ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THXA08

# REVIEW OF HEAT TREATMENTS FOR LOW BETA CAVITIES: WHAT'S SO DIFFERENT FROM ELLIPTICAL CAVITIES

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*Abstract*

Heat treatments done for low beta (low frequency) cavities are usually, due to the lack of feedback, inspired from elliptical (high frequency) cavity results.

Is that still relevant now that experimental data are available thanks to the florishing business of low beta structures (Spiral2, ESS, FRIB, C-ADS, MYRRHA, PROJECTX, ...).

These two families are moreover not usually operating in the same resistance regime (BCS and residual).

The paper will review procedures applied and results obtained on different type of cavities (Quarter-Wave resonator, Half-Wave resonator and Spoke) and different temperature treatments (low temperature baking, hydrogen degassing, nitrogen doping, ...) and compare these to elliptical cavities.

## INTRODUCTION

Heat treatments and specifically hydrogen degassing have been applied to L-band elliptical cavities to avoid severe irreversible degradations (see Figures 1 and 2) of the quality factor due to the precipitation of hydrogen (called Q-disease or 100K effect). This treatment typically done at 800°C during 3h under vacuum was compulsory to achieve performance requirements in terms of power dissipations ($Q_0$ > 1E10) at accelerating gradients targeted by superconducting linac projects ($E_{acc}$ > 20 MV/m).

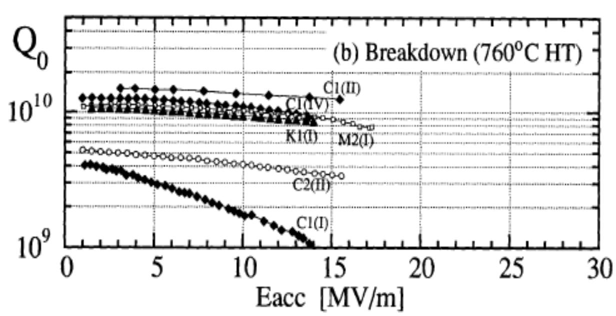


Figure 1: Study of the effect of heat treatments done on elliptical cavities at KEK in 1993. Curve C1(I) and Curve C1(II) represent respectively the quality factor at 2K versus accelerating gradient of a non-degassed and 760°C degassed cavity (figure extracted from [1]).

The heat treatment is followed by an Electro-Polishing (EP) or Buffered Chemical Polishing (BCP) of few tens of microns to remove the surface polluted by the residual gas re-absorbed by bulk Niobium at the end of the process. Indeed, at these temperatures, the oxide barrier is dissolved allowing any pollution to diffuse into the bulk.

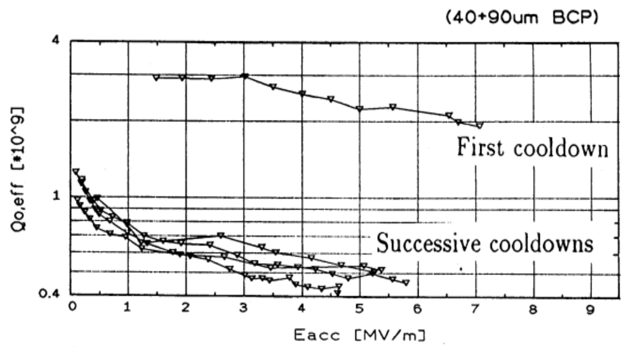


Figure 2: Irreversible degradation caused by hydrogen precipitation of an elliptical cavity after successive cooldown (figure extracted from [2])

Once this treatment optimized and part of the standard preparation of cavities, performances were then limited by what has been called the High Field Q-slope (HFQS). This very abrupt $Q_0$ degradation has been investigated for long. Even though the origin of this phenomenon is not totally understood, a cure has been reported by B. Visentin in 1998 [3]. Baking a cavity at 170°C during 70h would not only mitigate the HFQS but also decrease the BCS resistance as visible on Figure 3. Optimization of the baking treatment made it converge at 120°C during 48h.

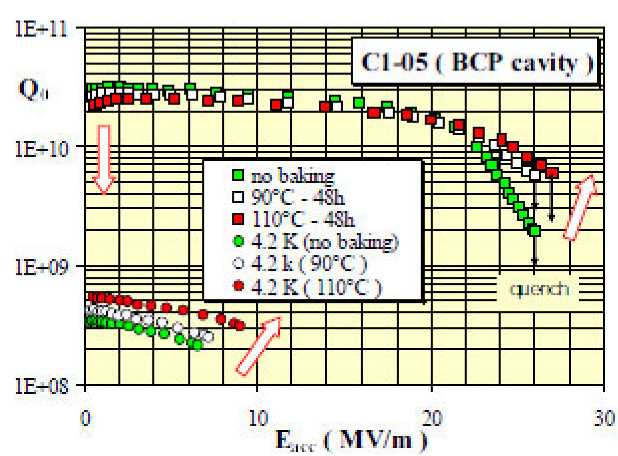


Figure 3: Improvement of the BCS resistance with a 110°C baking during 48h. A $Q_0$ improvement at 4.2K is the sign of reduction of BCS resistance. A $Q_0$ degradation at 2K is sign of residual resistance increase (figure extracted from [4]).

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As a final improvement, people were working hard in the 1.3 GHz community to keep on improving the quality factor at medium field (~ 15 MV/m) as new CW machines were required (for example LCLS2). Fermilab reported in 2013 a new treatment called nitrogen doping. It consists in venting the furnace with nitrogen gas at the end of the 3h at 800°C degassing for few minutes and then let it diffuse for few minutes after good vacuum conditions are recovered [5]. The real gain is revealed after a light chemical etching to remove the “over-doped” layer. The quality factor was actually surprisingly increasing with field (anti Q-slope) up to 7E10 at 2K as seen on Figure 4 but at the expense of a severe reduction of the quenching gradient (~ 25 MV/m) and a higher sensitivity to residual magnetic field [6].

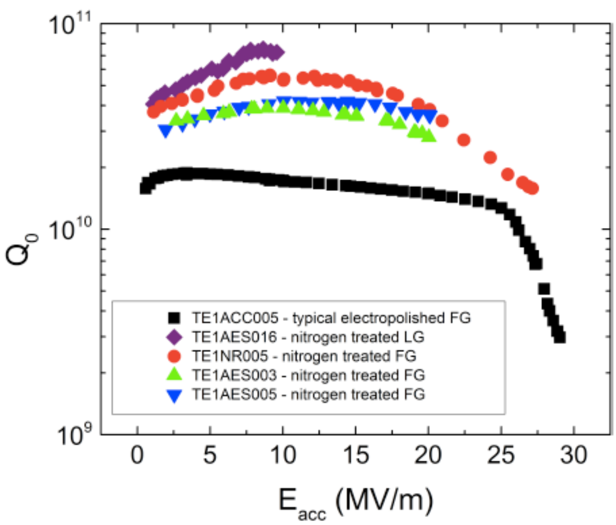


Figure 4: Improvement of the quality factor at 2K thanks to nitrogen doping. In black, the typical Q-curve of a cavity treated with standard recipe (figure extracted from [5]).

It has to be mentioned that in 2001, B. Visentin already reported an anomalously low BCS resistance when a degassed cavity at 800°C under vacuum is refilled with nitrogen gas at the end of the process [7]. However, he didn’t mention any anti Q-slope. In a different way, G. Ciovati reported in 2010 that the passivation of Niobium surface at temperature below 400°C with nitrogen gas directly following the hydrogen degassing without any subsequent chemical etching could improve significantly the $Q_0$ [8]. A process very similar to the so-called nitrogen infusion [9].

Because of the lack of statistics due to the fabrication and cryogenic testing costs of the cavities, the low beta community is applying the optimized heat treatments as presented above. This gives a good opportunity to study qualitatively the differences in behaviour at different frequencies. This paper aims at reviewing but not extensively all the improvements done after hydrogen degassing and low temperature baking.

## HYDROGEN DEGASSING OF LOW BETA CAVITIES

Contrary to elliptical cavities for which a hydrogen degassing is compulsory to achieve good performances, low beta cavity can be operated at 4.2K as demonstrated by several superconducting linacs like ISAC2 (TRIUMF), ALPI (LNL), Spiral2 (GANIL) and SARAF (SOREQ). This statement appears to be wrong for 352 MHz Spoke cavities as depicted in Figure 5. A simple room temperature cycle after a cryogenic test is enough to degrade permanently the $Q_0$ and its dependence with field [10, 11].

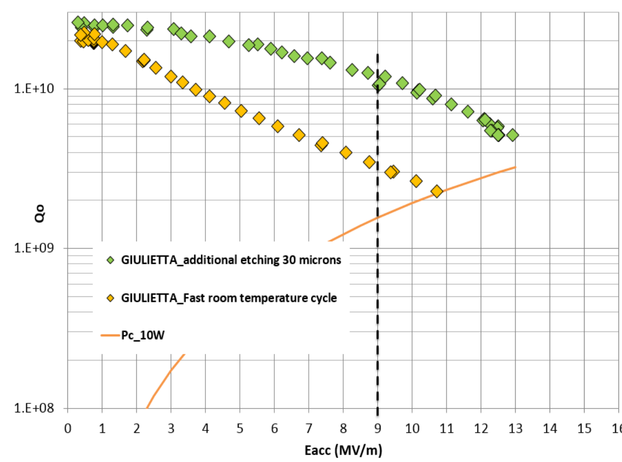


Figure 5: Degradation of the $Q_0$ visible at 2K after a room Temperature cycle of a double Spoke resonator for ESS.

Heating parameters have not been re-optimized as thoroughly as for 1.3 GHz elliptical cavities. Same parameters or scaled parameters (temperature and time) have been applied considering additional constraints as, for example, the presence of a titanium tank or brazed stainless steel parts.

The aim of this paper is to show examples of degassed cavities and more particularly those which have data available before and after treatment. Many low beta cavities are degassed by default (ATLAS upgrade, C-ADS or FRIB) but unfortunately no before/after comparison could be done.

### *ANL Experience with Spoke Cavities*

In 2005, ANL reported 2 Triple Spoke Resonators (TSR) [12] with a $\beta = 0.5$ and 0.63 treated in the furnace of Fermilab. The cavities have been degassed at 600°C during 24h [13]. This limitation in temperature is coming from the presence of brazed stainless steel parts on the cavity that wouldn’t withstand an 800°C heat treatment. A clear improvement of the low field $Q_0$ is noticeable at 4.2K and 1.9K, sign of the reduction of both residual and BCS resistances (See Figure 6). Moreover, the $Q_0$ degradation with field is significant before treatment whereas it becomes negligible afterwards.

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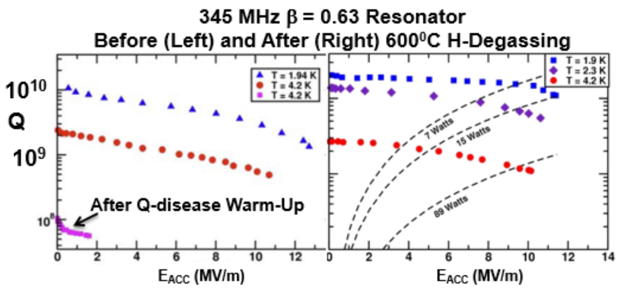


Figure 6: $Q_0$ versus accelerating gradient before (left) and after (right) hydrogen degassing at 600°C. At 4.2K, surface resistance is dominated by BCS contribution and is decreased of 50%. At 1.9K, residual contribution is divided by a factor of 2 (figure extracted from [13]).

## *IPNO Experience with Spoke Cavities*

A new furnace dedicated to heat treatments have been recently commissioned at IPNO. After several RRR measurements and SIMS (Secondary Ion Mass Spectrometer) analysis of Niobium samples showing a very limited pollution, 2 Double Spoke Resonators (DSR) for ESS [14] and one single Spoke for MYRRHA [15] have been degassed at 600°C during 10h. The same temperature limitation was experienced on these cavities for the same reasons as ANL TSR. The additional constraint of a titanium tank installed on the cavities implied to improve the cavity preparation. Indeed, the helium space in between the tank reservoir and the cavity has to be thoroughly cleaned. Additional cleaning has been performed in ultrasonic bath with a degreasing agent followed by ultra-pure water rinsing. Moreover, to limit pollution, Niobium caps have been installed on all cavity ports.

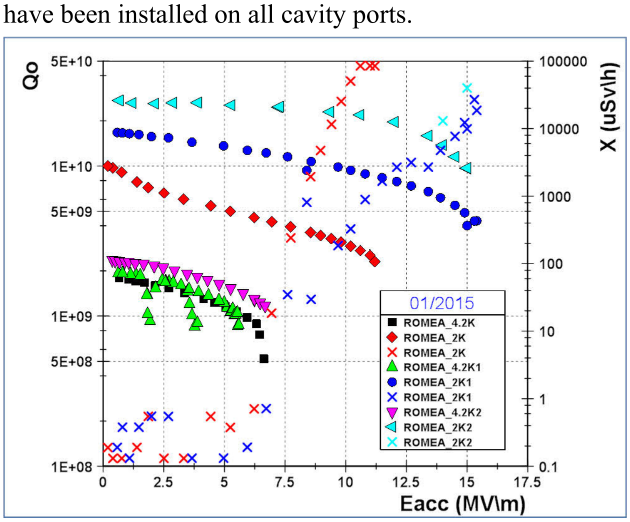


Figure 7: $Q_0$ factor at 2K versus accelerating gradient of an ESS DSR prototype after a warm BCP etching inducing hydrogen pollution (red diamonds), after a cold BCP etching (blue circles) limiting hydrogen pollution and finally after hydrogen degassing at 600°C during 10h followed by a 30 microns BCP etching (blue triangles). Respectively at 4.2K, black squares, green triangles and purple triangles. Presented at [16].

The ESS Double Spoke cavity has undergone a post BCP etching of 30 µm to remove any polluted layer, following the standard recipe of the community. As depicted on Figure 7, the $Q_0$ has been significantly improved at both 4.2K and 2K showing an improvement of both residual and BCS resistance.

As part of a European R&D program named MYRTE [17], the Single Spoke Resonator (SSR) and because people of the 1.3 GHz community were reporting that potentially no post heat treatment chemical etching was required if the cavity was equipped with Niobium caps covering all the cavity ports, this solution was then envisaged as this would simplify drastically the preparation procedure of cavities. Figure 8 shows that $Q_0$ is effectively improved even without any post BCP etching. However, the cavity was loaded with very strong field emission. This was attributed to the presence of iron detected by SIMS analysis done on a sample installed in the cavity beam tube during the heat treatment [16]. This polluted layer is too thin to deteriorate the $Q_0$ but increases significantly the Secondary Emission Yield (SEY) of the surface. An additional BCP etching of few microns helped to eliminate any problem of field emission but at the expense of a reduction of $Q_0$.

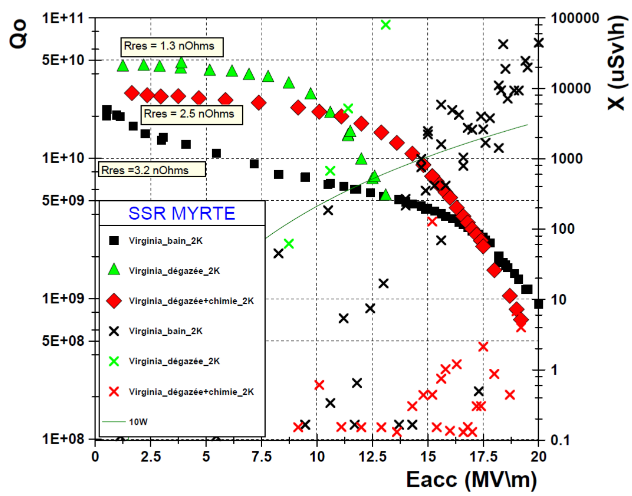


Figure 8: $Q_0$ factor at 2K versus accelerating gradient of an MYRRHA SSR prototype before hydrogen degassing (black squares), after 600°C degassing during 10h without post BCP etching (green triangles) and with post BCP etching (red diamonds). Field emission is represented by crosses with corresponding colours. Presented at [16].

## *CEA Experience with Low Beta Elliptical Cavities*

CEA Saclay is in prototyping and R&D phase for the β=0.67 6-cell elliptical cavity operating at 704 MHz for ESS project [18]. Vertical tests to assess cavity performances have been done before heat treatment. Although an intensive study for BCP etching optimization have been carried out, this cavity couldn't meet the specifications because of an anomalously high residual resistance, a behaviour very similar to 1.3 GHz cavities. After degassing the cavity at 600°C during 10h the $Q_0$ has been nota-

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bly improved (See Figure 9). It has to be mentioned that the cavity has not been equipped with Niobium caps during the process causing a surface pollution. This can explain why the $Q_0$ is improved after an additional BCP etching contrary to the previous example of MYRRHA.

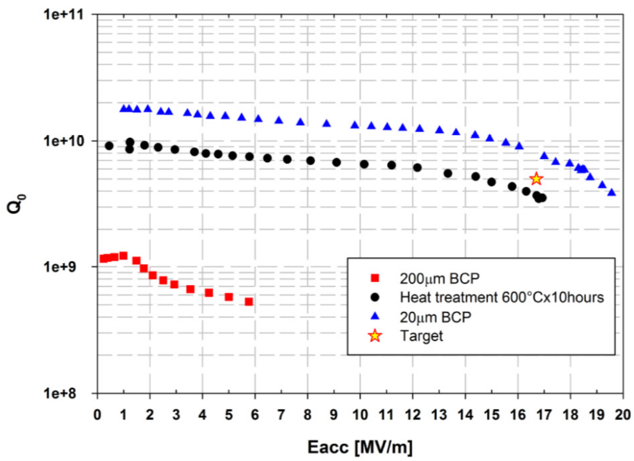


Figure 9: $Q_0$ at 2K versus accelerating gradient of the low beta elliptical cavity for ESS project before hydrogen degassing (red squares), after degassing (black circles) and after additional BCP etching (blue triangles). Figure extracted from [18].

To summarize about hydrogen degassing:

- There is no real difference between low beta and 1.3 GHz elliptical cavities in term of benefit even though this treatment looks to be compulsory for resonators operating above 300 MHz at 4.2K. We showed here that Spoke resonators at 352 MHz have to be degassed for operation at 2K.
- Both the residual and the BCS resistances are improved
- The medium field Q-slope is mitigated.

## LOW TEMPERATURE BAKING OF LOW BETA CAVITIES

Low temperature baking at 120°C showed on elliptical cavities very interesting benefits. This rather simple treatment allows to reduce drastically both the HFQS and the BCS resistance. This looks very interesting for low beta cavities operating in the BCS regime at 4.2K.

### *Experience at TRIUMF for RISP QWR*

The low temperature baking has been done at 120°C for 48h [19]. As seen on Figure 10, the $Q_0$ curve is shifted down at 2K showing that the residual resistance is increased. At 4.2K however, the low field $Q_0$ is comparable but the Q-slope has been significantly reduced already at low field. The increase of residual resistance compensated the BCS resistance improvement.

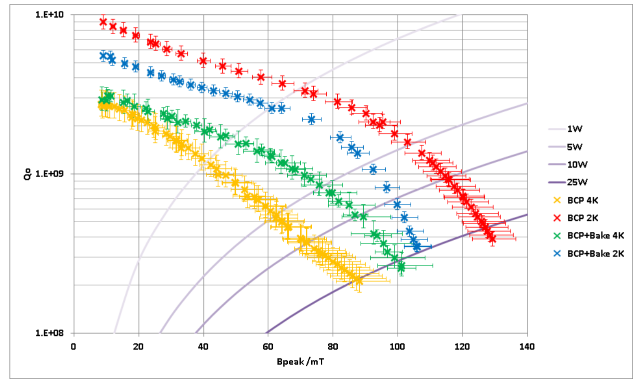


Figure 10: $Q_0$ versus accelerating gradient of the QWR prototype for RISP project before (yellow squares at 4.2K and red squares at 2K) and after 120°C baking (green squares at 4.2K and blue squares at 2K). This cavity has not been hydrogen degassed. Figure extracted from [19].

### *Experience at IPNO for SPIRAL2 QWR*

The same behavior has been observed after a 120°C baking during 48h [20]. Additional tests have been carried out to study the influence of baking time. As shown on Figure 11, after 24h, the low field $Q_0$ and Q-slope have been both improved but after 55h the low field $Q_0$ is lowered whereas the Q-slope has been maintained.

A model presented in [21] has been used to fit the curves. This is showing that the BCS resistance is constantly improved from 3.5 down to 2.4 nΩ whereas the residual resistance is increased from 1.9 nΩ up to 4.3 nΩ.

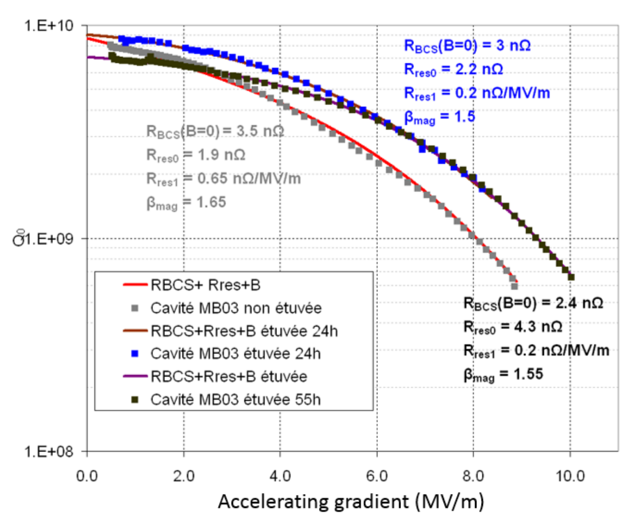


Figure 11: $Q_0$ at 4.2K versus accelerating gradient of a QWR prototype for Spiral2 project before (grey squares) baking, after 24h (blue squares) and 55h (black squares) at 120°. This cavity has not been hydrogen degassed (figure extracted from [20]).

The strong field dependence caused by the BCS resistance has been attenuated which divided by almost 2 the power dissipation at the nominal gradient (6.5 MV/m)

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### *Experience at MSU for ReA3 QWR*

The same benefits are observed on this cavity (See Figure 12). The Q-slope is significantly improved. The power dissipations at the operating gradient is divided by 2. Compared to the 2 examples presented previously this cavity has been hydrogen degassed. This could explain the improvement of the low field $Q_0$ that wasn't observed after such long baking. The degassing removed impurities (mainly hydrogen) that couldn't contribute anymore to Niobium hydrides formation.

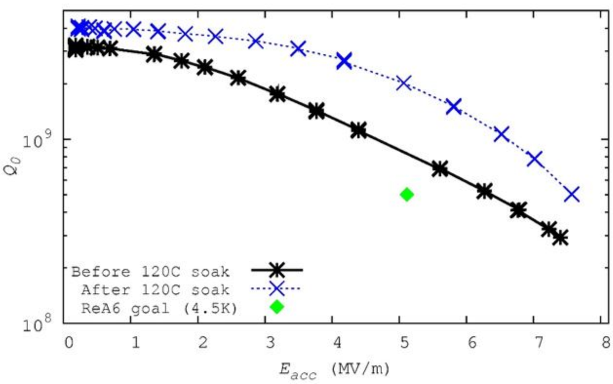


Figure 12: $Q_0$ versus accelerating gradient of a QWR prototype for ReA3 project before (black cross) and after (blue cross) baking at 120° during 48h. Figure extracted from [22].

As a conclusion on low temperature baking, this rather simple treatment has a huge impact at the operating gradient (i.e typically between 50-60 mT) as it can divide by 2 the power dissipations at 4.2K. It has however no advantages for accelerating structures operating in the residual regime (i.e at 2K for low frequency cavities).

The increase of the residual resistance observed is very similar to the 1.3 GHz elliptical cavities whereas the benefits of the baking are only visible above 80 mT (>20 MV/m).

## NITROGEN DOPING OF LOW BETA CAVITIES

So far, beside on 1.3 GHz and 650 MHz elliptical cavities (See Figure 13), nitrogen doping has not been tested on other low beta cavities. At 650 MHz, a significant difference is already observed: there is no anti Q-slope anymore and the $Q_0$ gain is way less impressive (60%). What would be the real benefit on cavities with even lower BCS resistance?

Nitrogen doping is known to keep residual resistance low and decreases BCS resistance [5]. This implies that to have a significant effect, the residual resistance has to be low compared to the initial BCS resistance to be really beneficial. Figure 14 is showing what would be the gain of $Q_0$ in percent depending on the residual resistance if we consider the typical improvement by 2 of the BCS resistance.

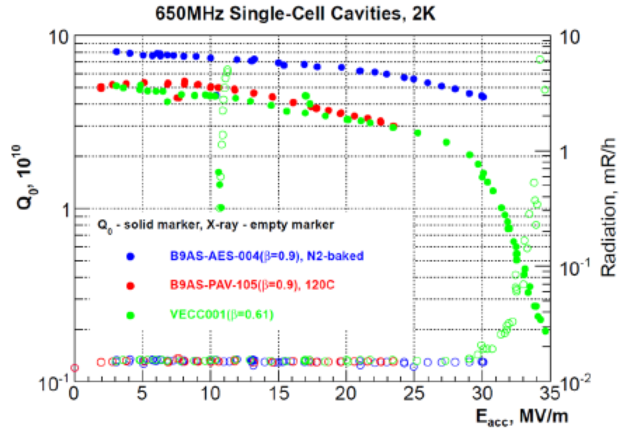


Figure 13: $Q_0$ versus accelerating gradient of an elliptical cavity at 650 MHz for PIP2 project after hydrogen degassing (green circles), after 120°C baking (red circles) and after nitrogen doping (blue circles). Figure extracted from [23].

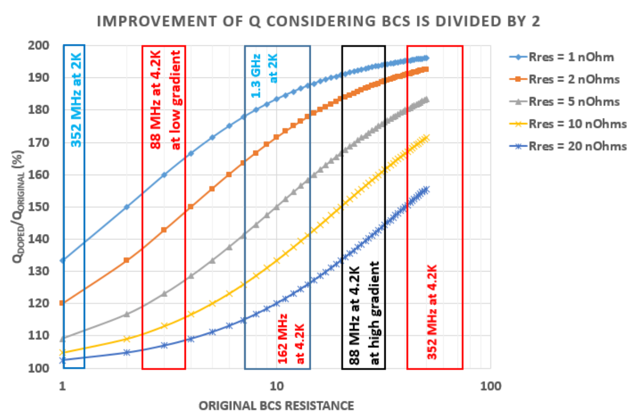


Figure 14: $Q_0$ gain versus the BCS resistance for different residual resistance. Here the assumption is a reduction of a factor of 2 of the BCS resistance.

The following statements can be done:

- Residual resistance has to be below 2 times the original BCS resistance to observe at least 50% improvement of the $Q_0$.
- Nitrogen doping of cavities below 350 MHz and operating at 2K is not beneficial as the Q-slope is negligible.
- Nitrogen doping of cavities between 50 and 100 MHz and operating at 4.2K is of great interest as it reduces significantly the Q-slope (if caused by BCS resistance) even though the low field $Q_0$ is not improved a lot.
- Nitrogen doping could allow 4.2K operation of cavities above 352 MHz if residual resistance is low compared to BCS resistance.

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THXA08

## CONCLUSION

Table 1 is giving the evolution of the BCS resistance at different temperatures and frequencies based on the following formula (See equation 1)

$$R_{BCS} = \frac{A(\lambda,\xi,l,...)\cdot\omega^2}{T}\cdot\exp\left(\frac{-\Delta}{k_B\cdot T}\right) \quad (1)$$

With A = 8·10$^5$ Ω·K·s$^{-2}$, T the temperature, ω the pulsation of the RF wave, Δ the energy gap and $k_B$ the Boltzmann constant.

Table 1: BCS Resistance

| $R_{BCS}$ (nΩ) | 4.2K | 2K | 1.8K | 1.5K |
|---|---|---|---|---|
| 1300 MHz | 585 | 15 | 6.5 | 1.2 |
| 700 MHz | 174 | 4.3 | 1.9 | 0.35 |
| 530 MHz | 97 | 2.5 | 1.1 | 0.2 |
| 352 MHz | 44 | 1 | 0.5 | 0.09 |
| 176 MHz | 11 | 0.3 | 0.1 | 0.02 |
| 88 MHz | 3 | 0.07 | 0.03 | 0.006 |
| 44 MHz | 0.7 | 0.02 | 0.007 | 0.001 |

Figure 15 is summarizing the main effects observed on the 3 heat treatments described in this paper. Hydrogen degassing shows the same improvements whatever the frequency of cavity. If properly done, only positive effects are observed. Recent tests have shown that the post chemical etching is not necessarily required if precautions are taken.

Regarding the 120°C baking, the effects are very different depending on the type of cavity and whether operating in BCS regime (BCS resistance is dominating) or in residual regime (BCS resistance is negligible). At 4.2K, the low temperature baking is improving the performances whatever the frequency is. However, at 2K and below 200 MHz, the increase of residual resistance is dominating. The performances are then degraded in this case. The low temperature is not recommended. For elliptical cavities at 1.3 GHz, the benefits of low temperature baking are only visible at magnetic fields above 80 mT. This treatment mitigates strongly the HFQS depending on the treatment history of the cavity [8, 24]. The reduction of the HFQS after low temperature baking is attributed to an increase of the energy gap. Is the mitigation of the Q-slope of low beta cavities correlated to the same effect? Are additional/different loss mechanisms existing at low frequencies inducing a different behaviour after low temperature baking?

Finally, nitrogen doping can't really be compared as only one cavity at lower frequency (650 MHz) has been treated. At first sight, the improvement after this treatment tend to be as well frequency dependent because mainly affecting the BCS resistance. In a near future, nitrogen infusion at a temperature above 160°C will be tested on one of the SSR for MYRRHA project at IPNO. This treatment is preferred than the original nitrogen doping as it doesn't require any post chemical etching.
This treatment may allow 4.2K operation of 352 MHz Spoke resonators. This is very interesting for ADS projects as these require very high reliability. The cryogenic infrastructure, distribution and cryomodule design for 4.2K operation is indeed way simpler than for 2K operation.

| | 1.3 GHz Elliptical Technical details | Observed effect | | Low beta cavities Technical details |
|---|---|---|---|---|
| Hydrogen degassing | - Compulsory<br>- Done without tank<br>- Done at 800°C<br>- Done during 3h | - Improvement of Residual<br>- Improvement of BCS<br>- Improvement of Q-slope<br>- Q-disease disappears | | - Not compulsory below 300 MHz<br>- Done with/without tank<br>- Done at 600°C (brazed parts)<br>- Done during 10h |
| 120°C baking | - Done during 48h<br>- Hot air/nitrogen blown around cavity | - Improvement of BCS resistance<br>- Increase of residual resistance | | - Done during 48h<br>- Hot air blown in helium tank<br>- Heating wires |
| | | - Mitigate High field Q-slope (above 80 mT) | - Mitigate Q-slope already at low field | |
| Nitrogen doping | - 800°C 3h + 2 min at 25 mtorr $N_2$ + 6 min in UHV + EP<br>- 800°C 3h + 160°C at 25 mtorr $N_2$ during 48h | - Improvement of BCS resistance<br>- Residual resistance stays low | | - Tried on 650 MHz only<br>- Will be tried on Spoke at 352 MHz |
| | | - Anti Q-slope | - No anti Q-slope | |

Figure 15: Comparison of observed improvements/degradations of the three heat treatments studied in this paper for elliptical cavities at 1.3 GHz and low beta cavities. When green, the observations are similar for both 1.3 GHz elliptical cavities and low beta cavities. When red, observations are different.

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## ACKNOWLEDGMENT

I would like to thank the following people for providing me material to illustrate this paper, Z. Conway (ANL), Z. Yao (TRIUMF) and E. Cenni (CEA). I would also like to thank all the technical staff from IPNO for their availability and the quality of cavity preparation for all the cryogenic tests done at IPNO on the 2 types of Spoke cavities (ESS and MYRRHA).

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