America's Overview of Superconducting Science and Technology of Ingot Niobium

Gianluigi Ciovati, Peter Kneisel and Ganapati R. Myneni

Thomas Jefferson National Accelerator Facility, 12000 Jefferson Avenue, Newport News, VA 23606, USA

Abstract. This contribution will present an overview of the results from R&D programs in the USA over the past four years towards the development of ingot Niobium as a viable alternative material to fabricate SRF cavities for particle accelerators. Activities at several laboratories and universities include fabrication, surface treatment and RF testing of single- and multi-cell cavities and studies of the thermal, mechanical and superconducting properties of samples from ingots of different purity. Possible advantages of ingot niobium over standard fine-grain (ASTM 6) are discussed and a streamlined treatment procedure to fully exploit those advantages is proposed.

Keywords: Superconducting cavities, niobium

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INTRODUCTION

Since the first successful RF tests of large-grain cavities made from Nb sheets directly sliced from ingots at Jefferson Lab in 2005, several activities have been pursued by laboratories and universities throughout the world to investigate the applicability of this new technology to the production of superconducting RF (SRF) cavities with performance comparable to that of cavities made of standard high-purity, fine-grain (ASTM 6) Nb. A comprehensive review of these activities up to about 2006 can be found in [1]. The requirements on fine-grain Nb for SRF cavity production can be found in [2]. Although progress in the surface treatments of fine-grain Nb cavities over the past decade has pushed their performance close to the theoretical limit of the material [3], large-grain/single-crystal Nb directly sliced from an ingot may have the following benefits over fine-grain material:

- removal of elaborate sheet fabrication steps, such as forging, annealing and rolling
- smooth surfaces achieved by buffered chemical polishing (BCP), which is a cheaper chemical treatment than electropolishing (EP)
- higher thermal stability because of the presence of a phonon peak in the thermal conductivity at about 2 K
- lower residual resistance, resulting in reduced cryogenic losses
- cleaner surfaces by high-pressure rinsing (HPR), resulting in higher values of the surface electric field corresponding to the onset of field emission [4].

The possible advantages listed above would result in reduced cost of fabrication, treatment and operation of SRF cavities and the R&D programs pursued over the past four years are trying to fully investigate those possibilities.

This contribution is organized as follows: the next section will review the progress on fabrication, treatment and RF testing of single- and multi-cell cavities. The next section will review material studies on samples while the following section will propose a streamlined cavity process procedure which could result in higher Q-values (reduced RF losses), which is an important aspect for continuous wave (CW) superconducting Linacs. Finally, some conclusions will be drawn in the final section.

SINGLE- AND MULTI-CELL CAVITIES FABRICATION AND RF PERFORMANCE

Cavity Fabrication

Because niobium sheets directly sliced from an ingot have many grains of different orientations with typical area of several cm^2 , the deep drawing process used to stamp the sheet in a half-cell result in some issues related to the non-uniform grain deformation which can be properly addressed, as discussed in [5, 6]. As more large-grain cavities have been built in the past few years, some issues with electron-beam welding (EBW) of the equator of dumbbells made of large-grain Nb has emerged. A full penetration electron beam weld at the equator line from the outside cavity surface is done to join dumbbells or half-cells together to form a cavity. Using the same welding parameters used for fine-grain Nb, the experience at Jefferson Lab showed that a hole was blown during EBW of at least one of the equators during the fabrication of multi-cell cavities (typically 5 to 9 cells). These events, which were much rarer when fabricating single-cell cavities, are not correlated with the particular dumbbell welding sequence but the hole location appears to be near grain boundaries.

The holes had to be repaired by machining the hole region to fit a Nb “plug” which is then electron beam welded. KEK also experienced difficulties with the equator EBW of large-grain cavities but the only company who has built large-grain cavities so far (Research Instruments, Germany) successfully produced eight 9-cell 1.3 GHz cavities with no welding issues [7].

The possibility of cutting Nb from an ingot with almost any shape or thickness prompted a company (Niowave, USA) to use industrial metal forming processes, other than deep drawing, (for example forging, extrusion, spinning and flow-forming) to fabricate cavity parts with reduced number of components and welds [8].

Single-Cell Cavities

Extensive RF testing at JLab of 1.3 GHz and 1.5 GHz single-cell cavities made from large-grain Nb from different manufacturers (CBMM, Brazil, Ningxia, China and W.C. Heraeus, Germany) resulted in excellent, reproducible performances: the average quench field at 2.0 K from 51 RF tests of 18 different cavities occurred at a peak surface magnetic field (B_p) of 140 ± 14 mT [5]. The residual resistivity ratio

(RRR) of the material used to fabricate these cavities ranged between 280 and 500 and no correlation between RRR and quench field value was found. The cavity treatments before RF testing are based on material removal by BCP, hydrogen degassing at 600 °C or 800 °C, HPR, baking at 120 °C, post-purification at 1250 °C with Ti. The cavity treatments are described in greater detail in [5].

Three sets of four additional 1.3 GHz single-cell cavities were built with large-grain Nb from CBMM, Ningxia and W.C. Heraeus in order to further study the reproducibility of the RF performance. The cavities were subjected to the same treatments consisting of 25-50 µm material removal by BCP, heat-treatment at 600 °C/10 h, 50-90 µm BCP, HPR and baking at 120 °C/12 h. The average quench field at 2.0 K measured for the W.C. Heraeus and Ningxia cavities is 147 ± 19 mT and 141 ± 18 mT, consistent with the results from the earlier study [1]. In both cases, the Q_0 -value at the quench field is about 1×10^{10} . RF testing of the cavities built from CBMM material is ongoing.

The RF cavity tests also showed that the onset of the so-called high-field Q-drop, which is a common feature of cavities made of bulk Nb, is shifted to B_p -values of about 110-120 mT compared to about 90 mT for fine-grain cavities treated by BCP [9].

In one case, the RF performance of a 1.5 GHz large-grain single-cell cavity made from CBMM ingot B niobium showed a steep Q-degradation starting already at $B_p \cong 20$ mT [10]. Temperature maps showed few localized hot-spots in the equator region. Samples from the hot-spot regions were cut and different measurement techniques were applied to gain further insight on the possible loss mechanism, as will be discussed in greater detail in the next section.

Multi-Cell Cavities

Several multi-cell cavities in the GHz range have been built at JLab over the past 4 years:

- two 1.3 GHz 9-cell cavities of the TESLA/ILC shape [11] (LG1 and LG2)
- one 1.3 GHz 7-cell cavity of the Low Loss (LL) shape [12]
- two 1.497 GHz 7-cell cavities of the High Gradient shape proposed for the CEBAF Upgrade [13] (HG-A and HG-B)
- two 1.497 GHz 7-cell cavities of the Low Loss shape used for the CEBAF Upgrade [13] (J100-1 and J100-2)
- one 1.497 GHz 5-cell cavity of the High Current (HC) shape [14].

Most of these cavities were built using Nb from different ingots, named A, B, C and D, provided by CBMM. The RRR of the ingots is ~ 280 and the main difference is the Ta content: ~ 800 wppm in ingots A and B, ~ 1500 wppm in ingot C and ~ 1300 wppm in ingot D. As mentioned above, all of these cavities had a one hole burnt through at least one of the equatorial EBW. Pictures of some of the large-grain multi-cell cavities built at JLab are shown in Fig. 1.

The surface preparation, similar to that of the single-cell cavities, consisted of ~ 50 µm material removal by BCP, heat treatment at 600 °C/10 h, ~ 50 µm BCP, HPR. Cavities J100-2 and LG1 were also baked at 120 °C. Some cavities (HC) were also

post-purified at 1250 °C/3 h in a Ti box followed by ~50 μm BCP. Some cavities (LG1 and HC) were electropolished, removing about 35 μm . The best RF test results at 2.0 K of the 1.3 GHz and 1.497 GHz cavities are shown in Figs. 2 and 3, respectively. The average quench field value of all multi-cell cavities is 100 ± 18 mT. This value is 40% lower than achieved on single-cells and the standard deviation is about twice as large as that obtained from single-cell cavity tests. One reason for this poorer RF performance can certainly be attributed to the repaired holes at the equator (high magnetic field region). Cavities LG1 and LG2 were tested with a 2-cell thermometry system [15] to detect the quench location, which occurred at the place of the weld repair. In addition, features such as etch pits and “grooves” were observed on the inside cavity surfaces and those might have been caused by not sufficient agitation of the acid during BCP process in the production closed-cabinet system at JLab.

FIGURE 1. Picture of the 1.497 GHz, 7-cell cavities J100-1 and J100-2 (a) and of the 1.3 GHz 7-cell Low Loss cavity (b) built from large-grain Nb at Jefferson Lab.

FIGURE 2. Best RF performance measured at 2.0 K for the three 1.3 GHz multi-cell cavities at JLab. The cavity LG1 was electropolished and baked at 120 °C/48 h. The arrows indicate a quench.

FIGURE 3. Best RF performance measured at 2.0 K for the five 1.497 GHz multi-cell cavities at JLab. The cavity J100-2 was baked at 120 °C/12 h. The arrows indicate a quench.

Interestingly, the lowest performing multi-cell cavities were built from CBMM ingot B, the same one from which the single-cell with anomalous losses mentioned above was made from. Two other single-cell cavities made from the same material had good performance ($B_{p,quench} = 112 - 143$ mT) [16]. One such multi-cell cavity was the 5-cell, 1.497 MHz HC. The cavity was subjected to several different surface preparation methods which included, besides the ones mentioned in the previous paragraph, barrel polishing (BP) [17, 18]. A summary of the RF test results at 2.0 K after various preparation procedures is shown in Fig. 4. The quench field did not exceed 95 mT.

FIGURE 4. RF test results at 2.0 K for the 1.497 GHz, 5-cell HC cavity after different surface preparation processes.

A positive result from the multi-cell cavities was the confirmation of maintaining high Q_0 -values (greater than 1×10^{10}) up to $B_p > 100$ mT, as it was measured on single-cell cavities. The values of the residual resistance were deduced from the Q_0 -values at low field, as described in [19], for the test results shown in Figs. 3 and 4 and are lower than those obtained on fine-grain cavities at the same frequency, as shown in Fig. 5. The values for the fine-grain cavities are the average of several tens of cavities built, processed and tested at JLab: 805 MHz SNS cavities (treated by BCP), 1497 MHz CEBAF refurbishment cavities (treated by BCP) and 1300 MHz ILC cavities (treated by EP) [19].

FIGURE 5. Residual resistance values obtained from the low-field $Q_0(2.0$ K) for fine-grain (FG) [19] and large-grain (LG) cavities at different frequencies.

SAMPLE STUDIES

Samples Cut from Single-Cell Cavity

Twelve samples were cut by milling from the 1.5 GHz single-cell cavity made from CBMM ingot B niobium which exhibited anomalous RF losses at low fields. Nine samples were from regions of high RF losses (“hot-spots”) and three from region of low RF losses (“cold-spot”). Optical microscopy and Secondary Electron Microscope (SEM) images showed many etch pits on the samples’ surface, with size 20-80 μm and 2-10 μm in depth as measured by 3D profilometry [10]. Higher density of etch pits correlates with the hot-spot behavior. Nevertheless, the pit distribution is not uniform among the multiple grains but attempts to correlate higher density of pits with a particular grain orientation is not yet conclusive. Magnetic field enhancement at the pits’ edges is not sufficient to explain the steep decrease in Q_0 occurring already at $B_p \cong 20$ mT.

Point Contact Tunneling (PCT) was used at Argonne National Lab to measure the electronic density of states in several regions of the samples at different temperatures and applied DC magnetic field. The measurements showed a large peak in the center of the energy gap in hot-spot rather than cold-spot samples. The temperature and field dependence of the peak could be fitted using the Anderson-Appelbaum theory which is related to the presence of magnetic impurities within the tunnel oxide or at the oxide/metal interface [20].

Measurement of Superconducting Properties

Jefferson Lab

Hollow cylindrical samples were machined from a new ingot production series from CBMM. The ingots, which were also labeled A, B, C, and D, have Ta content between 520 wppm and 1320 wppm. The samples were mounted in a setup which allows measuring the thermal conductivity as a function of temperature, the magnetization and the penetration depth as a function of temperature and applied external DC magnetic field up to 1 T [21]. The sample preparation consisted of 180 μm material removal by BCP, 600 $^{\circ}\text{C}/10$ h followed by 24 μm BCP and baking at different temperatures, between 120 $^{\circ}\text{C}$ and 160 $^{\circ}\text{C}$, for 12 h. Although the RRR of the samples from the different ingots ranges between 61 and 146, as obtained from the thermal conductivity value at 4.2 K, measured after 180 μm BCP 1:1:1, there was no significant difference in the values of the field of first flux penetration, H_{ffp} , (the analogous of the lower critical field H_{c1} for a sample with reversible magnetization curve) nor of the upper critical field, H_{c2} , measured at 2.0 K. Those values were $H_{\text{ffp}} = 176 \pm 5$ mT and $H_{c2} = 400 \pm 5$ mT [22]. Measurements of the penetration depth at 270 KHz as a function of the applied DC magnetic field allows measuring H_{ffp} and H_{c2} closer to the surface. The data showed:

- $H_{\text{ffp}}(2.0 \text{ K})$ values of about a 110 mT, lower than the bulk value

- a weak dependence of H_{ffp} on RRR
- an increase of $H_{\text{ffp}}(2.0 \text{ K})$ up to about 130 mT, independent of RRR, after 120 °C bake.

These results are summarized in Fig. 6 and discussed in [22] in greater detail. H_{ffp} is of particular interest because fluxons entering the Nb surface is a possible cause of the high-field Q-drop observed in SRF cavities [23]. The same samples have been treated by EP, followed by baking at 120 °C/12 h and the measurement results of the samples' superconducting properties after these treatments are reported in [24].

FIGURE 6. Field of first flux penetration obtained from DC magnetization measurements and from AC measurement, before and after baking at 120 °C/12 h, as a function of the RRR of four large-grain samples [20].

It was attempted to measure the quench field in RF of the same cylindrical samples, by inserting them into a pill-box cavity to form a coaxial cavity, excited in the TE_{011} mode at 3.5 GHz. Heating followed by quench was measured at $B_p \approx 25 \text{ mT}$ on the sample and seems to be caused by the small cross section of the samples' channel, so that the RF dissipated power reaches the critical heat flux for He II at rather low RF fields. All these measurements will be repeated on new samples with larger diameter, machined from new ingots from CBMM.

Florida State University/Applied Superconductivity Center

Bi-crystal samples cut from large-grain Nb sheets have been studied to understand flux penetration and motion along grain boundaries using different techniques: magneto-optical imaging and DC transport measurements [25, 26]. Both techniques showed that preferential flux penetration at a grain boundary can occur and it is highly sensitive to the orientation between the grain boundary plane and the applied external field. Flux penetration and flux flow along a grain boundary was clearly observed when the applied external field is parallel to the grain boundary plane.

In addition, a micro Hall probe was used to measure the magnetic field enhancement at a grain boundary step. A field enhancement factor of about 5 was measured at the edge of a grain boundary step with height $\sim 7 \mu\text{m}$ and 10° slope, relative to the normal direction.

Measurement of Mechanical and Thermal Properties

Extensive studies of the metallurgical properties of large-grain and single-crystal Nb samples have been carried out at Michigan State University (MSU) [27]. In particular, stress vs. strain curves were measured on single-crystal samples of different orientation and the results were correlated with the most active slip systems for each particular orientation. In addition, electron backscatter diffraction (EBSD) was used to study the crystallographic properties of single-crystal samples after EBW and showed that welding plastically strained single crystals leads to recrystallization, while it is possible to maintain a single-crystal structure when welding unstrained samples. EBSD studies also showed that Nb sheets sliced from different ingots have different orientation gradient distributions which can impact their formability. Thermal conductivity measurements on large-grain and single-crystal samples were also carried out at MSU and showed the presence of a phonon peak at $\sim 2.0 \text{ K}$ [27]. Future work will evaluate the impact of strain and heat treatments on the phonon peak.

Tensile test measurements on single-crystal samples of different orientations were also done at the National Institute of Standards and Technology (NIST) and showed yield stress values between 25 and 40 MPa [28]. Multi-axial straining tests of tri-crystal plates were also done and the local strain rate was measured during the test in an attempt to determine the locally active slip systems during the deformation process [29].

Tensile tests at cryogenic temperatures on bi-crystal and single-crystal samples were done at Oak Ridge National Lab. The yield strength increased from about 50-80 MPa at room temperature to about 560 MPa at 77 K [30]. Tensile tests of large-grain Nb samples heat treated at $800^\circ\text{C}/3\text{h}$ and $600^\circ\text{C}/10 \text{ h}$ were recently contracted by JLab to St. Louis Test Lab. The yield strength at room temperature was generally greater than 48 MPa and increased up to 512 MPa at 4 K [31]. At JLab, the evaluation of the cavity design requires the allowable stress to be lower than $2/3$ of the measured yield strength under a variety of load conditions [32].

STREAMLINED TREATMENT PROCEDURE FOR LARGE-GRAIN CAVITIES

Although standard cavity treatment procedures, such as BCP and heat treatment, equally applied to cavities with different grain size (single-crystal, large-grain and fine-grain) did not show significant variations in cavity performance, grain boundaries are still preferred locations for impurity segregation. In particular, oxygen and hydrogen are interstitials which are known to affect significantly the cavity RF performance. In this respect, large-grain niobium may have some advantages over fine-grain because of the reduced grain boundary length. We think that it could be

possible to develop a streamlined cavity treatment procedure which could lead to Q_0 -values at 2.0 K and $B_p \cong 90$ mT about a factor of 2 greater than typically achieved in fine-grain cavities at the same temperature and RF field, at a reduced cost. The treatment procedure after cavity fabrication is outlined as follows and each step is further discussed in the following paragraph:

- ~ 80 μm material removal by centrifugal barrel polishing (CBP)
- ~ 20 μm material removal by BCP
- heat treatment at $800^\circ\text{C}/3\text{ h} + 120^\circ\text{C}/12\text{ h}$ in a clean UHV furnace
- surface passivation by depositing a thin nitride layer
- HPR.

CBP is a mechanical material removal method developed and routinely used at KEK [33]. Its main advantage is to remove the damaged layer after forming while obtaining a uniform, smooth finish of the inner cavity surface, particularly in the weld regions. The amount of material removal by CBP and BCP may need to be optimized.

Recent studies of the RF performance of cavities heat-treated at $800^\circ\text{C}/3\text{ h} + 120^\circ\text{C}/12\text{ h}$, without subsequent chemical etching, showed 25%-80% improvement of the $Q_0(2.0\text{ K}, 100\text{ mT})$ compared to a baseline test after BCP. The Q_0 improvement was due to a reduction, up to $\sim 50\%$, of the BCS surface resistance combined with low values of residual resistance ($\sim 5\text{ n}\Omega$) [34]. Samples were heat treated with the cavities and analyzed by Secondary Ion Mass Spectroscopy (SIMS) at North Carolina State University and showed almost two orders of magnitude reduction in hydrogen concentration, compared to the control, BCPed samples [34]. A study in Ref. [35] showed that baking at $140^\circ\text{C}/3\text{ h}$, besides $120^\circ\text{C}/12\text{ h}$, can also reduce the high-field Q-drop, while reducing the time the cavity sits in the furnace. The success of this treatment, without following chemical etching, requires a clean, UHV (pressure of $\sim 10^{-8}$ mbar at 800°C) furnace. In this respect, JLab is planning on purchasing an induction furnace with separate cavity and furnace vacuum.

In order to avoid the re-absorption of hydrogen and oxygen during the furnace cool-down to room temperature, the inner cavity surface could be passivated with a thin ($\sim 10\text{ nm}$ thick) NbN layer, grown with the cavity inside the furnace at some intermediate temperature ($\sim 500^\circ\text{C}$). Possible methods to grow the film could be plasma discharge or atomic layer deposition. The natural oxide, Nb_2O_5 , is also a passivating layer but the growth of a dry oxide in the furnace at high temperature could create a layer contaminated with interstitial oxygen between the oxide and the metal, due to thermal diffusion. Interstitial oxygen is known to suppress superconductivity in Nb: for example, the critical temperature is reduced by 0.93 K/at.%. [36].

The final surface preparation step would be the standard HPR to remove particulates from the inner cavity surface and avoid field emission.

SUMMARY AND OUTLOOK

Despite its first development in 2006, no large-grain cavity has yet been installed in a cryomodule. This suggests that a significant amount of R&D work is yet to be done for large-grain ingot Nb to become an acceptable technology to fabricate SRF cavities

to be installed in particle accelerators. In the US, cavity fabrication and RF testing in the past 4 years has been limited to JLab. Single-cell cavities consistently achieve B_p -values of ~ 140 mT, corresponding to accelerating fields of ~ 32 MV/m but significantly lower fields were reached in multi-cell cavities, mostly because of problems with EBW. In this respect, the industry has done a better job in fabricating multi-cell cavities without such problems.

A complete set of specifications to order large-grain Nb from the manufacturers is still missing. Sample studies are helping in reducing the parameter space for grain size, orientation and purity of the material which could lead to reproducible mechanical and superconducting properties.

There are at least two important advantages of large-grain Nb over fine-grain, high RRR, Nb, which at the moment haven't fully realized: lower cost and lower RF losses. In the past few years it became evident that lower cost could be fully realized only if the market for large-grain Nb will expand significantly. The introduction of possibly cheap slicing techniques [37] pushes towards this direction but the adoption of large-grain Nb technology for large-scale cavity production projects seems necessary to make this expectation reasonable.

The presence of fewer grain boundaries in large-grain vs. fine-grain Nb has the potential for lower values of residual resistance, which is supported by some cavity test results. In addition, the presence of a phonon peak in the thermal conductivity at 2.0 K could improve the thermal stability of the cavity and maintain high Q_0 -values at high RF fields. The development of a cost-effective treatment procedure, such as the one outlined in this article, which could result in Q_0 -values significantly higher than what is currently achieved with fine-grain Nb, would fully realize the potential of large-grain cavities.

Clearly, a large number of cavities will have to be built and tested according to specified material properties and treatment procedures to prove their effectiveness. This can only be achieved with strong commitments by many laboratories, not only in the US but throughout the world.

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