ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THPB007

NITROGEN INFUSION R&D ON SINGLE CELL CAVITIES AT DESY

M. Wenskat*, A. Prudnikava[1], D. Reschke, J. Schaffran
DESY, Hamburg, Germany
[1]Universität Hamburg, Hamburg, Germany

Abstract

A first series of single cell cavities underwent the "Nitrogen Infusion" treatment at DESY. Samples, which were in the furnace together with the cavities, underwent a series of SEM/EDX measurements and showed some unexpected structures. In parallel, the cavity performance deteriorated after the treatment. The furnace pressure and temperature and the residual gases during the treatment were analyzed to find the possible cause for the deterioration and next steps to prevent this deterioration in following treatments are discussed.

INTRODUCTION

Future upgrades of the European X-ray Free Electron Laser (XFEL) to operate in continuous wave (cw) mode at moderate to high gradients demand a high quality factor Q_0 at those gradients. A recently discovered treatment, the so called 'Nitrogen Infusion', seems to allow for such a behavior [1, 2], which could also lead to a cost reduction of a possible International Linear Collider.

FURNACE

The furnace in use has a volume of 7m^3 of which an effective volume of $1800 \times 625 \times 660$mm^3 has a homogenous temperature during baking, and was produced by the company Ipsen^{TM}. The maximum achievable, stable temperature is T_{max} = 1100°C and the furnace has a temperature stability of ±2°C. The door of the furnace is double sealed by EPDM O-rings with vacuum in between. Other accessories like pumps, gauges etc. are sealed by standard EPDM seals. The pre-pumps are a rotary vane pumps and a roots pump. The main pumps are two turbo molecular pumps (TMP) by Varian^{TM} with a pumping speed of 6000 l/s each. The installation of a single cell with niobium caps and two niobium samples on an aluminum-oxide ceramic in the furnace can be seen in Fig 1.

CAVITIES

Several single cell cavities are prepared for this R&D, and up till now, three of them have been baked. The first cavity which underwent the infusion treatment, 1DE18, underwent a minor HF treatment, prior to the baking, after a small fraction of the inner surface of one beam pipe was oxidized after the long term storage. All three cavities were tested last in 2006/2007 and have been stored since in double sealed clean room bags and with plastic caps. For the baking, all cavities were equipped with niobium caps, which underwent

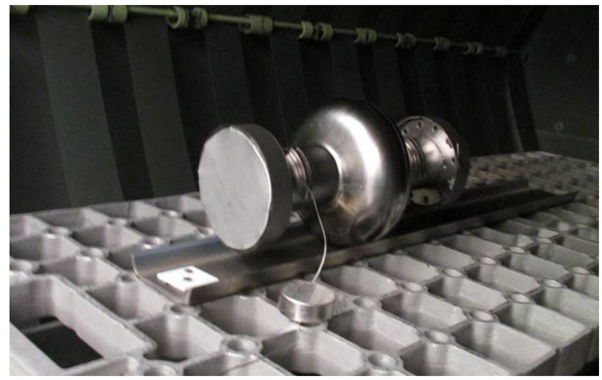

Figure 1: 1DE18 installed in furnace. Two samples are placed in front on a ceramic and the temperature sensor is attached to the cavity.

an etching and bake out before assembly onto a cavity. Table 1 shows a summary of the relevant cavity history details for each single cell. The nitrogen pressure was three order

Table 1: Cavity History and RF Results

	1DE18	1DE17	1DE16
Material	Ningxia fine grain	Ningxia fine grain	Plansee fine grain
RRR	300	300	300
Final Chemistry	100 μm EP	82 μm BCP	100 μm EP
RF Test 1 @ 2K			
$E_{acc,max}[\frac{MV}{m}]$	37.7 - BD	31.2 - BD	32.2 - BD
$Q_0(4\,\mathrm{MV/m})[\times 10^{10}]$	2.8	2.5	2.7
Baking Parameters			
p @ 800^oC [mbar]	2×10^{-5}	1.5×10^{-5}	1.3×10^{-6}
$p_{N=28}$ @ 800^{o}C [mbar]	2.2×10^{-7}	1.6×10^{-7}	2×10^{-8}
P_{N_2} @ 120°C [mbar]	7×10^{-5}	w/o	w/o
RF Test 2 @ 2K			
$E_{acc,max}[\frac{MV}{m}]$	20.2 no FE	19.5 no FE	-
$Q_0(4\,\mathrm{MV/m})[\times 10^{10}]$	0.5	1.2	-

of magnitudes lower than the recipe for 1DE18 during the 120°C. The problem was the location of a gauge, used to steer and control the mass flow, which was placed directly at the inlet and the high throughput of the turbo molecular pumps. This caused a pressure gradient across the furnace

* marc.wenskat@desy.de

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THPB007

and the low pressure at the cavity. In addition, the pressure at 800°C was a magnitude higher than the design value and the temperature was held for only 2 hours. The Q versus E_{acc} curves of the tested single cells are shown in Figure 2. An additional Q versus T measurement of 1DE18 showed

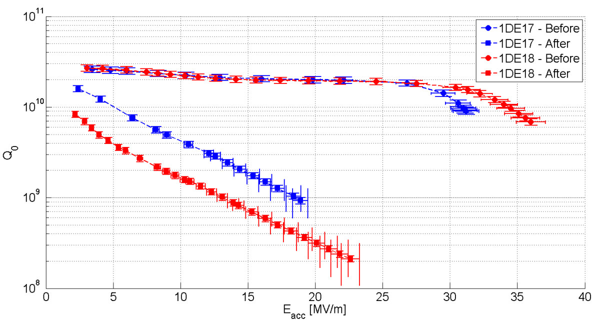

Figure 2: Quality factor vs. the accelerating field of the two single cells, treated in the DESY furnace. Circles are before, squares after treatment, red markers for 1DE18 and blue markers for 1DE17.

an increased residual resistance from 3.5nΩ to 35nΩ and a decrease of the reduced gap value from 1.97 to 1.79. After the deterioration of the performance of 1DE18 in RF Test 2, the same temperature cycle but without a nitrogen injection during the 120°C baking was done for 1DE17 and 1DE16, see Fig 3. This was done to exclude the nitrogen injection as the origin of the deterioration, since first analysis of the samples in the oven hinted on a possible nitride growth on the samples, see Figure 4. The performance of the second

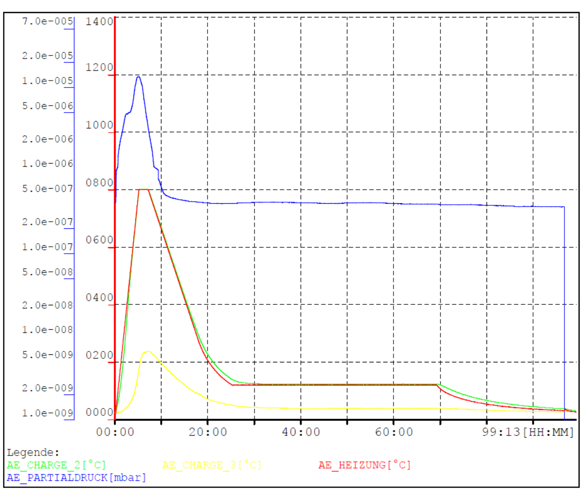

Figure 3: Oven pressure and temperature versus time of the 1DE17 cycle. No nitrogen was injected during the 48h at 120°C bake.

cavity, 1DE17, showed the same behavior as 1DE18. Hence, the origin for this cannot be the nitrogen injection and any possible contamination of the process, but the furnace pressure or a contamination of the furnace itself. A bake out cycle (1100°C for 4h) of the furnace and an extensive leak search was done to improve the overall and partial pressures prior to the treatment w/o nitrogen of the third cavity 1DE16. This cavity will be tested after the cryo shutdown beginning of August 2017. A subsequent improvement of the furnace pressure has been observed, see Table 1.

SAMPLES

All samples were made out of Heraeus material and underwent a BCP for their preparation. The samples which were in the oven with 1DE18 were placed on top of an aluminum-oxide ceramic. The samples for 1DE16 and 1DE17 were in addition covered with niobium pot. A total of three samples was prepared for each cavity treatment. One as a reference sample, which was brought to the furnace along the other two samples, but brought back into the clean room immediately after the cavity installation and not put into the oven. Two other samples were put into the oven and underwent the same backing procedure as the cavities. After the cavity is brought out of the oven, the samples are brought back into the clean room. Both samples underwent a rinsing to 18 MΩ, while only one underwent an additional HPR. In none of the samples for all cavities, a titanium contamination was found, which would have explained the shape of the Q vs. E curves [3]. Instead, star-like structures can be seen on all samples, see Figures 4 - 6.

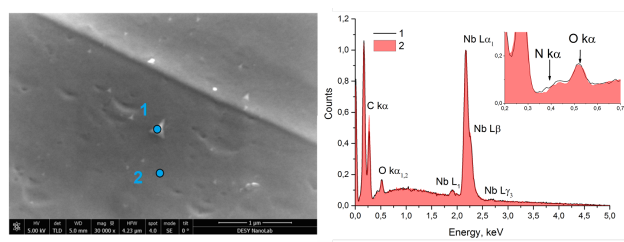

Figure 4: Left: SEM image of the sample which was in the oven with 1DE18. Right: EDX spectrum of two spots on the SEM image. A slight excess of nitrogen at roughly 0.4 keV in such a star-like structure is visible. EDX spectra on other samples were inconclusive, hence no unambiguous identification was possible.

DISCUSSION

The observed star-like structures are optically identical to the already identified β-Nb_2N structures, which are common for nitrogen doped cavities [4] but the EDX spectrum of those structures do not allow distinct identification. A Subsequent treatments of those samples, e.g. a 800°C bake out or HF rinsing, were done but did not remove those star-like structures. A TEM analysis of a treated and of a reference sample is now being prepared and carried out next to unambiguously identify those structures and possible other surface contamination. The two treated and tested cavities underwent an HF rinsing and will be retested. Assuming, that no additional contamination except the star-like structures is the cause of

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THPB007

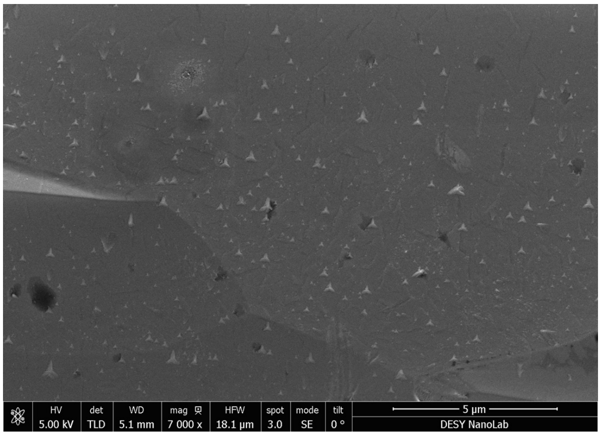

Figure 5: SEM image of a sample which was in the oven with 1DE17. The same structures were seen on samples which were together in the furnace with 1DE18.

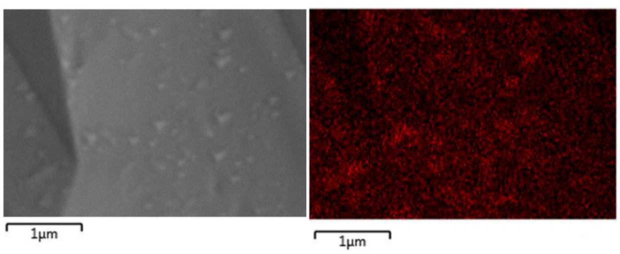

Figure 6: Left: SEM image of the sample which was in the oven with 1DE16. Right: EDX map of nearly the same region. One observation, which was not seen in any EDX map before, is an aggregation of carbon at the location of the precipitates.

the performance deterioration and using the sample results, no change is expected and would be an additional hint that the stars alone are the origin of the performance degradation. Although several steps were undertaken to improve the furnace pressure, first SEM/EDX investigation of the samples for 1DE16 showed the same star-like structures and hence, a deterioration of the performance can be expected. Other contamination, like certain types of niobium carbides as suggested in Figure 6, cannot be excluded as the cause for the performance drop and are also investigated.

REFERENCES

[1] A. Grassellino and S. Aderhold, "New Low T Nitrogen Treatments Cavity Results with Record Gradients and Q", presented at TTC Workshop 2016, Saclay.

[2] A. Grassellino *et al.*,"Unprecedented Quality Factors at Accelerating Gradients up to 45 MV/m in Niobium Superconducting Resonators via Low Temperature Nitrogen Infusion", ArXiveprint:1701.06077, 2017.

[3] A. Grassellino *et al.*,"Fermilab experience of post-annealing losses in SRF niobium cavities due to furnace contamination and the ways to its mitigation: a pathway to processing simplification and quality factor improvement", ArXiveprint:1305.2182, 2013.

[4] Y. Trenikhina *et al.*, "Characterization of Nitrogen Doping Recipes for the Nb SRF Cavities", in *Proc. SRF 15*, Whistler, Canada.