TANTALUM PRODUCERS INTERNATIONAL STUDY CENTER

QUARTERLY BULLETIN FEBRUARY 1976 FIRST QUARTER

CURRENT ACTIVITIES OF THE T.I.C.

The principal current activities of the T.I.C. since the Fourth General Assembly have been :

1. Offering membership in the T.I.C. to processors and consumers of tantalum. The response has been favorable and, at the Fifth General Assembly, the applications received will be submitted for approval to the membership.

2. Offering to companies within the tantalum community who are not yet members, the opportunity to participate in the T.I.C. study of end-use markets and the outlook as it affects the future requirements for tantalum source materials. To date, seven companies have subscribed to the study.

3. Developing future information for the T.I.C. " Bulletin ". Both members and non-members have been very helpful and the entire series for 1976 and early 1977 has been planned.

4. Extending the mailing list for the " Bulletin " to attain greater worldwide coverage of all interested parties.

1975 TANTALUM SHIPMENTS IN THE U.S.A.

As a result of the extensive recession during 1975, shipments by U.S. processors of tantalum reached only 865,000 lb. of contained Ta, 51.5 % of the peak 1974 level. Thus 1975 was the lowest year in shipments since 1968.

The breakdown by type of product is not generally available, but a survey of the capacitor producers indicates that capacitor powder shipments were about 45 % of the 1974 level as a result of extensive inventory reduction throughout the electronics industry. At approximately 420,000 lb. of capacitor powder, that use dropped back to about 5 % over the 1971 level, the last year of recession in electronics. Another large drop of about 115,000 lb. Ta content was in the export of potassium fluotantalate to Japan according to the Japanese Customs import statistics. From this it can be concluded that other uses, primarily tantalum carbide and metal products, dropped only 40 % from the 1974 level.

Consumption of source materials (tantalite, columbite, and tin slags) by U.S. processors would calculate to be 1,150,000 lb. contained Ta_2O_5 in 1975 compared to 2,250,000 lb. in 1974. Since G.S.A. sales of tantalite and columbite (that portion used as a source of tantalum) were also 600,000 lb. Ta_2O_5 less in 1975 than in 1974, the demand for materials from producers and inventories was only 30 % lower. The lack of large previously accumulated source material inventories in 1975 accounts for the market for new materials remaining reasonably firm during the year.

Industry sources forecast a better year for 1976, up about 30 % above the 1975 level. This forecast, if realized, will result in the shipment of products in the U.S. containing about 1,125,000 lb. Ta. The need for source materials in the U.S. alone will be almost 1,500,000 lb. Ta_2O_5. Proportional requirements in Europe and Japan coupled with restrictions on supply, as a result of continuing I.T.C. tin export controls, should result in a small shortage during 1976 which will have to be made up by further inventory reductions by processors.

T.I.C. FIFTH GENERAL ASSEMBLY

On March 25, 1976 at 10 . 00 a.m., the Fifth General Assembly of the T.I.C. will convene at the offices of Compagnie Geomines at Chaussée de la Hulpe 154, Brussels. All members will be represented and interested prospective members have been invited. The Agenda for the meeting will be :

1. **Approval of minutes.**
2. **Financial report for 1975.**
3. **Report of Committees :**
   a. **Executive and Membership**
   b. **T.I.C. Bulletin**
   c. **Technical Advisor**
   d. **Standards.**
4. **Statistics for 1975.**
5. **Discussion of the T.I.C. Program.**
6. **T.I.C. Study-Phase II**
7. **1976 Budget.**
8. **Sixth General Assembly.**
9. **Presentation - " Tantalite, The Exploration Challenge " by**
   — **Prof. Pierre Evrard, Professor at the University of Liège, Chairman of International Resources consultants.**
   — **Mr. W. Hans Schreiber, Behre Dolbear Inc.**

Others wishing to attend should contact Mr. H. Becker-Fluegel, President of T.I.C., care of National Resources Development Corp., 576 Fifth Ave., New York, N. Y. 10036, U.S.A., area code 212, 765-2680.

THAILAND SMELTING AND REFINING CO.

On December 19, 1975 Union Carbide Corporation announced that it had sold its holdings in a joint venture with Billiton N.V. to the Dutch-based Company. The joint venture was comprised of several companies owned by Union Carbide and Billiton and was engaged in mining, smelting and selling tin, primarily in Thailand. Thailand Smelting and Refining Co., Ltd. (a member of T.I.C.) was one of the joint venture companies.

Another of the companies, Thailand Exploration Mining Company, had its mining concessions in the Phang-Nga Bay area revoked by the Thai government in March 1975. Subsequently, Thailand's state-owned Offshore Mining Organization (OMO) agreed in principle to contract with a Thai subsidiary of Billiton to conduct offshore mining in Phang-Nga Bay. OMO will retain ownership of mining leases and any minerals obtained from the operation.

The sale by Union Carbide included also their interest in Billiton Handelsgesellschaft A.G. in Lucerne, Switzerland and Billiton Trading Co., Inc. of New York City. Both of the latter companies are engaged in tin and tantalite marketing, including the sale of the Thaisarco tin slags.

KAWECKI BERYLCO INDUSTRIES NEW ELECTRON BEAM FURNACE

The largest electron beam furnace in the free world for the melting and purification of non-ferrous metals has been placed in operation at the Boyertown, Pennsylvania plant of Kawecki Berylco Industries, Inc. Installed at a cost of approximately $5 million, the new KBI facility will produce ingots up to 16 inches in diameter by approximately 80 inches long. The new Leybold-Heraeus furnace will dramatically increase KBI's capacity to produce ingots of high-quality electronic and metallurgical grades of tantalum, niobium (columbium) and their alloys.

KBI has the world's most modern facility for converting raw materials to pure tantalum and niobium metal and alloys in powder, ingot, bar, rod, wire, strip, foil, tubing, and expanded mesh forms. In addition to the new electron beam furnace, the company recently put into operation an expanded liquid-liquid extraction facility at the Boyertown plant for digesting tantalum and niobium bearing ores. Also, a new vacuum annealing furnace makes possible the processing of wide sheets of KBI refractory metals.

TANTALUM MINING CORPORATION OF CANADA LIMITED (BERNIC LAKE)

Location and Access

The mine site is situated 110 miles by road northeast from Winnipeg in the Lac du Bonnet Mining Division of South-eastern Manitoba. A paved road leads from Winnipeg 25 miles beyond Lac du Bonnet, the last 12 miles to the mine is all-weather gravel road.

Lac du Bonnet, 37 miles from the mine, is the nearest point to which ores or concentrates can be trucked for rail shipment. The Canadian Pacific Railroad branch line connects Lac du Bonnet with the transcontinental lines in Winnipeg. Employees live in Lac du Bonnet and commute daily to the mining operation.

History

In the 1920's gold prospectors discovered a narrow dyke containing some tin values. Exploration of this dyke led to the discovery of the large Bernic Lake sill hidden below it. Some diamond drilling was done and a small shaft sunk at this time. In 1925 the property was acquired by Montgary Explorations Limited, now International Chemalloy Corporation. From 1954 to 1957, Montgary drilled a total of 44 vertical holes totalling 26,000 feet to investigate the lithium-bearing zones. In 1956, a 3-compartment shaft was collared and sunk to a depth of 305 feet.

In 1957, the property was optioned to the American Metal Company Limited (now American Metal Climax). A drilling program consisting of 21 holes totalling 6,693 feet was carried out, but the option was not exercised.

Between 1959 and 1961, International Chemalloy Corporation increased the depth of the shaft to 339 feet, and drove 6,030 feet of drifts in the spodumene, lepidolite and pollucite (cesium) zones. The mine was maintained on a standby basis until the spring of 1962, when all the equipment was hoisted from the workings and the mine was allowed to flood. The property remained dormant during the period from 1962 to October 1966. With the development of the tantalum market the mine was reopened, and during 1967 surface and underground exploration work was carried out. Ore was mined for metallurgical testing, and the Tantalum Mining Corporation of Canada Limited (TANCO) was formed in November 1967 to mine, process, and produce tantalum concentrates. The plant was run-in during the summer of 1969, and went into full production in September 1969. Tanco is a private company owned 50.01 % by International Chemalloy Corporation, 25 % by the Manitoba Development Corporation and 24.99 % by Kawecki Berylco Industries, Inc.

General Geology

The Bernic Lake area of Manitoba is underlain by a Precambrian volcanic-sedimentary sequence known as the Rice Lake group which forms a belt three to four miles wide, trending east-west, flanked on the north and south by granitic intrusive rocks of batholithic dimensions. A tongue of granite outcrops on the west end of Bernic Lake extending 4-1/2 miles westward and attaining a maximum width of three-quarters of a mile.

The pegmatite sill, currently being developed underground, is an almost flat-lying ellipsoidal body within 3000 feet of the nose of the granite tongue. The average dip is 20° north and the sill is arched in the center, plunging at 10° - 30° east and west. The extent of the north-south axis is not fully known but is at least 3,500 feet long. The maximum thickness is 280 feet. Diamond drill holes at 2 and 2-1/2 miles east of the Tanco shaft, on property owned by Tanco, have intersected two pegmatite sills which may either be continuations of the main sill or are other sills located parallel to and en echelon with the Tanco sill.

Wherever the pegmatite has been delineated by drilling, it is enclosed in the east-west trending amphibolite country rock. This country rock dips almost vertically at an angle normal to the pegmatite body. There is evidence of at least one other zoned pegmatite sill underlying the main one. Drill holes in the areas of the West tantalum orebody have intersected this lower sill about 100 feet below the Bernic Lake sill, and all zones, including pollucite, are represented.

The Bernic Lake pegmatite is a complex zoned structure where the outer zones tend to form shell-like concentric envelopes, with layer-like inner zones. Although some nine zones have been described, the main zone, based to some extent on their economic mineral content, can be summarized as follows :

1. The Wall Zones or outermost assemblage, containing albite, microline, perthite, quartz, tourmaline, beryl and coarse muscovite.
2. The Spodumene Zone, containing spodumene-quartz intergrowth ("squi") mixed with feldspars, quartz, and minor amounts of amblygonite and apatite.
3. A quartz-microline-mica assemblage, which contains the tantalum ore-zones, an albite tantaliferous section, minor beryl and spodumene, and a pure quartz core unit.
4. A pollucite zone, containing almost pure pollucite.
5. A lepidolite or lithium muscovite zone, replacing part of the quartz-microcline-mica zone.

Considerable work has been done on the mineralogy of the pegmatite, and numerous reports have been published, several in the Canadian Mineralogist. (e.g. Volume 11, Part 3, 1972.)

Wodgenite, tantalite, and microlite are the most abundant tantalum bearing minerals; pseudo-ixiolite, tapiolite, cassiterite and ilmenite also occur in the tantalum orebodies. The different minerals are all complex oxides of Ta, Sn, Cb, Mn, Fe, Ti, and by far the most abundant tantalum mineral is wodgenite.

Analysis of a number of specimens of wodgenite gave approximately the following average oxide contents :

Ta_2O_5	69.0 %
SnO_2	15.0 %
MnO	9.2 %
TiO_2	2.6 %
FeO	2.4 %
Cb_2O_5	1.8 %

It is practically impossible to distinguish the different minerals from each other, except by x-ray diffraction, and since the specific gravities are all considerably heavier than the host rocks the concentrating plant recovers all of them. Microlite and cassiterite are more abundant in the west tantalum orebody than in the shaft orebody.

Tantalum Reserves

There are two tantalum bearing zones in the pegmatite which are of economic significance at the present time, the Shaft area and West area. Between these two areas (700 feet apart), the tantalite zone is replaced mainly by lepidolite with minor microcline and aplitic albite, also by a mixed zone of all these minerals, however values of tantalite do occur in sections large enough to be mined. This area, between the Shaft and West area reserve blocks, has not been included in any of the reserve calculations but could possibly have an average content of about 1 pound of Ta_2O_5 over quite a considerable area, and will be studied for possible additions to the reserves. The area east of the Shaft area is still open and has potential to the east; a second sill below the main sill has tantalum bearing intersections, (e.g. an intersection 0.19 % Ta_2O_5 over a 12 feet thickness, and it is a parallel sill approximately 100 feet below the main sill).

The shaft orebody extends over 1,200' with an average width and thickness of 365' and 45' respectively. The West orebody covers an area approximately 375' by 315' averaging 42' in thickness.

The tantalum ore reserves, calculated from diamond drill intersections, and after applying a factor to the assay grade, were as follows on January 1, 1976 :

	Tons	% Ta_2O_5
Mineable, Shaft Orebody	463,303	.127
Mineable, West Orebody	194,878	.219
	658,181	.154
Pillars, Shaft Orebody	217,521	.139
Pillars, West Orebody	60,911	.188
Total	**936,613**	**.150**

Mining and Milling

A second article covering the mining and milling operations at Bernic Lake will appear in the Second Quarterly issue of the 1976 " T. I. C. Bulletin ".

ELECTRON BEAM MELTING OF TANTALUM

In order to produce bar, sheet, and other mill products of tantalum, it is first necessary that the tantalum be in a solid piece large enough to forge and roll. Since newly reduced tantalum is a fine powder, consolidation is required. The standard powder metallurgy techniques of pressing into bars can be and are often used. However, the size of the bar which can be made is limited. In addition, powder metallurgy processing does not do as much to improve the composition quality of the tantalum as does electron beam melting and only controlled-purity powder can be used. To obtain larger size starting ingots, melting is the obvious approach, particularly since additional refinement of the tantalum can be obtained at the same time.

Improvement of Tantalum Quality

Sodium reduction, the almost universal method of obtaining tantalum metal from refined salts, results in fine metal powder of varying quality. The powder contains contaminants of various types, the most significant being carbon (10 - 65 p.p.m.), oxygen (500 - 1,500 p.p.m.), nitrogen (10 - 70 p.p.m.), and hydrogen (20 - 100 p.p.m.). All of these elements are detrimental to the fabricability of tantalum. If high enough, they actually result in glass-like brittleness of the tantalum. In addition, there are metallic contaminants carrying over from the raw material and picked up from the equipment (tanks, piping, and retorts) and from process water. These elements are most frequently iron, nickel, chromium, silicon, and calcium. Small amounts of these affect the mechanical properties of the tantalum. Inherent from the sodium reduction are residuals of sodium, potassium, chloride, and fluoride which can not be completely removed by washing. Such interfere with obtaining optimum properties of the metal.

When melting of the tantalum is carried out in high vacuum, not only is further contamination from the oxygen and nitrogen in the air prevented, but contamination in the metal is actually reduced. Any element present which is more volatile than tantalum at the temperature and pressure used in melting will volatilize and be removed. Since tantalum melts at 2996°C., almost all contaminants will volatilize to some extent if the pressure is lower than 10^{-4} atmospheres. To establish this condition along with enough energy to melt the tantalum, electron beam furnaces are used.

The Electron Beam Furnace

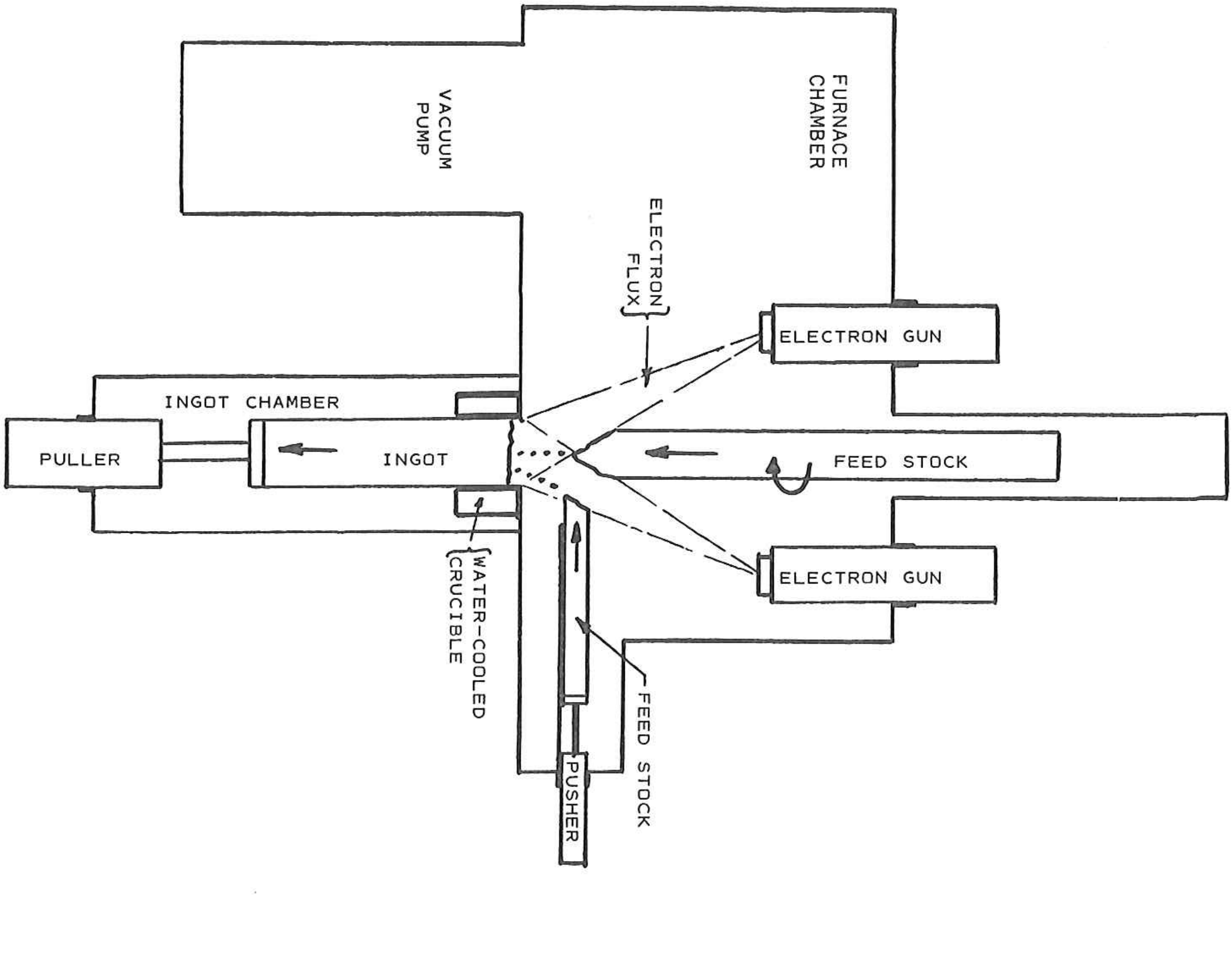
The electron beam furnace, as shown in the sketch, is essentially a large television tube without the scanning device. The furnace is an evacuated chamber with a hot electron emitter, an electrostatic accelerator, a magnetic focusing and steering device, and an anode as a target for the electron beam. The electrons are accelerated to a very high velocity in the vacuum developing tremendous kinetic energy. When they impinge on the anode (the metal being melted), they release their kinetic energy in the form of heat bringing the metal to the fusion point.

In practice, EB furnaces are sized in accordance with their apparent power capacity; however, effective power is determined by electron beam gun design and type of power supply. They range from 30 kw. for small laboratory furnaces to the newest installation in the United States with 1,200 kw. rating. Electron accelerating voltages range from 10,000 to 20,000 volts. The vacuum chambers range from less than one cubic meter to almost twenty-five cubic meters. The vacuum system consists of a series of pumps, oil-diffusion pumps backed up by oil-jet and mechanical pumps.

Tantalum ingots can be melted in a range of sizes, depending on the power applied, up to 250 mm. diameter by 3 m. long weighing 2.5 m.t. Melt rates range upward to 800 kg. per hour for clean feed stock, but are usually considerably lower since the degree of purification or refinement is a time-at-temperature effect requiring controlled melting. Also, the size of the ingot greatly determines the melt rate.

Operations

The tantalum in the form of pressed bars is fed into the electron stream from either the side or the top of the furnace. The continual stream of electrons progressively melts the metal and it drips into a crucible. Since the metal is melted



slowly, practically one molecule at a time, there is sufficient opportunity for the volatile contaminants to vaporize and be carried away by the vacuum system. The dripping metal is collected in a water-cooled copper crucible arranged so that the bottom of the crucible retracts to keep the molten surface at a constant level. Playing the electron beam on the surface keeps it molten to a controllable depth and provides additional time for the vaporization of the undesirable contaminants. As a result, the interstitial elements can be reduced, in general, as follows :

Contaminant	*Initial Level p.p.m.*	*Final Level p.p.m.*
Carbon	10 - 65	10 - 25
Oxygen	500 - 1500	10 - 50
Nitrogen	10 - 70	10 - 30
Hydrogen	20 - 1C0	1 - 5

At the same time metallic contaminants are vaporized and their level greatly reduced. Due to intermetallic bonding between elements and low partial pressures, total refining cannot always be accomplished using a single melt. Thus, it is often necessary to remelt the ingot to further improve the purity. Very dirty starting material may take up to four melts to attain the desired purity. Melting twice, however, is the general practice, the first melt to purify and the second melt to finish purification and to develop good side walls on the ingot.

The finished ingot is retained within the vacuum chamber until it has cooled almost to ambient temperature. Since tantalum readily absorbs oxygen and nitrogen from the air when hot, cooling in the vacuum prevents such and reduces the amount of material which would have to be removed to obtain a contamination free surface.

With properly controlled melting, very good side wall can be produced requiring little conditioning prior to forging into billets or slabs. One of the drawbacks, due to the relatively slow melt rate, is the formation of very large crystals in the ingot. Consequently, extensive cold work with intermediate heat treatments is necessary to break up the crystals and to refine them to the size which will give the desired mechanical properties when used as wire, foil, tubing, sheet, plate and rod.

Some grades of capacitor powder are made from EB melted ingots. Because of the high purity, such powder has higher breakdown voltage than that produced from sodium reduced tantalum directly. The EB melted ingot is converted to powder by heating it in a hydrogen atmosphere. The tantalum hydride which forms is easily broken up and milled to fine powder. After classifying, it is dehydrided by heat-treatment. A few years ago, this type of powder was predominant in the manufacture of capacitors, those used primarily by the aerospace industry. Since it is much more expensive than capacitor powder made directly from sodium-reduced tantalum, it is only used in those devices requiring extreme reliability.

Most of the tantalum processors have electron beam furnaces. They are listed with location and power rating in the table.

Tantalum Alloys

Since most of the metallic elements are vaporized when EB melting tantalum, it is difficult to make tantalum alloys in the EB furnace. One exception is the common tantalum-tungsten alloys which can be made in the EB furnace since the vapor pressure of tungsten is lower than that of tantalum. To make other alloys, however, containing hafnium, titanium, zirconium, etc., it is usually necessary to remelt the EB melted ingot in a consummable-electrode vacuum arc furnace. The alloying elements are welded in strips to the outside of the EB ingot. This ingot is used as the consumable electrode in arc-melting. Sometimes two such arc-melts are required to attain uniform distribution of the alloying elements throughout the tantalum.

Other applications of the EB Furnace

EB furnaces, although developed to melt niobium and tantalum, are used extensively to melt other special metals when high purity is required. Niobium metal particularly is EB melted since the reduced metal contains considerable aluminum carried over from the exothermic reduction of high purity niobium oxide by aluminum. Zirconium electrodes for vacuum-melting are produced from recycle scrap. The scrap can be side-fed into the EB furnace in aluminum boats eliminating the need to weld the scrap into electrode form as would be necessary for the arc-furnace. The aluminum boats volatilize and do not add contamination to the ingot. Titanium scrap could be similarly processed, but the recent development of a non-consumable electrode vacuum furnace has taken precedence in such melting. The EB furnace is also used to produce special iron and nickel base alloys, but a more complicated furnace is used than that needed for tantalum.

ELECTRON BEAM FURNACES USED TO MELT TANTALUM

COMPANY	LOCATION	NO.	CAPACITY
Cabot Chemical Co.	Kokomo, Ind., U.S.A.	1	1000 kw.
Fansteel Inc.	Muskogee, Okla., U.S.A.	2	450 kw.
Gesellschaft für Elektro-Metallurgie mbH.	Nurnberg, W. Germany	1	50 kw.
Kawecki Berylco Industries, Inc.	Boyertown, Pa., U.S.A.	1	1000 kw.
		1	1200 kw.
Heraeus GmbH.	Hanau, W. Germany	1	n.a.
Hoboken Overpelt	Hoboken, Belgium	1	260 kw.
		1	500 kw.
Murex Ltd.	Sussex, Eng., U.K.	1	260 kw.
Wah Chang-Albany	Albany, Ore., U.S.A.	1	350 kw.
		1	450 kw.
		1	600 kw.

TANTALUM SPINNERETS

A small but very important use of tantalum is in the manufacture of synthetic fibers for the textile industry. The demands of the fashion world require a variety of textures, weights and colors of yarns from which the new fabrics are made. Since these fibers are produced by the extrusion of a syrupy organic chemical fluid through a small orifice into an acid spinning bath to set up the solid fiber, the barrier between the two liquids must be resistant to the corrosive influence of both. Few materials are suitable; tantalum being one of the best. This barrier, containing up to hundreds of orifices, is called a spinneret.

Actually the spinneret is a small die ranging in size from 4 cm. to 6.5 cm. It is a flat disc with a collar in the base of which are the extrusion holes. These holes are funnel shaped tapering down to the diameter desired for the fiber, sometimes as small as 4 microns. The holes are made by hand under a microscope by skilled craftsmen. The accuracy of the drilled holes, the sharpness of the edges on the outlet side, and the smoothness of the bore walls are all of major importance in determining the quality of the synthetic fiber.

The tantalum used to produce the spinnerets must be very pure and fine grained. Prime tantalum powder is melted into an ingot in an electron-beam furnace, forged and rolled to reduce the grain structure. The spinneret blanks are cut from the resultant plate and precisely machined to form the disk before the die-holes are drilled. Considering that more than 7-1/2 billion pounds of synthetic fibers were produced in the United States during 1973 and the market keeps growing, this use of tantalum will continue to expand.

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