TANTALUM INTERNATIONAL STUDY CENTER

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Tenth Anniversary

In 1984 the T.I.C. will celebrate the tenth anniversary of its foundation. As a result of the efforts of a small group of people involved with the production of tantalum, the association named the Tantalum Producers International Study Center was formed in 1974, receiving Royal Assent on August 6th 1974 and officially publishing its Charter in the Moniteur Belge on October 24th 1974.

Membership has grown to 70 companies, whose interests include not only production of concentrates and slags but also processing of tantalum materials and uses in the manufacture of capacitors and carbides, and most recently several companies involved with production and processing of niobium have been welcomed. As well as organising two meetings each year for the delegates, the association is gradually expanding its services in the collection of data and the distribution of information. It is a truly international organisation, circulating this Bulletin world-wide, and drawing its members from 16 countries.

A special Cocktail Party and Buffet Supper will be held on October 29th in honour of the tenth anniversary, and it is hoped that all the member companies will be able to send delegates to celebrate the first ten successful years.

TWENTY-FIRST GENERAL ASSEMBLY

The Twenty-first General Assembly was held in Stockholm on Tuesday June 5th 1984, when the members decided to change the name of the T.I.C. to "Tantalum International Study Center", to reflect the wider membership interests of the expanding organisation. Six new companies were elected to membership, and the assembly agreed to reduce the period of notice required by applicants from three to two months before the relevant General Assembly.

A programme of presentations on the topic of tantalum in hard materials followed the General Assembly on June 5th, and the business of the day closed with a panel discussion. Papers presented were :

— The effect of tantalum on the microstructure and properties of cemented carbide, by Mr B. Aronsson, Sandvik Hard Materials.
— Production and properties of tantalum carbide and mixed carbides, influence of scrap reclamation, by Mr P. Borchers, Hermann C. Starck Berlin.
— The future of virgin tantalum carbide in steel grade cutting tools, by Mr W. Hantel, Metallurg.
— The role of tantalum in carbides for metal cutting, by Mr K. Nordlund, Seco Tools.
— Exploration for tantalum in Sweden, by Mr D. Bjurstroem, A. Johnson Ore and Metals.

The first three of these are printed in this issue of the Bulletin.

On June 6th participants had a choice of plant visits. One group toured the facility of Sandvik Hard Materials at Västberga, lunching afterwards as guests of Sandvik. The second group visited the factory of Seco Tools at Fagersta, were guests of the company for lunch in the town nearby, and paused on the return journey to see the old ironworks at Engelsberg — a great contrast to the modern installation of the host company.

The T.I.C. welcomed participants with a Cocktail Party on June 4th. On the following evening the four Swedish host companies — Sandvik Hard Materials, Seco Tools, A. Johnson Ore and Metals and Ekman and Co. — invited all the conference delegates and their ladies to a banquet on board the S.S. Gustafsberg VII while the boat sailed calmly through the Stockholm archipelago in the late sunset of the Swedish midsummer. The guests disembarked for a while at Boo Brygga for a demonstration of folk-dancing in which they afterwards joined.

Ladies' Tours were arranged for both Tuesday and Wednesday, with visits to Millesgarden, the Royal Palace, the "Old Town", Mariefred and Gripsholm Castle, giving the ladies a special opportunity to appreciate the warm sunny weather.

TWENTY-SECOND GENERAL ASSEMBLY

The Twenty-second General Assembly of the T.I.C. will be held at 9 a.m. on Tuesday October 30th 1984 in the International Association Centre at 40 rue Washington, 1050 Brussels.

AGENDA

1. Voting proxies.
2. Presidential address.
3. Minutes of the Twenty-first General Assembly (held in Stockholm on June 5th 1984).
4. Applications for membership.
5. Financial matters.
6. Report of the Executive Committee.
7. Statistics :
   production
   processing
   capacitors.
8. Statutory elections.
9. Twenty-third General Assembly.
10. Other business.

The formal business of the association will be followed by the presentation of papers by speakers including Professor Jean Vereecken of Brussels University, and Dr Harry Stuart of Niobium Products Co. Ltd.

On the evening of October 29th there will be a Cocktail Party and Buffet Supper. All delegates are invited to celebrate the tenth anniversary of the foundation of the T.I.C.

Presidential address

The following is taken from Mr R.W. Franklin's address to the General Assembly in Stockholm.

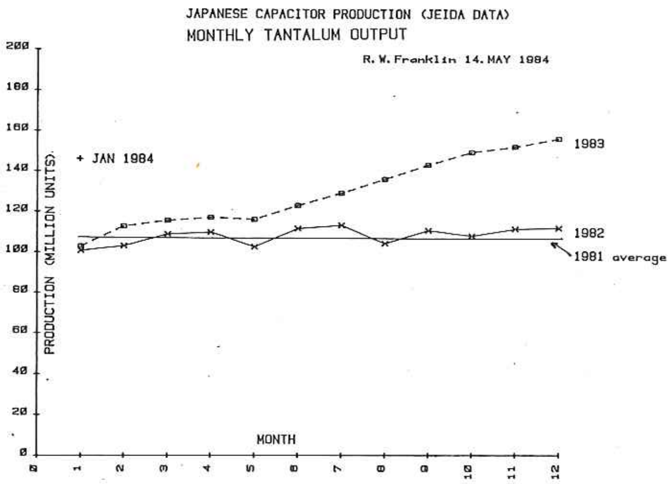
Welcome to the Twenty-first General Assembly of the T.I.C. It is especially pleasing to be able to welcome those member companies which have just joined the T.I.C. and also our various guests and observers.

It is fitting that we should be holding this meeting in Sweden as it was here that tantalum was first discovered. Sweden has made many contributions to the development of Europe — in the days of the Vikings in particular — and also more recently to the development of world trade. In the case of tantalum and niobium this is particularly true in the carbide cutting tool industry, which is the reasoning behind the theme of this General Assembly.

At this point it is appropriate to thank the four host companies, AB Sandvik Hard Materials, Seco Tools AB, Ekman and Co. AB, and Axel Johnson Ore and Metals AB, for organising arrangements in Stockholm, including the social events and the plant tours. The T.I.C. is most grateful for their contributions to all aspects of this General Assembly and we express our warmest thanks to them.

When I said that tantalum was discovered here, that is not the whole story. You will be hearing in the first of today's papers how the discovery of tantalum was interlinked with that of niobium and how the work which sorted out the chemical aspects left us with the problem that still exists today, namely that element 41 has two names — either columbium or niobium. When I chose to follow the decision of the International Union of Pure & Applied Chemistry in 1950 to call it niobium, this is a personal choice and does not reflect any preference of the T.I.C. The destinies of the two elements tantalum and columbium/niobium have been intermingled from the very beginning and so it is appropriate that the T.I.C. should be extending its services to cover both materials.

Returning now to the state of the industry, there is no doubt at all that the manufacturing of end products containing tantalum has increased significantly during the past year. For example, the graph below shows how the output of tantalum capacitors in Japan has increased. The demand for scrap tantalum for re-cycling has also been markedly higher. However, these improvements are not yet sufficient to get hard rock mining back to where it must be for long term stability of the industry as a whole. Some of this disparity is due to out-of-phase supply and demand time cycles. Another part is due to reduced demand per item produced due to technological advances.



The position in the niobium industry is somewhat different, both from the point of view of sources of raw materials and the nature of the end users. I hope that we will be able to persuade one of our new members to give us a detailed analysis of the niobium industry on a future occasion.

An important function of the T.I.C. is to publish statistics and we now have obtained permission to publish tantalum capacitor statistics from both the U.S.A. and Japan. At the same time we are attempting to provide a more detailed split of the tantalum production and processing data which have been published over the years. Bare statistics on their own can only be taken as one index of trends. They never tell the whole story. Therefore it is necessary every now and then to crawl inside the data and establish the meaning of the movements. Life is certainly not a steady-state condition. Taking for instance the graph to which I referred earlier, the increase in shipments of capacitors cannot be related directly to demand for tantalum. Over the years the ratio of tantalum used to capacitors produced tends downwards. The change for powder is greater than that for wire. (Another way of saying that is that the ratio of wire weight to powder weight increases over the years.) Also, the increase in production of tantalum capacitors is less than that for other types of capacitors, so it looks as if substitution is an important factor. The deeper analysis of trends and their causes must be an essential part of the T.I.C.'s activities. We cannot rest content with the publication of totals. That is not to say that the data already being published are of little use. Quite the contrary! However, they are essentially a first order indicator of the movement of materials. We must be able to relate the different numbers together. For instance, what is the conversion rate from capacitors to tantalum raw materials? We know that it is quite different for the U.S.A., for Japan and for Europe and we have past estimates in the surveys carried out for the T.I.C. by Ayers, Whitmore. This ratio needs regular updating.

New alloys containing tantalum and niobium are being developed : we need to know how these are progressing. The carbide cutting tool technology is continuously advancing and we need a guide to these trends, some of which will be described during today's sessions. There is a lot more work to do in this area but it has to be done step by step and in a manner which does not compromise the commercial confidentiality of the reporting organisations. One area of statistical data which we do not attempt to cover is price movements. This is because the T.I.C. is not involved in any activity designed to control the market forces which determine prices. We take great care that we do not infringe antitrust regulations anywhere throughout the world. It could be claimed that we are over-reacting to that risk because the information is published elsewhere. However, it is better for us to concentrate our limited resources on matters where there is maximum interest and limited availability of data from other sources.

Whilst it is necessary for us to consider total supply and demand movements, we must not ignore the smaller applications. Some of these are very small in relation to the total demand levels but they still can add to our knowledge of the behaviour of the elements and their compounds. For instance the silicides of tantalum and niobium are being actively investigated as interconnections in some integrated circuits because of their high conductivity. Almost as conductive as the metals themselves, the silicides are stable in contact with the other materials of the integrated circuit. The sulphides and selenides of the two elements also have unusual properties and research is being carried out in connection with charge density waves. The interest here comes from their two-dimensional crystal structure : investigators are checking whether this behaviour pattern can be utilised to develop a new electronic component. Niobium, and to a lesser extent tantalum, is of value for superconductors, materials which at very low temperatures conduct electricity without power loss. Lithium niobate has a good future in surface acoustic wave and surface skimming bulk wave devices and in integrated optics.

All these investigations may reveal possible new volume usages, but also they create a deeper understanding of the chemical and physical properties of the elements and their compounds. The more lines of study that are proceeding, the greater is the chance of an interaction of properties which would lead to a real winner, either as a product or as an improvement in the processing of the raw materials. I hope that we will be giving some space to reports of these investigations in future Bulletins.

Turning to the papers to be presented at this meeting, we are, naturally, concentrating on the cemented carbide industry because of the important place held by Sweden in the manufacture of this product : the cemented carbide industry is the second largest user of tantalum and is an important user of niobium.

R.W. FRANKLIN
President

T.I.C. tantalum production and shipments

The T.I.C. data for the production and shipment of tantalum-bearing tin-slags and concentrates were released to the members at the Twenty-first General Assembly. They follow, including the total production and shipments for 1979 through 1982 :

(figures given in lb. Ta_2O_5 contained)

	Slags	*Concentrates*	*Total*
1979			
Production	1,204,945	893,157	2,098,102
Shipments	1,182,163	938,723	2,120,886
1980			
Production	1,383,704	792,528	2,176,232
Shipments	1,589,729	726,480	2,316,209
1981			
Production	1,228,246	926,241	2,154,487
Shipments	1,020,598	738,628	1,759,226

	Slags	Concentrates	Total
1982			
Production	1,210,140	685,845	1,895,985
Shipments	857,802	442,184	1,399,986
1983			
Production			
1st quarter	304,840	87,228	392,068
2nd quarter	303,398	51,497	354,895
3rd quarter	395,454	83,154	478,608
4th quarter	276,576	146,956	423,532
Total	1,280,268	368,835	1,649,103
Shipments			
1st quarter	125,003	75,684	200,687
2nd quarter	7,100	43,844	50,944
3rd quarter	154,688	61,899	216,587
4th quarter	360,837	133,042	493,879
Total	647,628	314,469	962,097

In the first quarter of 1983, 25 out of 29 companies reported; in the second quarter, 22 out of 29; in the third quarter, 24 out of 27; and in the fourth quarter, 26 out of 27. Failure of two or three companies to report probably results in only a small error as the total reporting will not be made by the data collection agency unless at least two-thirds of the producers have reported, including the six major producers.

The total production in 1983 shows a further decrease in production of about 13 % and an even greater reduction in shipments of about 30 %. Thus, once again producers have built their inventories by 687,000 lb. contained Ta_2O_5. Added to the apparent increase in producers' inventories during 1981 and 1982, these inventories are now almost 1,600,000 lb. Ta_2O_5.

70 % of the producers' inventories are in tin slag, about 1,100,000 lb. The declining market for tin slags is evident when it is considered that the production of tin slags has been essentially constant from 1981 through 1983 at an average of about 1,250,000 lb. per year. Shipments of slag equalled 93 % of production in 1981, 79 % in 1982 but only 51 % in 1983. It is probable that most of this slag inventory is of the low-grade type, less than 3.5 %. These low-grade slags are not economically usable at the price of concentrates in today's market.

Although the shipments of concentrates continued to decline in 1983, a higher percentage of the current production was shipped increasing from 80 % in 1981 and 64 % in 1982 to 85 % in 1983. Once again it is evident that, when the supply of source material is larger than the processors' demand for additional material, the processors prefer the higher tantalum-containing concentrates. With a breakdown by grade available for the first time in 1984, the preference of processors is confirmed on the basis of the data now available for the first quarter :

	Production	Shipments
Tin slag	226,705	46,452
Tantalite under 25 % Ta_2O_5	13,319	8,267
Tantalite between 25 % and 60 % Ta_2O_5	56,312	66,075
Tantalite over 60 % Ta_2O_5	9,471	31,244
Other materials	9,259	Nil
Total	315,066	152,038

(Data have been received from 19 of the 26 reporting companies.)

Process shipments are again available from data received from the processing members. 16 out of 16 companies reported in 1981 and the first three quarters of 1982, 16 out of 17 in the fourth quarter of 1982. In 1983 19 companies were asked to report : in the first and second quarters 16 did so, in the third quarter 18 and in the fourth quarter 19 reported. In the first quarter of 1984, 19 out of 19 companies responded.

(figures given in lb. Ta contained)

1981	1,755,139
1982	1,463,850
1983	
1st quarter	326,363
2nd quarter	440,074
3rd quarter	529,280
4th quarter	612,817
Total	1,908,534

Thus, 1983 shows a total greater than either of the two previous years and a very significant increase of 30 % over 1982, about double the increase forecast by industry members a year ago. The gain in each quarter of 1983 portrays a steadily improving market sustained in the first quarter of 1984 :

1984 - 1st quarter :

Tantalum oxide	27,471
Alloy additive	29,130
Carbides	124,411
Powder	257,066
Ingot, unworked metal, scrap, other	88,812
Mill products	82,160
Total	609,050

Conversion of the product shipments data for each year into equivalent pounds of Ta_2O_5, including an allowance for unrecoverable losses during processing, can be used to demonstrate the reduction in processors' inventories, these occur even if it is assumed that 10 % of the source material (considered high on the average) was purchased scrap.

	Equiv. Ta_2O_5 incl. losses	10 % Purch. Scrap	Use of Slag & Concentrates
1981	2,370,000	240,000	2,130,000
1982	1,560,000	160,000	1,400,000
1983	2,580,000	260,000	2,320,000

Comparing to the shipment of source materials by producers for the three year period indicates that processors have reduced their inventories by about 1,700,000 lb. contained Ta_2O_5. Thus, the combined inventories of both producers and processors has shown a three-year reduction of only about 100,000 lb. This, again, follows the historic cycle of inventories shifting into the hands of the processors in periods of under-supply and back into the hands of producers in periods of over-supply.

1983 should be noted separately, however, as it may indicate a trend for the next few years. The total demand for virgin source materials by processors was about 2,300,000 lb. Ta_2O_5 whether obtained from their own inventories or from producers. The production of new source materials was, however, only 1,650,000 lb. Thus, the net reduction of inventories in 1983 alone was about 650,000 lb, about 25 % of the need for the year.

If the processors' shipment levels of the fourth quarter of 1983 and the first quarter of 1984 are sustained throughout 1984, the consumption of virgin source materials could reach almost 3,000,000 lb. Ta_2O_5 with an allowance for 10 % scrap use. Adding to this amount the 240,000 lb. of concentrates to be delivered to the G.S.A. for the U.S. National Stockpile during 1984, the total need could be as high as 3 1/4 million lb. There could then be a very large reduction in inventories in 1984, as much as 1 1/2 million lb. since it seems unlikely that the processors' demand for new shipments from the producers would be any larger than in 1983 in view of the remaining high level of processors' inventories. Even if the processors should choose to buy more material during 1984, it seems evident that the producers could supply as much as 3 1/4 million lb. contained Ta_2O_5 without additional production by drawing down their accumulated inventories.

It seems likely, therefore, that the tantalum raw material market will remain stable through the balance of 1984 and, probably, well into 1985 before it will be necessary for the producers to increase their production.

Japanese Tantalum Capacitor Sales

(000's units)

(Data from Japanese Electronic Industry Development Association)

	Production	Of which-export
4th quarter 1983	457,249	107,870
1st quarter 1984	479,731	105,431

The effect of tantalum on the microstructure and properties of cemented carbide

The following article has been extracted from a paper presented by Mr B. Aronsson, Sandvik Hard Materials, to the Twenty-first General Assembly of the T.I.C. in Stockholm on June 5, 1984.

It may seem easy to give an answer to the question inherent in the title. The properties of cemented carbide with varying tantalum contents have been determined in a large number of investigations published over the last five decades. To present these in a systematic way and draw some conclusions from them should be rather easy. If I claim that this is not the case, it is not only in order to justify continued costly R&D activities (for a small part of which I am responsible), but because the effects of a particular element on the properties of a material really are a more complicated problem than one would think. The reason is that a given amount of an element will have different effects depending on the concentration of other elements, on variations of processing parameters and on the particular properties which are relevant in view of the applications.

In an attempt to simplify, I shall begin by presenting some facts about tantalum carbide and hard materials based on this compound. Subsequently, information on how tantalum additions affect the composition of phases, microstructure and more fundamental properties of cemented carbide will be given.

COMPOSITION AND PROPERTIES OF TANTALUM CARBIDE

Tantalum carbide has a variable composition corresponding to formulae in the range of $TaC_{0.7}$ to TaC. In addition to variable carbon contents, a fraction of the carbon can be replaced by oxygen and nitrogen.

As to mechanical properties, the hardness has been extensively studied. The hardness at temperatures below 700 °C increases as the carbon content decreases from the composition TaC to $TaC_{0.8}$. The higher hardness of the carbon deficient tantalum carbide is associated with the ordering of occupied and vacant carbon sites and this, in turn, could be influenced by chemical history.

The hardness of TaC is considerably lower than that of the related carbides (TiC, ZrC, HfC, NbC) within a broad temperature range up to 700 °C. The same is true for the mixed carbides of the type (Me, W)C with Me = Ti, Zr, Hf, Nb in which tungsten substitutes for part of the Me. That is, the (Ta, W)C mixed carbide has a lower hardness. Other observations of the mechanical properties of MeC and (Me, W)C carbides show that those containing tantalum tend to have a lower strength and a higher ductility.

As a final comment, the (Ti, Ta, Nb, W)C carbide present in cemented carbide can be more or less homogeneous, depending on variations of the processing parameters and this could somewhat influence the effects on Me properties of cemented carbide.

TaC-RICH HARD MATERIALS

By mixing tantalum carbide with a ductile metal, such as nickel or cobalt, useful materials, very similar to conventional cemented carbide, can be obtained. There exists a two-phase TaC-binder metal region, at least in the Ta-C-Ni system, which makes the preparation of dense materials possible. Reasonably good properties have been obtained for TaC-Ni and TaC-TaN-Ni alloys. Some tests on TaC-Co materials have also been reported but these seem less promising than those with nickel.

It seems, however, very unlikely that TaC-rich hard materials should have such combinations of price and properties that they will become commercially interesting.

PHASES AND MICROSTRUCTURE OF CEMENTED CARBIDE WITH DIFFERENT TANTALUM CONTENTS

Cemented carbide generally consists of a mixture of three phases. Each of these has the same composition and properties wherever it is found in the material and it is separated from the other phases by sharp boundaries, usually with a thickness of only a couple of atomic distances. The phases are the hexagonal tungsten carbide WC (often called the alpha phase) which in all common grades has a composition closely corresponding to this formula, the cobalt binder phase (beta phase) and a complex cubic carbide phase (Ti, Ta, Nb, $W)C_x$ (gamma phase) in which x is generally close to but always somewhat less than one. The effect of the tantalum on these three phases is easy to describe since virtually all tantalum is dissolved in the cubic phase. Knowing the total contents of tantalum, niobium and titanium in a cemented carbide grade, the composition of the cubic carbide, as well as the amount of it, can be calculated using the phase diagram.

Thus the first effect observed when increasing the amount of tantalum is that the cubic carbide will contain more tantalum, and some of the changes in properties may, in a straight forward way, depend on this. However, only by considering an isothermal section of the TiC-TaC-WC phase diagram, care is needed in jumping to quick conclusions. If, for instance, titanium is replaced by an equal (atomic) amount of tantalum, not only does the tantalum content of the cubic phase increase, but, at the same time, it also becomes more tungsten-rich and, in addition, the ratio of cubic carbide to hexagonal tungsten carbide (gamma/alpha ratio) increases. Hence, the extent to which a change in properties depends on the higher contents in the cubic carbide or the higher gamma/alpha ratio is not known.

The cubic carbide contains considerable amounts of oxygen and also nitrogen. These amounts probably decrease as tantalum replaces titanium and this may also contribute to changes in properties. As yet, experimental results on the composition of the cubic carbide phase, in particular the contents of carbon, oxygen and nitrogen, lack precision. But, with the recent application of atom probe techniques in the studies of cemented carbides, the situation will soon be improved permitting more precise conclusions to be drawn.

Whereas the influence of tantalum on the cubic carbide phase is somewhat complicated, its effect on the composition of the other phases is very simple. In contrast to tungsten, tantalum does not dissolve to a detectable extent in the cobalt binder phase nor does it substitute for tungsten in the tungsten carbide. This has recently been corroborated with atom probe analysis.

What is the effect of tantalum on the microstructure of cemented carbide ? What influence does it have on the grain size and its distribution, the degree of contiguity of the carbide skeleton, etc. ? One effect is well established : that is the tendency of tantalum to inhibit grain growth. Because of this, small amounts of tantalum are often added to straight WC-Co grades. In this respect, tantalum is found to be more efficient than other carbides except those of other transition metals in the fifth group, e.g. vanadium and niobium. It has been claimed that the effects of tantalum are due to interface phenomena.

In the context of cemented carbide it is important that the two carbide phases can exist together with a molten binder phase (without other phases being formed) within a sufficiently broad region of temperature and carbon activity to permit large scale production. In this respect, tantalum behaves very much like niobium and there are no practical limitations regarding tantalum contents due to this.

It can be added that tantalum (mainly as carbide but also as nitride) is a constituent in other hard materials than just conventional cemented carbide. Those based on TiC-Ni should be particularly mentioned. As yet, only few results of the composition of phases and a microstructure of these materials (often called Cermets in American and Japanese literature) have been published. In general, the effects of tantalum would be analogous to those in cemented carbide with a cobalt binder.

THE PROPERTIES OF TANTALUM-CONTAINING CEMENTED CARBIDE

Tantalum can, of course, be used as an addition in order to achieve a desirable ratio between WC and cubic carbide and part of the results can be rationalized by considering this effect. However, a suitable ratio of the two carbide phases can as well be obtained by judicious additions of titanium or niobium. The reason why the more expensive element tantalum is chosen must be because it has some particularly beneficial effects. It appears that high tantalum content is associated with an improved toughness behaviour. This would be compatible with the relatively high toughness of TaC and (Ta, W) C.

The results of more fundamental mechanical properties, such as fracture toughness and high temperature strength, are too fragmentary to permit any far reaching conclusions. The best documented proof of the superior toughness behaviour comes from technological tests, e.g. in toughness-demanding metal cutting operations such as milling.

The good oxidation resistance of tantalum carbide may also be a contributory factor to the use of tantalum containing cemented carbide in demanding cutting operations.

THE SUBSTITUTION OF OTHER ELEMENTS FOR TANTALUM

When the metal cutting cemented carbide grades were introduced in the 1930's, tantalum was abundantly used as addition in order to obtain an appropriate amount of the cubic carbide phase. This was particularly so in the United States, whereas higher contents of titanium were used in Europe. This difference has decreased lately. With the rocketing tantalum prices a couple of years ago, there have been many activities on both sides of the Atlantic to reduce to a minimum the contents of tantalum. One would expect this to eliminate the earlier difference between American and European grades. As with other raw materials, those used in cemented carbide have also been readily available as a result of the recent recession with a concurrent reduction in prices. The rapid increase in the use of coated grades as well as more efficient recycling has reduced the demand on tantalum and made tantalum substitution a less urgent business.

A number of papers and patents have presented results on effects of replacements of tantalum carbide, partially or wholly, by other carbides. Hafnium and niobium carbides have been particularly studied. At least partial substitution is possible without significant deterioration in properties and, in some cases, minor improvements have also been reported. However, if we accept the very tentative conclusions concerning tantalum addition that its beneficial effect would be associated with the relatively low hardness and high ductility of TaC and (Ta, W) C, then a complete replacement of tantalum by elements that form harder carbides or mixed carbides does not seem possible without some sacrifice in properties.

So, unless tantalum becomes very scarce and expensive, it will remain an important element in cemented carbide. And, with the very satisfactory supply situation and the stable and reasonable prices, it will do so for a long time to come.

Production and properties of tantalum carbide and mixed carbides, influence of scrap reclamation

The following article has been taken from a presentation made by Mr P. Borchers of Hermann C. Starck Berlin at the T.I.C. Twenty-first General Assembly in Stockholm.

The standard high quality of premium grades of cemented carbides, among them many Swedish products, is known worldwide and their good name is linked with the extensive use of TaC. There are some new developments in the technology of cemented carbides which will influence the consumption of TaC and which make the subject of this conference very timely :

— The growing use of coated cutting grades with the resultant increase in durability as well as lower concentrations of TaC in the substrate will reduce its consumption,

— Improved scrap reclamation techniques resulting in the repeated use of valuable carbides, and,

— Strong efforts to substitute for expensive carbides.

The last development is a consequence of the price explosion of tantalum products in the late seventies which influenced the consumption of virgin TaC. Fortunately, the prices have stabilised in the last two years and, based on realistic consumption figures, a very favourable picture for the long term supply situation can now be seen.

This presents a challenge to encourage the carbide and tantalum industry to develop new grades and applications for the future, making use of the outstanding properties of TaC.

APPLICATION

There are two quite different reasons for using TaC.

— Small amounts are added to straight WC-Co grades in the 1.5 micron and below range as an effective grain growth inhibitor.

— The substantial quality improvement in cutting tools mainly in the area of long-chipping steel-cutting grades.

TaC forms mixed crystals with NbC, TiC and WC which are effective in improving the properties of cemented carbides. Higher contents of TaC in these mixed crystals increase remarkably the thermal shock resistance, hot hardness and resistance to cratering and oxidation.

In earlier times the incorporation of the single carbides was achieved by adding them separately to the mixture of WC with the binder metal. Later, TiC was introduced in the form of a solid solution with WC adding TaC or TaNbC separately to the mixture. This method of application is still preferred worldwide.

The method of prealloying the perfect solid solution (W, Ti, Ta, Nb)C, called « triple » without NbC or « tetra » with NbC, in ratios required for a special cemented carbide grade, is increasing and only limited by the boundaries of solubility and by economic considerations. The use of prealloyed mixed crystals in cemented carbides yields several advantages. The mixed crystals are harder and also tougher compared to single and unalloyed carbides. The contents of oxygen, nitrogen and free carbon are distinctly lowered by purification during the diffusion process, whereby the wettability for cobalt and other binder metals is increased. The temperatures for the preparation of mixed crystals are about 500° higher than the sintering temperatures which are normally applied to hard metal production, which is an advantage for the exact formation of mixed crystals.

Quality requirements for all the different grades of pure single carbides, as well as mixed carbides, are the same and generally very strict. Normal requirements are high chemical purity, high combined and low free carbon, low oxygen and nitrogen contents with the correct grain size and grain size distribution. Additionally, all mixed crystals must be homogeneous and in complete solid solution.

Fine grain size and the requirements for solid solutions are often affected adversely by the necessary treatment conditions. Special fine powders in the one micron range are requested increasingly.

PRODUCTION

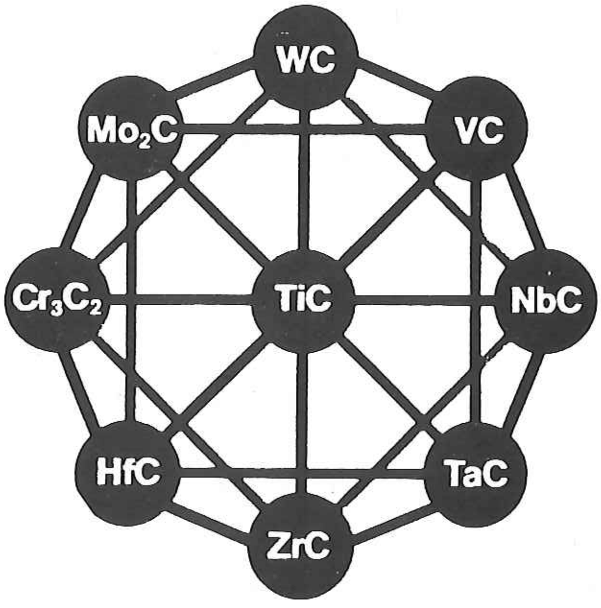
Production methods have to follow the above mentioned quality requirements.

When Balke of Fansteel first used TaC in the Ramet cemented carbides in 1930, it was made from tantalum powder by solid state reaction with carbon. The first TaC from Starck was produced by carbon reduction of tantalum oxide made by alkali-fusion of K_2TaF_7. It was the purest Ta-compound available at that time. The furnace was a gas fired kiln with graphite crucibles normally used for tungsten carburization.

With the introduction of the Solvent Extraction process for the separation and purification of tantalum and niobium, sufficient pure oxides of constant quality can be extracted from any kind of raw materials such as natural concentrates, high grade slags, artificial concentrates from low grade tin slags or from the products of chemical scrap treatment. It has little bearing on the cost if tantalum and niobium are first separated totally or are co-precipitated from a combined strip solution to produce a mixed oxide. However, the oxide purity is very important because it determines the amount of work which has to be done during carburization. Some of the impurities in the oxide can be eliminated during high temperature treatment in vacuum. Others, like calcium oxide, aluminium and silicon remain in the carbide. Normally, a two-step carburization at 1600° to 2000° C, with a carbon and oxygen adjustment between the two steps, and a final vacuum treatment will result in the desired quality.

The methane carburization of tantalum chloride in hydrogen in a plasma, or other suitable heat source, is still a very attractive but expensive method for the production of submicron ultrafine carbides. To remove residual chlorides, an additional heat treatment is required. The pure chlorides represent a valuable source for chemical vapour deposition of metal or carbide.

Well formed TaC crystals with nearly theoretical carbon content and low free carbon and gas content are produced by the menstruum process in an auxiliary metal bath. The nice golden colour of the menstruum carbide should by no means raise any associations with the price of this precious metal. High prices for tantalum carbide would again create a demand for substitution.



From all of the cubic carbides shown on the right side of the diagram, TiC acts as the host lattice for the mixed carbide phase and is, therefore, added more or less regularly together with TaC.

Niobium carbide has similar properties to tantalum carbide and is soluble with tantalum carbide in all ratios. It is, therefore, the natural and most commonly used carbide in combination with TaC. Regarding its valuable properties in cemented carbide, it can be ranked between TaC and TiC. There are different opinions about how far

NbC can replace TaC in the different grades without reducing their performance. U.S. producers still prefer the pure or tantalum-rich grades; in Europe, for a long time, 90:10, 80:20 and 60:40 TaC:NbC are used. Japanese producers fall between the two extremes.

Some authors claim a replacement of 30 % of the TaC with the NbC to be the maximum limit without affecting the strength. An overall value of 20 to 30 percent seems to be correct which can be seen from some typical HCST figures :

	1979	1980	1981	1982	1983
% NbC in TaNbC :					
— In new deliveries	22 %	29 %	34 %	33 %	33 %
— In Treated Cemented Carbide Scrap	18 %	18 %	21 %	14 %	18 %

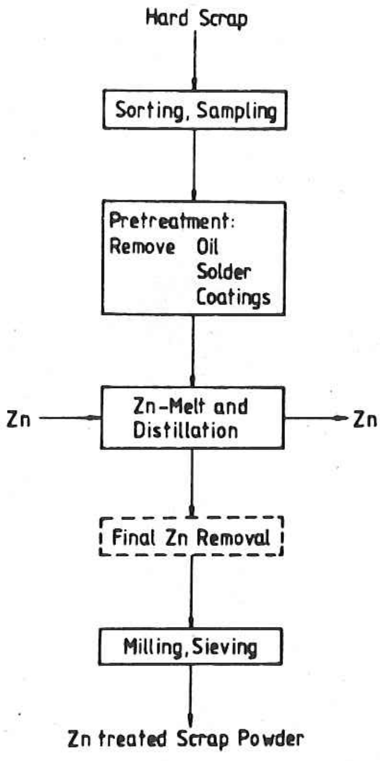
It was proven many years ago that NbC alone will not work.

Mixed crystals of HfC + NbC were proposed years ago to replace (TaNb)C. Recently, mixed crystals of ZrC + HfC and ZrC + NbC in solid solution with TiC + WC were tested with encouraging results. But it seems that so far no significant advantage over the existing systems can be found which would justify their introduction in industry. In the case of HfC, the uncertain supply and price situation may be another reason for the limited acceptance of this new system. From the standpoint of scrap treatment, the incorporation of too many different carbides into the mixed carbide phase can result in difficulties in re-using carbide scrap.

RECLAMATION PROCESSES FOR SCRAP

Reclamation processes for cemented carbide scrap have a high degree of influence on the consumption of virgin TaC and mixed crystals.

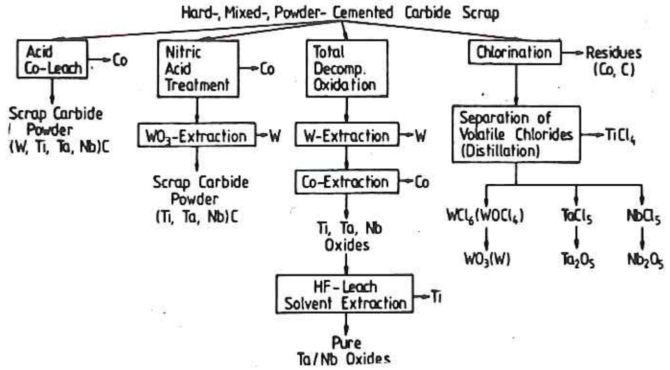
The zinc-process is especially suitable for treating scrap from cutting grades and other small size carbide scrap. The zinc-treatment attacks only the binder phase without changing the composition of the carbide phases.



In the temperature range used in the zinc-process, the composition of mixed carbides, (W, Ti, Ta, Nb)C, is conserved and the treated carbide can be re-used in a new batch of cemented carbides which requires the same or similar composition. This is normally done by mixing the recovered carbides with virgin material. Since the zinc-process is a disintegration process and not a purification process, impurities picked up during or after usage of the tools remain in the treated scrap thus limiting its use. Careful sorting with pretreatment to remove oil, solder and refractory coatings is essential for satisfactory reclamation.

Computerised test work to find out the right proportion of scrap to virgin material can optimize reclamation but cannot prevent completely the danger of introducing impurities into high quality products.

The Chemical Processes are suitable for all kinds of scrap with different contents of W, Co, Ti, Ta, Nb, Ni, Fe, Cu and impurities from their usage.



Hard and mixed scrap of all sizes, dust and sludges can be treated by selected chemical production steps. In the simple cobalt-leach process, which has only limited applications, the original composition of all the carbide phases is preserved. In the nitric acid treatment, this is the case for the mixed carbide phase. The latter interesting method could not be introduced up to now in industry due to environmental considerations. Only in the total-decomposition process are all of the valuable contents of the scrap recovered, separated and purified resulting in virgin products like APT, tungsten metal and carbide and cobalt metal powder. Titanium, tantalum and niobium are collected in an oxide sludge which can be used as a feed material for a solvent extraction plant. Finally, tantalum and niobium are recovered in the form of pure oxides or carbides. Recovery rates have been increased remarkably in the last few years by improved technologies. This chemical route has a very broad range of applications. The higher costs of this process are at least partly compensated by the high quality of the recycled products and the flexibility of their application.

The chlorination-process appeared at one time to be very promising but has not been fully accepted by the industry. The tungsten recovery and separation from the other elements is much more complicated and costly than in the other established processes.

Both main lines of scrap reclamation — the zinc process and the total chemical decomposition process — complement each other and result in a better utilisation of valuable and expensive materials.

It is difficult to judge how especially the intensively applied zinc-process will influence the demand and supply situation of tantalum. There is only a limited amount of high grade scrap, treatable in the zinc-process, available and it is questionable whether all of the existing capacities can be filled in the up and down movements of the cemented carbide industry. On the other hand, most of this high grade scrap was already treated before by other processes, mechanical or chemical, certainly with somewhat lower recovery rates in respect to the tantalum content and at higher cost. Therefore, the reduction of demand for tantalum will be less dramatically affected as the consequence of other changes which have already taken place, like coating technology and substitution.

The 15 % to 20 % figures for TaC recovered, as mentioned in the last Ayers Report, may be realistic for the recycling of high grade scrap. Another 10 %, depending on scrap grades and tantalum value, should be estimated for recycling of lower grade scrap through chemical processes.

The future of virgin tantalum carbide in steel grade cutting tools

The following paper was presented by Mr W. Hantel, Metallurg Inc., at the Twenty-first General Assembly of the T.I.C. in Stockholm on June 5, 1984.

Since 1980, the market for virgin tantalum carbide has been in decline. This has been brought about as a result of the worldwide recession and, particularly, as it affected the sale of durable goods. The first sharp decline was in automobile production, then farm and construction equipment and finally, by mid-1982, the sharp fall in energy-related equipment. Contributing to this decline were rising interest rates, fear of a tantalum shortage and the resulting rise in tantalum prices.

As prices rose during the 1979-1980 period, end-users began to look for replacement material, for reduction of tantalum content and for recovery of tantalum from scrap. Each has resulted in a permanent effect on the future demand for virgin tantalum carbide.

Let us look at business activity in the United States as measured by new orders for durable goods from 1979 through 1983 to attempt to correlate such with the demand for tantalum carbide.

1979	79.5 billion dollars
1980	79.3 billion dollars
1981	83.7 billion dollars
1982	87.9 billion dollars
1983	88.0 billion dollars

There was a slight drop from 1979 to 1980 but from 1981 through 1983 the economy showed expansion. In fact, the increase from 1981 through 1983 was 4.8 % and at the end of 1983, the increase over 1979 was 9.6 %. Thus, it is difficult to relate new orders for durable goods to the decline in sales of tantalum carbide and hard-metal.

A more realistic view would be to correlate with U.S. automotive production. On this basis, the decline in metal cutting requirements is apparent.

U.S. Auto-truck Manufacture
(units produced)

1979	Auto	9,208,074	
	Truck	3,053,033	12,261,107
1980	Auto	6,776,582	
	Truck	1,638,259	8,414,841
1981	Auto	6,251,300	
	Truck	1,689,778	7,941,078
1982	Auto	5,157,477	
	Truck	1,912,099	7,069,576
1983	Auto	6,780,245	
	Truck	2,424,191	9,204,436

From 1979 to 1982 automobile production fell by 56 % and truck production declined by 62% even though 1982 was somewhat higher than 1980. The total automobile and truck production was off by 58 %. There is an increase in 1983 over 1982 of 30 % and the forecast for 1984 (based on annualizing the first quarter production) for automobile production is 8,612,000 units to within 87 % of the 1979 level.

Another factor is the shift in the number of cylinders in the engines of the automobiles.

No. Cylinders per Engine

	4	*6*	*8*
1978	10 %	25 %	65 %
1979	18 %	24 %	58 %
1980	32 %	35 %	33 %
1981	38 %	35 %	27 %
1982	41 %	31 %	28 %

In the same manner, the cubic inch displacement of engines from 1978 to 1981 was :

% of Total Engines each Year

cubic inch displacement

	200	*201-250*	*251-300*	*301-350*	*351-400*	*400+*
1978	16.8 %	16.2 %	5.1 %	40.7 %	13.3 %	7.7 %
1979	26.7 %	13.5 %	7.5 %	40.4 %	7.0 %	4.9 %
1980	45.2 %	20.0 %	1.1 %	19.7 %	3.9 %	
1981	51.1 %	19.6 %	9.9 %	16.7 %	2.7 %	

Four cylinder engines increased during this period by 127 % while eight cylinder engines decreased by 70 %. In the same manner, the proportion of engines with small cubic inch displacement increased by over 200 % and the ones with large displacement decreased by 59 % in the 301-350 category and by 80 % in the 351-400 category. Thus, an idea of the magnitude of the decrease in metal removal is obtained and, by inference, the influence of the oil-crisis on metal removal. Moreover, there have been other factors affecting the use of tantalum carbide :

— Designed reduction of TaC in steel cutting grades of cemented carbides,
— Recovery of used TaC through reclaiming,
— Increased use of coated tools,
— Production of near-net shaped parts.

These, combined, have led to a permanent reduction of the use of virgin tantalum carbide and, dependent on the economy and the prices, could amount to as much as a 70 % reduction at any particular time. If the economy is strong and prices are stable, virgin tantalum carbide should be the primary feed-stock. But if prices begin a sharp rise, substitution will again increase and there will be more recovered scrap used.

How each of the stated factors will affect the total usage of virgin tantalum carbide is hard to predict. The use of coated tools will definitely increase, meaning that less TaC will be used in the substrate. The amount of TaC designed in the tool, either coated or uncoated, has decreased with, as a result, a permanent reduction. The significance of scrap and the effect of parts being formed to near-net shape, however, is variable.

The tantalum carbide feedstock obtained by recycling scrap is obtained by either chemical separation or by the "Zinc-process". The tantalum carbide that has been chemically separated is returned for use as a virgin pure material where as the product of the zinc-process has not been separated and is returned in graded form along with the other carbides in the scrap. The percentage of TaC in the latter case ranges from small amounts, 5 % to 10 %, to large amounts of over 50 % and even up to 100 % as reported by some consumers. How much can be used has yet to be determined. During the past recession, tools were not put to their full usage. The amount of metal removed, because of the recession, was greatly reduced and, therefore, tool life was not truly measured. Not until full production is again reached will it be possible to determine how much reclaim material can be practical. Even though the amount of metal removed will continue to decrease, the newer machine tools, better controlled and more rigid, will lead to higher cutting speeds producing higher cutting temperatures and thus putting more demand on the tool. These conditions will require continued need for tantalum carbide.

To look at the usage of tantalum carbide over the next several years at various levels of industrial activity, several assumptions must be made :

— The base period of 1979-1980 is used as 100 % capacity.
— There will be 20 % reduction in the TaC contained in cutting tools due to redesign of the tools.
— There will be a 30 % reduction in the use of virgin TaC as a result of scrap recovery and the increased usage of coated tools.
— There will be a stabilization of inflationary forces on the price of tantalum raw materials and finished products.
— The total usage of TaC/NbC will be the same in the U.S. and in Europe with Japan using one-half as much but the ratio Tac/NbC will be 100/1 in the U.S., 80/20 in Europe and 90/10 in Japan.
— Europe and the U.S. will rely on 30 % from scrap recovery and Japan will probably not begin using recovered scrap until 1985 and then only at a 10 % level.

	1979-1980	*1981-1983*	*1984*	*1985-1986*	*1987*
U.S.					
Capacity level	100 %	40 %	65 %	80 %	100 %
Hard Metal Shipments - lb.	17,000,000	6,800,000	11,000,000	13,600,000	17,000,000
Metal Removal - 45 % of total - lb.	7,650,000	3,060,000	5,000,000	6,120,000	7,650,000
Steel cutting grades 50 % of metal removal - lb.	3,825,000	1,530,000	2,500,000	3,060,000	3,825,000
TaC contained - steel cutting grades-lb.					
10 % 1979-1980	382,500				
8 % 1981-1987		122,400	200,000	244,800	306,000
TaC via scrap - lb.		(50 %) 61,200	(30 %) 60,000	(30 %) 73,440	(30 %) 91,800
TaC via virgin - lb.		61,200	140,000	171,360	214,200
EUROPE : 80/20 TaC-NbC - lb.	305,600	98,000	160,000	195,840	244,800
TaC via scrap - 30 % - lb.		29,400	48,000	58,800	73,440
TaC via virgin - lb.		68,600	112,000	137,100	171,360
JAPAN : 90/10 TaC/NbC - lb.	154,800	60,000	81,000	110,200	137,700
TaC via scrap - 10 % - lb.		—	—	—	13,800
TaC via virgin - lb.		60,000	81,000	110,200	123,900
Total Tac via virgin - lb.	843,000	189,800	333,000	418,700	509,460
Permanent loss of TaC 1979/80 to 1987					333,640

These figures are, of course, estimates and were used to determine the percentage loss, which we feel is the most important figure. We must reiterate that this represents a loss of virgin TaC which would be made from a feedstock other than scrap originating from former TaC production. Finally, the columns for 1985, 1986 and 1987 do not represent a forecast for that period but simply are used to illustrate what demand could be if the recovery continues for several years to come. As stated, these results are based on inflationary control and stable prices in the free-world economies. Sharp rises or rises in a short period would lead to the same imbalance that occurred during the 1979-1980 period which would lead, in turn, to further substitution by design and to greater use of scrap. Such could result in a permanent loss of 60 % of the virgin TaC required by the cemented carbide industry.

To look at this, a comparison of the previous chart at the 65 % and 80 % level of activity can be made assuming that the contained TaC will drop to 6 % (by redesign) and the use of scrap will increase to 50 % :

U.S.

Capacity level	65 %	80 %
Hard Metal Shipments	11,000,000	13,600,000
Metal removal - 45 % of total	5,000,000	6,170,000
Steel cutting grades-50 %	2,500,000	3,060,000
TaC contained at 6 %	150,000	183,600
TaC via scrap - 50 %	75,000	91,800
TaC via virgin	75,000	91,800
TaC via virgin from previous chart	140,000	171,360
Loss in use of virgin TaC	65,000	79,540
Additional loss	46 %	46 %

The result is a further 46 % reduction in the use of virgin TaC.

In conclusion, TaC in virgin form will continue to be used in hard metal products but it will be reduced by as much as 40 % and could be further affected if there is erratic behaviour of prices and an imbalance of supply and demand develops.

MEMBERSHIP

The following six companies were elected to membership by the Twenty-first General Assembly :

Howmet Turbine Components Corporation,
475 Steamboat Road,
Greenwich, CT 06830, U.S.A.

KBI Division, Cabot Corporation,
Box 1462,
Reading, PA 19603, U.S.A.

Mallory Capacitor Company,
P.O. Box 372,
Indianapolis, IN 46201, U.S.A.

Reading Alloys, Inc.,

P.O. Box 53,
Robesonia, PA 19551, U.S.A.

Teledyne Wah Chang Albany,
P.O. Box 460,
Albany, OR 97321, U.S.A.

Waycom Holdings Ltd.,
Wokingham Road,
Bracknell,
Berkshire RG12 1ND, England.

Pilgan Mining Company resigned from membership.

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