TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

BULLETIN N° 60 DECEMBER 1989 FOURTH QUARTER

Thirtieth General Assembly and associated meeting

The Thirtieth General Assembly of the Tantalum-Niobium International Study Center took place on Thursday October 19th 1989 in the charming setting of the Hotel Gravenbruch Kempinski, near Frankfurt, as part of a three day meeting which also included a technical session, a plant tour of W.C. Heraeus GmbH, social events and a ladies' tour.

The President, Dr Harry Stuart, Niobium Products Company, reported on a very satisfactory year for the association : the International Symposium on Tantalum and Niobium in November 1988 had been highly successful and the number of member companies was rising. The Assembly approved the accounts for the year ended June 30th 1989 and was pleased to note that the financial position of the T.I.C. was sound.

Dr George Korinek, NRC Inc., was elected President for the coming year, his second term in this office. Mr Yoichiro Takekuro, Vacuum Metallurgical Company, and Mr Peter Adams, Thailand Smelting and Refining Company, were elected to the Executive Committee, and all other Committee members were re-elected.

The T.I.C. welcomed the appointment of Mr Rod Tolley as the association's Technical Adviser and editor of the quarterly Bulletin. He will work on a part-time basis and may be contacted through the T.I.C. office in Brussels. The association decided to organise an international standard for tantalum powder for capacitors, and also to review its collection of tantalum and niobium statistics, and both these activities will be co-ordinated by Mr Tolley, among his other tasks.

Ten companies were elected to membership (see last page of this Bulletin), bringing the total to 68 at this time.

The next General Assembly will be held in Perth, Western Australia, on November 6th 1990, during a three-day meeting which will include a tour of the Greenbushes Ltd. mine. An informal assembly is also planned for April 27th 1990, in Brussels.

Following the official business meeting, these technical presentations were made :

General overview of the tantalum industry, by Mr Lawrence S. O'Rourke, Consultant

Characterisation and mineral processing of European ores, type Echassières (France) and Penouta (Spain), presented by Dr I. Gaballah of the Centre de Recherche sur la Valorisation des Minerais

New process for the production of niobium and tantalum oxide, by Dr Norbert Krummen, Gesellschaft für Elektrometallurgie mbH

Update on the outlook for tantalum capacitor demand and tantalum materials demand for capacitors, by Mr David E. Maguire, Kemet Electronics Corporation, presented by Mr Derek Payne, Kemet Electronics S.A.

Tantalum powder dopants and the effect of their residues on tantalum oxide quality of low oxygen tantalum surfaces, presented by Mr John VanVoorhis. Sprague Technologies

The technical session closed with a panel discussion on the expansion of the association's activities, particularly in the field of statistics for raw materials and for the capacitor industry and the establishment of a standard for capacitor powder. It was chaired by Dr Harry Stuart, and panel members were Mr Robert Franklin, Mr John Fredrickson, Dr George Korinek and Mr John Linden.

On October 20th the delegates to the meeting were given a most interesting and informative tour of the plant of W.C. Heraeus GmbH, at Hanau. They visited the rolling mill, electron beam melting furnace, metals laboratory, chemical department, thick film department and medallion mint — where a special commemorative medallion had been struck for the occasion — and they were impressed by the up-to-date methods and equipment of this active and developing company.

President's letter

Based on the positive comments of quite a number of participants, it can be concluded that the Thirtieth Assembly and Meeting of October 18-20, 1989 in Frankfurt was a success.

We appointed Rod Tolley to serve as the technical adviser and I am happy to report that he is already firm in the saddle and running.

I would like to take this opportunity to thank our outgoing president, Dr Harry Stuart, for his dedication to the T.I.C. during his term of office and for his great help in organizing the Symposium in Orlando.

George J. Korinek
President

November 17, 1989

THE PROCEEDINGS OF THE ORLANDO SYMPOSIUM

There are still copies available of this book which, with its up-to-date description of all aspects of the tantalum and niobium industries, deserves a place in all technical libraries.

The price, US$80 per copy, includes post and packing : please send your order and bank draft to the Secretary General, T.I.C., 40 rue Washington, 1050 Brussels.

MAILING LIST

Is your copy of the Bulletin correctly addressed ? Is it reaching the right people in your organisation ? Do you receive the number of copies you would find useful ?

The mailing list is on a computer programme : please help us to keep it up to date by telling us of any changes which should be made.

T.I.C. statistics

TANTALUM

PRODUCTION AND SHIPMENTS

(quoted in lb Ta_2O_5 contained)

2nd quarter 1989

	Production	*Shipments*
Tin slag (over 2 % Ta_2O_5)	253 714	126 760
Tantalite (all grades), other	303 795	228 772
Total	557 509	355 532

Note :

17 companies were asked to report, and all 17 replied. The companies which reported included the following, whose reports are essential before the data may be released :

Datuk Keramat Smelting
Greenbushes
Malaysia Smelting
Mamoré Mineração e Metalurgia
Metallurg Group
Tantalum Mining Corporation of Canada
Thailand Smelting and Refining

PROCESSORS' SHIPMENTS

2nd quarter 1989

Product category	*lb Ta contained*	*lb Ta_2O_5 equivalent*
Ta_2O_5, K_2TaF_7	19 746	26 657
Alloy additive	17 413	23 508
Carbides	193 166	260 774
Powder/anodes	202 132	272 878
Mill products	82 277	111 074
Ingot, unworked metal, other, and scrap	13 703	18 499
Total	528 437	713 390

Notes :

1. 16 companies were asked to report, and all 16 replied. Reports by the following companies are essential before the data may be released :
   Cabot Corporation, Electronic Materials and Refractory Metals
   Fansteel
   W.C. Heraeus
   Kennametal
   Metallurg Group
   Mitsui Mining and Smelting
   NRC Inc.
   Showa Cabot Supermetals
   Hermann C. Starck Berlin
   Treibacher Chemische Werke
   Vacuum Metallurgical Company
   V Tech
2. Reports were made in lb tantalum contained.

QUARTERLY PRODUCTION ESTIMATES

(quoted in lb Ta_2O_5 contained)

LMB quotation :	*US \$ 30*	*US \$ 40*	*US \$ 50*
3rd quarter 1989	220 000	353 500	421 500
4th quarter 1989	217 000	351 500	446 500
1st quarter 1990	212 000	331 500	446 500
2nd quarter 1990	215 000	313 500	451 500
3rd quarter 1990	215 000	313 500	451 500

Note :

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

Capacitor statistics

EUROPEAN TANTALUM CAPACITOR SHIPMENTS

(thousands of units)

2nd quarter 1989	173 672

(Data from ECTSP)

JAPANESE TANTALUM CAPACITOR PRODUCTION AND EXPORTS

(thousands of units)

	Production	*Of this, exports*
2nd quarter 1989	916 492	205 039

(Data from JEIDA)

U.S. TANTALUM CAPACITOR SALES

(thousands of units)

	2nd quarter 1989	**3rd quarter 1989**
Foil	220	181
Metal-cased	25 499	21 673
Moulded	68 464	61 282
Dipped	98 237	90 663
Chips	48 859	48 822
Wet slug	2 052	1 729
Total	243 331	224 350

(Data from EIA)

Note :

The data did not provide separate figures for U.S. shipments and exports for these two quarters.

Update on the outlook for tantalum capacitor demand and tantalum materials demand for capacitors

This paper was prepared by Mr David E. Maguire of Kemet Electronics Corporation for presentation to the technical session following the Thirtieth General Assembly on October 19th 1989

INTRODUCTION

The outlook for tantalum capacitor demand and the usage of tantalum materials for such capacitors through 1994 will be updated in this paper. An outlook for tantalum capacitor demand in 1994 was first presented to the Twenty-third General Assembly of the Tantalum International Study Center in June of 1985. The 1985 presentation included the design driver factors which would propel the growth of tantalum capacitors through the forecast period. The second forecast presentation through 1994 was made to the International Symposium on Tantalum and Niobium in November of 1988. This latter presentation identified the potential impacts of rising material costs and other technical factors on the future demand for tantalum capacitors.

The paper reviews and updated forecasts for the worldwide electronics industry, capacitors usage in total, a breakdown of future capacitor demand by dielectric, the future demand for tantalum capacitors by styles, and a forecast of tantalum material demand for capacitors by style and by total.

THE ELECTRONICS INDUSTRY

	1988	1994	Growth Rate %
Total Value $ Billions	570	960	11
Total Capacitors Units-Billions	180	270	8
Total Capacitors $ Billions	8.0	10.8	6
Capacitors Content Value	1.4%	1.1%	

Table I

ELECTRONICS INDUSTRY
Worldwide Total

Table I shows the 1988 and 1994 projected total market value of the worldwide electronics industry. The 1988 volume is over one-half trillion U.S. dollars and is projected to grow to about one trillion U.S. dollars by 1994. This represents an 11% compound average annual growth rate over the next five years. This growth rate has been demonstrated over the past fifteen years, albeit with short term interruptions and accelerations primarily due to inventory accumulations and rundowns within the component, circuit, hardware, and distribution consumption stream. Fortunately, the future fluctuation in growth should be much less severe than in the past as the world goes towards a 'just-in-time' manufacturing and distribution philosophy with a concurrent dramatic reduction of 'work-in-process' inventories.

The total worldwide consumption of capacitors in 1988 is estimated to have been 180 billion units. This estimate and the further updated estimates to follow in this paper are based on newly available and more accurate worldwide consumption data.

The compound average annual growth rate in the total unit demand for capacitors over the next five year period to 1994 is 8% to a total of 270 billion units. The concurrent compound average annual growth rate in the dollar value of capacitors is forecast to be 6% from a total of $8.0 billion in 1988 to $10.8 billion in 1994. The slower growth in value versus units is the result of future growth in smaller size and less expensive capacitors plus the inexorable cost learning curve within the electronics industry. (The cost learning curve, in its simplest generic form, states that every time the cumulative volume of production of an electronic device **doubles**, the unit average cost of such a device **declines** by about 30 % in constant dollar terms.)

The value of the capacitor content of the total electronics industry is slowly declining from 2.9% in 1968 to 1.4% in 1988 and is forecast to decline further to about 1.1% in 1994.

This gradual decline is due to the increasing complexity and functionality of integrated circuits. The internal function of an integrated circuit has risen from hundreds of active elements in 1968 to millions of active elements today, and the trend is continuing. Meanwhile, a fixed number of capacitors is required to decouple and support each individual integrated circuit.

WORLDWIDE CAPACITOR MARKET

Dielectric	1988			1994			Growth Rate	
	Quantity Billions	Cost $/1000	Value $MM	Quantity Billions	Cost $/1000	Value $MM	Quantity	Value
Ceramic (Single Layer)	54.1	15	785	54.1	15	785	0	0
Ceramic (Multilayer)	63.0	34	2160	130.8	29	3795	16	12
Tantalum	5.6	186	1045	10.3	139	1430	13	6
Aluminum	39.0	64	2495	52.2	64	3035	6	4
Film	17.0	79	1350	19.7	79	1565	3	3
Other	0.5	290	145	0.5	290	145	0	0
TOTAL	179.2	45	7980	267.6	40	10755	8	6

Table II

CAPACITOR MARKET
Worldwide Total

The breakdown of the 1988 worldwide capacitor market by dielectric is shown in Table II. The projected 1994 worldwide consumption has been estimated along with the compound average annual growth rate for each dielectric. The fastest growth rate dielectric continues to be the monolithic ceramic dielectric capacitor. The monolithic ceramic capacitor is already suited to the trends in electronics equipment where, in general, everything is getting smaller, more reliable, less expensive, and faster in terms of current speed. Traditional electronic components have been produced with lead wires which served to connect the capacitor element into the electronic circuits. Modern circuit designs, called SMT or Surface Mount Technology, are dispensing with superfluous and costly lead wires and attaching the chip elements directly into the electronic circuit on a substrate. The monolithic ceramic capacitor elements have always been produced as chip elements which can be directly used for SMT. These monolithic ceramic capacitors also have no intrinsic dielectric constant or dielectric thickness restraints and thus will gradually displace other dielectric types. Such displacement has eliminated, and will continue to eliminate, the growth in single layer ceramic capacitors and will displace some lower value tantalum capacitors. The monolithic ceramic capacitor will also limit the growth of film dielectric capacitors through gradual displacement.

Aluminum electrolytic capacitors are the lowest cost approach to providing circuit decoupling and filter functions where larger values of capacitance are required. The growth rate of aluminum capacitors will continue at a rate of about 6% per year which is a little lower than their traditional growth rate of about 8% per year. The lower growth rate is caused by the inability of this capacitor dielectric to be produced as a true surface mounted chip capacitor and its inability to withstand the over 250°C temperatures encountered in the chip mounting process. The higher capacitance function in circuits using modern surface mounted technology will be filled by solid tantalum capacitor chips.

The solid tantalum capacitor element can endure the temperatures required to surface mount chips in modern circuits because it undergoes similar temperature treatments during its manufacturing. However, the basic capacitor element is not of intrinsic chip form (like the monolithic ceramic capacitor element) and thus must be connected to suitable chip terminations and must further be encapsulated to protect the element from mechanical damage. The extra cost of termination and encapsulation works to the competitive disadvantage of solid tantalum chip capacitors. The other major cost limitation to the even higher growth of solid tantalum chip capacitors is the high cost of the tantalum materials required. Tantalum is roughly the same cost as silver !

Not withstanding the above limitation, solid tantalum capacitors should experience a growth from 5.6 billion units in 1988 to 10.2 billion units by 1994 for a compound average annual growth rate of 13 %. The value of solid tantalum capacitors is forecast to grow at 6 % per year from about $ 1.0 billion in 1988 to $ 1.4 billion by 1994.

THE TANTALUM CAPACITOR MARKET

Style	1988 Quantity Millions	1988 Cost US Cents	1988 Value $MM	1994 Quantity Millions	1994 Cost US Cents	1994 Value $MM	Growth Rate Quantity	Growth Rate Value
Metal	285	44	125	227	48	109	(4)	(2)
Molded	750	23	173	700	24	168	(1)	0
Dipped	2390	13	321	2265	13	299	(1)	(1)
Chips	2175	16	350	7081	11	779	27	17
Wets	15	507	76	13	575	75	(2)	0
TOTAL	5615	19	1045	10286	14	1430	13	6

Table III

TANTALUM CAPACITOR MARKET
Worldwide Total

The 1988 worldwide market for tantalum capacitors, detailed by physical styles, is shown in Table III, along with the 1994 projection and compound average annual growth rates.

The metal style is encased in a hermetically sealed can and is primarily driven by military usage and older industrial and computer designs. It is not used for new designs. The volume of the metal can style tantalum capacitor has begun to shrink and is projected to continue declining.

The molded style has either axial or radial lead wires and is encased in a molded plastic case. The usage is primarily in industrial, telecommunications and current computer designs. This style is less expensive than the metal-cased style and is equivalent in electrical performance except in very harsh physical environments involving a high level of moisture. The demand for the molded style of tantalum capacitor is expected to stop growing and slightly decline as new designs move to surface mount chip styles.

The dipped tantalum capacitor has historically represented the largest volume usage. The capacitor element is connected to radial lead wires and encapsulated by an inexpensive dip coating of epoxy.

This style displaced some aluminum capacitors in critical consumer applications twenty years ago. The consumer driven high volume demands drove the production costs down an expanded volume learning curve. However, as the world turns to surface mount technology, many of the newer designs will use a tantalum chip capacitor.

The chip style tantalum capacitor is the only style where growth is expected. The future growth rate is only limited by the intrinsic cost of the capacitor. The projection for 1994 of 7 billion units with a compound average annual growth rate of 27% per annum assumes that the combined efforts of the capacitor manufacturer and the tantalum materials supplier will permit rapid progression down a high volume cost learning curve. Please recall that the 1994 forecast for aluminum capacitors is over 50 billion units. An estimated 5 to 10 billion units of the aluminum capacitor market is available for displacement by the solid tantalum capacitor chip style **if** and **when** the aluminum capacitor production costs can be more closely approximated !

Wet tantalum capacitors are used almost solely in military applications and as such will gradually decline in volume with time.

GROWTH IN TANTALUM CAPACITORS

An empirical relationship between the demand for tantalum capacitors and the demand for integrated circuits is shown in Table IV. There appears to be a good correlation between the worldwide demand for each circuit element. Note the overheated demand for both tantalum capacitors and integrated circuits in 1984 due to system inventory building followed by an inventory rundown effect for both circuit elements in 1985 and 1986. The projected demand for tantalum capacitors previously forecast is remarkably close to the demand projected by using the established relationship to the forecast future demand of integrated circuits.

The mathematical relationship between the demand for integrated circuits and the demand for tantalum capacitors may be expressed (in billions of units) as

$$\text{Tantalum Capacitors} = 0.167 \times \text{Integrated Circuits} + 0.39.$$

In words, this relationship says that **one** tantalum capacitor will be required for each **six** integrated circuits used in the world. A remainder of about 390 million tantalum capacitors will be required for other basic and support circuit functions.

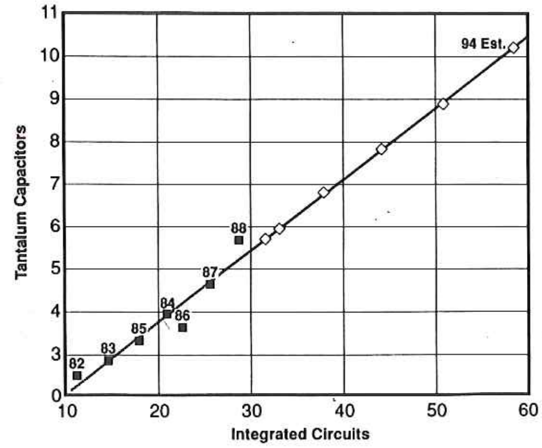

Table IV

TANTALUM CAPACITORS VS INTEGRATED CIRCUITS
World Shipments in Billions of Units

As a point of information, a similar excellent empirical relationship between the usage of integrated circuits and the demand for monolithic ceramic capacitors also exists. In this case, the ratio is 2.3 monolithic ceramic capacitors for each integrated circuit.

TANTALUM POWDER AND WIRE CONSUMPTION

	1988 Quantity Millions	1988 Tantalum Powder lbs/1000	1988 Tantalum Powder Total	1994 Quantity Millions	1994 Tantalum Powder lbs/1000	1994 Tantalum Powder Total
Metal	285	0.25	71	227	0.25	57
Molded	750	0.20	150	700	0.18	126
Dipped	2390	0.22	526	2265	0.18	408
Chips	2175	0.15	326	7081	0.15	1062
Wets	15	4.00	60	13	4.00	52
Total	5615	0.20	1133	10286	0.165	1705
TA Wire @ 15%			170			256
Total Tantalum Required			1303			1961

Table V

TANTALUM POWDER & WIRE CONSUMPTION
Gross Pounds (OW's)

Table V shows the estimated 1988 demand for tantalum capacitors by style, the average tantalum powder usage in pounds per 1000 pieces by style, the total tantalum powder usage, and an estimate of the total tantalum wire usage in pounds. The 1994 projections for both the unit capacitor volume by style and the powder consumption by style are shown.

The unit powder requirements for the metal case solid tantalum and the wet tantalum capacitor will probably remain static. Both styles involve some very large and/or high voltage ratings. The unit powder requirements for the molded style tantalum capacitors should decrease modestly as higher charge powder is utilized. The unit powder requirements for dipped tantalum capacitors will decrease more rapidly as this style has more low voltage ratings and thus very high charge powder can be designed into the anodes. The unit powder requirements for chip style tantalum capacitors will probably remain flat on average. The declining unit weight of anodes allowed by higher charge powders will be offset by the demand for higher CV (Capacitance times Voltage) ratings within the individual physical case sizes.

Tantalum wire usage will probably remain at about 15% of the total powder usage. This implies that the learning curve reductions in tantalum wire diameters and lengths will closely match the reductions in overall unit anode powder weights.

The overall tantalum capacitor unit volume is forecast to grow at an average annual rate of 13% while the total tantalum materials required for capacitors is forecast to grow at an average annual rate of 8.5%.

A new process for the production of tantalum and niobium oxide

This article has been written from the presentation made by Dr Norbert Krummen, Gesellschaft für Elektrometallurgie mbH, to the meeting of the T.I.C. on October 19th 1989

INTRODUCTION

Over the last decades the negative environmental impact of nitrogen-containing compounds such as ammonia and nitrates has been officially recognized.

Recent regulations on industrial and municipal waste water demand drastic reductions of these compounds as well as restrictions on the use of those chemicals or processes which may generate them.

This situation affects nearly all industries, especially in those cases where large volumes of critical chemicals are used and large volumes of solid, liquid or gaseous wastes are generated.

As it is practically impossible to reverse history, today's challenge is the development of new environmentally positive techniques which are as efficient as or even better than the existing ones.

The present chemical process (see Figure I) to extract the tantalum and the niobium from the various raw materials and the final conversion into salts, oxides and the metals makes use of large quantities of two chemicals :

— hydrofluoric acid (HF)
which is very corrosive and poisonous
and

— ammonia
whose environmental impact especially on fresh water is known.

Neither chemical is contained in the final products such as tantalum and niobium metal, oxides or carbides : they leave the system in the waste water and the solid residues at different stages.

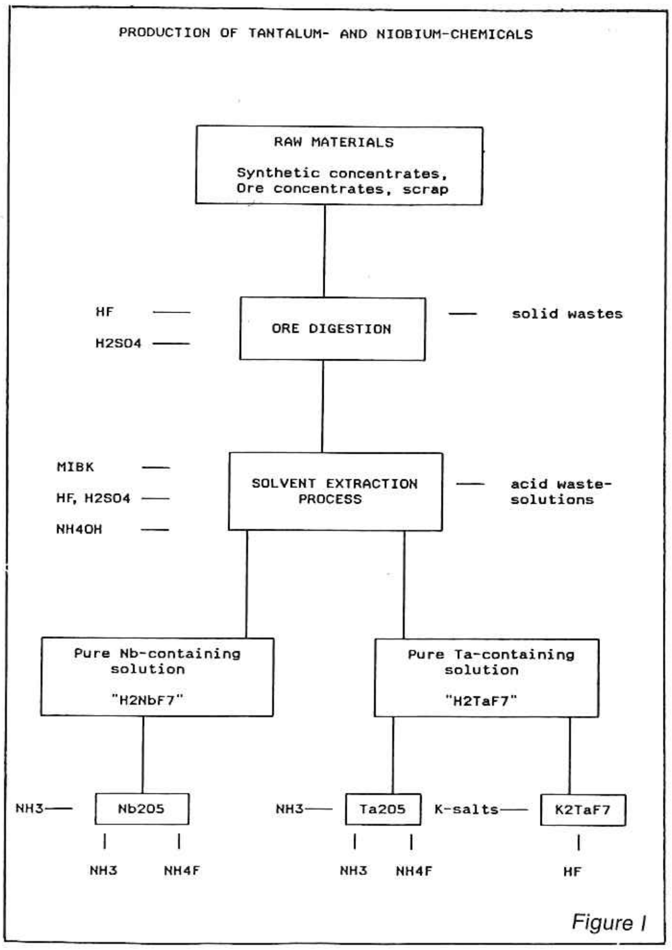

Figure I

Hydrofluoric acid and fluorides contained in the waste water are usually transformed into solid calcium fluoride which is environmentally neutral.

Ammonia has been discarded with the waste water as long as this was permitted.

Over the last decade most processors have installed a distillation process to recover ammonia from alkaline waste waters and recycle it into the tantalum process.

At GfE we have now developed an alternative technique which does not make use of ammonia at all, and in addition recovers part of the fluorine as sodium fluoride.

In this paper, both old and new methods are compared.

PRODUCTION OF Ta_2O_5/Nb_2O_5 VIA THE NH_3 ROUTE

The pure Ta-fluoride- or Nb-fluoride-containing solutions coming out of the solvent extraction process are precipitated with gaseous ammonia or ammonia-water (see Figure II). The product of precipitation is a complex ammonium-oxo-fluoro-niobate or tantalate. During the chemical reaction an NH_4F-containing solution is formed, which is removed from the system by decantation and/or filtration.

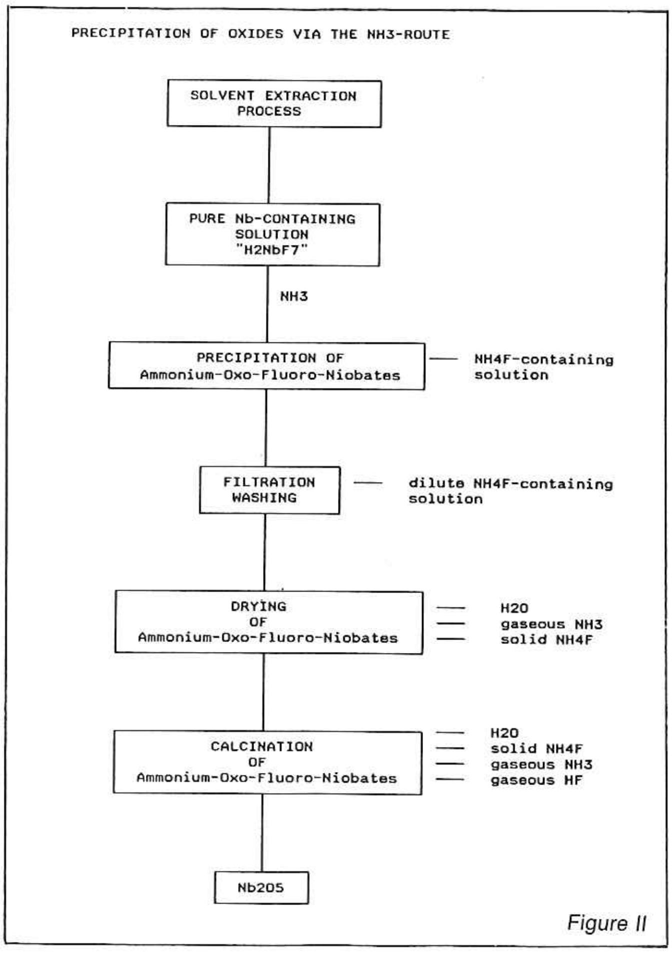

Figure II

After several washing operations and filtration the material is dried in a rotating furnace where mainly water and a small amount of ammonia is removed.

The final conversion to the oxide is effected during the final calcination at 900°C.

The complex ammonium-oxo-fluoro-niobate decomposes thermically at this temperature.

During calcination the remaining water and ammonia, hydrofluoric acid and ammonium fluoride are evaporated. These compounds are absorbed in a wet scrubber and the solution generated is treated in the waste water purification plant.

We can summarize the by-products of the oxide production as follows :

By-products	**Action**
NH_4F-containing solution	waste-water treatment precipitation as CaF_2
solid NH_4F	thermal decomposition to NH_3 and HF followed by wet absorption (H_2SO_4, NaOH)

The F-containing compounds are completely precipitated as CaF_2 and dumped, but the NH_4-containing compounds were until recently released without any treatment into the public waste water system.

PRODUCTION OF Nb_2O_5/Ta_2O_5 VIA THE NaOH-ROUTE

As mentioned before, new regulations specify that ammonia in waste waters must be drastically reduced, and eventually eliminated. After successful examination and development, GfE decided to use another process and to substitute ammonia by sodium hydroxide. The new process is based partly on the old tantalum (Marignac) process, where raw materials were fused with NaOH to the resulting sodium niobates or tantalates. After acid hydrolysis and dissolving in hydrofluoric acid, niobium and tantalum were separated by fractional crystallisation. The old Marignac process for the separation of niobium and tantalum was abandoned in the late 50's in favour of the process of solvent extraction with MIBK. The solvent extraction process yields pure tantalum- and niobium-containing solutions which are directly processed to the chemical products such as the oxides and K_2TaF_7.

We decided to use these pure solutions to substitute ammonia by NaOH and to precipitate sodium tantalate or sodium niobate.

The principle of the new process for the production of Nb_2O_5 is shown in Figure III.

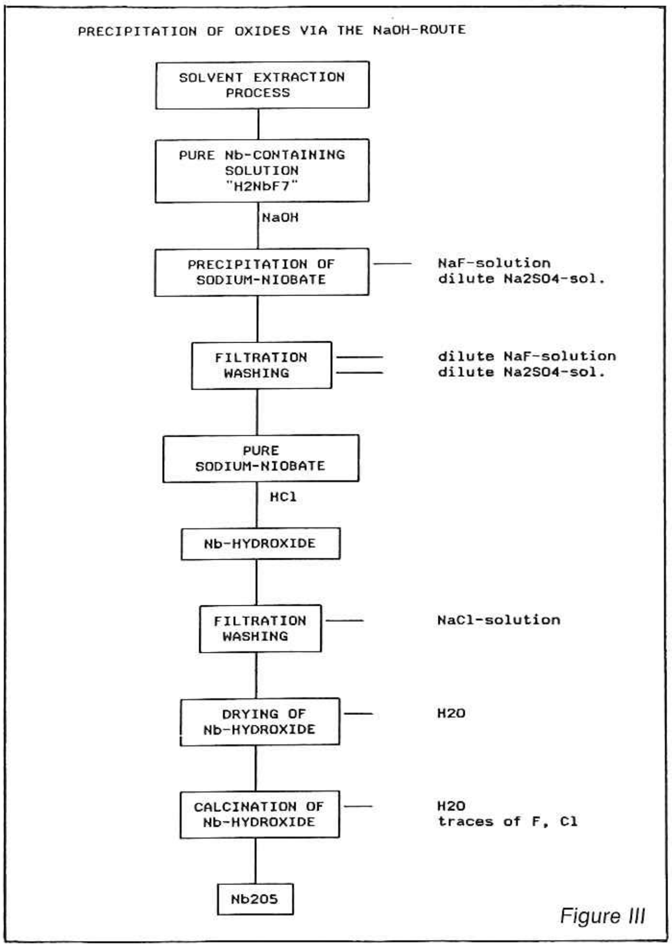

Figure III

The niobium-fluoride-containing solution from solvent extraction is mixed with NaOH solution and sodium niobate is precipitated. Unlike the classical NH_3 process this procedure gives a crystalline product which is $NaNbO_3.xH_2O$ and not a complex mixture of ammonium-fluoro-niobate.

After decantation the precipitated Na-niobate is filtered and washed.

During these procedures practically all the fluorine is removed from the system and is carried completely into the waste water. As sodium tantalate and sodium niobate are virtually insoluble under these conditions, a pure sodium fluoride solution is generated. This solution can be treated with calcium chloride, precipitating very pure calcium fluoride. Another alternative is to evaporate the water and produce pure sodium fluoride.

The remaining F-content after these operations is well below 0.1 % in the $NaNbO_3$ product. This is clearly different from the classical NH_3 process in which the fluorine has to be reduced by repeated washing operations and finally by calcination.

The precipitated and washed product is then hydrolyzed with hydrochloric acid. The final product of the hydrolysis is niobium hydroxide, and the by-product is sodium chloride. The niobium hydroxide is filtered off and washed with deionized water to remove the sodium chloride. The removal of sodium chloride is controlled by measuring the conductivity of the washing water.

After these operations, the hydroxide is dried and calcined. Another advantage over the previous process is that during this calcination only water is removed from the system. As there are no fluorine emissions, a simple filter may be used instead of a scrubber. The retained oxide is directly fed back into the furnace.

The significant differences compared to the classical NH_3-route are :

— the process consists of two chemical reactions and

— requires two filtration operations.

Especially closely controlled washing of the niobium hydroxide after hydrolysis is important for the quality of the product. Residues of sodium chloride cannot be removed thermically even at calcination temperatures of 900°C.

EQUIPMENT

The precipitation of the Na-niobate takes place in different reactors depending on the use of either a batch-process or continuous precipitation. Careful pH-control is required in each case.

Filtration and washing of the sodium niobate is carried out in a membrane filterpress, which proved to be the most efficient equipment for the removal of fluorine and sodium chloride.

The acid hydrolysis is done batchwise in rubberlined reactors by addition of hydrochloric acid to the Na-niobate.

The final filtration and washing operations take place again on a membrane filterpress.

The washed material is then dried and calcined in rotary kilns.

The equipment used is shown diagrammatically in Figure IV.

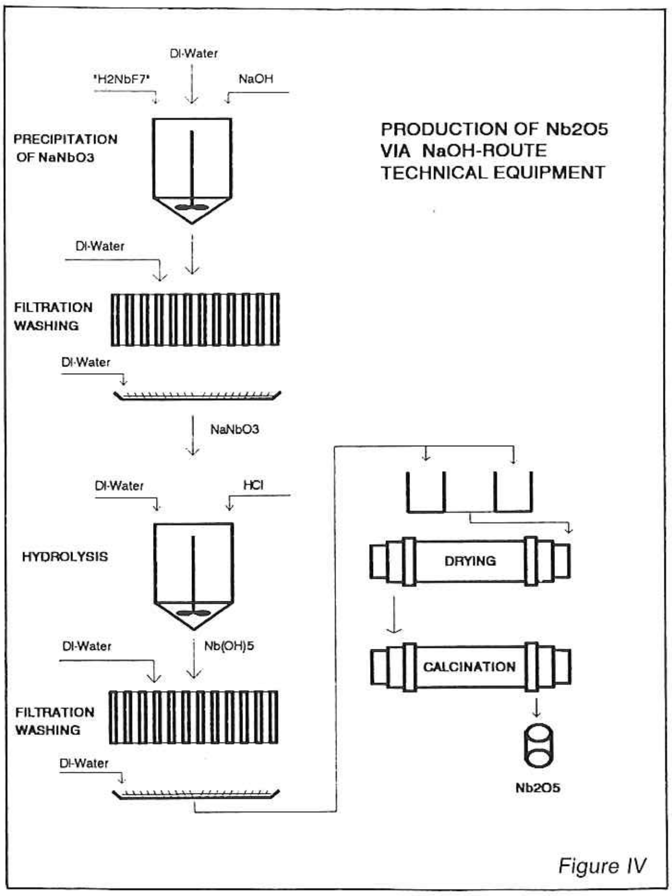

Figure IV

QUALITY

The quality of oxides made by the new process compares well with that of the existing one. The analysis for impurities of both the Ta_2O_5 and Nb_2O_5 differs only with regard to residual sodium and chlorine, and in each case this is less than 100 ppm in the calcined oxide.

Standard grade oxides for carbide manufacturing require only technical grade sodium hydroxide and hydrochloric acid, ceramic and optical grade qualities require pure chemicals.

Grain size is controlled via the concentration of the solutions.

SUMMARY

The process using NaOH as alternative precipitation agent for the production of Nb_2O_5 and Ta_2O_5 has been developed and technically introduced by GfE.

The advantages of the new process can be summarized as follows :

- ammonia-free waste water as a result of substitution of ammonia
- possible recovery of fluorine as pure CaF_2 or NaF
- substitution of wet scrubbers by dry filters as a result of simplification of the calcination process.

The remaining waste waters from this oxide production process only contain NaCl.

The Frankfurt General Assembly

Mr Pilgram, of Treibacher Chemische Werke, has sent the T.I.C. a set of the photographs he took at the Frankfurt General Assembly, and they are so good that we asked for his permission to print some of them here. This he kindly gave, and a selection is shown below.

Dinner after the Assembly

Ready for the plant visit

Heraeus briefing

Buffet lunch at Heraeus

MEMBERSHIP

The following companies were elected to membership by the Thirtieth General Assembly :

Carr Boyd Minerals
24 Outram Street,
West Perth 6005, Western Australia.

Ethiopian Mineral Resources Development Corporation
P.O. Box 2543,
Addis Abeba, Ethiopia.

Ginatta Torno Titanium
Via Brofferio 3,
10121 Torino, Italy.

Goldrim Mining Australia
317 Hunter Street,
Newcastle, NSW 2300, Australia.

Hi-Temp Specialty Metals
Beverly Rancocas Road & Industrial Dr.,
Willingboro, New Jersey 08046, U.S.A.

Institut National de Recherche Chimique Appliqué
Centre de Recherche, B.P. No 1,
91710 Vert-le-Petit, France.

Kennametal
P.O. Box 231,
Latrobe, PA 15650, U.S.A.

Special Metals Fabrication
Horndon Industrial Park,
West Horndon, Brentwood,
Essex CM13 3XG, England.

Alex Stewart (Assayers)
Caddick Road, Knowsley,
Merseyside L34 9ER, England.

Titanium International, Cookson Group
Thornhill Road, Solihull,
West Midlands B91 2HF, England.

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