salts were separated from the tantalum powder by water and acid leaching after the reaction mass was 'drilled out' of the steel tubes. Undissolved sodium would ignite and burn during this procedure. Washing continued until the powder was chloride-free. Drying produced the finished metal powder. Siemens and Halske reportedly produced 300 to 350 kg per month.

The process used by Murex Limited⁽³⁾ to produce tantalum metal powder was very similar to that used by Siemens and Halske.

H.C. Starck⁽³⁾ in Germany began investigations in 1948 into the recovery of tantalum from residues left from the processing of hardmetal scrap obtained from the United States. Tantalum values were converted into potassium tantalum fluoride or tantalum oxide. The former was used for conversion to the metal via sodium vapor reduction in nickel tubes and the oxide for conversion to tantalum carbide, which became a very important business for this company.

Early attempts by H.C. Starck to produce pure tantalum oxide from potassium tantalum fluoride were not successful via wet chemical processes due to contamination with large amounts of potassium. A sodium hydroxide fusion of the impure oxide was required to release and remove the chemically-bound potassium. Commercialization of this process was completed in 1952.

A patent was issued to H.C. Starck in 1951^(2,4) for the preparation of tantalum metal powder via sodium reduction of potassium tantalum fluoride. This unique technology utilized sodium vapor as the reducing agent, with the tantalum salt and sodium isolated in separate chambers. The sodium vapor was

passed over the tantalum salt resulting in a controllable reaction rate. This process was conducted in vacuum. The powder was recovered via acid leaching, washing, filtration, and drying. Yields of 90% and purities of 99.5% were achieved.

H.C. Starck never processed tantalum raw materials using the Marignac procedure, but instead purchased potassium tantalum fluoride from GfE. H.C. Starck, in conjunction with GfE, established a research institute known as Elchem in 1951, which developed a solvent extraction process based upon the research studies conducted and published by the U.S. Bureau of Mines during the 1950s. The application of this technology was installed in production by GfE and some years later by H.C. Starck.

Kawecki Chemical Company⁽⁵⁾ in Pennsylvania was a fledgling chemical processor of uncommon elements that pursued an interest in tantalum, beginning in about 1955. A rejection by the U.S. Government concerning a lithium carbonate production proposal pushed KCC into a tantalum programme using the Marignac technology for production of potassium tantalum fluoride and the sodium reduction of this same tantalum-bearing salt. Electrolysis of molten salts containing potassium tantalum fluoride was also employed to produce an early tantalum product.

The sodium reduction process for the production of tantalum powder was developed at KCC in the mid-1950s using processing technology similar to that employed by other processors. A major difference however was the development of 'high' capacitance powder, ranging from 2000 to 3500 CV/g. These high CV powders were generally in the shape of flakes, which provided additional surface area in comparison to the electrolytic and competitor sodium-reduced powders of that period.

The tantalum industry was developing in Japan during the 1955-1960 period: an early record of tantalum capacitor production can be documented in 1959⁽⁶⁾ at the Matsuo Electric Company Ltd in Osaka, Japan. The source of the tantalum powder for these early efforts is unknown, but it could have been either Showa Denko K.K. or Mitsui Mining and Smelting Co., Ltd.

The Ulba Metallurgical Plant in Kazakhstan⁽⁷⁾ began production of potassium tantalum fluoride in 1951 and tantalum powder via sodium reduction in 1960. Information concerning the processing of tantalum in China, India, and other countries in the 1950s and earlier was not available.

Numerous alternate powder production schemes were attempted by various researchers during these formative years of the industry up through the late 1950s. They are reviewed in some detail in the book by G.L. Miller.

One of the most important technical developments during the 1950s was based on research conducted by the U.S. Bureau of Mines aimed at the clean separation of niobium and tantalum from ore concentrates utilizing a solvent extraction scheme. The process took advantage of the differences in extraction coefficients between an aqueous acidic solution of complex fluoride anions of the numerous elements found in the ores and a family of ketones. The tantalum processors adopted this technology from the early 1960s. Methyl-isobutyl ketone (MIBK) was the extractant of choice. Carefully controlled conditions of

acidity permitted niobium and tantalum to be isolated from each other as well as from troublesome impurities such as silicon, titanium, and zirconium. In addition to achieving high levels of purity in the potassium tantalum fluoride and other products, the process was adaptable to continuous operation.

The second major technical innovation in the processing area was the development of the extraction and refining of tantalum and niobium from tin slags. Tin slags were being produced in Africa and Southeast Asia as well as in other countries where tin ores were mined and smelted for tin recovery. The resultant slag from the smelting furnace contained the niobium and tantalum that had been present in the cassiterite concentrates. In the 'glory days' of tin slag, it could have a combined niobium and tantalum oxide content of 15% as a minimum, with some sources exceeding 20%, such as those from the early production of the Thaisarco smelter in Thailand. The very high grade slags could be processed directly through acid digestion and solvent extraction purification. Lower grade slags, however, faced severe economic penalties for direct use.

H.C. Starck initiated studies in 1954⁽⁹⁾ aimed at increasing the niobium and tantalum contents of tin slags so they could be used as feedstock for the standard acid digestion process. The tin slag⁽⁸⁾ was reduced in an electric arc furnace, usually with the addition of scrap iron or iron oxide. The formation of a high-carbon ferroalloy collected the niobium and tantalum content from the original slag. The ferroalloy was treated via chlorination to recover the niobium and tantalum chlorides in highly purified form or was treated chemically, resulting in a niobium-tantalum concentrate which could be processed in a manner similar to that for tantalite-columbite mineral concentrates. Process details were eventually developed that allowed the conversion of the ferroalloy to an oxidic product of high niobium and tantalum oxide content that could be subjected to standard processing technology.

The U.S. government had enough interest in tantalum in the 1950s to provide encouragement to companies to enter the business. This effort was extended to potential refiners of the metal as well as downstream users of the element, such as capacitor manufacturers. The net effect of this encouragement was the rapid development of the tantalum industry in the United States. Similar activity took place in Europe at the same time.

Besides Kawecki Chemical Co. and the Fansteel Metallurgical Co., there were additional companies that became involved with the processing of tantalum-bearing raw materials, various chemicals, and/or powder production in the United States during the late 1950s and 1960s. Former processors are shown below. (The lists below have been compiled with the best of the author's knowledge, but please see his 'special request'.)

Former Processors, United States

Fansteel Metallurgical Co., Muskogee, OK and North Chicago, IL
Union Carbide Corporation, Buffalo, NY
Mallinckrodt Chemical Works, St. Louis, MO
Shieldalloy, NY and NJ
Metallurgical International, Newfield, NJ

Former Processors, Europe

Siemens & Halske, Berlin, Germany
Murex, Rainham, Essex, U.K.
Gesellschaft für Electrometallurgie mbH (GfE), Laufenberg,

Germany

Société Générale Métallurgique de Hoboken, Hoboken, Belgium
Ciba Limited, Basle, Switzerland

Former Processors in Asia

Showa Denko K.K., Tokyo, Japan

THE EARLY TANTALUM CAPACITOR INDUSTRY

The very first commercial tantalum capacitor was produced and marketed in 1930 by Fansteel⁽²⁾. These capacitors were used primarily in telephone service applications. One of the designs was the size of a 'pint fruit jar' and contained electrodes of crimped tantalum sheet. This device provided a rating of 800 mfd at 24 volts. During the 1940s, technology improvements led to further commercial production of tantalum capacitors. Numerous microfarad and working voltage ratings were developed utilizing tantalum foil, gelled sulfuric acid electrolyte, and hermetically sealed cans of silver.

A volumetric comparison⁽¹¹⁾ of different capacitor types provides one of the driving forces for the success of the tantalum capacitor (see Table 2). The comparison is based on a standard of 100 volt capacitors at the same microfarad rating.

Type	Percent volume
Paper	100
Ceramic	95
Aluminum foil	23
Tantalum foil	15

Table 2: Volumetric comparison of different types of capacitor

The volumetric advantages are clearly evident.

The tantalum foil capacitor⁽¹⁰⁾ was constructed by the preparation of the anode from very high purity tantalum foil, slit to design width and length and fitted with a tantalum lead wire. This assembly was submerged in a hot electrolyte, such as phosphoric acid or ethylene glycol, and a dielectric film was formed to 40% higher than the rated working voltage. For example, a 60 volt working volt capacitor would be formed to 84 volts. With a deposition of 17 angstroms of tantalum oxide per volt, the resultant oxide thickness on the foil would be 1425-1430 angstroms. When the final dielectric film thickness was reached, the target forming voltage was held for one hour to maximize anodic film quality. A high electrolyte temperature enhanced the formation of low leakage tantalum oxide film. Film quality was determined by measuring the d.c. leakage at room temperature at rated voltage.

The capacitor was completed by rolling the anodized foil with another piece of foil, equal in area, with a high quality paper spacer between the two foil lengths. The second foil served as the cathode. The purpose of the spacer was to protect the anodic film and absorb electrolyte throughout the contact area between the two foil layers. A schematic of this rolled configuration is shown in Figure 2.

The rolled foil, containing a tantalum lead wire on both anode and cathode foils, was inserted into a can and impregnated with a gelled sulfuric acid electrolyte (produced by the addition of ethylene silicate to the sulfuric acid) and hermetically sealed with

an elastomer or glass-to-metal seal. Silver cans were commonly used. This method of construction resulted in the anode and cathode wires (connection to the circuit) exiting from the ends of the sealed unit.

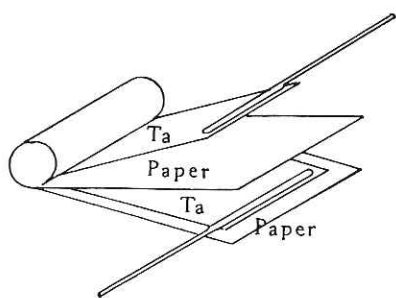


Figure 2: Rolled foil construction, tantalum capacitor ⁽¹¹⁾

Both polar and non-polar capacitors were produced. A polar device is one where one foil is anodized. If an a.c. signal is applied to the finished capacitor, the foil containing the tantalum oxide layer will change polarity during half of the cycle. In turn, the cathode will become anodic and develop an oxide film by the normal mechanism. A d.c. polarizing voltage is required to maintain proper polarity.

Some capacitors had a very low voltage film on the cathode to protect against small polarity reversals. These were known as semi-polar tantalum capacitors.

The third type of tantalum foil capacitors was that containing identical oxide films on both foil electrodes, which then become two capacitors in series. The total capacitance is about half of the first example where only one foil is anodized. This configuration is known as a non-polar or a.c. capacitor. The General Electric Company⁽¹⁰⁾ was one of the early producers of these capacitors in the late 1940s.

It was reported that Fansteel first produced the porous-slug tantalum capacitor as early as 1936⁽¹⁰⁾. During the late 1940s, Fansteel developed a sub-miniature, porous-slug type tantalum capacitor in conjunction with Bell Laboratories and the U.S. Air Force. The design of the tantalum anode could be adjusted in terms of its shape, but was generally cylindrical with L/D ratios of 0.5 to 3.0 as well as wafers, and rectangular bodies. The typical design is shown in Figure 3.

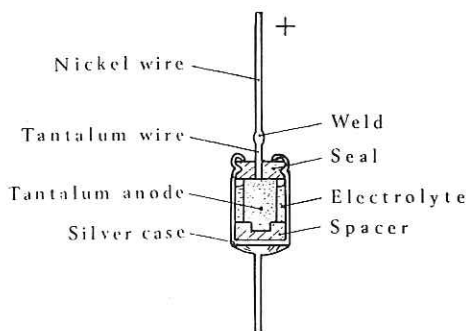


Figure 3: Porous-slug design of tantalum capacitor ⁽¹¹⁾

The external can is the cathode and the lead wire is connected to the anode. Generally, the cathode lead wire is welded directly to the external container, which was steel, silver, or tantalum.

The porous tantalum slug⁽¹²⁾ was produced by pressing tantalum powder (sodium-reduced) of controlled particle size distribution into a powder compact and embedding a tantalum wire during

the powder pressing process. This 'green compact' was sintered at a temperature required to cause the powder particles to form a strong bond between particles while at the same time leaving an open porosity throughout the entire anode body. Pressed densities of 8.0 to 10.0 gm/cm³ were standard. Sintering temperatures ranged from 1800° to over 2000°C in high vacuum, and at these temperatures, impurities in the powder such as carbon, nitrogen, oxygen, and metallic elements with high vapor pressures were removed. There are always tradeoffs between pressed density and sintering temperatures, and the resultant anode porosity and the strength of the anode.

Anodization of the sintered tantalum anode was conducted in a manner similar to that for foil, resulting in a tantalum oxide film covering all of the surface area of the sintered pellet. The thickness of the oxide film is directly proportional to the applied voltage. Electrolytes were sulfuric or phosphoric acid compositions, or ethylene glycol. Electrical testing of the anode quality was required for each powder lot.

During the early 1960s, a solid counter electrode was developed in the form of manganese dioxide for use in slug-type tantalum capacitors⁽¹³⁾. This represented a technical breakthrough that virtually eliminated the disadvantages and use of the gelled sulfuric acid electrolyte. The manganese dioxide cathode was formed by impregnating the anodized, porous tantalum slug with manganese nitrate solution, followed by pyrolysis to MnO₂, after which the tantalum oxide film was reformed in liquid electrolyte to 'heal' any damaged areas of the tantalum oxide film. Multiple applications of manganous nitrate, thermal decompositions, and reforms are generally performed to insure the integrity of the anode. The rated working voltage of this style of tantalum capacitor was set at a maximum of 35 volts.

A variation on the design of the slug-type tantalum capacitor is shown in Figures 4 and 5.

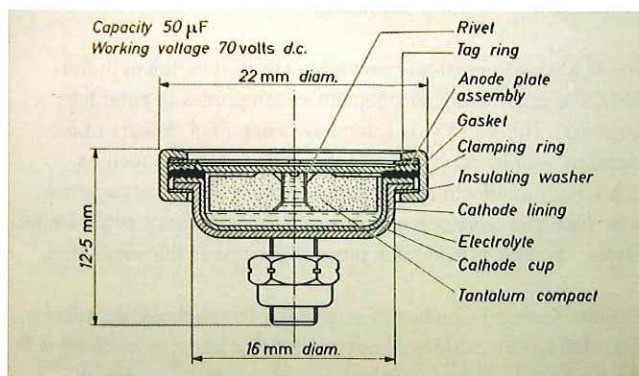


Figure 4: Plessey castanet capacitor⁽¹²⁾

This design contains a porous sintered tantalum disk anode, a liquid electrolyte cathode, and a silver cathode container. The external shell is nickel which provides a robust construction. The purity and particle size of the tantalum powder are tightly controlled and it is sintered at high temperature and vacuum to insure optimum electrical performance. The dielectric film is formed in the normal manner with film thickness dictated by the capacitor rating and application. A wide range of capacitance values can be achieved ranging from 50 mfd at 70 V to 750 mfd at 3 V. 'Stacked' designs were employed for ratings up to 200 volts.

These capacitors have a long shelf life, measured in years, and are designated as high reliability, low leakage capacitors. Their temperature range of operation is -60°C to +200°C. This same style of capacitor is still produced today.

Almost all the tantalum anodes required during the early years of this capacitor industry were actually produced by the powder manufacturers and were sold to the capacitor manufacturers. Sintered anodes were a standard product line into the mid 1960s.

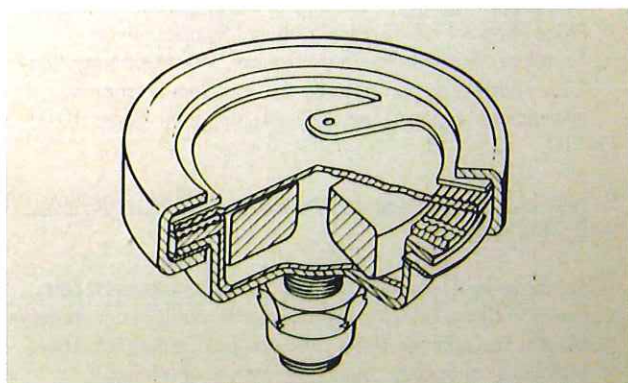


Figure 5: Interior design of the Plessey castanet capacitor⁽²⁾

The Fansteel Company was the only company in the U.S. in 1955 that was processing tantalum into finished capacitors. By 1960 there were nine capacitor manufacturing companies, and that list expanded dramatically over the next 10 years, not only in the United States, but in Europe and other areas of the world. The chart below lists the companies in both the U.S. and Europe that were producers of tantalum capacitors during the period from 1955 up through the current time that are no longer in business or not in the same hands. It is obvious that failures and consolidations, etc. took their toll on the industry. It was also in the mid 1960s that the producers of tantalum powder came to the conclusion that they could no longer 'compete' with their customers by making anodes and capacitors. As a result, the processors made the powder, and the use of it to manufacture capacitors was taken over by other firms.

Former Ta Capacitor Manufacturers, North America⁽¹⁴⁾

Aerovox, New Bedford, MA
 Astron, Newark, NJ
 Bell Laboratories, Murray Hill, NJ
 Cornell Dubilier, Plainfield, NJ and Sanford, NC
 Components → Corning (now AVX), Phoenix, AZ, El Paso, TX, Biddeford and Kennebunk, ME
 Carolina Components, Columbia, SC
 DCA Relays, Fairfax, VA
 Dickson → Siemens, Scotsdale, AZ
 EFCO, Garden City, Long Island, NY
 Electro Capacitors, Oakland, CA
 Electra Manufacturing, Independence, KS
 Elpac, Torrance, CA
 Erie Resistor, Erie, PA
 Fansteel, North Chicago, IL
 Faradyne, Newark, NJ
 General Electric, Hudson Falls, NY & Irmo, SC
 General Instrument, Newark, NJ & Long Island, NY
 Gentronics, Norwalk, CT
 Goodall – TRW, Ogallala, NE
 Hilton Industries, Glenn Falls, NY & Sarasota, FL
 Hunter-Stanley, Nashville, TN
 International Electronic Industries, Nashville, TN
 Illinois Tool, Chicago, IL
 ITT, Palo Alto, CA & West Palm Beach, FL
 John Fast Co., Chicago, IL
 Johnson Matthey, Toronto, Canada
 LenKurt, Redwood City, CA
 Magnavox, South Bend, IN

Micamold, Newark, NJ
 Minitronics, Newark, NJ
 National Capacitor → Mepco Electra (now Vishay Sprague), West Palm Beach, FL
 PRMallory → North American Capacitor (now Vishay Sprague), Indianapolis & Greencastle, IN
 Ohmite, Skokie, IL
 Pacific Semiconductor, El Segundo, CA
 Pyramid Nytronics, Newark, NJ
 Plessey → Westlake → Arcotronics, Westlake Village, CA
 RCA, Sommerville, NJ
 Sangamo, Springfield, IL & Pickens, SC
 Special Tantalum Components, El Paso, TX
 Sprague Electric Co., North Adams, MA
 Sprague Electric Co., Concord, NH
 Tansitor (now Vishay Sprague), Bennington, VT
 Telegraph Condenser, Toronto, Canada
 Texas Instruments, Dallas, TX
 Tital, Oakland, CA
 Union Carbide, Cleveland, Ohio
 US Semcor/Components, Phoenix, AZ & El Paso, TX
 Western Electric, various locations in PA, MA, NC
 Westinghouse, Monroeville, PA

Former Ta Capacitor Manufacturers, Europe⁽¹⁴⁾

AEG Telefunken, Nuremburg, Germany
 CELM (General Instrument), Naples, Italy
 Componentes Electronicos Thomson (CSA), Barcelona, Spain
 Dubilier, London, UK
 Electronic Industries, Belgrade, Yugoslavia
 Elpi, Romorantin, France
 EPI, Bordeaux, France
 Erotantal → Roederstein → Vishay Sprague, Landshut, Germany
 Eurotaag/Hughes, Glenrothes, Scotland
 Kapsch & Sohne, Vienna, Austria
 Le Capacitor Ceramic (LCC) → Tekelec → CSA, Dijon & Bordeaux, France
 Lignes Téléphoniques et Télégraphiques (LTT) → ITT, St. Honorine, Paris, France
 Philips, Eindhoven, Netherlands
 Piher, Barcelona, Spain
 Plessey/Arcotronics → (now Nissei Arcotronics), Towcester, Northants, UK
 Plessey → Plessey/Hughes, Bathgate, Scotland
 Pro Condenser/Vanzetti, Belluno, Italy
 Precis, Milan, Italy
 RIFA, Stockholm, Sweden
 Roussell – Uclaf (now Firadec), Paris France
 Siemens Corp. (now Epcos), Heidenheim, Germany and Evora, Portugal
 Soveril, Bagneaux-sur-Loing, Paris, France
 Sprague/Creas, Milan, Italy
 Sprague → Vishay, Tours, France
 Standard Electric Lorenz (SEL) à ITT à STC à AVX, Bislohe, Germany
 Standard Telephone Co. (STC) à ITT (now AVX), Paignton, Devon, UK
 Tekelec, near Lisbon, Portugal
 Tekelec → Firadec, Paris & Bordeaux, France
 Thompson CSF, Lille, France
 Union Carbide, Aycliffe, Darlington, UK
 Unita/Elwa, Warsaw, Poland

Former Ta Capacitor Manufacturers, Japan

Oomori Electric Co., Ishikawa Prefecture

The porous slug anode coupled with the manganese dioxide solid electrolyte led to the manufacture of epoxy coated solid

capacitors in the 1970s in radial, axial, molded and a number of other designs. The advantage was the elimination of the external metal can (cathode) as well as the gelled sulfuric acid electrolyte and a serious reduction in the cost of manufacturing a capacitor due to automation. This opened up new opportunities for tantalum capacitors and their characteristics of high reliability, long life, wide temperature range with minimal change in performance, low d.c. leakage, and occupation of 'small real estate' on a circuit board in comparison with alternative technologies. The lead wire configuration required manual insertion into the circuit board.

The early 1980s saw the development of the chip design which eliminated the external wire leads and replaced them with an internal connection to a lead frame that becomes the soldered connection to the circuit board. This development created the opportunity for 'pick and place' positioning on the circuit board.

Technical developments in the increase of the surface area of tantalum powders led to the reduction in anode weights for an equivalent rating during this same period. A much greater percentage of the anode weights were measured in milligrams instead of grams. In the early 1960s, anode weights of 1 to 5 grams were common. Today the average anode weight across all production is 50 to 55 mg with the smallest anode weighing about 0.5 mg and applications are found today in almost every electronic application imaginable where high reliability and small footprint are common requirements.

SPECIAL REQUEST

The research for this paper involved the use of a few of the member companies' publications that were written and published for special celebrations or notable anniversaries. The ones we have are listed in the bibliography. The T.I.C. office would appreciate a copy of any other such publications from member companies that may be available. These are invaluable sources not only of historical information about the niobium and tantalum industry as it evolved in each of the member companies, but also represents a substantial technical resource for general publication about these two elements. Please send any copies of your company publications to the office in Brussels, Belgium.

Also, there are probably a number of additional companies that have ceased processing niobium and tantalum as well as tantalum capacitors. Submission of additional company names and correction of any errors are encouraged. Information is sought for areas of the world not covered in this paper.

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UNITED NATIONS

On January 24th 2003 the United Nations Security Council adopted resolution 1457, concerning the resources of the Democratic Republic of the Congo, and asked for a further six-month mandate for the panel of experts investigating the 'illegal exploitation of natural resources'.

DLA

The Defense Logistics Agency, Defense National Stockpile Center (DNSC), announced on January 10th 2003 that it was offering concentrates with about 80 437 pounds of contained tantalum pentoxide, offers to be considered on January 28th 2003.

In February the DNSC was seeking to convene an Industry Meeting on Tantalum to discuss the DNSC's Basic Ordering Agreement sales process, as it intended to use this process for the remainder of tantalum sales in fiscal year 2003. A meeting has been arranged for Thursday March 20th in Arlington, Virginia. DNSC expected to post a draft Basic Ordering Agreement on its web page before the meeting.