PERFORMANCE OF SINGLE CRYSTAL NIOBIUM CAVITIES*

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Abstract

We have fabricated and tested a total of six single cell niobium cavities, made from single crystal, high purity niobium. Two of the three cavities of the TESLA shape (1300 MHz) were made from Heraeus niobium by extending a smaller single crystal by rolling and annealing steps; the third cavity was made by spinning from CBMM material. The three other cavities of the scaled "Low Loss" (LL) shape (two) and "High Gradient" (HG) shape (one) resonated at 2.3 GHz and were fabricated from "as received" single crystals, both from Heraeus and CBMM niobium.

After appropriate surface treatments by buffered chemical polishing and electropolishing most cavities performed quite nicely and peak surface magnetic fields of ~ 160 mT or above corresponding to accelerating gradients between 38 MV/m and 45 MV/m were reached.

This paper reports about the performance of these cavities.

INTRODUCTION

High purity niobium sheets with RRR–values > 250 are exclusively used for the fabrication of high performance cavities for SRF accelerator projects/future projects such as SNS, XFEL, ILC, RIA, ERL's or the 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. By now it has been demonstrated [1] that cavities fabricated from high purity niobium sheets directly cut from a large grain ingot show performances comparable to those manufactured from polycrystalline material with the additional benefit of possibly being less expensive because of a "streamlined" process.

Some of the ingot material provided by the suppliers had very large single crystals, which could even be enlarged by appropriate mechanical and thermal treatments as discussed below. We have manufactured several cavities of different frequencies and shape from single crystals of two suppliers. Our interest was in exploring whether single crystal niobium performs superior in rf fields to large grain or polycrystalline material of equal purity. This investigation reports about our experiences with these cavities.

PROPERTIES OF INGOT MATERIALS

CBMM Niobium

Our initial research on large grain niobium started with two ingots (A and B of about 220 lbs each with a diameter of 9 ¹/₄ inches), produced by Companhia Brasileira de Metalurgia e Mineração (CBMM, Sao Paulo, Brazil) Ingot "A" had a very large single crystal of 7 inches diameter in its center, with a few smaller grains at the periphery as shown in Fig. 1, 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. From the center crystal of ingot "A" we fabricated two cavities of frequencies of ~ 2.3 GHz, the lowest frequency, which we could obtain with a single crystal sheet of 7" diameter. One cavity had the HG shape, whereas the second cavity was of the LL shape.

W.C.Heraeus Niobium

W.C. Heraeus has for a long time supplied high quality polycrystalline niobium of high purity (RRR grade) for rf cavity fabrication. Shortly after the successful tests on large grain niobium W.C. Heraeus also introduced high purity, large grain ingot material. However, as in the case of the CBMM material, the single crystals in the ingot were not large enough to be used for a 1.3 GHz, TESLAtype cavity.

At DESY a method was developed as described below to enlarge the single crystal by rolling and appropriate heat treatments to a sheet size adequate for 1.3 GHz halfcell deep drawing (265 mm in diameter) without destroying the single crystal [2]. Furthermore, by matching the crystal orientations of both half-cell single crystals during electron beam welding, the single crystal characteristics could be maintained [2]. The material used for the single crystal 1.3 GHz cavities is shown in Fig. 1.



Figure 1: W.C.Heraeus (left: large grain, center: enlarged grain) and CBMM (right) material used for the fabrication of single crystal cavities.

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Table 1: Properties of single crystal cavities. E_{peak}/E_{acc} is 2.07 for the ILC_LL shape, 1.67 for the HG shape and 1.9 for the TESLA shape. B_{peak}/E_{acc} is 3.56 mT/(MV/m) for the ILC_LL shape, 4.29 mT/(MV/m) for the HG shape and 4.31 mT/(MV/m) for the TESLA shape.

Cavity #	Supplier	RRR	Shape	Frequency
1	CBMM	280	HG	2.2 GHz
2	CBMM	280	ILC_LL	2.3 GHz
3 (1AC6)	CBMM (spun)	280	TESLA	1.3 GHz
4 (1AC8)	Heraeus (enlarged)	~470	TESLA	1.3 GHz
5	Heraeus (enlarged)	~470	TESLA	1.3 GHz
6	Heraeus	~470	ILC_LL	2.3 GHz

CAVITY FABRICATION

The 2.3 GHz cavities were fabricated by "standard" fabrication methods: deep drawing of half cells from single crystal sheets, electron beam welding (EBW) of beam-tube/beam-pipe subassemblies to half cells after trimming for EBW, mechanical grinding of half cells and EBW at equator weld. No attempts were made to match the crystals during equator welding.

As mentioned above, the single crystals for the 1.3 GHz cavities had to be enlarged to a diameter of 365 mm. The method developed at DESY has been reported elsewhere in detail [3]; here we give only a short summary of the essential results and steps:

- Defined enlargement of the disc's diameter is possible without destroying the single crystal structure of an existing configuration
- Appropriate heat treatment will not destroy the deformed single crystal.
- The single crystals keep their crystallographic structure and orientation after deep drawing and annealing at 800 °C.
- Two single crystals will grow together by EBW, if the crystal orientations are matched.

Once the single crystal discs had been prepared, the cavity fabrication proceeded as described above for the 2.3 GHz cavities, except for cavity #3, for which the half cells and the enlargement of the single crystal were accomplished by spinning. No problems during deep drawing of the enlarged crystals were encountered. Fig. 2 shows the different cavity shapes.



Figure 2: Single crystal cavities (left: TESLA-type, 1.3 GHz, center: HG, 2.3 GHz, right: ILC_LL, 2.3 GHz).

07 Accelerator Technology Main Systems

TREATMENT AND TESTING

The 2.3 GHz cavities were made from sheets, which were cut off the ingot by wire EDM. This process introduces large amounts of hydrogen into the material; therefore a hydrogen degassing heat treatment at 600 °C for 10 h was carried out after the removal of ~ 60 μ m from the surface.

The initial discs for the 1.3 GHz cavities were cut from the ingot with a diamond saw and received several heat treatments during the crystal enlargement. Therefore no additional heat treatment was done before the testing.

After the heat treatments different amounts of material were removed by BCP 1:1:1 and in some cases by EP. The cavities then followed the standard preparation procedures: high pressure ultrapure water rinse for ~ 1 h drying in a class 10 clean room for several hours, assembly and evacuation to below 3×10^{-8} mbar. During cool-down and testing the cavities were actively pumped with a turbo-molecular pump.

The cryogenic tests consisted of measuring the temperature dependence of the surface resistance between 4.2 K and 2 K and the Q_0 vs. E_{acc} at 2 K.

RESULTS AND DISCUSSION

The best cavity performances of all single crystal cavities are summarized in Table 2. Results from cavities #1 and #2 have been reported earlier [4]; the same is true for cavities #3 and #4 [2]. Since cavity #4 was the first cavity made from an enlarged single crystal by the process described above, we made a series of tests with successive material removal by BCP only in order to evaluate the depth of "damage" in the material. Whenever there was no field emission in the initial test, after the surface treatment, a test after "in situ" baking (120 °C for 12-48 h) followed. In Fig. 3 the evaluation of the gradient as a function of material removal depth is shown. The cavity had its best performance after ~216 µm (as determined by resonance frequency shift) material removal: $E_{acc} = 38.9 \text{ MV/m}$, corresponding to a peak surface magnetic field of $B_{peak} = 168 \text{ mT}$



Figure 3: Evaluation of gradient with material removal.

Cavity #	E _{acc} (MV/m)	B _{peak} (mT)	Q ₀ (B _{peak})	Rres (nΩ)	∆/kT _c	Treatment
1	38	162	4×10 ⁹	1.5	1.94	200μm BCP, 800°C 3h, HPR, 120°C 48h
2	45	160	7×10 ⁹	n.a.	n. a.	200μm BCP, 800°C 3h, HPR, 120°C 24h
3 (1AC6)	41	177	1.2×10^{10}	4.6	1.91	140μm BCP, 750°C 2 h, 110μm BCP, 120μm EP, HPR, 135°C 12h
4 (1AC8)	38.9	168	1.8×10^{10}	4.5	1.96	216µm BCP, HPR, 120°C 12h
5	38.5	166	7.6×10 ⁹	4.1	1.90	170µm BCP, HPR, 120°C 12h

Table 2: Summary of test results after baking.

Based on the experience with cavity #4, more material was removed from the surface of cavity #5 prior to the first test and less material removal steps were performed. After a removal of ~170 µm, the cavity reached Eacc=38.5 MV/m (Bpeak=166 mT). Both results are plotted in Fig. 4. Cavity #5 showed a different behavior than all other single crystal/large grain cavities after baking: the Q-drop did not disappear after 12 hours. The crystal orientation of the single crystals of this cavity was (110) with a tilt against the surface; for cavity #4 the crystal orientation was (100). The surface of both cavities appeared quite different after BCP: whereas cavity #4 exhibited a very smooth, shiny surface, the surface of cavity #5 was "rough" (orange peel/fish scale appearance) and less shiny. Obviously, there is a difference in the reaction of the BCP chemicals at different crystal orientations [5]. Cavity #3 (1AC6) achieved E_{acc} = 41 MV/m after large amounts of material removal by BCP $(250 \,\mu\text{m})$ and EP $(120 \,\mu\text{m})$ and baking at 137 °C for 12 h.

Cavity #6 performed rather poorly so far; the initially high Q-value at low field started to drop significantly with increased field in the cavity, indicating some heating of the surface. This behavior is not yet understood, especially since four large grain cavities from the same material batch performed very well [1]. We suspect that a manufacturing defect is presently limiting the cavity and further work is needed to understand the behavior and to improve the performance.



Figure 4: Q_0 vs. E_{acc} at 2 K for single crystal cavities #4 and #5 (best performance).

SUMMARY

The results from this set of cavities seem to indicate that single crystal niobium does not show superior performance in rf fields compared to large grain and polycrystalline niobium; therefore excluding intrinsic limitation due to grain boundaries in polycrystalline niobium. Presumably however, cavity performance can be influenced by crystal orientation and this subject needs to be investigated.

In addition, no difference in terms of maximum field was found between RRR 280 and ~500 cavities. The spread of data is possibly narrower and data are more consistent. BCP surface treatment produces good surfaces and generally – except for cavity #5 – "in-situ" baking at 120C for 12 h removes the "Q-drop". Three more single crystals are presently in preparation in an attempt to produce more statistics.

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