PRODUCTION OF HIGH-PURITY-NIOBIUM UNDER INDUSTRIAL SCALE FOR UPCOMING LINEAR COLLIDER PROJECTS

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Abstract

Sheet, tubes and formed parts made of high-purity Niobium with a high residual resistivity ratio (RRR-Niobium) is the key component for future linear accelerators based on the superconducting radio-frequency technology. To be prepared for large production scale quantities, which are demanded for the upcoming projects like XFEL and ILC respectively, W. C. Heraeus GmbH (D) and PLANSEE Metall GmbH (A) joined their competencies in the field of RRR-Niobium. In 2007 the qualification procedure as material supplier for the XFEL project could be successfully finished and a complete product and technology package for products made of RRR-Niobium was established.

Based on the combination of the high expertise and long-term experience in electron beam melting of different RRR-Niobium qualities, the knowledge and availability of various processing technologies for manufacturing of semi-finished and ready to assemble components, and the unique analytical capabilities for advanced quality control along the process chain, customized product solutions can be realized for the accelerator industry. Besides, a general overview about the production capabilities available investigation methods for characterization and quality assurance of RRR-Niobium large production scale quantities will be discussed.

INTRODUCTION

In 2007, at the German Electron Synchrotron (DESY) in Hamburg the starting signal was given for the construction of XFEL (X-ray free electron laser). For the heart of the linear accelerator high purity niobium, so called RRR-Niobium grade, will be used for the cavity resonators [1]. In preparation of this cutting edge international project W. C. Heraeus GmbH (D) and PLANSEE Metall GmbH (A) started in 2004 a project to bundle the competencies of both companies on RRR-Niobium.

W. C. Heraeus GmbH (part of the Heraeus group) is working since many years on RRR-Niobium and is worldwide supplying research laboratories with melt-ingots and semi-finished products and components for cavity resonators [2]. PLANSEE Metall GmbH (part of the PLANSEE group) is specialized in manufacturing of products made of Molybdenum, Tungsten, Tantalum, Niobium and Chromium [3]. Based on available equipment PLANSEE Metall GmbH set up a production line for RRR-Niobium to allow the manufacturing of semi finished products and components according to customer material requirements. For RRR-Niobium sheet material in December 2007 the PLANSEE Metall GmbH was successful qualified as supplier for RRR-Niobium sheets [4,5]. Both companies are now joining their competencies in the field of RRR-Niobium - from the electron beam melting, the processing into semi-finished products by use of advanced manufacturing technologies and numerous analytical capabilities – to offer a complete product and technology package for the accelerator industry.

MELTING

Production of RRR-Niobium starts with standard Niobium ingots melted by electron beam or electro-arc-melting. The further refining is done by electron beam melting where Niobium is purified in a vacuum at over 2500 °C. In Fig. 1 the several reactions between the metal and the gases, as well the reactions between the gases itself are shown [6]. In particular, Niobium reacts readily with Oxygen and Nitrogen wherever it gets the chance. Vacuum remelting and special conditioning technique during melting leads to Oxygen contrentations lower than 5 µg/g. With a crucible diameter of around 300 mm and a weight in the range of 1,4 tons a modern melting plant for production of RRR-Niobium is available at W. C. Heraeus GmbH.

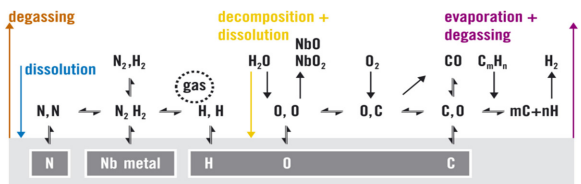

Figure 1: Metal-gas and gas-gas reactions during Niobium refining.

PRODUCTS AND COMPONENTS

Starting material are melted blocks with a typical diameter of around 300 mm and a length between 800 and 2000 mm. Forging is the first step for refining the as-cast grain structure and setting up the proper shape for further processing. After surface conditioning subsequent rolling for sheet production or forging and drawing respectively for production of rods or tubes is applied.

Due to the wide range of available production equipment within both companies semi-finished products in various geometries and sizes such as sheets, plates, discs and seamless tubes are available. Further processing

on the basis of machining and chipless forming allows the supply of RF cavity components and supporting parts ready to assemble such as HOM-coupler, housings, end-tubes and flanges. In Fig. 2 the production steps for the manufacture of RRR-Niobium products and components are given. In order to ensure product quality along the process chain, all the RRR-Niobium output passes through advanced quality control procedures including chemical analysis, characterization of mechanical properties and RRR measurement repeatably.

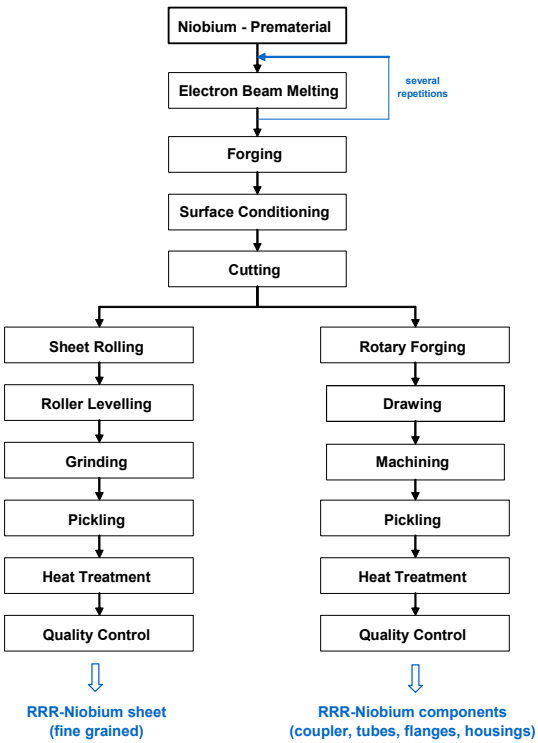

Figure 2: Production steps for manufacturing of RRR-Niobium products and components.

For sheet manufacturing the parameters for rolling and heat treatment are critical for production of fine grained sheet with equiaxed material properties meeting customer specification. For production of large scale quantities of RRR-Niobium sheet a rolling mill with useable width of 800 mm and a high vacuum annealing furnace with a useable length of about 3000 mm at PLANSEE Metall GmbH are available.

Final conditioning operations for sheet manufacturing like e.g. roller levelling, grinding and pickling according to costumer specification can be made on fully automized equipment.

QUALITY ASSURANCE

RRR values of 300 and higher are achieved by marked reduction in the contents of interstitially dissolved non-metal element impurities by several electron-beam-melting refining steps. Mainly the content of the interstitially disolved elements Oxygen and Nitrogen have a strong influence on the RRR value (Fig. 3). But nevertheless also metallic elements, especially those with higher melting points as Niobium (e.g. Ta, W), must be controlled very careful over the whole supply and production chain due to thermal stability and increase of concentration during purification by melting [6,7,8].

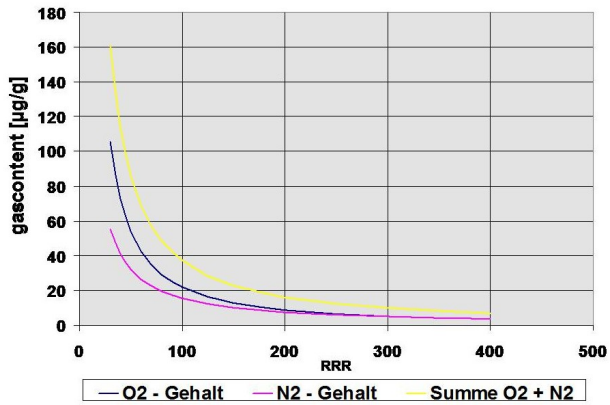

Figure 3: RRR value for Niobium as function of the interstitial solved gases Oxygen and Nitrogen.

In order to maintain the high purity level over the whole production route the parameters for deformation, heat treatment and machining must be well defined and controlled to prevent reaction with the atmospheric gases. Furthermore, due to the mechanical softness of the material and the cold welding tendency of Niobium a careful handling during processing is mendatory to prevent surface defects or surface contaminations.

Spot like concentrations of interstitially disolved gases or metallic impurities, small defects in the microstructure or scratches or inclusion of foreign material in the surface can lead to problems in the performance of the cavity resonator. Therefore, analytical techniques with high detection sensitivity are applied for characterization and quality assurance of RRR-Niobium. In both companies advanced quality control procedures were established and used for characterization and quality assurance of industrial RRR-Niobium production as listed below [9,10].

Gas analysis in solid materials:

- Trace determination of of C, O, H, N and S in the bulk by fusion extraction using the platinum flux technique.
- Determination of moisture and carbon on the surface.

Physical properties:

- RRR measurement (according established standard for XFEL).
- Determination of thermal conductivity and thermal expansion coefficient.
- Measurement of heat capacity.

Mechanical and non-destructive testing methods:

- Tensile strength, bending strength and hardness tests.
- Determination of strain hardening coefficient and plastic strain ratio.

- Ultrasonic testing.
- SQUID (eddy current testing with superconducting magnetic field sensors).
- USM (ultrasonic microscopy for determination of volumetric defects).

Metallographic analysis:

- Qualitative and quantitative microstructure analysis.
- Micro hardness and low-load hardness tests.
- High resolution electron scanning microscopy.
- EBSD analysis for determination of crystallographic orientation and micro-texture.

Surface analysis:

- EDX/WDX analysis by use of scanning electron microscopy.
- X-ray microanalysis.
- Scanning Auger microanalysis.

SUMMARY

Based on the combination of the high expertise and long-term experience in accelerator-technology, W. C. Heraeus GmbH and PLANSEE Metall GmbH are joining their competencies in the field of RRR-Niobium since 2004. The partnership offers a complete product and technology package for the accelerator industry from electron beam melting to processing of semi-finished products and components.

The constant quality and purity control over the whole process chain has an important role in the manufacture of RRR-Niobium products and will be guaranteed by use of adequate analytical capabilities, available within the laboratories of both companies. The applied production technologies and analytical methods are regularly audited with regards to compliance to the quality documents and the guidelines of ISO 9001 and the customer specification respectively.

Both companies are fully qualified as supplier of RRR-Niobium for the XFEL-project and are ready for industrial production of semi-finished products and components made of RRR-Niobium.

REFERENCES

[1] R. Brinkmann, "The European XFEL SC Linac Project", LINAC 08, Victoria BC, September 2008, MO102, p.6 (2008); http://www.JACoW.org

[2] W. C. Heraeus GmbH, D-63450 Hanau, Germany; www.wc-heraeus.com/special-metals-technology

[3] PLANSEE Metall GmbH, A-6600 Reutte, Austria; www.plansee.com

[4] A. Brinkmann rt al., "Progress of the test cavity program for the European XFEL", SRF 2007, Beijing, October 2007, TUP74, (2007)

[5] Communication between DESY (D) and PLANSEE Metall GmbH (A), December 2007

[6] B. Spaniol "W. C. Heraeus GmbH and its activities regarding large grain Niobium discs", International Niobium Workshop, Araxá, October 2006, p179-185 (2006)

[7] K. K. Schulze, "Preparation and Characterization of Ultra-High-Purity Niobium", Journal of Metals, May 1981, p33-41 (1981)

[8] P. Kneisel et al., "Influence of Ta content in high purity Niobium on cavity performance", PAC 05, Knoxville, May 2005, TPPT075, p.3955 (2005); http://www.JACoW.org

[9] A. Brinkmann et al., "Nondestructive Testing of Niobium Sheets for SRF cavities using Eddy-Current and SQUID flaw detection", LINAC 08, Victoria BC, September 2008, THP012, p.787 (2008); http://www.JACoW.org

[10] R. Grill et al., "Nb-RRR sheet inspection by means of ultrasonic microscopy", LINAC 08, Victoria BC, September 2008, THP001, p.758 (2008); http://www.JACoW.org