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Mid-T Baking:
Development of a Recipe for a Simple and Inexpensive *in-situ* Processing

A. Prudnikava, Y. Tamashevich, A. Matveenko,
A.Neumann, O. Kugeler, J. Knobloch

Alena.Prudnikava@helmholtz-berlin.de

Making Defects: Mid-T Baking Worldwide

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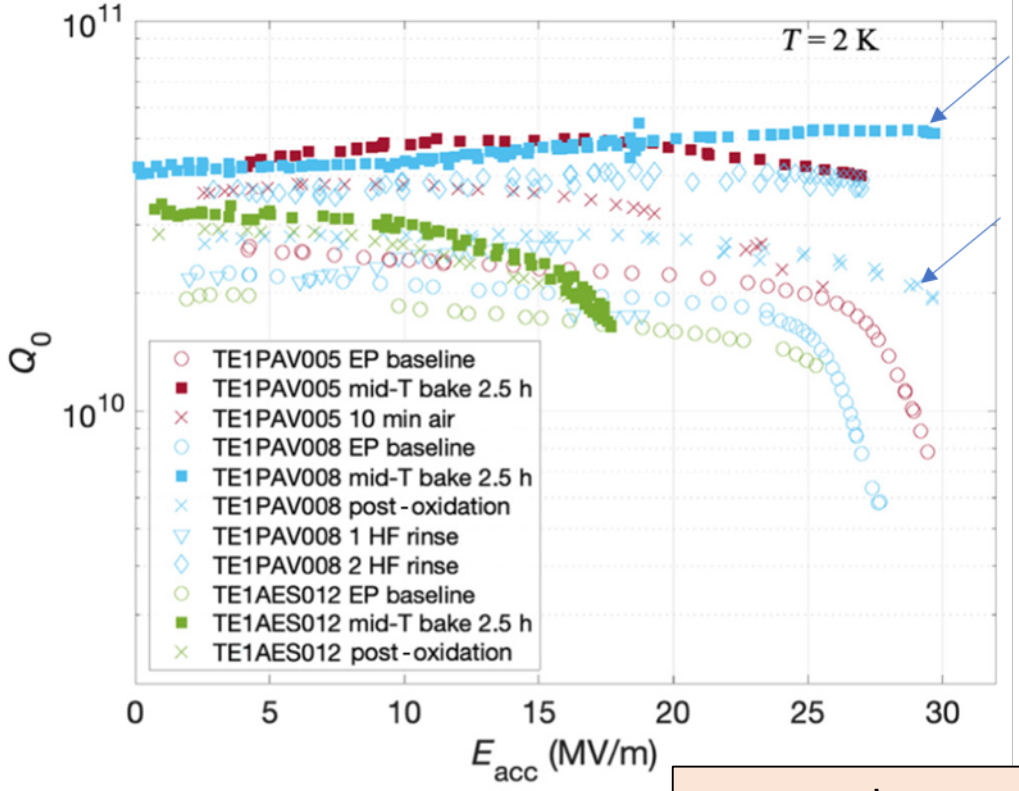
Ultralow Surface Resistance via Vacuum Heat Treatment of Superconducting Radio-Frequency Cavities

S. Posen , A. Romanenko, A. Grassellino, O.S. Melnychuk , and D.A. Sergatskov, 2020

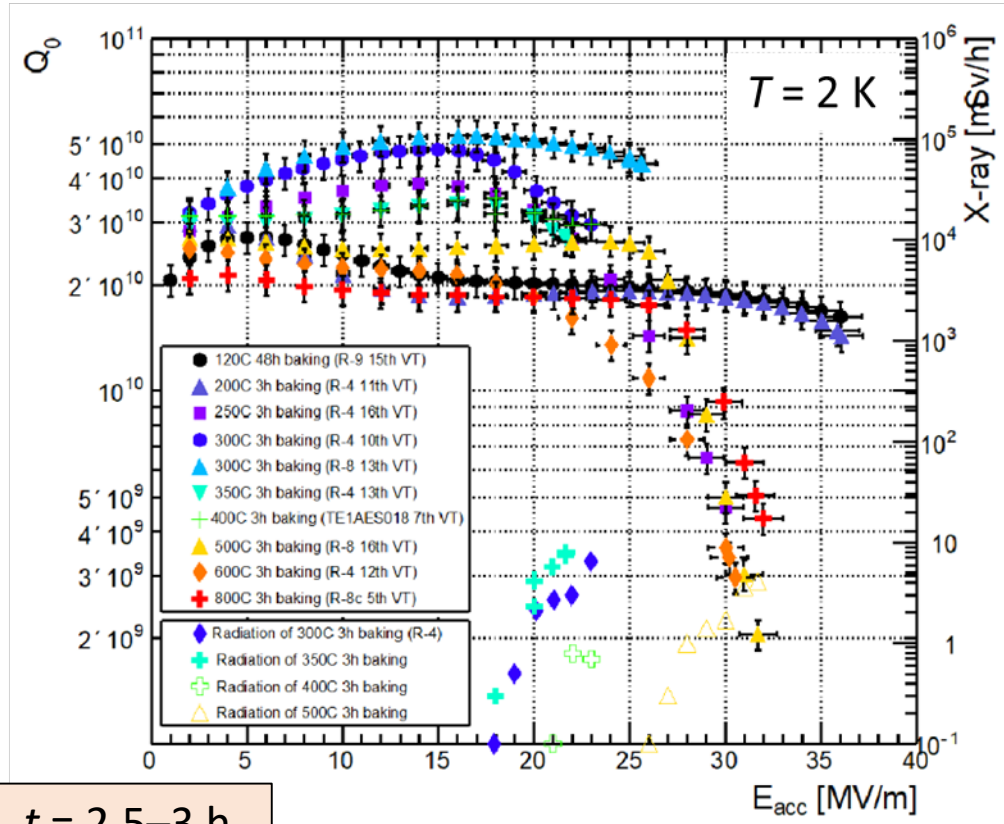
Systematic Investigation of Mid-T Furnace Baking for High-Q Performance.

Ito, H., Araki, H., Takahashi, K. and Umemori, K., 2021.



300 °C, 2.5 h

after air exposure



Best results at T = 300–350 °C, t = 2.5–3 h
Some Q-degradation after air exposure
Often: E_{max} reduction and FE or Quench

Why using Nb Samples?

Different labs give different “best” recipes for cavities. Reasons:

- before baking: cavity **treatment** and **cleaning** history is very important
- **state of the vacuum furnace** has huge impact on results -> can cause Q_0 and E_{max} degradation and FE
- after baking (before test): careful cavity **handling** is very important

It is important to study the Mid-T baking process under controlled conditions

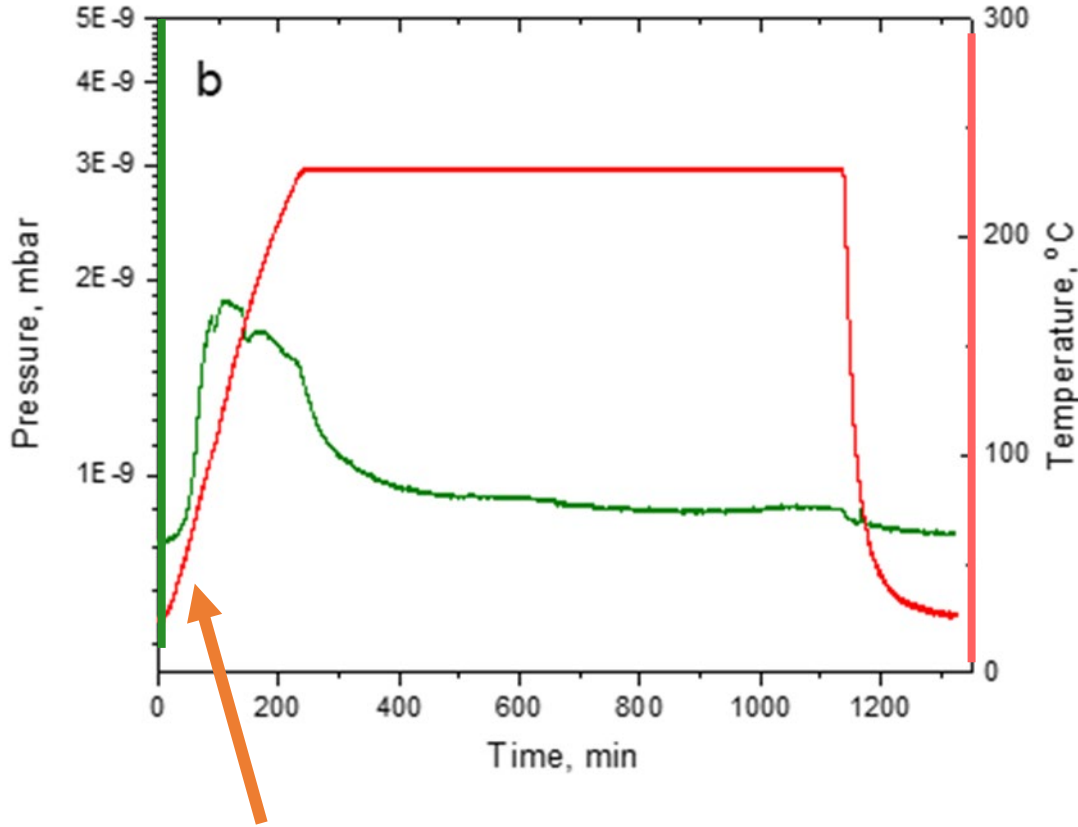
Nb samples by *in-situ* **synchrotron XPS** at BESSY II beamline:

- **Ultra High Vacuum** in the analytical chamber eliminates the influence of the furnace condition
- High brightness **synchrotron X-Ray** source gives very strong signal => fast data acquisition
- *In-situ* measurements: kinetics of oxides dissolution, carbides growth and Nb interactions with impurities

In-situ Study of Mid-T Baking via Synchrotron XPS

Series of baking experiments at 200-400°C

- Before baking: all samples treated together
- UHV: p = 3e-9 mbar and better during baking
- Baking T behaves as in real furnace (**no fast heating**)
- Determined all **NbO_x phases** at Nb surface
- Analyzed interaction of surface impurities
- **Kinetics** of the native oxide dissolution studied

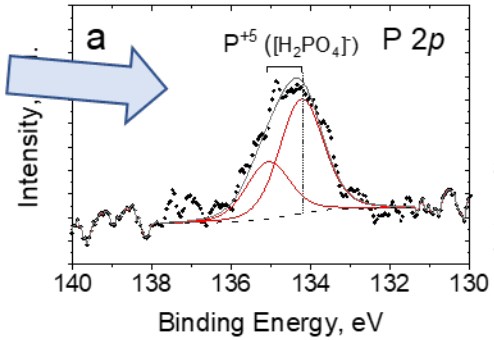


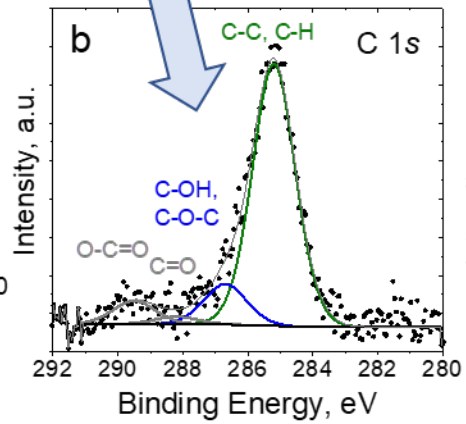
Slow temperature rise to mimick real furnace

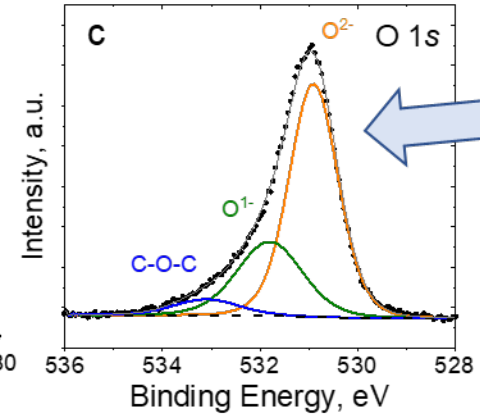
Composition of the cavity surface

Carbon

Phosphorus



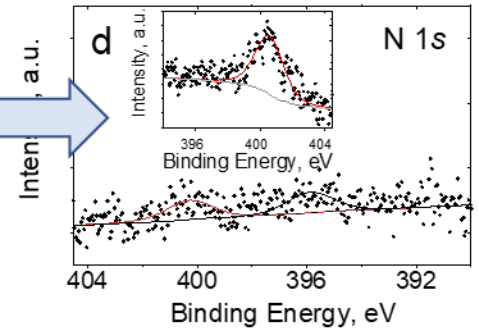




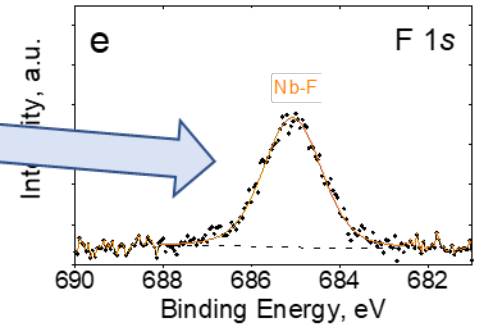
Oxygen

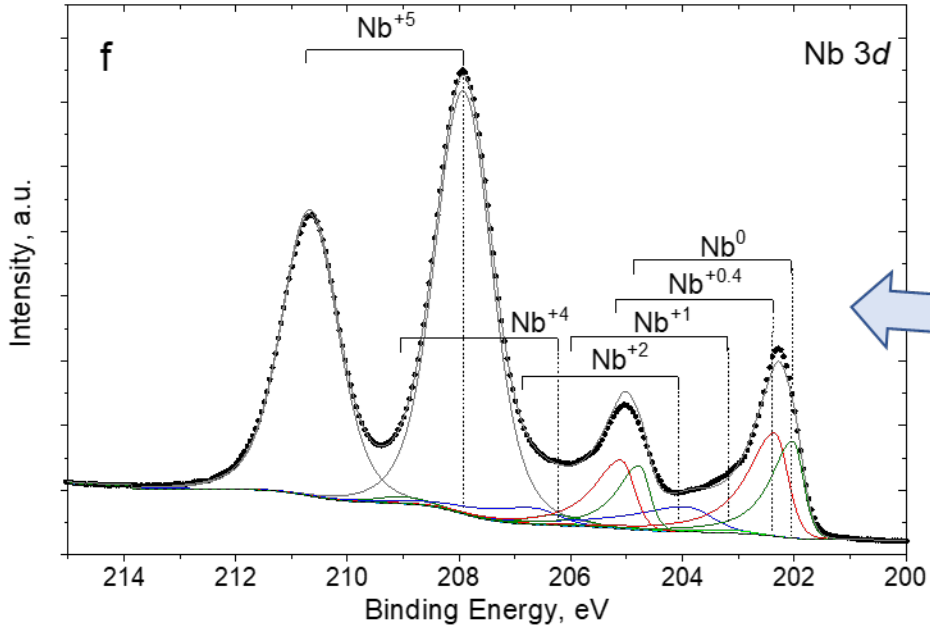
Set of spectra every 20 min!

Nitrogen



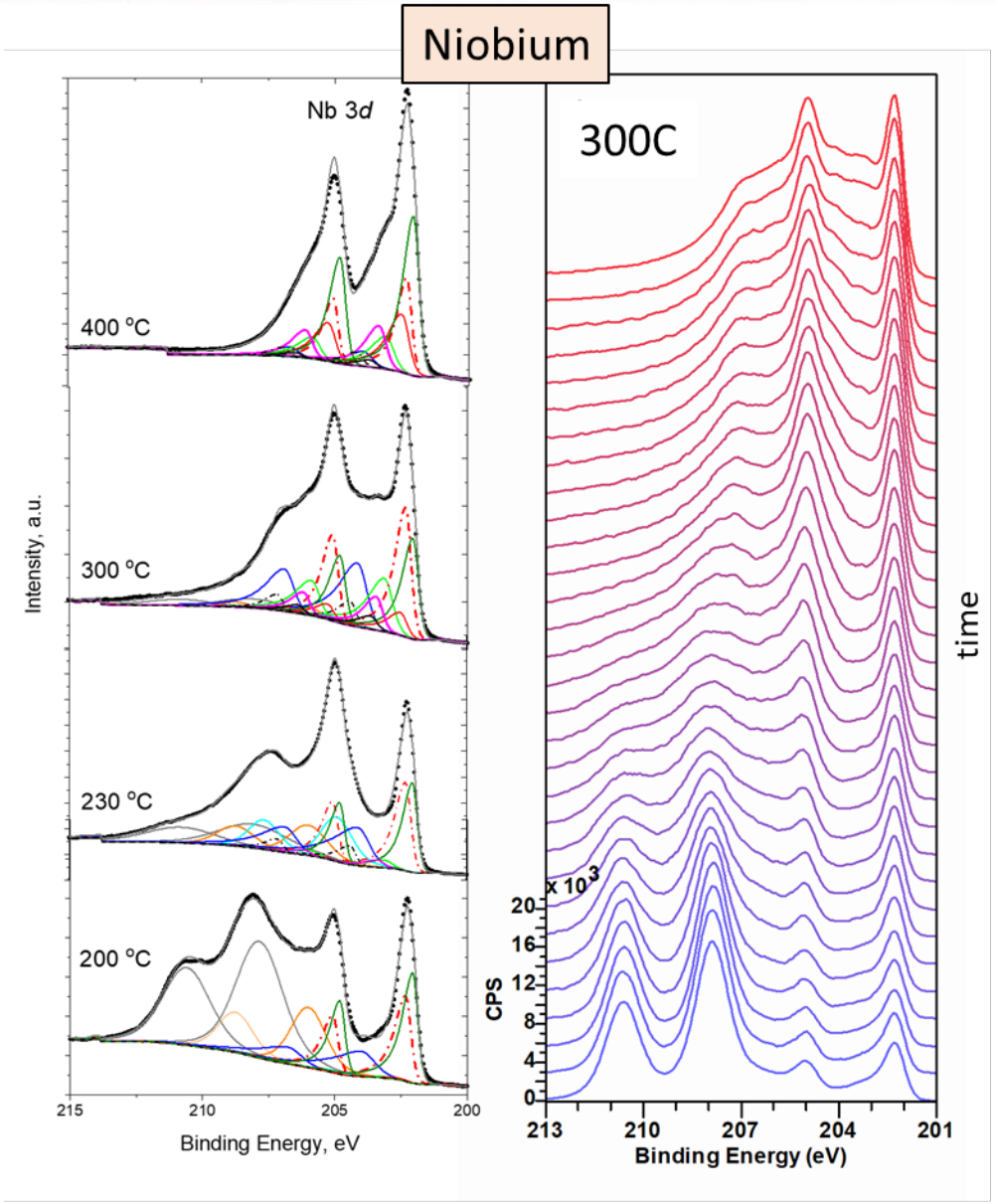
Fluorine

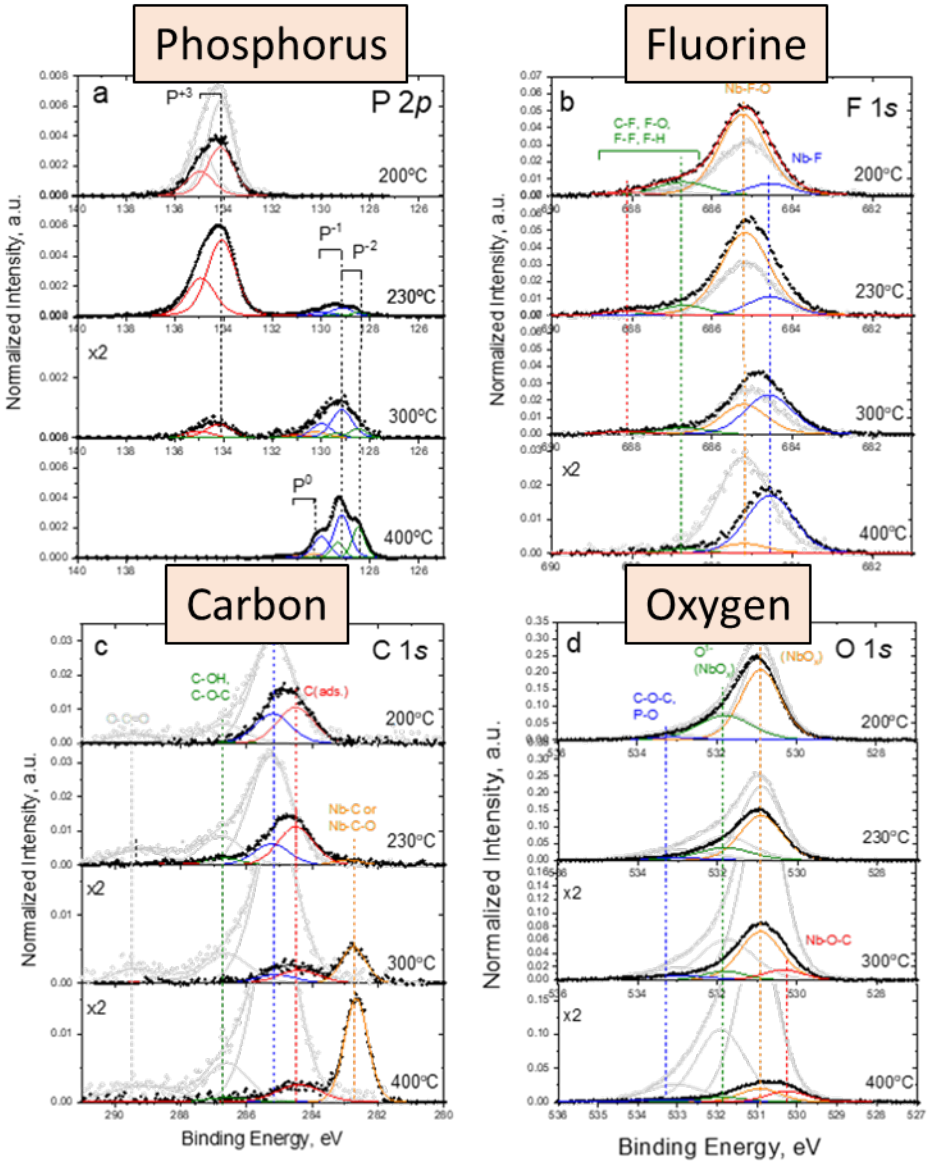




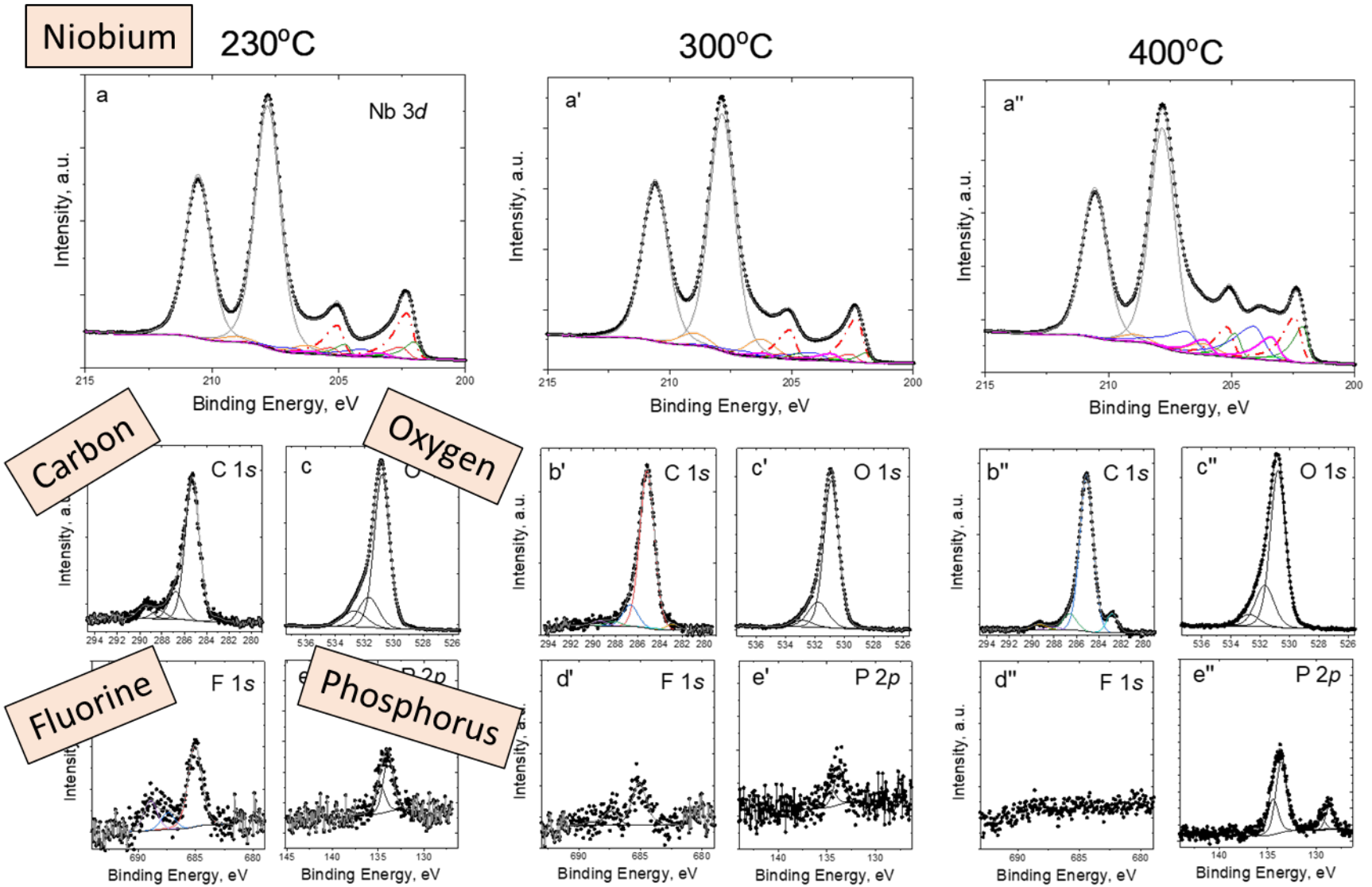
Niobium

Evolution of spectra: kinetics of reactions





Open the cavity: air oxidation

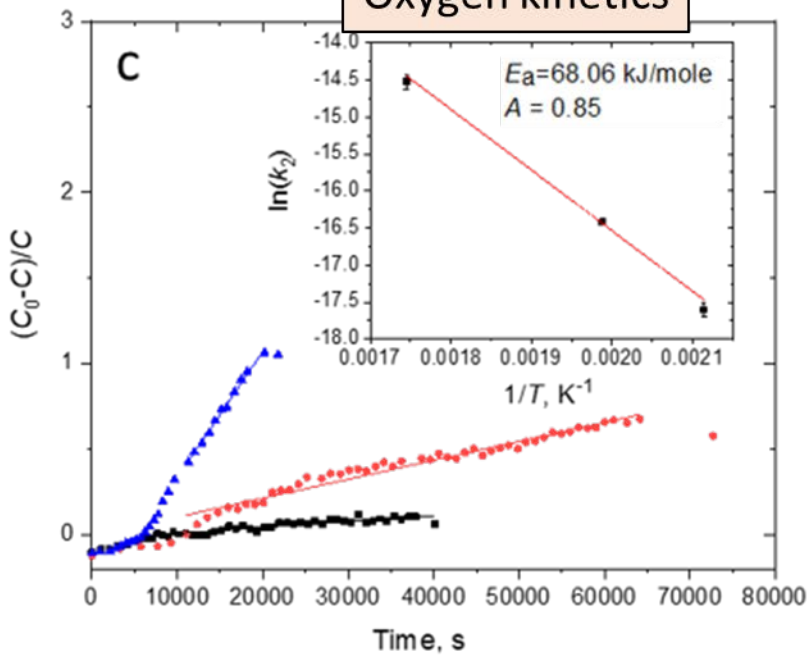


XPS Results

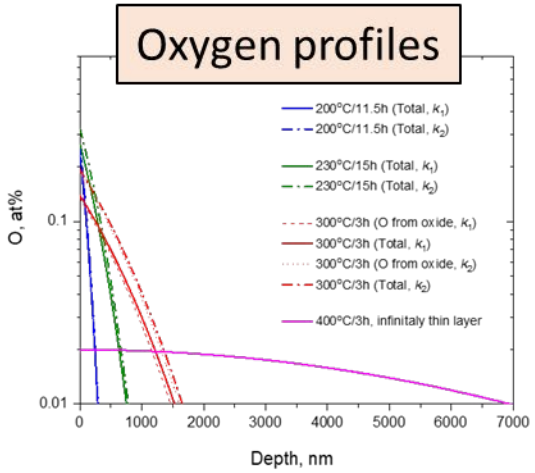
- Oxides dissolution kinetics
- Oxygen profiles
- **Huge role of fluorine** (masked by Nb2O5 !)
- Carbides formation kinetics

Carbides grow even in UHV at elevated temperatures.

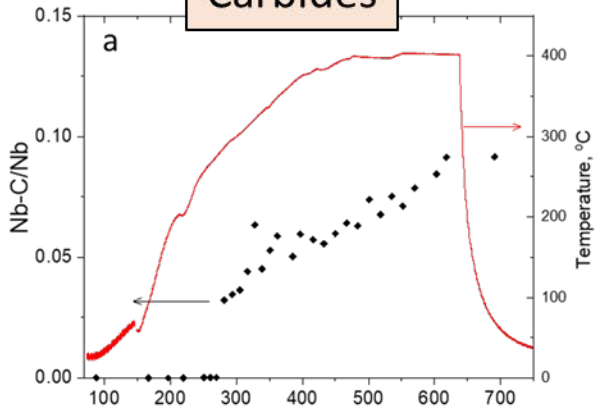
Oxygen kinetics



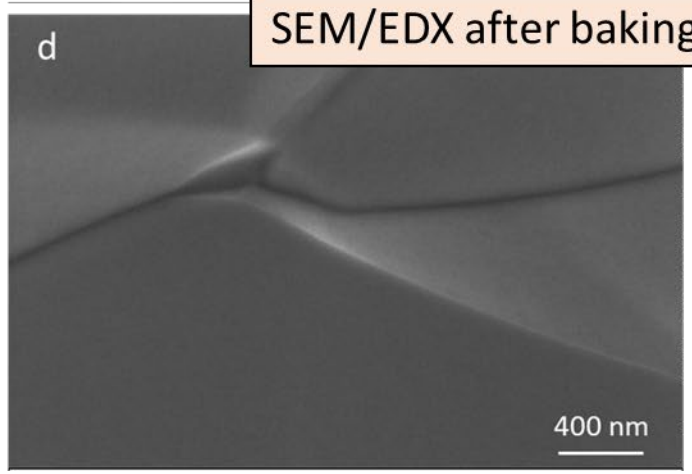
Oxygen profiles



Carbides

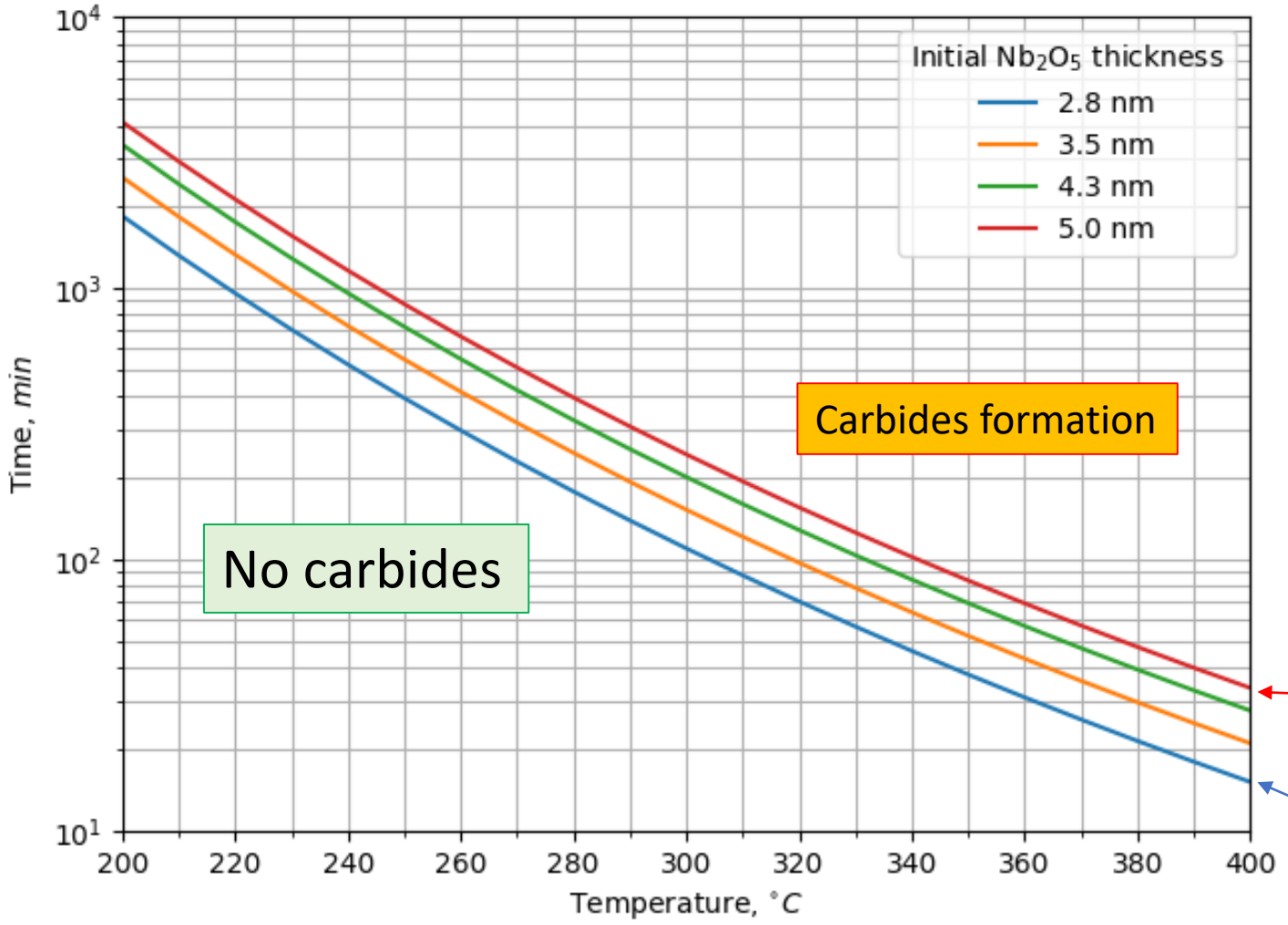


SEM/EDX after baking



Protective layer of Nb_2O_5

Knowing O and C kinetics we can calculate a phase diagram



Protective layer of Nb_2O_5

- If pentoxide is thicker than **1 nm**, **carbides growth is suppressed**
- Based on kinetics of O and C we calculated the safe regions for baking even in non-UHV furnaces

"old" cavity or after intense HPR

fresh BCP, EP or HF rinsing

Is 300-350 °C required?

What we learned from the XPS data

(kinetics of oxygen dissolution, impurities and contaminations):

- Higher temperature -> faster reactions -> shorter time to achieve “the best” profile
- Higher temperature -> faster Nb carbides formation -> high R_s

Can the **controlled** oxide dissolution be realized on cavities?

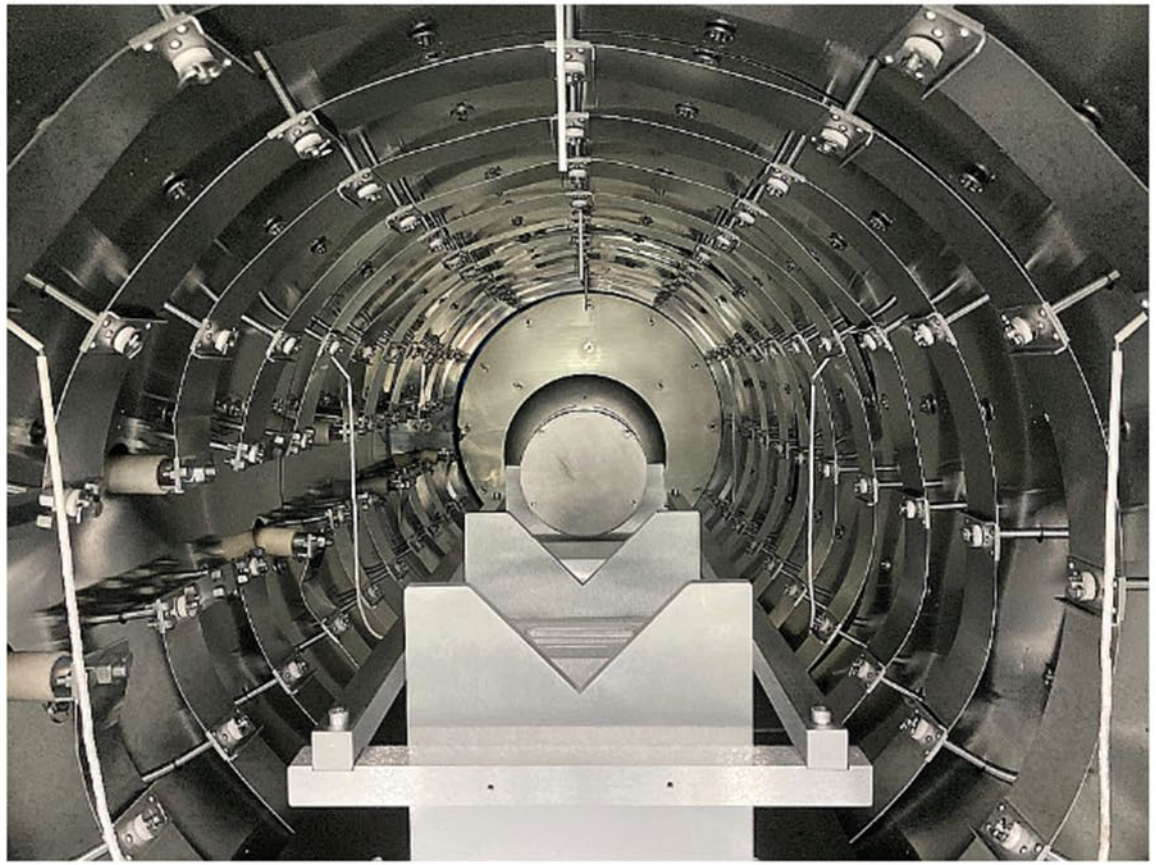
Mid-T baking at temperatures: 230 - 270 °C

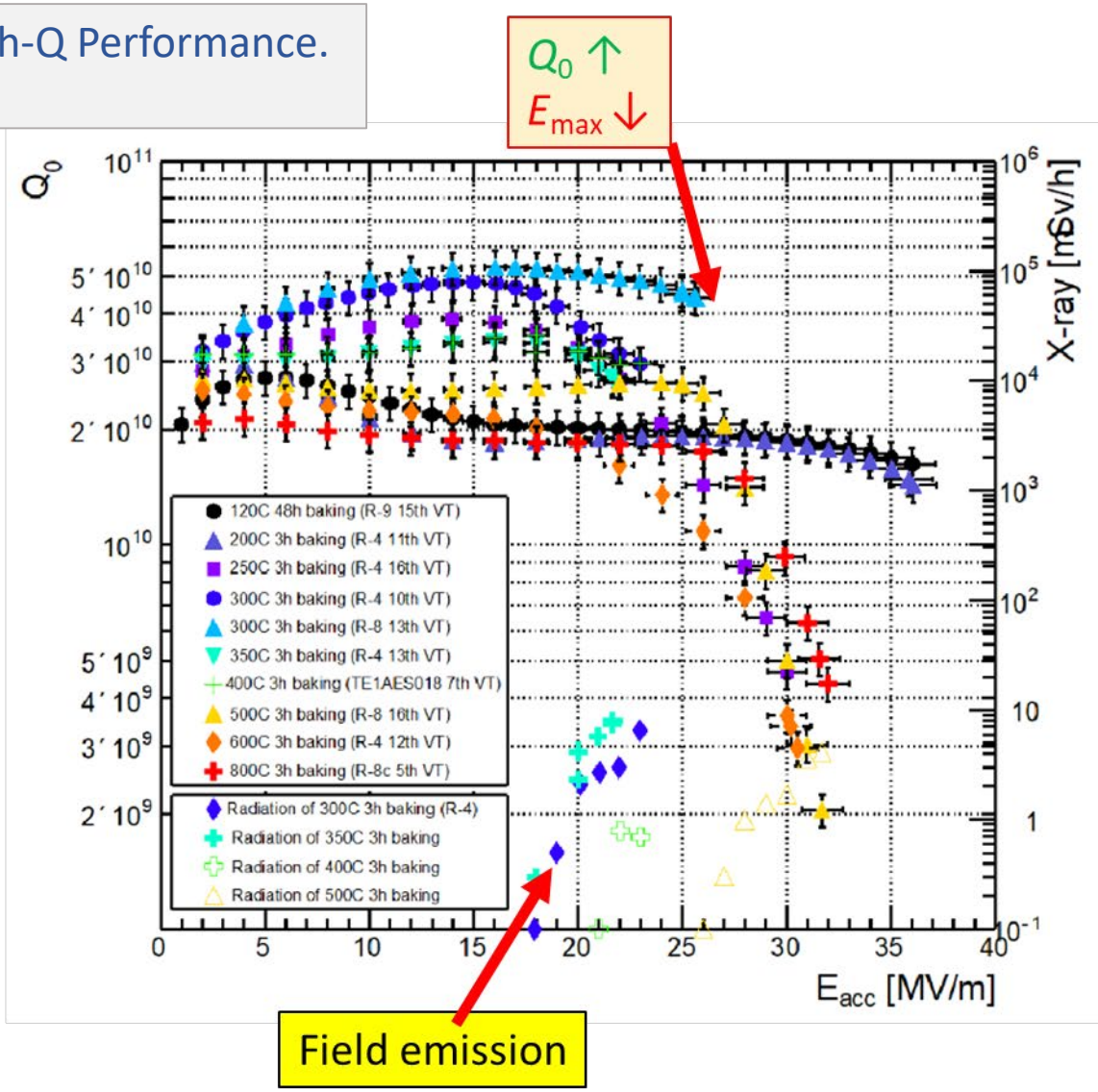
- reasonable baking duration (ca 12 h)
- **Low amount of carbides**

Let’s prove it with a cavity!

Mid-T in the furnace: risk of contamination

Systematic Investigation of Mid-T Furnace Baking for High-Q Performance.
Ito, H., Arakio, H., Takahashio, K. and Umemorio, K., 2021.

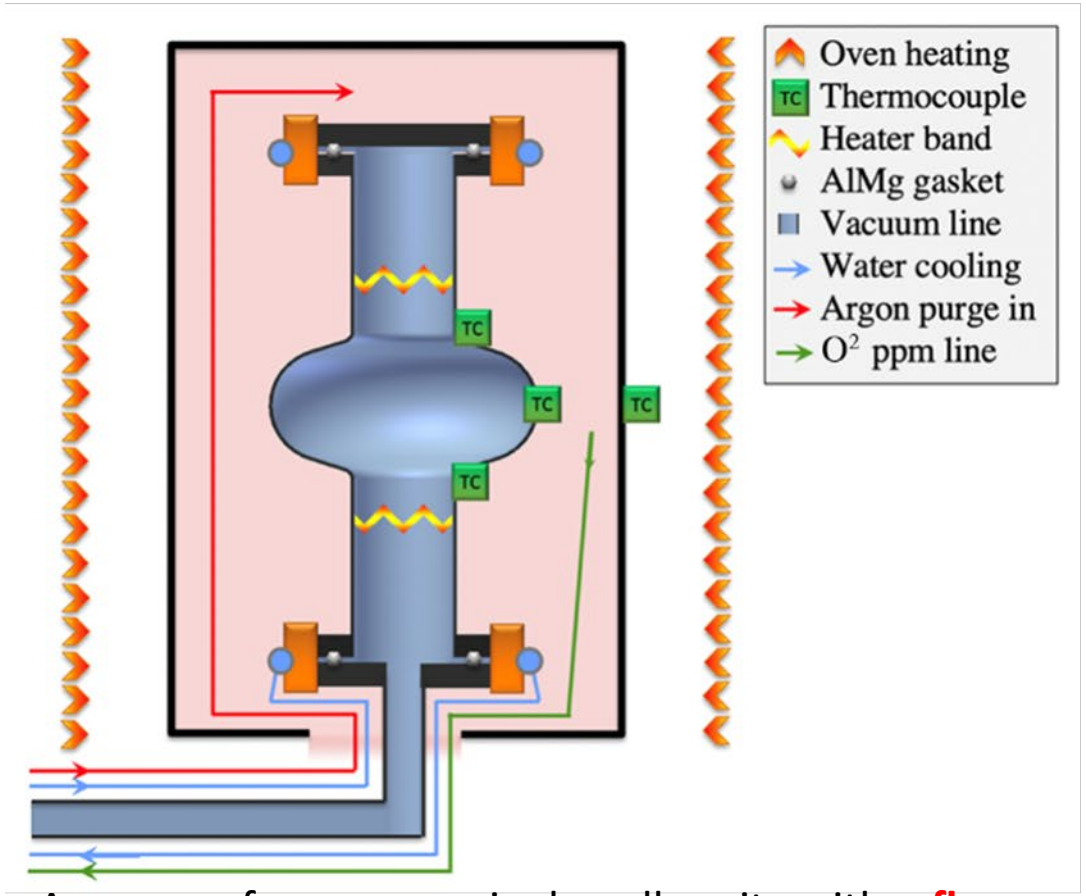


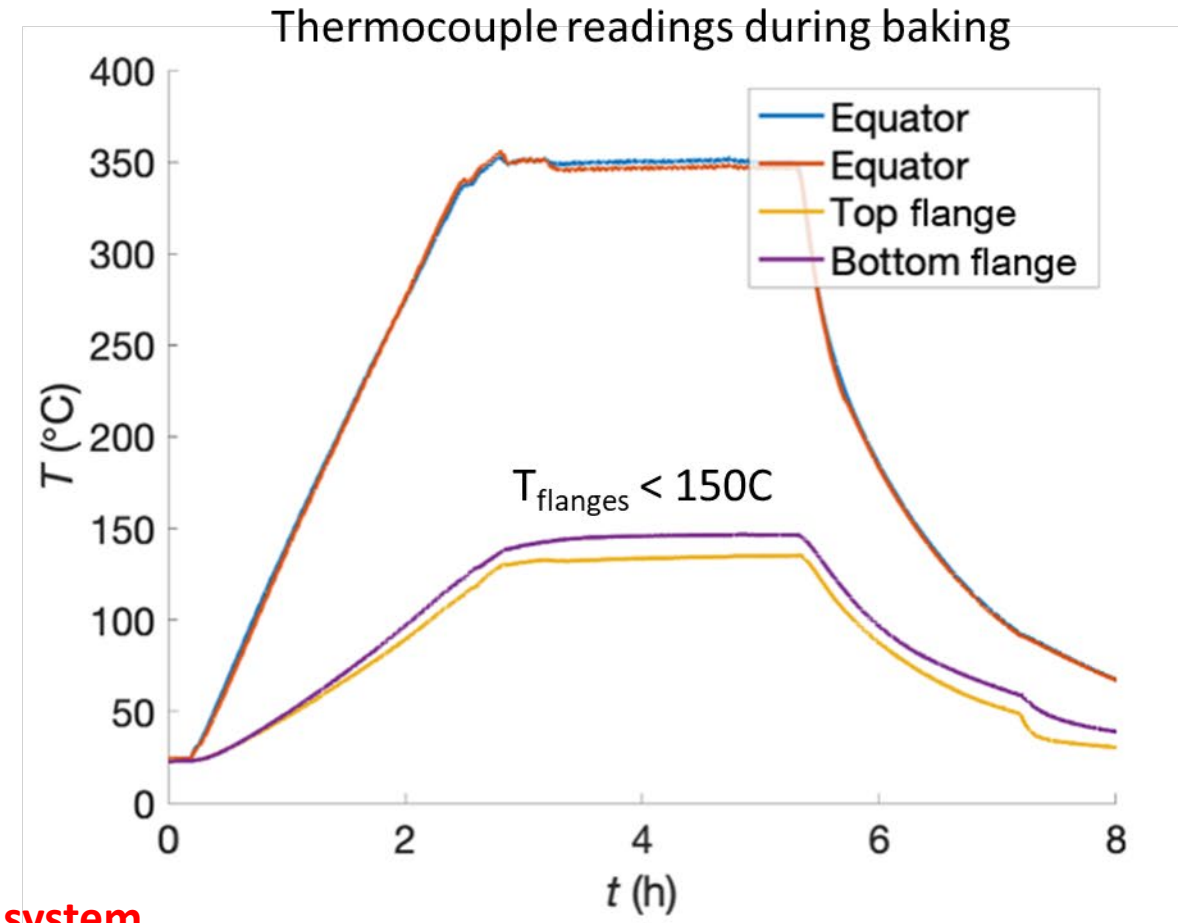


Vacuum furnace and single-cell cavity with Nb caps
(to protect the cavity from contamination)

Mid-T in the furnace: double volume

Ultralow surface resistance via vacuum heat treatment of superconducting radio-frequency cavities.
Posen, S., Romanenko, A., Grassellino, A., Melnychuk, O.S. and Sergatskov, D.A., 2020





- A vacuum furnace + a single-cell cavity with **a flange-cooling system**
- rf test can be performed, but one need to break vacuum for installation into cryomodule => **post-oxidation, particles, etc.**

Other Options to Perform Baking?

Furnace

Functions

Vacuum vessel

Cavity heating

Advantages

Uniform heating

Disadvantages

Uniform heating (flanges also!)

Requires intermediate disassembly

Poor vacuum

Contamination

Extra mounting process

High power

High cost

And we do not have it (ca. 500 kEuro)

Any alternative?

We can use any vacuum chamber

We can use “local” heaters

We can use several heaters

We can heat only High B-field region

Cavity vacuum can be separated

No contamination if separate vacuum

Much less work required

Low power

Low cost

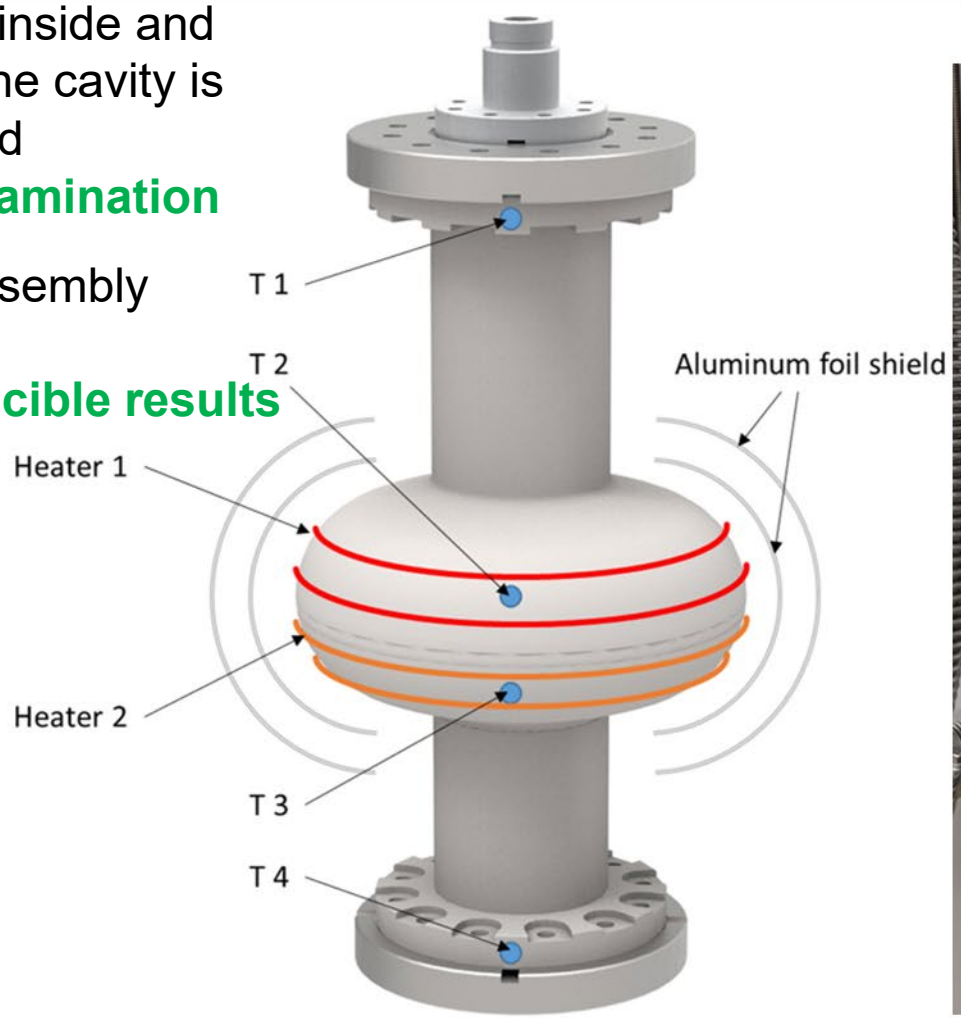
Baking in Cryostat!!!

- ✓ Vacuum inside and around the cavity is separated

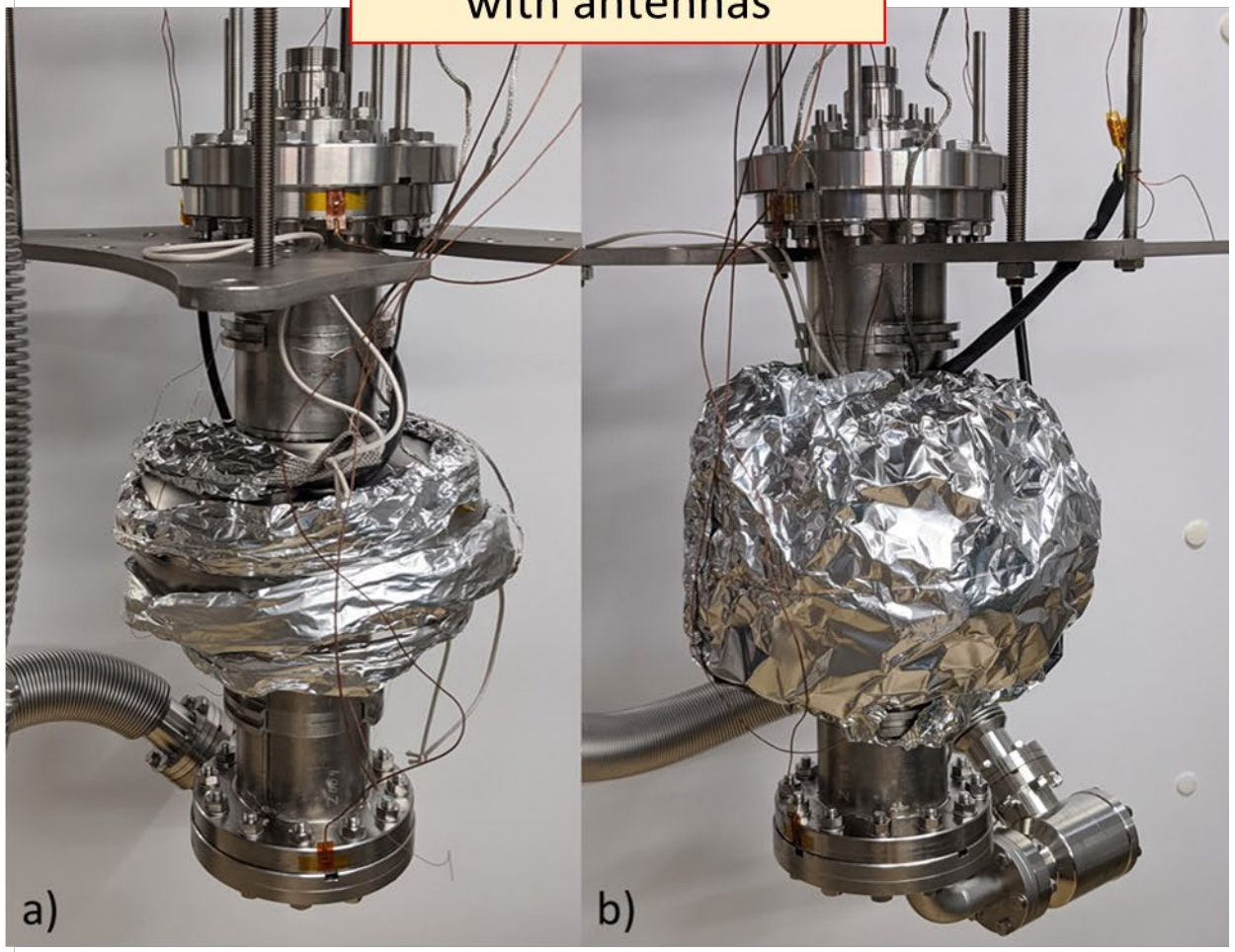
=> no contamination

- ✓ No disassembly required

=> reproducible results



1-cell 1.3GHz Nb cavity with antennas



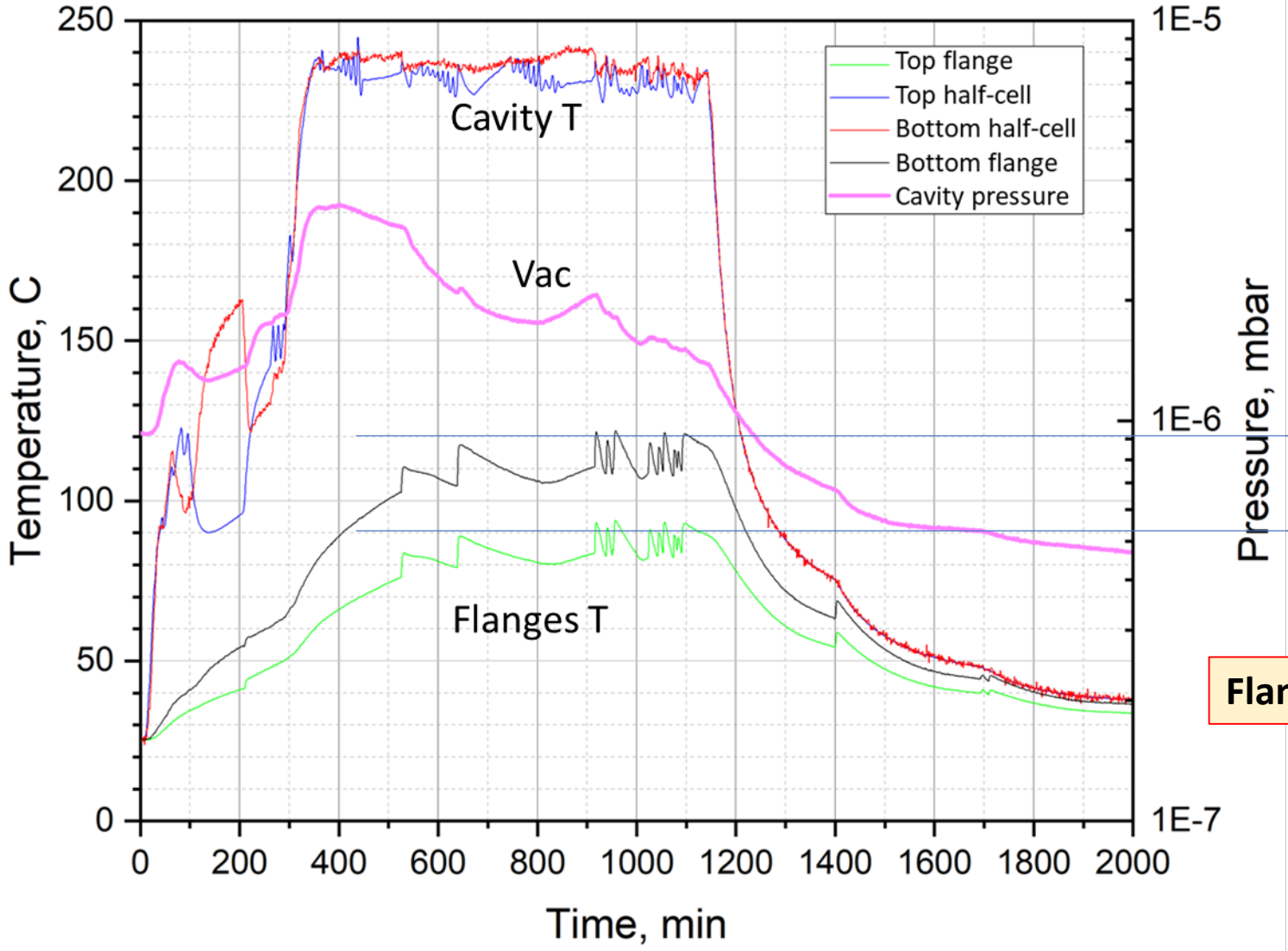
The set up can be used for cavity baking and rf cavity testing

✓ in a vertical test stand;

✓ **directly in a cryomodule!**

see Y. Tamashevich et al, arXiv:2307.09094

1st heat treatment: 13 hours



Goal: keep 1nm Nb_2O_5

230°C baking during 13h

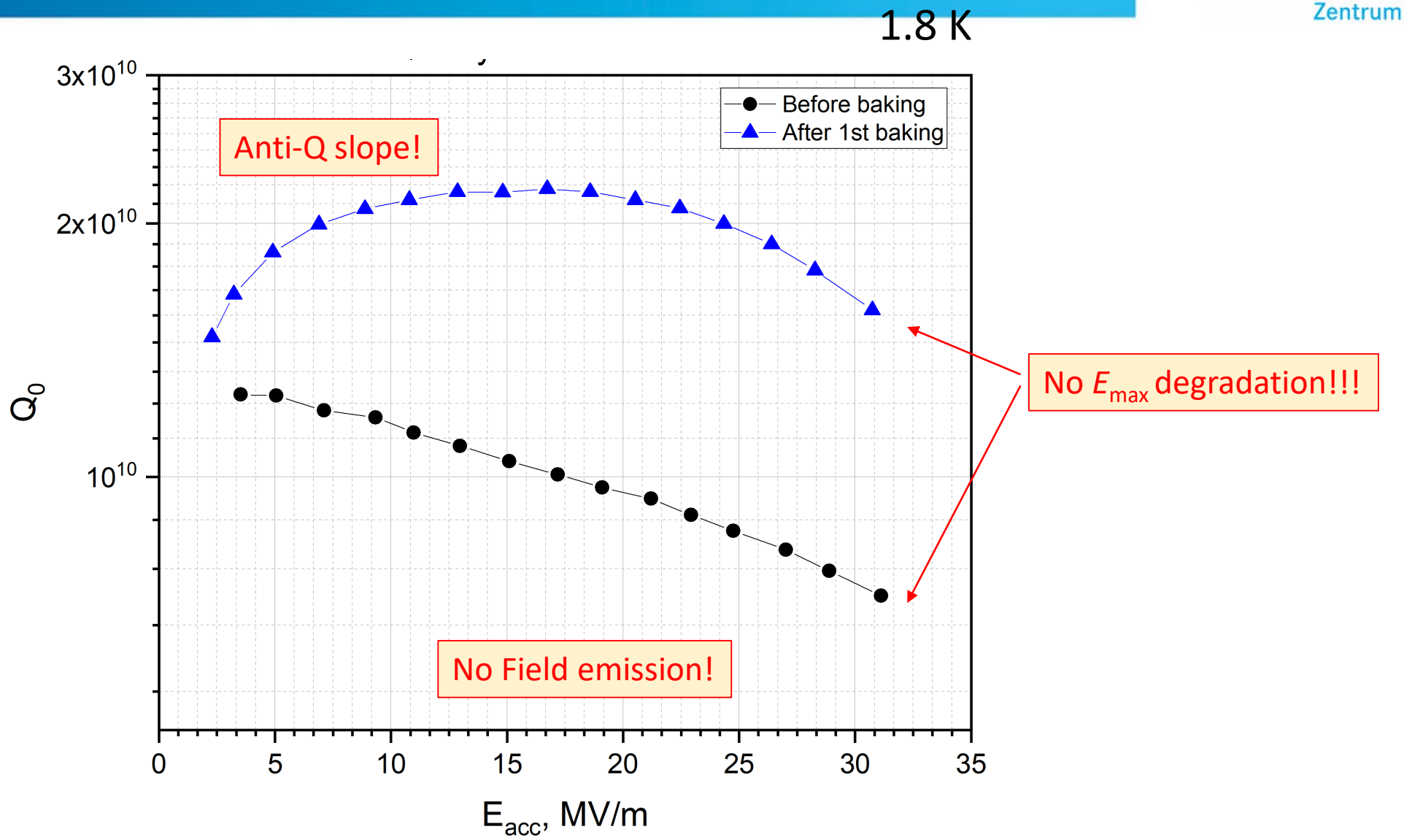
Flanges and gaskets

Below 120C

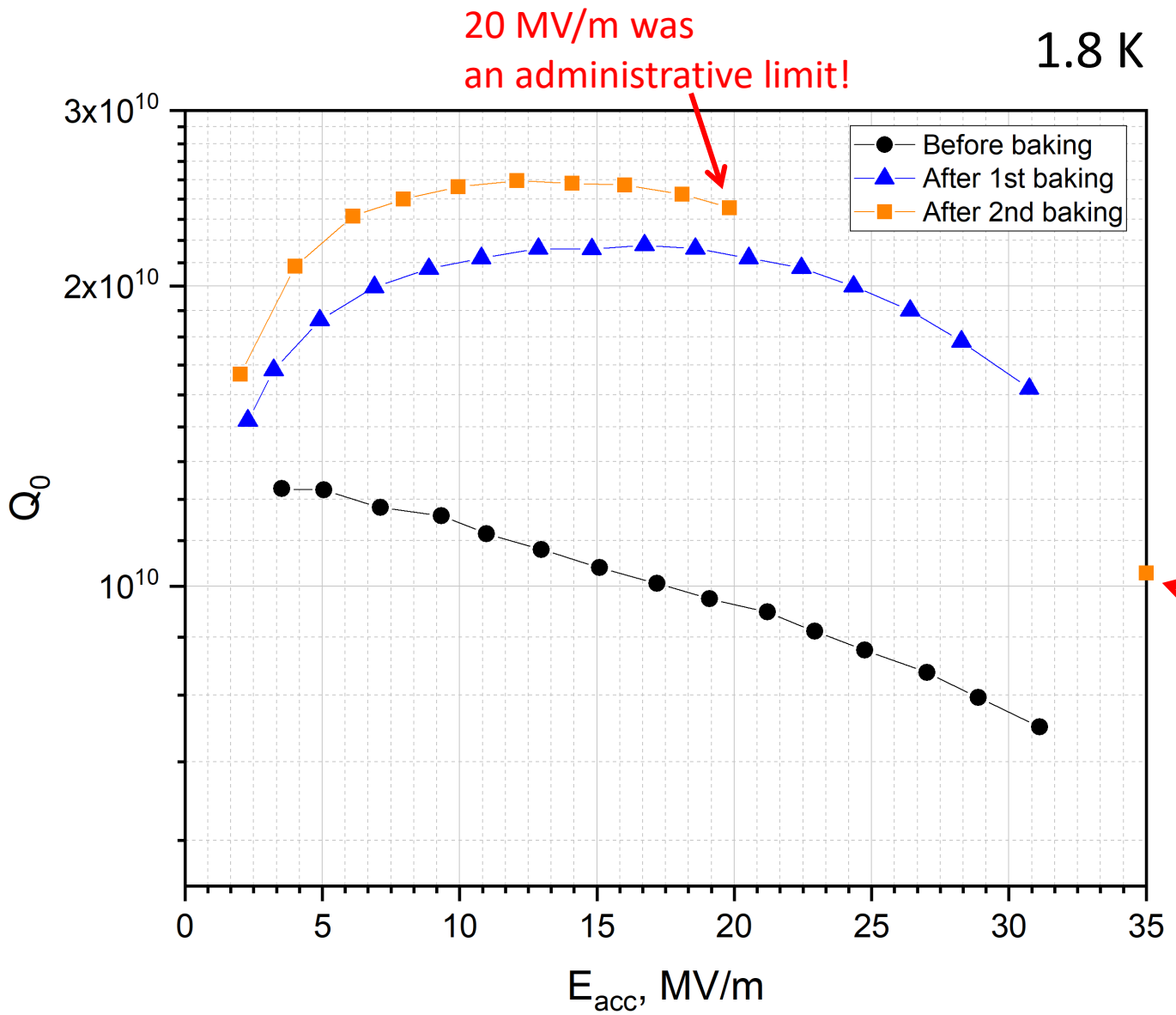
Below 90C

Flanges below 120°C without cooling!!!

Cold test results at 1.8K



2nd baking (+11h): even better



Continue

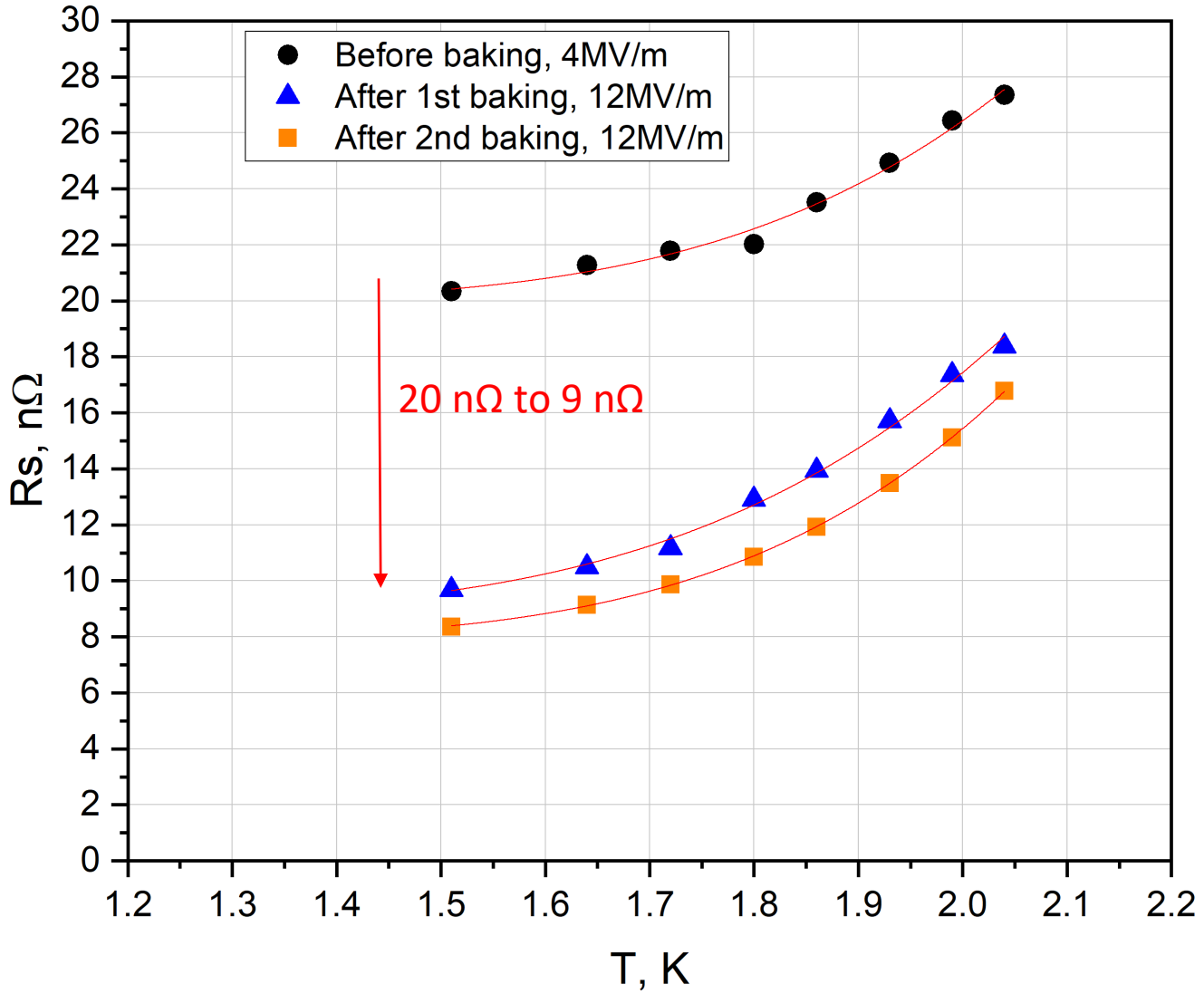
230°C baking during 11h more

No E_{max} degradation!!!

E_{max} check

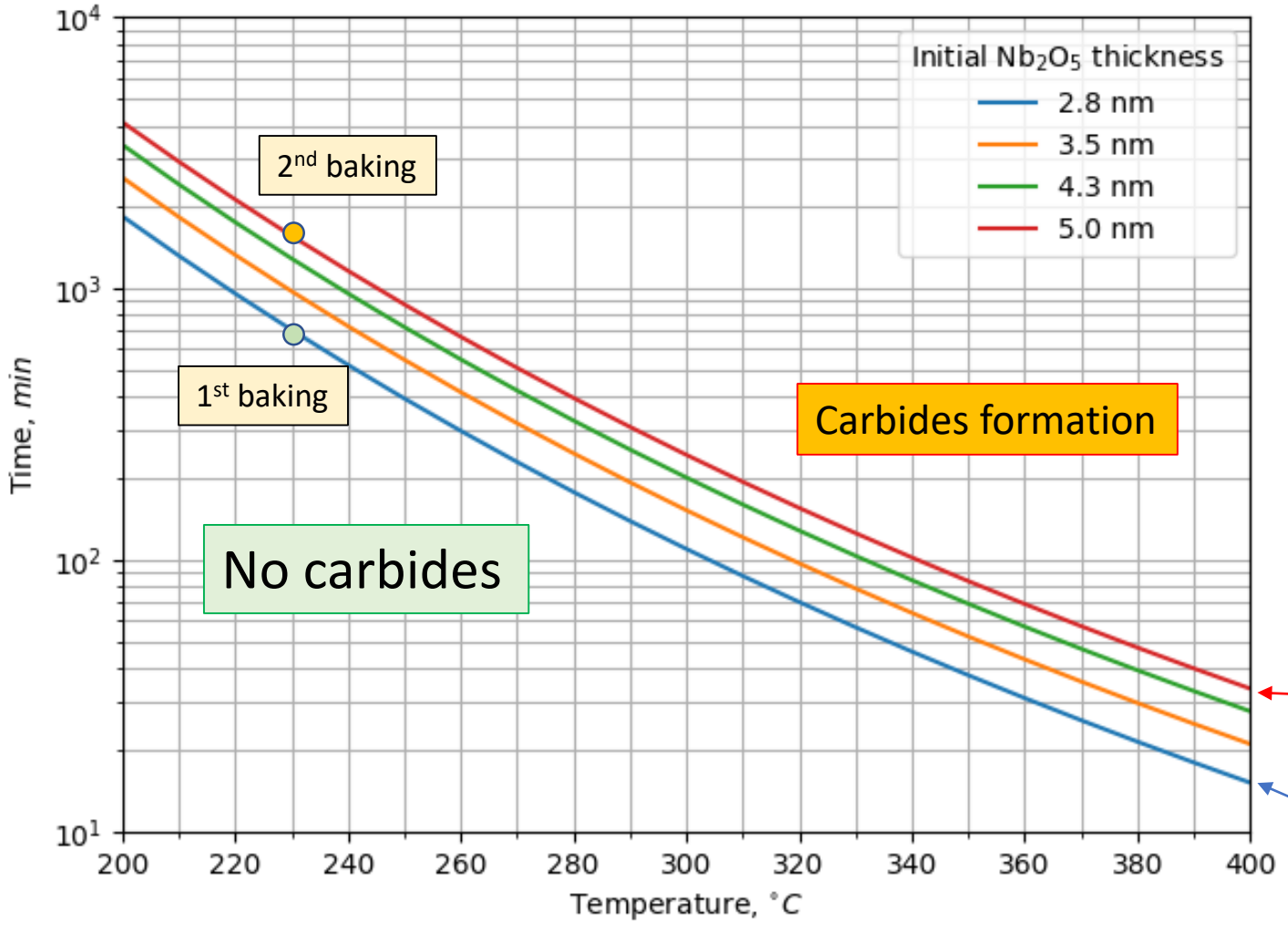
HZB PC network down.
No allowance from safety officers for systematic tests >20MV/m
But we did E_{max} check

Surface Resistance, R_s



Protective layer of Nb_2O_5

You can do clean baking in the furnace too!



- Carbides will start formation after crossing the 1nm line
- At low temperatures they will form slowly
- Above 300C carbides form very fast

"old" cavity or after intense HPR

fresh BCP, EP or HF rinsing

It works! No furnace needed!

- ✓ successful Mid-T baking without oven demonstrated
- ✓ reliable and inexpensive procedure for cavity baking established.
- ✓ This procedure can be used for the systematic study of heat-treatment effects in cavities:
  - cavity vacuum is always isolated.
- ✓ Low-power local heating allows all RF cables stay connected ➡

➡ **Mid-T baking can be done directly in the cryomodule!**

(Patent pending!)

- ✓ Potentially the method can be used to treat the existing infrastructure

Paper pre-print: A. Prudnikava et al, arXiv:
Paper pre-print: Y. Tamashevich et al, arXiv:2307.09094

Thank you!

use this plot in your experiments