NITROGEN INFUSION SAMPLE R&D AT DESY.

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The so-called "Nitrogen Infusion" process applied to 1.3GHz TESLA type cavities was reported to achieve higher Q-values compared to the standard surface treatments. The recipe consists of a heat treatment at 800C for 3h under vacuum conditions followed by a ramping down to 120C. During the hold time of 48h at 120C a partial pressure (3x10e-2 mbar) of nitrogen is applied. It is our goal to find the key parameter for a stable and reproducible recipe. An intense sample study is carried out and the results are presented here.

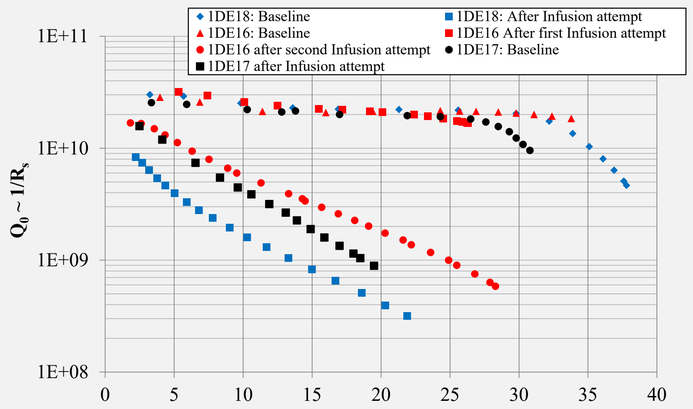


Figure 1: Cavity performance of the single-cell cavities 1DE16, 1DE17 and 1DE18 before and after first heat treatment without post chemical surface removal at DESY. The cavities shown here were treated at 800C for 3h under vacuum conditions followed by a ramping down to 120C without nitrogen.

Precipitation of carbides observed on cavity inner surface and witness samples!

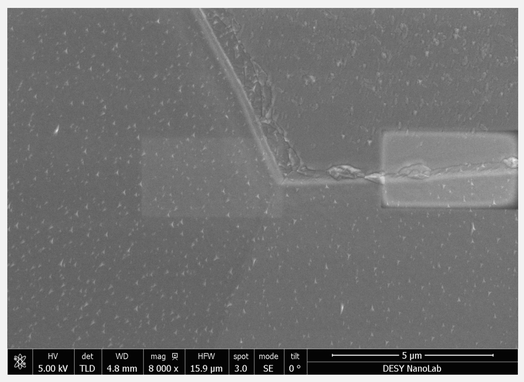


Figure 2: SEM of cavity cut-out from 1DE16 after the second heat treatment. Star shaped precipitates are observed on the surface. A more detailed analysis of 1DE16 is discussed in [1].

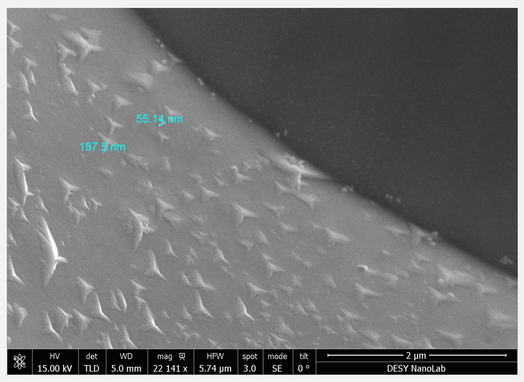


Figure 3: SEM of a witness sample heat treated together with one of the degraded cavities shown in fig. 1. Precipitates were found on witness samples of each run and identified as carbides via TEM EDS. The optical appearance is very similar to those of nitrides.

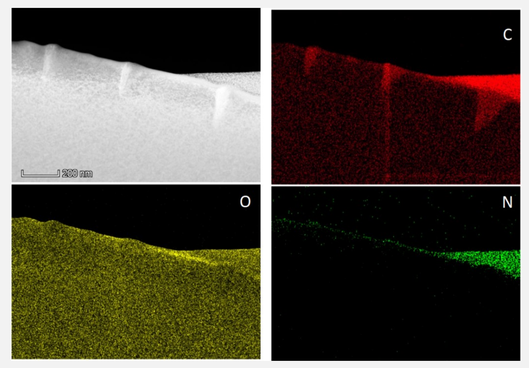


Figure 4: TEM EDS of cross section from a witness sample with precipitates. Star shaped precipitates have been identified as carbon enriched and as possible hexagonal β-Nb_2C phase.

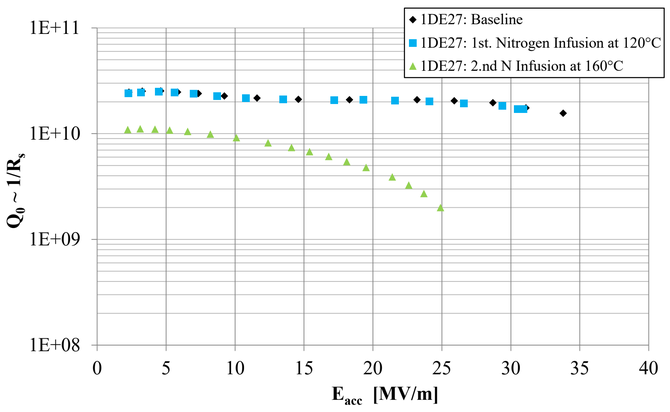


Figure 5: Cavity performance of 1DE27 before and after nitrogen Infusion treatment at two different temperatures without post chemical surface removal at DESY. There was no chemical surface removal between the two nitrogen Infusion's.

Carbide precipitates observed inside of niobium box only! No precipitates on samples outside of niobium box!

Figure 6a: Witness samples for the nitrogen Infusion of 1DE27 were placed inside and outside of a niobium box to mimic the line of sight protection of the niobium caps during heat treatment. Sample No. #68(1.st run) and #10(2.nd run) were placed inside the box. Sample No. #61(1.st run) and #1(2.nd run) were placed outside.

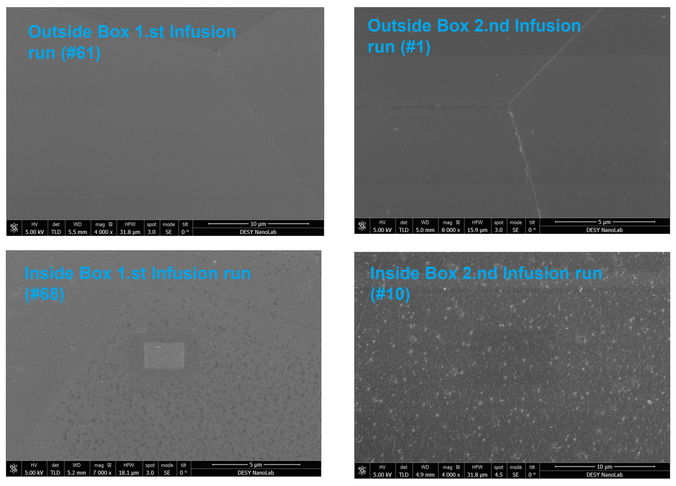


Figure 6b: SEM images of samples inside and outside a niobium box during the nitrogen Infusion treatments of 1DE27. In both cases carbide precipitation occurred on samples inside the box, only.

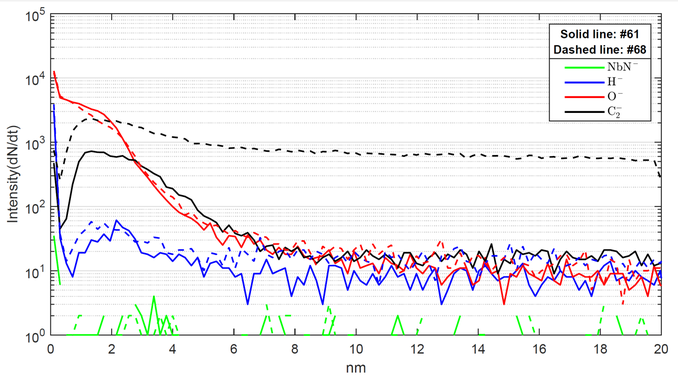


Figure 7: SIMS measurement of samples inside (dashed line) and outside the (solid line) niobium box from the 1.st Infusion of 1DE27 at 120°C. A clear difference in the carbon signal is observed.

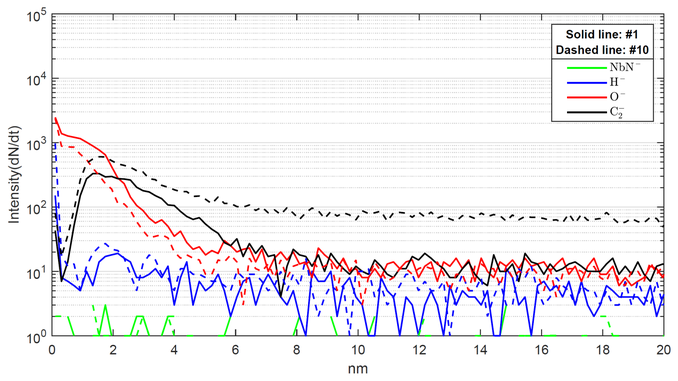


Figure 8: SIMS data of samples inside (dashed line) and outside the box (solid line) from the second Infusion of 1DE27 at 160°C. The relative difference in the carbon signal between the two samples here is less pronounced compared to the first Infusion run. Furthermore the amount of oxygen is lower for the sample inside the niobium box compared to the sample outside.

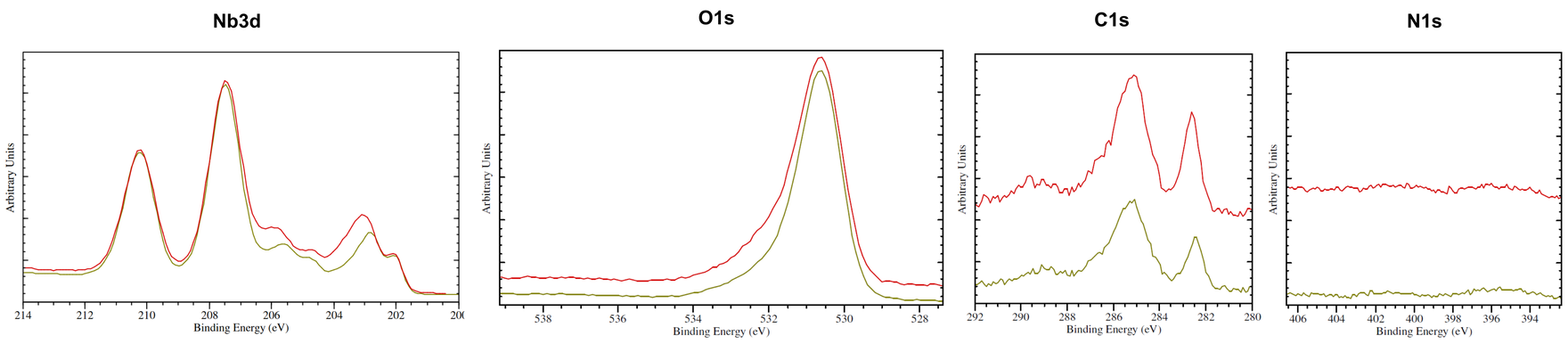


Figure 9: XPS measurement on sample #68 (green curve) from the 120°C nitrogen Infusion and sample #10 (red curve) from the 160°C nitrogen Infusion. Both samples were treated inside of the niobium box. A higher oxygen and carbon amount is present for sample #10 which has been infused at 160°C. No nitrogen signal is present for both samples. The curves of C, N and O are offset in vertical direction.

Figure 13: Image of the small sample furnace. The furnace has a ceramic tube with a diameter of 80 mm. An Residual Gas Analyzer and a mass-flow controller for nitrogen inlet is installed. The maximum achievable, stable temperature is 1350°C. The pump system is completely oil free and reaches p<2*10^{-7} mbar. The furnace has been setup for explicit nitrogen Infusion studies on samples.

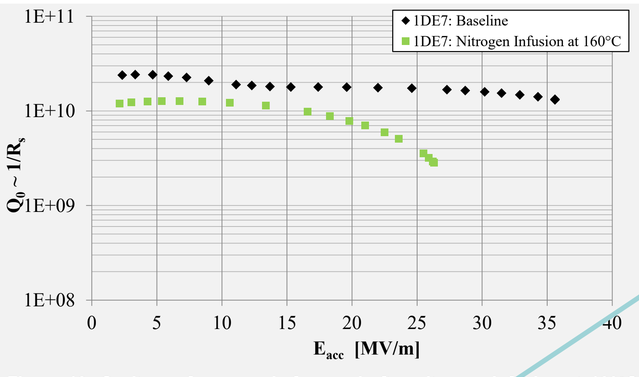


Figure 10: Cavity performance before and after nitrogen Infusion at 160°C without post chemical surface removal at DESY.

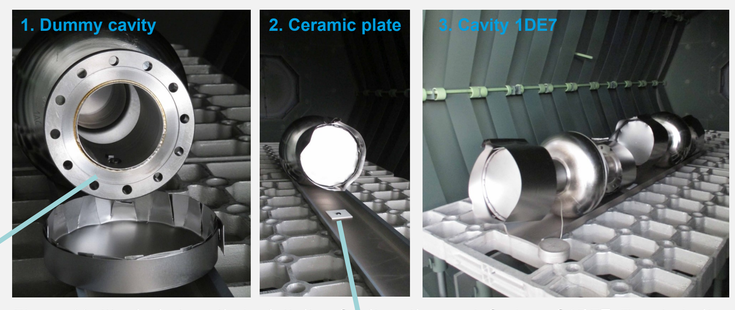


Figure 11: Illustrated cavity installation for the nitrogen Infusion of 1DE7. To mimic the line of sight protection a dummy cavity (instead of the niobium box) is used shown on the left picture. The sample "outside" is placed on a ceramic plate to make sure to have a clean underground for the sample.

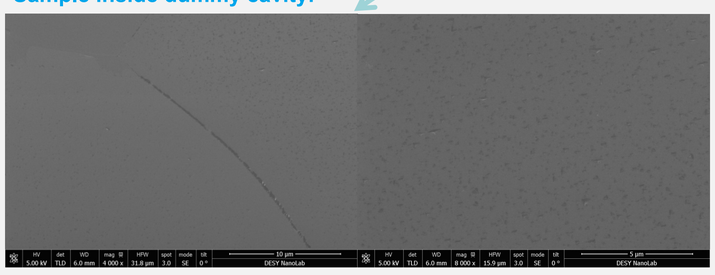
Table 1: Summary of so far observed carbides on witness samples during heat treatment with and without a line of sight (LOS) protection. For the cavities 1DE18, 1DE17, 1DE16 and 1DE9 no nitrogen has been used but the same heat ramping to 800°C for 3h and then 120°C for 48h in p<10^{-5} mbar. In almost each case with LOS carbides are observed while only once without LOS.

Cavity	perf.	w. LOS	w/o LOS
1DE18	degr.	✓	n.a.
1DE17	degr.	✓	n.a.
1. 1DE16	same	✓	n.a.
2. 1DE16	degr.	✓	n.a.
1DE9	degr.	✗	n.a.
1DE10	degr.	✓	n.a.
1DE11	degr.	✓	n.a.
1. 1DE27	same	✓	✗
2. 1DE27	degr.	✓	✗
1DE7	degr.	✓	(✓)
1. 1DE3	degr.	✓	n.a.
2. 1DE3	same	?	n.a.

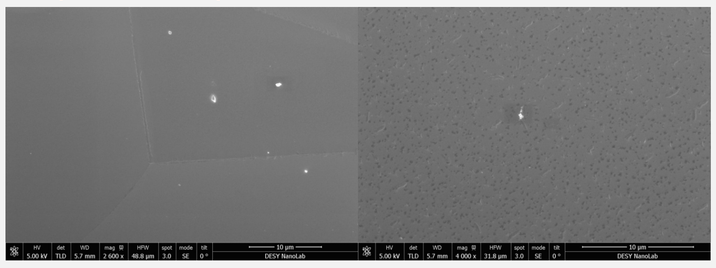
Table 2: Summary of so far observed carbides on witness samples treated without cavities in the sample furnace (see fig. 13) and the big DESY ZM furnace. No case of carbide precipitation has been observed when no LOS is used. This together with the observation of table 1 shows a very strong correlation between the usage of LOS and carbide precipitation.

Heat treatment	w LOS	w/o LOS
180°C N-Inf sample furnace	n.a.	✗
800°C cavity furnace	✓	✗
800°C sample furnace	n.a.	✗
800°C sample furnace	n.a.	✗
800°C sample furnace	n.a.	✗
800°C sample furnace	n.a.	✓

Sample inside dummy cavity:



Sample on ceramic plate:



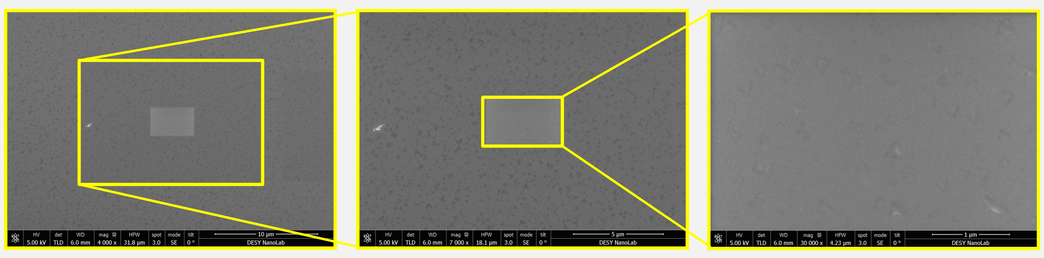


Figure 12: SEM images of witness samples from 160°C nitrogen Infusion of the cavity 1DE7. On the left the sample infused inside the dummy cavity shows star shaped precipitation. A dark layer accumulated on the top which can be sputtered away by focusing with the SEM as illustrated. This suggests a very thin carbon monolayer on the surface is present.
On the right we have the sample on the ceramic plate which also shows precipitation on some grains and none on others.

Conclusions

- **Several samples have been investigated for surface changes after heat treatments without any post chemical cleaning.**
- **Star-shaped precipitates were found on witness samples as well as cavity cut-outs and are suspected to be a source for cavity performance deterioration.**
- **This study shows a strong correlation between carbide precipitation and the use of line of sight protection (e.g. niobium foils or boxes as shown in fig. 6a) during heat treatment. This can be seen from the summary in table 1 and 2.**
- **However, cases are observed were carbides grew without line of sight protection and can be attributed to other sources too. SIMS and XPS measurements of the 1DE27 witness samples show more carbon contamination during the second Infusion treatment were the cavity started to deteriorate. The SIMS data in fig. 7 and 8 shows this by the relative comparison of each run between sample outside and inside the box.**
- **The witness samples of 1DE7 which were nitrogen infused at 160°C show carbides inside and outside of protection but the sample outside the dummy cavity also has precipitate-free grain spots as shown in the SEM images.**
- **The cavity performance after the nitrogen Infusion has a similar characteristic deterioration for 1DE7 and 1DE27 but differ from the first attempts without nitrogen of 1DE16, 1DE17 and 1DE18. Different from the first attempts a carbon mono layer is observed on sample surfaces.**

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[1] Wenskat et al, MOP025, SRF 2019 Dresden

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