QUALITY FACTOR MEASUREMENTS OF THE ULTRAMET 3 GHz CAVITY CONSTRUCTED USING CHEMICAL VAPOUR DEPOSITION*

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Abstract

A seamless 3 GHz bulk niobium cavity constructed by Ultramet using rapid chemical vapor deposition (CVD) techniques has been tested on the vertical SRF test stand at Cornell. The cavity received a 25 μm buffered chemical polish (BCP) and 700°C heat treatment for 4 days. First test results gave an intrinsic quality factor of $Q_0 = (1.55 \pm 0.12) \times 10^7$ and $(2.00 \pm 0.15) \times 10^7$ at 4.2 K and 1.5 K, respectively. A second BCP removed 100 μm of material, after which test results improved to $Q_0 = (7.59 \pm 1.52) \times 10^7$ and $(4.16 \pm 0.31) \times 10^8$ at 4.2 K and 1.5 K. During the first test poor coupling to the input amplifier impeded tests at accelerating fields >0.2 MV/m, while during the second test the cavity quenched at 1.3 MV/m when operating at 1.5 K. An optical inspection of the cavity after the second test revealed the presence of at least 4 pits on the upper hemisphere suggesting an area of higher than average surface resistance that may have contributed to the low field quench via thermal runaway. The potential of CVD as a construction method for SRF cavities is discussed.

INTRODUCTION

A seamless 3 GHz ILC-style SRF bulk niobium cavity was delivered to Cornell in September of 2012 for testing. The cavity was produced using a fabrication process incorporating rapid chemical vapor deposition (CVD) techniques developed by Ultramet, Inc. (Pacoima, CA USA), under research funded by the U.S. Department of Energy. The cavity was subjected to two vertical performance tests, the results of which are presented in this paper. Also presented are RRR measurements of three samples of Ultramet CVD niobium conducted by Cornell in July 2012.

Ultramet's CVD-based cavity fabrication methodology uses low cost commercially available niobium source materials, and allows for the construction of seamless niobium cavities and components that require a minimal amount of assembly welds, includinge achieved. Furthermore, XPS measurements conducted by Ultramet on CVD niobium samples confirm that contamination from the mandrel of the as-fabricated niobium is confined to a region <25 μm from the surface [1]. This suggests that CVD niobium cavities will require only minimum chemical treatment to achieve peak performance.

CAVITY CONSTRUCTION METHOD

The cavity is formed by depositing niobium, using proprietary CVD techniques developed by Ultramet, onto a sacrificial interlayer metal-coated graphite mandrel, such as those seen in Fig. 1 (A). The mandrel, immediately after application of the interlayer metal, is shown in Fig. 1 (B).

For the scaled-down 3 GHz ILC cavity tested, a two-layer CVD niobium process was adopted for cost-saving purposes; first, a thin initial layer of higher quality niobium was deposited on the mandrel, after which the surface was sanded using a CNC grinder. Subsequently, a second layer of standard grade niobium was deposited to extend the average thickness of the cavity walls to 3 mm for the sake of structural integrity. The cavity immediately after CVD is shown in Fig. 1 (C).

To prevent contamination of the niobium surface layer with hydrogen and other impurities, the mandrel must be thoroughly out-gassed prior to the CVD process. Upon completing the niobium deposition, the outside of the cavity is machined on a CNC lathe to achieve uniform wall

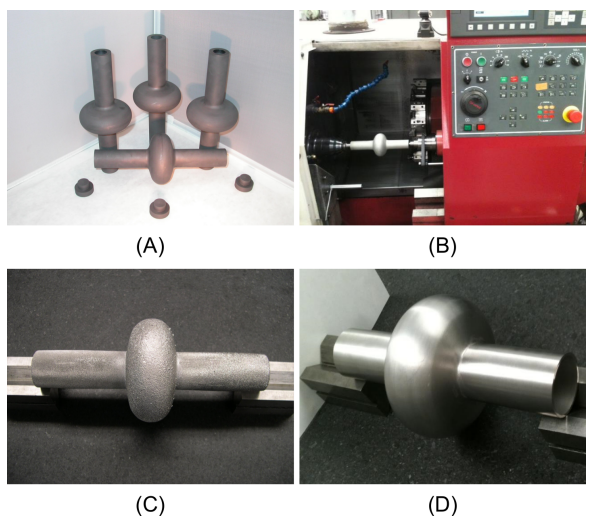

Figure 1: (A) Graphite mandrel prototypes machined using CNC methods. (B) The mandrel immediately following application of the sacrificial interlayer metal. (C) The cavity immediately after CVD. (D) The completed cavity, after removal of the mandrel and exterior finish. Photographs courtesy of Ultramet, Inc.

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thickness, after which the mandrel is removed by chemical methods. The cavity in its final as-fabricated form prior to leaving Ultramet for Cornell is shown in Fig. 1 (D).

RRR RESULTS FROM CVD SAMPLES

In July 2012 Cornell performed RRR measurements of three samples of CVD niobium provided by Ultramet. Each sample was deposited using different parameters in the CVD process (time, temperature, pressure, reactor design, etc.). The results of the RRR measurements are given in table 1. These, together with the RRR measurements performed separately by ORNL on behalf of Ultramet [2-4], demonstrate that the CVD process can be optimized to produce high quality niobium with a bulk RRR >200, on par with high-RRR niobium used in traditional cavity fabrication.

CAVITY PREPARATION AND RESULTS OF THE FIRST TEST

The as-fabricated cavity included beam pipes and was received without beam pipe flanges; these were fabricated and added at Cornell upon arrival. The cavity received a 5 μm buffered chemical polish (BCP) in preparation of the flanging operation, after which two reactor-grade niobium indium-seal flanges were electron-beam welded at either end of the cavity. The CVD niobium responded well to beam welding and demonstrated good structural integrity. An additional 10 μm of material was removed via BCP to remove any possible contaminants introduced during the welding process.

The cavity then received a 700°C bake in a UHV furnace for 4 days, followed by a further 10 μm BCP. Since work carried out separately by Ultramet with samples of CVD niobium indicate that surface impurities from the fabrication process are present only to a depth of <25 μm , the standard 100 μm BCP etch was not performed for the first test.

Cavity preparation culminated in a final low temperature bake to improve the BCS surface resistance of the niobium. Although the bake was intended to be 120°C for 48 hours, a failure in the furnace control system instead caused the cavity to be baked at 210°C for 3 hours. The cavity received a final high pressure rinse before being mounted on the test stand for a vertical test.

Results for the first test measuring intrinsic quality factor Q_0 against accelerating field E_{acc} are shown in Fig. 2.

Table 1: Results of the RRR measurements at Cornell on CVD deposited niobium. Each sample was fabricated using different sets of critical parameters in the CVD process.

Sample No	RRR
1-1	(129 ± 24)
2-1	(253 ± 44)
3-1	(47 ± 9)

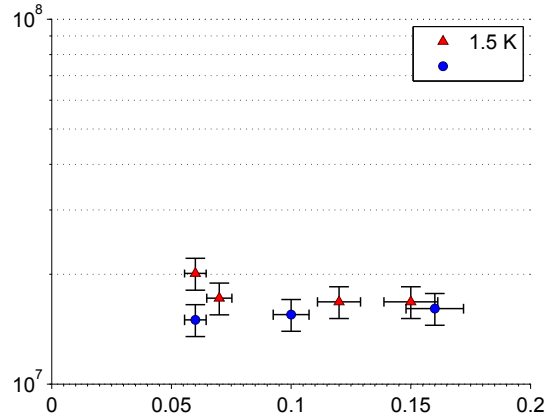

Figure 2: Quality factor Q_0 vs. accelerating field E_{acc} measurements made during the first vertical test. Poor coupling to the cavity impeded testing at fields >0.2 MV/m.

Values of Q_0 were taken at temperatures of 1.5 K and 4.2 K. Initial results demonstrate no discernible difference between the two temperatures, although poor coupling between the input antenna and the cavity impeded testing at fields greater than 0.2 MV/m. Results from the first test indicate a Q_0 of $(2.00 \pm 0.15) \times 10^7$ at 1.5 K, sub-par for a 3 GHz cavity when compared to standard fabrication methods. The measured surface residual resistance of $(1.4 \pm 0.1) \times 10^{-5}$ Ω suggests the presence of surface contaminants and/or areas of poor superconductivity or normal conductivity. The cavity was removed from the vertical test stand and preparations were made for a second test.

POST-TEST CHEMISTRY AND RESULTS OF THE SECOND TEST

To remove the surface contamination suggested by the results of the first test, the cavity was subjected to a 100 μm BCP etch. No other chemistry or further baking was performed, and the cavity was returned to the test stand for a second test. Measurements taken at 1.5 K indicate a Q_0 of $(4.45 \pm 0.33) \times 10^8$ at 1.1 MV/m, and a measurement taken at <0.5 MV/m shows a low-field Q_0 of $(7.59 \pm 1.52) \times 10^7$ at 4.2 K. Accordingly, the average residual surface resistance improved to $(6.50 \pm 0.49) \times 10^{-7}$ Ω. The Q_0 at 4.2 K indicates a BCS resistance of $(2.91 \pm 0.22) \times 10^{-6}$ Ω, which is in good agreement with the expected BCS resistance for niobium of 2.49×10^{-6} Ω obtained using SRIMP [5].

A quench at approximately 1.3 MV/m impeded testing at higher fields despite the improved coupling factor. A Q_0 vs. E_{acc} curve for the second test is shown in Fig. 3.

OPTICAL INSPECTION OF THE CAVITY INTERIOR AND ANALYSIS OF RESULTS

An optical inspection of the cavity was performed after the second test to investigate for possible causes of the

quench at 1.3 MV/m observed during the second test. A number of pits and surface defects were found on the upper hemisphere of the cavity in a band parallel to the equator, as shown in Figs. 4 (A) and (B), ranging in size from small indentations to pits approximately 0.2 mm across and 0.05–0.1 mm deep. Epoxy casts of the inside of the cavity confirmed that the larger pits are visible to the naked eye, although the small size of the cavity impeded the creation of a reliable cast from which to obtain depth measurements.

Discussion with Ultramet suggests that these pits may be evidence of the BCP etching through the first niobium layer and exposing the interface between the first and second layers of niobium. It is likely that damage to the mandrel locating datum, and/or small disparities in the CNC grinding profile during coating and machining operations and the physical geometry of the cavity, led to regions of the first niobium layer along the length of the cavity being thinner than expected or even being penetrated, creating microscopic pin holes exposed during cavity preparation etching operations. This is strongly supported by the aligned nature of the pits and their being confined to a narrow band on the cavity inner surface. Subsequently, this thin region of the niobium layer interface was the first to be exposed by surface chemistry. Further BCP may exacerbate existing pits or expose additional pits, and/or artifacts of the CNC grinding process.

The results of the first test suggest the presence of surface contaminants. Discussions with Ultramet conclude that the metal interlayer coating on the mandrel may not have been sufficiently outgassed, hence leading to contaminants being introduced into the surface niobium layer. Although more aggressive surface chemistry removed enough contamination to lower the average residual surface resistance

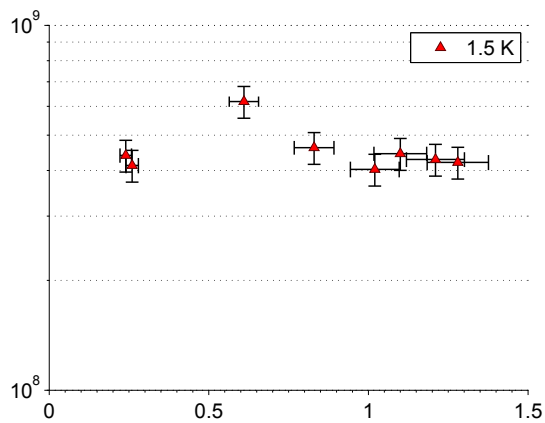

Figure 3: Quality factor Q_0 vs. accelerating field E_{acc} measurements taken during the second vertical test, following the removal of 100 µm of material via BCP. Only one point at 4.2 K was taken during this test, not shown here.

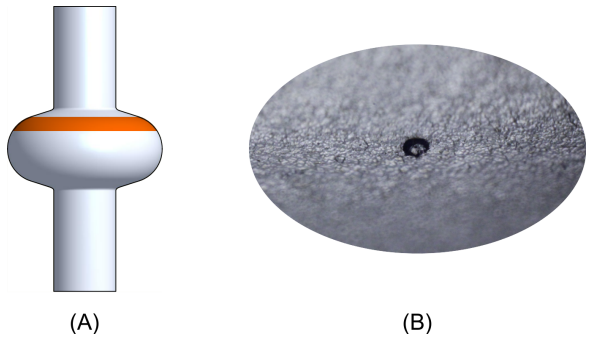

Figure 4: (A) Extent of the band (superposed in red on the cavity diagram) in which all pits were found. (B) An example of one of the pits. The pit shown is approximately 0.2 mm in diameter.

which, coupled with possible contaminants left from the CNC grinding process, contributed to a thermal runaway that caused the quench observed at 1.3 MV/m.

CONCLUSION

The CVD cavity has been shown to be capable of achieving a quality factor of $(7.59 \pm 1.52) \times 10^7$ at 4.2 K, close to the expected BCS value for niobium at 3 GHz; a promising result for a first prototype. Furthermore, the CVD process has demonstrated that it can create niobium samples of RRR >200. The results of the tests and subsequent inspection indicate that the current limitations of the cavity lie primarily in easily adjusted construction methods. In future cavities, to prevent contamination of the niobium surface layer, the mandrel must be thoroughly degassed prior to deposition. Furthermore, Ultramet is currently conducting DOE-funded research to develop improved mandrelling techniques due to undergo validation at Cornell in September of 2013 [6]. The mandrelling techniques under development will allow for the net deposition and/or substantially thicker initial deposits of the RRR>200 CVD niobium material, hence avoiding or minimizing the risk of interlayer penetration or contamination by machining deposits.

In summary, the CVD fabrication process shows considerable promise with respect to the creation of seamless superconducting structures, with potentials for cost savings and the construction of complex geometries, and warrants the need for further investigation.

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