ECONOMICAL MANUFACTURE OF SEAMLESS HIGH-PURITY NIOBIUM

V. Arrieta and S. McNeal, Ultramet, Pacoima, CA 91331, U.S.A.

ABSTRACT

Ultramet continues to develop a methodology to fabricate high-performance (RRR>200) seamless niobium superconducting radio frequency (SRF) cavity cells using advanced chemical vapor deposition (CVD) methods. Optimization has resulted in the consistent rapid deposition of RRR>200 niobium at structural thicknesses suitable for accelerator applications. In addition, CVD niobium joining/welding capabilities have been demonstrated as a potential alternative to electron beam welding, and preliminary mechanical and microstructural characterization of the joined area has been performed.

INTRODUCTION

Ultramet manufactures advanced materials by CVD using a broad variety of refractory metals and ceramics for high temperature and/or extreme environment applications. Founded in 1970, Ultramet specializes in the research, development, prototype fabrication, and low- to medium-volume production of advanced materials to meet the needs of government, commercial, and academic customers.

The technical objective of the current ongoing research is to develop an efficient, cost-effective means of fabricating seamless ultrahigh-purity niobium SRF cavities and components suitable for particle accelerator applications.

The work to date, performed under two Small Business Innovation Research (SBIR) Phase I projects for the U.S. Department of Energy (DOE), has demonstrated the suitability of CVD for minimizing current SRF cavity fabrication challenges and the ability to fabricate freestanding CVD niobium cavity structures and to CVD-weld niobium components together. Ultramet's CVD niobium processing has also been optimized to consistently deposit structural high-purity niobium with purity ratings greater than RRR200 [1,2].

NIOBIUM SRF CAVITY FABRICATION BY CVD

The following inherent characteristics of CVD are unique to the process.

- Deposition is not line-of-sight, so uniform coating and seamless forming of freestanding structures on complex-shaped mandrels (e.g. low-beta cavities) is possible. Figure 1 shows a CVD niobium cavity prototype fabricated by Ultramet.
- Low-cost, medium-purity niobium chloride precursor material can be used because CVD is also a metal-refining process; reactions are induced with the metal of interest, which is then transferred to the mandrel for deposition while unwanted elements/impurities in the starting material are left behind. Non-heat-treated niobium has been successfully deposited with RRR values of 213–282.
- Decontamination heat treatments are minimized or potentially eliminated.
- Seamless cavity processing eliminates or substantially reduces the need for expensive electron beam welding.
- High reproducibility and economical near-net-shape fabrication are promoted.
- CVD is a practical and economical approach to fabricate complex and custom one-of-a-kind research cavities and test components, because simple removable mandrel techniques are employed and no fabrication molds are required.
- The need for and/or extent of electropolishing/etching is minimized, so target cavity profile designs are preserved during manufacture. CVD coatings reflect/reproduce/mimic the mandrel surface roughness, and conventional outer diameter mandrel polishing can be used to economically produce inner diameter cavity surfaces with low surface roughness.
- CVD grows inherently large, stress-free grains on a net-shaped mandrel surface. In contrast, mechanical deforming processes create a highly stressed inner cavity niobium surface layer that must be chemically removed during etching, further compromising the final cavity profile and maximum performance potential.
- Flanging features to accommodate joining and CVD welding of components can be fabricated *in situ*, thereby substantially reducing the need for electron beam welding.
- High niobium deposition rate (up to 0.012"/hr) makes formation of large, complex structural components practical.

Niobium CVD Process Optimization

Niobium CVD process optimization has included variations in reactor design, niobium source material, and process conditions (e.g. gas ratio, flow rate, temperature).

RRR Characterization

Multiple CVD niobium deposition runs were performed to fabricate specimens suitable for characterization at Oak Ridge National Laboratory (ORNL, Oak Ridge, TN). The characterization effort focused on determining the residual resistivity ratio (RRR) of the niobium materials deposited (without using a heat treatment purification step), along with that of niobium fabricated by conventional powder processing. The results are presented in Table 1 and show that four of the CVD materials exhibited RRR values ≥213 and one material had a RRR value of 282.

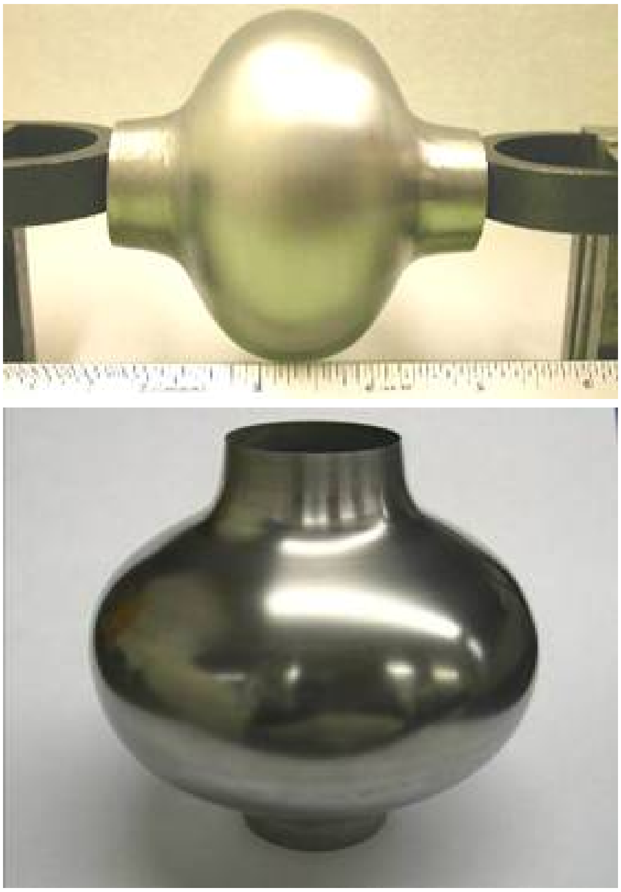

Figure 1: Freestanding seamless CVD niobium cavity prototype fabricated by Ultramet.

Table 1: RRR Measurements Performed by ORNL

CVD condition	RRR*
1	100
2	NA
3	150
4	160
5	282†
6	233†
7	226†§
7	213†§
Control	238†

* No heat treatment before RRR measurements.
† RRR measured in 1-T magnetic field.
§ CVD of as-received commercial-grade niobium scrap.

Performance Testing

A superconducting quantum interference device (SQUID) magnetometer was used to determine the magnetization of CVD niobium specimens at 5 K, yielding the overall magnetic hysteresis (energy loss per cycle) and critical fields (Figure 2). The low field of first flux penetration, related to the lower critical field Hc1, indicated the CVD niobium material's potential high gradient performance capabilities if it were to be used in a superconducting accelerator cavity application.

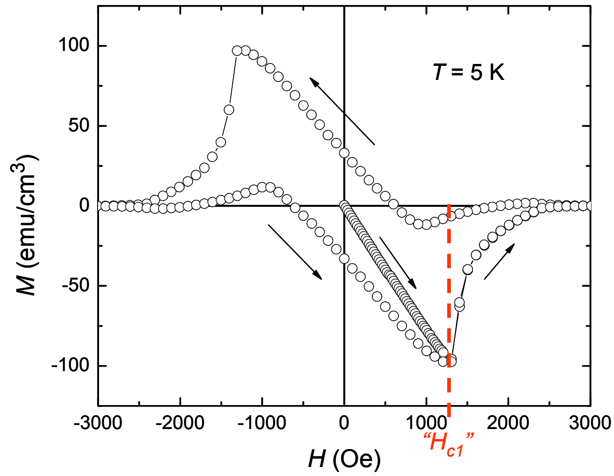

Figure 2: Complete magnetization loop indicating gradient performance potential conducted at 5 K, Hc1 (i.e., field of first significant flux penetration) ~1300 Oe, and Hc2 at high field ~2750 Oe.

CVD Welding

The CVD niobium joining process does not induce the unwanted recrystallization that occurs during electron beam welding. In the cross-sectional optical micrograph in Figure 3, the inherently large grains of the as-deposited CVD niobium weld material can be seen on the left, and unaffected and significantly smaller grains of the niobium tubes fabricated by powder metallurgy are apparent on the right.

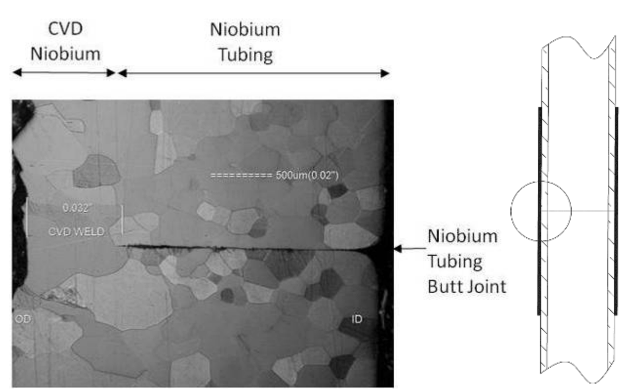

Figure 3: Cross-sectional optical micrograph of CVD-welded tensile butt-joint test specimen (*left*) and schematic showing orientation (*right*) of butted niobium tubes with ~0.03" thick CVD niobium. Measured ultimate tensile strength was 39.327 ksi vs. reported strength of pure annealed niobium of 43.5 ksi.

SUMMARY

The niobium material optimization conducted and the demonstration of near-net-shape cell prototype fabrication capabilities continue to support further development of this innovative approach to the fabrication, joining, modification, and repair of SRF accelerator components.

CVD niobium was deposited under various conditions to maximize purity using a relatively low-cost, moderatepurity niobium chloride starting material, and resistance and magnetic properties were characterized. Impressive RRR values up to 282 were measured in testing at ORNL. The initial feasibility of using CVD to join prefabricated niobium components, as a means to reduce or eliminate expensive electron beam welding, was demonstrated, and preliminary development of a low-cost mandrel for cavity fabrication in a production environment was performed. The fabrication and testing of a subscale single-cell International Linear Collider (ILC) cavity has been identified as the simplest and most economical way of measuring the RF properties of the CVD niobium material produced by Ultramet and has been proposed to DOE.

2012 UPDATE

In January 2012, Ultramet was awarded a third DOE SBIR Phase I project for the proposed subscale ILC cavity fabrication and testing using CVD niobium. Performance testing will be conducted by the SRF Group at Cornell University (Ithaca, NY).

REFERENCES

[1] V.M. Arrieta and S.R. McNeal, “High-Purity Niobium Superconducting Radio Frequency Cavities,” Final Report (ULTRA-TR-06-4205), Grant DE-FG02-05ER84175, Ultramet for U.S. Department of Energy, Washington, DC, June 2006.

[2] V.M. Arrieta and S.R. McNeal, “Economical Manufacture of Seamless High-Purity Niobium,” Final Report (ULTRA-TR-10-4935), Grant DE-SC0002721, Ultramet for U.S. Department of Energy, Washington, DC, July 2010.