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INVESTIGATION OF HIGH TEMPERATURE BAKING OF JACKETED QUARTER WAVE RESONATORS

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Abstract

The Superconducting booster Linac at IUAC has been delivering accelerated ion beams for scheduled experiments since 2013 [1, 2]. It has three accelerating modules with eight Quarter Wave Resonators (QWR) ($\beta_{opt}=0.08$) in each module. The QWRs for the first module were built at Argonne National Laboratory while those for the second and third modules have been built in-house. During the electropolishing of one of the indigenously built resonators (QWR # I03) the RF surface got spoiled due to a wrong mixture of acid that was meant for etching. In subsequent cold tests of the cavity, its performance was poor (2.6 MV/m @ 4 W). There was evidence of Q disease also, as the performance deteriorated further (~20%) when the cavity was held at 100-120 K for ~8 hours. In an attempt to recover the cavity, it was baked at 650 °C for 10 hours along with its outer stainless steel jacket. A series of tests were conducted thereafter, wherein a substantial improvement (A factor of two) in the performance was observed. Encouraged by these results, another QWR designed for a lower beta ($\beta_{opt}=0.05$) was also heat treated identically. This improved its performance in a similar fashion. This paper presents the different treatments followed to enhance the cavity performance vis-à-vis the test results.

INTRODUCTION

The Superconducting Linac at IUAC, which serves as a booster to the 15 UD Pelletron accelerator [3], uses Niobium Quarter Wave Resonators (QWR) as the accelerating element. It has a total of 27 QWRs installed in 5 cryostats. These are, a Superbuncher cryostat housing one, three Linac cryostats each housing eight and a Rebuncher cryostat with two QWRs respectively. Figure 1 shows the schematic of the Pelletron-Linac system at IUAC.

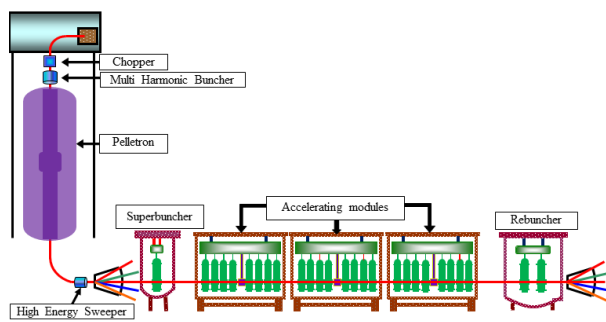


Figure 1: 15 UD Pelletron-Linac system.

The QWR was designed and developed in collaboration with Argonne National Laboratory (ANL) [4, 5]. The first batch of 12 QWRs were built at ANL. Resonator fabrication facilities [6] were thereafter setup at IUAC and the remaining QWRs for the Linac were built in-house. These facilities include an electron beam welding machine, a surface treatment lab and a high vacuum annealing furnace. The first indigenous QWR (QWR # I01) was fabricated using the Niobium sub-assemblies left over from the ANL project. This was completed in the year 2003. The resonator achieved an accelerating gradient of 5 MV/m at 6 W of input power at 4.2 K, surpassing the design goal of 4 MV/m @ 6 W. It was subsequently installed in the Linac. Two more cavities (QWR # I02 and I03) were thereafter fabricated to freeze the fabrication algorithm and develop confidence in the indigenous fabrication procedures. QWR #I02 was successfully tested offline and was installed in the Linac. QWR # I03 was also tested offline after a light Electropolishing (EP) and the results were promising. To further improve the cavity performance it was given another round of EP. During the process it suffered an accident which resulted in an uneven etching of the RF surface. In a subsequent test the cavity performance was very poor and there was significant frequency detuning. The resonator was henceforth used as a test piece for doing microphonics measurement and other developments at room temperature.

QWR # I03 HISTORY

QWR # I03 was one of the first cavities that could be termed as a complete indigenous fabrication in the sense that all the niobium sub-assemblies were built in-house. Post fabrication, the cavity was given a light EP (~20µm) (this was besides ~250 µm of EP done during fabrication). In the subsequent cold test it achieved a gradient of 3.1 MV/m @ 3.5 W ($Q=1.8 \times 10^8$) @ 4.2 K. Q measurements could not be done at higher powers due to shortage of LHe supply. It was decided to EP the cavity to remove another ~50 µm from the surface before installing it in the Linac. During the second EP, as soon as the electrolyte was transferred into the cavity, the acid temperature shot up to ~55 °C and spontaneous uncontrolled etching of the surface started. This was accompanied with the emission of brown fumes (possibly NO₂). All these symptoms pointed towards the contamination of the EP electrolyte with HNO₃. The contaminated electrolyte was immediately pumped out but the damage had already been done. The RF surface became very rough (almost like scales of a fish), as shown in

Figure 2, with the grain boundaries prominently visible. The presence of HNO_3 in the EP mixture (which contains 98% H_2SO_4 and 40% HF in a volume ratio of 85:15) was later confirmed by chemical analysis wherein, 1.3 % by mass of HNO_3 was found in the electrolyte.



Figure 2: RF surface of QWR # I03 post EP accident.

The resonator was thereafter electropolished to remove $\sim 50 \mu\text{m}$ as decided earlier. This however, failed to improve the surface roughness. In the subsequent cold test the cavity performance was very poor. It achieved a gradient of 1.2 MV/m @ 16 W of power ($Q = 5.9 \times 10^6$).

INVESTIGATIONS AND CORRECTIVE ACTIONS

Attempts were made to recover the performance of the cavity, wherein it was decided to give it a high temperature bake in the vacuum furnace. The logic behind this conclusion was that probably there was a large amount of hydrogen up-take during the acid accident which was resulting in the formation of hydride phases during cooldown. The exposed grain boundaries were further aiding in the process by providing nucleation sites for the niobium hydrides. The cavity was first tested after cleaning it with warm ultrapure ($18 \text{ M}\Omega\text{-cm}$ at $55 \text{ }^\circ\text{C}$) water in a high frequency (68 kHz) ultrasonic bath for $\sim 1 \text{ hr}$. The aim of this test was to establish a baseline performance of the cavity. It achieved a gradient of 2.6 MV/m at 4 W ($Q = 1.1 \times 10^8$). This was surprisingly a substantial improvement from the test conducted just after the accident. The reason for this is not clear, save perhaps the fact that the cavity was thoroughly rinsed in warm ultrasonic bath followed by low pressure DI water, before the test. In the previous test (just after the accident) the ultrasonic cleaning was not done. Nevertheless, the accelerating field was still $\sim 40\%$ lower than that obtained before the accident. Furthermore after the initial measurements, wherein, the cooldown was done at a fast rate (cavity temperature fell from 200 K to 40 K in $\sim 45 \text{ mins}$), the cavity was warmed up and held at the temperature of $\sim 100 \text{ K}$ for 8 hrs before it was cooled again to

4.2 K (at the same rate as before) for another set of Q measurements. There was a $15\text{-}20\%$ deterioration in the cavity Q (Q at 4 W decreased to 8.8×10^7 from an earlier value of 1.1×10^8). The Q recovered once the cavity was warmed up to 300 K and rapidly cooled back to 4.2 K , confirming the presence of ‘ Q disease’.

After establishing a baseline performance of the cavity it was put in the High Vacuum Furnace (HVF) for the bake-out. In preparation for the procedure the annular space between the outer SS jacket and the inner Niobium housing, as shown in Figure 3, of the cavity was thoroughly cleaned in an ultrasonic bath using soap solution followed by analytical grade Iso-Propyl alcohol.

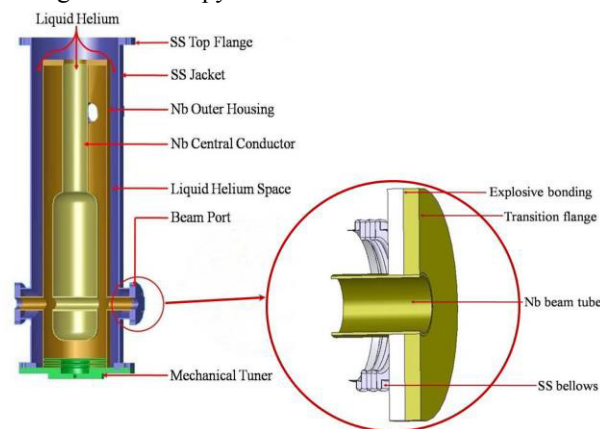


Figure 3: Schematic of IUAC QWR.

The cavity was the loaded in the HVF (Figure 4) and baked at a temperature of $650 \text{ }^\circ\text{C}$ for 10 hrs . The temperature was not increased beyond $650 \text{ }^\circ\text{C}$ for the fear of developing leaks in the SS jacket especially in the SS bellows which are installed in the four ports to take care of the differential contraction between the SS and niobium (see Figure 3).

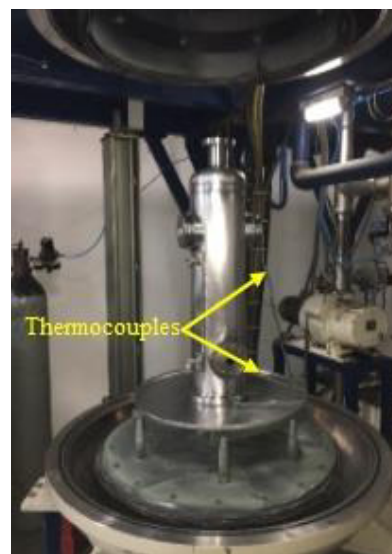


Figure 4: QWR # I03 being loaded in HVF.

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The partial pressure of hydrogen inside the furnace chamber was monitored and the baking time was adjusted to allow the pressure to fall to the background ($<1e-9$ mbar). Figure 5 shows the RGA spectrum during the baking cycle.

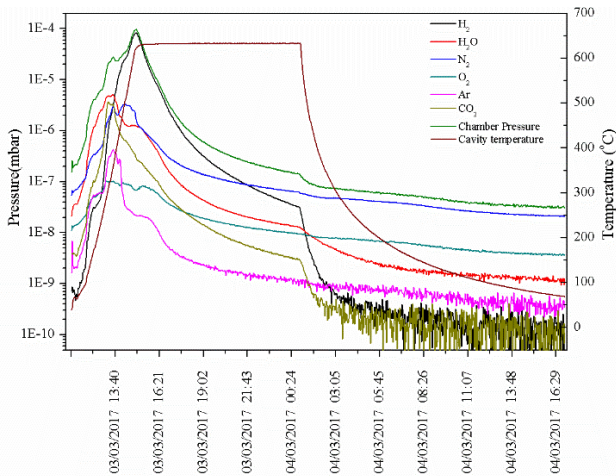


Figure 5: RGA spectrum during 650 °C bake of QWR#103.

After the bake cycle the cavity was given a high frequency ultrasonic rinse in warm ultrapure water as before and loaded in the test cryostat. Q measurements were done after a fast cooldown (cavity temperature fell from 250 K to 20 K in ~1hr.) to 4.2 K. A substantial improvement in the performance was observed. The accelerating gradient of the cavity increased to 3.86 MV/m at 4 W ($Q = 2.51 \times 10^8$) from its pre bake value of 2.6 MV/m at 4 W ($Q = 1.1 \times 10^8$). To rule out the possibility of any Q degradation due to hydride formation, the cavity was warmed up and held in the temperature zone of 95-110 K for ~ 8 hrs. Thereafter, it was cooled to 4.2 K and another set of Q measurements were taken. Surprisingly the quality factor showed further improvement. The cavity field increased by ~12% to 4.33 MV/m at 4 W ($Q = 3.16 \times 10^8$). The reason for this anomalous increase is not clear. Figure 6 shows the Q curves for QWR # 103 before and after the 650 °C bake.

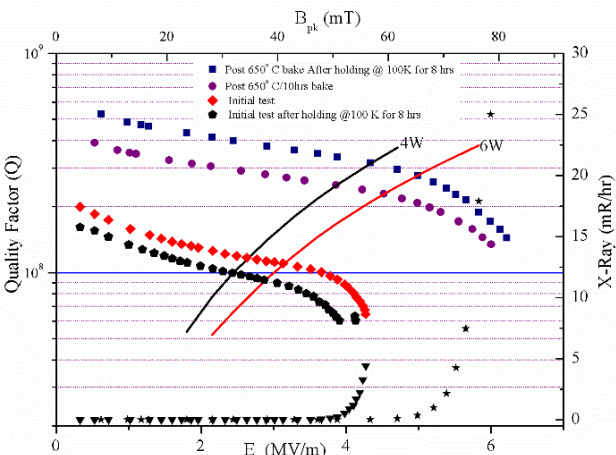


Figure 6: Q curves for QWR # 103 pre and post 650 °C bake.

HIGH TEMPERATURE BAKING OF $\beta=0.05$ QWR

Encouraged by the promising results obtained with QWR # 103, it was decided to repeat the process on a QWR of a different design. This resonator, which has a $\beta_{opt}=0.05$ and the same operational frequency of 97 MHz, has a fully indigenous electromagnetic design and fabrication [7]. This was prototyped for use as a velocity matching structure with the High Current Injector (HCI) [8] to the superconducting Linac. The HCI project is presently under development at IUAC. In the baseline test of the cavity at 4.2 K it achieved a gradient of 8.3 MV/m at 4 W ($Q = 2.73 \times 10^8$). It was thereafter baked at 650 °C for ~10 hrs and processed in an identical manner as QWR # 103. Subsequent cold test indicated a significant performance enhancement. The accelerating field in the cavity increased to 9.79 MV/m at 4 W ($Q = 3.79 \times 10^8$). In-fact, the improvement in the quality factor at lower fields was even more significant (Q at 2.25 MV/m increased by a factor of ~2.5 from 3.94×10^8 in the baseline test to 1.03×10^9 after baking). Increased field emission compared to the baseline test was responsible for the fall in the Q value at higher fields. This is evident from the X-Ray data in the Q curves of the cavity shown in Figure 7. It is planned to give a high pressure rinse to the cavity, for which the high pressure wand and its movement mechanism is being modified. This will most likely reduce the Q slope at higher fields.

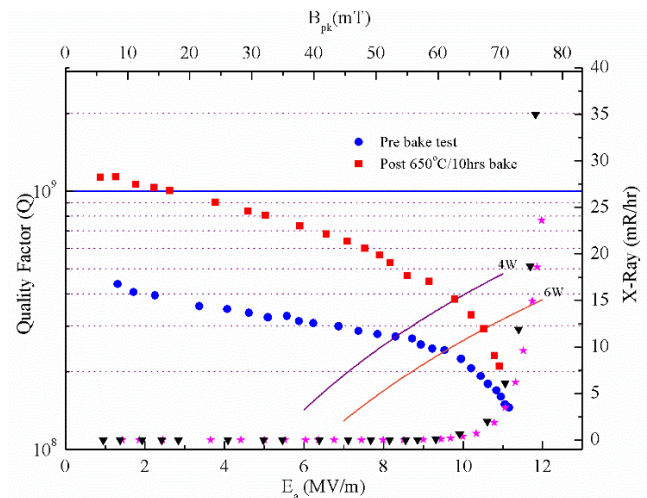


Figure 7: Q curves for $\beta=0.05$ resonator pre and post 650 °C bake.

CONCLUSIONS

The results shown in the previous sections indicate that a high temperature bake @ 650 °C does lead to an improvement in the quality factor @ 4.2 K. It also makes the cavity immune to the losses due to hydride precipitation during cooldown. Most of the baking studies in the past have been done on elliptical cavities operating in the 2 K regime, where a significant reduction in the BCS resistance (R_{BCS}) has been reported [9]. This result, is however, not applicable to low frequency cavities, such as the ones used at

IUAC, which operate at 97 MHz at 4.2 K. For them the total surface resistance is predominantly residual resistance, as the R_{BCS} component is very small. Quoting numbers, the total surface resistance of the $\beta=0.05$ cavity at 2.2 MV/m ($B_{pk}=14$ mT) is ~ 41 n Ω and ~ 16 n Ω respectively pre and post 650 °C bake whereas the pre-bake R_{BCS} is ~ 3 n Ω only. Improvement in the quality factor after baking is therefore, due to a reduction in the residual surface resistance. Removal of hydrogen also cannot fully account for the Q improvement, as the degradation in Q due to hydride precipitation is significantly less than the improvement observed after baking.

As a concluding remark, it is worth mentioning that cleanliness of the cavity surfaces (specially the annular liquid helium space, since these are jacketed resonators) and of the HVF are imperative in achieving good results. This gains even more importance since no electropolishing is done after baking and the resonators are only rinsed with ultrapure water.

More studies have been planned in the future, with niobium samples, to gain a better understanding of the phenomenon. It has also been planned to do Nitrogen doping [10] in these resonators and see its effect on the performance.

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