Heraeus

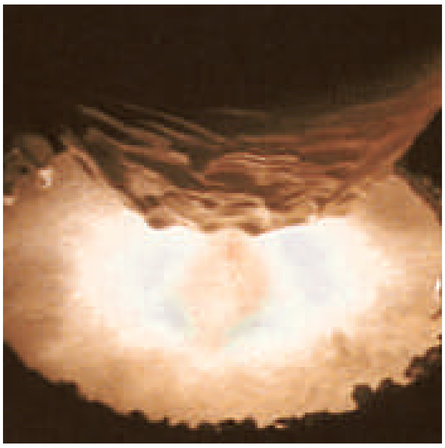
Precision in Special Metals

Materials
Technology Division

Product Line
Precision Technology

In 1856 the pharmacist and chemist Wilhelm Carl Heraeus became the first person to succeed in melting platinum jewellery scrap in industrially interesting quantities using an oxygen-hydrogen burner. He founded the present company W.C. Heraeus GmbH in Hanau. Since then all precious metals and the special metals niobium, tantalum, titanium and their alloys have played a central part in the company's activities.

Since that time Heraeus has always been at the forefront of innovation, transforming unusual technologies into viable industrial processes, thereby becoming a competent partner not only for the chemical and pharmaceutical industries but also for the electronics, electrical and aerospace industries. Heraeus is also active in science, research and medicine.



Melting niobium under high vacuum in the electron beam furnace at a temperature of approx. 2600°C

In the Materials Technology Division we manufacture, fabricate and machine valuable materials for technological applications. Many of these materials require very complex processing.

The central competence of the Division lies in the industrial application and technological usage of noble and special metals. Our demanding products find application in many different industries and in research centres throughout the world.

Pioneer in special metals technology

Heraeus started sintering tantalum powder in the late 1940's and in 1955 began melting titanium and zirconium in the vacuum arc furnace. Electron beam melting of tantalum dates from 1959 and that of niobium from 1960.

The melting capacity for tantalum and niobium was markedly increased in 1984 when Europe's largest electron beam furnace was taken into service.

Niobium crystallized from the melt

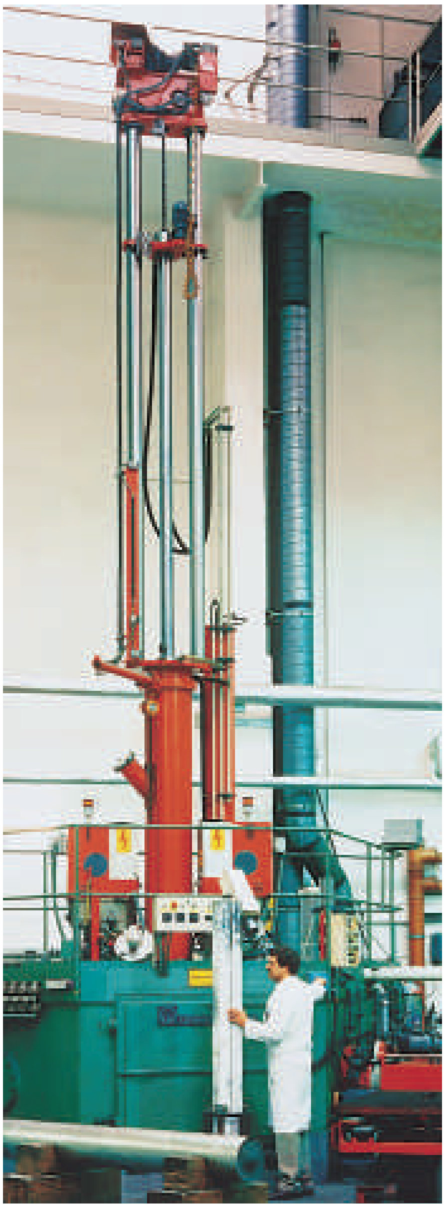
Close co-operation and constant, intensive dialogue with our customers provide the basis for the overall success of our efforts.

If we know our customer's wishes and the precise product requirements we can supply technically and economically optimized products to meet the required specifications.
After intensive consultation with the customer we design the product from the characterization and manufacture of the precursor material to the selection of suitable process parameters.

We have acquired a well-founded and comprehensive knowledge of high purity metals and special materials, their properties and possible process technologies. This is a result of many years experience in the manufacture and treatment of these materials and our close co-operation with international research institutes and standards committees.

This enables us to select optimally suited production methods and to develop special analysis processes.

Everything in one hand: from the primary production of the metals, through design and manufacture to the inspection and analysis of the finished product. That is the greatest advantage of working with Heraeus.



Europe's largest electron beam furnance

Above: CAD work-station
Centre: Continuous process control
Below: Scanning electron microscope with energy and wave-length dispersive X-ray analysis

Melting technology

Induction furnaces for melting precious metals. Electron beam furnaces for melting niobium, tantalum and their alloys. Vacuum arc furnaces for melting special alloys.

CAD design and tool-making

Basis for highest production technology

Machining

Turning and milling on CNC equipment, spark and wire electric discharge machining, precision drilling and honing, precision grinding.

Forming

Drawing, rolling, swaging, spinning, impact extrusion, deep drawing, stamping, cladding and roll cladding, hydro-mechanical forming

Finishing

Heating-treatment and surface treatment

Gas analysis

Mass spectrometer for determining degassing temperatures

Hydrogen, nitrogen, oxygen and carbon analyses for high melting materials

Surface analysis

Scanning Auger microanalysis and secondary ion mass-spectroscopy

C/H analyzer for determining surface coverage

Physical properties

Measurement of electrical resistance including temperature coefficients and thermal conductivity

Non-destructive testing

Ultrasonic testsing

Mechanical testing

Hardness, tensile, compressive and bending strength

Cyrogenic laboratory

Measurements of physical properties at temperatures down to approx. 4.2 K

Determination of important material properties such as residual resistivity ratio and thermal conductivity as a measure of interstitially and substitutionally dissolved impurity atoms in the metal lattice (integrated process from the melting of the raw materials to the production of the precision parts).

Precision parts are products that are manufactured by forming and/or machining and display a high level of quality and application technology.

Our precision parts of special materials are distinguished by their specific properties:

- ☐ Precise dimensional tolerances
- ☐ High surface quality
- ☐ Closely specified mechanical properties
- ☐ Tightly specified metallurgical features
- ☐ High resistance to corrosion and thermal oxidation
- ☐ High chemical stability
- ☐ Excellent biocompatibility
- ☐ High reproducibility

The Certificate according to international standard DIN EN ISO 9001 was awarded to the quality management of the Materials Technology Division in 1994.

This standard guarantees not only product quality but also ensures the documentation of all work steps and all production processes from the beginning of the planning to the dispatch to the customer.

Furthermore the additional standard requirements of QS 9000 were installed and applied in our company in December 1997.

TÜV CERT

CERTIFICATE

The TÜV CERT Certification Body
of TÜV Management Service GmbH

certifies in accordance
with TÜV CERT procedures that

W.C. Heraeus GmbH
Divisions Materials Technology and Electronic Materials
D-63450 Hanau

has established
and applies a quality system for

Precious Metals Technology, Semi-Finished Products, Medicine Technology, Plastic Metal Composites, Precision Technology, Thin Film Technology, Packaging Materials

An audit was performed, Report No. 24022261

Proof has been furnished that the requirements according to

DIN EN ISO 9001 : 1994

are fulfilled. The certificate is valid until Dezember 2000

Certificate Registration No. 12 100 6497

Deutscher Akkreditierungs Rat
TGA-ZM-18-96-00

Munich, March 05, 1998

TÜV
MANAGEMENT SERVICE

TÜV-CERT Certification Body
of TÜV Management Service GmbH
Unternehmensgruppe TÜV Süddeutschland

Due to their high resistance to temperature and corrosion, a wide variety of precision parts made from special and precious metals such as tantalum, titanium, niobium and platinum-tungsten find particular application in lighting technology. Heraeus produces a wide variety of customer-specific components and offers tailor-made technologies.

High pressure sodium vapour lamps

In this field of application a wide variety of small tubes and caps made from a niobium 1%-zirconium alloy have proved particularly successful. They are used world-wide in high pressure sodium vapour lamps.

The high pressure sodium vapour lamps, which were developed around 1960, have a broader spectral distribution than the older low pressure sodium vapour lamps with their dull, yellow light of essentially one single wave length. For this reason they have a pleasant, warm, whitish-golden glow.

High pressure sodium vapour lamps are excellent energy-saving lamps with a long service life (approx. 25,000 hours) and an especially high degree of efficiency. They produce five times as much light (approx. 160 lumen per watt) as conventional lamps for the same energy consumption.

A gas discharge is necessary to create light in the high pressure sodium vapour lamps. The discharge is induced between two electrodes in a thin, ceramic tube, the discharge tube, which is to be found inside the lamp's glass body. The electrically conducting feed-throughs for these electrodes are made of niobium. The advantages of the niobium 1%-zirconium alloy are: the excellent high temperature resistance to liquid and vaporized sodium which is present in the discharge tube, and the very favourable expansion coefficient for ensuring a reliable seal to the lamp's alumina ceramic tube.

Niobium 1%-Zirconium tubes and closed caps

Heraeus precision parts are largely produced using forming processes, for instance, deep drawing and straight tube-drawing, impact extrusion, stamping and upsetting.

One of the properties of niobium which causes problems is its tendency to galling or cold welding with other metals, for instance with tools during forming operations.

This poses particular technological demands on the forming process. Heraeus has therefore developed appropriate processes for use during manufacture so that the exceptional properties of these metals can be fully utilized in special areas of application.

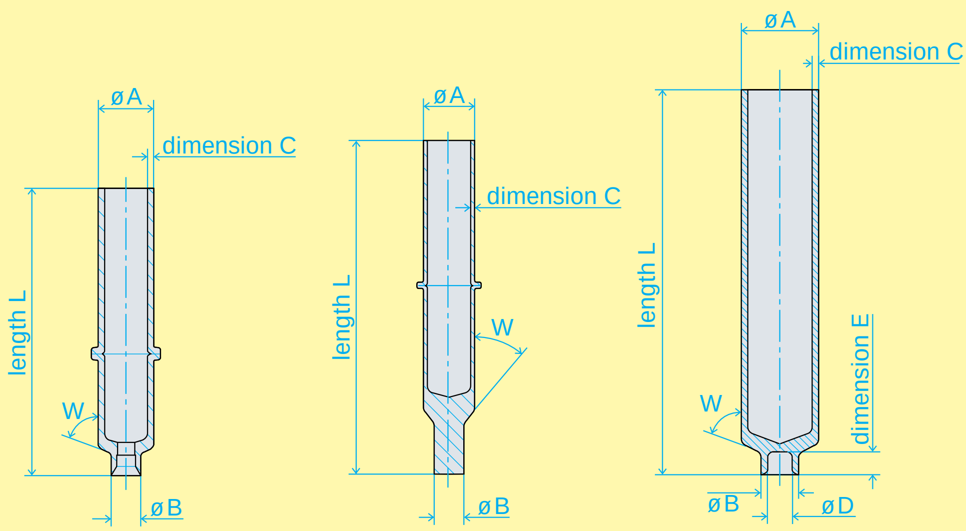
The extremely tight dimensional tolerances and the very high surface quality which are achieved when manufacturing the precision parts are the most important prerequisites for optimum joining elements.

The following dimensional ranges are available:

- ☐ External diameter ≤ 5 mm
- ☐ Length ≤ 60 mm
- ☐ Wall thickness from 0.1 mm to 0.5 mm (according to customer requirement)

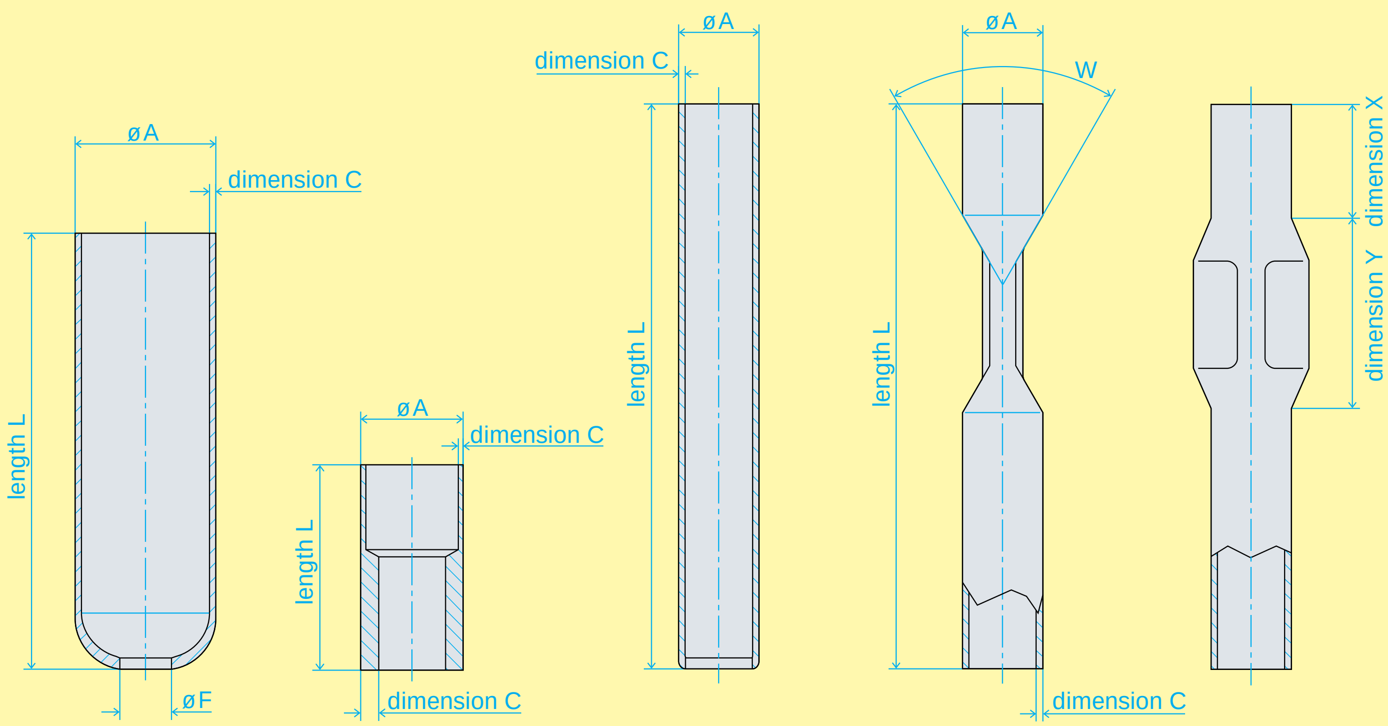
Typical dimensions of niobium 1%-zirconium precision parts

øA:	from 2 - 4 mm
øB:	max. 0.4 x øA
dimension C:	0.2 - 0.4 mm
øD:	according to customer requirement
dimension E:	max. 1.2 x øD
øF:	according to customer requirement
length L:	according to customer requirement
angle W:	40° - 70°
dimension X:	according to customer requirement
dimension Y:	according to customer requirement



- ☐ Pure niobium and niobium 1%-zirconium wires with closely defined surfaces and tolerances
- ☐ Pure niobium and niobium 1%-zirconium cut wires or bent parts, e.g. for halogen metal-vapour lamps
- ☐ Cut wires and formed pins of nickel and other materials, with and without surface coating
- ☐ Formed precision parts of special and precious metals
- ☐ Precious metal alloy wires, e.g. platinum-tungsten
- ☐ Coated expanded metal
- ☐ Precious metal foils and strips
- ☐ Sputter targets for thin film technology
- ☐ Ceramic colours
- ☐ Precious metal recycling
- ☐ Surface coating of parts supplied by customer
- ☐ Close co-operation during the development of new materials and technologies

Halogen metal-vapour lamp
with niobium wires



Due to their excellent stability precision tubes made of special and precious metals are used in a wide range of applications.

Above all the following properties deserve special mention: their resistance to various aqueous solutions and acids, and to metal melts and vapours, their high temperature stability and mechanical strength, their good corrosion properties and last, but not least, their biocompatibility.

Our strength lies in innovation. Wherever the greatest demands are made on special and precious metal fine tubes, Heraeus is your partner. Heraeus with its special manufacturing processes, its progressive technology and its comprehensive experience will answer your questions and advise you.

Areas of application for fine tubes

- ☐ Medical technology
- ☐ Analysis techniques
- ☐ Pharmacy
- ☐ Lighting technology
- ☐ Measurement technology
- ☐ Electronics
- ☐ Electrical engineering
- ☐ Laboratory equipment

Dimensions

External diameter approx. ≥ 0.5 mm

Internal diameter approx. ≥ 0.2 mm

Length up to approx. 4 m

Typical materials

Tantalum, titanium, titanium 0.2%-palladium, niobium 1%-zirconium and precious metals

Products forms

Seamless drawn tubes

Cut lengths of tubes

Needles with turned or ground points

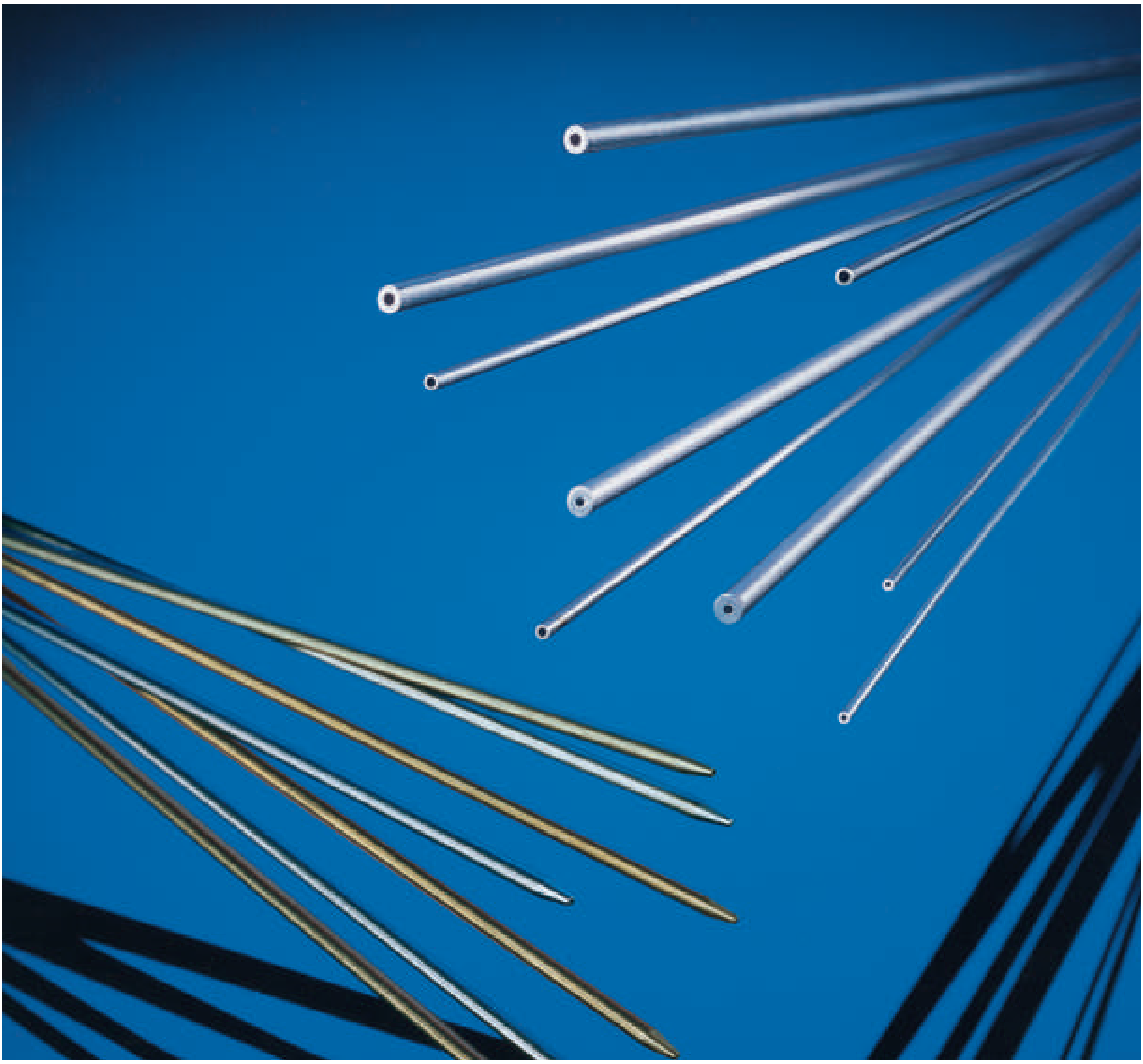
Tube-in-tube products/composite tubes

Precision turned parts on the basis of fine tubes

Upset tubes

Special forms to customer specification

Fine tubes of special and precious metals



In 1911 Heike Kamerlingh-Onnes at the University of Leiden, Holland, discovered the phenomenon of superconductivity in metals. Superconductivity is the abrupt disappearance of electrical resistance at temperatures close to absolute zero (–273°C).

The wide use of this technology became possible once the so-called hard superconductors, i.e. those which are still superconducting even at high currents and in strong magnetic fields, had been developed. In the meantime, superconductivity has been confirmed in more than 30 metals and approx. 2000 alloys and compounds. The metal niobium in its purest form has proved to be particularly suitable for technological applications.

A special quality of niobium metal with very high thermal conductivity is used in the construction of so-called cavities. For fundamental research, charged particles (protons, electrons, positrons) are accelerated by means of these cavities in accelerators to speeds approaching the speed of light.

The high thermal conductivity of the niobium is achieved by a marked reduction in the contents of interstitially dissolved non-metals (C, O, H, N) and by defined contents of metallic elements. This is accomplished by melting the material in the electron beam furnace under high vacuum at temperatures around 2500°C.

A measure of the purity is the RRR (Residual Resistivity Ratio). This is the ratio of the resistance at room temperature to the resistance at the critical temperature. The higher this value, the better the quality of the niobium. The RRR value for standard qualities of niobium is in the range 20 to 40.

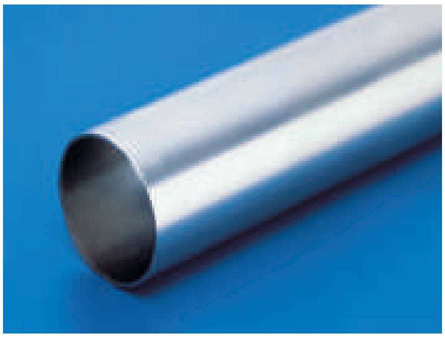
The Product Line Precision Technology of W.C. Heraeus GmbH melts high purity grades of niobium with RRR values of 300 and higher. Various types of semi-products are produced from the melted and refined ingots to meet customers' wishes.

Semi-product forms:

- ☐ For high frequency superconductors: Sheets, discs, plates, rods and seamless tubes
- ☐ As a precursor for the manufacture of superconducting cables: Rods, seamless tubes, foils and strips
- ☐ As a precursor for the manufacture of niobium alloys: Ingots and slabs

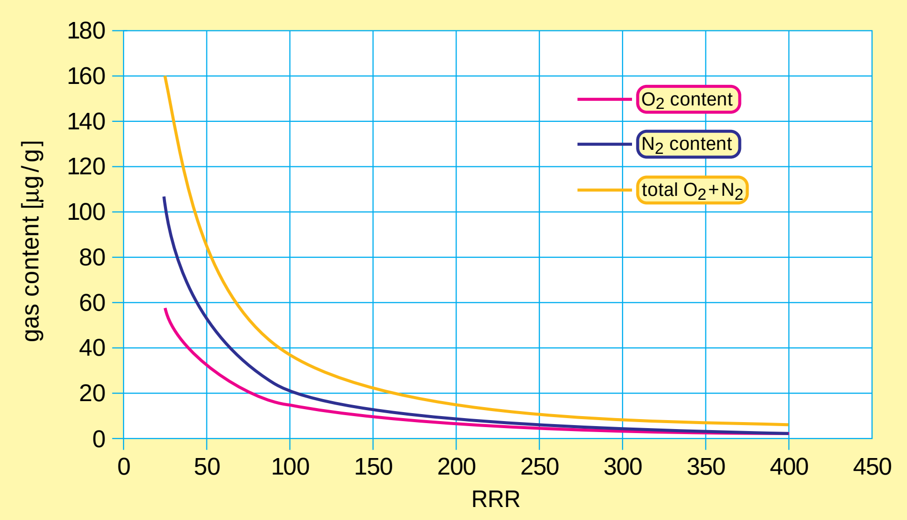
Superconducting cavities are normally produced from niobium sheets by welding. Seamless tubes with an external diameter up to 150 mm or larger are intended for an alternative, more economical production technology.

Heraeus has developed a process for the seamless manufacture of large dimensioned tubes of special metals. One of the most important properties of such tubes is the high degree of plastic deformation which can be attained during the subsequent forming steps in manufacturing the cavity. The original properties of the starting material – the high purity and a high residual resistivity ratio – should not be detrimentally affected by this processing.



Large dimensioned niobium tube, manufactured without seams

Cavity of pure niobium



Residual resistivity ratio in niobium samples dependent on N_2 and O_2 content

1801	The English chemist Charles Hatchett discovers the element “columbium”. He obtained the ore from New England, USA.
1844	The German Heinrich Rose separates columbium from tantalum and, thinking that he has discovered a new element, names it “niobium”.
1925	First applications of niobium as a substitute for tungsten in the hardening of tool steel.
1940	Manufacture of superalloys, e.g. for gas turbines.
1948	The International Union of Chemists accepts the element name niobium. Metallurgists from the USA, Canada and Mexico, however, still use the name columbium.
1953	The pioneers Becket and Franks investigate the influence of niobium on the increase in strength of steel containing carbon.
1957	Heraeus develops electron beam melting – a key process in the production of special metals such as niobium and tantalum.
1958	The Araxa deposit is discovered in Brazil. The ore is particularly rich in niobium.
1960	Niobium manufacture begins on a large scale. Niobium becomes essential in steel production. World-wide some 15,000 tons of niobium are now consumed annually. That is approx. 85% of the entire niobium production.
Today	Today niobium 1%-zirconium products for lighting technology are standard products of the W.C. Heraeus Product Line Precision Technology.

material	melting point / melting range [°C]	density [g/cm³]	linear expansion coefficient (20-100°C) 10^{-6} [K^{-1}]	thermal conductivity at 20°C [$Wm^{-1}K^{-1}$]	yield point [MPa] annealed	yield point [MPa] hard	tensile strength [MPa] annealed	tensile strength [MPa] hard	tensile elongation [%] annealed	tensile elongation [%] hard	Vickers hardness annealed	Vickers hardness hard	Young's modulus [GPa]
Nb	2470	8.57	7.1	55	75	105	170	205	35	25	80	120	105
Nb-1%-Zr	2407	8.55	7.5	42	130	–	240	400	25	10	120	150	100
Ta	3000	16.60	6.5	54	140	–	210	–	40	20	90	140	185
Ti, Grade 1	1668	4.50	8.3	22	170	–	240	–	20	–	120	–	105

All figures are non-binding values for guidance

Material properties

Tantalum, niobium and titanium are ductile metals. They tend to absorb gases so that they can only be used in a limited temperature range in spite of their relatively high melting temperatures. Hydrogen, especially atomic hydrogen, is particularly dangerous because it tends to cause rapid embrittlement even at low temperatures. The risk of embrittlement at high temperatures does not arise in pure noble gas atmospheres or under high vacuum.

Areas of application

- ☐ Lighting technology
- ☐ Electrical engineering
- ☐ Superconducting cable
- ☐ High frequency superconducting
- ☐ Medical technology
- ☐ Analysis
- ☐ Chemical equipment construction

Delivery forms

- ☐ Foils, wires, tubes
- ☐ Caps, rounds
- ☐ Formed parts according to drawings
- ☐ Stamped parts
- ☐ Boats

Chemical and physical properties

The transition metals and their alloys display exceptionally good resistance to corrosion in aqueous solutions, metal melts and vapours. The aqueous corrosion resistance is based on the formation of a stable, dense, strongly adhesive and self regenerating oxide film.

Tantalum, along with the precious metals, belongs to the most corrosion resistant metals and is only attacked significantly by hot concentrated sulphuric acid over 200°C, hot caustic soda and hydrofluoric acid. Tantalum displays the best corrosion resistance in both oxidizing and reducing acids and also in the presence of chloride ions. It also has good high temperature strength and excellent biocompatibility.

The metals have a low resistance to oxidation in air at elevated temperatures.

The very favourable strength/density ratio of titanium and its alloys makes the material interesting for various areas of application.

Titanium's good resistance to organic substances has secured areas of application for it in the food industry. In medical technology titanium is used as a material for prostheses, bone nails and implants.

Niobium is resistant to metals such as Hg, Na and K in liquid form and in the vapour phase. The linear expansion coefficient lies close to that of many ceramic materials.

Crystallized niobium melt

Heraeus

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