

PRELIMINARY RESULTS FROM SINGLE CRYSTAL AND VERY LARGE CRYSTAL NIOBIUM CAVITIES*

P. Kneisel[#], G.R. Myneni, G. Ciovati, TJNAF, Newport News, VA, USA
 J. Sekutowicz, DESY, 22603 Hamburg, Germany
 T. Carneiro, Reference Metals Co, Bridgeville, PA, USA.

Abstract

We have fabricated and tested several single cell cavities using material from very large grain niobium ingots. In one case the central grain exceeded 7" in diameter and this was used to fabricate two 2.2 GHz cavities. This activity had a dual purpose: to investigate the influence of grain boundaries on the often observed Q-drop at gradients $E_{acc} > 20$ MV/m in the absence of field emission, and to study the possibility of using ingot material for cavity fabrication without going through the expensive rolling process. The sheets for these cavities were cut from the ingot by wire electro-discharge machining (EDM) and subsequently formed into half-cells by deep drawing. The following fabrication steps were standard: machining of weld recesses, electron beam welding of beam pipes onto the half cells and final equator weld to join both half cell/beam pipe subunits. The cavities showed heavy Q-disease caused by the EDM. After hydrogen degassing at 800 °C for 3 hrs in UHV and about 200 μm total removals from the inner surface by BCP 1:1:1, the cavities showed promising results, however, the Q-drop was still present. In the two cavities made from large grain material accelerating gradients of 30 MV/m have been reached. After "in-situ" baking the Q-drop disappeared. The smaller cavities made from single crystal material showed very low residual resistances and accelerating gradients up to $E_{acc} = 45$ MV/m were reached (one of the highest ever achieved), corresponding to a peak surface magnetic fields (B_p) of 160 mT. In one rf test at 2 K, a $B_p = 185$ mT was reached for few hundred milliseconds, close to the theoretical critical field of this material.

INTRODUCTION

High purity niobium sheets with RRR-values > 250 are exclusively used for the fabrication of high performance cavities for SRF accelerator projects such as SNS, TESLA, RIA or CEBAF upgrade. These sheets are formed from a multiple electron beam melted ingot by an elaborate process of forging, annealing, rolling and chemical etching. Until now it has been believed that uniform and fine grain material (ASTM grain size ≥ 6) is needed for deep drawing of cavity half cells. The sheet manufacturing process bears the risk of foreign material inclusions in the material and stringent QA procedures such as eddy current or squid scanning of the sheets are administered to ensure defect-free material prior to the

cavity manufacturing. On the other hand, the use of niobium cut from of a large grain ingot could be an alternative and conceivably less expensive manufacturing process, if the mechanical properties and the formability of this material is comparable to sheet material. This has now been proven to be the case as will be shown later.

For many years it has been observed that rf cavities made of high purity niobium suffer from the so-called "Q-drop" [1] even in the absence of field emission at accelerating gradients/magnetic surface fields above ~ 20 MV/m / ~ 100 mT. The reasons for this Q-degradation are not well understood, and explanations range from magnetic field enhancements at grain boundaries to features of the metal-oxide interface. [2, 3] Since grain boundaries are "weak" areas in a niobium surface that can easily be contaminated by segregated impurities and form "weak links", they are a prime candidate for causing a degradation of the performance of the material. By testing cavities with fewer or no grain boundaries it should be possible to investigate their influence on cavity performance and their impact on the Q-drop.

We have pursued these ideas and in the following will report on our preliminary results.

PROPERTIES OF INGOT MATERIAL

Ingot Production

Two ingots [(A and B) of about 220 lbs each with a diameter of 9 1/4"] were produced by Companhia Brasileira de Metalurgia e Mineração (CBMM, Sao Paulo, Brasil) from pyrochlore as found at CBMM's deposit in Araxa, Brazil. Standard processing procedures [4] converted the ore to niobium ingots after multiple electron beam melting of aluminothermically reduced niobium oxide. Ingot A had a very large single crystal of 7" diameter in its center, with a few smaller grains at the periphery, whereas ingot B had several large grains, but not as large as ingot A. The RRR value of the material was ~ 280 with a Ta content of ~ 800 ppm. The RRR value did not significantly improve (~ 10 %) during post-purification with Ti at 1250 °C.

Tensile tests performed with the large grain from the center of ingot A are shown in Figure 1. Surprisingly, this material is far superior in ductility to fine grain material and an elongation of 100 % was achieved. In addition, no yielding was observed in the elastic region of the stress-strain curve, a favorable property of the material in comparison to small grain niobium.

* Supported in part by DOE contract DE-AC05-84ER40150

[#]kneisel@jlab.org

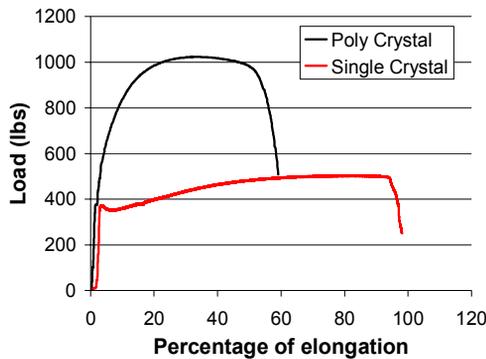


Figure 1: Load-elongation curves for the single and polycrystalline niobium samples.

CAVITY FABRICATION AND TESTING

Cavity Fabrication

Sheets of 0.125" thickness were sliced from the ingots by wire electro-discharge machining (EDM). Standard fabrication techniques were applied subsequently: half-cells were deep drawn, machined to dimension with a self centering welding recess and a beam pipe/flange assembly was electron beam welded (EBW) to each of the half cells. After mechanical polishing to remove any surface imperfections and removal of approximately 30 μm from the surface by Buffered Chemical Polishing (BCP), the two half-cells were joined at the equator by EBW. One 1.5 GHz cavities of the high gradient (HG) shape [5] was fabricated from each ingot. In addition, a scaled HG cavity at a frequency of 2.2 GHz, was built which used only the center large crystal of ingot A (Fig. 3). Additionally, we fabricated a scaled version of the proposed low loss (LL) cavity for ILC [6] from single crystal material at a frequency of 2.3 GHz. For comparison to the single crystal cavities we also manufactured and tested a 2.2 GHz cavity from 'standard' small grain niobium.

Surface Treatment

All cavities were subjected to the following –equal– surface treatments:

- Degreasing in a soap/water solution with ultrasonic agitation for 30 min.
- Buffered chemical polishing (BCP), with a 1:1:1 solution of hydrofluoric, nitric and phosphoric acids at room temperature, removing approximately 100 μm of material. Because of the wire EDM cutting of the sheets, a hydrogen degassing at 800 $^{\circ}\text{C}$ for 3 hrs was performed. Subsequently additional BCP of ~ 100 μm at room temperature was done followed by thorough rinsing with ultra pure water and 30 min of high pressure rinsing (HPR) with a fan generating spray nozzle.
- After HPR each cavity was dried in a class 10 clean room for 2 hrs prior to assembly of the rf input and output coupling probes and evacuation on the test stand.

- After 12 hrs on the test stand the cavity vacuum had improved to the low 10^{-8} mbar range, and the cavity was cooled down to liquid helium temperature.
- After the initial tests the cavities were baked "in-situ" for ~ 40 hrs at 120 $^{\circ}\text{C}$ and re-tested.

RF Tests

Subsequent rf tests consisted of measuring the surface resistance between 4.2K and 2K and the dependence of the Q-value on accelerating gradient (Q vs. E_{acc}) at 2K or below. The following observations were made:

- The 1.5 GHz cavities from the large grain material (ingots A and B) reached accelerating gradients of $E_{\text{acc}} = 30$ MV/m and 32 MV/m, limited by quenches. In the absence of field emission, a Q-drop was observed which disappeared after "in-situ" baking. (Fig. 2).
- The single crystal cavities improved after "in-situ" baking to gradients up to 45 MV/m. Peak surface magnetic field up to $B_p \sim 185$ mT, which is considered to be the fundamental limitation of high purity niobium, was reached for about 300 ms. Again, a strong Q-drop was observed before baking with on-set value shifted towards higher surface magnetic fields, relative to the large grain results, and "in-situ" baking removed the Q-drop, but some field emission was detected.
- Residual resistances as low as 0.8 $\text{n}\Omega$ were measured on the single crystal cavity; this value degraded to about 8 $\text{n}\Omega$ after "in-situ" baking and makes us to believe that the optimum baking conditions for single crystal material might be somewhat different from the one used here.
- The 2.2 GHz cavity (HG shape) made from standard fine grain material also performed very well with comparable performance (Fig. 4). On the other hand, the Q-drop did not recover after baking.
- Energy gap value Δ/kT_c ranged between 1.72 and 1.82; no systematic deviation from the values observed on fine grain material was observed.

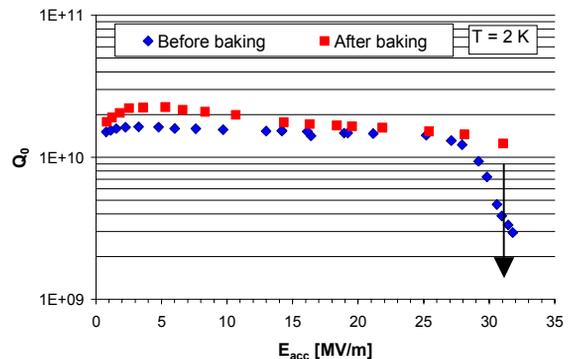


Figure 2: Performance of 1.5 GHz single cell cavity fabricated from ingot A niobium, before and after "in-situ" baking at 120 $^{\circ}\text{C}$ for 48 h. $E_p/E_{\text{acc}} = 1.77$, $B_p/E_{\text{acc}} = 4.47$ mT/(MV/m).

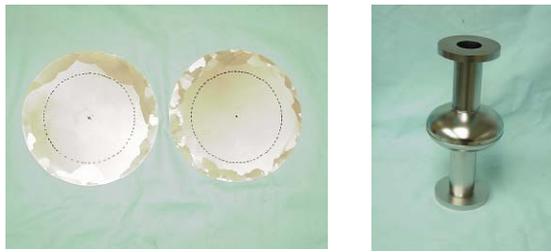


Figure 3: Single crystal niobium (ingot “A”) used to fabricate the 2.2 GHz cavity on the right side of the figure.

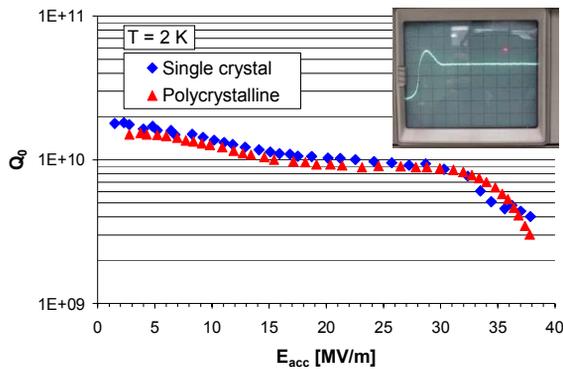


Figure 4: Performance of single crystal cavity from Figure 3 and identical polycrystalline cavity after “in situ” baking. As shown on the scope screen, the transmitted signal during the test of the single crystal cavity reached a value of 185 mT ($E_{acc}=43$ MV/m), but leveled off at ~ 162 mT after app. 300 msec.

Fig. 5 shows the field distribution of the LL-ILC cavity shape scaled to 2.36 GHz and the actual cavity.

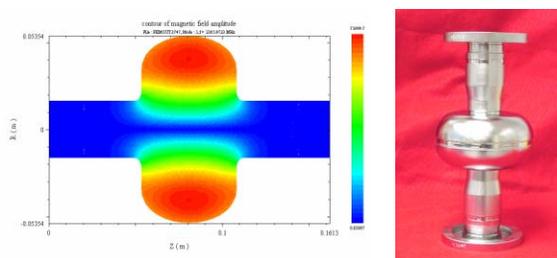


Figure 5: LL-ILC cavity shape with reduced surface magnetic field ratio: $B_p / E_{acc} = 3.56$ mT/(MV/m), E_p/E_{acc} increased to 2.07.

In the first tests a multipacting barrier was found at about $B_p = 71$ mT. After additional HPR, the cavity reached a gradient of 42.5 MV/m, limited by Q -drop and some field emission. After baking at 120 °C for a shorter time (24 h), the Q -drop recovered and the cavity reached $E_{acc} = 45$ MV/m (Fig. 6). After a few minutes at that gradient, an emitter switched on, lowering the quality factor due to field emission.

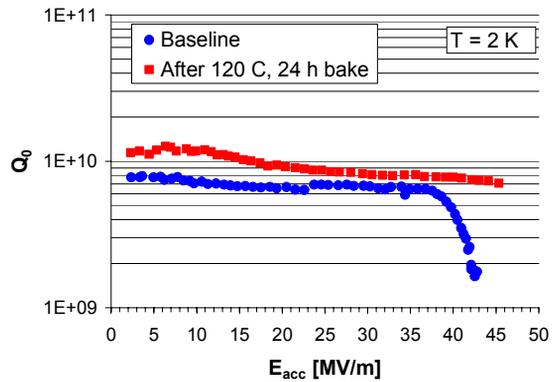


Figure 6: Test results from the LL_ILC single crystal cavity.

SUMMARY

Large grain or single crystal niobium seems to be an alternative to polycrystalline niobium for rf cavities with high performance. In our judgment, this material has the following potential advantages: it might be less expensive because of the elimination of the sheet fabrication process; the associated reduction in the rate of defect creation could eliminate laborious QA procedures such as eddy current scanning. After BCP the surfaces are very smooth and shiny; the measured surface roughness over an area of 0.2 mm x 0.2 mm was 27 nm, a factor of 10 smoother than electropolished polycrystalline material and 50 times smoother than bcp'd polycrystalline Nb [7]. Electropolishing as a final surface treatment step for high performance cavity might not be necessary. Also, the onset of the Q -drop might be shifted to higher gradients, which would eliminate the need for a lengthy “in-situ” baking process.

In the future we plan to test a multi-cell cavity of the LL_ILC shape, made from single crystal material.

ACKNOWLEDGEMENT

We would like to thank our colleagues from Jefferson Lab – L. Turlington, R. Manus, G. Slack, S. Manning and P. Kushnick – for their support of this work.

REFERENCES

- [1] B. Visentin, Proc. of the 11th Workshop on RF Superconductivity, Travemuende, Germany (2003) paper TuO01.
- [2] J. Knobloch et al., Proc. of the 9th Workshop on RF Superconductivity, Santa Fe, New Mexico (1999), p. 77.
- [3] J. Halbritter, Proc. of the 10th Workshop on RF Superconductivity, Tsukuba, Japan (2001), p. 292
- [4] H.R. Salles Moura, “Melting and Purification of Niobium”, Proc. Intern. Symposium Niobium 2001, Dec. 2-5, 2001, Orlando, FL, p. 147.
- [5] G. Ciovati, et al. JLAB-TN-01-015 (2001).
- [6] K. Ko et al, this conference.
- [7] A. Wu, private communications.