CAVITY CUT-OUT STUDIES OF A 1.3 GHz SINGLE-CELL CAVITY AFTER A FAILED NITROGEN INFUSION PROCESS

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Abstract

R&D on the nitrogen infusion process at DESY produced at the beginning a series of 1.3 GHz single-cell cavities which have shown severe deterioration in the vertical cold test which was completely unexpected and could not be explained at the time. To investigate the origin of the deterioration, one of the cavities was optically inspected and a Temperature- and Magnetic Field-Mapping was done in collaboration with HZB. Together with 2nd Sound data, regions of interests were identified and cut from the cavity. Subsequent surface analysis techniques (SEM/EDX, SIMS, PIXE, EBSD) were applied in order to identify the reason for the deterioration. Especially the differences between hot and cold spots as well as quench spots identified by T-Mapping were investigated.

NITROGEN INFUSION R&D AT DESY

The discovery of the nitrogen infusion process [1] led to a global R&D effort to understand the procedure itself, its influence on the cavity surface and how the surface evolution relates to the cavity performance. The first three cavities treated at DESY showed a severe deterioration after baking procedures with and without nitrogen injection [2, 3], see Figure 1.
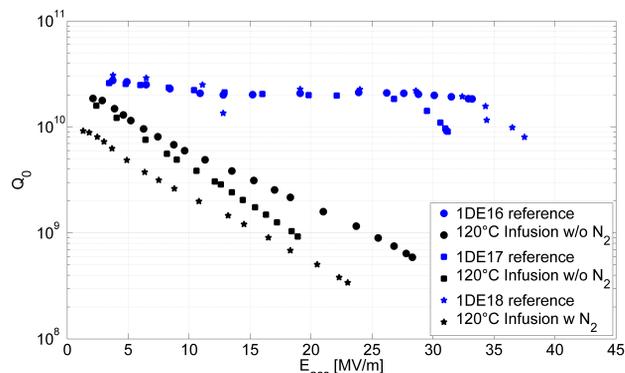

Figure 1: Quality factor vs. accelerating field of the first three single cell cavities used for infusion R&D. Reference measurements (blue) and measurements after treatment (red) are shown. The same kind of deterioration for all cavities was observed.

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Two interesting observations emerged from these three runs

1. Samples which were baked in the furnace inside a Nb box to mimic the caps on the cavities together with the cavities formed $\beta - Nb_2C$ on their surface

2. The quality factor at $4\,MV/m$ at $2\,K$ for the cavities increased, as the pressure during the baking at $800^{\circ}\,C$ decreased

These observation let us conclude that, that the formation of carbides during the $800^{\circ}\,C$ phase of the infusion was the origin of the deterioration. Following infusion runs on cavities showed a more complex situation, in which samples outside the Nb box during the same run did not form niobium carbides and cavities which showed a less severe or no deterioration still resulted in samples covered with niobium carbides. Hence, the decision was taken to check the assumption that niobium carbides are forming inside the cavity and that they are the cause of the cavity performance degradation by cutting a cavity was done.

IDENTIFYING REGIONS OF INTEREST

The single-cell cavity 1DE16 was then transported to Helmholtz-Zentrum Berlin (HZB) to test the cavity with their T-Map and H-Map system [4]. This information, together with previous DESY T-Map data [5] and 2nd Sound measurements was used, to define regions of interests, see Fig. 2.
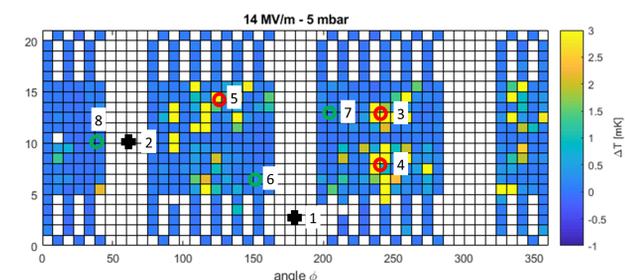

Figure 2: Temperature map of 1DE16 taken at HZB – white areas are the position of the H-Map cards. A rather homogenous heating with only three dominant heating spots is observed. Markers for the samples cut from quench spots (black), hot spots (red) and cold spots (green) are shown.

Fundamental R&D - Nb

processing (doping, heat treatment)

In total, 8 square-shaped samples, with a side length of ≈ 1.5 cm, were cut:

- 2 Quench spot samples: sample 1 is the quench spot prior to the failed infusion run and sample 2 the quenchspot after the first failed infusion

- 3 Hot spot samples: samples 3-5 are cut from regions with a rather local heating of ≈ 3 mK

- 3 Cold spot samples: samples 6-8 are cut from regions with no heating

Quench spot 1 was not the limiting region after the infusion procedure but still showed significant flux trapping of 1.5 μT during cooldown in the H-Map data. No H-Map of quench spot 2 was obtained due to a malfunctioning card.

ORIGIN OF DETERIORATION

All samples were investigated with a scanning electron microscope (SEM) to check whether niobium carbides were forming on the surface. On all samples, the same star-shaped precipitates were found, which were identical to the precipitates found on samples, see Fig. 3. To further identify the observed structures as carbides, energy dispersive X-ray spectroscopy (EDX) was applied and a carbon-enriched signal was observed in those precipitates compared to its surroundings. From all samples, a set of images was taken
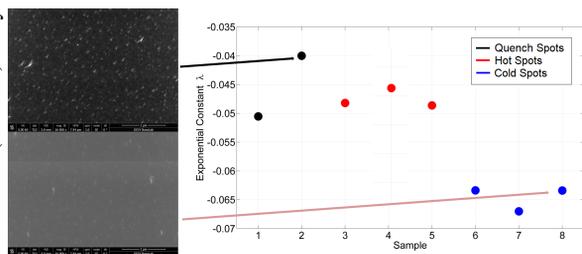

Figure 3: SEM Images of sample 2 (top - quench) and 8 (bottom - cold spot). The right plots shows the exponential decay constant λ of the size distribution of the niobium carbides.

with the same parameters w.r.t. applied voltage and magnification, to have a comparable set between the samples. Counting the precipitates and sorting them by size created an exponential decaying distribution and the decay constant λ for the different samples is shown in Fig. 3. A smaller decay constant for quench spots and hot spots than for cold spots is observed, hence more and larger precipitates are observed on quench spots and hot spots. In addition, all samples underwent time-of-flight secondary-ion mass spectrometry (ToF-SIMS) measurements and the normalized carbon signals are shown in Fig. 4. In agreement with the SEM image analysis, a higher carbon signal was observed in the quench spot and hot spot samples compared to the cold spot samples.
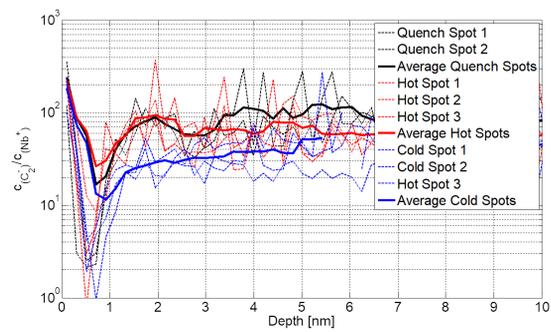
Figure 4: Normalized C_2^- - signal of each sample in the first 10 nm obtained with a ToF-SIMS. Quench spots and hot spots showed a higher carbon content in this first layer.

INFLUENCE ON CARBON SOLUBILITY

The question arose, what is the origin of a higher or lower carbon aggregation. The crystal structure was mapped utilizing electron backscatter diffraction (EBSD) were two examples are shown in Fig. 5. Analyzing the local misorientation
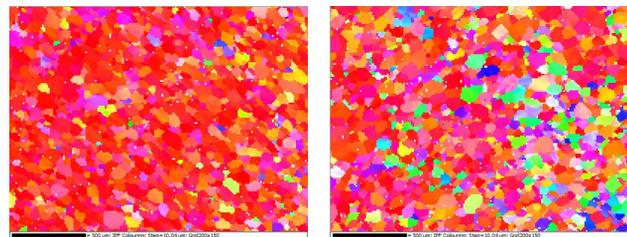

Figure 5: Inverse pole figures of hot spot sample 3 and cold spot sample 7.

obtained during this measurement lead to the distribution of the low angle grain boundaries below 2.5°, which is shown in Fig. 6. The curves shown are the average over the two classes of samples. On each sample two different spots were scanned with an 200×150 grid with a 10 μm step size. An excess of
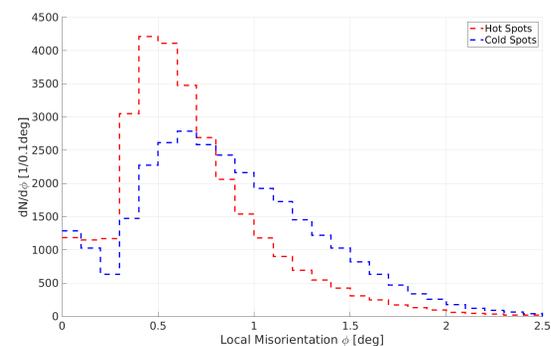

Figure 6: Local misorientation distribution for hot spots (red) and cold spots (blue).

low angle grain boundaries for hot spots compared to cold spots was found. In addition, to check the samples for heavier elements such as titanium as a potential cause for degrada-

tion, four samples were analyzed by proton-induced X-ray emission (PIXE). No titanium contamination was found and from all other elements only zirconium showed a relevant signal, shown in Figure 7.
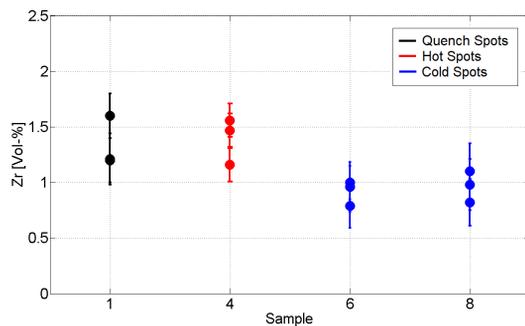

Figure 7: Zirconium concentration for some samples according to PIXE. A slightly higher concentration in the hot spot and quench spot samples compared to the cold spots samples was found.

CONCLUSIONS

From the SEM/EDX analysis, the assumption that both, the samples within the Nb box and the inner cavity surface, form niobium carbides has been proven to be correct and results from samples are transferable to cavities and vice versa. In addition, a correlation between the carbon signal and the local heating were established. Hence, the statement that infusion process in this specific furnace forms niobium carbides which are then the cause for additional losses is valid while the origin of the carbon pollution is still unclear. To identify the reason for the different carbon aggregation, EBSD measurements on all samples were performed. Crystal orientations, grain sizes and internal strain based on local misorientations were investigated. A small excess of low angle grain boundaries for hot spots compared to cold spots was found and it is known that these type of grain boundaries have a strong influence on the flux pinning behavior and vacancy and interstitial trapping [6,7]. Additional elemental analysis of the samples with PIXE showed a slightly higher zirconium content in the hot spots. Zirconium is a known pollution on Nb material [8]. Furthermore, Zr can act as a catalyst on the formation of niobium carbides Nb-Zr-C alloys are known to be stable [9]. These two factors - local misorientation and zirconium content - might have been the cause for the difference observed in the carbon content and hence local heating during T-Map. This is reasonable, considering the rather homogenous heating in the T-Map in Fig. 2 which is the result of a homogenous 'coating' of the surface with niobium carbides during the baking.

REFERENCES

[1] A. Grassellino *et al.*, "Unprecedented quality factors at accelerating gradients up to 45 MVm^{-1} in niobium superconducting resonators via low temperature nitrogen infusion", *Supercond. Sci. Technol.*, vol. 30, no 9, p. 094004, 2017.

[2] M. Wenskat, D. Reschke, J. Schaffran, and A. L. Prudnikava, "Nitrogen infusion R&D on single cell cavities at DESY", in *Proc. 18th Int. Conf. RF Superconductivity (SRF'17)*, Lanzhou, China, Jul. 2017, pp. 759–761. doi:10.18429/JACoW-SRF2017-THPB007

[3] M. Wenskat *et al.*, "Nitrogen infusion R&D for CW operation at DESY", in *Proc. 29th Linear Accelerator Conf. (LINAC'18)*, Beijing, China, Sep. 2018, pp. 652–657. doi:10.18429/JACoW-LINAC2018-TH2A01

[4] B. Schmitz *et al.*, "Magnetometric mapping of superconducting RF cavities", *Rev. Sci. Instrum.*, vol. 89, no. 5, p. 054706, 2018.

[5] M. Pekeler *et al.*, "Thermometric study of electron emission in a 1.3 GHz superconducting cavity", in *7th Workshop on RF superconductivity*, Gif-sur-Yvette, France, 1996, vol. 1, pp. 79-93.

[6] Z. Sung *et al.*, "Development of low angle grain boundaries in lightly deformed superconducting niobium and their influence on hydride distribution and flux perturbation", *J. Appl. Phys*, vol. 121, no. 19, p. 193903, 2017.

[7] M. Wang *et al.*, "Investigation of the Effect of Strategically Selected Grain Boundaries on Superconducting Properties of SRF Cavity Niobium", in *Proc. 18th Int. Conf. RF Superconductivity (SRF'17)*, Lanzhou, China, Jul. 2017, pp. 787–791. doi:10.18429/JACoW-SRF2017-THPB026

[8] W. Singer *et al.*, "Superconducting cavity material for the European XFEL", *Supercond. Sci. Technol.*, vol. 28, no. 8, p. 085014, 2015.

[9] T. Grobstein *et al.*, "Characterization of Precipitates in a Niobium-zirconium-carbon Alloy", NASA Rep. NASA-TM-100848, 1986.