DESY Infusion R&D

Results and status at DESY

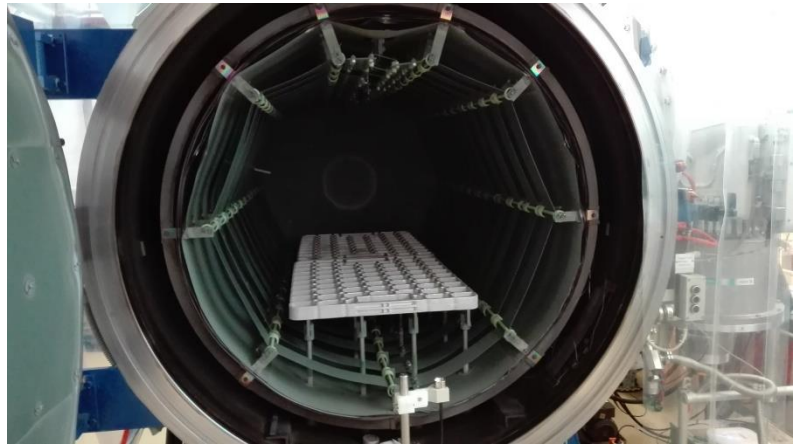
Bate Christopher on behalf of the SRF Team
Sendai, 31.10.2019

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

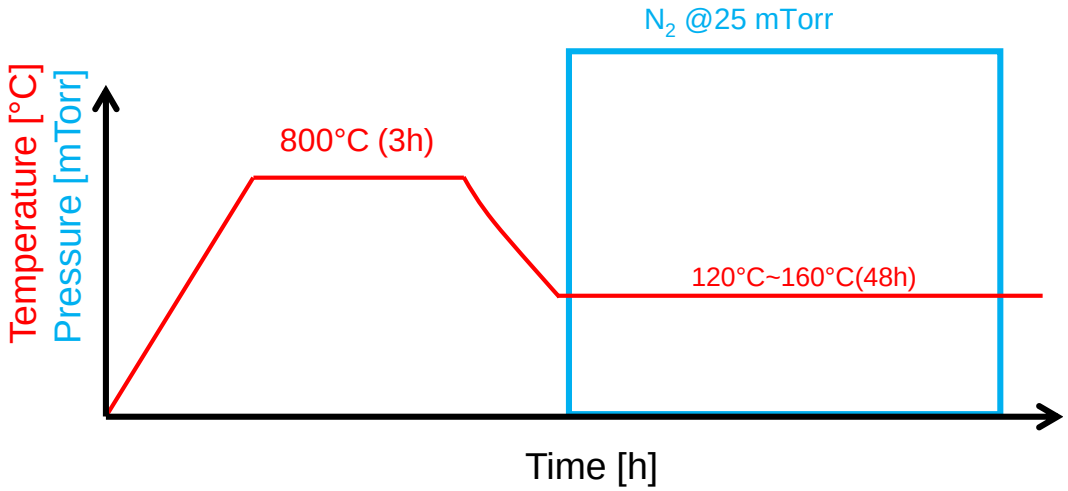
DESY.

Infusion cavity treatments at DESY

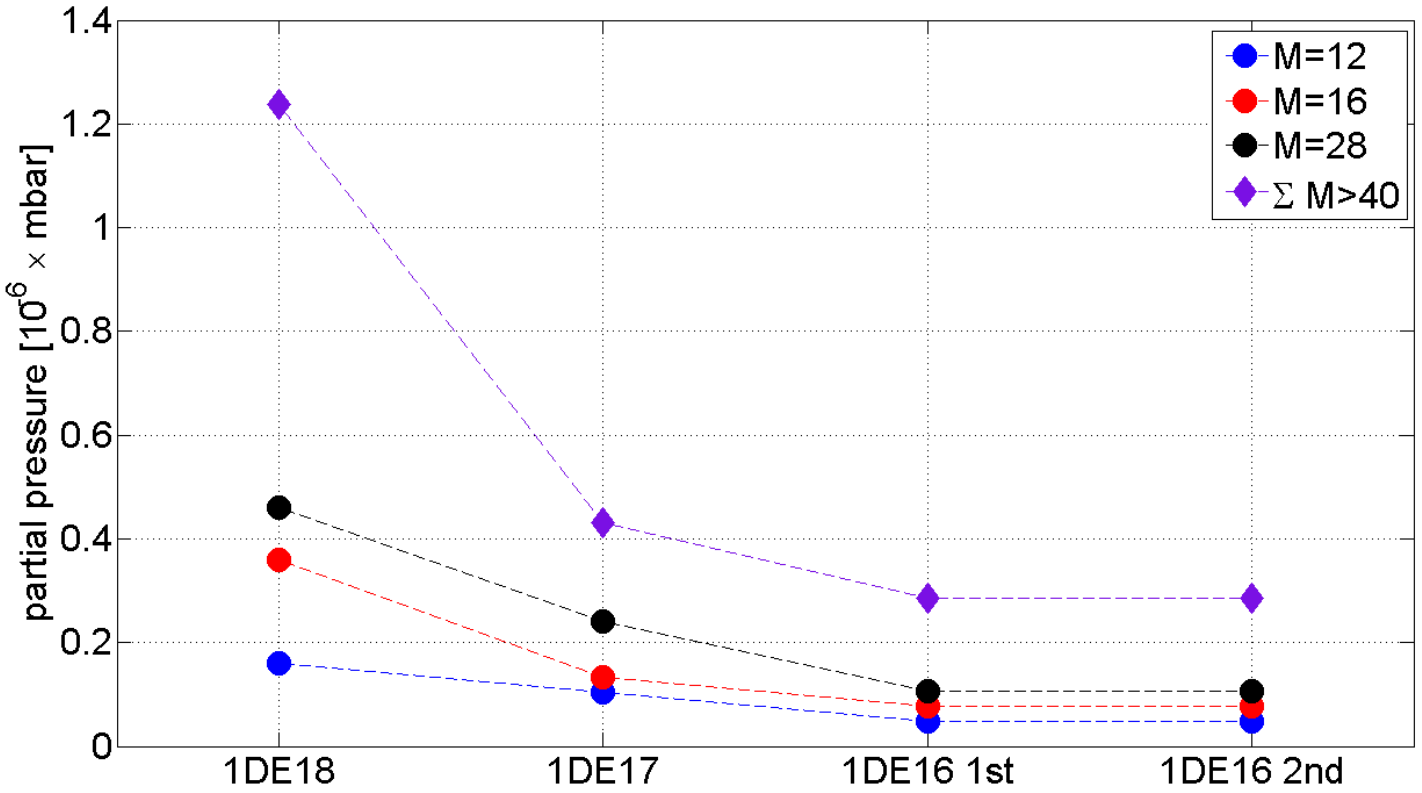
No improvement of baseline performance yet



Infusion recipe:



All so far heat treated cavities without post chemical etching:



Infusion cavity treatments at DESY

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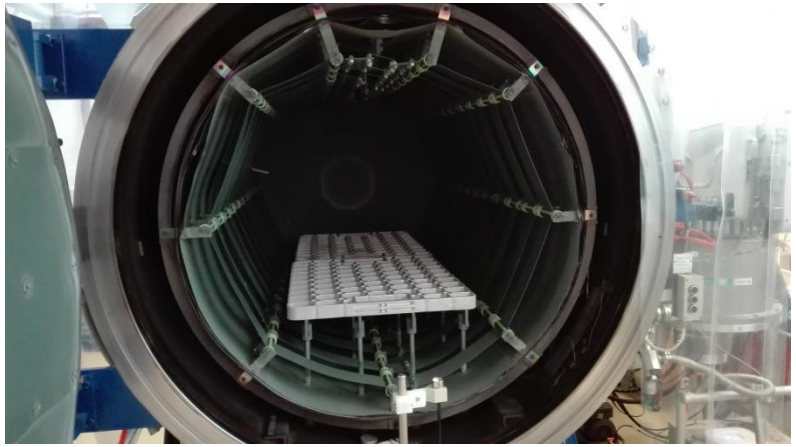
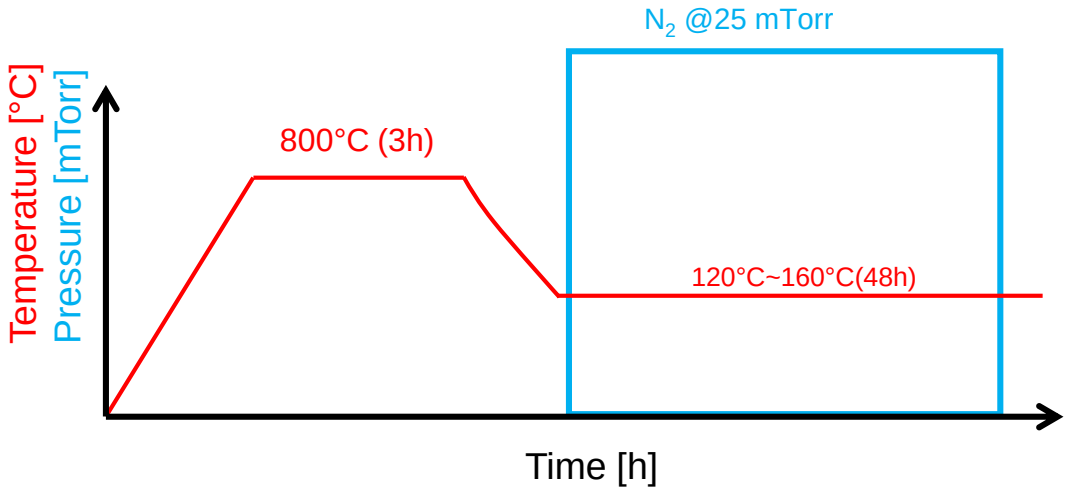
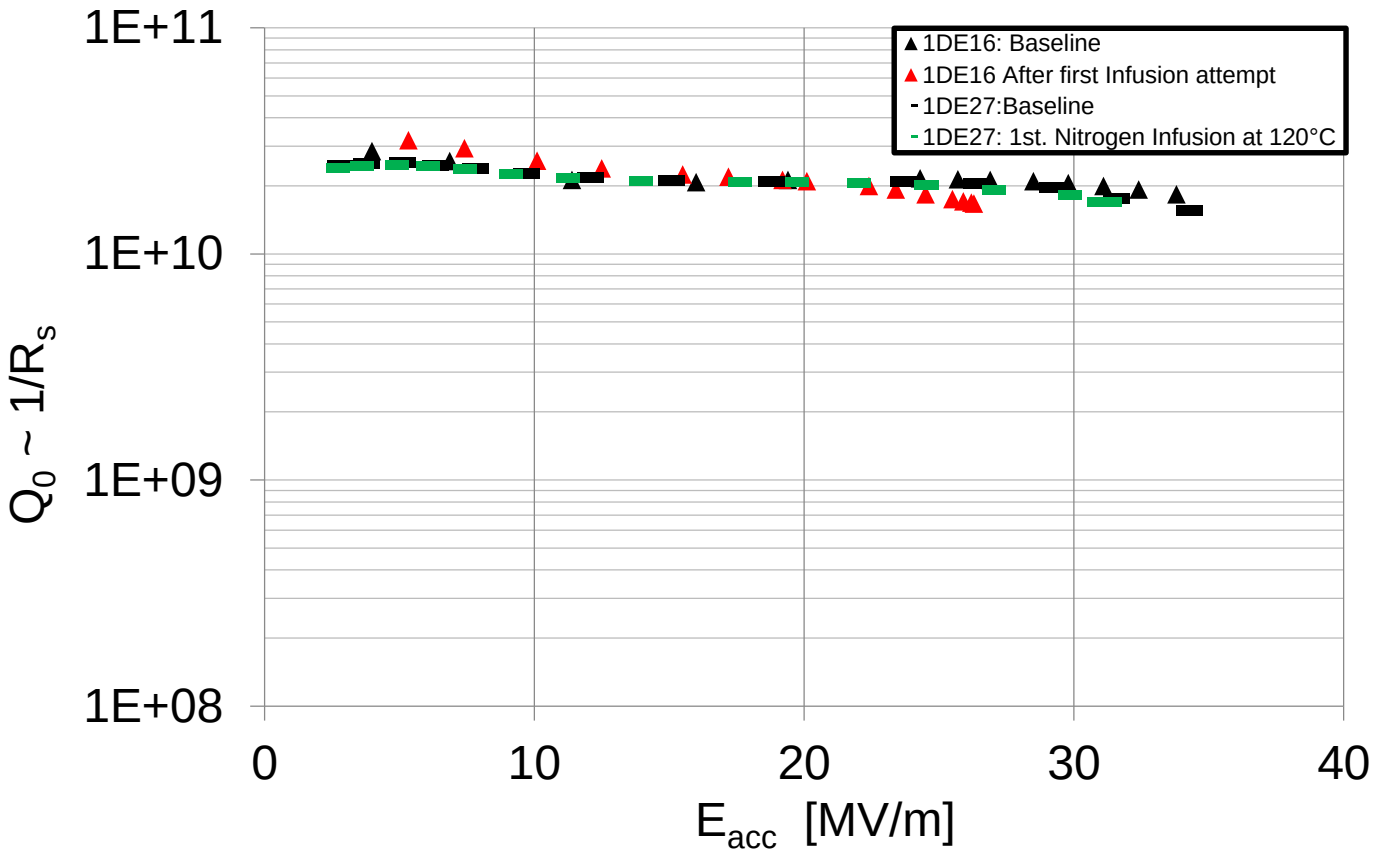


Infusion recipe:



Baseline could be reproduced twice:



Infusion cavity treatments at DESY

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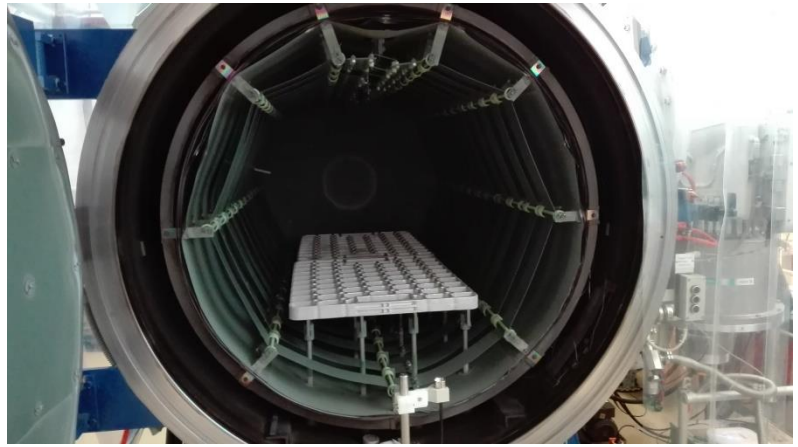
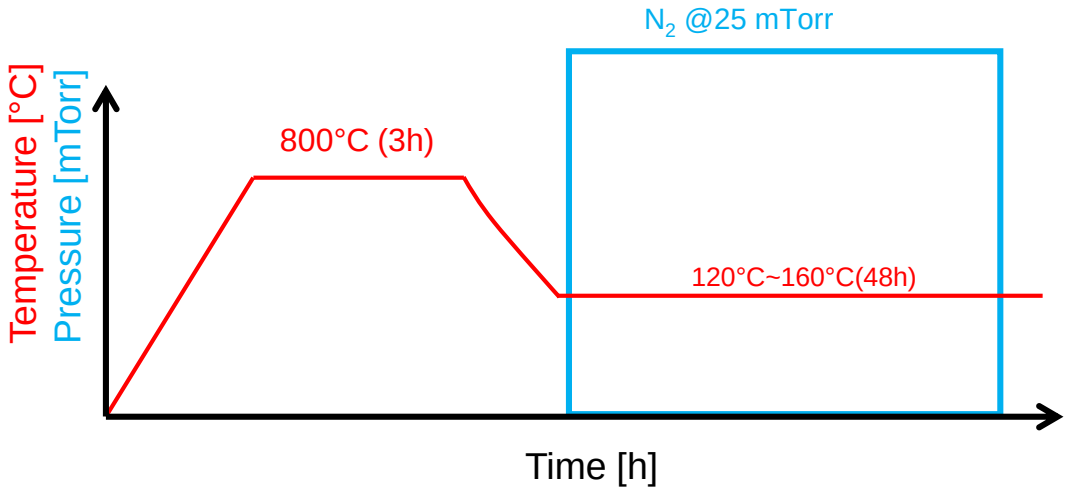
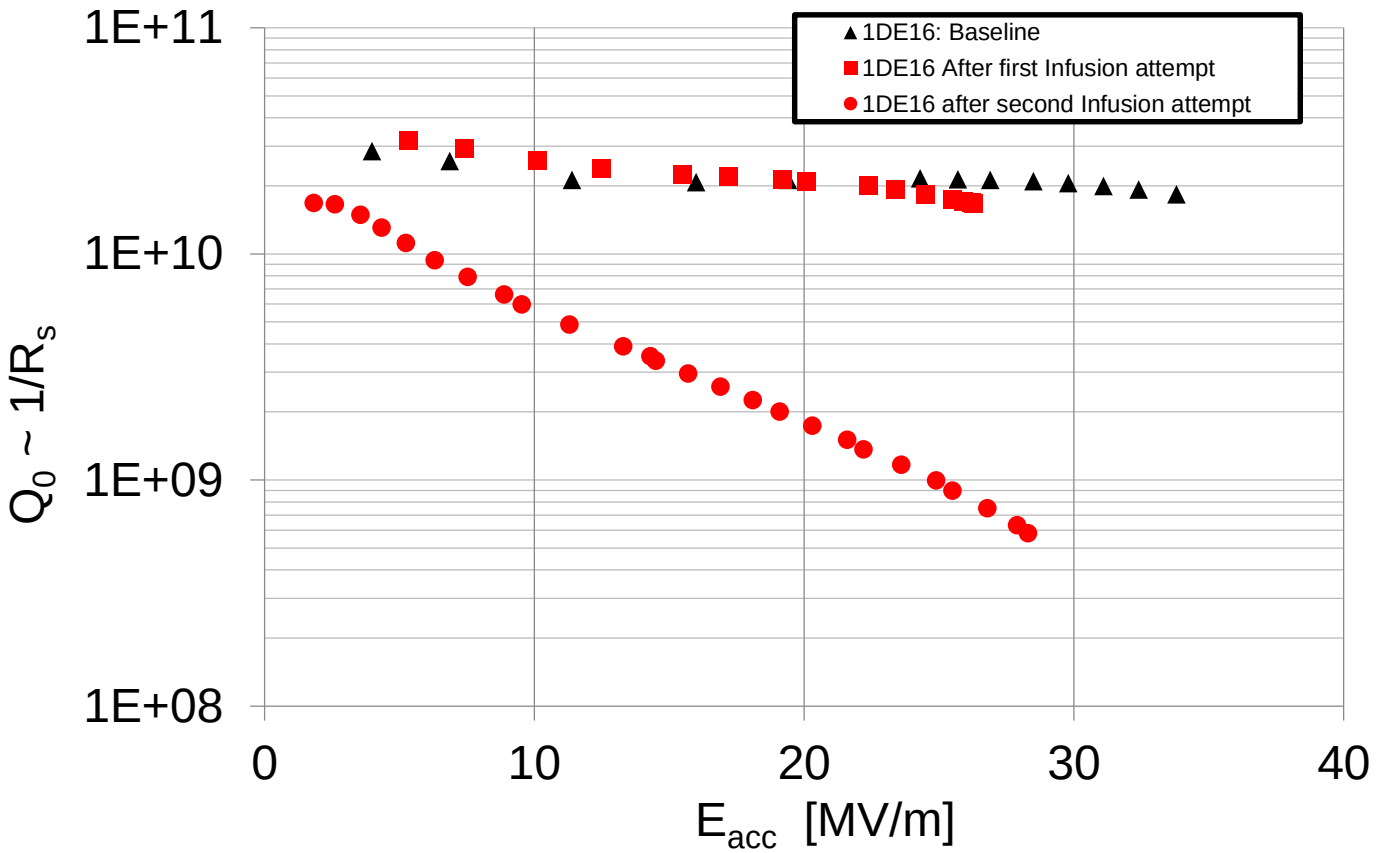


Infusion recipe:



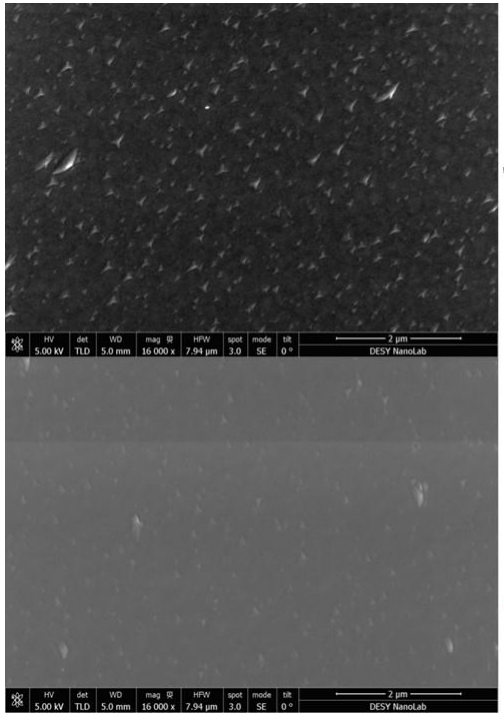
This cavity has been cut:

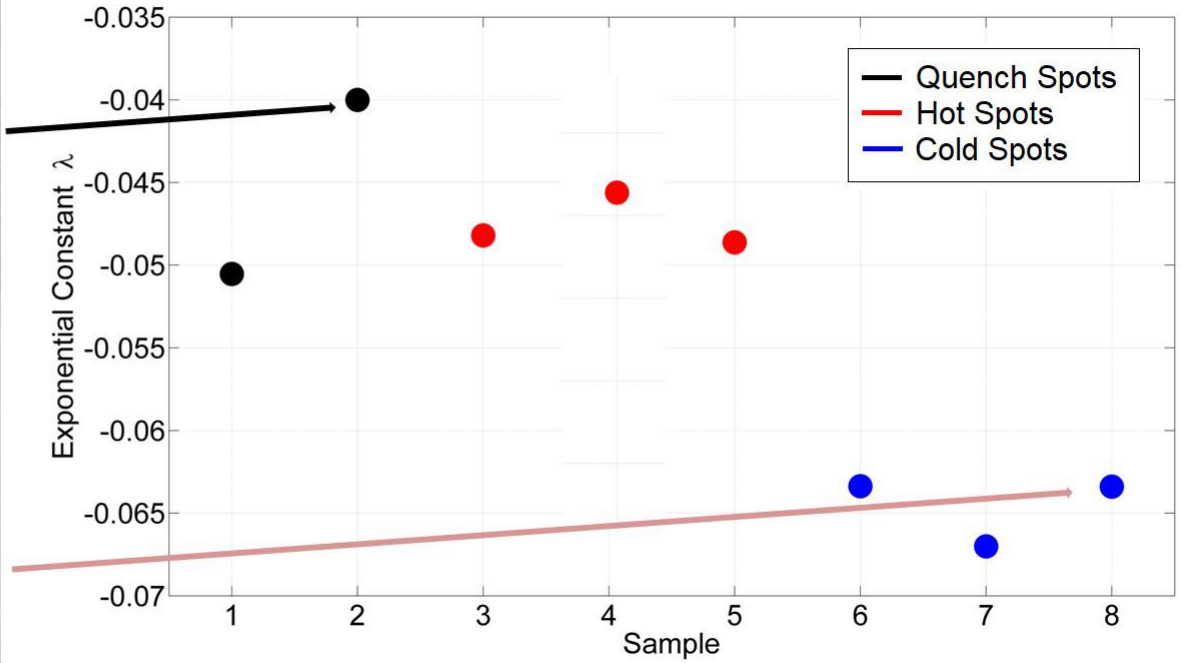


Cavity cutout

Analysis of cutouts from degraded cavity

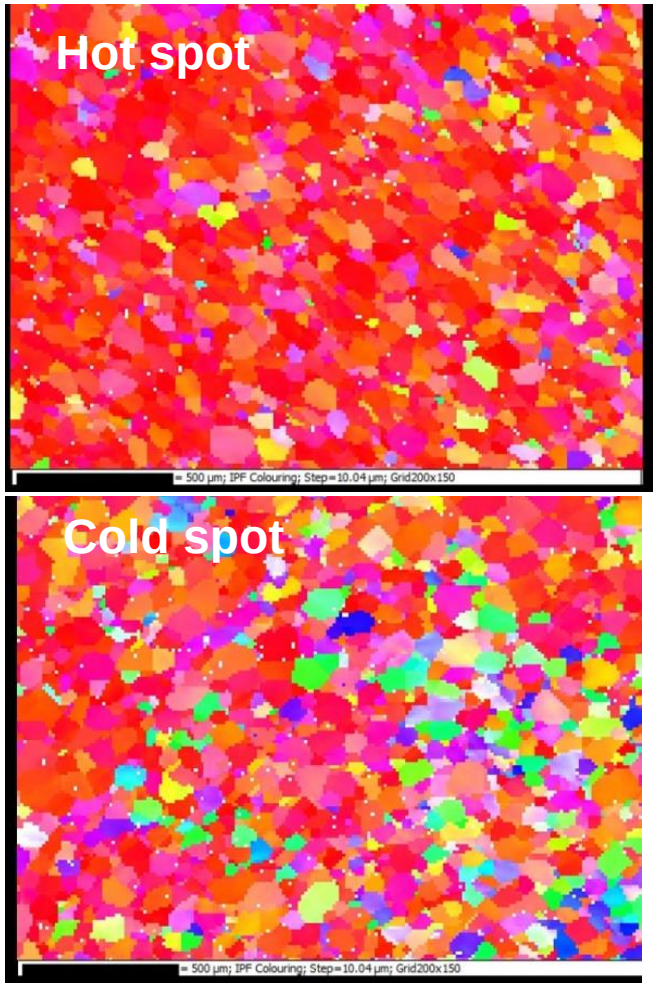
- Cold and hot spots were identified via Tmap and Hmap at HZB
- SEM image analysis shows higher size distribution of carbides on quench and hot spots compared to cold spots

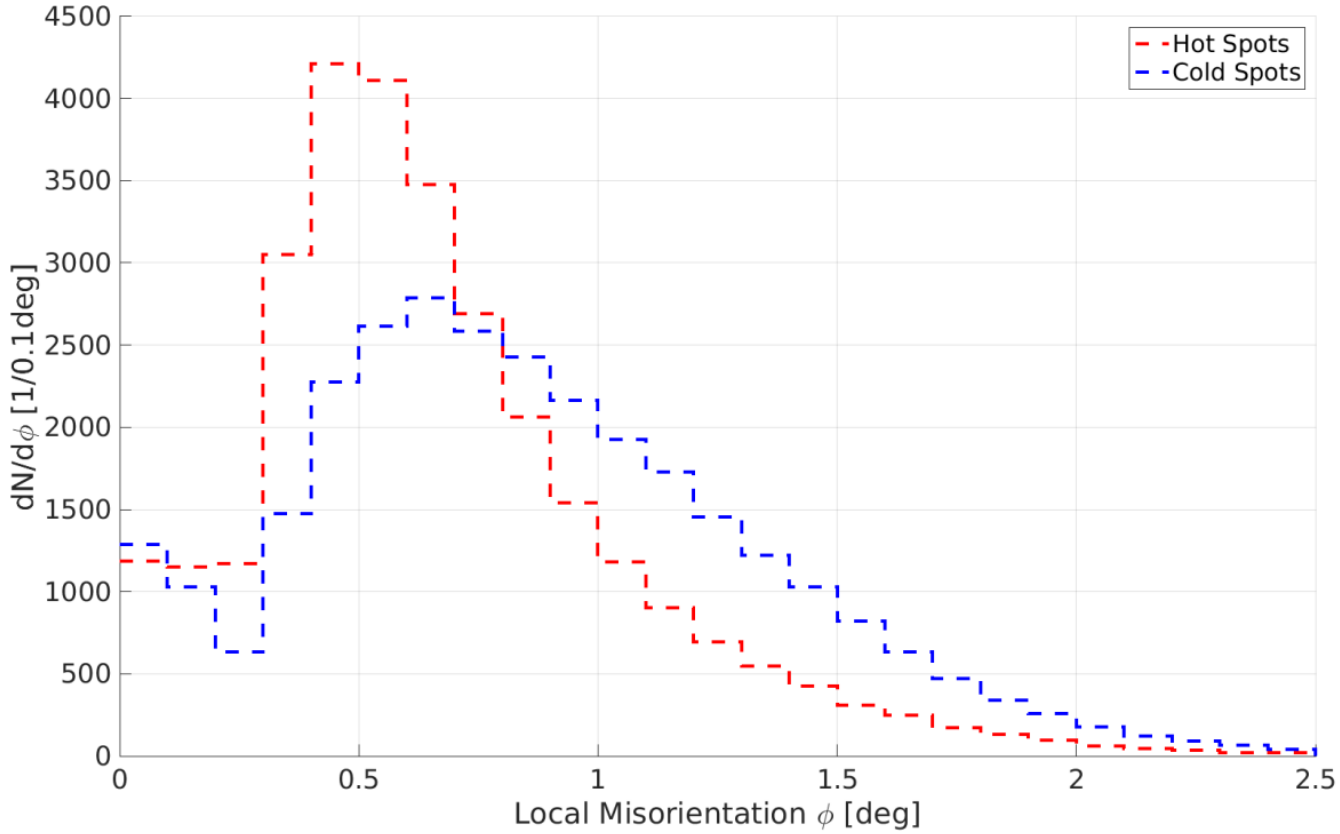




Cavity cutout

Origin for locally different carbon aggregation



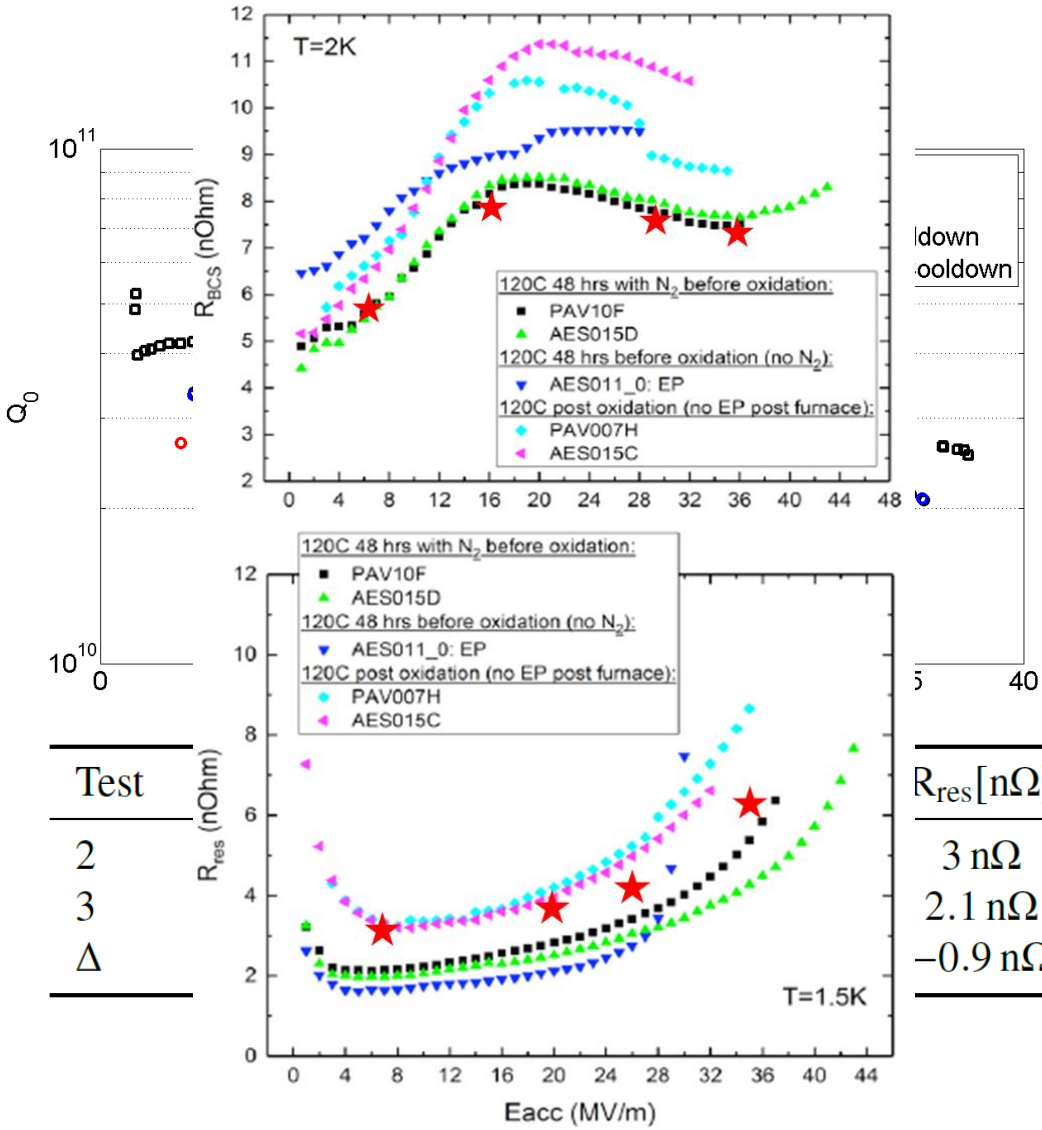


- Electron backscatter diffraction measurement shows more low angle grain boundaries for hot spots compared to cold spots

Infusion cavity treatments at DESY

Tackling the problems

- No successful Infusion at DESY yet
- Sample/Cutout studies revealed carbide formation as cause for degradation
- Assumptions for ‘failing’:
  - Carbide formation due furnace contamination with hydrocarbons
  - Carbide formation due lack of cleanliness and vacuum conductance of ‘line of sight’ protection
- No degradation but also no improvement
  - Recent runs show that we are at the limit of our furnace, the cavities seem to be unchanged after treatment -> now detailed studies on furnace status
- Infusion of DESY-Cavity at FNAL
  - Test at FNAL and DESY for comparison
  - Difference in Q0 mainly due to difference in Rres; RBCS is the same at the labs
  - Origin for this unclear
    - Magnetic hygiene of test infrastructure? No evidence
    - Cooldown parameters? Infused Cavities don’t show dependency on those



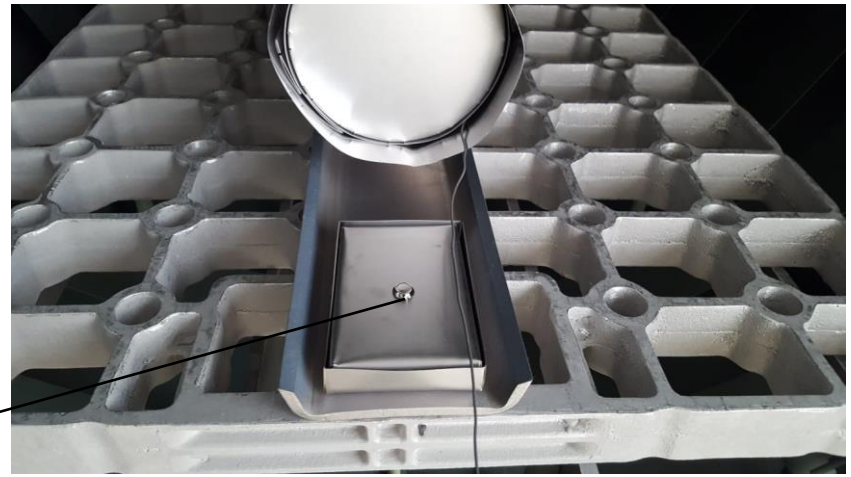
Test	$R_{res}[n\Omega]$
2	3 nΩ
3	2.1 nΩ
Δ	−0.9 nΩ

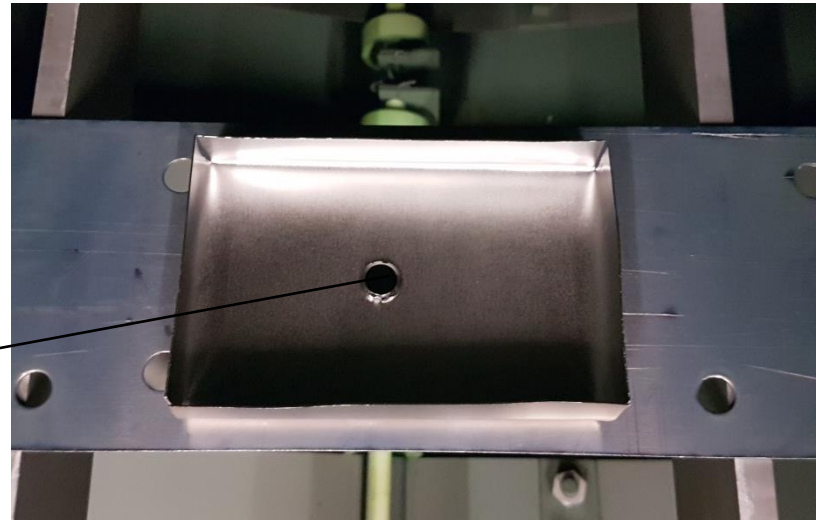
[Wenskat et al., RF2019, MOP025]

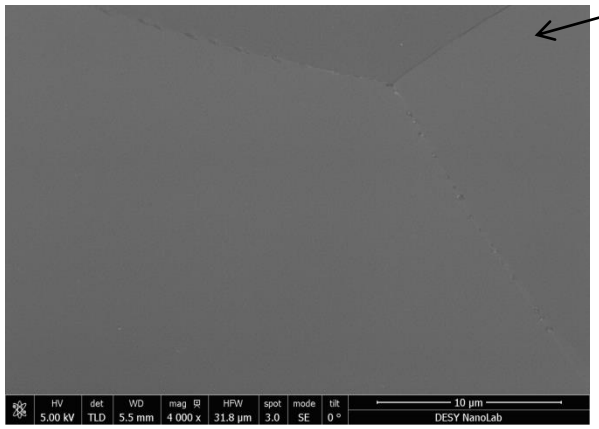
Carbide precipitation during heat treatment

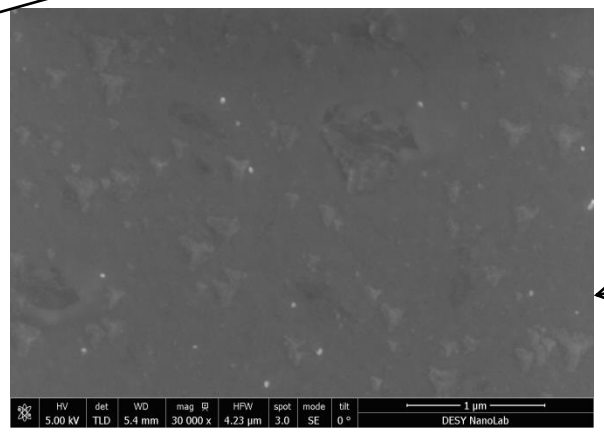
Carbide precipitation occurred on niobium samples

- A niobium box for the samples was used to mimic the ‘line of sight’ protection of the niobium caps for the cavities
- For most runs sample inside the box showed carbides while the sample outside showed none
- The box was bcp’ed before each treatment just as the caps





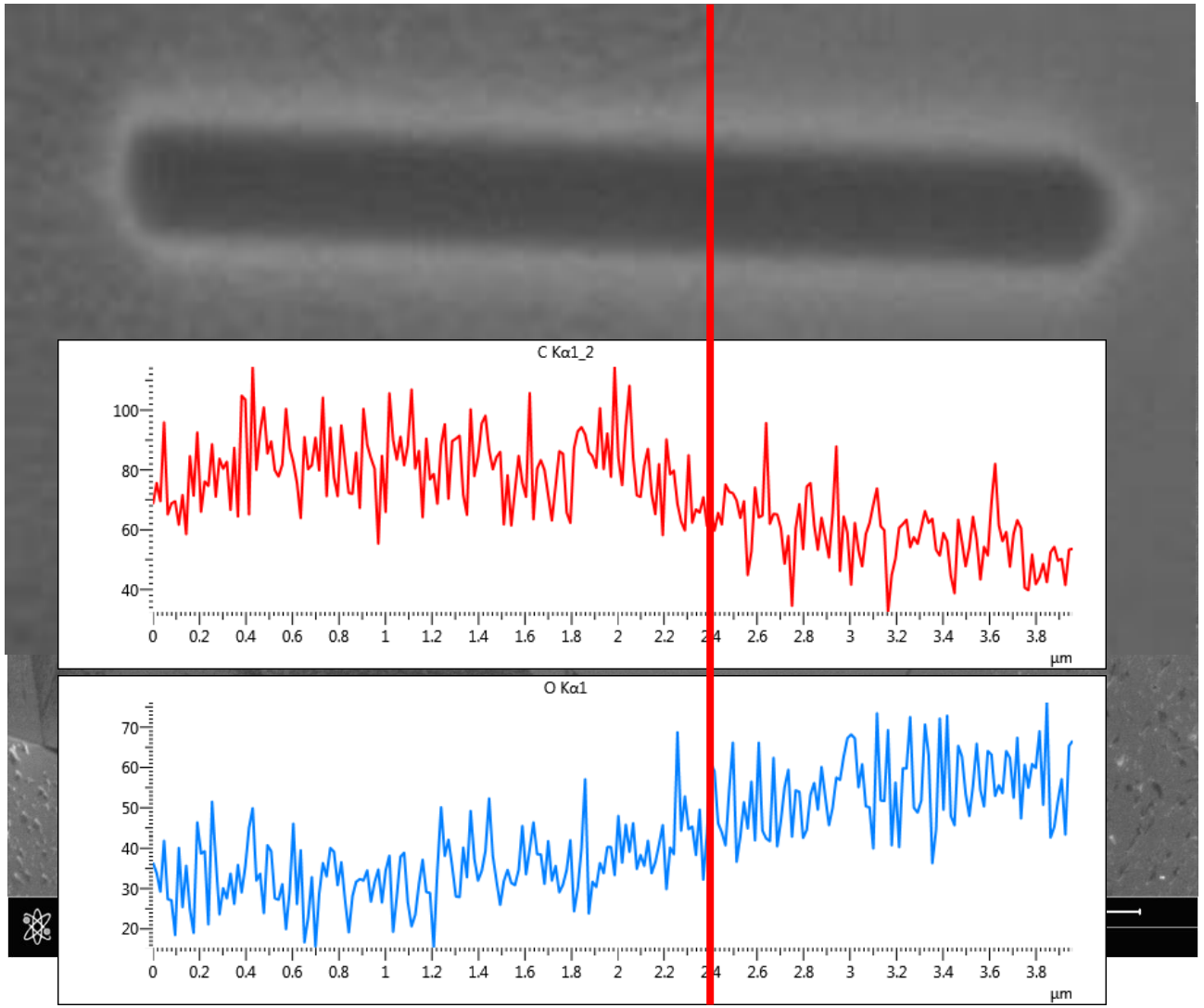




Carbide precipitation during heat treatment

Carbide precipitation occurred on niobium samples

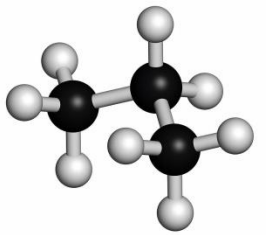
- Besides carbides, dark areas on sample surfaces can be found in the SEM images
- EDX line scan show increase in carbon signal on those areas showing this must be some thin carbon layers that accumulated on the surface
- The carbon layers avoid grain boundaries and some certain kind of precipitates
  - This shows the diffusive absorptive behavior of grain boundaries for carbon
  - In agreement with known importance of grain boundaries for the N diffusion process

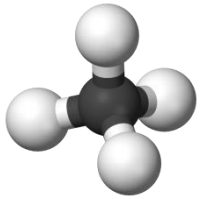


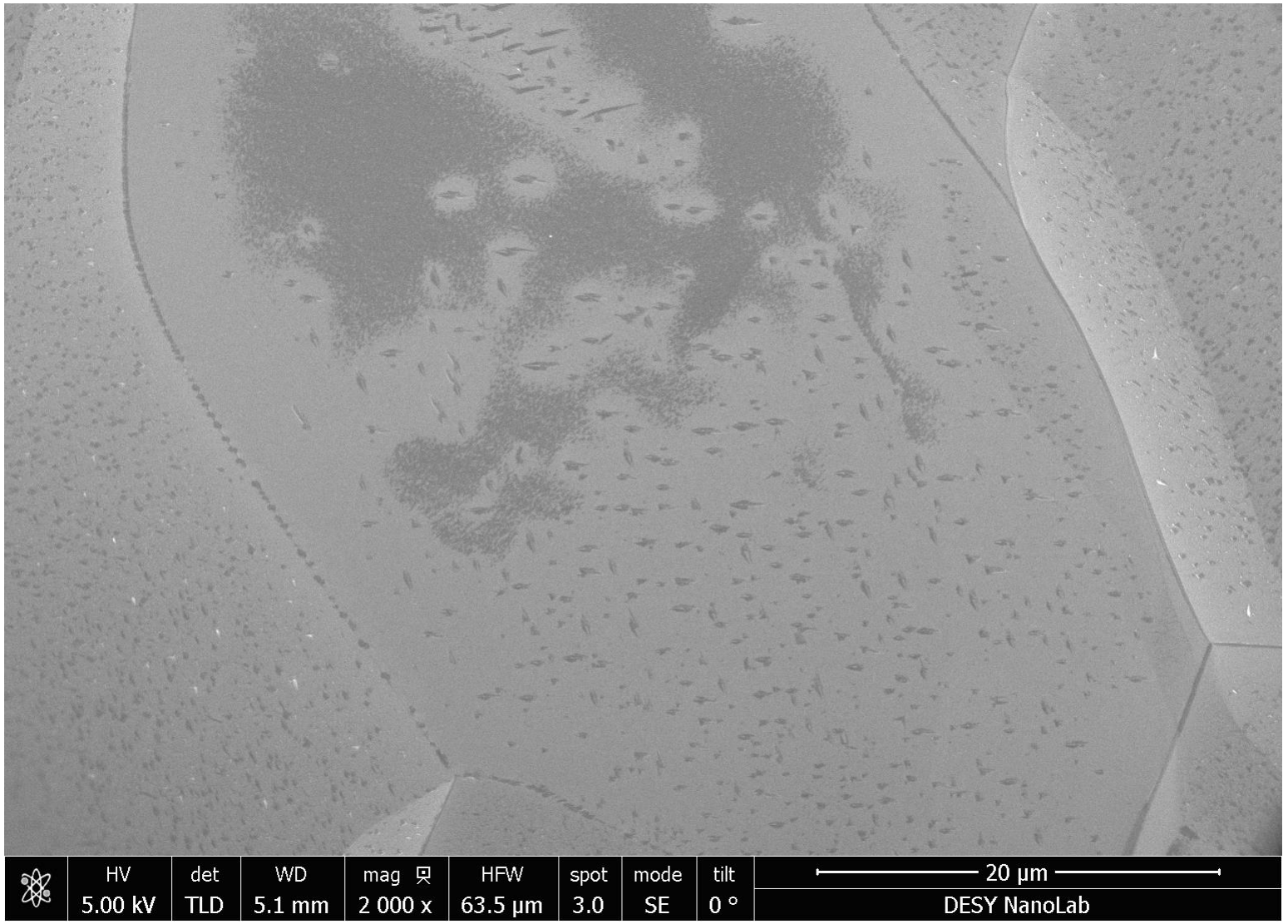
C-doping

Where does the carbon come from?

- Different phases of carbon precipitation are observed
- They might depend on
  - Crystal orientation
  - The composition of the carbon enriched environment (hydrocarbons)
- Possible sources are:
  - Remains of alcohol or during the bcp of the niobium caps/box could exist
  - The furnace itself or the pumping system

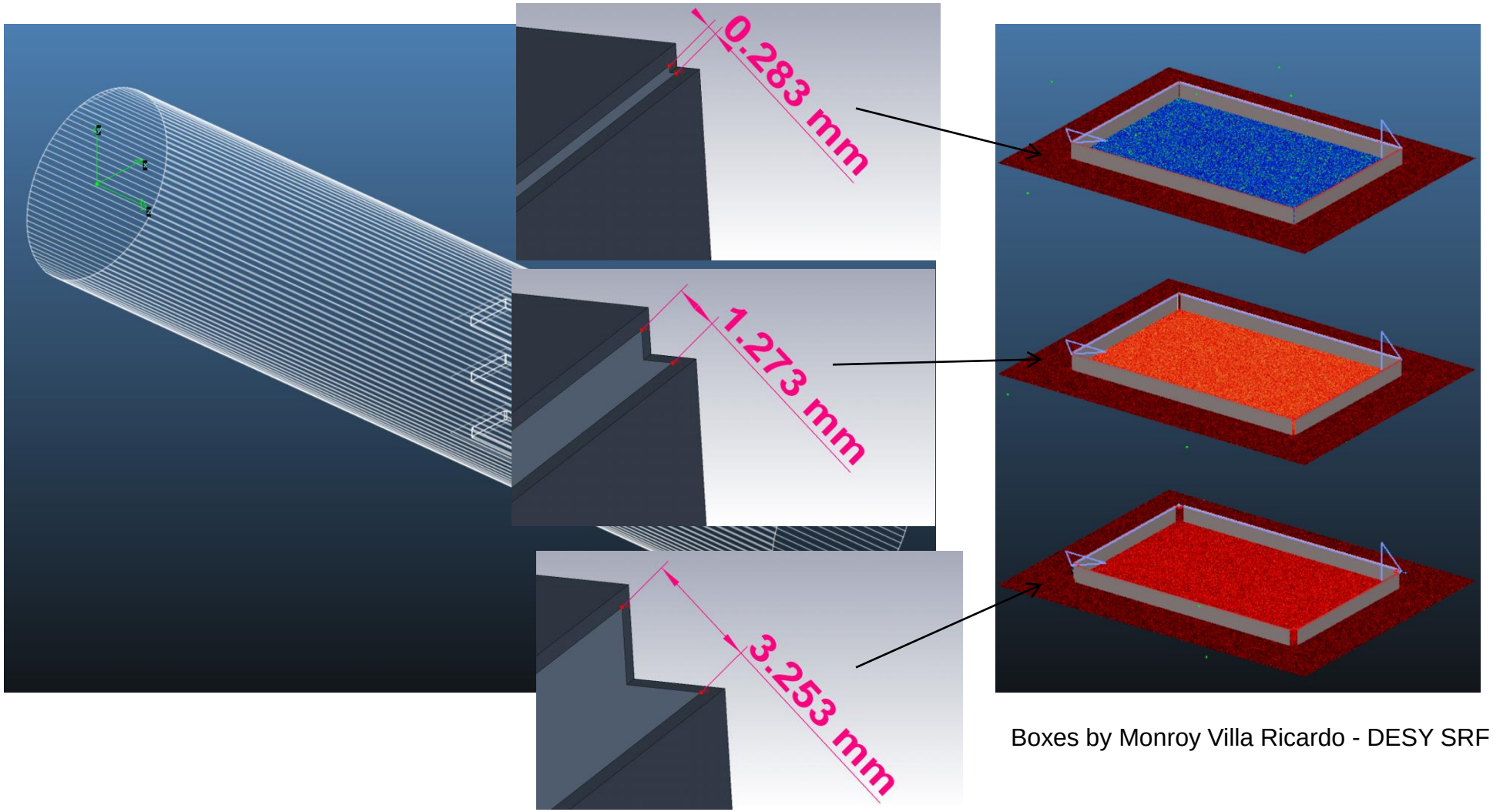






Vacuum conductance of line of sight protection

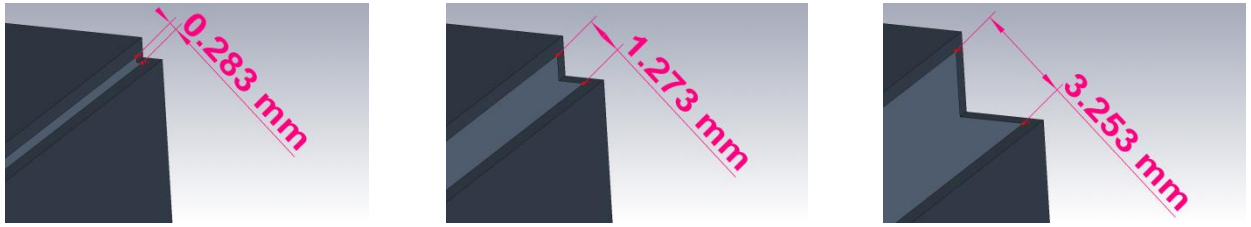
Molflow simulation on niobium boxes in a pipe

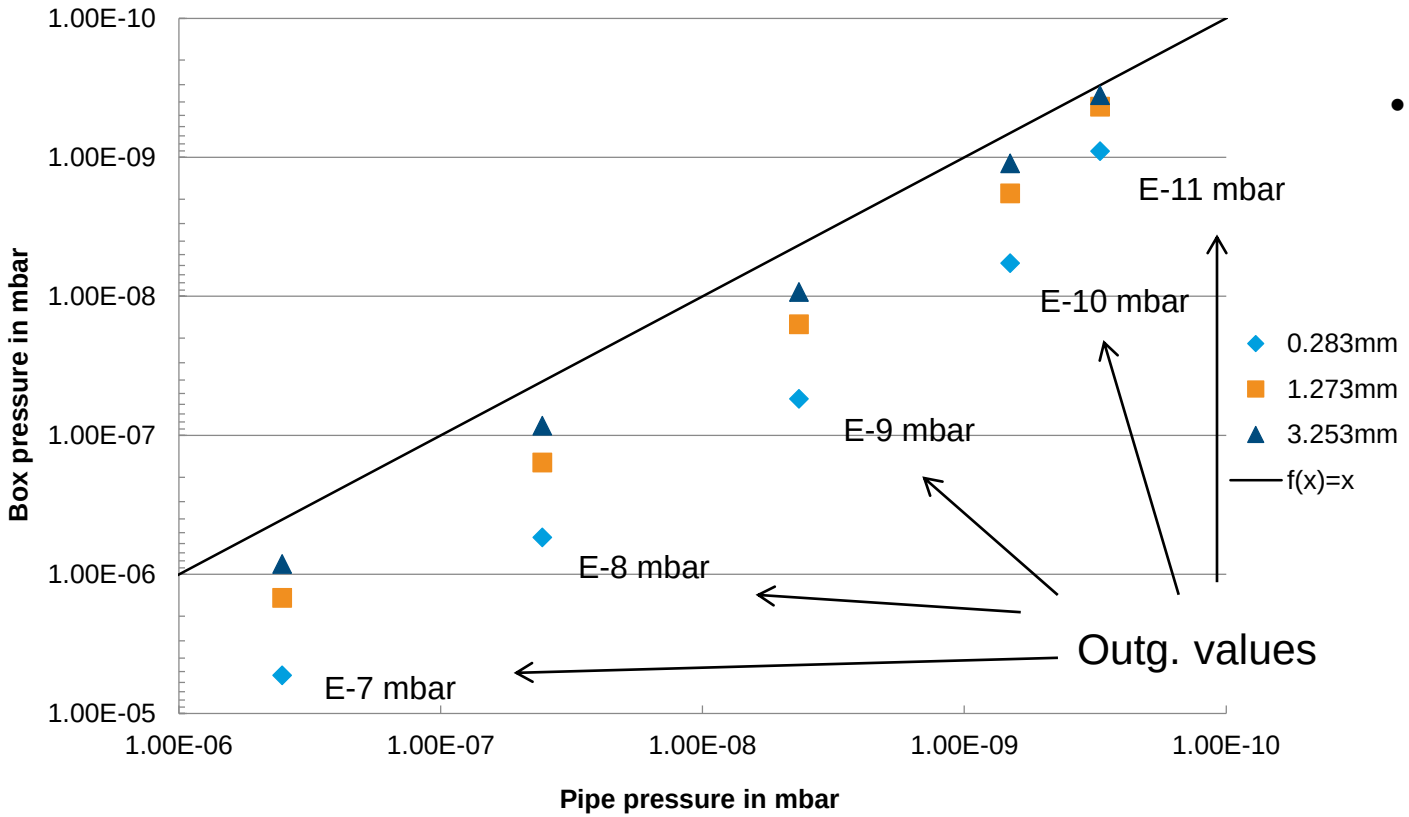


Boxes by Monroy Villa Ricardo - DESY SRF

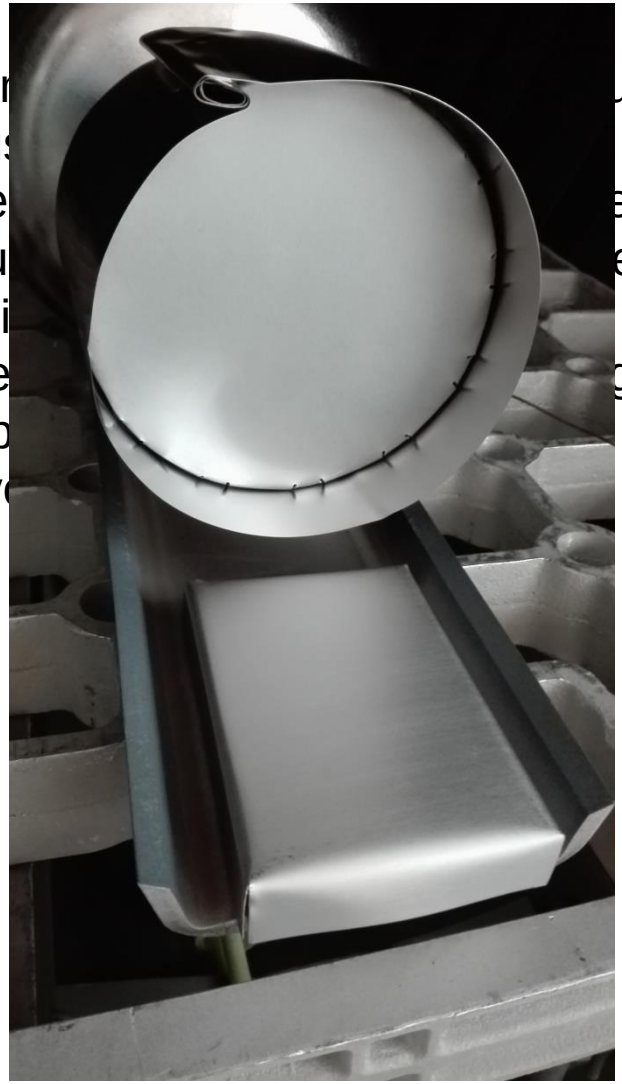
Vacuum conductance of line of sight protection

Molflow simulation on niobium boxes in a pipe





- Differer um box were us
  - Le essures
- The qu e (gap width) i
- For the g value of E-7 mb is observe

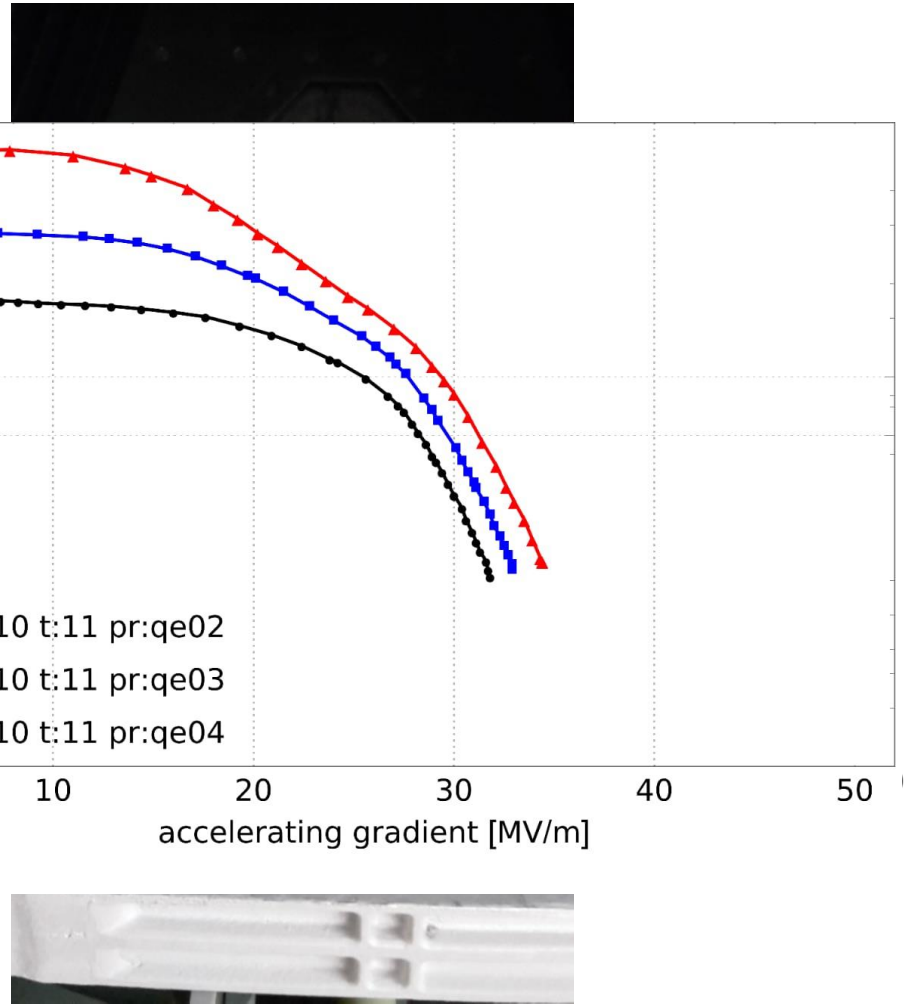


New runs with different line of sight protection

Furnace assessment

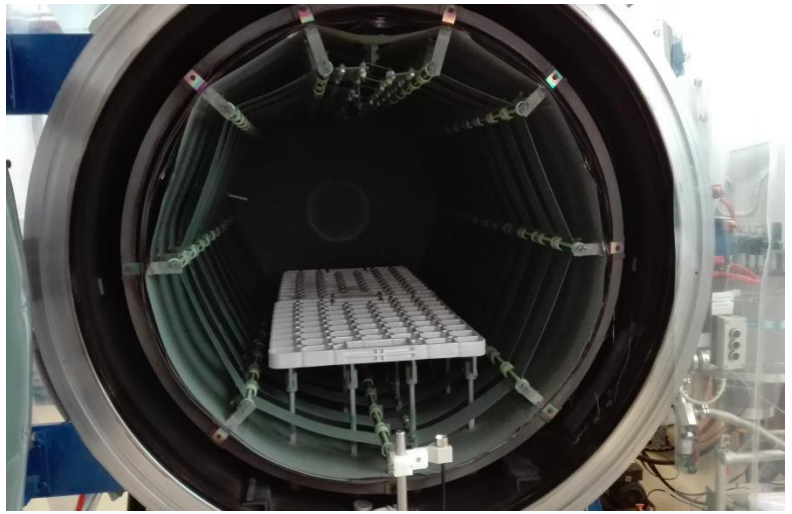
1. 800°C w/o caps and w/o Nitrogen:
   - Expectation: A clean furnace would simply re-set the surface to a status prior to 120°C bake; HFQS should re-appear
   - Observation: Strong degradation of Q at low fields, similar to first infusion runs

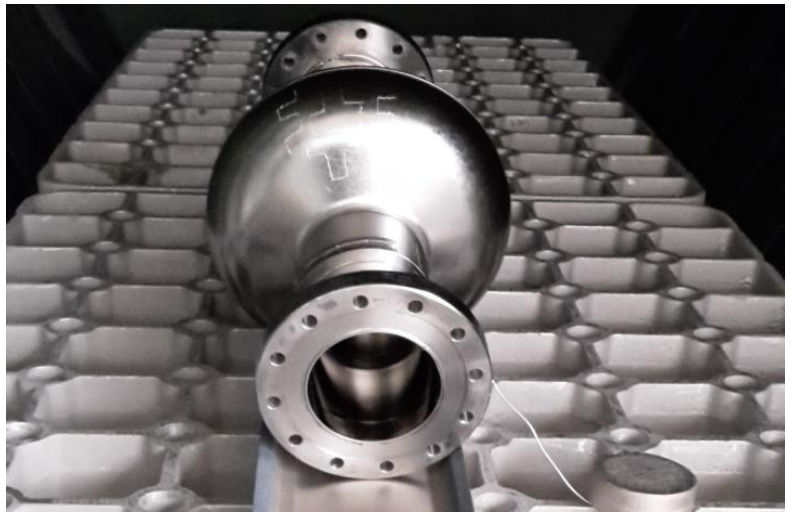
2. 650°C with modified caps and w/o Nitrogen:
   - Expectation: Lower T leads to a better pressure but T is sufficient to outgas the cavity and break up the oxides. Hence, HFQS should re-emerge.
   - Observation: HFQS re-emerges. Measurements at 1.8K and 1.5K show another loss mechanism kicking in at ~18MV/m before HFQS starts.



Summary

- Evidence for correlation between carbides and cavity performance degradation
- Carbide formation is in connection with
  - Cleanliness and vacuum conductance of line of sight protection
  - Furnace conditions
- Our cavity furnace will undergo an upgrade till Q3 2020 (oil free pre-pumps and software upgrade)
- Magnetic hygiene will be studied and Helmholtz-Coils for active field cancellation will be prepared for future tests.
  - Origin for difference in Rres for FNAL and DESY tests of 1DE20 is unclear



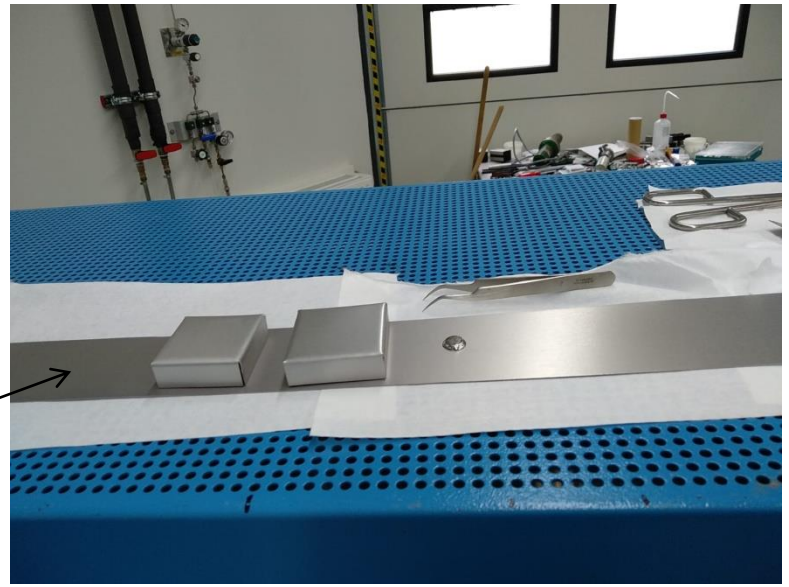


Thank you

BACKUP - pre-treatment of line of sight protection

Influence from line of sight pre-treatment on sample surface

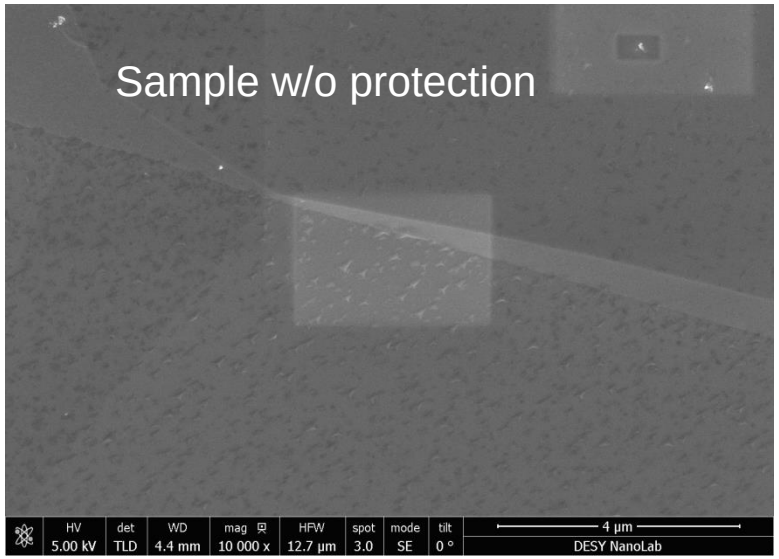
- To investigate the influence of the line of sight preparation on carbide precipitation a test with 3 samples was done in the sample furnace
- The samples were all bcp'ed
  - Sample 1: placed without line of sight protection
  - Sample 2: Beneath a 'only bcp'ed' niobium box
  - Sample 3: Beneath a niobium box that was bcp'ed and pre annealed at 1000°C in the sample furnace
- The samples were then baked at 800°C for 2h (Pressure in sample furnace @800°C about 2*10^-7mbar)

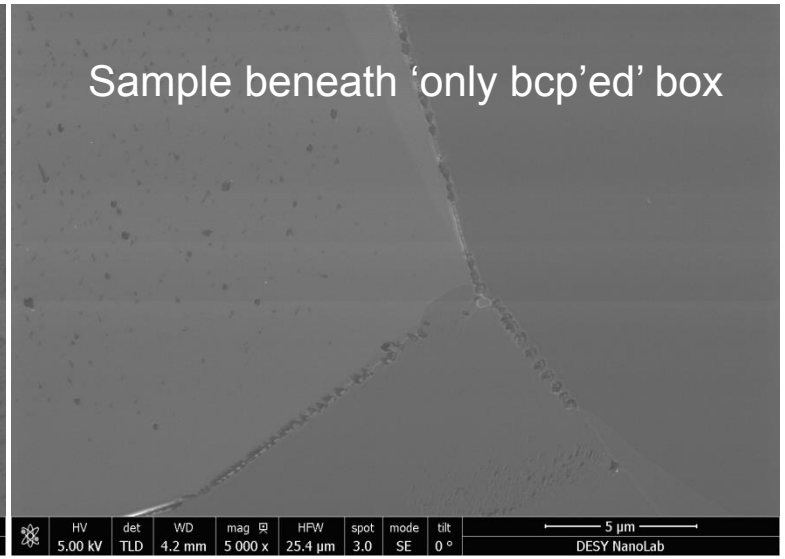


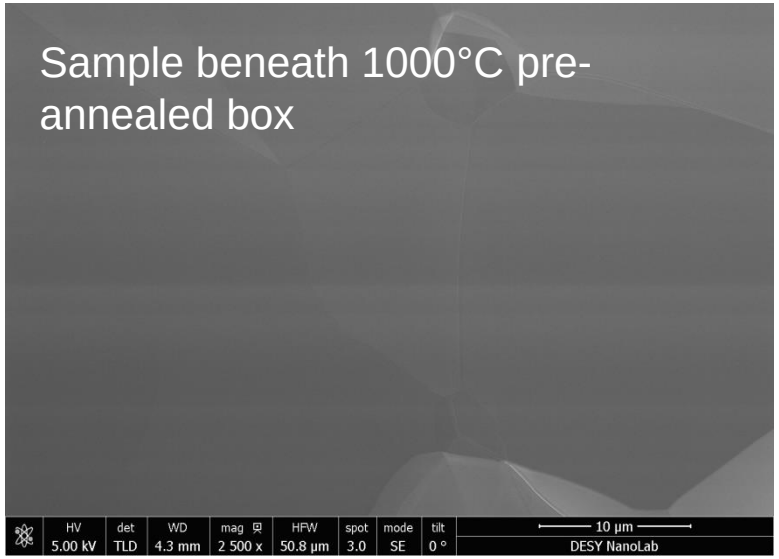
Niobium sheet

BACKUP - pre-treatment of line of sight protection

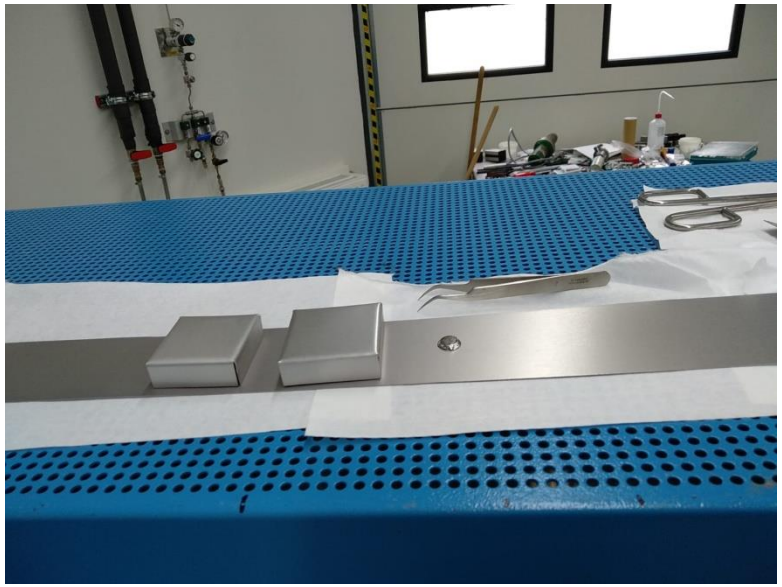
Influence from line of sight pre-treatment on sample surface







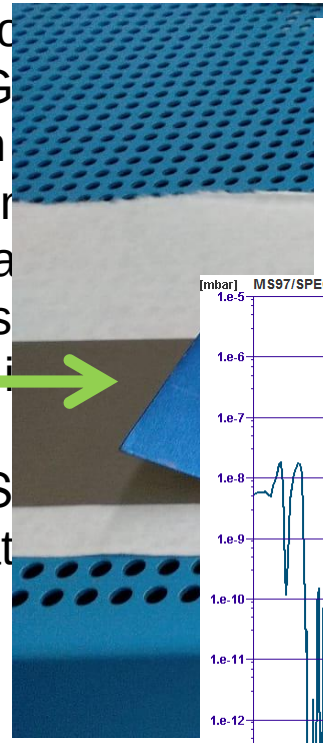
- The test was repeated 3 times with same results
- Carbide formation on sample without line of sight protection indicate some hydrocarbon contamination in the furnace
- This test shows pre annealing the line of sight protection helps to avoid carbide precipitation

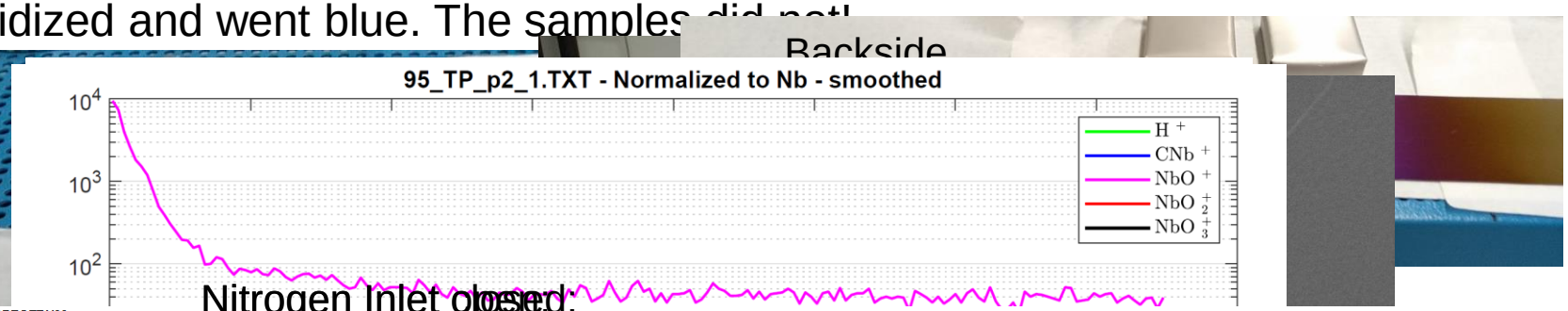


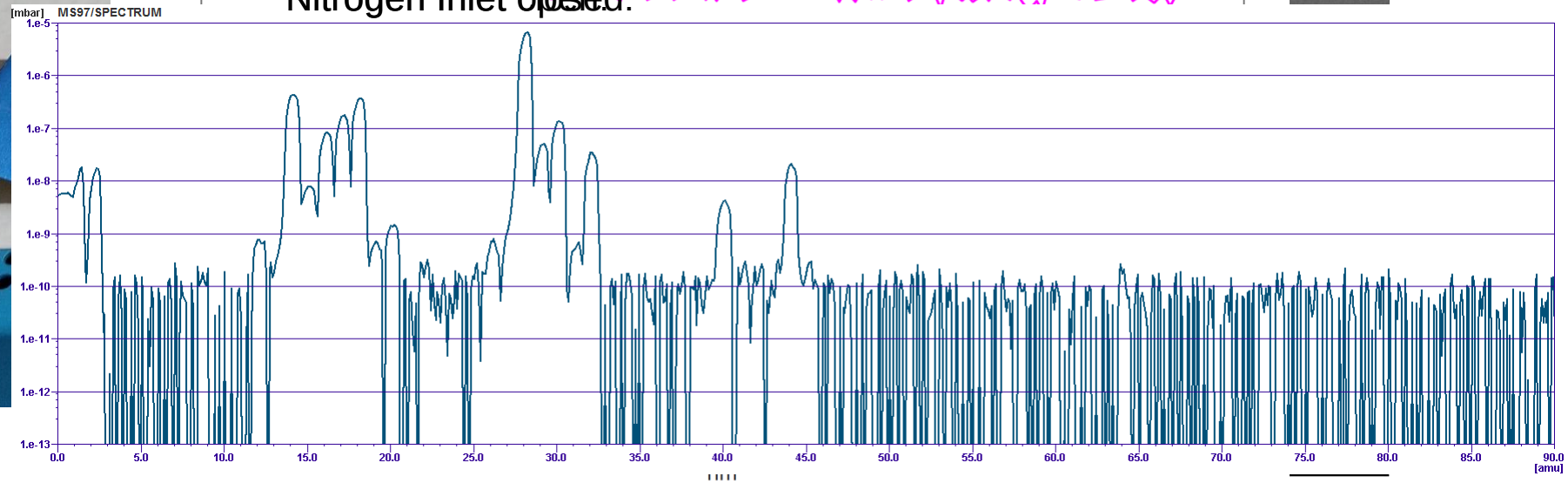
BACKUP - Nitrogen Infusion in sample furnace

So far no nitrogen has been found via TOF-SIMS on our samples

- To force a Nitrogen signal we did Nitrogen Infusion at 400°C (first ramping to 800°C) in the sample furnace
- 1 fine grain and 1 large grain sample were covered each with a niobium box
- The box was pre-annealed at 1000°C after BCP
- Parts of niobium the sheets oxidized and went blue. The samples did not!
- SEM revealed nc
- TOF-SIMS of LG
  - No Nitrogen
  - High Oxyger
    positive pola
- RGA Spectrum s
  leak at Nitrogen i
- Next step TOF-S
  result for FG mat

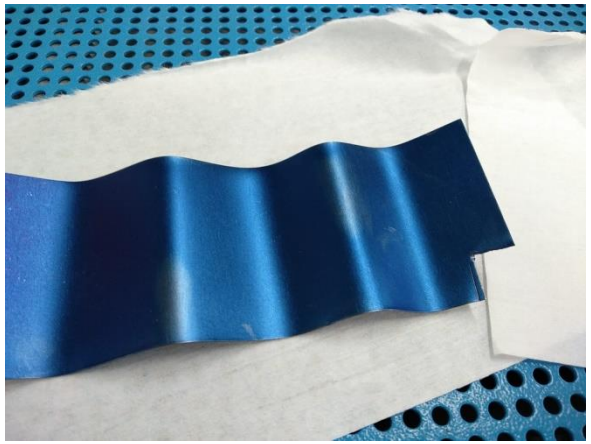


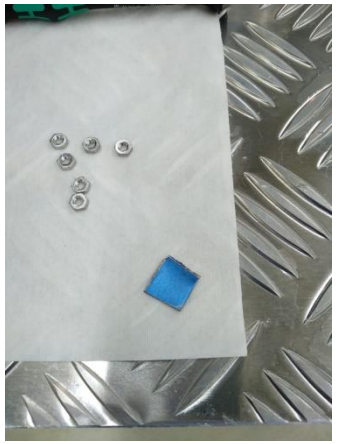




BACKUP - Nitrogen Infusion in sample furnace

- EDX of sheet cutout shows high oxygen peak





Spectrum Label	Spectrum
N	0.00
O	21.60
Si	6.75
Nb	71.65
Total	100.00