

Tungsten – a very hard business

History

The history of tungsten goes back to the end of the Middle Ages. The miners in the Erz Mountains of Saxony noticed that certain ores disturbed the reduction of cassiterite and induced slagging. “They tear away the tin and devour it like a wolf devours a sheep”, a contemporary wrote in the symbolic language of the Middle Ages. The miners gave this annoying ore German nicknames like “wolfart,” “wolframite” or “wolfrahm.”

In 1758 the Swedish chemist and mineralogist Axel Fredrik Cronstedt discovered and described an unusually heavy mineral that he called “tungsten,” which is Swedish for heavy stone. He was convinced that this mineral contained a new and as yet undiscovered element. It was not until 1781 that a fellow Swede, Carl Wilhelm Scheele, who worked as a pharmacist and private tutor in Uppsala and Köping, succeeded in isolating the oxide (tungsten trioxide). Independent of Scheele, two Spanish chemists, the brothers Elhuyar de Suvisa, first reduced the mineral wolframite to tungsten metal in 1783.

Jöns Jacob Berzelius (1816) and later also Friedrich Wöhler (1824) described the oxides and bronzes of tungsten and gave the new metal the name wolframite. While the name wolfram established itself in Germany and Scandinavia, the Anglo-Saxon countries preferred Cronstedt’s “tungsten” for the name of this element. Even though the “International Union of Pure and Applied Chemistry,” better known as the IUPAC, recommends that tungsten be abandoned worldwide in favor of wolfram, the name tungsten will be used in this book.

A metal of many superlatives

There is no doubt that tungsten is a metal of many superlatives. It not only has the highest melting point sources in scientific literature vary between 3,387° C and 3,422° C – but also the best high-temperature mechanical properties and the lowest expansion coefficient of all metals. A temperature of some 5,900° C is needed to bring tungsten to a boil – which corresponds to approximately the temperature of the sun’s surface.

With its density of 19.25 gcm³, tungsten is also among the heaviest metals. Its electric conductivity at 0° C is about 28 percent of that of silver.

Pure tungsten is a shiny white, cubic body-centered, latic-type metal, which experts refer to as a “tungsten type.” Compared with air, it is extraordinarily stable at room temperature. It only begins to oxidize gradually to tungsten (VI)-oxide (WO₃ or tungstic ocher) at red-heat temperatures. Mineral acids such as hydrofluoric acid and nitro-hydrochloric acid corrode tungsten very slowly. However, it will dissolve very quickly in a mixture of nitric and hydrofluoric acid.

Tungstates form when tungsten is fused with alkali hydroxides. They are derived from a monotungstic acid in highly alkaline solutions. So-called polytungstates are obtained when such solutions are acidified. Sodium carbonate and sodium nitrate melts will also dissolve the metal..In its purest form, tungsten is a quite pliant, easily processed metal. However, it usually contains small amounts of carbon and oxygen, which give tungsten metal its extreme hardness and brittleness.

In contrast, ultrapure tungsten single crystals are still ductile at the temperature of fluid nitrogen, or -196° C. Because of the different amounts of added carbon, the levels of hardness documented in scientific literature vary from 4.5 to 8.0 according to the Mohs' scale.

Not much is known about the toxicity of tungsten, but it is generally estimated to be quite low. Biologically speaking, tungsten is an adversary of molybdenum. Consumption of very high amounts of tungsten in rats induces a decrease in the activity of the liver xanthine oxidase. A similar effect was observed in bacterial molybdenum proteins.

Tungsten is usually found in the form of tungstates in nature. The most important minerals are wolframite and scheelite. Others include lead tungstate (stolzite) and tungstic ocher (tungstite). Another mineral with the name mpororoite, which is an iron aluminum tungstate, has been found in Uganda.

China has the world's largest deposits of tungsten – followed by Canada, the United States, the countries of the CIS, Australia, Korea, Turkey, Bolivia and Burma. The largest European reserves are in Portugal, France, Austria, Sweden and southern England; smaller deposits are found in the Erz Mountains, near Reichstein (Slask) and also in Baden-Württemberg, Germany.

H. C. Starck imports tungsten concentrates from all regions of the world – until recently, also in large amounts from China. The significance of China for raw materials markets has increased substantially in recent years due to the decrease in prices of the metal.

Because of the downward pressure on prices, a number of mines in the West were forced to lower output; in some cases the mines were shut down altogether. This collapse of prices was truly dramatic: by the end of 1986, tungsten was valued at only about 20 percent of the value that it had had in the late 1970s and early 1980s.

One of the most important tungsten producers

Worldwide demand for tungsten is currently slightly less than 50,000 tons per year. A more exact estimate is not possible because both of the most important tungsten-producing countries – the former Soviet Union and the People's Republic of China – publish incomplete data on the amount produced and used.

About half of the estimated total consumption is attributed to countries with market economy systems. H.C. Starck contributes some 15 percent of the supplies for the West. Consequently, the company is among the most important producers of tungsten and tungsten products, along with Osram Sylvania, formerly GTE Sylvania Products Company in Towanda, Pennsylvania, in the United States.

A historical view of tungsten metal processing

For Gebr. Borchers AG, the year 1889 was a milestone in the history of the company. It was the year that Gebr. Borchers decided to buy a shipment of tungsten ore –wolframite –from Cornwall in the U.K. for 40,000 goldmarks. The aim was to produce a metallic tungsten product suitable for use in the steel industry.

By today's standards, the flow chart of the first tungsten production process in 1899 seems quite simple. It was set up in an outdated factory for the production of permanganate and was based on the extraction process for native black manganese oxide.

The production site was in the plant located in the old city of Goslar, the so-called Pfalz borough. Decomposition took place in four separate steps:

- 1) fusing wolframite with sodium carbonate in an iron crucible.
- 2) leaching and crystallizing sodium tungstate.
- 3) decomposing sodium tungstate with hydrochloric acid to tungstic acid.
- 4) reducing the dried and calcined tungstic acid with charcoal in a sealed clay crucible.

With a tungsten content of between 96 and 98 percent, and less than 0.5 percent carbon, the metal obtained as sintering material from this method – and freed from marginal areas that had not been completely reduced – largely fulfilled the demands of alloy producers. The yield of usable metal was initially scarcely more than 50 percent, although this figure soon improved.

However, difficulties with a residual content of alkali in the metal arose as a result of the first supplies. This was given little attention and probably not properly analyzed. In order to avoid carbon absorption during alloy production, steelworks smelted a master alloy containing 30 percent tungsten from tungsten metal and refluxes. The clay and graphite crucibles they used for smelting were destroyed after just a few passes if the alloy contained higher amounts of alkali.

For several years, Gebr. Borchers had difficulties matching the very clear-cut specifications offered by the company's English and American competitors: "tungsten metal, free from substances harmful to steel, free from tungsten in the form of oxide and free from soluble tungstates." It was a demand that German tungsten producers refused from the start and probably resulted in a negative impact on their businesses.

These strict specifications were not met in the first delivery contracts of Gebr. Borchers AG, and consequently, customers refused to accept the products. The result was litigation that lasted several years. In fact, the lawsuit reached the German supreme court and was finally resolved through an out-of-court settlement with the metal traders.

In retrospect, the alloying industry's high demands also had a positive side. Gebr. Borchers was repeatedly forced to make greater efforts to improve the quality of its products – an investment that later proved to be worth the effort.

For example, Gebr. Borchers chemists added two more steps to their four-step process. After leaching the sodium tungstate, the tungsten was first precipitated as calcium tungstate. This product – synthetic scheelite – was then decomposed with hydrochloric acid to a much purer tungstic acid.

A further improvement was the so-called preliminary precipitation. In this process, the alkaline tungstate solution was neutralized with hydrochloric acid, which caused a precipitation of the silicic acid and stannic acid also contained in the solution. Both these additions to the process remained a part of Goslar's tungsten chemical production methods for decades.

As the story goes, Dr. Karl Borchers – along with Senator Hermann Borchers, one of the Borchers brothers referred to in the company name – was a particularly creative chemist. He was dissatisfied with the fact that insufficient yields were obtained from fusion decomposition – especially from mixed ores – and that large quantities of impurities were dissolved with the charges. Even prior to 1905 he had already performed successful tests with an autoclave decomposition process. This method was then introduced in a new factory at the plant in Oker, and a patent for the process was granted in 1908. Within the company it was referred to as the “Hempel patent” in recognition of a former employee’s contribution to its development.

In the years before World War I, substantial sales – in many cases of products that were to be modified – were achieved with this higher quality, 96 to 98 percent tungsten metal, whose carbon content was under 0.2 percent. Customers for these products were not only located in Germany, but also in England and the United States. Production increased by leaps and bounds, as the following figures clearly illustrate:

The outbreak of the first World War led to a severe setback in business – mainly due to the loss of foreign business and increased difficulty in securing raw materials.

Difficult working conditions

The conditions of the first metal production were not to be compared with today’s safety standards. It is remarkable how employees in those days managed their production problems. The hydrochloric acid was heated in boilers made from chiseled sandstone. Corrosive liquids were conveyed along clay pipes in so-called acid eggs. Effective exhaust air scrubbers were not available. The universal remedy for all gas and dust problems was the chimney stack. A limestone pit was used to treat acidic waste water.

The transport of raw materials, auxiliary substances, products and hazardous materials was not much better. Horse and cart were the only available means of transportation for delivery to the factory yard or to the train station.

On the other hand, the standard of furnace construction and firing technology was astoundingly high to the benefit of the tungsten project. The furnaces equipped with crucibles were in many cases fired regeneratively and could be brought to temperatures of around 1,400° C by blast heating. The reduction furnaces used in those days were fired with coaltar oil. They were loaded from the side, and some 300 kg of tungsten metal could be produced at one time in the furnace chamber.

The further development of tungsten chemistry

Goslar developed a special problem after World War I. In 1917 the plant was allotted a tin slag from Geisingen in the Erz Mountains that contained some 10 percent tungsten (VI)-oxide and nine percent tin. In very short period of time, a completely new decomposition method had to be developed to process these slags. As it later turned out, this move would pave the way for further pioneering developments.

Thanks to the chemists Dr. Karl Borchers and Georg Hüffner, a tungsten oxide that could be used for metal production was produced from this raw material as early as 1918. The key to this success was fusion decomposition with sodium sulfate. Borchers and Hüffner could not have imagined in their wildest dreams that 20 years later this process would be accepted as the standard method for processing molybdenum-containing furnace pigs from Mansfeld copper-ore smelting. Neither could they have known that H.C. Starck would one day be the “world champion” in concentrating tin slags – not only to recover tungsten, but also to obtain tantalum concentrate from slags originating from Southeast Asia.

Technological input from Altherzberg

Goslar's problems were not shared by the Altherzberg plant, since this facility used the traditional ammonium paratungstate process (APT) based on imported scheelite. In simplified terms, this production method consists of three steps:

- 1) decomposing pre-roasted scheelite with hydrochloric acid
- 2) dissolving the still contaminated (technical) tungstic acid in a watery ammonia solution
- 3) crystallizing the ammonium paratungstate.

The Altherzberg plant thus had ammonium paratungstate very early on – both in the form of a raw material for the production of tungsten metal and also for special, very pure tungstic acid. This production and the corresponding know-how were later transferred to Goslar.

For a very long time Gebr. Borchers maintained the process based on alkaline decomposition of tungsten with subsequent alkali treatment through preliminary precipitation and calcium precipitation. With a widely varied raw material mix, this process was only practicable if sufficiently large calcination and leaching capacities for treating residues and extracting byproducts of the ores, such as tin and manganese, were available.

The crowning touch to cooperative efforts, which also made use of the know-how from Altherzberg, came with the development of a combined process. Introduced in the mid 1930s, this method, which guaranteed nearly total flexibility in the use of raw materials, essentially continued to be used for many years – right up to recent times. There are four main steps to this process, whereby the last one already represents a recycling step:

- 1) decomposition of wolframites and intermediates rich in tungsten oxide in an autoclave
- 2) calcination with sodium carbonate for low percentage ores, tungsten tin mixed ores and residues; production of calcium tungstate as an intermediate through alkali treatment and calcium chloride precipitation
- 3) hydrochloric acid decomposition of highpercentage scheelites or calcium tungstate with subsequent dissolution of technical tungstic acid in ammonium and then crystallization of ammonium paratungstate with mother liquor processing
- 4) concentration of residues, scrap and production tailings.

The last processing method was designed to fit in perfectly with production steps 1 to 3.

Advances also in technology

Parallel to developments in processing technology, there were also many substantial improvements in chemical technology. These had to be taken into consideration during expansion. For example, Dr. C. Otto & Company GmbH in Bochum, which formerly belonged to Salzgitter AG, developed a new plastic based on a phenolic resin and called it "Haveg." With this new material it was possible to solve acid boiler, mixer and washer corrosion problems. For the first time ever, stainless steel evaporators, condensers, mixers and pipeline systems could be used for handling ammonia solutions, nitric acid and ammonium paratungstate – as long as it was considered acceptable in terms of costs.

Nevertheless, pitch-pine wood from the North American *Pinus rigida* pine tree (parquet yellow pine) was still the ideal material for conventional leaching equipment. The hard, tough, elastic and durable wood was particularly suitable for leaching vats, filter chambers and frames. This equipment was produced in the company's own workshops.

The infrastructure of the new plant near the Oker train station and the drainage outfall at the Oker river were virtually ideal. All kinds of raw and auxiliary materials were available via train. Chemicals supplied directly from chemical plants in central Germany were delivered in tank cars. Energy supplies were tied to the ironworks and steelworks of Salzgitter. Fresh water, an important auxiliary material for the production of sophisticated chemical products, was also available in abundance and in adequate quality. However, disadvantages have since become evident: residential developments were later built too close to the plant, and the topographical situation in an inversion basin cannot be considered ideal in light of today's requirements for air purification.

The era of tungstic acid chemistry

Tungstic acid chemistry reigned from the 1940s until the 1960s. This period was spurred by the need to develop purer intermediates with very specific properties. These intermediates would be used to obtain pure tungsten for mill products and for high-quality hardmetals and heavy metals. The crucial criteria included bulk and tap densities, as well as a specific surface. H.C. Starck was able to fulfill these exacting requirements with two completely independent product lines:

- 1) light tungstic acid: a bright yellow tungstic acid produced from crystallized sodium tungstate via calcium tungstate (Gebr. Borchers' process) and under certain conditions, precipitated with hydrochloric acid.
- 2) fine crystalline tungstic acid: this product type stems from Altherzberg and is closely tied to the foreman who was transferred to Goslar from Altherzberg, W. Boche. This process begins with ammonium paratungstate, which is converted by acid treatment to tungstic acid.

The second product line became a great economic success. In the 1960s up to 100 tons of this acid were produced every month and processed mainly to fine tungsten carbide.

The amount of equipment required for the different acid precipitations and for the subsequent washing processes was enormous in those days. For example, the first units for demineralized water were installed for the process. Freshly precipitated tungstic acids had the irksome feature that impurities stuck firmly to the large surfaces of the acids. One example, in particular, is silicic acid (SiO_2).

The difficult problem of drying the tungstic acid also took time to get under control. And the fight against corrosion was a real ordeal for employees as well. It was the era of the rubberlined reactors, mixers and pumps, which in many cases were only protected from abrasion with an acid-proof ceramic lining. The new plastic Haveg was not at all suitable for lining the large wet chemical equipment.

The year 1923 represents an important milestone in the tungsten chronology. It marks the invention of hardmetal by Schröter of the "Osram Studiengesellschaft" and the corresponding application for a patent, which was eventually granted to Krupp in Essen in 1926. This development was to have a revolutionary impact – not only on tungsten, but also on other carbide-forming metals, including cobalt. It also heralded a decisive change in application and consumption. In this case, tungsten was not used as an alloying component, but rather as a carbide in powder form in a new metallurgical application for the production of tools. Krupp chose the name "WIDIA" for this hardmetal, a trademark that was created from the German words "wie" and "Diamant," meaning "like a diamond." Tungsten carbide and hardmetal.

The initial WIDIA grades that were first marketed in 1923 were composed of 94 percent tungsten carbide and six percent cobalt as auxiliary metal and binder. In those early days, the hardmetal was used for machine tools, cutting tools and shell cores.

Today's WIDIA hardmetal is more complex in its structure and, depending on the kind of application, is often composed not only of pure tungsten carbide, but also of mixed crystals with carbides of titanium, niobium and tantalum. This results in improved oxidation resistance, hardness and heat resistance.

A prerequisite for the production of a WIDIA hardmetal was that the intermediates for the tungsten carbide that was to be sintered – and the binder cobalt – had to be very pure. The physical properties of the powder also had to fulfill particular demands. H.C. Starck was well-prepared for this new trend. The young hardmetal industry initially preferred to reduce the tungsten to metal and produce the carbide in their own plants. But soon, the calculation proved right to choose one of the pure products from the sequence ammonium paratungstate, oxide, metal and carbide.

H.C. Starck participated at Krupp and other German processing companies in hardmetal development with products from its tungsten chemical processes and with cobalt oxide and metal. Thus tungsten chemistry saw a significant upswing in the time following the worldwide economic crisis of 1929, mainly because of the hardmetal boom.

But H.C. Starck had also prepared itself for the new powder metallurgy applications. Because of increasing quality requirements over the years, the original, rather simple process of reduction of tungsten oxide with charcoal in a crucible was changed decisively, as the following milestones in the process development illustrate.

- 1) The technical tungsten oxide is replaced by the purer ammonium paratungstate.
- 2) Pitch is used in the reduction crucible instead of charcoal. Unlike charcoal, pitch could be ground and mixed with ammonium paratungstate in an empirically calculated weight ratio. It can also be added to the crucible in a relatively dense packaging.
- 2) "Schmidt's circular furnace" takes the place of the former double-chamber furnace with coaltar oil heating. The circular furnace has an optimum draft ratio and therefore, a nearly ideal temperature distribution – an important prerequisite for a high metal powder yield of good quality.

A furnace shrouded in mystery

Although the circular furnace process would be considered labor-intensive from today's point of view, it was nearly ideal back then. While the output of up to two tons of metal during the life of the furnace was enormous, costs for reducing agents and energy were low.

Quality was reproducible and could be modified, for example by selecting different types of pitch. The capacity of the facility was supposed to be increased to 70 tons per month between 1937 and 1940. But in actuality, it only ran at a monthly maximum capacity of 45 tons.

In those days, the circular furnace process was one of the bestkept secrets at H.C. Starck. In addition to metal powders, carbides could be produced in the same furnace – although with different crucible equipment. Named by the former plant manager Berthold Stinsky, the metal and carbide powders were designated with the abbreviation ST – naturally as a reminder of the ST in HCST – to distinguish them from the subsequently developed hydrogen-reduced HC grades. Watched distrustfully by competitors, the ST 250 grade gained worldwide success and acceptance.

Regular production of ST grades was halted in the early 1980s, but only after the continuously running hydrogen reduction in large multitube furnaces achieved equally high outputs at comparable costs with improved quality.

In the many years before the unit was shut down, 70 to 100 tons of metal were produced in the circular furnace every month. The qualitative peak was a metal powder called ST 100, which had an average size of one micron. The quantitative peak of the “spikes boom” came in the years 1969/1970.

Tungsten raw materials and their processing

Ores of many types and qualities were used in the more than 90-year history of tungsten in Goslar. This variety of raw materials was one of the main reasons why the company chose to introduce the most universal processing program possible. Ores were supplied from the following regions, which are listed according to their historical importance:

- 1) tungsten tin ores from Cornwall in the U.K.
- 2) mixed ores and tin slags from the Erz Mountains
- 3) often highly intergrown wolframite and scheelite from Spain and Portugal.

After the trade routes reopened in 1947 and 1948, ore supplies also came from Central and South America, particularly Bolivia and Peru. Canada, but above all Burma and Thailand, also played a major role. In more recent years, China has become enormously important.

Actually, this could be considered a revival of an old business relationship, although under completely different circumstances: in the 1920s Starck purchased ore concentrates through English trading companies from mines in southern China that had first been opened during World War I. For a limited time there was also a kind of “division of labor” in that H.C. Starck took over the tungsten part of tungsten-tin mixed ores that had been pre-separated or chemically pretreated in England or Holland.

Hermann C. Starck participated in a German-Chinese trade agreement on the delivery of tungsten ores in the mid-1930s. However, China turned out to be an unreliable partner at that time. Starck ultimately solved the problem by drafting a separate contract which expanded on existing complicated international provisions for the evaluation of tungsten deliveries. The second agreement, known as "Contract B," provided for sampling of Chinese ores at the port of arrival.

There is an interesting passage in a book by K.C. Li entitled, "Tungsten," Third Edition 1956, about Hermann C. Starck's role in stocking tungsten raw materials before the outbreak of World War II. Among other things, he wrote that the Purchase Pool in Berlin, which was under the direction of the "very capable tungsten expert, Hermann C. Starck," bought up practically the entire yield of South America's off-grade qualities at a substantial discount.

It is very doubtful whether a deal of this magnitude ever really took place. However, the thought of having this kind of ore as the only raw material for processing must have caused the tungsten chemists many a sleepless night.

It is a proven fact that by this time Hermann C. Starck had gained a worldwide reputation as an expert for tungsten raw materials, especially for low-percentage material. This fame earned him the nickname, "Mister Tungsten."

It is also true that he was very interested in the question of whether these so-called off-grade concentrates actually stemmed from ores with a large number of intergrown gangue or were just poorly separated at the mines because of inadequate technology. Magnet separation to enrich the wolframite fraction and to separate cassiterite was thus one of the first industrial activities of the company.

Hermann C. Starck himself arranged to conduct the necessary tests with the so-called "Ulrich circular separator" at Krupp in Magdeburg and acquired one of these separators for the tungsten department of his company.

In cooperation with the Mining Academy in Clausthal, Germany, the company subsequently performed concentration tests, which eventually led to the installation of a special ore concentration facility in Goslar.

The core part of this ore concentration facility was a high-performance Starkfeld dry magnet separation, which was ultimately equipped with two Krupp circular separators (1953), a cross-belt separator (1970) and a double circular separator (1976). A wet magnet separator was added to the facility in 1980. With this technology the separation of very fine-grained ore sludge was made possible for the first time ever.

Parallel to these developments, completion of a number of other technologies was stepped up, including

- gravity sorting by means of quick percussion tables and Humphrey separators in 1968
- the introduction of the flotation process for sulfides in 1978, and
- the introduction of the flotation and electrostatic processes for scheelite.

Processing residues and scrap

Because of the serious shortage of raw materials during World War I, Gebr. Borchers had already begun to process ironwork tailings containing tungsten (tungsten hammer scale). These tailings, which contained a lot of metallic iron and only about five percent tungsten, were decomposed by an alkaline-oxidizing treatment. It soon became clear that a roasting process similar to the one for chrome iron ore would be the most practical. The resulting tungsten liquors were diluted and processed by precipitation of calcium tungstate. In the 1930s, H.C. Starck was faced with a large number of problems related to the reconcentration of the first hardmetal scrap.

The first dilemma was the question of crushing and oxidation. To overcome this, Gebr. Borchers developed a process that roasted the hardmetal pieces and then ground the oxide layers in heavy ball mills on an alternating basis. By comparison, powder scrap could be directly oxidized.

Hardmetal oxide mixtures of tungsten, cobalt, titanium and tantalum/ niobium – and including other residual metals and carbides – were an extremely high-quality raw material for autoclave decomposition.

By comparison, isolating such useful materials as cobalt/nickel and tantalum/niobium from the dissolved residues of the autoclave, and removing residual tungsten, which required calcination with sodium carbonate, proved to be more difficult.

The residues from processing heavy metals and contact materials were relatively problem free. Worst of all – even for the experts – was and is processing hardmetal grinding residues that have a high silicon and boron carbide content.

Postwar years and a new beginning

Similar to the years during the First World War, work conditions were difficult in the early 1940s. A continuous development of the tungsten business in those days was out of the question. The end of the war, the capitulation and the collapse of the Third Reich represented an important turning point in the development of the company.

Difficult times for Hermann C. Starck

The era of the Third Reich and the years immediately after World War II were an extremely difficult time for Hermann C. Starck personally. Following the seizure of power by the Nazis, many of Starck's friends had to flee Germany. He was left with a permanent sense of fear for his family. Like many of his contemporaries, Starck had to fight to protect his two children, Barbara and Gerhard, because his wife, Klara, was Jewish.

These circumstances did not, however, prevent Hermann C. Starck from hiring other people who were oppressed by the Nazi regime. In October 1936 Dr. Hans Adelung joined the company because he was not able to continue his education as a lawyer for political reasons.

Another example was the 20-year-old Hellwarth Lahusen, who began his career at H.C. Starck as a commercial clerk in February 1940. Lahusen was lucky that he was largely able to escape persecution by the Gestapo through his constant travels between Berlin and Goslar.

When within two days in November 1943, the “Romanische Haus” office building and his house in the Bellevue Street in Berlin were destroyed in bombings. Hermann C. Starck thus transferred his offices complete with the remaining employees to his private home in Potsdam.

In June 1945 when the Soviet army marched into Germany, Hermann C. Starck was arrested in his home in Potsdam. He was first detained at the internment camp, Fünfeichen, in Brandenburg, but was then moved to the former Nazi concentration camp at Buchenwald. In retrospect, Starck’s mistake was not to have fled from the influence of the Soviet military government. He apparently had too much confidence in the fairness of the occupation forces, since not only had he remained politically neutral during the Nazi period, he had also experienced difficulties himself. As it turned out, he miscalculated the situation.

As an industrialist who had worked in the armaments industry and employed foreign workers during the war, he was classified as a “war criminal” by the military government – with bitter consequences: Together with some 3,000 other prisoners, Starck was turned over to East German jurisdiction in 1950 and sentenced at the Waldheim trial to 20 years imprisonment at Bautzen for “supporting Nazism,” and all of his assets and property were confiscated.

From the beginning of his imprisonment Hermann C. Starck had made sure that his company would not be left without proper management, even temporarily. In a secret message smuggled out of the Fünfeichen camp, he entrusted general power of attorney to his close friend and legal advisor of many years, Dr. Wilhelm Königswarter. The paper was immediately recognized by the subsequent shareholders’ meeting. During Starck’s internment, Königswarter secured the continuation of the company and took care of the family.

After the currency reform, when the increasing importance of the Rhine/Ruhr area became clear, the office in Düsseldorf was founded on the initiative of Dr. Hans Adelung, Hellwarth Lahusen and Walter Stolle. During Starck’s absence, Dr. Adelung and Lahusen acted as joint-managing directors and saw to it that the business was successfully continued according to Starck’s wishes.

In the end, Starck got lucky: Through a declaration by the Saxon ministry of February 22, 1951, of the 13 particularly spectacular cases Starck and one other prisoner were granted a pardon. He was released from prison on February 24, 1951.

It is very unfortunate that Hermann C. Starck did not live to see the illegality of his conviction officially confirmed. The District Court of Dresden established the invalidity of the June 6, 1950 decision of the Chemnitz-Waldheim regional court on October 28, 1991 – long after his death. The original conviction was thus finally nullified.

The suffering during imprisonment had left its mark on Hermann C. Starck’s health, but mentally and emotionally, he was unbroken. Immediately following his release, Starck devoted himself to the reconstruction of his company, which, thanks to the dedication and hard work of his loyal employees, had developed successfully in his absence.

Adjusting to the new situation

In 1952, Hermann C. Starck sent Hellwarth Lahusen to the United States to reestablish contact with the former business associates. Starck himself later traveled to the States and to Japan several times. He expanded the Düsseldorf office that had been founded in his absence and set up an apartment in the two upper floors of the building. Karl Ludwig Frege, whom Starck had hired in Berlin in 1955, transferred to Düsseldorf as his personal assistant.

For the Goslar plant, a new beginning was made possible through the understanding and fairness of the British military government, which had already started granting limited production permits – also for hardmetal intermediates – as early as 1946. This permission offered the company the opportunity to process the intermediates and residues that had accumulated in great quantities during the last years of the war.

But in the meantime the tungsten situation had also changed. The heyday of the large scheelite mines had now emerged which, with their own processing facilities, had to some degree even outmatched the importance of the traditional wolframite deposits in Bolivia and Portugal. Faced with competition from Western mines equipped with modern technology, China was in a difficult position and was suffering from political difficulties as well.

Important impulses for the further development of extractive metallurgy came in those days from the United States. The possibilities offered by the use of the newly developed solvent extraction process for reducing impurities in ammonium paratungstate were particularly interesting to H.C. Starck. Among other things, a patent for the production of an ammonium paratungstate of improved quality resulted. In addition, ammonium metatungstate was produced both in the form of a solution and also in a crystallized state. This substance is an important intermediate for catalysts containing tungsten. Their development was decisively spurred by this new process.

Reducing energy and raw material costs

As a consequence of radically increased energy and raw material prices in the 1970s, previous methods of raw material processing had to be replaced with new strategic concepts. The main emphasis of the corresponding development work was on avoiding dust losses, and saving and recovering energy. Economic pressure caused by high prices prompted major simplification of the processes and corresponding reductions in costs. For example, the autoclave decomposition facility was expanded in a way that calcination with sodium carbonate could be replaced by pressure leaching with a soda solution.

Environmental protection efforts benefited from these innovations as well. For example, less dust resulted by changing the processing of raw material to a hydrometallurgical decomposition process. A number of scrubbers and filter units were installed at other locations in the plant. They were soon amortized because of the recovered raw materials.

Under the direction of the tungsten chemical plant manager at that time, Dr. Wolfgang Müller, H.C. Starck initiated an extensive conversion of the alkali purification process to solvent extraction and developed a special processing method adjusted to the needs of the Goslar plant. This meant an expansion of the autoclave capacity with the aim of less corrosive alkaline decompositions.

The preliminary precipitation was also improved: new filtration techniques were introduced, as were modern testing and control methods. In this way a far more constant level of production and quality was achieved, despite the constantly changing raw materials. The quality requirements of customers, for example in the wire industry, could be fulfilled without any special guarantee in respect to the origin of the starting materials.

There were also remarkable innovations in the reduction and carburization technologies of postwar times. Although in former times H.C. Starck had been accustomed to working solely with crucible furnaces, development of electrically heated furnaces for the hydrogen reduction of tungsten was accelerated in 1950.

Working in close cooperation with the company Elino of Düren, H.C. Starck was able to introduce increasingly more efficient furnaces in the tungsten production facilities at the Goslar plant right up into the 1970s.

Higher and higher demands on quality

This was also a time of significant advances in the improvement of metal powder grades. The following developments contributed to the results:

- 1) choosing the optimum starting material (acid hydrate, yellow oxide, blue oxide, APT)
- 2) varying process parameters.

One should mention at this point that the entire range of grain size distributions from 0.3 to 150 micrometers was supposed to be covered in the product program. The grades at the upper and lower ends of this spectrum were a particular strength of the company at that time.

By 1968 reduction capacities had also been set up at the Elektro-Nitrum (ENAG) plant (see also the chapter on the history of Laufenburg). The guarantee of production through a second facility, a lower price for electricity, and water supplies were decisive factors in taking this step.

In the 1970s H.C. Starck introduced for certain qualities the highly efficient, natural gas heated multitube pusher furnace preferred in the United States. The reduction capacities are set up so that up to three quarters of the tungsten chemical production can be reduced internally, and today can also be delivered to a large degree as carbides.

The development of carbide technology at H.C. Starck had a different background than other suppliers of hardmetal intermediates. The first efforts took place under the pressure of not only having to be involved in the carburization of tungsten, but also of tantalum, niobium, titanium, chromium, molybdenum and vanadium. This included the production of various mixed carbides as well.

Carburization first took place in crucible furnaces. By 1950 induction heated graphite crucibles – also called open coils – with a carbon granule covering became available. Shortly thereafter, such carburization was performed for the first time under a vacuum.

The next technological advance came in 1968 when large capacities for the carburization of tungsten were made available. Conversion took place in small covered graphite boatlike vessels in furnaces that were equipped with molybdenum heating elements.

Ceramic lined furnaces that could be charged with hydrogen were available as reaction space. By comparison, resistance-heated graphite tube furnaces were used for particularly high-temperature carburization processes – especially for coarser carbide grades. H.C. Starck made decisive improvements to this technology, which the company had developed itself.

During this period advances were also made in the development of particularly fine carbide grades in the submicron range.

The customer as a stimulus for innovation

Developments like these always reflected the needs of the market. For example, fine-grained hardmetals were required for tools that had to be extremely hard and have sharp cutting edges, such as the microdrills used to make components for electronics or in dental technology.

Quality requirements in modern microelectronics are particularly high. For example, an individual hardmetal drill has to bore some 10,000 exact holes when producing circuit boards – plastic sheets with printed circuit diagrams. These thin tool tips with diameters of as little as 0.2 mm can break easily. This is especially true of sensitive cutting edges. A flaw in the structure because of an impurity or a coarser grain is often the cause of a break.

By comparison, hardmetals that are used for coal-mining chisels have to be as tough as possible and extremely wear resistant. A coarse-grained carbide, in which fine particles would spoil the effect, is required for this application. Fine particles decrease toughness, and the chisel would not last long. The same is true of mining drills. Bore crowns with hardmetal inserts are used for deep mining in relatively soft rock, whereas diamond-studded bore crowns, primarily made from cast tungsten carbide and a binder consisting of various copper alloys, are used for hard rock.

There was a special development in the Laufenburg plant which resulted from the fact that H.C. Starck had already begun supplying defined tungsten metal grades for the production of an eutectic mixture made of ditungsten carbide and tungsten carbide in the 1950s. The oil industry in the United States was a very large market for this product.

Back then, the Elektro-Nitrum facility in Laufenburg succeeded in varying the melting and cooling conditions to such a degree that the desired fine-grained structure of the carbide was preserved. This clever adjustment made it possible to use the product for more demanding applications in the field of hardfacing. The cast carbides represented an important supplement to the powder varieties based on tungsten carbide that H.C. Starck had developed for hard-facing and flame and plasma spraying.

Keeping abreast of hardmetal technology

As H.C. Starck started to establish itself as a major supplier of carbides for the hardmetal industry, the accompanying hardmetal development soon became a dominating factor. Obviously, there was a great temptation to become a hardmetal producer and thus to complete the cycle from raw material to final product. However, H.C. Starck resisted this temptation. The company was going to abide by its philosophy not to compete with its own customers in the hardmetal industry. In other words, H.C. Starck's product program ended with carbide powders and cobalt metal powders. Even hardmetal mixtures have been and still are only produced at the company for its own requirements in producing thermal spray powders.

Despite this decision, it was and is essential for H.C. Starck to keep abreast of modern technology in hardmetal production. This commitment is necessary in order to adjust the intermediate grades to customers' needs and at the same time, guarantee a consistency in quality.

After encouraging results with a simple hardmetal test, a central laboratory for hard materials was set up that contained all the equipment necessary to produce hardmetal composites for molding and sintering and also to test sample objects. For this purpose, several suitable production and test systems had to be developed and kept in balance with the interests of the large processing companies, the different scientific opinions and the realities of powder production.

The development of quality is a process that requires permanent innovation. A supplier dedicated to fulfilling customers demands must regularly take part in technical discussions. New product grades are the result of such challenges. These are the kinds of products that first made it possible for H.C. Starck to penetrate such competitive markets as the United States and Japan. The information that emerges from discussions with customers is valuable capital that, also in light of the company's own interests, should be treated confidentially.

Every challenge spurs new qualities

The prerequisites for quality improvements were expanded substantially in 1985 and 1986. This was made possible through the installation of a furnace pilot plant in which smallscale test production was performed. Such a project obviously involved costs. Yet the expansion offered the company two major advantages:

- 1) Before the furnace pilot plant went on stream, a number of development projects previously had to be interrupted because of the high capacity utilization of the facility. Production and punctual delivery to customers had priority.
- 2) Accommodating test products that did not always fit into the production scheme was no longer a problem because much smaller quantities were required.

The quality demanded of a product, especially tungsten products for powder metallurgy, is coming closer and closer to the requirements of high-tech industries. For H.C. Starck this was a further impetus to supplement the already existing quality control system. The result is a comprehensive quality assurance system that functions completely independently of production.

For certain customers, mainly in the aircraft industry, such systems were successfully introduced at the end of the 1970s. These had organizational consequences in the tungsten and tantalum activities in 1986. This reorganization aimed to adjust production methods to meet the requirements of particularly demanding customers and to structure the entire production process according to quality assurance standards.

The successes achieved so far are impressive. In tungsten production, H.C. Starck is proud to be able to serve loyal customers who place particularly high demands on quality assurance.