

High Performance Metal Solutions

High Performance Solutions for the Synthetic Diamond Industry

H.C.Starck 

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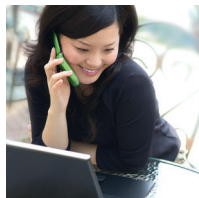
Our Commitment to Engineering Excellence

H.C. Starck Solutions' complex product fabrication capability delivers superior quality with material consistency and product reliability. H.C. Starck Solutions achieves world class quality through continuous research of new products, development of engineering solutions, and continuous improvements in H.C. Starck Solutions' manufacturing environment to deliver premium products for the most challenging applications.

Nearly 100 years of powder metallurgical experience is the cornerstone of H.C. Starck Solutions' success in producing advanced technology metals for fast growing industries including aerospace, chemical processing, electronics, industrial, medical, and energy. As one of the world's leading suppliers of molybdenum, tungsten, tantalum, niobium, and their alloys, H.C. Starck Solutions is at the forefront of creating solutions with next-generation materials and fabricating engineered components for a diverse spectrum of markets.

- > **Product Quality and Service**
- > **Research and Development**

- > **Manufacturing Excellence**
- > **Reclamation and Recycling**



Strategic Advantages of Working with H.C. Starck Solutions

H.C. Starck Solutions understands market trends and the latest cutting-edge technologies are providing us the opportunity to create value added solutions for complex applications. In addition, our robust and sustainable, vertically integrated supply chain enables us to deliver high performance materials and products seamlessly to the marketplace. Paramount to H.C. Starck Solutions is securing materials from reliable sources. Our raw material procurement relies on the continuous expansion of our recycling activities and the fair, conflict-free and sustainable procurement of raw materials.

A recognized leader in refractory metal technology, H.C. Starck Solutions' knowledge and technical expertise benefit customers through joint collaborations with our dedicated team of research engineers. This collaborative effort facilitates new and improved product designs through a study of the product's life-cycle. Extensive in-house state-of-the-art laboratory facilities with the latest in analytical tools, testing equipment, modeling and simulation software assist engineers in evaluating product performance. For example, innovative material solutions provide texture control thus enhancing the uniformity and performance consistency.

Spanning the globe with over 30 locations worldwide including Asia, Europe, and the Americas, H.C. Starck Solutions offers exceptional customer care with local sales and technical support. Our local presence coupled with multiple global manufacturing sites permits us to effectively respond to our customer's requests.

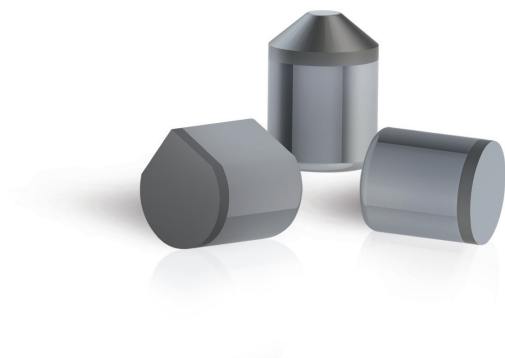
Innovation in Producing Synthetic Diamonds

H.C. Starck Solutions' niobium (Nb) is formed into crucibles for the manufacture of synthetic diamonds in a wide range of applications. Certain applications require block-like synthetic diamonds, which are produced by placing a carbonaceous material in the niobium crucible and subjecting it to a combination of high pressure and high temperature (HPHT).

The niobium crucibles are produced from sheet material by deep drawing. The quality and surface finish of these crucibles is critical to the final quality of the synthetic diamonds. To meet the deep draw challenges and quality requirements for synthetic diamonds, H.C. Starck Solutions' development engineers made significant improvements to our niobium sheet that addresses the challenges and issues inherent to manufacturing the crucibles.

Applications using Synthetic Diamonds

- > **Machining**
- > **Cutting**
- > **Thermal management**
- > **Magnetometry**
- > **Acoustics**
- > **Drilling**
- > **Optical (Laser, IR, RF)**
- > **Detectors in high energy physics**
- > **Waste water treatment**
- > **Ozone generators**



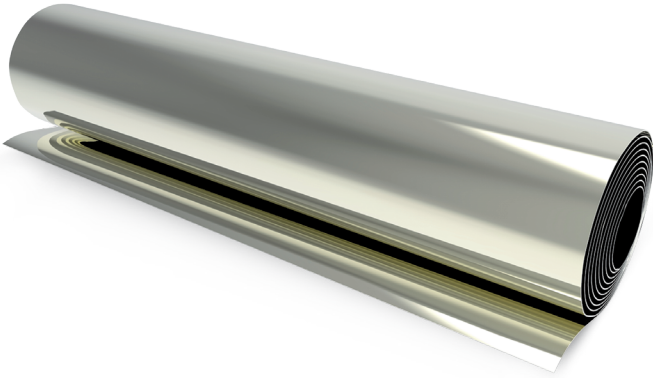
Industries Served

Synthetic Diamonds are used as an abrasive tool for non-ferrous and non-metallic materials:

- > **Stone: oil & gas exploration, mining, quarrying and building process**
- > **Construction: building infrastructure**
- > **Woodworking: wood-based products such as flooring, furniture and consumer products**
- > **Transportation: automotive and aviation industry**
- > **Machining: general industrial machining includes non-ferrous metals, plastic and other materials**
- > **Electronics: ingot and wafer slicing includes silicon, glass, sapphire and other materials**



Niobium Sheet Deep Drawn to Form Crucibles



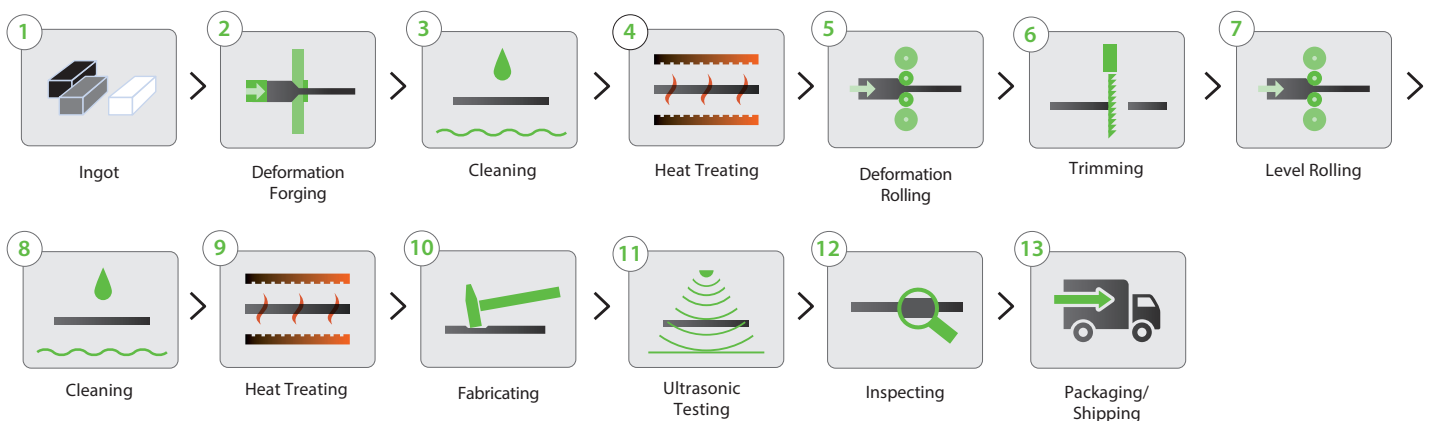
The niobium crucible is a single-use container designed for the high pressure and high temperature (HPHT) process in making synthetic diamonds. The production of these crucibles starts with relatively simple and straightforward cold rolling and annealing processes to produce niobium sheet material. The niobium sheet is deep drawn into crucibles through a punch process that uses one or more dies to manufacture the small crucibles.

One of the key advantages of H.C. Starck Solutions' niobium is our ability to roll sheet evenly, so it does not become thin, crack, or fail during the deep drawing process. In order for the niobium sheet to withstand multi-axial tensile stresses without failure, it has to be extremely ductile and possess suitable anisotropic mechanical properties to reduce wall-thinning during deep drawing.

Anisotropy in sheet is gauged by a number, called the r-value (or Lankford coefficient), which refers to the ratio of true strain along the width to true strain along the thickness during a tensile test. The r-value usually varies as a function of the sheet rolling direction.

Niobium Process

- > In-house expertise in forging, rolling, machining, cutting and fabrication
- > Expertise in thermal processing
- > Chemical, mechanical, and microstructure monitoring in-house
- > Consistent grain size and texture control



Avoiding Crucible Defects with Niobium Sheet

There are many variables to consider when designing a deep draw process. An incorrect setting on any of the steps can lead to defects¹ in the final deep drawn component.

- > **Wrinkling in the flange occurs due to low blank holding force.**
- > **Wrinkling in the wall occurs when a wrinkled flange is drawn into the cup or clearances are very large.**
- > **Tearing occurs from high tensile stresses that cause sheet thinning. Tearing also occurs from sharp corner radii, high length-to-diameter (L/D) ratios, high pressure pad loads, and high punch loads.**
- > **Earring occurs when the material is anisotropic (i.e., directionality in properties).**
- > **Surface scratches occur if the punch and die are not smooth or if process lubrication is inadequate.**
- > **Radial cracks in the flanges and edge of the cup due to insufficient metal ductility.**
- > **Orange peel (surface roughness) occurs in coarse grain metals.**

The most common material related defect issue is “orange peel” because of the coarse grain sizes. A related niobium microstructure can show poor flatness and smoothness of the deep drawn cup as each grain tends to deform independently and non-uniformly. Poor flatness and/or smoothness can produce a synthetic diamond that requires excessive grinding. A coarse grain structure can also cause tearing of the sheet during deep draw operations².

¹ MP Groover, “Sheet Metal Cup Drawing Lab Presentation”, Ohio State University, February 2008.

² PR Aimone, Internal H.C. Starck reports KDM01-02 and KDM01-08, 2001.

Grain Stabilized Niobium (GSNb)

H.C. Starck Solutions’ grain stabilized niobium sheet is a single-phase micro-alloyed niobium material that has a grain size of approximately 2 ASTM numbers finer than commercial grade niobium. This reduces the “orange peel” affect during drawing and forming operations and is particularly well suited for deep draw applications where uniform deformation is required.

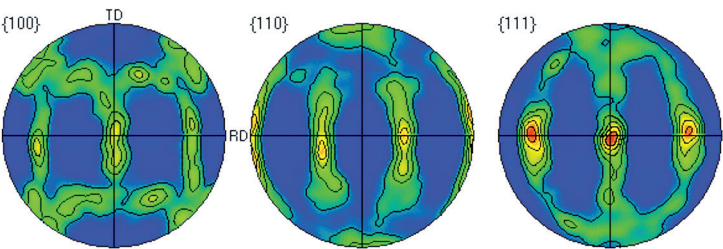
GSNb is used extensively in the industrial diamond market. Additionally it has corrosion resistant qualities equal to commercial grade niobium for applications in the chemical process industry. GSNb can also be used in sputtering targets for fiber optic applications and architectural glass. In nuclear reactors it has low thermal neutron cross-section and superior corrosion resistance. It is an excellent getter and finds use in high temperature vacuum furnaces, and is resistant to attack by the molten alkali metals found in sodium vapour lamps.

Optimizing Deep Drawing Capabilities

Deep drawing capabilities of niobium sheet are impacted by the amount of silicon (Si) added to the niobium composition. The effect of the Si concentration on the r-value or drawability in niobium sheet has been studied in detail. Thick sheets of 0.01" (254 μm) with Si concentrations between 40-135 ppm were measured in tensile testing, and it was found that the average r-value decreases linearly as the Si concentration increases. A value of less than 1.0 can be obtained when the Si concentration is greater than 85 ppm. Additionally, it was discovered that the Si concentration has its maximum effect on r-0 degree and r-90 degree texture measurements, and it is proposed that Si atoms may be pinning grains with <100>//RD and thereby, effecting the r-value.

For best deep drawing characteristics, an optimal Si concentration of 85 ppm was determined to ensure that the mean r-value remains greater than 1.0 while also maintaining a small grain size. Good control of the Si concentration is critical to obtain consistent performance during deep drawing.

EBSD pole figure results for 60 ppm Si annealed Nb sheet of 254 μm thickness



As the Si-concentration increases, the mean r-value decreases from greater than 1.0 to lower than 1.0, with an optimal Si concentration of 85 ppm.

Corrosion Resistance

The corrosion resistance of GSNb is identical to that of commercial grade niobium and can be used in all applications where commercial grade niobium is used. Like tantalum, GSNb is resistant to most acids with the exception of hydrofluoric, however it is not as resistant as tantalum to strong acids at high temperature. It should not be used with strong bases (alkalis).

AVAILABLE FORMS AND PROPERTIES GRAIN STABILIZED NIOBIUM

Material	Thickness inches	mm	Width inches	mm
Foil	0.001- 0.015	0.025 - 0.381	up to 12	up to 304.8
Sheet	0.015 - 0.1875	0.381 - 4.763	up to 36	up to 914.4
Plate	0.1875 - 1	4.763 - 25.40	Common widths	

Many variations of thickness and width are available to meet the needs of the application.

PHYSICAL PROPERTIES OF NIOBIUM

Physical Properties	
Atomic Number	41
Atomic Weight	92.91
Density	8.47 gm/cc (0.31 lbs/in ³)
Melting Point	2468°C
Coefficient of Expansion (20°C - 100°C)	7.1 x 10 ⁻⁶ /°C
Specific Heat (27°C)	0.065 cal/gm/°C
Thermal Conductivity	0.125 cal/sec-cm-°C
Electrical Resistivity (0°C - 100°C)	14.5 microhm-cm
Crystal Structure	bcc

MECHANICAL PROPERTIES OF GSNB (ANNEALED)

Mechanical Properties	
Tensile Strength	18,000 psi (125 MPa) minimum
Yield Strength	10,500 psi (73 MPa) minimum
Elongation	20% minimum (>= 0.010" thick) 15% minimum (< 0.010" thick)
Hardness (Typical)*	HV 60-100
Grain Size (ASTM)*	6 or finer (45 ums) for thicknesses < 0.010"

*Additional information not included in ASTM B393
GSNb meets the mechanical properties requirements of ASTM B393 Type 2 Commercial Grade Niobium (UNS R04210).

Olsen Cup Testing is available on request for thinner gauges. GSNb has similar values to Commercial Grade Niobium. Typical Olsen Cup depth values for 0.005" to 0.010" thick GSNb are 0.240" (6.1) min.

CHEMISTRY

The chemical properties of GSNb are as follow:

Element	ppm (max)
C	100
O	250
N	100
H	15
Zr	200
Ta	3000
Fe	100

Other trace elements are less than 50 ppm each.

The elements CONH are tested at the ingot stage and may be higher in finished material.

Element	ppm (max)
Si	100
W	500
Ni	50
Mo	200
Hf	200
Ti	300
Nb	balance

Fabrication

Machinability	While challenging, GSNb can be machined using high rake angle tools, slow feeds and speeds, and water-soluble oils.
Weldability	GSNb can be resistance welded to itself and other metals such as tantalum, nickel, platinum, titanium and niobium. It can be welded using GTAW (TIG) using proper shielding and cleanliness techniques. It can also be Electron Beam (EB) welded.
Heat Treatment	GSNb will recrystallize at temperatures above about 1650°F (900°C) (Heat treat in vacuum only).

Typical Applications

Sheet, strip and foil for forming applications such as crucibles, cups and formed parts.
Any application that can use commercial grade niobium but requires improved forming and surface finish.

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DPAP 03/2020

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