ITIA

Tungsten

Newsletter – December 2013

Cemented Carbides in the Soviet Union – The Unknown History

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Prof G A Meerson (front row, second left), father of the Soviet Cemented Carbide Industry, with assistants and students late 1940s.

The ITIA is grateful to Dr Igor Konyashin and Dr Lev Klyachko, the authors of this article which was specially written for this Newsletter. Their brief CVs may be found on page 15.

The Soviet Union, which was in former times a powerful and influential country, one of two major world superpowers, collapsed in 1991 and the country with this name disappeared from the map. It is still a matter of discussion in Russia and other independent states of the former Soviet Union, as well as the rest of the world, whether this event was positive or negative. Nevertheless, many aspects of Soviet history are as yet unknown in the West. In particular, the history of many branches of the Soviet industry related to the military-and-industrial complex remains almost unknown outside Russia, mainly as a result of the great extent of secrecy in Soviet times. In some cases this secrecy was reasonable, but in many cases it was excessive. This led to the fact that many important discoveries and inventions made in the Soviet Union were unknown worldwide and were "re-invented" in the West after many years, so that the pioneering role of the Soviet Union in many fields was lost.

Also, the names of many outstanding scientists and engineers working in the Soviet Union are unknown in the West. Legend has it that, after the launch of the first artificial Earth satellite in 1957, the Nobel Prize Committee sent a letter to Nikita Khrushchev, the Soviet leader at that time, asking who was the author of this revolutionary achievement in order to award him the Nobel Prize. Khrushchev's answer was: "The author is the whole Soviet nation", and Sergey Korolyov, the father of the first artificial Earth satellite, did not receive the Nobel Prize, as his name was kept secret at that time.

The history of the Soviet cemented carbide industry, which was presumably one of the biggest carbide industries worldwide in former Soviet times, is almost unknown in the West. It comprised 10 huge plants with the total annual production of over 7,000 tons of cemented carbides, a large R&D center with a number of R&D nodes at each carbide plant and a Production-and-Technological Bureau for the implementation of new carbide grades in the Soviet industry with 10 branches in all the Soviet regions; its staff was over 25,000 employees including nearly 3,000 engineers and scientists. The history of the Soviet carbide industry is unknown in the West partially because all the information about its annual production, capacities, etc was kept secret in Soviet times. Another reason is that there is only one work on this issue [1] published in a Russian journal and, of course, in Russian.

The production of cemented carbides in the Soviet Union started in the late 1920s and was based on the tungsten metal manufacture at Moscow Bulb Plant, which was re-named as "Moscow Electro-Plant", in 1926 (**Figure 1a**).

Figure 1a: The Moscow Electro-Plant in the 1920s when the first soviet cemented carbide POBEDIT was developed and its pilot production was started.
Source: http://www.melz-invest.ru/about/history/

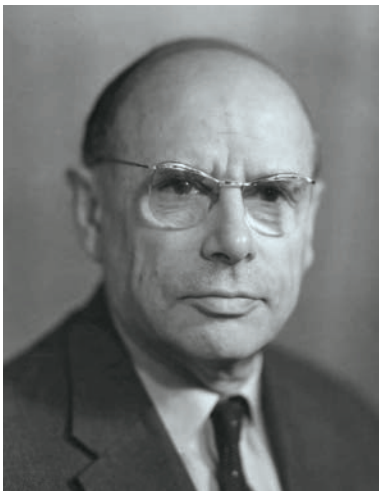

Figure 1b: Prof G A Meerson, the father of the first Soviet carbide grade POBEDIT.
Source: http://www.melz-invest.ru/about/history/

The manufacture was launched by G A Meerson, who was the father of the Soviet cemented carbides, in 1922. The picture of G A Meerson, who became later one of the leading Soviet scientists in the field of refractory and rare metals, as well as cemented carbides, is shown in **Figure 1b**. At the Moscow Electro-Plant, G A Meerson and L P Malkov developed the first Soviet WC-10%Co cemented carbide in 1929 and gave it the brand name "POBEDIT", the word originating from the Russian word POBEDA – victory. The brand name POBEDIT soon became a common noun and even now many people use it to designate cemented carbide in Russia instead of the technically correct terms "hard alloy" or "hardmetal". It is interesting to note that the same happened in Germany, where the first brand name of cemented carbide "WIDIA" also became a common noun, so that many people still employ it when they talk about cemented carbides. The development of POBEDIT was presumably a result of disclosure of the Schröter patents on cemented carbides and the first demonstration of "Widia-N" by Friedrich Krupp AG at the Leipzig Spring Fair in 1927, which caused a sensation [2]. The Soviet Union did not need to be granted a licence from Friedrich Krupp AG to fabricate WC-Co materials because the Schröter patents were applied only in Germany, Great Britain and the USA and the priority date in other countries was missed.

The pilot production of POBEDIT for metal-cutting, dies for wire-drawing, etc. was started soon after its development in 1929. Therefore, the beginning of the cemented carbide production in the Soviet Union was only 3 years later than the start of the carbide fabrication in Germany, which was launched in 1926 [2]. Valerian Kuybyshev, one of the major Soviet leaders at that time, reported the high efficiency of employing POBEDIT in various industries in his presentation at the XVI Communist Party Congress in 1930. Nearly 16 tons of POBEDIT were fabricated in 1931, which was more than the estimated total production of cemented carbides in Western countries at the beginning of the 1930s [3]. According to the report of G J Trapp et al "The German Hard Metal Industry" written as a result of a trip to Germany in September 1945 undertaken at the request of the British Intelligence Objectives Sub-Committee (CIOS Black List Item No. 21. METALLURGY), the carbide output by Krupp WIDIA was some 9 tons in 1929, dropping during 1931 and 1932, but rising again to nearly 24 tons in 1935. The large amount of cemented carbides produced in the Soviet Union in the beginning of the 1930s was presumably related to the low cost and price due to inexpensive raw materials and the absence of a need for licence payments for the Schröter patents. In contrast to that, according to Kolaska [2], the price of cemented carbides in the West was nearly 1 US dollar per gram of WC-Co in the 1930s, so that they were more expensive than gold at that time. POBEDIT was produced by dry mixing/milling of WC and Co powders in ball-mills by use of sugar water as a plasticizing agent and cold pressing and sintering in hydrogen furnaces using the equipment employed for the tungsten metal production.

The subsequent history of cemented carbides in the Soviet Union was related to the foundation of the Moscow Plant of Rare Chemical Elements in 1928, production at which was started in 1930. This plant and a number of mines and factories producing rare metals, as well as the cemented carbide shop of the Moscow Electro-Plant, became parts of the association "SOUZREDMET" (abbreviation of "Soviet Rare Metals") in 1931. Later on, the carbide production was completely transferred to the Moscow Plant of Rare Chemical Elements from the Moscow Electro-Plant and full-scale carbide production – from the fabrication of tungstic anhydride (WO_3) to sintered carbide articles – was started there in the beginning of the 1930s. Also, the production of hard-face materials on the basis of melted tungsten carbides was launched at the plant in 1931. A research laboratory headed by V Riskin was founded at the Moscow Plant of Rare Chemical Elements in the beginning of the 1930s. The first important achievement of this laboratory was a new technology for the fabrication of WC-Co mixtures, which substituted the dry mixing/milling process employed for the POBEDIT production. This technology was developed by V I Tretyakov – a young engineer just graduated from university at that time – who became later one of the leading Soviet scientists in the carbide field. The new patented technology [4] was based on the deposition of Co on WC powders from its ammonium complex solution as a result of chemical reaction with zinc dust when continuous mixing a Co- and WC-containing slurry. This technological process heralded all the modern technologies for fabrication of WC-Co mixtures by coating WC with cobalt (see eg [5]) as early as in 1932. A new carbide grade with 8 wt% Co designated as "RE8" was developed and launched on the basis of the new technology. This grade was the first one in the range of carbide grades with Co contents varying from 6 wt% (RE6) to 15 wt% (RE15).

The POBEDIT and RE8 grades were employed for the first large-scale production of drilling bits for mining in 1933. It should be mentioned that there were attempts to employ cemented carbide for mining tools at the beginning of the 1930s in Germany by Friedrich Krupp AG [2]. According to the report "The German Hard Metal Industry" mentioned above, the production of percussive drilling bits with carbide plates was started at Krupp WIDIA before the Second World War. Nevertheless, presumably because of the high prices of cemented carbides, no large-scale production of mining carbide tools was performed in Germany in the 1930s. The first experimental production of carbide rock-drilling tools was launched in other Western countries significantly later than in the Soviet Union and Germany – only in 1940 at Seco in Sweden [6]. Cemented-carbide-coated rock drills were developed by Sandvik even later (in 1942) and cemented carbide tipped rock drills were used by this company for the first time in 1944 [7]. Therefore, the pioneer in the field of the large-scale industrial manufacture of mining tools with carbide inserts was the Soviet Union. Nearly 150,000 carbide inserts, mainly of the RE12 and RE15 grades, had already been fabricated for mining bits for rock-drilling in 1936.

The technology of depositing Fe-group metals on WC powders was further developed with respect to Ni binders and the first WC-Ni grades with the brand name "RENIKS" were developed and implemented at the Moscow Plant of Rare Chemical Elements in 1933. These grades were of great importance during the Second World War, when they were widely employed for fabricating ammunition cores for missiles.

Figure 2a: The entrance check-point of the Moscow Cemented Carbide Plant (MKTC) in the 1950s. The slogan right of the image of Lenin says - „Under the banner of Lenin and Stalin, forward to the victory (of communism)". Source: http://oldmos.ru/old/photo/view/109514

In the beginning of the 1930s a number of Ti-containing WC-based carbide grades for steel-cutting, with the brand names of "Alpha 21", "Alpha 15" and "Alpha 5", were developed and their production was started in 1935. These grades became the basis of the standard metal-cutting grades with different amounts of (Ti,W)C developed later (T30K4, T14K8, T5K10, etc.), which were widely used in the Soviet industry after the Second World War.

In the late 1930s, the manufacture of rare metals was removed from the Moscow Plant of Rare Chemical Elements and the Plant was re-named as the Moscow Cemented Carbide Plant (MKTC), which is now a part of Sandvik (**Figures 2a, b**). MKTC was the only plant fabricating cemented carbides before the Second World War. Besides carbide production, intensive research activity was performed at the Plant Laboratory, which included both basic research on the W-C-Co phase diagram, carbide – metal phase diagrams, complex Ti-containing carbides, etc. and applied research on the development of novel processes of the whole carbide technological chain – from raw materials to sintered carbide articles. The State Prize, one of two major prizes for scientific and industrial achievements in the USSR, was granted to the researchers of the Laboratory for the development of new technologies for the fabrication of (Ti, W)C carbides and Ti-containing cemented carbides in 1948.

The Second World War started for the Soviet Union on 22nd June 1941 and in August 1941 it was decided to transfer the carbide production from Moscow to the Ural region. Nevertheless, carbide manufacture partially remained at MKTC during the war with the emphasis on the fabrication of carbide ammunition cores. For this work MKTS was also granted the State Prize after the war.

Figures 2b: Recent photos of the carbide workshop (left) and the entrance check-point (right) of the MKTC-Sandvik Plant. Source: Authors

Unexpectedly, MKTC, as the leading industrial enterprise in powder metallurgy, was involved in the Soviet Atomic Project in the mid 1940s. After the test of the first US atomic bomb in 1945, research focused on the creation of an atomic weapon was greatly reinforced and accelerated in the Soviet Union. At that time special Ni-based filters were needed for units intended for the separation of different uranium isotopes by the thermo-diffusion method schematically illustrated in **Figure 3a** [8]. The filters had to possess very peculiar properties, for example they had to have millions of tiny holes with a diameter of roughly 0.01 µm (10 nm) and their size had to be constant for long-term performance. A new technology for the fabrication of such filters was developed at the MKTC Plant Laboratory in a very short time and their large-scale production was quickly launched. The first Soviet atomic bomb tested in 1949 (**Figure 3b** [9]) was to some extent a result of carbide research.

The second Soviet carbide plant, Kirovgrad Hardmetal Plant (KZTS), was founded in the small town of Kirovgrad in the Ural region using equipment evacuated from MKTS in the autumn of 1941. There were no industrial buildings available in Kirovgrad at that time and, because of the great urgency, the equipment for the tungstic anhydride (WO_3), tungsten carbide and WC-Co production was placed in buildings of a city bath-house shown in **Figure 4a** and a garage.

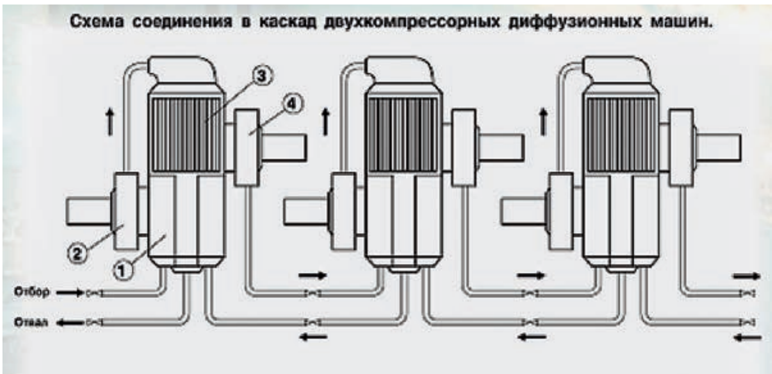

Figure 3a: Scheme of the first Soviet unit for the separation of different uranium isotopes by the thermo-diffusion method. The gas (uranium hexafluoride) is fed from the mixer (1) via the compressor (2) onto the surface of nickel filters (3), which were made at the Moscow Cemented Carbide Plant. Subsequently the partially separated gas is pumped by another compressor (4) into the next unit for further isotope separation.
Source: http://sob.znate.ru/docs/299/index-37027.html?page=4

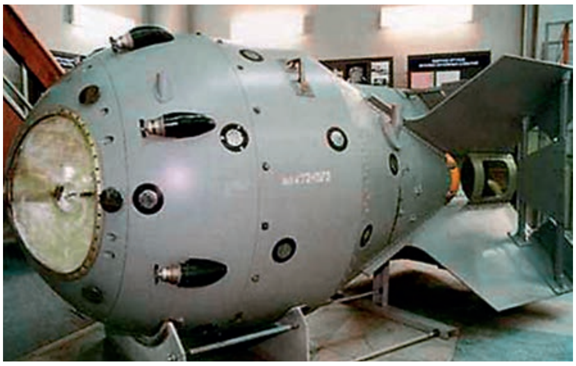

Figure 3b: The separated uranium isotope was used in the first Soviet atomic bomb.
Source: http://rocketpolk44.narod.ru/yas/rds1.htm

Figure 4a: In 1941 carbide production was transferred from Moscow into an adapted city bath-house in Kirovgrad.
Source: http://www.kzts.ru/istoriyz_kompanii

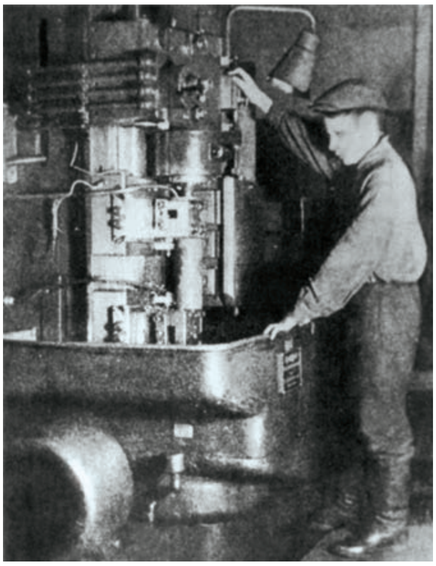

Figure 4b: The Kirovgrad Hardmetal Plant (KZTS) in the 1940s, in which during the war mainly women and children worked (right). Tungstic anhydride was produced by calcination of tungstic acid (above). Source: http://www.kzts.ru/istoriyz_kompanii

Besides the industrial equipment, the research and technical personnel were evacuated from Moscow including G S Kreimer, I S Brochin, V A Ivensen and M M Babich, who later became the major Soviet scientists working in the carbide field. The re-assembling of the equipment evacuated from Moscow and launch of the production were carried out in very unfavorable conditions in a short time, so that everybody had to work for 12 to 14 hours a day including weekends. As a result, the first cemented carbides for metal-working and ammunition cores were produced by the beginning of 1942. The ammunition cores fabricated at KZTS played an extremely important role during the famous Tank Battle of Kursk in 1943, when nearly 70% of German tanks were destroyed by missiles with the carbide ammunition cores fabricated at MKTS and KZTS. Almost all the adult men were mobilised as soldiers at that time, so that mainly women and children of 11 to 16 years worked at the plant during the war (**Figure 4b**). Some of these young workers were so small that special wooden benches had to be made in order that they would be able to work on presses and other machines. These were very hard times with a severe shortage of bread and other food stuffs, and many workers lived on the verge of starvation. Many children used to exchange the so-called “bread cards” for sweets and, as a result, had nothing to eat, so that it was decided to hand out the bread cards only on the same day that bread was delivered. Nevertheless, in spite of all the difficulties and complications, the carbide production was steadily growing at KZTS during the war.

After the Second Word War the production capacity of KZTS grew rapidly and novel carbide grades and products were developed and implemented. Also, coated cermet indexable cutting inserts with a special chromium carbide based coating obtained by a new patented technology [10] were produced at the Plant during Soviet times. Nowadays KZTS is the

Figure 4c: A contemporary view of the Kirovgrad Hardmetal Plant (left) and up to date Sinter-HIP furnaces in the sintering workshop today (right). Source:http://www.kzts.ru/istoriyz_kompanii

biggest carbide plant in Russia with an annual production of about 700 tons and modern equipment as shown in **Figure 4c**.

The history of the third Soviet carbide plant was related to the fact that the Soviet Government decided to purchase a licence and the equipment for fabrication of Ti-containing cemented carbide from the Fagersta Company (Sweden) in 1940. Because of the Second World War, the equipment was shipped to Moscow only at the end of the war. The equipment for carbide production was placed in the building of a former refining plant in the village of Nigny Kotly in the suburbs of Moscow (**Figure 5a**) and carbide manufacture was started in 1945. At that time, carbide production was kept secret and the plant was given a number 522; it was later re-named as the Moscow Pilot Plant of Refractory and Hard Metals. The Chief Engineer of this plant was G S Kreimer who later became one of the leading Soviet scientists (**Figure 5b**) and whose book "Strength of Hard Alloys" translated into English in 1968 [11–13] is the most comprehensive monograph about mechanical properties of cemented carbides even today.

As a whole, carbide production in the Soviet Union dramatically grew during the Second World War in comparison with that of 1941: by a factor of 8.7 in 1942, 13.9 in 1943 and 17.7 in 1944, mainly due to the large-scale production of ammunition cores. Because of the growing machine-building, mining industry, oil-and-gas drilling, etc. the demand for cemented carbides in the Soviet Union significantly rose after the war. As a result, the construction of a new carbide plant having the same name as the first Soviet cemented carbide grade "POBEDIT" was started in Vladikavkaz in the South of Russia in 1946 and the first Ti-containing cemented carbides were produced there in 1947. The full technological cycle of carbide production starting from tungsten ore concentrates was launched in 1950. Many at that time novel technologies for the fabrication of W and WC powders were developed and implemented at the Plant. The first Chief Engineer of the POBEDIT Plant was M M Babich, who became later one of the leading Soviet experts in the carbide field. Also, one of the authors of this article started working at the POBEDIT Plant as a foreman in 1951. The plant was one of the biggest carbide producers in the USSR, fabricating a wide range of cemented carbides from ultra-fine grades and coated grades for metal-cutting to coarse-grain grades for mining and oil-and-gas drilling. Some new technologies, for example a technology for depositing multilayer CVD wear-resistant

Figure 5a: The Hard and Refractory Metals Research Institute (VNIITS) from afar, indicated by the arrow, in the 1950s.
Source: http://moskva-bez-prik.livejournal.com/25840.html

Figure 5b: Session of the Institute Research Council in 1979. From left to right I N Chaporova, G S Kreimer, V S Rakovsky, L I Klyachko, I V Andryushin, Y N Smirnov, T N Levin, V N Strakov; the slogan: „Congratulations on the 50th Anniversary of Soviet Cemented Carbides". Source: Authors

coatings at special parameters allowing one to eliminate a decarburised η-phase underlayer, were developed and implemented at the Plant. The production at the Plant noticeably decreased after the collapse of the Soviet Union in 1991 and annual carbide production is presently around 300 tons.

Designing another carbide plant – Uzbek Plant of Refractory and Hard Metals (UzKTGM) in the city of Chirchik, now the Republic of Uzbekistan – started in 1952 and carbide fabrication at one of its shops was launched in 1959. The plant had the whole technological chain of the W and Mo fabrication starting from W and Mo ore concentrates, sourced from the Koshtanky and Ingichkinsky mines in Uzbekistan. The most advanced technologies for fabrication of tungsten metal, tungsten carbide and cemented carbides were implemented during the design of the Plant. New plasma technologies for the fabrication of nano and near-nano W and WC powders, which will be briefly described below, were developed and implemented at the Plant. There was a branch of the Hard and Refractory Metals Research Institute, which will be described in detail below, on the plant premises. The production of mainly WC-Co grades for metal-cutting and mining as well as cermets for metal-cutting was performed at the Plant in Soviet times. Carbide production at UzKTGM dramatically dropped after the collapse of the Soviet Union in 1991.

Another carbide plant – Dneprovsky Cemented Carbide Plant (DZTS) – was opened in the city of Svetlovodsk, now Ukraine, in 1970. Mainly WC-Co grades as well as coated grades for metal-cutting and Al_2O_3-based ceramics were fabricated at DZRS. Some new technologies, for example a technology of depositing wear-resistant coatings on carbide indexable inserts for metal-cutting by combining PVD and CVD, were developed and implemented at the Plant. Carbide production at DZTS also dramatically dropped after the collapse of the Soviet Union in 1991.

Besides the plants mentioned above, a part of the Soviet system of carbide production was the so-called "Special Production-and-Technological Bureau "Orgprimtverdosplav" intended to implement new carbide grades in the Soviet industry, which comprised 10 branches in different regions of the USSR. The Bureau, Plants and Hard and Refractory Metals Research Institute mentioned above were a part of the Industrial Corporation "Soyuztverdosplav", an abbreviation of "Soviet Cemented Carbides", which also comprised a number of plants producing refractory metals, hard-face materials, mining tools, etc.

One of the major parts of "Soyuztverdosplav" was its R&D Centre, the Hard and Refractory Metals Research Institute (VNIITS), founded in 1946. The first laboratories of the Institute were located in the building of the Moscow Pilot Plant of Refractory and Hard Metals shown in **Figure 5c**. A dedicated building for the Institute also shown in **Figure 5d**, which remains almost unchanged nowadays, was constructed in 1948. The research staff of the Institute was

Figure 5c: The building of the Moscow Pilot Plant, which today belongs to the Institute, where the first Institute laboratories were located. Source: Authors

Figure 5d: The first dedicated Institute building constructed in 1948. Source: Authors

Figure 5e: The new Institute building constructed in 1982. Source: Authors

around 120 scientists, engineers and technicians in 1951. In the middle of the 1960s, the Institute significantly grew and started doing research in the fields of refractory metals (W and Mo), designing industrial enterprises, developing metal-cutting and mining tools, standardisation work, etc. A new modern building of the Institute shown in **Figure 5e** was constructed in 1982. Besides cemented carbides and refractory metals the Institute had laboratories dealing with ceramics, superhard materials (diamond and c-BN), hard-face materials, mining tools, PVD and CVD coatings, etc. Both basic research in the field of materials science of cemented carbides and refractory metals and applied research on the development of new carbide grades and technologies were carried out at the Institute. The Institute comprised departments dealing with raw materials (Dr V G Bukatov), analytical chemistry (Dr S N Suvorova), examination of carbide physico-mechanical properties (Dr G A Travushkin), metal-cutting (Dr E F Eichmans), rock tools and rock-drilling (Dr A M Chuvilin), development of mining grades and grades for special applications (Prof V A Falkovsky), development of grades for metal-cutting (Dr A I Anikeev), ceramics for metal-cutting and wear-applications (Dr V N Anikin), etc. A number of new grades for mining, construction, metal-cutting, high-pressure high-temperature components, etc from ultra-fine to ultra-coarse were developed and implemented in industry by the Institute scientists. The implementation process included a pilot-scale production at the pilot plant (Moscow Pilot Plant of Refractory and Hard Metals mentioned above) with further up-scaling at the large carbide plants with the aid of the R&D nodes at each plant. Also, all the Soviet Standards (GOST) in the carbide field were elaborated at the Institute. Drafts of a number of the ISO standards in this field were developed by the Institute employees too, as the Institute headed the ISO sub-committee dealing with control of chemical purity and mechanical properties of cemented carbides from 1969 till the mid 1990s.

A number of books on cemented carbides, which are listed below, were written and published by the Institute researchers and some of them became “bibles” in the carbide field in the Soviet Union and other Socialist counties, eg the books of G S Kreimer [11–13] and V I Tretyakov [14, 15]. Unfortunately, only the book of G S Kreimer [11] was translated into English [12]. The list of the books is below:

- G S Kreimer. Strength of cemented carbides/Strength of hard alloys [11–13]
- V I Tretyakov. Bases of materials science and technology of fabrication of sintered cemented carbides [14, 15]
- V I Tumanov. Properties of alloys of the tungsten carbide – cobalt system [16]
- V I Tumanov. Properties of alloys of the tungsten carbide – titanium carbide – tantalum carbide – niobium carbide – cobalt system [17]
- I N Chaporova, K S Chernyavsky. Structure of sintered cemented carbides [18]
- T B Gorbatscheva. X-ray diffractometry of cemented carbides [19]

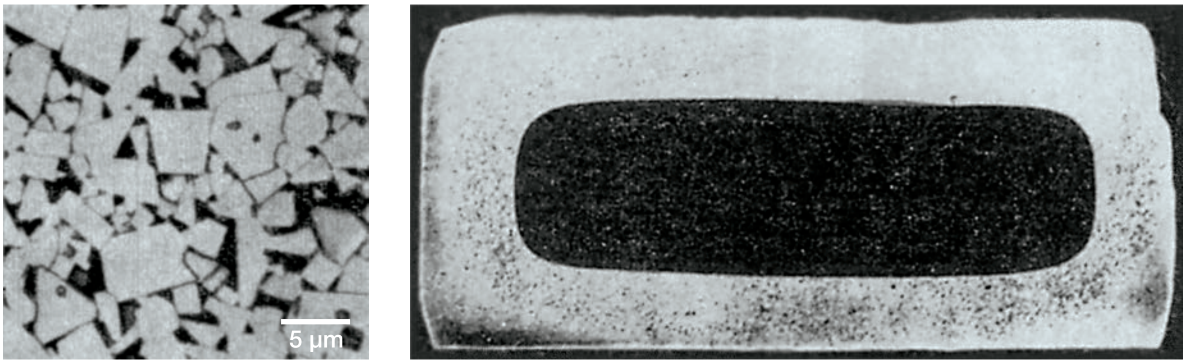

Figure 6: Cemented carbide grades developed in the Soviet Union during the 1960s pioneering present state-of-the-art carbide grades: microstructure of ultra-coarse WC-Co cemented carbide, produced before 1966 [11-13] (left); cross-section of a functionally graded cemented carbide part (cm size, Murakami etched). Production was done in a two step process, first sintering a part with η-phase and secondly carburizing the part from the outside during a second liquid phase sintering run (right). This technology was developed before 1962 [14, 15]. Source: Authors

A great number of novel technologies and carbide grades were developed at the Institute. Unfortunately, very frequently, Russian patents or so-called "Author Certificates" on novel technologies and materials developed in Soviet times were not published, so that, in many cases, one cannot trace the novel technologies and materials patented at that time even nowadays. Nevertheless, some information on the technologies and materials, which were novel at that time, was disclosed and it is possible now to trace when they were developed. **Figure 6** illustrates two examples of cemented carbides developed at the Institute, which heralded materials being now state-of-the-art in the carbide industry. One of them is an ultra-coarse WC-Co grade with WC mean grain size of about 5 μm, the microstructure of which was published [11–13] in 1966, so that this cemented carbide was presumably developed well before 1966. Such ultra-coarse cemented carbides were afterwards "re-invented" in the West (see eg [20]) and are presently widely employed for the fabrication of road-planing and coal-cutting picks. Another example is functionally graded cemented carbide, the microstructure of which (shown in **Figure 6**) was published in 1962 [14, 15]. The functionally graded cemented carbide was obtained as a result of carburising a fully sintered carbide article, which originally comprised η-phase, during liquid-phase sintering. This method is very similar to that used for the fabrication of functionally graded WC-Co materials today known as the "DP (Dual Properties) carbides" (see eg [21, 22]), which are now widely employed in mining applications.

Below is a list of technologies and materials developed at the Institute and afterwards implemented in the Soviet industry on a large scale, which appear to pioneer research in the particular carbide field heralding those described in numerous publications today. Of course, it cannot be ruled out that other carbide companies developed similar technologies and materials earlier but did not disclose them. Nevertheless, according to the authors' knowledge the developments briefly described below were new and original at the time they took place in the Soviet Union.

1. High-temperature processes for fabrication of WC powders

It is difficult to trace where and when the first production of coarse-grained W and WC powders at elevated temperatures was started. The report entitled "The German Hard Metal Industry" mentioned above provides evidence that temperatures of hydrogen reduction of W powders did not exceed 920°C and carburisation temperatures did not exceed 1600°C at the German and Austrian carbide plants in 1945. Experiments on increasing the W reduction temperatures up to 1200°C were carried out at the Institute by GS Kreimer at the end of the 1940s and the first pilot production was launched in the beginning of the 1950s followed by the large-scale production started at the POBEDIT Plant in 1956 [23]. Further research on increasing the carburisation temperatures was carried out at the Institute by V I Ivensen, V A Falkovsky, O N Eiduk, etc in the 1950s and 1960s. As a result of the new technology for the fabrication of WC powders, cemented carbides were developed and patented worldwide (see eg [24]). This technology included the hydrogen reduction of W powders at temperatures of up to 1200°C and high-temperature carburisation of the

W powders at temperatures of up to 2200°C. The fact of significantly greater plastic deformation before failure of WC-Co cemented carbides obtained from the high-temperature carburised WC powders was well established and described in detail in ref [14] in 1962. The employment of coarse-grain WC powders after their high-temperature carburisation for mining grades is currently state-of-the-art in the cemented carbide industry.

2. Nano and near-nano W and WC powders obtained in hydrogen-based plasmas

The development of first high-power arc plasma guns in the Soviet Union in the 1950s allowed research on the fabrication of near-nano and nano powders, particularly W and WC powders, at the beginning of 1960s [25]. It was established that the W powders obtained by the reduction of WO_3 in hydrogen plasmas had a mean grain size of nearly 100 nm or finer [26, 27]. Pioneering research on obtaining nano and near-nano WC powders, which are shown in **Figure 7**, was started in the Soviet Union in the 1960s and resulted in the development of a plasma technology for fabrication of such powders and thus cemented carbides [28, 29]. The technical development and implementation were carried out by a number of Research Institutes including the Institute of Metallurgy of the Soviet Academy of Sciences (Y V Zvetkov, Y V Blagoveshensky, etc) and the Hard and Refractory Metals Research Institute (V K Rumayntsev, L I Klyachko, T A Elemyanova, etc) [30]. The large-scale production of near-nano and nano W and WC powders by use of the industrial unit PUV-300 was started at the Uzbek Plant of Refractory and Hard Metals in 1985.

3. Nanostructured WC-Co powders obtained from solutions of mixed tungsten and cobalt salts

A process for the fabrication of WC-Co powders by obtaining a solution of their salts, its evaporation, calcination, hydrogen reduction of the mixed oxides and their final carburisation was developed and patented by G A Meerson, the father of Soviet cemented carbides and pioneer of all the later research works in this field, in 1930–1936 [31, 32]. Further R&D on this process was carried out at the Institute in the 1970's by N K Vaskevich, V I Tretyakov, V K Senchikhin, et al. and resulted in the large-scale production of nanostructured WC-Co powders at the POBEDIT Plant [33–37]. These powders were employed mainly for plasma- and thermally sprayed hard-facing due to the very fast grain growth of WC during conventional sintering of such nanostructured powders. A similar technology for the fabrication of nanostructured WC-Co powders developed in the West by Nanodyne was "re-invented" only in 1991 [38]. The approach of obtaining nanostructured WC-Co powders starting from a mixture of their salts and oxides still continues and is the subject of recent publications (eg [39]).

4. WC powders with uniform grain size distribution as the basis for cemented carbides with uniform microstructure

A technology for the fabrication of W and WC powders with uniform grain size distribution and cemented carbides with uniform microstructure was developed at the Institute by

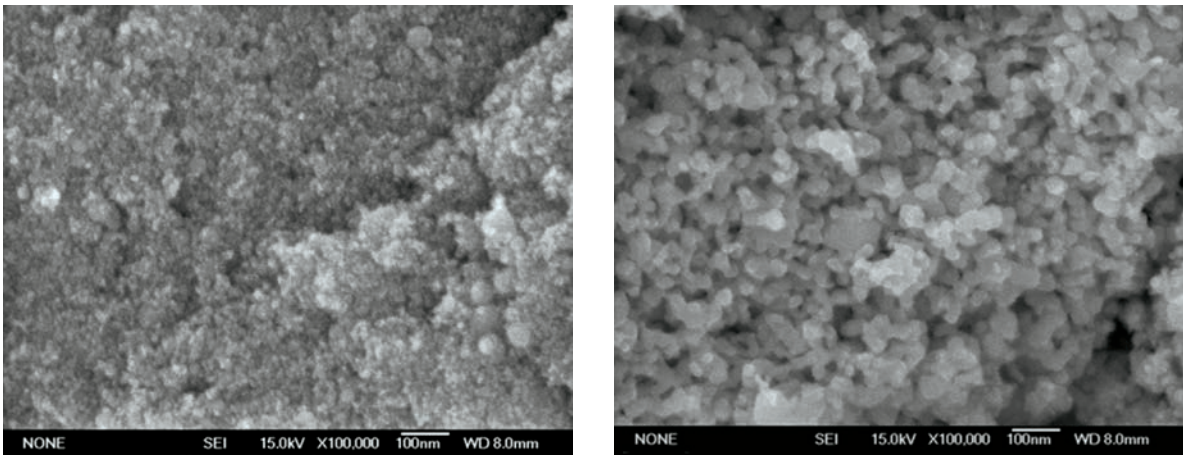

Figure 7: Nanostructured WC powders with different BET specific surface areas, produced in hydrogen plasma. left: 27 m²/g; right: 12 m²/g. Source: the authors

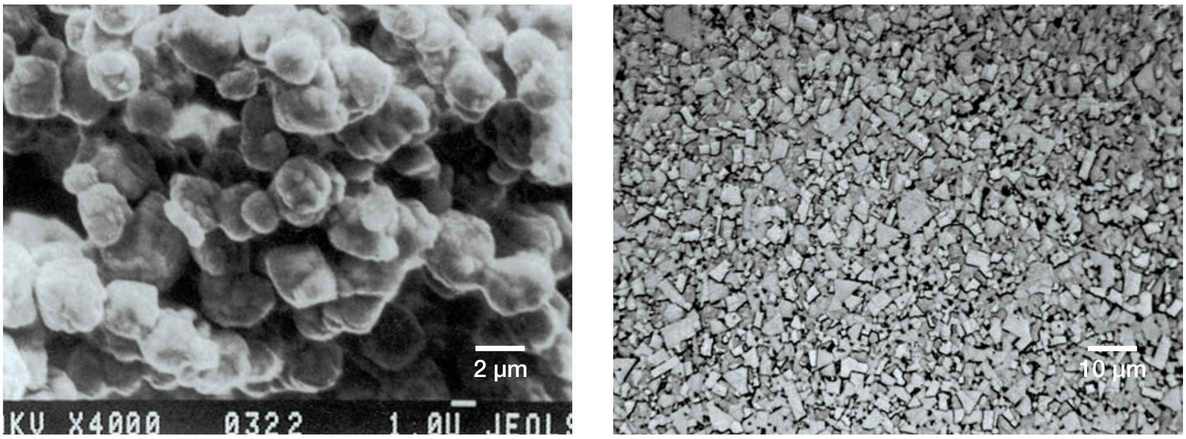

Figure 8. WC powders with uniform grain size distribution (left) and cemented carbides (WC-8%Co) made from these powders. Source: the authors

I Konyashin, V K Senchihin, V K Bukatov, M M Smirnova et al. and implemented in industry on a large scale in the 1980's [40–42]. The morphology of such a WC powder and the microstructure of the cemented carbide without grain growth inhibitors are shown in **Figure 8**. Both medium- and coarse-grain cemented carbides with uniform microstructure completely free of large WC grains can be produced by use of the WC powders without employing grain growth inhibitors, thus solving the important problem of the presence of large and abnormally large WC grains in the carbide microstructure [43]. As a result, the cemented carbides are characterised by improved combinations of hardness, fracture toughness and transverse rupture strength and prolonged tool lifetime in metal-cutting. The cemented carbides with uniform microstructure are currently employed on a large scale in metal-cutting as substrates for wear-resistance coatings and for other applications.

About 1,000 scientists, engineers and technicians were employed at the Institute in 1990 before the collapse of the Soviet Union, but this number dramatically dropped in the 1990s because of the lack of government funding and the very poor general situation in the Russian carbide industry at that time. There remain presently less than 100 employees at the Institute and it still suffers from the shortage of government funding.

It should be mentioned that carbide research was performed not only in Moscow but also at two research Institutes, which did not belong to the Industrial Corporation "Soyuztverdosplav", in Kiev, Ukraine. These were the Frantsevich Institute for Problems of Materials Science founded in 1955 and the Bakul Institute for Superhard Materials. The researchers at both Institutes dealt mainly with the WC based grades, covering a wide range of directions that includes the liquid and solid state sintering, the heat-treatment of cemented carbides, as well as the fabrication of high-pressure high-temperatures components for diamond synthesis. The Frantsevich Institute for Problems of Materials Science presently employs about 1,700 people and is a large scientific and research complex, including a Special Design Bureau with Pilot Plants and a Computer Centre. The most known scientists from FIPMS in the field of cemented carbides are S S Ponomarev, Yu V Milman, A V Shatov, A V Laptev, S A Firstov, A A Mikhailov, V V Sverdel, and M S Kovalchenko. The Bakul Institute for Superhard Materials was founded on the back of the Central Design- and-Technological Bureau of Carbide Tools in 1961. M M Babich mentioned above many times actively participated in the Institute's foundation and was one of its chief executives for many years. He wrote a book entitled "Non-Uniformity of Cemented Carbides with Respect to Carbon Content and Method of Its Elimination" containing lots of useful information on materials science and technological aspects of WC-Co cemented carbides. The Bakul Institute for Superhard Materials is still active in carbide research employing a number of outstanding scientists in the field of cemented carbide including N V Novikov, V P Bondarenko, I V Andreev, A F Lisovsky, M G Loshak, V T Golovchan, N V Novikov, etc. The history of the Bakul Institute for Superhard Materials and the present state of research in the carbide field are summarised in the book [44].

Figure 9a: View of the Moscow Mining Academy in the 1920s. Source: http://old.misis.ru/ru/65

Figure 9b: Professors and researchers at the Department of Metallurgy of Rare Metals and Cemented Carbides in 1949: top left O E Krein, bottom left G V Samsonov, center G A Meerson, top right A N Zelikman, bottom right I P Kislyakov. Source: Authors

The history of cemented carbides in the Soviet Union would be incomplete if one does not mention the major University providing highly qualified engineers and researchers in the carbide field – the National University of Science and Technology MISIS, formerly the Moscow Steel and Alloys Institute (State University). The University was a part of the Moscow Mining Academy (**Figure 9a**) in the 1920s and became an independent university "Moscow Institute (State University) of Non-Ferrous Metals and Gold" in 1930. G A Meerson founded the Department of Refractory Metals at the University in 1931 and the first group of 9 students were granted an MS diploma at the Department in 1933. The department was transferred to the Moscow Institute (State University) of Fine Chemical Technologies in 1935 and back to the Moscow Institute (State University) of Non-Ferrous Metals and Gold in 1943 and re-named as "Department of Metallurgy of Rare Metals and Cemented Carbides". Five professors and researchers, shown in **Figure 9b**, were employed at the Department in 1946. They were, among others, its Head and Founder Prof G A Meerson,

Figure 9c: Prof G A Meerson teaching foreign students at the Department in the 1950s. Source: Authors

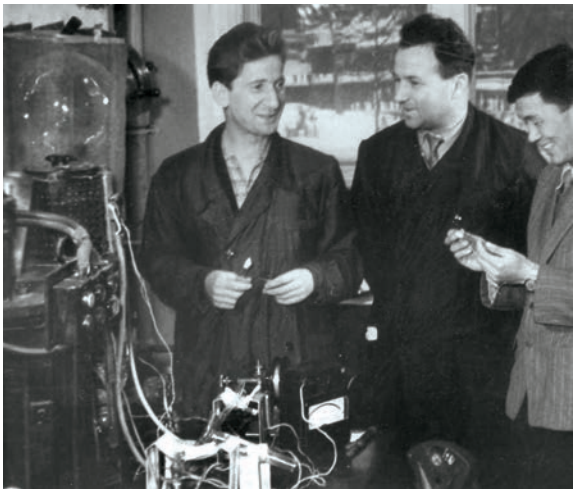

Figure 9d: The Departments laboratory in the 1950s. Source: Authors

Figure 9e: The University today: The University building hosting the Department of Powder Metallurgy and Functional Coatings.

Figure 9f: Prof E A Levashov, Department Head, granting masters diploma to foreign students.

Dr G V Samsonov who later became famous for his research works and books on refractory compounds (see eg [45–52]), and Prof A N Zelikman, one of the major Soviet experts in the field of refractory metals, author of a number of books on metallurgy of tungsten, molybdenum, rhenium and other refractory metals (see eg [53, 54]). The first group of five students with the specialty “Cemented Carbides” graduated from the University in 1947. The Department (**Figures 9c, d**) became a part of the Moscow Steel and Alloys Institute (State University) in 1962 and after a number of re-organisations, was renamed as “Department of Powder Metallurgy and Functional Coatings”. More than 3,000 students graduated from the Department, including foreign students, currently working all over the world (**Figures 9e, f**), as well as over 370 PhD students. The Department is today headed by Prof E A Levashov and has a special English language course on powder metallurgy and protective coatings for foreign students. A new English language course on nanostructured materials, including nanostructured hard materials, for foreign students is being prepared and will be launched in 2014. The Department is very active in research on cemented carbides and hard coatings, which is performed by Prof V S Panov, Prof V K Narva and Prof V N Shumenko.

It should be noted that in spite of the very strong research potential of the Soviet scientists and many outstanding achievements in R&D, the technical state of carbide production and the quality of cemented carbides in the USSR were in many cases insufficient. Because of the fully planned and centralised Soviet economy and the lack of completion among industrial enterprises there was no driving force for large-scale implementations of new technologies and materials. In many cases, Managing Directors of industrial enterprises were at the same time their Research Directors in order to solve this problem or, as they said at that time, “to reinforce ties between research and production”. For example, G A Meerson headed the Department of Metallurgy of Rare Metals and Cemented Carbides at the University and was at the same time the Technical Director of the Industrial Corporation (Trust) of Cemented Carbides. One of the co-authors of this paper was Managing Director of the Industrial Corporation “Soyuztverdosplav” and at the same time headed the Hard and Refractory Metals Research Institute for about 11 years to make it easier to implement the Institute’s developments in industry. Nevertheless, all the work on implementation of new technologies and materials for each industrial enterprise was governed by special plans annually created at a high level by Soviet Ministers, which was not the best way for innovation. Therefore, if even a new carbide grade was successfully implemented at a pilot-production level, its quality sometimes became unsatisfactory after the large-scale implementation at huge Soviet industrial plants.

One of the co-authors of this paper, as Managing Director of "Soyuztverdosplav", visited both research and production facilities of various Western carbide companies in Soviet times. As a result, he had an impression of how the Western system of implementation of innovations worked in the environment of very strong competition among different companies. He came to the conclusion that the Soviet system of innovations and their implementation had to be changed and gave a number of lectures on this issue at different Soviet industrial and research organisations. After some time, he was summoned by the KGB and requested to end giving such lectures; the Soviet system was very conservative and extremely resistant to any changes. Another big disadvantage related to the socialist planned economy was that there was no need for any cost reduction, as all the prices of industrial products were set by central government organisations and ministries. This also significantly hampered the implementation of any innovation in industry, leading to the dramatically reduced effectiveness and productivity of labor in the Soviet Union. Also, the fact that a huge proportion of the Soviet economy worked for the military-and-industrial complex had a negative effect on the economy as a whole, leading to shortages of up-to-date equipment and instruments for the civilian industry.

Nevertheless, as a result of the efforts of the very highly qualified research and management personnel, the Soviet cemented carbide industry was one of the biggest in the world and relatively technologically advanced. Its history, therefore, can be designated as a success story.

The authors:

Dr Igor Konyashin graduated from the National University of Science and Technology MISIS, Moscow, in 1982, obtained PhD at the University in 1985 and worked at the Hard and Refractory Metals Research Institute, Moscow, until 1995, when he moved to the Max Planck Institute for Metals Research, Stuttgart, Germany. He is presently Manager of Research and Development at Element Six GmbH, Germany.

Dr Lev Klyachko graduated from the North-Caucasus Mining-and-Metallurgy Institute (University) in 1951 and afterwards worked at the POBEDIT Plant in Vladikavkaz, first as a foreman and finally as Managing Director. He moved to Moscow in 1965 and since that time headed the Soviet Cemented Carbide Industry being the General Manager of the Industrial Corporation "Soyuztverdosplav" until 1986. He is presently Senior Expert at the Joint-Stock Company VNIIINSTRUMENT and Chief Scientist at KZTS, Russia.

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ITIA news

The 26th Annual General Meeting, 23–25 September, Sydney

The meeting surpassed expectations as the President, Claude Lanners of CERATIZIT SA, welcomed 220 delegates and laid to rest fears that the long journey for Europeans and North Americans might be detrimental to attendance, despite the fact that this was the first time that Australia was a venue for an ITIA meeting. Added to this concern was the fact that Sydney had recently been rated the third most expensive city in the world, never before having appeared in the top 50! Fortunately the thought of missing an occasion to see old friends, renew business contacts and make new deals was, as always, the prime consideration and the magnetism of the tungsten industry was irresistible.

Andreas Lackner with Ulrika Wedberg, President of Wolfram Bergbau. Both are members of the Executive Committee.

A number of delegates did indeed have journeys with long delays but, smiles soon returned, deals concluded and attention given to the authoritative guest speakers and their presentations. For the first time since 1990, the ITIA decided to revisit the subject of pricing mechanisms and, under the watchful eye of Counsel, Claire Hack of Metal Bulletin and Nigel Tunna of Metal-Pages, took turns to explain the modus operandi their journals employed to formulate and publish tungsten quotations. As one observer commented afterwards, looking back to reports of the first such session at the Tungsten Symposium in 1979, little had changed either in terms of the concerns of the industry or the explanations of the compilers.

Visit to Mt Carbine Tungsten Mine

The largest US consumer of tungsten, Global Tungsten & Powders Corp, led by its President & CEO, Andreas Lackner, with the support of Wolfram Camp Mining Pty Ltd, generously hosted a dinner at the famous Doyles restaurant on Watson Bay where delicious grilled seafood, the local Barramundi and succulent steak were enjoyed with local wines.

The last issue of the Newsletter (June) covered in detail the mine visits which were to be hosted by Wolfram Camp Mining, an open-pit tungsten and molybdenum mine, and the Mt Carbine Tungsten Mine. Some 70 delegates flew from Sydney to Cairns to participate in the guided tours and several seized the opportunity to explore the Great Barrier Reef and do some fishing. At least one encounter with a two metre barracuda was accidental, not to say alarming, but another resulted in a black marlin weighing in at 400kgs. Congratulations to Steve Nance!

Group photo at Wolfram Camp Mine

HSE Database

In the course of his opening address, the President announced that, after two years' effort, the HSE Database in the ITIA members' Area of the website, compiled and constructed by Dr Philip Mitchell (Emeritus Professor, Reading University) and Mr Carmen Venezia (Manager, Safety and Environment, Global Tungsten & Powders Corp) would go live at the AGM.

The Database had been derived from the original literature search with more than 4,700 citations and provided ITIA members with easy access to the literature on tungsten and its compounds in biology and the environment. The Database would be the first port of call for members seeking information on the occurrence, uptake and transport of tungsten in humans, animals and plants; tungsten in the work place and the environment (soil, groundwater, rivers and the oceans); the impact of tungsten on human health; its toxicity, and regulatory legislation; and the chemistry and biochemistry of tungsten related to its biological impact.

Before Mr Carmen Venezia elaborated on the mechanics of the website, the new HSE Director, Dr Ranulfo Lemus (introduced in the June Newsletter), gave his first presentation in this capacity on ITIA's HSE work programme, to widespread applause.

ITIA membership

Welcome to:

Almonty Industries Inc, a tungsten concentrate producer in the Iberian Peninsular

Saxony Minerals & Exploration AG has received the mining rights to mine tungsten, tin and other amended ore in Pöhla-Globenstein, Ergebirge, Germany.

Thor Mining Plc which proposes to develop its 100% owned Molyhil tungsten deposit in the Northern Territory of Australia

Wolf Minerals Ltd which is a listed specialty metals company focused on developing the Hemerdon Ball tungsten and tin project in Devon, in the UK

Election of Vice-President

Members at the AGM unanimously approved the election of Mr James Oakes, Vice President, Materials Engineering, Kennametal as Vice-President in 2014. Jim has been connected with ITIA in one way or another for many years and was elected to the Executive Committee in 2010.

James Oakes, newly elected ITIA Vice-President

Election to the Executive Committee

Members at the AGM unanimously approved the election as members of the Executive Committee of:

- Mrs Silke Gray, Director, Procurement Business Segment AMCP, HC Starck GmbH
- Mr Akira Kawaguchi, General Manager, Procurement and Logistics Department, Japan New Metals Co Ltd

ITIA's 27th AGM, 21–25 September 2014, Canada

The ITIA's 27th Annual General Meeting in Canada (probably Toronto) and the provisional outline programme is as follows:

Date	Meeting / Function
Sunday 21 Sept	• Tungsten Consortium Technical Committee • ITIA HSE Committee
Monday 22 Sept	• Tungsten Consortium Steering Committee • ITIA Executive Committee • ITIA Reception and Dinner
Tuesday 23 Sept	• AGM • Tungsten Consortium Committee • Dinner
Wednesday 24 Sept	• AGM
Thursday 25 Sept	• Optional Plant Visit(s)

Further details of this annual event, at which the worldwide industry gathers, can be found on our website – www.itia.info and will be updated to include the expanded programme and registration form in May. Companies which are not ITIA members may attend (there is a fee) and receive presentations on a variety of industry and general topics.

Dr Burghard Zeiler
Secretary-General