Cavity Cut-Out Studies of a 1.3GHz Single-cell Cavity after a failed Nitrogen Infusion Process

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Nitrogen Infusion R&D at DESY

- Nitrogen infusion [1] is a promising but not well understood process to improve cavity performance.
- Severe deterioration of cavity performance after first infusion runs in the DESY furnace was observed [2].
- The deterioration was inverse proportional to the pressure at 800°C
  - $p_{1DE16} < p_{1DE17} < p_{1DE18}$ at 800°C
  - $Q_0(4MV/m)_{1DE16} > Q_0(4MV/m)_{1DE17} > Q_0(4MV/m)_{1DE18}$
- Samples showed niobium carbides forming during that phase.
- To test the transferability of sample studies to the cavity surface and to investigate the deterioration of the cavities, 1DE16 was cut.

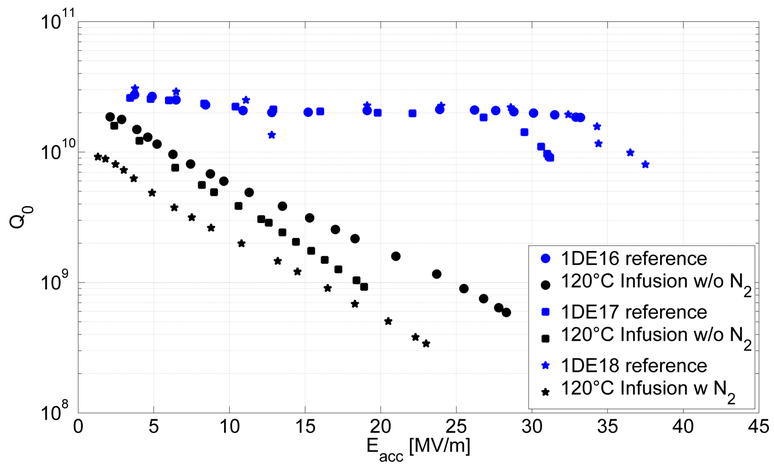

Figure 1: Quality factor vs. accelerating field of the first three single cell cavities used for infusion R&D. Reference measurements (blue) and measurements after treatment (red) are shown. The same deterioration for all cavities are observed.

1DE16

- Fine grain single-cell cavity (Ningxia).
  - Reference: $E_{acc,max}$ = 32 MV/m (BD); Q_0(4MV/m) = 2.7 x 10^{10}
- Baked twice according to nitrogen infusion temperature profile at 120°C w/o nitrogen
  - 1st Bake: $E_{acc,max}$ = 27 MV/m (BD); Q_0(4MV/m) = 2.7 x 10^{10}
  - 2nd bake: $E_{acc,max}$ = 26 MV/m (PWR); Q_0(4MV/m) = 3.2x 10^{9}

Identifying Regions of Interest

- T-map data, 2nd Sound data, optical inspection and H-map [3] data was gathered and 8 regions of interest were identified.

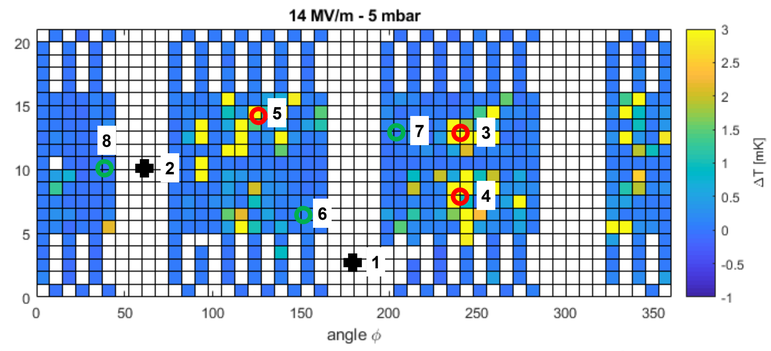

Figure 2: Temperature map of 1DE16 – white squares are the position of the H-Map cards. A rather homogenous heating with only three dominant heating spots is observed.

Table 1: Sample list of cut-outs.

Diagnostics		Position			Sample
T-map DESY	Quench Spot 1	170° - Below E			1
2nd Sound Test 3		170° - E-24mm			
H-map HZB		170° - near beam pipe			
2nd Sound Test 4	Quench Spot 2	70° - on Equator			2
T-map HZB	Hot Spots	240° E+2cm	240° E-1cm	115° E+1.5cm	3/4/5
	Cold Spots	155° E-1cm	215° E+1cm	30° E	6/7/8

- Quench spot 1 was not the limiting region after the infusion procedure but still showed significant flux trapping of 1.5 µT in the H-Map.
- No H-Map of quench spot 2 was obtained due to a malfunctioning card.
- The samples were square shaped with a side length of ~1.5cm.

Origin of Deterioration

- β-Nb_2C has been found on the surface of samples treated in the DESY furnace [2]. If those are forming on the inner cavity surface and if they are related to the observed heating was investigated.

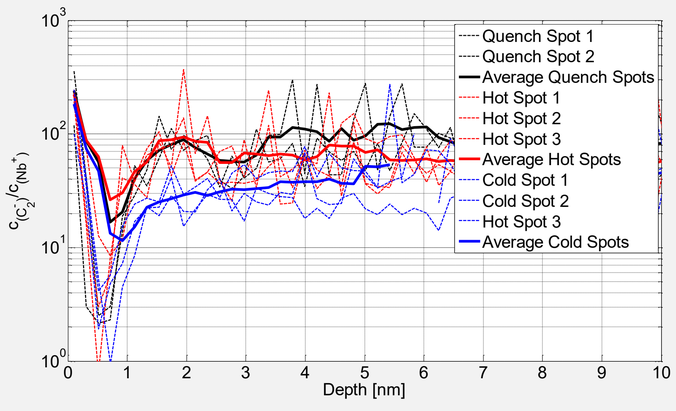

Figure 3: Normalized C_2^- - signal of each sample in the first 10nm obtained with a TOF-SIMS. Quench spots and hot spots showed a higher carbon content in this first layer.

- A smaller carbon signal was observed in the SIMS measurements for cold spots compared to hot spots.
- SEM images showed that niobium carbides form on the inner surface as well.
- Cold spot samples showed less and smaller carbides on the surface than hot spots samples.

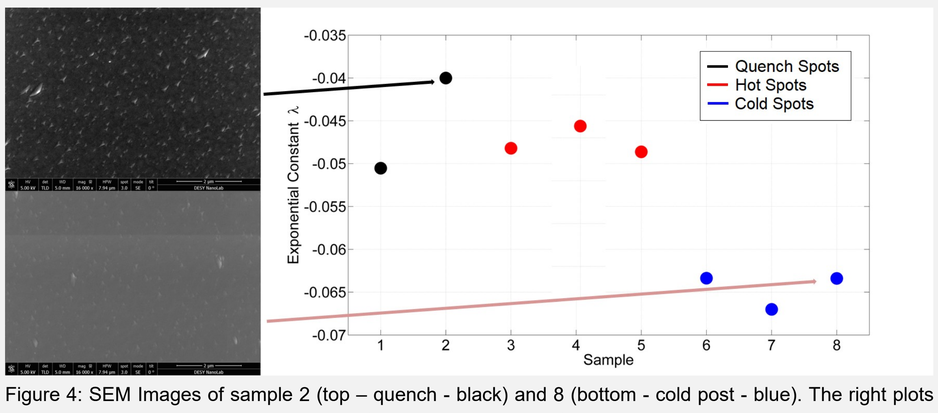

Figure 4: SEM Images of sample 2 (top – quench - black) and 8 (bottom - cold post - blue). The right plots shows the exponential decay constant λ of the size distribution of the niobium carbides.

Influence on Carbon Solubility

- What causes the difference in the carbon solubility and formation of niobium carbides? Lattice structure and interstitials were investigated.

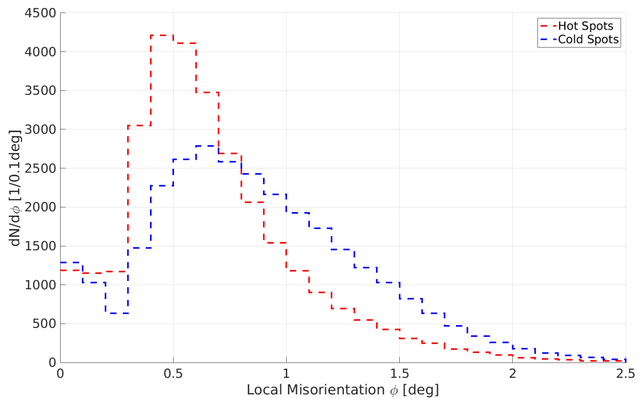

Figure 5: Local misorientation data for hot spots (red) and cold spots (blue). Average over 2 measurements per sample, covering a 200x150 grid with a 10 µm step size. An excess of low angle grain boundaries for hot spots compared to cold spots was found.

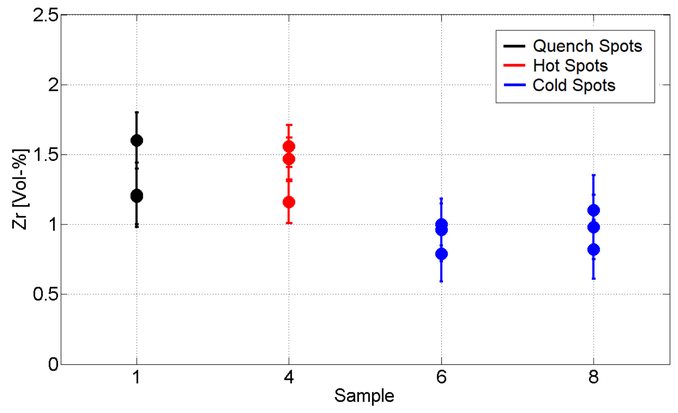

Figure 6: Zirconium concentration for some samples according to PIXE. A slightly higher concentration in the hot spot and quench spot samples compared to the cold spots samples was found.

Conclusions

- Niobium carbides form also on the inner cavity surface and their size and density are correlated to the local heating.
- They form during the 800°C bake in the DESY furnace. The origin of the residual carbon is still unclear.
- EBSD data showed a small excess of low angle grain boundaries on hot spot samples compared to cold spot samples.
- Additional elemental analysis of the samples with PIXE showed a higher zirconium content in the hot spots. Zirconium is a known pollution on Nb material [4] and in Nb-Zr-C alloys Zr can act as a catalyst on the formation of niobium carbides [5].

References

1) Grassellino et al., Supercond. Sci. Tech 30.9 (2017): 094004.
2) Wenskat et al., Proceedings of LINAC2018, TH2A01
3) Schmitz et al., Rev. Sci. Instrum. 89.5 (2018): 054706
4) Singer et al., Supercond. Sci. Tech 28.8 (2015): 085014
5) Grobstein et al. “Characterization of Precipitates in a Nb-Zr-C Alloy”, DOE/NASA/16310-6 (1986)

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