

# T I C

## TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

### PRESIDENT'S LETTER

Dear Friends,

This issue of the T.I.C. Bulletin will presumably reach you just a few days prior to the beginning of the Tantalum/Niobium Symposium in Goslar. This meeting will no doubt offer ample opportunities for an exchange of opinions on the current situation of the tantalum/niobium industry. On the "eve" of this important event, please allow me to recapitulate what I consider to be the key functions of our organization: providing information and promoting business.

Information about raw materials resources, production, consumption and new trends in developments is provided in the form of statistics, reports or presentations at various meetings and is distributed, for example, by this publication.

This system has certainly contributed to the stability of our industry in recent years. For example, since all steps of production have become more transparent, capacities can be adjusted in time to meet future needs.

Luckily, we are once again experiencing an upturn in demand. This development can no doubt be handled without speculative price movements, as all members of our association know how to use the information provided.

Promoting business by introducing new applications for tantalum and niobium is another important task of the T.I.C. Unfortunately, details about these successful activities are not always easy to obtain, as most projects are either long-term or pursued by member companies directly.

The forthcoming T.I.C. Symposium in Goslar offers an excellent forum to serve both these key functions. In fact, we already expect a record turnout.

Before closing, I should like once again to thank all those who have devoted so much time and effort to the organization of this ambitious event. As you can see from the programme, the diversity of speakers and subjects is truly remarkable. Therefore, I look forward to welcoming as many of you as possible in Goslar.

Sincerely,

Peter Köhlert,  
President

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### INTERNATIONAL SYMPOSIUM : ABSTRACTS

#### TANTALUM - AN OVERVIEW

George Korinek  
*Tantalum-Niobium International Study Center*

The last Tantalum-Niobium International Symposium sponsored by T.I.C. took place in November 1988 in Orlando, Florida, U.S.A.. The following highlights describe the developments in the tantalum industry that have since taken place:

1. The primary production of tantalum decreased to about one half.
2. The processors' shipments remained constant until 1993 when an increase of 10% was realized.
3. No new major applications for tantalum were reported.

The trends and developments in the major consumption areas will be discussed.

#### TANTALUM RAW MATERIAL SUPPLY

John Linden  
*Gwalia Consolidated Ltd.*

Tantalum raw material supply and availability have undergone significant changes over the seven years since the last time I addressed this subject in Orlando in 1988.

The changes that have developed include an almost complete elimination of tantalum bearing tin slags as a by-product from tin-smelting operations, the development of significant new hard rock primary tantalite mines in Australia, the significant increase in recycling of tantalum units by the processing industry and changes in the supply and demand balance in the CIS and China.

Supply from unsophisticated operations in Africa and Brazil is difficult to quantify but is a significant proportion of total supply to processors.

Inventories of low grade slags and synthetic concentrates continue to make up the primary supply and demand imbalance.

#### AN OVERVIEW OF THE NIOBIUM INDUSTRY

Harry Stuart  
*Niobium Products Company Inc.*

A summary of the niobium industry will be presented to illustrate the great diversity of uses for niobium in all its purities.



All product forms will be covered. Special emphasis will be given to applications of niobium not covered in other papers in the symposium. Some commercial aspects of the niobium business will be explained where appropriate, and an attempt will be made to forecast future prospects for this unusual specialty metal.

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### **HYDROMETALLURGICAL PROCESSING OF Ta/Nb COMPOUNDS - PRESENT STATE OF THE ART**

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Joachim Eckert  
*H.C. Starck GmbH & Co KG*

Hydrometallurgy has made considerable progress in processing of Ta/Nb compounds. Ta/Nb separation shifted from fractional separation to solvent extraction in the early seventies. This decisive change has been modified and continuously improved: today Ta/Nb compounds of highest purity are processed on an industrial scale in large tonnage. Within the last few years, HCST's efforts have been concentrated on improving the wet chemical process by installing internal recycling routes to reduce the consumption of chemicals such as  $H_2SO_4$ , to reduce the amounts of sludges and to recycle  $NH_3$  and HF. Recent process variations make it possible not only to process raw materials with different Ta/Nb ratios but even to produce highly concentrated solutions of tantalum starting from a raw material containing only 4-6%  $Ta_2O_5$  but 30-35%  $Nb_2O_5$ .

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### **RECENT DEVELOPMENTS IN Ta ORE PROCESSING**

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Patrick M. Brown  
*Cabot Performance Materials*

The tantalum-niobium technical community continues to face ever increasing environmental challenges. As part of Cabot Performance Materials' commitment to operating its facilities in an environmentally responsible manner, a new MIBK Recovery System has been installed. Presented are some of the technical details which assisted in establishing design and operating parameters.

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### **THE PRODUCTION OF FERRO-NIOBIUM IN AN ELECTRIC ARC FURNACE AT CBMM**

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C.A. de Faria Sousa  
*Cia. Brasileira de Metalurgia e Mineracao*

From 1966 to 1991, the standard ferro-niobium used in microalloyed and heat resistant steels was produced at CBMM by aluminothermic reduction in an open reactor, which is the classic process for refractory alloy production.

In 1989, CBMM decided to build a new metallurgical plant for FeNb production, with the main objective of improving environmental controls and working conditions.

In 1991, the new plant began running using a closed reactor with fume control in a bag house, slag granulation, FeNb casting in cast iron moulds and automated mixing of raw materials. This plant was in operation from July 1991 to May 1994.

In June 1994, CBMM started up the operation of an electric arc furnace in its metallurgical plant, with the main objective of reducing aluminium consumption, replacing iron oxide with metallic iron powder. All other improvements, which had already been incorporated were maintained.

This new 4.25MVA submerged arc furnace has a capacity of 22 000 tons/year of alloy.

This paper describes the new CBMM metallurgical plant for the production of ferroniobium and presents the results obtained during the first 12 months of operation with the combined EAF melting-aluminothermic reduction process.

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### **FeNb BY THE READING ALLOYS PROCESS AND METHODS OF CONTINUOUS IMPROVEMENT**

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Brian J. Higgins  
*Reading Alloys, Inc.*

Advances in analytical detection methods have led to more stringent nickel-niobium master alloy specifications from super alloy producers, as well as an increase in the number of elements which must be reported. By building on its technically advantageous "Water Cooled Copper Furnace Thermite Process", Reading Alloys continues to meet these increasing quality demands of the vacuum melt industry. Through the use of statistical process controls not only in the manufacturing processes, but also in the characterization and directed use of raw materials, the nickel-niobium thermite process capability has been finely tuned to new levels of product consistency in controlling impurities. State of the art analytical instrumentation is required to support the process control improvements, as are well documented quality assurance procedures.

This paper not only describes the use of modern process control techniques by Reading Alloys in improving niobium master alloys' quality and consistency, but also explains how the same information can be used to negotiate realistic customer specifications.

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### **REVIEW OF THE TECHNOLOGY OF MICROALLOYING IN PIPELINE AND AUTOMOBILE STEELS**

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Friedrich Heisterkamp  
*Niobium Products Company GmbH*  
and Harry Stuart  
*Niobium Products Company Inc.*

Steels for pipeline and automotive application count for approximately 50% of niobium microalloying. In particular steels for large diameter pipe have been the workhorse in the development of high strength microalloyed steels in the last 20 years, fulfilling the steadily rising requirements for oil and gas transportation. In this context the combination of niobium microalloying with a thermomechanical treatment process provides the means to improve simultaneously strength and toughness of such steels. The key to this development is grain refinement, which results from the special processing. If additional strength is required, besides niobium other microalloying additives such as vanadium and titanium are applied.

For automotive steels the formability is the most important property. Therefore, a new type of steel was developed - a dual phase steel - which exhibits a better combination of strength and formability; besides the generation of second phases in the microstructure niobium is added for extra grain refinement.

For very severe sheet forming processes, interstitial free steels have been developed, in which after a severe decarburising and denitriding process the remaining carbon and nitrogen is fixed mainly by titanium and combinations of titanium and niobium. This type of steel is also increasingly used for galvanized or other continuously annealed sheet steels.



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## THE ROLE OF NIOBIUM IN MODERN STAINLESS STEELS

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Anthony J. DeArdo  
*Basic Metals Processing Research Institute  
University of Pittsburgh*

The use of niobium in modern stainless steels has increased remarkably within the last decade. This increase has accelerated within the last two years in North America, especially in grades used in the automotive industry. The reasons for this increase in use of niobium are lower net production costs within the steel plant, better formability and weldability in the fabrication shop, and superior performance in service applications.

Modern stainless steels contain combined carbon and nitrogen levels of less than 200-300 ppm as a result of advanced steelmaking. However, even these levels are too high for optimum processing and performance. Lower levels of carbon and nitrogen can be achieved through the addition of titanium and/or niobium that are much stronger carbide and nitride formers than is chromium. This phenomenon is called stabilization.

This paper will review the physical metallurgy of the stabilization of stainless steels, and will highlight and explain the benefits of using niobium.

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## NIOBIUM IN STEEL CASTINGS, FORGINGS AND STRUCTURALS

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Geoffrey Tither  
*Niobium Products Company Inc.*

The well-established fields of application for Nb-containing, microalloyed steels are automotive and related areas, and linepipe. Together with other microalloyed steel usage these account for about 75% of all niobium consumption. However, recent developments in the production of higher quality steel castings, forgings, and long products have opened up the opportunity for increased usage of Nb-containing microalloyed steels.

The present paper will discuss the metallurgy, steel design, properties and applications of Nb-microalloyed cast steels, forgings and structural sections.

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## TRENDS AND REQUIREMENTS OF VEHICLE ELECTRONICS

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Thomas Raith  
*Daimler-Benz AG*

In the future, the requirements faced by vehicles in terms of

- environmental protection (reduced fuel consumption and pollutant output)
- customer demands (for individual, functional and economical vehicles) and, at the same time
- optimization of lead times and resource utilization

will continue to grow. In order to develop vehicles which face up to these requirements and to overcome traffic problems the automotive industry will be forced to use electronic systems to an increasing extent. The objectives will include reduced cost, space requirements and low weight.

The use of electronics to meet these requirements will largely

determine whether the automotive industry remains competitive in the future.

Environmental protection and customer requirements mainly lead to improved or completely new vehicle functions. The time required to implement these functions in vehicles can be significantly reduced by new organizational forms of co-operation between vehicle manufacturers and their component suppliers, for example by sharing development methods and tools.

In order to master the new and additional functional requirements and to master the implementation of these functions in high-quality systems as rapidly as possible, it will be essential to optimize the interaction between numerous electronically controlled functions and between electronic and mechanical components. Of necessity, the optimization of a large number of electronic functions in vehicles will lead to fewer but more complex electronic systems and structures characterized by an ongoing integration of individual functions and by optimized interfaces.

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## OUTLOOK FOR TANTALUM CAPACITOR DEMAND

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David E. Maguire  
*Kemet Corporation*

The paper reviews the basic drivers for the growth in capacitor demand, the growth history of capacitors by generic type, and a projection of demand for the year 2000. Historical and projected demands are then focused on tantalum capacitors by leaded and surface mount styles. The paper concludes with a review of the 'learning curve' consumption of tantalum in capacitors and a projected tantalum powder and wire demand forecast for the year 2000.

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## HISTORY AND FUTURE VIEW ON THE DEVELOPMENT OF TANTALUM CAPACITOR TECHNOLOGY

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Takehiko Nakata, K. Morimoto, Y. Saiki, T. Nishiyama  
*NEC Toyama Ltd.*

Tantalum capacitors are widely used as the most suitable high charge small capacitors for surface mount technology.

After a brief review of the history of tantalum capacitor technological developments, the introduction of newly developed tantalum capacitors will be discussed with particular reference to their great possibilities in the growing field of electronic industries.

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## EXPECTATIONS OF TANTALUM CAPACITORS FOR WIRELESS COMMUNICATIONS

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James B. Crego  
*Motorola Inc.*

The paper will discuss the expected growth of the cellular/wireless communication industry, and the possibility of using the recent growth in the wireless communication industry to predict the future, such as easing of the growth rate and when this might happen. The significance of the growth rate in the wireless industry for tantalum capacitor demand, and the capacity of the industry to keep pace will be assessed. A shortage of tantalum capacitors could stimulate replacement of tantalum capacitors by ceramic capacitors. Different case sizes could be



required in the next few years, and almost certainly a change of mix of sizes will be needed.

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### **APPROACHING THE LIMITS? THE LATEST DEVELOPMENTS IN SOLID TANTALUM ELECTROLYTIC CAPACITORS**

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William A. Millman  
*AVX Limited*

The revolution in circuit board component technology, SMT, and its inherent severe demands upon performance, is today's driving force behind the tantalum capacitor displacing less compatible technologies which today enjoy major shares of the capacitor markets. They are used in the most demanding of circuit applications because of their SMT compatibility, small size, performance and high reliability. Circuit designers are demanding new requirements and conditions of use such as switching speeds, higher operational frequencies, lower voltage with higher capacitances, all within smaller and lower profiled packages.

These circuits and assembly requirements have led to advances in tantalum powder technology and counter electrode development which have brought about a need to understand the latest improvements in tantalum capacitor technology available to the circuit designers.

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### **TANTALUM POWDER AND TANTALUM WIRE FOR CAPACITORS**

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Terrance B. Tripp  
*H.C. Starck Inc.*

The explosive growth in the demand for surface mount chip (SMC) solid tantalum capacitors since 1990 has created many challenges for both the tantalum capacitor and the tantalum powder and wire manufacturers. The capacitor manufacturer is expected to supply parts with more capacitance in an ever smaller package. For the tantalum manufacturer, this translates into the need to provide wire and higher capacitance powders more suitable for SMC technology. The trends in powder physical properties are to higher usable surface area, a more uniform particle size distribution, and improved flow. Chemically, the latest generation of capacitor grade powders has lower concentrations of alkalis, transition metallics, and carbon. Controlling the oxygen content of these high performance powders is a major challenge which involves using deoxidation, nitriding, and vacuum packaging. The new SMC technology has forced a trend to finer, straighter wire. There has also been a move to harder wire and less need for grain size stabilization.

In this paper, we will examine developments in the manufacturing of capacitor grade tantalum powder and wire. The presentation will close with speculation about future trends.

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### **TANTALUM CAPACITOR POWDERS FOR THE FUTURE**

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Hongju Chang  
*Cabot Performance Materials*

Tantalum capacitors are highly reliable, surface mount compatible, and volumetrically efficient. The future capacitor market will continue to demand these desirable qualities; in

addition, it will also require low ESR and low cost. In this paper, the physical and chemical characteristics of the tantalum material needed to help tantalum capacitors maintain their competitiveness are discussed; suitable examples are also provided.

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### **THE CHANGE FROM LEADED TO SURFACE MOUNT TANTALUM CAPACITORS AND THE EFFECT ON RAW MATERIAL USAGE AND SUPPLY**

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Dennis M. Zogbi  
*Paumanok Publications, Inc.*

This presentation will establish the model for tantalum metal powder consumption in the worldwide tantalum capacitor industry, and indicate how the change from leaded to surface mount configurations and the subsequent downsizing of tantalum chips has affected and will continue to affect consumption of capacitor grade tantalum powder, by world region. The presentation will also show how trends in tantalum capacitor production by case size will affect future demand for capacitor grade tantalum powder, and how important the stability of the production of the physically larger leaded tantalum capacitors is to the stability of raw material usage and supply. Also, comparisons will be made between tantalum metal powder consumption in the tantalum capacitor market and titanate and palladium consumption in the ceramic capacitor market.

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### **DECOUPLING REQUIREMENTS OF INTEL MICROPROCESSORS**

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James Neal  
*Intel Corporation*

#### **The Market**

The personal computer is rapidly entering the home market due to advancements in both hardware and software which have made the PC indispensable for the home office and popular as an entertainment device. Unit shipments are steadily increasing, with total PC system shipments expected to reach 58 million units in 1996, 36 million based on Pentium processors, according to In-Stat. If a typical Pentium processor based PC is assembled with 10 tantalum chip capacitors, the total demand in 1996 would exceed 360 million units due to Pentium motherboards alone. Add-in cards and peripherals would increase this number further.

#### **Capacitor Design Considerations**

Three types of capacitors are widely available commercially: ceramic chip, aluminum electrolytic, and tantalum. Ceramic capacitors have excellent parasitic properties (ESR and inductance), are stable over temperature, highly reliable, low cost and surface-mountable, but are only available in relatively low values. Aluminum electrolytic capacitors are available in extremely large values and at low cost, but have higher parasitics and lower reliability, and are typically not surface-mountable. Tantalum capacitors have reasonable parasitics, good reliability, are available in reasonably large values, are surface-mountable, but at a higher cost.

These trade-offs are facing PC designers as they work with more powerful processors with ever-increasing decoupling requirements. Demand for tantalum and tantalum capacitors is likely to continue to increase rapidly if availability increases and pricing remains stable.



## RECENT DEVELOPMENT OF NON-ACTIVE INTEGRATED COMPONENTS IN MULTI-LAYER MODULES

Bernhard Mussler  
IBM Produktion GmbH

The various possibilities of multi-layer modules and the user markets of HTCC (High Temperature Cofired Ceramic) will be briefly reviewed and a few examples will be given. The opportunities and future importance of LTCC, especially for automotive and telecommunication applications, will be emphasized.

The basic material requirements and key parameters for the production of multi-layer substrates will be summarized. Furthermore the current production processes will be presented.

The material properties of currently used metals and ceramics will be compared critically and their limitations for LTCC applications will be reviewed.

The new LTCC technology offers for the first time on a broad basis the possibility of integrating non-active components, e.g. embedded resistors, capacitors and inductances, into the multi-layer module. Recent development activities and new results will be presented and examples of future opportunities will be shown.

## THE USE OF TANTALUM AND NIOBIUM COMPOUNDS IN THE ELECTRONICS INDUSTRY

Karlheinz Reichert  
H.C. Starck GmbH & Co. KG

Tantalum and niobium compounds such as oxides, niobates and tantalates are playing an increasingly important role as starting materials for the production of electronic components. These include multi-layer ceramic capacitors, resonators, filters, buzzers, actuators and sensors. The use of Ta- and Nb-compounds in the electronics industry can be divided into single crystal applications and electroceramic applications, which can be additionally subdivided into dielectric ceramics, microwave ceramics, piezoelectric ceramics, electrostrictive ceramics and pyroelectric ceramics. Especially for the electroceramic market an annual growth of between 5 and 10% is estimated over the next five years and therefore this market is of particular interest.

This presentation will give an overview of present and future use of Ta/Nb compounds in the electronics industry.

## RECENT DEVELOPMENT OF TANTALUM PASTES FOR SCREEN PRINTING

Rudolf Huenert  
H.C. Starck GmbH & Co. KG

The application of metal pastes in electronics industries is widespread: well-known examples are hybrids, multi-layer ceramic capacitors, single- and multi-chip modules and, of course, the numerous types of printed circuit boards. Screen printing technology has made it possible to develop and produce finely structured conductive layers on various types of substrates.

As the dimensions of capacitors became smaller and smaller in recent years, the idea, well-known in principle, simply to print a tantalum capacitor, was revitalized. Recent development work performed at H.C. Starck's central research shows that it is

possible to define a solvent-free, water-dilutable rheological system of synthetic additives in combination with a special type of tantalum powder to form a screen printable tantalum paste.

## PREPARATION AND PROPERTIES OF NIOBIUM- AND TANTALUM-OXIDE-BASED FUNCTIONAL COMPONENTS

Andreas Schoenecker and F. Schlenkrich  
Fraunhofer-Institut, Ceramic Technologies and Sinter Materials

Niobium-oxide-containing functional ceramic materials of perovskite and tungsten bronze crystal structure are showing attractive properties for applications in electronics. The use of ferroelectric phases for building capacitors, actuators, pyroelectric sensors, and optical waveguides is well-known.

By adjusting the quality of semi-finished products (powders, powder mixtures, liquid phase precursors), the preparation technology, and the ceramic microstructure, the following material properties have been achieved:

- Low sintering ( $T_s < 900^\circ\text{C}$ ) PMN-ceramic with excellent dielectric properties.
- Ferroelectric relaxor ceramic with high electrostrictive strain, e.g.  $s = 0.12\%$  at  $10 \text{ kV/cm}$ .
- Ferroelectric thin films of complex chemical composition based on water soluble precursors, for example in the system PMN-PNN-PT.

The preparation, structure and property relationships will be discussed in the paper.

## DIELECTRIC CHARACTERIZATION OF FERRO- ELECTRIC $\text{Pb}_3[\text{Mg}_2\text{Nb}]\text{O}_9$ RELAXOR CERAMICS

Detlev Hennings  
Philips GmbH Forschungslaboratorien

Complex ferroelectric perovskites, e.g. "lead magnesium niobate" (PMN), are increasingly used as high-permittivity dielectrics in ceramic multilayer capacitors.

PMN and related complex perovskites exhibit a number of peculiar dielectric effects. These are:

- Diffuse ferroelectric phase transitions, resulting in very high and broad dielectric maxima at the Curie point;
- Frequency dependence of the permittivity and losses ("relaxation" effect);
- Strong ageing of the dielectric properties.

Relaxation and ageing effects are strongly dependent on frequency and temperature. Moreover, these effects depend also on the chemical composition, the preparation technology, the amount of impurities and added heterovalent dopes.

## $\text{Nb}_2\text{O}_5$ AND THE MLCC INDUSTRY

Alan Rae  
TAM Ceramics Inc.

$\text{Nb}_2\text{O}_5$  has been used for many years in the Multi-Layer Ceramic Capacitor (MLCC) industry to suppress grain growth and modify electrical properties. There was great promise of growth in



relaxor dielectrics based on lead magnesium niobate about five years ago, but this consumption did not develop rapidly due to concerns of lead emission during firing, poor physical strength and plating problems.

New generation Y5V and X7R materials have greatly enhanced processing, physical and electrical properties and consumption is growing very rapidly. The combination of high dielectric constant and low electrode metals costs makes these products extremely attractive for low-cost, high capacitance and small size applications.

Potential Nb<sub>2</sub>O<sub>5</sub> requirements in terms of performance and volume will be outlined.

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### **Ta/Nb OXIDES IN OPTICAL GLASSES FOR MODERN OPTICAL SYSTEMS**

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Hans F. Morian  
*Schott Glaswerke*

The development of high performance optical systems (i.e. lenses with high speeds or large zoom ranges) is closely linked with the invention of high index lanthanum optical glasses containing Ta<sub>2</sub>O<sub>5</sub> and/or Nb<sub>2</sub>O<sub>5</sub>.

Criteria for the introduction of Ta/Nb oxides into glass without crystallization will be discussed.

The significance of these glasses in modern lens systems is presented.

In the ophthalmic industry one also needs high index eye glasses for the correction of high myopia. Refractive indices of 1.7, 1.8 and even 1.9 are now available in Nb<sub>2</sub>O<sub>5</sub>-containing lightweight glasses.

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### **TRENDS OF LiTaO<sub>3</sub> AND LiNbO<sub>3</sub> SINGLE CRYSTALS FOR ELECTRONIC DEVICE APPLICATIONS**

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Toshio Nishimura and Sadao Matsumura  
*Toshiba Corporation*

Since 1965, when LiTaO<sub>3</sub> and LiNbO<sub>3</sub> single crystals were first grown by the Czochralski method, there have been numerous developments on their applications to electronic devices such as piezoelectric devices, pyroelectric detectors, nonlinear optical devices and surface acoustic wave (SAW) devices. Among these, the crystals have been gaining the most warranted position in the SAW device application field. For applications to these electronic devices, many intensive efforts have been made to establish mass-production conditions of high quality crystals and their scaling up in diameter, with lower mass-production cost.

In this report, trends of LiTaO<sub>3</sub> and LiNbO<sub>3</sub> single crystal growths and the crystal qualities will be demonstrated. Moreover, application to various electronic devices and properties of various cut substrate of LiTaO<sub>3</sub> and LiNbO<sub>3</sub> single crystals for their electronic devices will be reviewed.

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### **SYNTHESIS, PROPERTIES AND PERFORMANCE OF YTaO<sub>4</sub> X-RAY PHOSPHORS**

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William Zegarski  
*DuPont Imaging Systems*

Tantalum oxide is the basis for the preparation of radio-luminescent rare earth tantalates for medical imaging. The high

density of these phosphors and the ability to modify their emission to suit radiographic film sensitivity makes them very useful in this application. High levels of diagnostic acuity for the radiologist and reduced x-ray exposure for the patient result from the use of tantalate phosphors. These materials are very versatile in that their performance can be adjusted to suit a variety of imaging application needs. Adding various activators controls the emission and efficiency of the resultant phosphor. Crystal properties, such as average size and morphology, are determined by the flux used during preparation. These properties determine the manner in which the phosphors are used in x-ray intensifying screens for medical diagnosis. The various factors involved in phosphor and screen preparation will be described in this talk.

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### **CATALYSIS BY TANTALUM AND NIOBIUM OXIDES**

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Takashi Ushikubo  
*Mitsubishi Chemical Corporation,  
Yokohama Research Center*

Recently, niobium oxide has attracted attention as a component of catalysts, e.g. catalyst support, solid acid catalyst, selective oxidation of catalysts. However, there have been very few papers concerning catalysis by tantalum oxide, although the structure and physical properties of these oxides are similar. In this paper, the catalytic properties of tantalum oxide and the recent development of catalysis by tantalum and niobium oxides will be reported, with special reference to niobium-containing mixed metal oxides and highly acidic hydrated tantalum and niobium oxide catalysts.

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### **HIGH-PURITY NIOBIUM ELECTRO-WON FROM NIOBIUM PENTACHLORIDE**

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Gregor Mori, G. Brecka, T. Wittner, P. Paschen  
*Department of Non-ferrous Metallurgy  
University of Leoben*

Niobium electrolysis has been performed in a NaCl-KCl-NaF-NbCl<sub>5</sub> electrolyte. By means of electrolytic refining, the average valency of the niobium ions was determined as well as the crystallization behaviour. In electro-winning runs, the influence of the main parameters, such as temperature, R<sub>F</sub> (mole ratio F/Nb), chemical prerelution, niobium content of the melt and current density, has been determined with respect to low specific energy consumption and high purity of the deposit.

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### **SOLUBILITY OF NIOBIUM AND TANTALUM PENTACHLORIDES IN APROTONIC SOLVENTS AND HIGH MOLECULAR COMPOUNDS AND DEVELOPMENT OF METHODS FOR PRODUCTION MATERIALS**

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Dmitri V. Drobot  
*Department of Rare Metals,  
Moscow State Academy of Fine Chemical Technology*

Results of the investigation of the solubility of higher niobium, tantalum and zirconium chlorides and their mixtures with aluminium chloride in titanium tetrachloride and its solution with carbon tetrachloride are discussed. Equipment was elaborated for investigation of the solubility of those systems where the total pressure was less than 150 kPa. The association degree of niobium and tantalum pentachlorides in TiCl<sub>4</sub> is shown to be determined by the concentration of MCl<sub>5</sub> (M=Nb, Ta). The mutual influence of chlorides in solutions is discussed. The results



described can be used in a reduction process for production of niobium and tantalum alloys with zirconium, aluminium and other metals. Niobium and tantalum pentachlorides dissolve in alcohols and high molecular weight organic compounds (for example, oligomeric derivatives of alkyl phenols). The results of interaction in both cases are compounds which can be used for synthesis of oxides and carbides. Thermal stability of such compounds, and methods of producing niobium and tantalum carbides, are discussed.

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## **TANTALUM AND NIOBIUM ELECTROREFINING AND ELECTROPLATING**

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E. Polyakov  
*Institute of Chemistry, Kola Science Centre,  
Russian Academy of Sciences*

The high cost of tantalum and niobium is an obstacle to their wide application, notwithstanding the unique properties of these metals. In cases where the properties of the material depend on those of its surface, articles of common metals coated with tantalum or niobium can be used. The most appropriate method of making such articles is electrolysis of molten salts. This provides a uniform distribution of the coating and purification in the process of deposition. The merits and the shortcomings of various electrolytes are compared, the mechanisms of electrode and chemical reactions which are the basis for the method are discussed.

The process is realized in a hermetic electrolyser under an atmosphere of inert gas, in the temperature interval between 650 and 750°C. The rate of coating deposition reaches 50 µm/hour for tantalum, and for niobium 100 µm/hour. The production rate of the process of obtaining coarse powders and dendrites is several times faster. The content of metallic impurities - less than 10 ppm of each - can, if necessary, be reduced 10-fold. Data on the coating structure and properties, and corrosion resistance, are given. The trends for further research are considered.

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## **TANTALUM-CONTAINING HARD ALLOYS FOR SURFACE TECHNOLOGIES**

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Erich Lugscheider  
*Materials Science Institute,  
Aachen University of Technology*

Surface technologies nowadays are an integral part of modern production lines and strategies.

Characterized by a variety of processes and materials, surface technologies offer the possibility of refining cheap components with comparatively thin surface coatings. Custom made composite systems can be realized either in repair and overhaul or in new part production.

Processes extend from physical and chemical vapour deposition, electroplating and thermochemical processes to thermal spraying, deposition welding and plating.

Numerous coating materials lead to a multitude of obtainable functions. Among functions such as resistance to corrosion, hot gas corrosion and oxidation; thermal and electrical insulation; medical, optical and decorative properties, the wear resistance is of greatest importance for economic reasons and potential. In this area hard alloys, especially nickel based ones, are of particular interest.

These so-called self-fluxing alloys, materials with characteristic, congruent hard phases based on borides, carbides

and occasionally also silicides, are mainly processed by thermal spraying and surfacing by welding.

Conventional nickel hard alloys show, besides complex carbides, only binary borides as wear resistance phases in the nickel matrix. Refractory metals differ fundamentally in their boride structures. Developments in the 80's verify the possibility of stabilizing more complex borides in nickel based alloys. When alloying tantalum, or even niobium, these tau-borides with their cubic structure occur.

One of the remarkable features of this new group of alloys in surface technology is to be seen in the high values of wear and also corrosion resistance.

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## **OXIDATION PROTECTION FOR METALLIC FASTENERS ON SPACE VEHICLES**

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Peter Roedhammer and W. Knabl  
*Technology Center,  
Metallwerk Plansee*

Advanced re-entry vehicles and orbital planes are designed to employ fastening elements made of refractory metal alloys for the assembly of hot structures (thermal protection systems, scram jet intakes, etc.). Operating temperatures will range between -150°C and 2000°C depending on application and design. Coatings will be required in order to protect the fasteners against oxidation upon multiple re-entries.

We have investigated the manufacture and the performance of coated fasteners made of niobium and tantalum alloys in combination with different coating systems (silicides, noble metals, SiC/TiN multi-layers). Different types of coating techniques (electron-beam ion-plating, CVD, slurry deposition, pack-cementation) were adapted to allow uniform deposition onto the complexly shaped fasteners.

Cyclic oxidation tests have been carried out in air at pressures up to 1 bar and at temperatures up to 1450°C. Life-times up to 700h have been achieved at 1300°C and 1 bar. Oxidation and failure mechanisms will be discussed based on the results of micro-structural and compositional analyses.

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## **TANTALUM AS A MATERIAL OF CONSTRUCTION FOR THE CHEMICAL INDUSTRY - A CRITICAL SURVEY**

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Michael Renner  
U. Gramberg and H. Diekmann  
*Bayer AG*

Materials selection for chemical plant equipment is based on economic considerations; the least expensive material withstanding service conditions for a given service time is chosen. For integrity of equipment in the CPI chemical inertness is an essential factor. Of all metallic materials used for CPI equipment, tantalum is second to none regarding chemical inertness. The price of tantalum, however, prohibits its broad application for construction. There are only two ways by which the demand for tantalum in this field could be promoted significantly. First, a major increase in the aggressivity of service conditions in process engineering caused by process development or modification. Second, cost reduction via tantalum price dilution by means of duplex systems application, utilizing the advantages of a cheap base metal and a thin tantalum lining or coating. The more important systems of this type are discussed and evaluated regarding their potential of development.



## MODELIZATION OF THE FORMING OF TANTALUM PARTS WITH A FINITE ELEMENTS CODE

Christophe Thiebaut  
C. Bonnet and J.M. Morey  
*Commissariat à l'Energie Atomique,  
Centre de Valduc*

Tantalum, a refractory material, has many advantages and combines the following interesting characteristics:

- good resistance to corrosion
- high conductivity
- high density
- good processability

It is this last property which we will consider mainly in our presentation. Tantalum can be processed in ways such as: rolling, forging, winding and drawing. Such experiments as forging are currently used in the Valduc Center, one of the research centers of the French Nuclear Energy Department.

We are now developing new tools for assessing our competitiveness in this field and tend to use finite elements code in connection with the industrial use of metalforming. In this presentation, a comparison between the setting-up of a tantalum part and its modelization with such a code will be given.

Numerical and experimental results, such as the forming load and the strain variations in the equatorial region, will be compared. The results will be used to modelize the forming of a hemispherical cup of 150 mm diameter.

## TANTALUM CARBIDE AND NIOBIUM CARBIDE IN HARD METALS

Bernard North  
*Kennametal Inc.*

The carbides of tantalum and niobium are common additives used to optimize the properties of cemented carbides and, more recently, cermets used primarily for metal cutting. An overview is presented of trends in the metal cutting process itself, and in the development of tooling materials and the manufacturing processes employed to make them. At least qualitative statements are made on how these trends might be expected to affect the demand for tantalum and niobium carbides.

## ADVANCED COATING TECHNOLOGY FOR AERO GAS TURBINES

Mohinder P. Malik  
*Lufthansa*

The performance and efficiency of gas turbines is directly related to the gas inlet temperature and pressure. It is well established that high pressure turbine components rely on meeting the high performance and efficiency challenge on the incorporation of suitable coatings in the design stage.

Modern gas turbines also rely on light weight components and structures with high temperature capability for achieving a higher thrust to weight ratio. For thin walled components the substrate and coating integrity assumes a particular significance.

In this highly competitive field the direct operating costs of gas turbines are related to durability and repairability of the turbine components. A multitude of processes and techniques is

needed during the manufacture and repair of gas turbines and the environmental issues, the disposal and recyclability aspects must be brought into line with the first costs of procurement and direct operating costs of the power generating equipment.

Developments in coating technologies allow the use of methods which may be used to repair cost intensive polycrystalline and anisotropic nickel base turbine components.

The control of tip clearance is critical for maintaining higher fuel efficiency and the developments in innovative turbine seals and blade tipping emphasize that coatings go a long way towards meeting these demands.

The presentation will focus on the following areas:

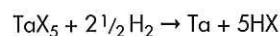
- Oxidation and corrosion resistant coatings
- Thermal barrier coatings (TBC's)
- Sealing systems
- Advanced repair techniques for gas turbines.

The objectives will be to give a broad overview of the status of coatings development to meet the challenge of high efficiency low emission systems.

## NANOSIZED-TANTALUM POWDERS

Gerhard Winter  
*H.C. Starck GmbH & Co. KG*

Nanosized tantalum powders have been synthesized by the so-called CVR process (chemical vapour reaction). The basic chemical reaction is the reduction of tantalum halides with hydrogen:



The specially constructed CVR reactor makes it possible to adjust the particle size of the tantalum powder from a few nm up to several hundred nm.

The sinterability of these powders depends very much on the particle size and influences the structure of the densified polycrystalline tantalum ingot.

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