

## T.I.C. activities

### NOVEMBER 1992 : PHUKET, THAILAND

The Thirty-third General Assembly will be held on Tuesday November 17th 1992 as part of a conference at the charming Meridien Hotel on the island of Phuket in Thailand.

The registration desk will be open on Monday November 16th, and on that evening the programme will feature a welcoming cocktail party and buffet supper. Following the General Assembly on Tuesday morning, the rest of the day will be taken up by the presentation of technical papers. In the evening all the delegates and their ladies will be the guests of Thaisarco at a formal banquet.

A field trip on Wednesday will visit the tin smelter of Thailand Smelting and Refining Company (Thaisarco), and also a tin dredge and the ore dressing plant of S.A. Minerals. The tantalum processing plant of Thai Tantalum should be in commission by November, and an additional tour to Mab Ta Phut is planned as an option for Thursday November 19th.

The location of the meeting in Phuket and association with sources of raw materials have prompted the technical session to focus on primary production, with papers promised by Thaisarco on tantalum in tin slags and by Greenbushes (of Gwalia Consolidated) on the hard rock project at its mine in Western Australia. But the programme will also range more widely, with an outlook over the industry and a survey of statistics collected by the association, new developments in the niobium industry and in the use of niobium in steels, and news of research in trace analysis, among other papers whose topics are about to be finalised.

Nominated delegates of member companies will be sent invitations in August, but participants from non-member companies are also welcome to join the technical sessions, social events and plant tours: those interested should contact the Secretary General of the T.I.C., 40 rue Washington, 1050 Brussels, Belgium, telephone 02 649 51 58, fax 02 646 05 25 (please note the change of number), telex 65080.

### OCTOBER 1993 : AUSTRIA

The Executive Committee has accepted a kind invitation by Treibacher Chemische Werke to visit its plant as part of a meeting in early October 1993, with the conference taking place in Vienna.

### APRIL 1992 MEETING : NEWS

The Executive Committee reviewed the financial situation of the T.I.C. in April, and decided to maintain the annual membership fee for 1992-93 at the same level as for 1991-92. Some applications for membership are already lined up for the next General Assembly. In addition to planning forthcoming assemblies and the administration of the T.I.C., the Committee is to undertake an expansion in its own numbers to broaden the field of activity covered by members. It is also giving some thought to holding another International Symposium to follow that in Orlando in 1988.

The Technical Adviser, Mr Rod Tolley, is assembling the material for display on the T.I.C.'s stand at the exhibition of the 1992 Powder Metallurgy World Congress, to be held in San Francisco from June 21st to 26th 1992. He is also preparing a publicity brochure on tantalum and niobium: readers of the Bulletin who may wish to have copies should let us know.

New fax number for T.I.C.  
(02) 646 05 25

## President's message

*(Abridged from his speech to the Brussels informal meeting on April 7th 1992)*

Previously the T.I.C. was composed of members from Western nations only, but, with the addition of Russia and the prospect of greater participation from China, it has taken on added responsibility, becoming a more global international organisation comprising a network in tantalum- and niobium-related industries worldwide. However, because Russia, along with its partners in the C.I.S., and China are unfamiliar with free-market economics, in order to avoid confusion due to unpredictable behaviour on their part as has been seen in other nonferrous metal markets, it will be necessary to help to deepen their understanding of the world niobium and tantalum markets, and to work together to develop tantalum- and niobium-related industries.

At a time of worldwide recession it is not easy to consider what form that development should take, but perhaps we can learn from Japan's reaction to the first oil crisis: it directed its efforts toward low-energy-consumption, high-technology industries, which resulted in a succession of new electronic products. From that came a major growth in markets for many new materials, including those using tantalum.

In my opinion future development needs to be based not on producing existing materials of ever higher quality but on determining what new capabilities our present materials have.

With regard to tantalum and niobium we have already seen in recent years important new applications for them, and their commercial exploitation has been greatly helped by resource availability. A real international development programme, involving the C.I.S. and China, will make them truly universal commodities.

Yoichiro Takekuro  
President

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## T.I.C. statistics

### TANTALUM

#### PRIMARY PRODUCTION

(quoted in lb Ta<sub>2</sub>O<sub>5</sub> contained)

	1st quarter 1992
Tin slag (2 % Ta <sub>2</sub> O <sub>5</sub> and over)	147 565
Tantalite (all grades), other	222 368
<b>Total</b>	<b>369 933</b>

Note : 14 companies were asked to report, 13 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, Pan West Tantalum (Wodgina Mine production), Tantalum Mining Corporation of Canada, Thailand Smelting and Refining

#### QUARTERLY PRODUCTION ESTIMATES

(quoted in lb Ta<sub>2</sub>O<sub>5</sub> contained)

LMB quotation :	US \$ 30	US \$ 40	US \$ 50
2nd quarter 1992	309 400	398 600	414 000
3rd quarter 1992	309 400	398 600	414 000
4th quarter 1992	369 400	398 600	414 000
1st quarter 1993	377 400	398 600	414 000
2nd quarter 1993	377 400	398 600	414 000

Note :

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

#### PROCESSORS' RECEIPTS

(quoted in lb Ta contained)

	1st quarter 1992
Primary raw materials (e.g. tantalite, columbite, struverite, tin slag, synthetic concentrates)	412 530
Secondary materials (e.g. Ta <sub>2</sub> O <sub>5</sub> , K <sub>2</sub> TaF <sub>7</sub> , scrap)	70 272
<b>Total</b>	<b>482 802</b>

Note : 17 companies were asked to report, 16 replied.

#### PROCESSORS' SHIPMENTS

(quoted in lb Ta contained)

Product category	1st quarter 1992
Ta <sub>2</sub> O <sub>5</sub> , K <sub>2</sub> TaF <sub>7</sub> and carbides	117 735
Powder/anodes	203 683
Mill products	100 089
Ingot, unworked metal, other, scrap, alloy additive	67 268
<b>Total</b>	<b>488 775</b>

equivalent to 659 847 lb Ta<sub>2</sub>O<sub>5</sub>.

Notes :

In accordance with the rules of confidentiality, categories have been combined as shown.

17 companies were asked to report, 16 replied. For both receipts and shipments by processors, reports by the following companies are essential before the data may be released :

Cabot Performance Materials, W.C. Heraeus, Kennametal, Metallurg Group, Mitsui Mining and Smelting, NRC Inc., Showa Cabot Supermetals, Hermann C. Starck Berlin, Thai Tantalum, Treibacher Chemische Werke, Vacuum Metallurgical Company, V Tech

### NIOBIUM

#### PRIMARY PRODUCTION

(quoted in lb Nb<sub>2</sub>O<sub>5</sub> contained)

	1st quarter 1992
Concentrates : columbite, pyrochlore	13 947 713
Occurring with tantalum : tin slag (over 2 % Ta <sub>2</sub> O <sub>5</sub> ), tantalite, other	168 574
<b>Total</b>	<b>14 116 287</b>

Note :

16 companies were asked to report, 14 replied. The companies which reported included the following, whose reports are essential before the data may be released : Cambior, Mineração Catalao de Goiás, Niobium Products Co. (CBMM)

#### PROCESSORS' SHIPMENTS

(quoted in lb Nb contained)

	1st quarter 1992
Compounds and alloy additive : chemical and unwrought forms (e.g. NbCl <sub>5</sub> , Nb <sub>2</sub> O <sub>5</sub> , NiNb, FeNb [excluding HSLA grades])	1 001 554
Wrought niobium and its alloys in the form of mill products, powder, ingot and scrap	
(i) Pure niobium	33 950
(ii) Niobium alloys (such as NbZr, NbTi and NbCu)	113 469
HSLA grade FeNb	8 366 870
<b>Total</b>	<b>9 515 843</b>

Note : 18 companies were asked to report, 17 replied. Reports by the following companies are essential before the data may be released : Cabot Performance Materials, W.C. Heraeus, Kennametal, Metallurg Group, Mitsui Mining and Smelting, Niobium Products Co. (CBMM), NRC Inc., Hermann C. Starck Berlin, Teledyne Wah Chang Albany, Thai Tantalum, Treibacher Chemische Werke, Vacuum Metallurgical Company

## Capacitor statistics

### TANTALUM CAPACITORS IN THE U.S.

#### U.S. CONSUMPTION OF TANTALUM CAPACITORS

(thousands of units)

	4th quarter 1991
Metal-cased (incl. wets & foil)	22 066
Dipped (radial)	93 454
Chips	195 276
Other (molded axial & radial)	97 517
<b>Total U.S. consumption</b>	<b>408 313</b>
Add U.S. production for export (all types)	109 884
<b>Total worldwide U.S. sales</b>	<b>518 197</b>

(Data from EIA)

#### EUROPEAN TANTALUM CAPACITOR SHIPMENTS

(thousands of units)

4th quarter 1991 164 725

(Data from ECTSP)

#### JAPANESE TANTALUM CAPACITOR PRODUCTION AND EXPORTS

(thousands of units)

4th quarter 1991  
Production of which exports 1 208 200  
262 416

(Data from JEIDA)



# Production of tantalum and niobium capacitors in the C.I.S.

This paper was given by Mr Y.M. Mudrolyubov, Positron Research & Manufacturing Corp., St Petersburg, at the Philadelphia meeting of the T.I.C., October 1991

It is the purpose of this communication to present a general view of the range of tantalum and niobium capacitors manufactured in the C.I.S., their specifications and the requirements imposed upon the tantalum and niobium materials used in the production.

The range of our commercially available capacitors comprises all of the principal versions of both solid- and liquid-electrolyte tantalum capacitors as well as their niobium counterparts whose production is due to the severe restrictions on the use of tantalum materials which applied in our country until recently.

It is for this reason that the field of use of this class of capacitors was limited exclusively to aerospace equipment, which affected the volume and structure of the production of these products, resulting for example in an increased production volume of capacitors housed in a metal case and thus creating a serious shortage in the most popular groups of products used in consumer equipment, particularly those for surface mounting and automated placement.

The capacitor types include :

(a) Liquid-electrolyte tantalum capacitors :

- Sealed capacitors in a silver case. To ensure better reliability in multiple thermal cycle conditions, capacitors in a palladium-silver case are optionally available.
- Hermetically encapsulated all-tantalum capacitors (in a tantalum case).
- Capacitors with metal foil electrodes.

The standard capacitance range is essentially 1.0 to 1000  $\mu$ F, the working voltages ranging from 3.2 to 125 VDC. The leakage currents must not exceed 0.002 to 0.003 CV.

On a special order, e.g. for ultradeep drilling equipment, hermetically encapsulated capacitors are produced in limited quantities with a maximum operating temperature of up to 250 °C and a limited lifetime.

(b) Solid-electrolyte capacitors :

- Tantalum capacitors in a hermetic metal case with axial leads, and their niobium counterparts.
- Tantalum capacitors in a plastic case with radial leads and their niobium counterparts.
- Dipped tantalum capacitors with radial leads and their niobium counterparts.

(c) Surface-mount capacitors, moulded or open.

The process for the manufacture of the above groups of tantalum capacitors is essentially quite traditional, as used by any manufacturer of tantalum capacitors.

An exception is the technique for the formation of the cathode coating of solid-electrolyte capacitors. In the vast majority of cases, at our factories, the cathode electrode is formed by successive deposition, on to the manganese dioxide, of a varnish-carbon coating and silver-bearing conducting pastes.

In our opinion, a cathode assembly prepared in this way provides for enhanced operational reliability of capacitors and reduces the need for costly silver.

I would like to call your attention to the original construction of our K53-28 solid-electrolyte capacitors designed for use in a wide frequency range and finding application in pulseforming and processing circuits, in particular in the output circuits of switched-mode power supplies. The use of an original technique for the formation of a flat


			
MODEL	DIMENSIONS	CAPACITANCE RANGE	WORKING VOLTAGES VDC
K53-28 TANTALUM	10x7.1x5.0 to 20x17x5.0	10-470 $\mu$ F	6.3-40
K53-31 NIOBIUM	10x7.1x5.0 to 15x12x5.1	0.68-150 $\mu$ F	6.3-20

Figure 1 : Flat capacitor

anode makes it possible to lower by almost an order of magnitude the equivalent series resistance and impedance of such capacitors. Figures 2 and 3 show the typical frequency characteristics of flat capacitors as compared to their counterparts with a sintered porous anode.

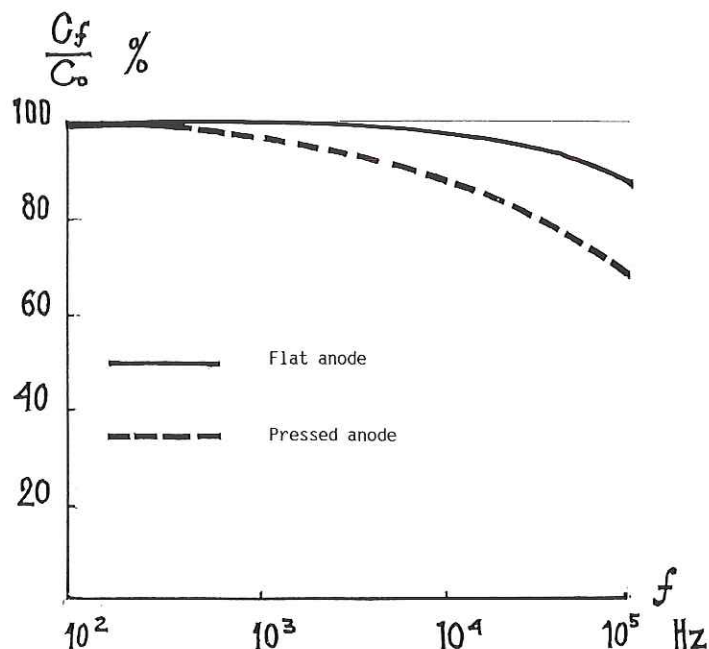


Figure 2 : Frequency vs capacitance (25v x 47  $\mu$ F)

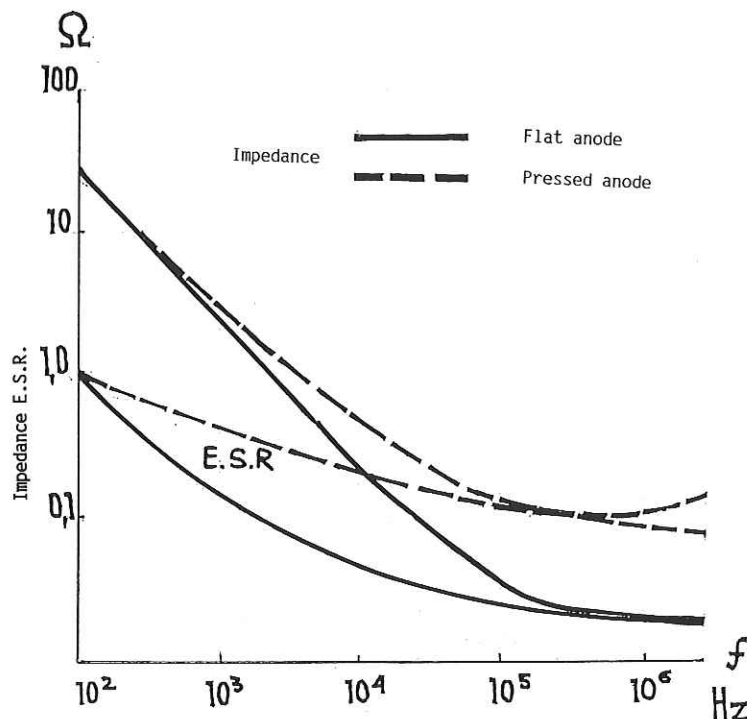


Figure 3 : Frequency vs impedance (25v x 47  $\mu$ F)

As a rule, a set of most stringent general requirements is put on tantalum capacitors. Capacitor lifetimes of 15 000 to 50 000 hours are specified for the operating conditions with a maximum allowable voltage bias whereas in less severe conditions the lifetimes may reach 100 000 hours and above. A set of mechanical stresses specifies an allowable vibration of 40 g at 5000 Hz and single impacts of 1500 g. The specified operating temperature range is -60... +125-155 °C.

Also, salt fog and funginertness requirements are specified.

The manufacture of niobium capacitors involves the same set of steps which, however, feature a number of essential parametric differences relating to sintering, formation and pyrolysis.

Clearly, niobium capacitors are inferior to their tantalum counterparts in some performance characteristics such as the maximum working voltage or leakage current. But this is compensated for by the low cost and availability of niobium materials.

The C.I.S. industry produces capacitor-grade tantalum and niobium materials.



Characteristics of powder	Type, Grade								
	BB-1	BB-2	BB-3	2 A	2 B	5 A	5 B	HB-1	HB-2
Capacitance characteristics, μF.V/g	600 1000 <sup>±</sup>	1100 1500 <sup>±</sup>	1800 2200 <sup>±</sup>	1800 2000 <sup>±</sup>	2200 2600 <sup>±</sup>	4000 4900 <sup>±</sup>	4900 5600 <sup>±</sup>	10000	12000
DC leakage, μA/μF • V × 10 <sup>-3</sup>	≤ 1			≤ 0,45 ÷ 0,65		≤ 2		≤ 1	
Sintering temperature, °C	2000 ÷ 2100			1900 ÷ 1950		1600 ÷ 1650		1500 ÷ 1600	
Pressed density, g/cm <sup>3</sup>	8,4 ÷ 9,6		7,9 ÷ 8,2	7,9 ÷ 8,2		6,5 ÷ 7,2		5 ÷ 6	
Chemical composition, ppm									
Niobium	2000			2000		2000		2000	
Iron	20			50		20		60	
Silicon	10			30		20		300	
Molybdenum	10					10		30	
Tungsten	30			100		30		30	
Titanium	8			8		8		10	
Hydrogen	100			100		100		1000	
Oxygen	1500			400 ÷ 1500		2500		3000	
Nitrogen	120			120		120		2000	
Carbon	30			100		100		100	

Table 1 : Tantalum powder, capacitor grade

Characteristics of powder	Type, Grade				
	I	II	III	V	agglomerated niobium powder
Capacitance characteristics, $\mu\text{F.V/g}$	1500	2500	4000	9000	20 000
DC leakage, $\mu\text{A}/\mu\text{F} \cdot \text{V} \times 10^{-3}$	$\leq 15$	$\leq 15$	$\leq 15$	$\leq 15$	$\leq 25$
Sintering temperature, °C	1850 ÷ 2000	1850 ÷ 2000	1850 ÷ 2000	1530 ÷ 1560	1300 ÷ 1350
Pressed density, g/cm <sup>3</sup>	4,7 ÷ 5,1	4,4 ÷ 5,0	4,3 ÷ 4,8	3,8 ÷ 4,4	3,4 ÷ 4,0
Chemical composition, ppm					
Iron	30	30	30	30	20
Silicon	30	30	30	30	50
Tungsten	50	50	50	30	30
Molybdenum	30	30	30	30	30
Tantalum	1000	1000	1000	600	—
Copper	30	30	30	30	50
Nitrogen	150	150	150	150	150
Oxygen	2000 ÷ 3000	2000 ÷ 3000	2000 ÷ 3000	2000 ÷ 4000	—
Carbon	50	50	50	50	200
Hydrogen	20	20	20	20	20

Table 2 : Niobium powder, capacitor grade

Table 1 presents the classification of our commonly used tantalum powders and the principal requirements put on them. This is a series of VV<sub>1</sub>, VV<sub>2</sub> and VV<sub>3</sub> fragmentation-type powders with a specific charge of 500 to 2000  $\mu\text{F V/g}$  for the manufacture of capacitors with maximum working voltages, the 2A, 2B, 5A and 5B fragmentation-type powders with a specific charge of 2000 to 5000  $\mu\text{F V/g}$  as well as the NV-1 and NV-2 sodium thermal reduction-type powders with a specific charge of 10 000 to 12 000  $\mu\text{F V/g}$ .

It is clear to the specialists that a very important group of powders with a high specific charge is lacking in this list whereas the existing ones are still far from being perfect.

Table 2 presents the classification of our niobium powders.

We use a total of four classes of fragmentation-type powders with a specific charge of 1500 to 9000  $\mu\text{F V/g}$  as well as a thermal reduction-type powder with a developed surface area of particles which has a specific charge of 20 000  $\mu\text{F V/g}$ .

Finally, compact materials. These are tantalum wire, 0.15 to 1.0 mm in diameter, niobium wire, sheet tantalum, 0.25 to 1.0 mm thick, for the manufacture of cases and anodes for flat capacitors as well as foil, 0.02 mm thick, for the manufacture of electrodes for foil capacitors.

I have presented a summary of the results of the activity of an industry coordinated by our corporation. This industry has been, for many years, isolated in a vast but closed area, which, in many respects, accounts for the drawbacks doubtless noticed by the specialists. We hope that our joining such an authoritative organisation as the Tantalum-Niobium International Study Center will favour integration of our industry into the world community on a mutually beneficial basis, and we thank our sponsors, Hermann C. Starck and AVX, for their support.

## Developments in copper-niobium and copper-tantalum alloys

[The following is an abridged version of the talk given by Dr W.A. Spitzig of Iowa State University to the TIC Philadelphia meeting in October 1991.]

### CONSUMABLE ELECTRODE ARC CASTING

During the late 1970s three independent research groups (including one group at Iowa State) developed a technique to prepare a superconducting wire containing an aligned structure of Nb<sub>3</sub>Sn filaments in a bronze matrix. This structure was called an "in-situ processed" composite due to the formation of niobium filaments produced during the mechanical deformation of a Cu-Nb alloy. Further processing of the Cu-Nb wire entailed coating the drawn wire with Sn and then diffusing the Sn into the wire where it reacts to form the Nb<sub>3</sub>Sn superconducting compound.

Large castings of Cu-Nb alloy were required to produce the large quantities of wire needed for superconductor applications. A process called consumable electrode arc casting was developed at Ames which provided larger castings of Cu-Nb and eventually other Cu-X alloys.

At the same time, interest was growing in high strength Cu-Nb alloys. It was first demonstrated by Bevk that a levitation melted alloy of Cu-18wt%Nb could be drawn to very large reductions having tensile strengths of over 2200 MPa (319 ksi). Unfortunately, these strengths were achieved in very fine wires (0.024 mm) (Fig. 1).

In addition, Bevik demonstrated that other refractory elements such as vanadium produce very similar filamentary microstructures with high tensile strengths.

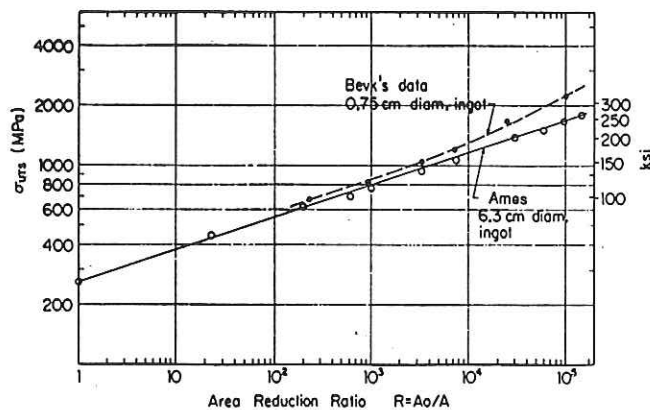


Fig. 1 : Tensile strength achieved by drawing Cu-18wt%Nb to very large reductions

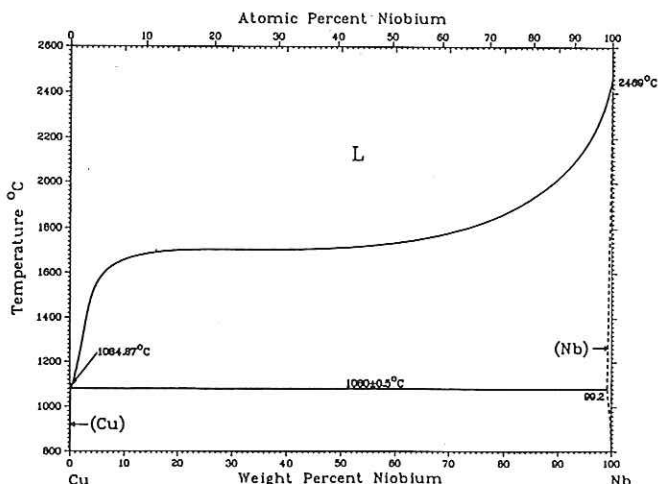
If we require high strength and high conductivity in the same alloy, we must reconcile the following considerations :

- (i) High strength requires a metal alloy because strength cannot be achieved by the intrinsic properties of a pure element without altering the element by cold work or second element additions. Any such alteration will affect the conductivity adversely.
- (ii) High conductivity requires a high conductivity base element. Few good choices are available (copper, silver, gold or aluminium) and any alteration to these pure elements will reduce the intrinsic conductivity.

We are therefore faced with trading either strength for conductivity or conductivity for strength.

The choice of the ideal material should therefore start with one of the four high conductivity metals mentioned above. That will need to be alloyed with an element which has no solubility in the parent metal (in order to avoid reduction in conductivity). The alloying element must be in a form which gives the highest strength while still leaving the alloy formable. It is known that this is met by having the alloying metal dispersed through the parent as lamellae or dispersoids, less than 100 nm apart. The known alloy systems to meet all these requirements are copper alloys with chromium, molybdenum, niobium, tantalum, vanadium, and tungsten.

Cu-Nb is the typical phase diagram of such alloy systems. Upon solidification, the as-cast microstructure consists of an array of refractory element dendrites in a matrix of nearly pure copper.



D.J. Chakrabarti and D.E. Laughlin, 1982.

Fig. 2 : Phase diagram : copper-niobium

The consumable electrode arc casting process requires the electrodes not only to contain the right proportion of the two alloying elements, but also to be of a design to produce a homogeneous ingot.

Electrode configurations were refined over a period of several years gradually moving toward a more symmetrically homogeneous configuration. Most electrodes were composed of a rod of OFHC copper with slots machined along the longitudinal axis to accommodate the placement of the refractory metal strip or rod.

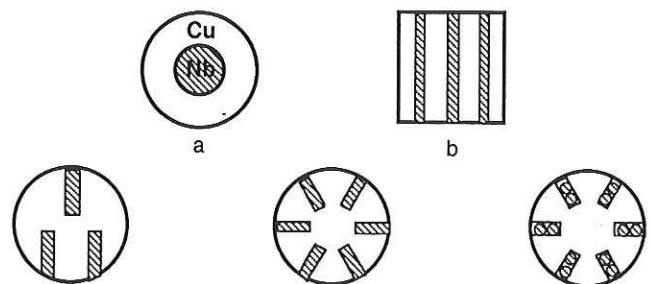


Fig. 3 : Electrode designs

Early electrode designs, such as shown in a and b, figure 3, were found to produce unalloyed globs of refractory metal requiring a second melting for complete alloying. Further dispersion of the refractory metal in the electrode helps to alleviate these unalloyed globs.

### MELTING AND CASTING IN GRAPHITE MOLDS

Consumable electrode arc casting initially began by melting the electrode into a split graphite mold liner placed inside a water cooled copper mold using an arc power of approximately 62 kW (28 Volts, 2200 Amps.) (see Fig. 4).

The melting containment was under a static partial pressure of approximately 380 mm of welding grade argon during the melting process.

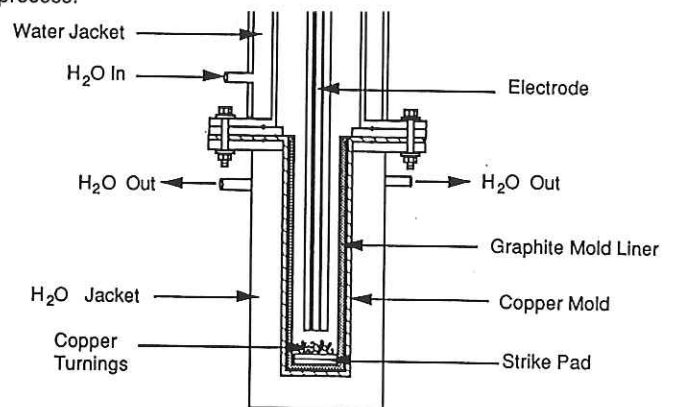


Fig. 4 : Consumable electrode arc casting apparatus (with graphite mold)

Graphite molds had disadvantages. Ingots cast into graphite molds were characterized by the following features :

- Carbon contents of 120 to 400 ppmw.
- Dendrite sizes of 6-8  $\mu\text{m}$ .
- Melt pool depths of 5-6 cm.
- Hot tears extending up to 1/4 of the ingot length.

High carbon contents were detrimental to the formation of well defined dendrite structures causing them to have shorter aspect ratios and resulting in poor filament development. Carbon contents were reduced by using a split graphite mold which had been plasma sprayed with an yttria stabilized zirconia coating. Carbon contents dropped to levels around 30-50 ppmw.

Although these carbon levels were still suspected of having detrimental effects on the formability of the niobium filaments, it was subsequently found that this could not be substantiated.

Later, thick-wall water cooled copper molds were used (see Fig. 5).

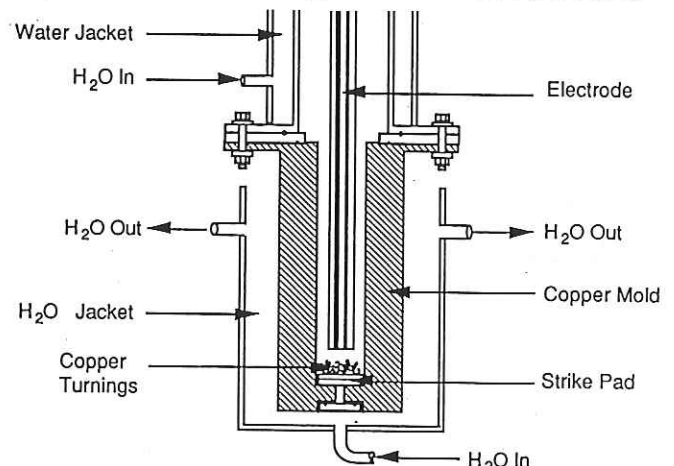


Fig. 5 : Consumable electrode arc casting apparatus (with copper mold)



These had the advantages of a reduced carbon contamination (to 10 ppmw) and an increased cooling rate, producing smaller dendrites (3-4  $\mu\text{m}$ ), a shallower melt pool (2-3 cm), and reduced hot tearing (2-3 cm depths). An additional improvement resulted from replacing welding grade argon with ultra high purity grade argon, evacuated and purged several times. Interstitial oxygen levels then dropped by nearly an order of magnitude.

## INGOT HOMOGENEITY

Ingot homogeneity remains a problem in the as-cast ingots despite improvements in the electrode homogeneity and casting procedures.

Transverse sections from the top and bottom of as-cast ingots were compared. These revealed several interesting characteristics:

Ingots of Cu-Ta were found to be more inhomogeneous than those of Cu-Nb.

Dendrite size across the radius of the Cu-Ta ingot varied slightly while the Cu-Nb alloy has much smaller dendrites toward the outer periphery.

A more thorough evaluation of the inhomogeneity was conducted by longitudinally sectioning several as-cast ingots. The inhomogeneity patterns revealed the following:

Cu-Nb alloys appeared to be fairly homogeneous after the first melt.

Both Cu-Nb and Cu-Ta alloy ingots show a thin Cu rich layer at the exterior ingot surface. The thickness of this layer is much smaller for Cu-Nb (1-2 mm) than for Cu-Ta (4-6 mm) alloys.

Cu-Ta alloys contained a dense annular region of Ta dendrites near the outer surface and distinct globs of unalloyed Ta.

A model of the electrode tip was proposed by Verhoeven which suggests that during the melting process the copper melts and runs down along the extending tips of the refractory metal dissolving the refractory metal and forming a liquid alloy (Fig. 6).

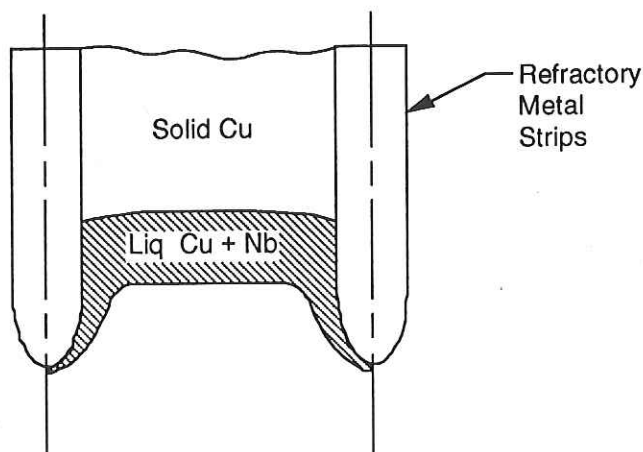


Fig. 6 : Model of electrode tip configuration

The following observations have been made during melting:

- 1) The arc is established between the refractory metal tips of the electrode and the melt pool.
- 2) During melting the tip length decreases until the arc gap voltage becomes greater than that of a near neighbour and the arc then jumps to the near neighbour.
- 3) Since the arc originates on the refractory metal tip there is a high probability that local melting will occur on the tip before alloying with the copper. This would produce a distinct refractory metal droplet that enters the molten pool and solidifies.
- 4) The likelihood of this event is increased as the distance between the refractory metal tips and the copper rod is increased. Such is the case for Cu-Ta when compared to Cu-Nb alloys.

The as-cast ingot crowns of Cu-Nb versus Cu-Ta alloys exhibited distinct differences similar in nature to those in the longitudinal sections:

A pronounced annular ridge in the Cu-Ta ingot crown compared to the Cu-Nb ingot crown.

This annular ridge occurs just inside the outer 4-6 mm Cu rich layer and corresponds to the dense region of Ta dendrites seen in the longitudinal section.

These variations in the ingot homogeneity are believed to be related to the difference in:

- 1) The melting point of the alloys where the liquidus temperature of Cu-20 vol. % Ta is 150 degC higher than that of Cu-20 vol. % Nb causing the Cu-Ta alloy to freeze more rapidly.

The increased rate of heat extraction in the copper mold may result in a mushy condition near the mold wall. The mushy condition results in a higher viscosity.

The Ta dendrites in this mushy region are then allowed to coarsen during the solidification of the interdendritic liquid.

- 2) The relative differences in density between the refractory metal and copper. The density ratio at the liquidus temperature is 2.1 for Cu-Ta and 1.1 for Cu-Nb.

Homogeneity, particularly in the case of copper-tantalum, is achieved by a second melt for which the electrodes are prepared from the first melt ingot.

These electrodes have a very fine dispersion of refractory metal dendrites and are less likely to produce distinct refractory metal droplets during melting.

Consumable arc casting technology has now reached the point where ingots of copper-niobium alloys weighing up to 1500 kg have been produced (at the NRC Inc. plant at Newton, Mass.). A large supply of this material is now available for commercial applications.

## POWDER METALLURGY

The powder metallurgy approach can provide an alternative route to high strength copper-niobium and similar alloys. Fig. 7 compares the strength of drawn wire produced from a consumable arc melted cast alloy of Cu-20%Nb, and from the same composition hot extruded from the mixed powders, degassed and sealed in a copper can. The strength of pure copper subject to the same deformation is also shown and it highlights the great increase in strength obtainable with the niobium addition and cold working.

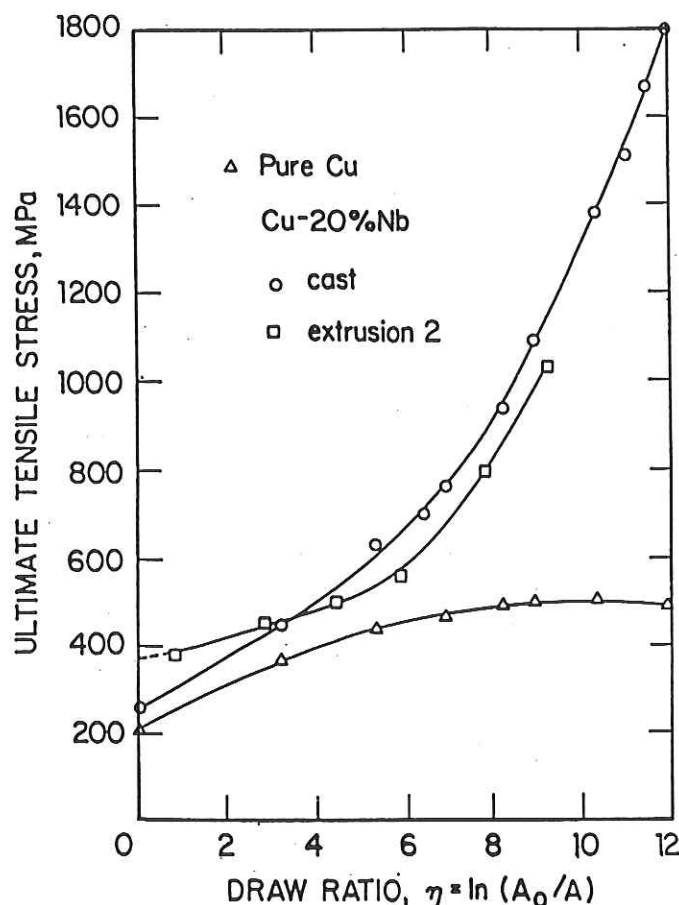


Fig. 7 : Strength of drawn wire : pure copper compared with Cu-20%Nb both cast and extruded

The powders used need not be of the constituent metals. The high pressure gas atomization plant developed at Ames can be used to produce the alloys in ultrafine rapidly solidified particles, of spherical form. The equipment is shown in figure 8 : the melting furnace is at the top of the 4.60 metre tall unit, and the metal stream is met by cold argon or nitrogen gas. Copper-20vol% niobium has now been atomized in the plant and the particles show a very fine distribution of niobium dendrites and spheres within a copper matrix. Compacts formed from them have very good strength properties.

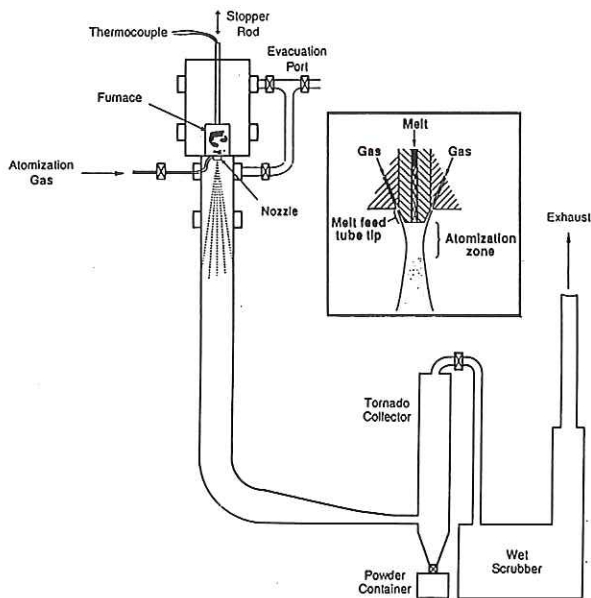


Fig. 8 : Ames high pressure gas atomizer

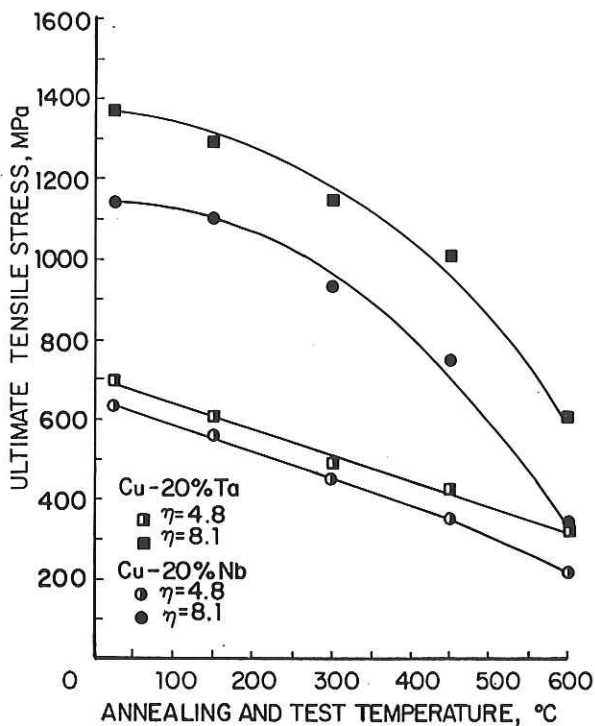


Fig. 9 : Strength of Cu-20%Ta and Cu-20%Nb

Engineers are already realising the useful combination of properties offered by copper-niobium alloys, and if performance above room temperature is of importance, the equivalent alloy of tantalum has merit.

As figure 9 shows, Cu-20%Ta already has at room temperature higher strength than Cu-20%Nb, due to the higher elastic modulus of Ta, and this advantage is maintained up to at least 600 degC if it has been subject to draw ratios of 5 or more.

Finally the chart in figure 10 shows the relationship between electrical conductivity and strength of these alloys compared with competing copper-based alloys.

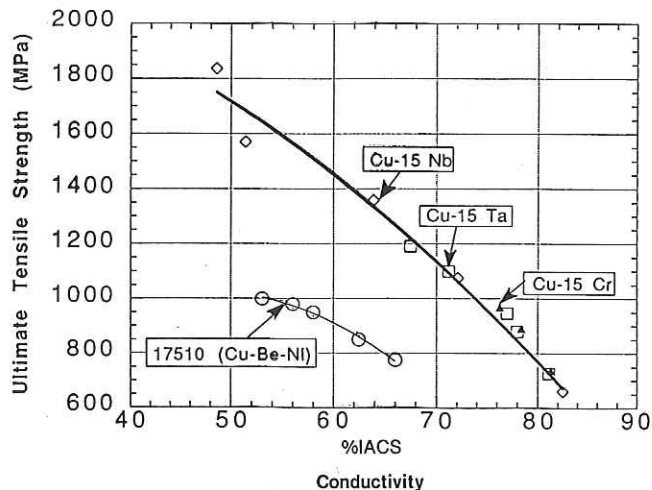


Fig. 10 : Copper-refractory metal microcomposites : strength/conductivity properties

## SUMMARY

Improved techniques have been developed to prepare Cu-Nb and Cu-Ta alloys whose deformation processed strengths plus electrical and thermal conductivities make them attractive composites for many applications.

Alloys of Cu-Nb and Cu-Ta have been prepared by consumable arc casting, while alloys of Cu-Nb have also been prepared by powder metallurgy processing of elemental powders and gas atomized alloy powder particles.

Ingots of Cu-Nb alloys weighing up to 1500 kg have been produced by NRC Inc. using the consumable arc casting technology. As a result of this technology transfer a large supply of this material is now available for commercial application.

