

HIGH-TEMPERATURE HEAT TREATMENT STUDY ON A LARGE-GRAIN NIOBIUM CAVITY*

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Abstract

Improvement of the cavity performance by a high-temperature heat-treatment without subsequent chemical etching has been reported for large-grain Nb cavities treated by buffered chemical polishing (BCP), as well as for a fine-grain cavity treated by vertical electropolishing [1]. Changes in the quality factor, Q_0 , and maximum peak surface magnetic field achieved in a large-grain Nb single-cell cavity have been determined as a function of the heat treatment temperature, between 600 °C and 1200 °C. The highest Q_0 improvement of about 30% at a peak surface magnetic field of 90 mT was obtained after heat-treatment at 800 °C – 1000 °C. Measurements by secondary ion mass spectrometry on large-grain samples heat-treated with the cavity showed large reduction of hydrogen concentration after heat treatment, compared to samples treated by BCP.

INTRODUCTION

A high temperature heat treatment (HT) at 600 °C for 10 h or 800 °C for 2 h in a ultra-high vacuum (UHV) furnace is part of the standard preparation procedure for SRF cavities made of bulk niobium. This treatment is done after cavity fabrication and heavy chemical etching to provide some stress-relief in the material and degas interstitial hydrogen from the bulk to avoid the so-called Q-disease [2]. Because of the re-absorption of residual gases in the furnace into the Nb cavity during cool-down to room temperature and possible surface contamination with foreign materials during HT, about 20 μm of Nb are typically removed from the inner cavity surface by either Buffered Chemical Polishing (BCP) or Electropolishing (EP) after the heat treatment. Nevertheless, measurements on Nb samples [3-5] show surface hydrogen concentrations of up to about 40 at.% after such chemical treatments. Such high concentrations are also to be expected from the theoretical point of view [6]. High concentrations of hydrogen near the surface can degrade the SRF properties of Nb, for example by increasing the residual resistance and therefore lowering the Q_0 of the resonator.

Initial studies were carried out at JLab to evaluate the performance of single-cell cavities after HT at 800 °C for 3 h, without subsequent chemical etching [1]. In some cases, a low-temperature (120 °C for 12 h) bake was also

included in the heat-treatment, after the hold-out at high temperature. Significant improvement of the Q_0 -value after HT without BCP/EP was measured in cavities made of large-grain niobium. Sample measurements by Secondary Ion Mass Spectrometry (SIMS) [1] revealed much reduced hydrogen content after heat treatment.

In this contribution we present the cavity test results and SIMS data on samples after heat-treatments at different temperatures and durations, in the range 600 °C – 1200 °C for 2 – 10 h to evaluate the parameters which would give the largest Q_0 improvements. Initial data have shown that lower residual resistance values can be already achieved using large-grain Nb, instead of fine-grain Nb, using standard cavity preparation procedures. Therefore we are focusing on this material to develop a cost-effective preparation procedure to obtain cavities with high Q_0 at peak surface magnetic field, B_p , of the order of 90 mT (accelerating gradients of about 20-25 MV/m). This would be beneficial for the use of new Continuous Wave (CW) accelerators for a variety of applications.

CAVITY PREPARATION AND TEST RESULTS

Cavity Preparation

The cavity used for this study is a 1.47 GHz single-cell of the original CEBAF shape and was made of large-grain Nb from Ningxia, China. This cavity was also used for the initial study of Ref. [1]. The treatment and test sequence was as follows:

- ~3 μm removal by BCP 1:1:2
- RF test (baseline 4)
- ~30 μm removal by Barrel Polishing followed by ~27 μm removal by BCP 1:1:2 and HT at 1000 °C/3 h + 120 °C/12 h
- RF test
- ~17 μm removal by BCP 1:1:2
- RF test (baseline 5b)
- HT at 1200 °C/2 h + 120 °C/12 h
- RF test
- ~16 μm removal by BCP 1:1:2
- RF test (baseline 6b)
- HT at 600 °C/10 h + 120 °C/12 h
- RF test

Common to the preparation steps mentioned above are: ultrasonic degreasing for 30 min prior to BCP or HT and after HT, High Pressure Rinse (HPR) with DI water for 30 min, after BCP or HT. After HPR the cavity is dried for ~3 h in a class 10 clean room and stainless steel blanks with pump-out port and RF antennae are assembled on the cavity flanges with In wire. The cavity is then evacuated

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to $\sim 10^{-8}$ mbar on a vertical stand and the thermometry system is attached. The RF tests consisted of measurements of $Q_0(2\text{ K}, B_p)$ and $Q_0(T, \sim 10\text{ mT})$. Temperature maps of the outer cavity surface were also taken during the RF tests.

The partial pressures of several gases are monitored with a Residual Gas Analyzer (RGA) during the HT and the average values of the three species with the highest partial pressure (H_2 , H_2O and CO) are shown in Fig. 1 for the different HT. The highest average partial pressure of hydrogen occurred during the HT at $1000\text{ }^\circ\text{C}$ and this is most likely due to the barrel polishing being done prior to the HT. It is known that large amounts of hydrogen are absorbed into Nb during barrel polishing [7].

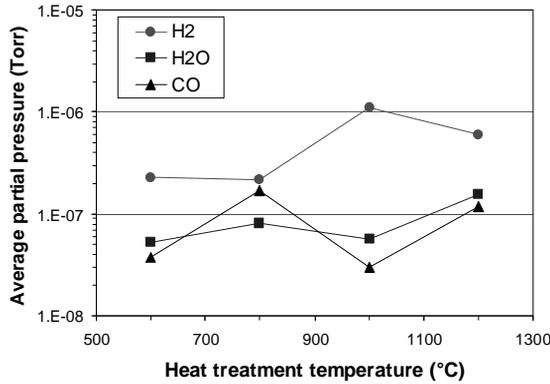


Figure 1: Average partial pressures of the main gases in the furnace during HT at different temperatures.

Cavity Test Results

All baseline cavity tests were limited by the high-field Q-slope (or “Q-drop”), except for “baseline 6b” test which was limited by quench at $B_p \approx 122\text{ mT}$. All tests after HT were limited by quench. No field emission was detected in any of the tests. The temperature maps showed that the quench locations changed after each HT and were located at $\sim 2\text{ cm}$ from the equator, with the exception of RF tests “baseline 6b” and subsequent HT. In that case, the quench location was the same, at $\sim 1\text{ cm}$ from the iris.

Optical inspection of the quench locations showed no outstanding features. Grain boundaries were visible at the quench locations after $1000\text{ }^\circ\text{C}$ and $600\text{ }^\circ\text{C}$ HT. Summary plots of all baseline tests and after HT are shown in Figs. 2 and 3, respectively.

The changes in the BCS surface resistance, R_{BCS} , at 4.3 K and $\sim 10\text{ mT}$, before and after HT, are shown in Fig. 4. In this and in the following figures, a data point before and after HT at $800\text{ }^\circ\text{C}$ for 3 h, followed by $120\text{ }^\circ\text{C}$ for 6-12 h, has been added as weighted average of “baseline 1” and “baseline 2” and of the corresponding “HT 1” and “HT 2”, as shown in Table III of Ref. [1]. A reduction of $R_{\text{BCS}}(4.3\text{ K})$ by almost 50% was obtained after HT at $1000\text{ }^\circ\text{C}/3\text{ h} + 120\text{ }^\circ\text{C}/12\text{ h}$. Fits of the surface resistance $R_s(T, \sim 10\text{ mT})$ with a computer program based on calculation of R_s using the BCS theory [8, 9] indicate that such reduction is due mainly to an increase of the energy

gap at 0 K , Δ/kT_c , from about 1.8 to about 2. T_c is the critical temperature, kept fixed at 9.25 K during the fit.

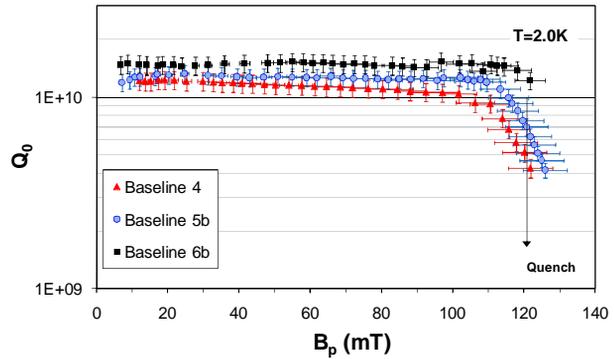


Figure 2: $Q_0(2\text{ K}, B_p)$ measured during baseline RF tests after BCP. The Q_0 -degradation at $\sim 110\text{ mT}$ is the so-called Q-drop

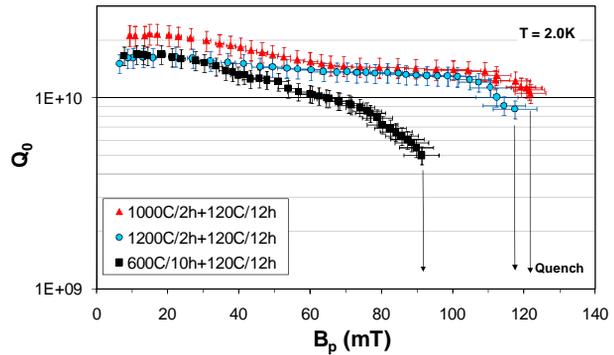


Figure 3: $Q_0(2\text{ K}, B_p)$ measured after HT. All tests were limited by quench.

Residual resistance values before and after HT are shown in Fig. 5. The maximum change from the baseline is about $2\text{ n}\Omega$. Figure 6 shows the values of the maximum B_p -value achieved during the RF tests at 2 K , which is $\sim 120\text{ mT}$ after BCP. The only significant change was a 25% reduction after HT at $600\text{ }^\circ\text{C}/10\text{ h} + 120\text{ }^\circ\text{C}/12\text{ h}$. Finally, Fig. 7 shows the change in $Q_0(2\text{ K}, 90\text{ mT})$ before and after HT. A $\sim 30\%$ improvement was obtained after HT at $800\text{ }^\circ\text{C} - 1000\text{ }^\circ\text{C}$.

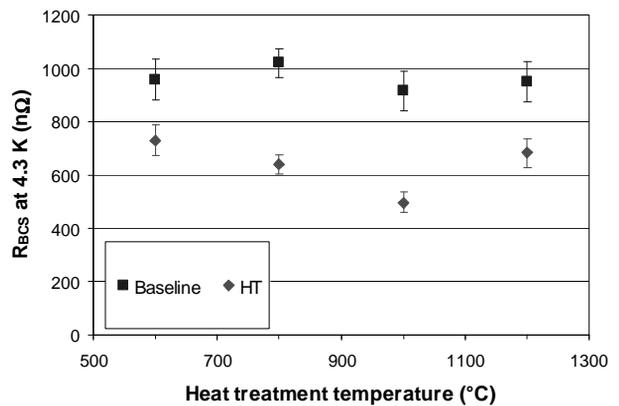


Figure 4: BCS surface resistance at 4.3 K and $\sim 10\text{ mT}$ measured before and after HT.

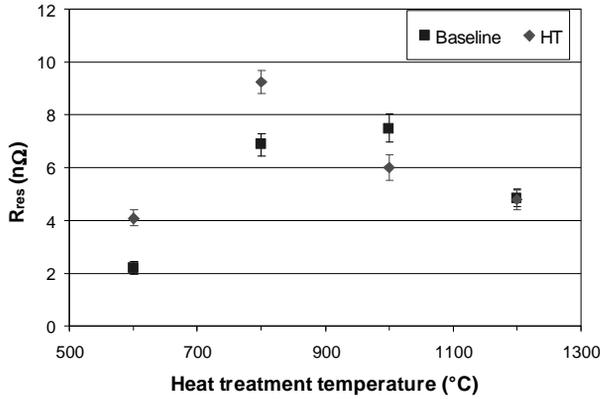


Figure 5: Residual resistance obtained from a curve fit of $R_s(T, \sim 10 \text{ mT})$ data before and after HT.

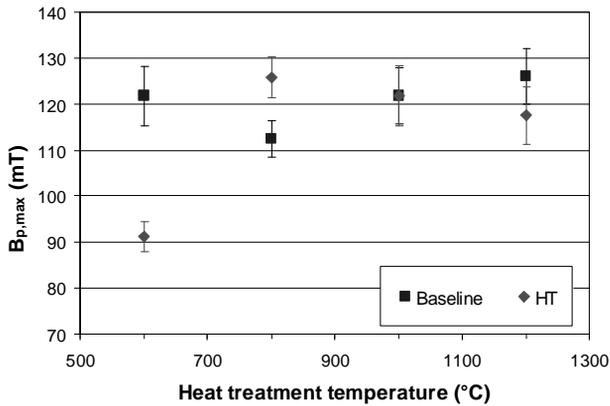


Figure 6: Maximum B_p -value measured at 2 K, before and after HT.

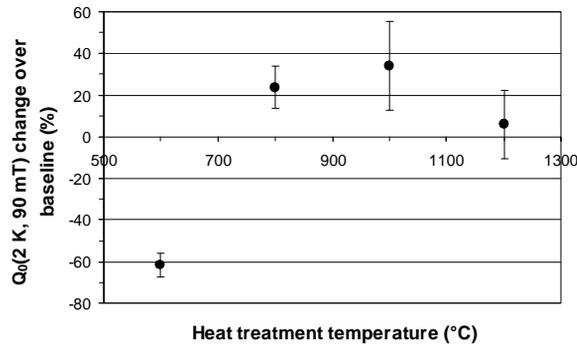


Figure 7: Change in $Q_0(2 \text{ K}, 90 \text{ mT})$ obtained before and after HT.

SAMPLE MEASUREMENTS

Samples with area $5 \text{ mm} \times 7.5 \text{ mm}$ cut from large-grain Nb discs from CBMM, Brazil, were prepared as described in Sec. V-B in Ref. [1]. Some samples were heat treated at different temperatures while others were kept as “control” samples, treated by BCP and without HT. The samples were analyzed by SIMS to measure the depth profile of impurities such as O, H, C, N within a $1 \mu\text{m}$ depth from the surface. This analysis was done using Cs^+ ion beam at

14.5 keV on a CAMECA IMS-6F. Because the furnace used for this study is sometimes used for post-purification of cavities in a titanium box at $1250 \text{ }^\circ\text{C}$, the depth profile of Ti in Nb was also measured to check for potential contamination. In this case the primary ion beam used was O_2^+ at 5.5 keV.

The concentration of C, N, and O and the ratio H/Nb for samples which underwent different HT and for a “control” sample, without HT, are listed in Table 1. Results on samples heat treated at $800 \text{ }^\circ\text{C}/3 \text{ h} + 120 \text{ }^\circ\text{C}/12 \text{ h}$ and $600 \text{ }^\circ\text{C}/10 \text{ h} + 120 \text{ }^\circ\text{C}/48 \text{ h}$ were already reported in Ref. [1].

Table 1: SIMS Measurements

Sample & Heat treatment	C (at.%)	N (at.%)	O (at.%)	H/Nb
L8: $800^\circ\text{C}/3\text{h} + 120^\circ\text{C}/12\text{h}$	0.036	0.005	0.010	0.8
L7: $600^\circ\text{C}/10\text{h} + 120^\circ\text{C}/48\text{h}$	0.013	0.005	0.014	1.4
L32: $1000^\circ\text{C}/3\text{h} + 120^\circ\text{C}/12\text{h}$	0.015	0.006	0.006	1.82
L37: $1200^\circ\text{C}/2\text{h} + 120^\circ\text{C}/12\text{h}$	1.000	0.09	13.910	0.6
L14: none	0.002	0.006	0.003	87

Figure 8 shows the depth profiles for Ti in samples L37, L32 and sample L16. Sample L16 had been heat treated at $800 \text{ }^\circ\text{C}/3 \text{ h}$, $400 \text{ }^\circ\text{C}/20 \text{ min}$ with N_2 , $120 \text{ }^\circ\text{C}/6 \text{ h}$. Ti contamination is significant in samples L37 and L32, heat treated at or above $1000 \text{ }^\circ\text{C}$, compared to L16, heat treated at a lower temperature.

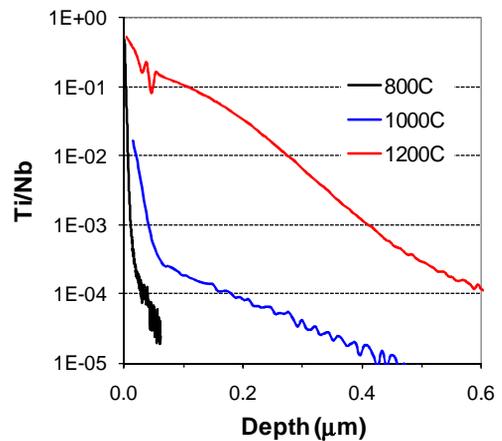


Figure 8: Depth profile of Ti in Nb samples heat treated at different temperature.

DISCUSSION AND CONCLUSION

The data shown in the previous sections indicate the possibility to improve the $Q_0(2\text{ K}, 90\text{ mT})$ of an SRF cavity made of large grain Nb by about 30% by a heat-treatment in UHV at 800 °C – 1000 °C followed by a 120 °C/12 h bake, without subsequent chemical etching. The reason for this optimum temperature range is unclear. The choice of the HT temperature and duration for this study was based on achieving similar quantities of desorbed hydrogen, so that HT at higher temperatures was carried out for shorter time. Improvements of the Q_0 -value after heat treatment at 800 °C/2 h and 1250 °C/3 h, without subsequent chemical etching, had been obtained also on single cell cavities made of single-crystal Nb [10]. The origin for the performance degradation after 600 °C/10 h + 120 °C/12 h is unclear and similar results were obtained after this heat treatment was tried on the same cavity during the initial study, as shown in Fig. 8 of Ref. [1].

While it is well known that a reduction of $R_{BCS}(4.3\text{ K})$ up to 50% is obtained by baking a BCP-treated cavity at 120 °C-145°C/48 h [4, 11], a reduction of $R_{BCS}(4.3\text{ K})$ by about 25 % was achieved already by HT at 800 °C/3 h, without 120 °C baking [1]. In this study, the 120 °C baking in the furnace was added to the high temperature HT mainly to reduce the Q-drop above 90 mT.

Sample measurements show significantly reduced hydrogen concentration after HT, independently of HT parameters. Sample measurements also indicate that HT at temperatures greater than 1000 °C lead to Ti contamination from the furnace. During the cavity HT at 1000 °C and 1200 °C, the cavity openings were partially closed with Nb foils to mitigate this issue. The origin of the quenches at ~120 mT after HT is also unclear.

It is to be expected that the cavity performance depends on the “cleanliness” of the furnace and that different results from the ones presented in this contribution may be obtained with a different furnace. We are presently commissioning a new induction furnace at JLab which will be dedicated to further HT studies on large-grain single-cell cavities. The heat treatment study reported in this contribution will be repeated with this new furnace. We expect the hydrogen partial pressure in this new furnace to be about one to two orders of magnitude lower than in the current furnace. The new furnace is in a class 10,000 environment. As the furnace contamination with foreign elements, other than Nb, will be controlled and minimized with the new furnace, better reliability of cavity performance after HT is expected, compared to the production furnace which was available for the initial studies.

The successful development of this treatment could result in large-grain Nb being the material of choice for SRF cavities for CW applications.

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