Electropolishing study on nitrogen-doped niobium surface

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- Effect of temperature
- Effect of voltage
- EP process to improve the surface
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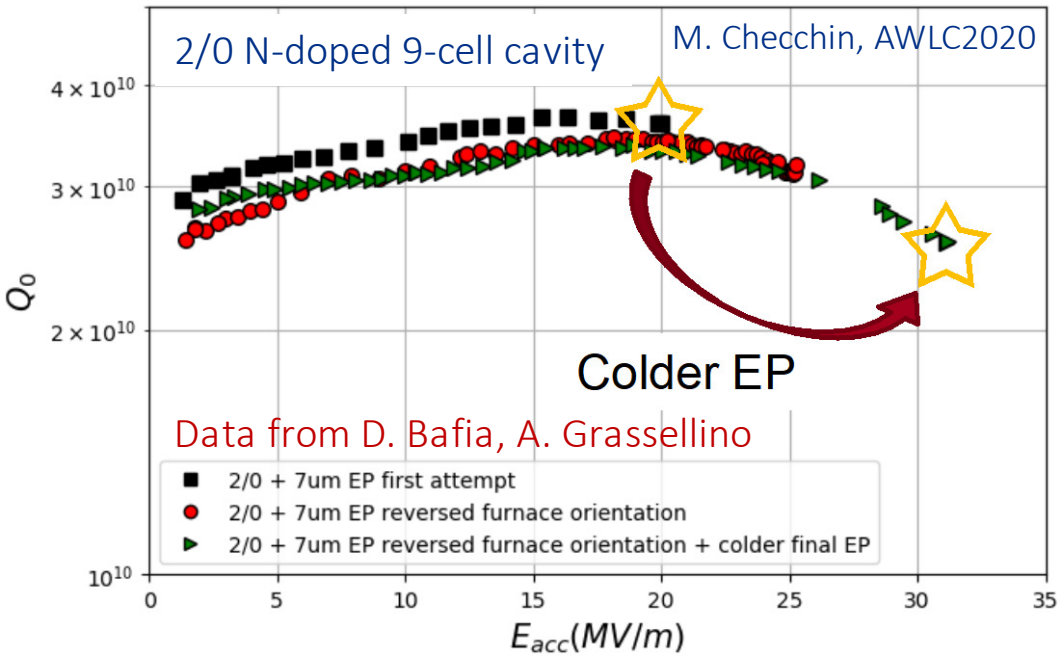
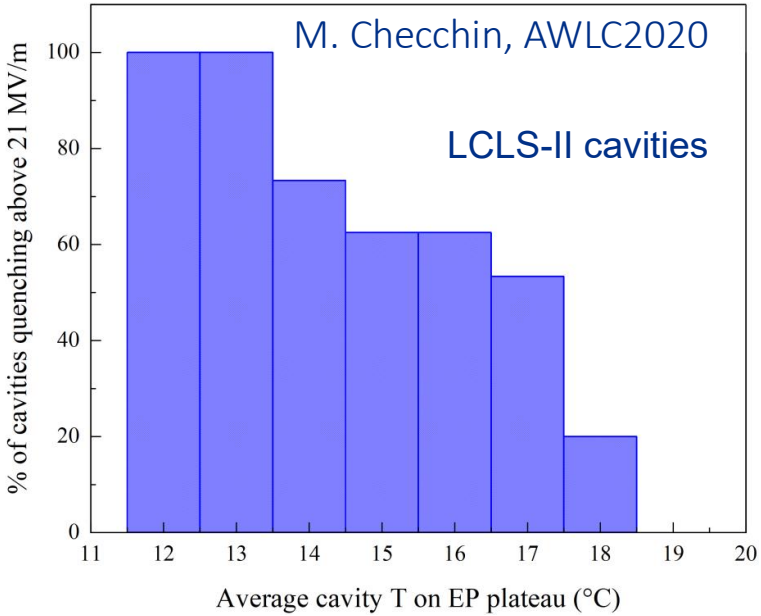
Motivation of study

R&D results at Fermilab





- The low quench field at high temperature was explained by pit formation on the surface. *J. K. Spradlin et al., MOP030, SRF2019*

Motivation:

- Understand the effect of EP on the N-doped surface.

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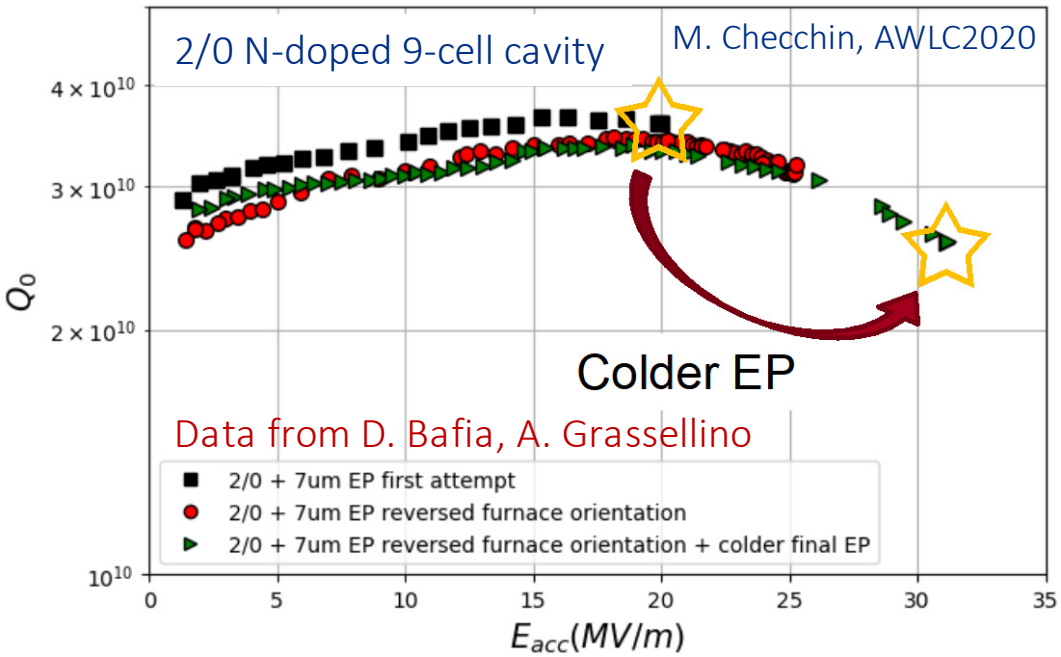
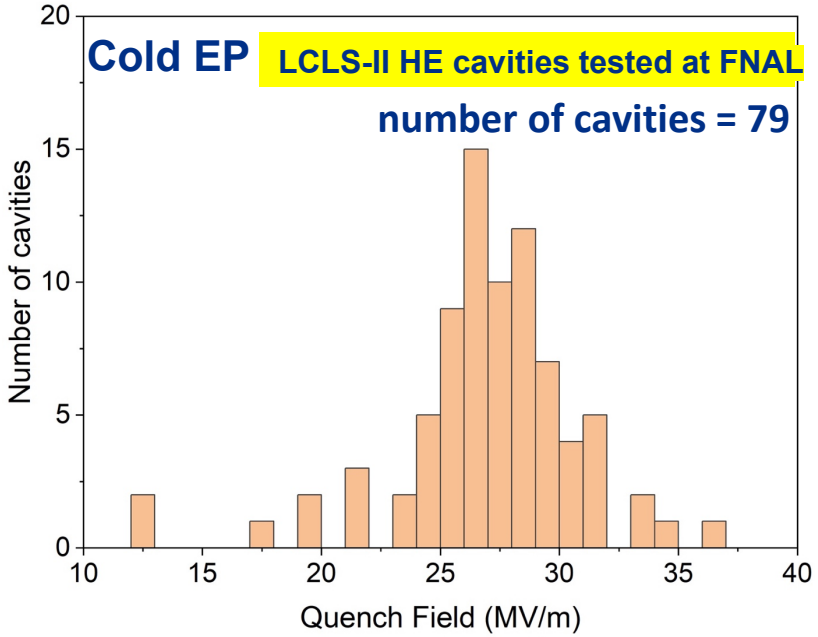
Motivation of study

R&D results at Fermilab





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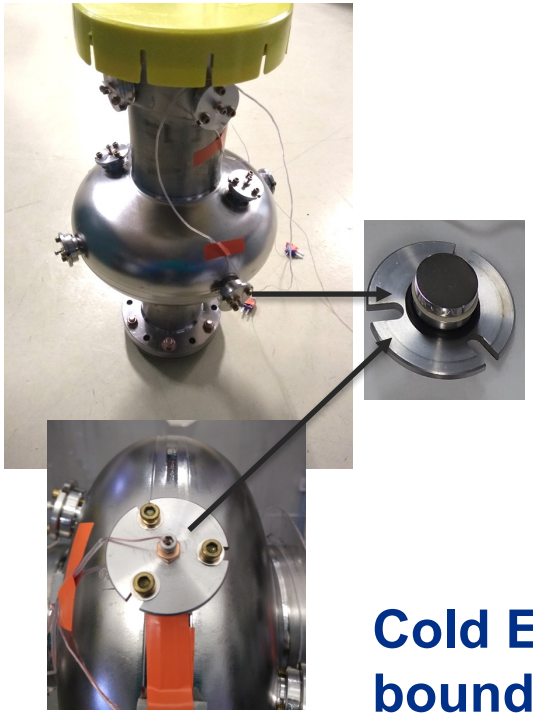
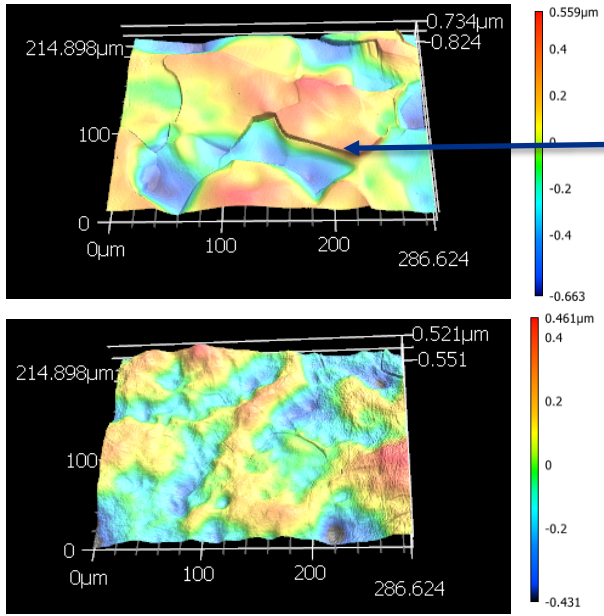
Undoped cavity surface at different EP temperatures

Equator Coupon Surface

Coupon cavity





Sharp steps at the grain boundaries

Cold EP makes the surface smooth with no sharp grain boundaries.

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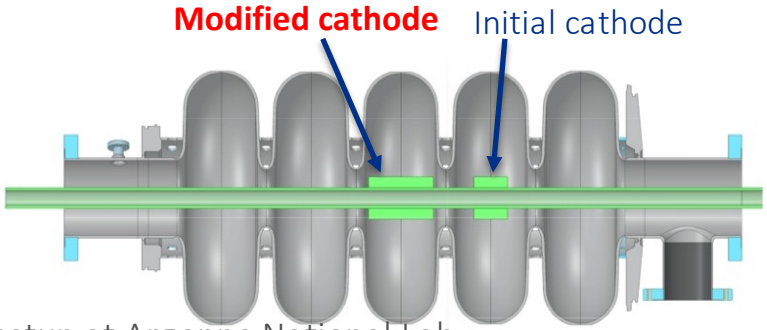
Electropolishing of 650 MHz PIP-II cavities



EP setup at Argonne National Lab

Parameters	Initial EP	Modified EP	
	Warm/cold EP	*Warm EP*	*Cold EP*
Voltage	**18 V**	**~25 V**	**22 V**
Cavity temperature	22 °C/12 °C	22 °C	12 °C
Cathode type	**Cathode-I (initial)**	**Cathode-M (modified)**	

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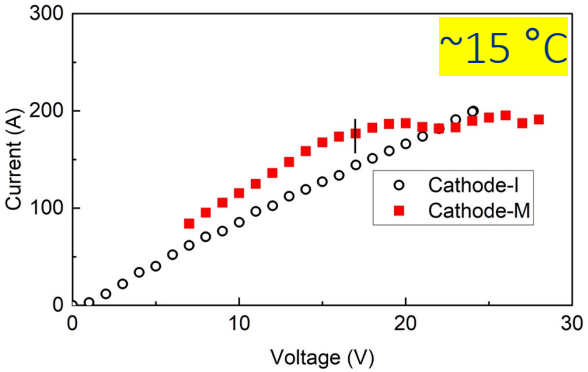
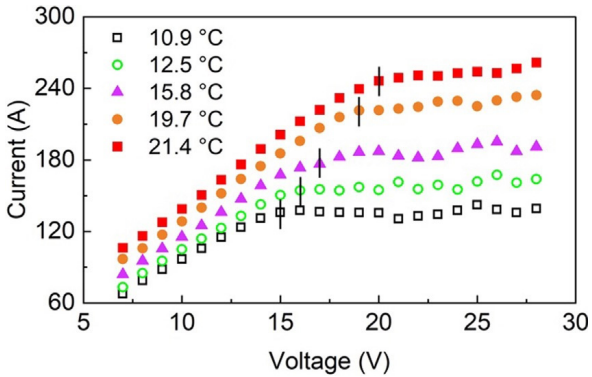
V. Chouhan et al., **TUPTB041, TUPTB042** SRF2023.

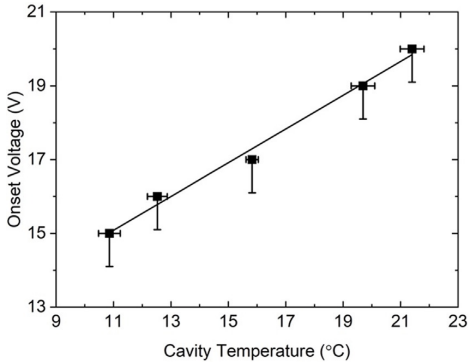
Comparison with initial cathode



I-V curves with modified cathode



Onset voltage vs cavity temperature



EP should be performed in the current plateau region.

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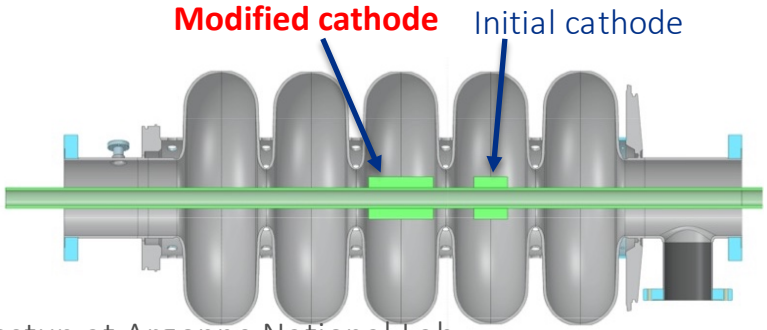

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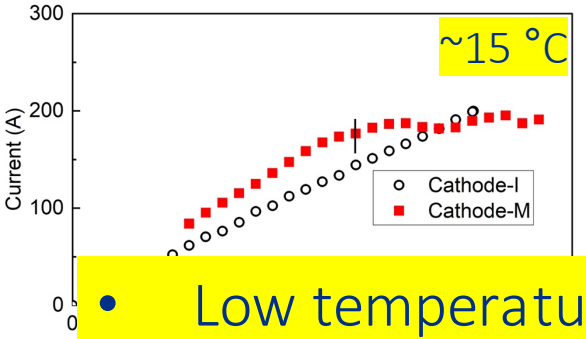

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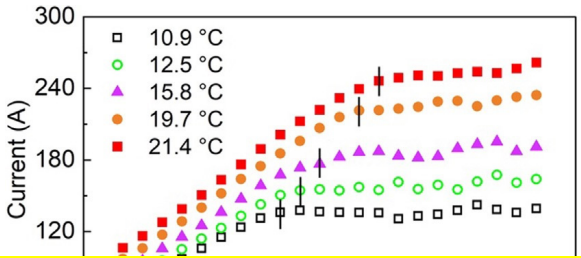

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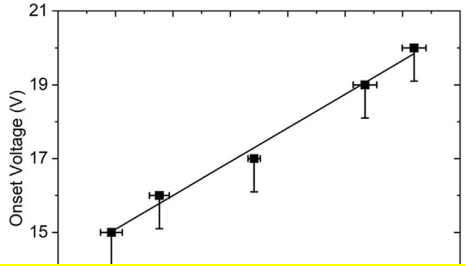

- Low temperature maintains EP plateau at a lower voltage.
- 650 MHz cavities require voltage higher than standard 18 V.

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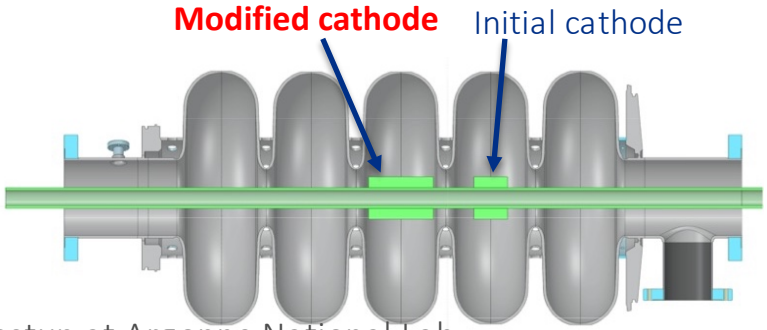
Electropolishing of 650 MHz PIP-II cavities



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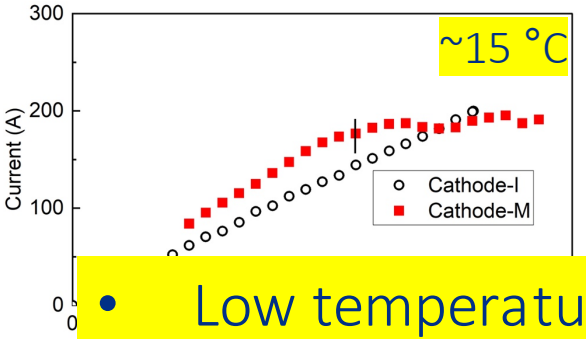

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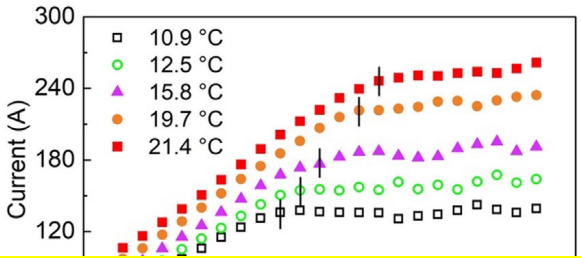

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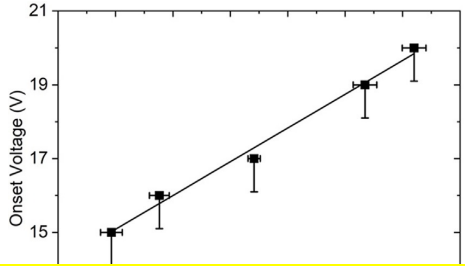

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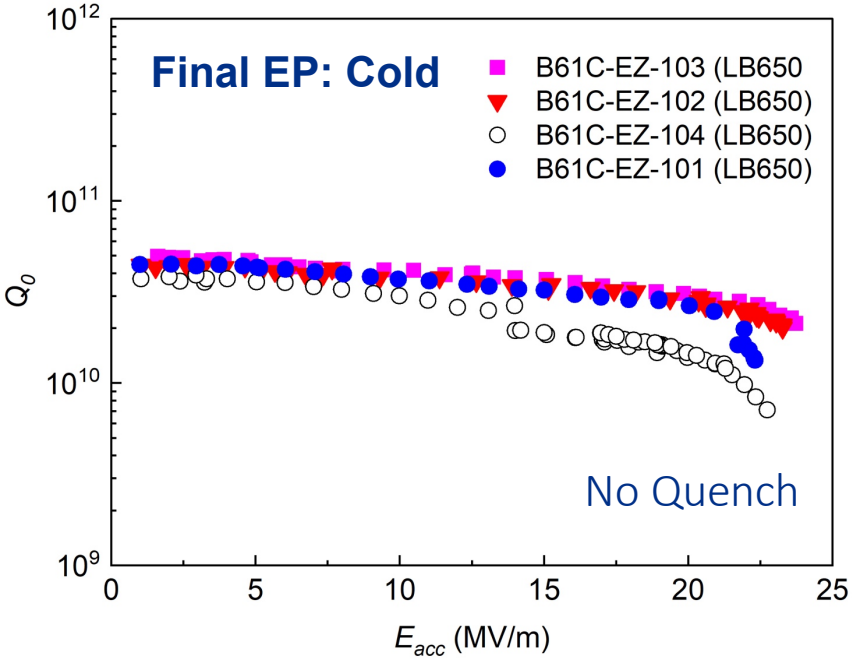
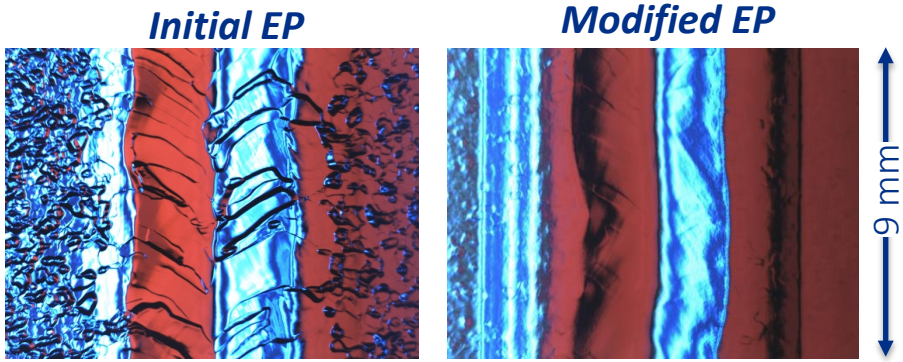
Performance in baseline tests (LB650 cavities)

LB650 PIP-II cavities after modified EP



Cavities qualified in baseline tests.
Mid-T bake was applied (see poster **MOPMB030**, SRF2023)



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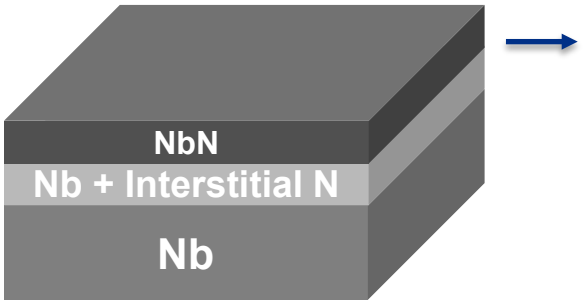
- Cavity B61C-EZ-103 after EP with initial conditions quenched at 15 MV/m.
- The cavities after EP with modified conditions significantly improved the E_{acc} in vertical tests.

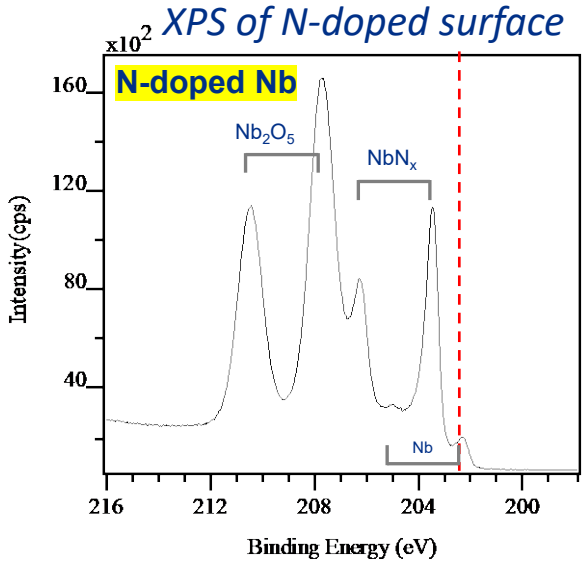
See **TUPTB042** for details on EP of the high-beta 650 MHz cavities.

EP of N-doped surface

As N-doped



XPS of N-doped surface



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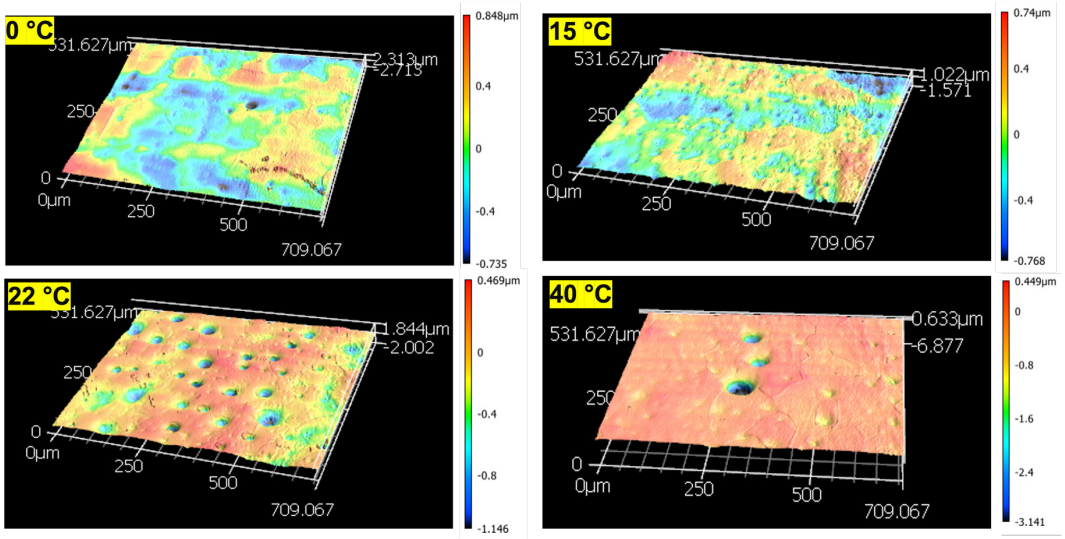
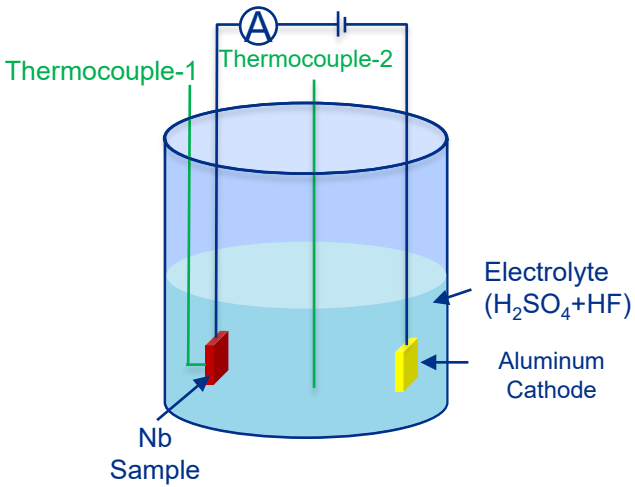
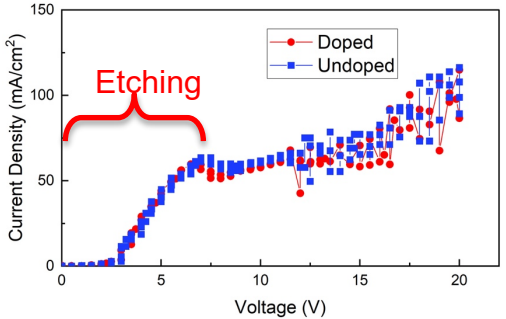
Effect of temperature in EP

- Nb surface: 2/0 nitrogen doping
- Post-doping EP was performed for ~5 µm:
  - at **Nb surface temperature** ranging from **0–40 °C**
  - **without acid agitation**
  - at **18 V**



Two-electrode system





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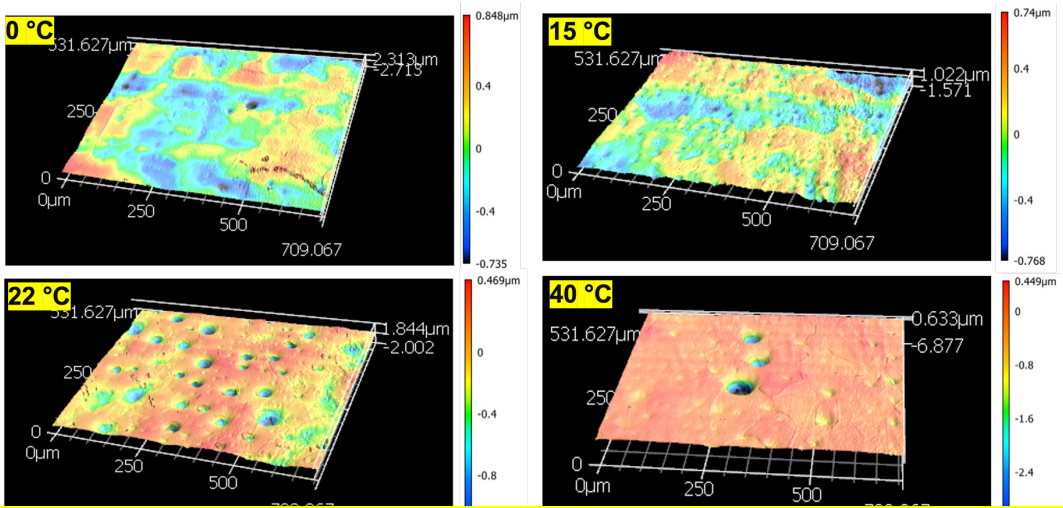

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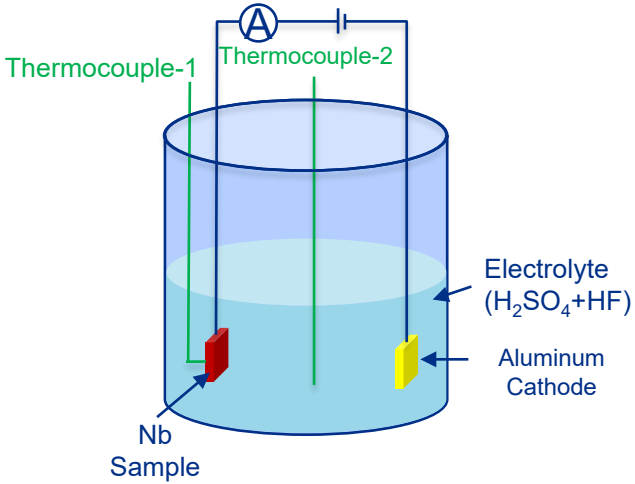

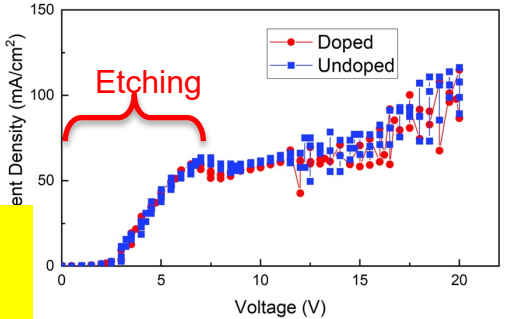

- Higher temperature produced more pits on the surface.
- No preferential etching of grains due nitrogen-doping.

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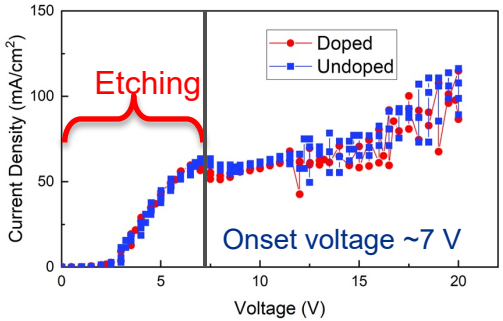
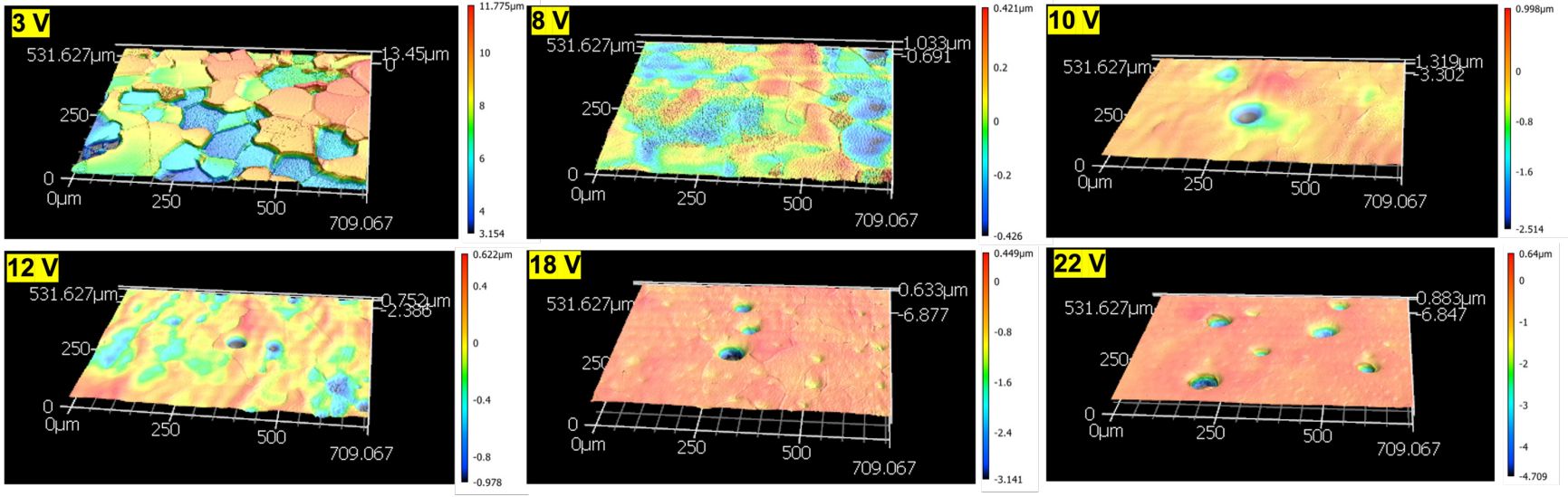
Effect of applied voltage

- Nb surface: 2/0 nitrogen doping
- EP was performed for ~5 µm removal:
  - at **Nb surface temperature** of **40 °C**
  - **without acid agitation**
  - at **voltage** ranging from **3-22 V**





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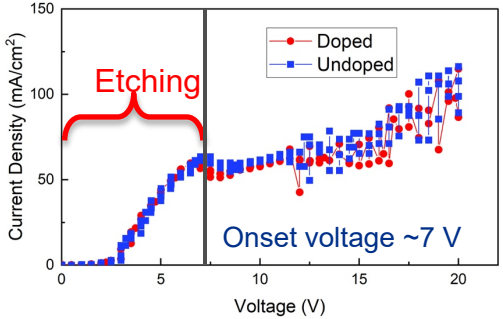

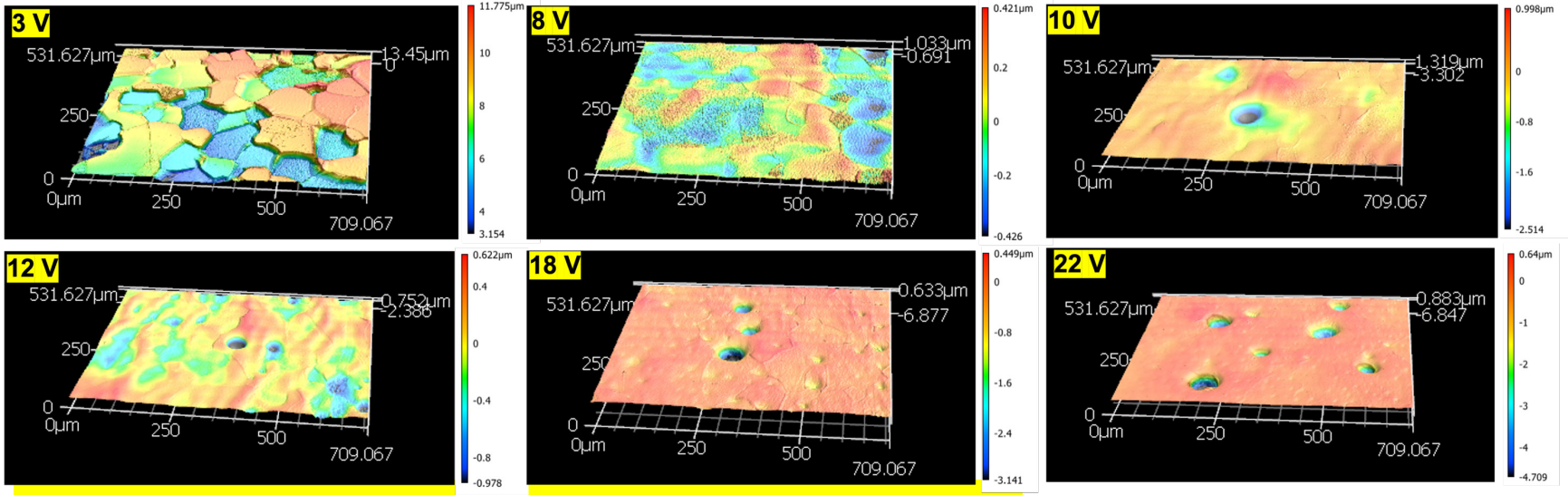

Higher voltage produced more pits on the surface.

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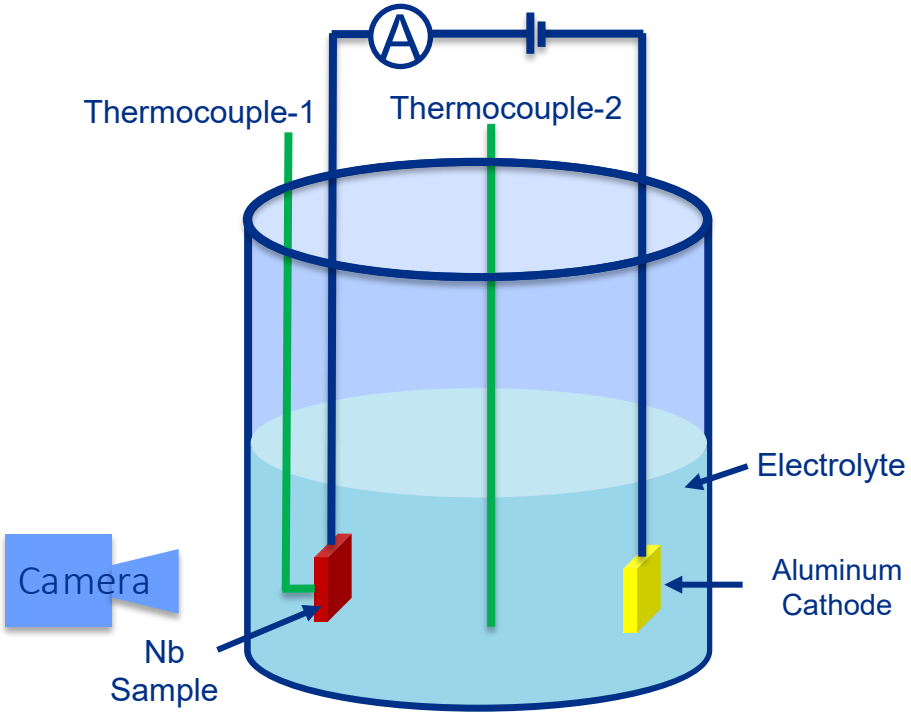
Pit formation: Gas evolution from N-doped surface



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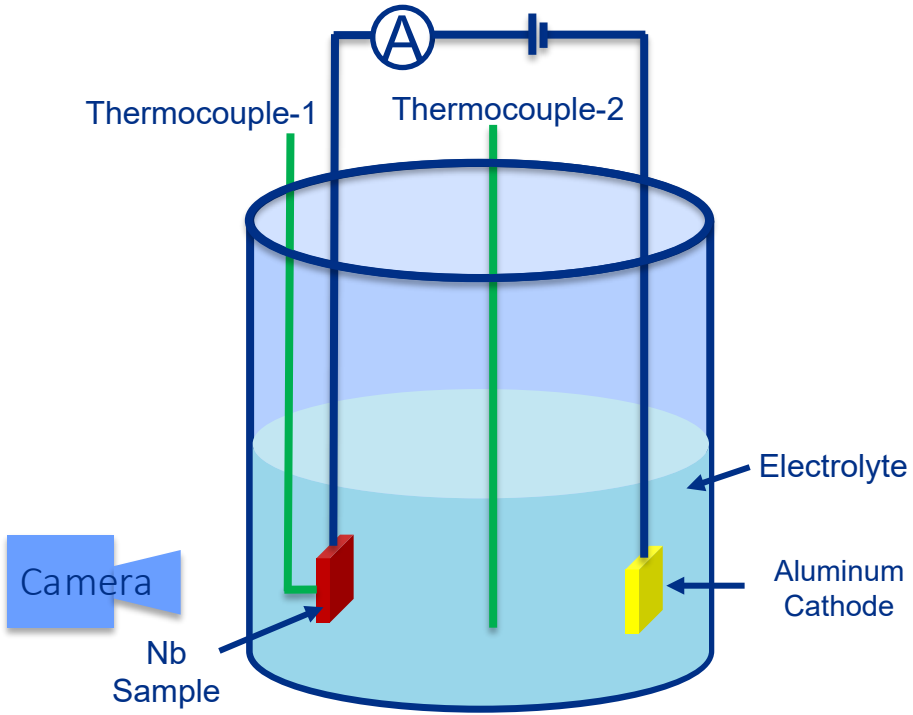
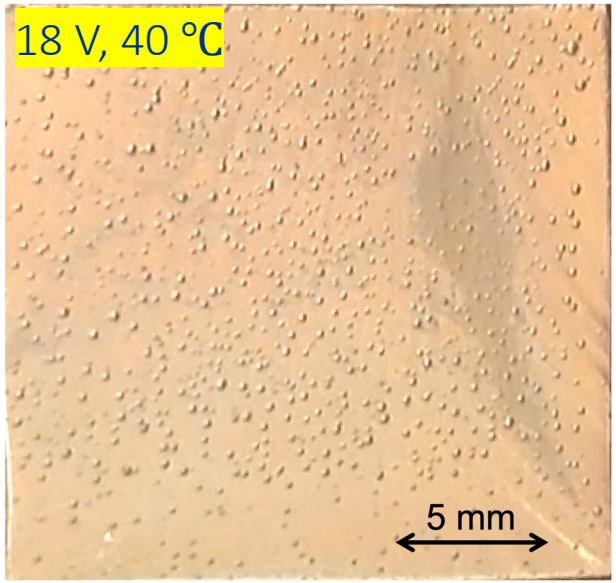


Bubble formation on N-doped surface during EP



There might be physical damage of the diffusion layer due to bubbles.

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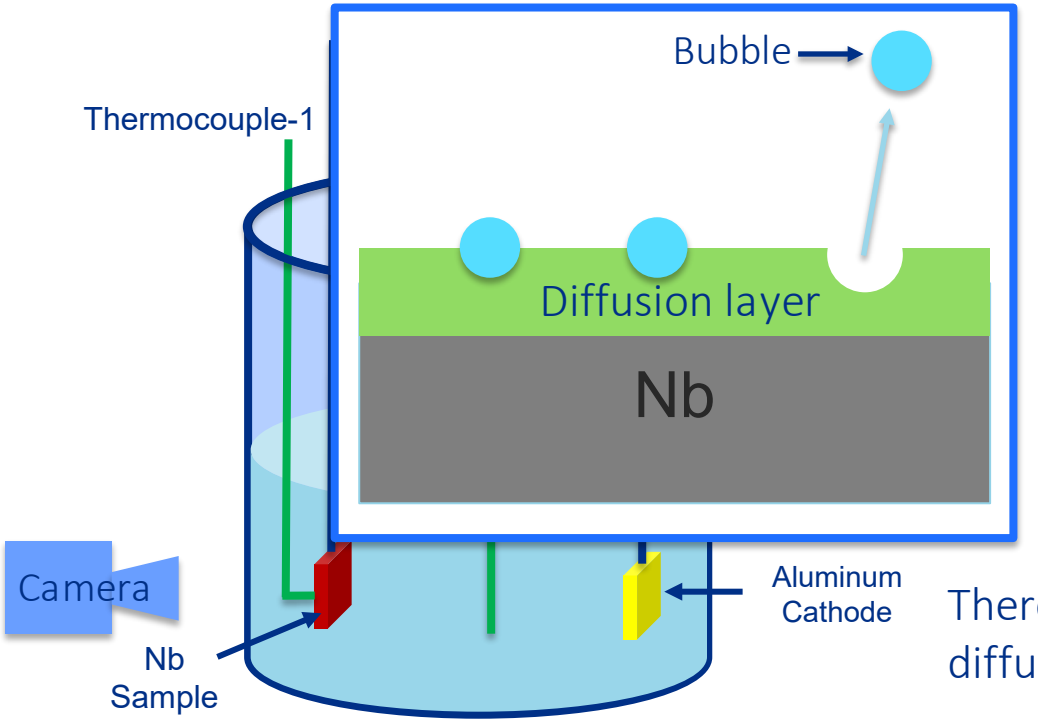

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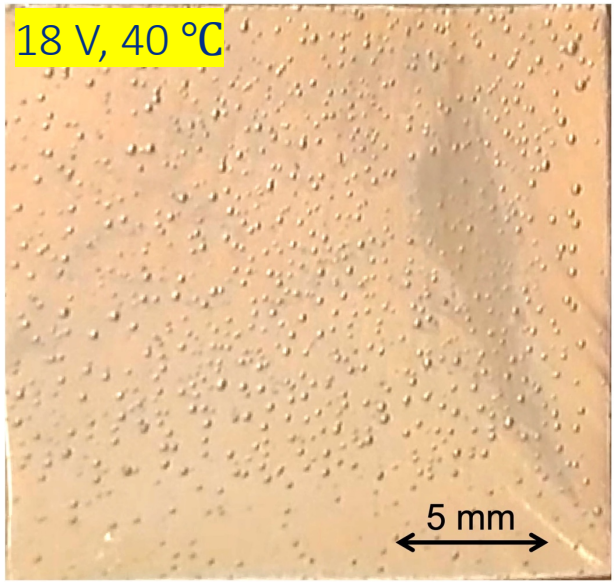

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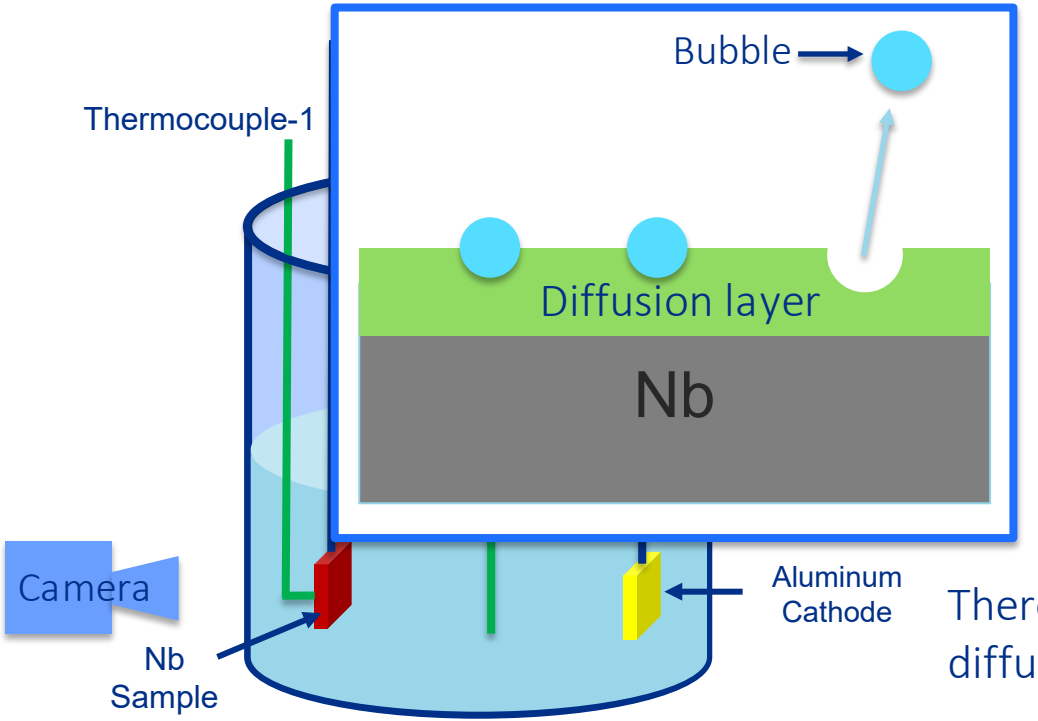

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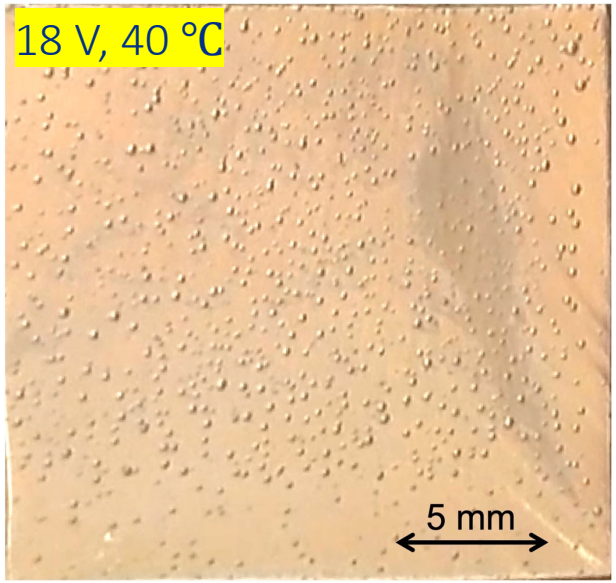
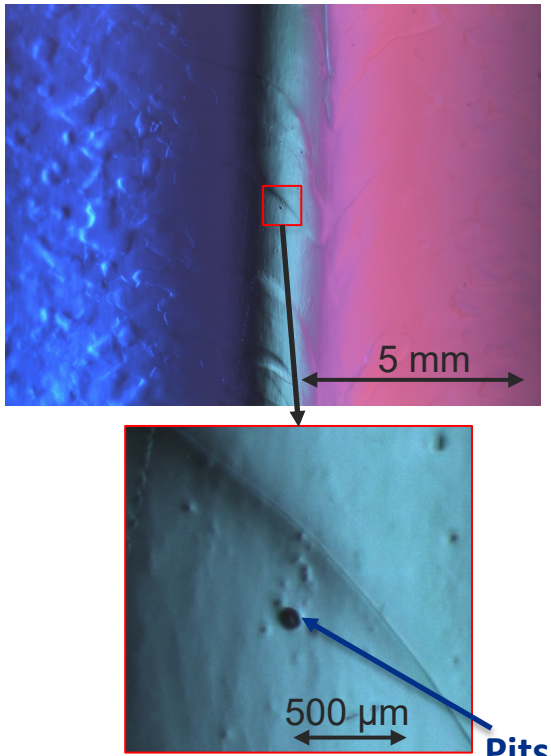


There might be physical damage of the diffusion layer due to bubbles.

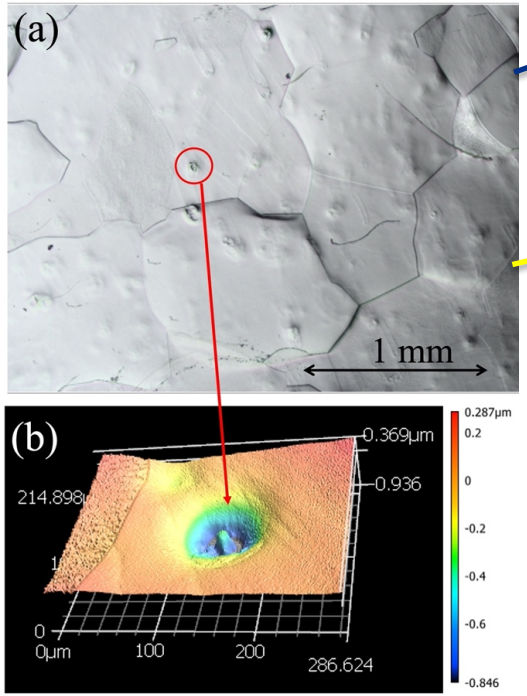
The top nitride layer was responsible for the bubble formation.

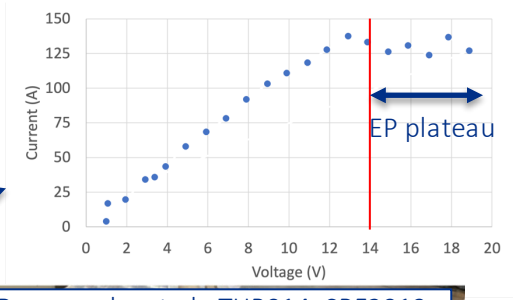
N-doped cavity surface after EP

1-cell cavity
EP at 32 °C & 18 V



Cutout from 9-cell cavity (CAV0018)
EP at 24 °C & 14 V





- The number of pits were significantly less on the cavity surface as compared to that on the samples.
- Cavity rotation removes bubbles periodically and forms a fresh diffusion layer on the surface.
- Cold EP could further reduce the pitting on the cavity surface. (to be confirmed).

Pits found on multiple locations

Highlights

- The top niobium nitride layer is responsible for pitting.
- Parameters that affect the surface pitting:
  - **Temperature**
  - **Applied voltage**
  - Acid agitation
  - Cavity rotation
  - HF concentration in acid (varies due to evaporation in acid mixing process)
- **Cold EP** at temperature **< 15 ℃** with a voltage **< 18 V** may reduce the risk of pitting and sharp grain boundaries. However, voltage should be above the onset voltage to avoid preferential etching of the grains.
- The pitting risk may be higher for the cavities (like 650 MHz cavities) that require a higher voltage of 22–25 V for EP.
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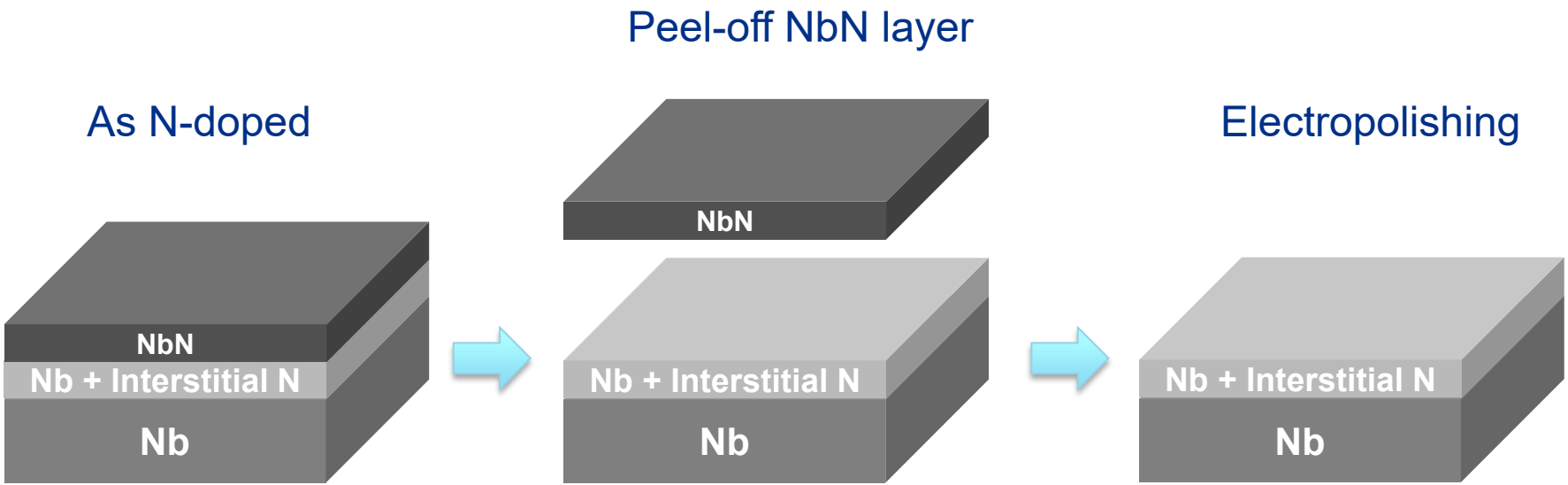
Can we have a process that eliminates the risk of pitting?

Process to eliminate pitting risk



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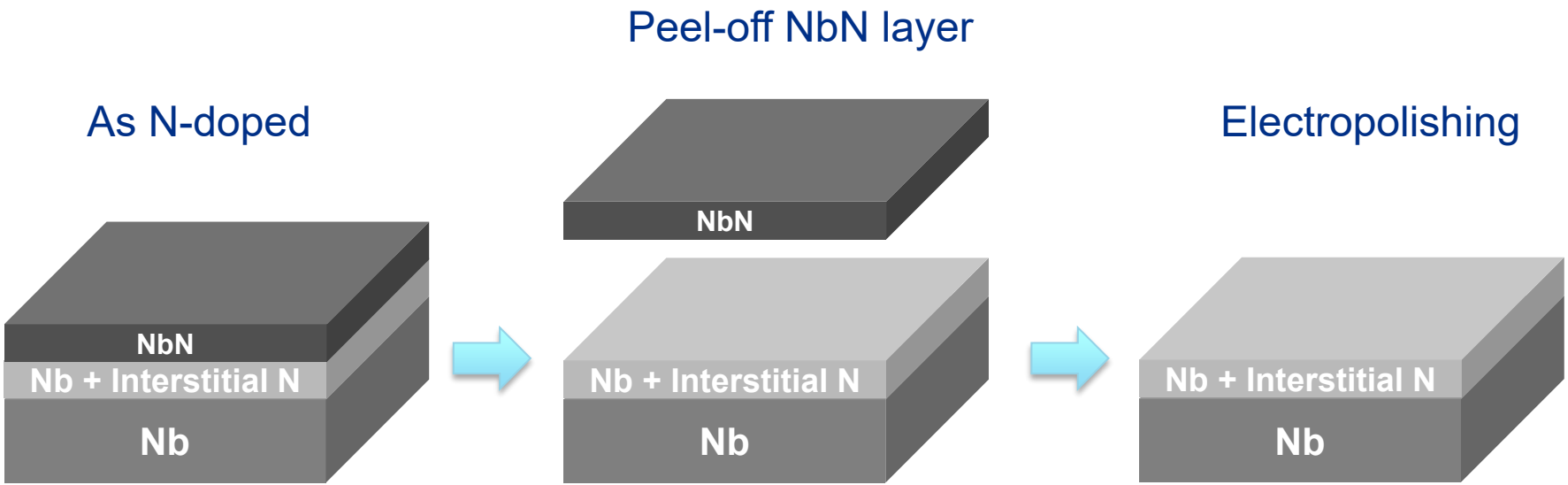
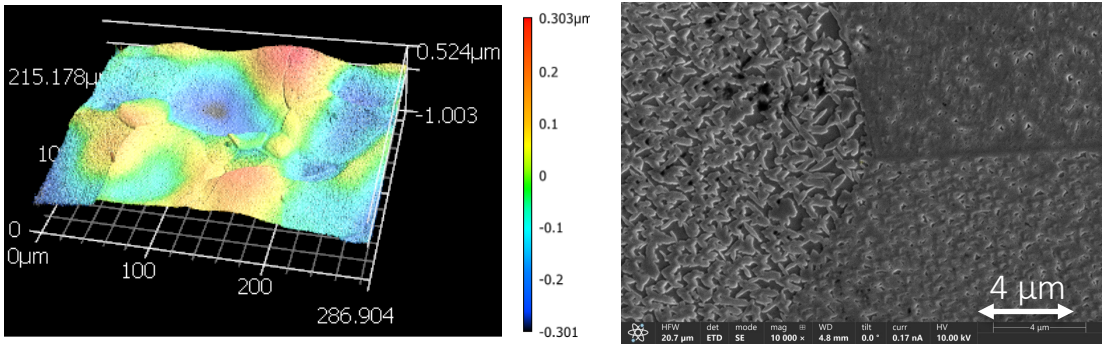


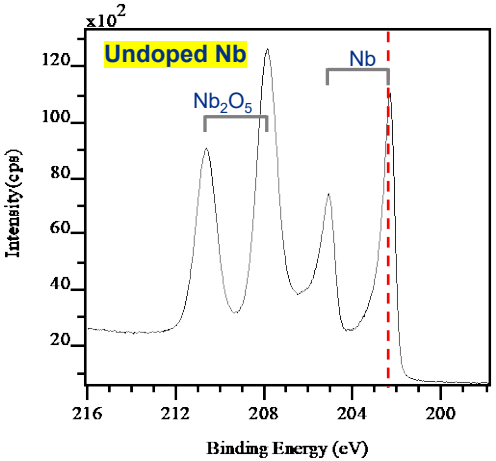
How can we peel-off the top thin nitride layer?

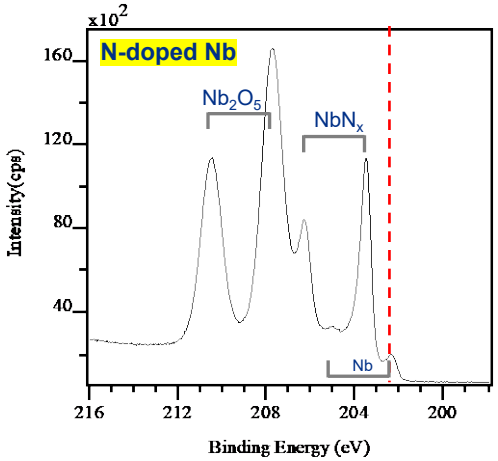
Can we dissolve nitride layer by EP-acid soak?

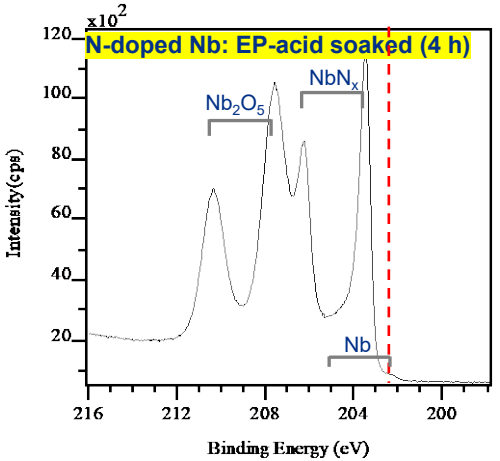
Surface after EP-acid soaking for 4 h



- Surface features altered.
- No pitting after 4 h of EP-acid soaking.







XPS analysis confirmed that nitride layer cannot be dissolved in several hours.

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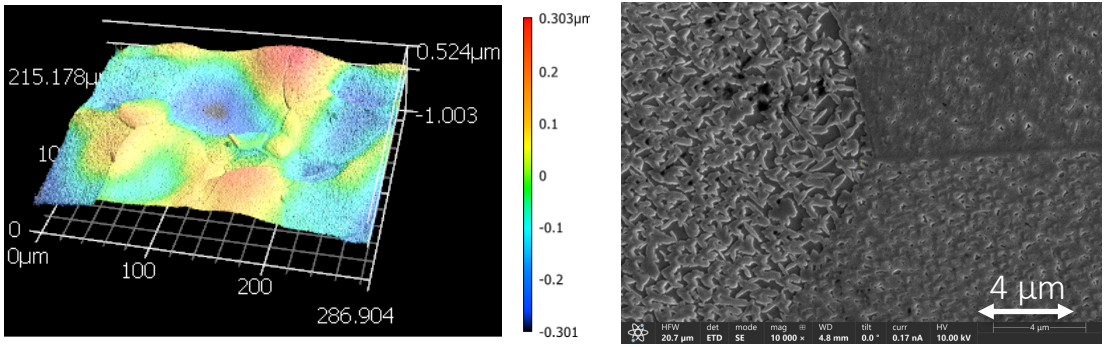
Can we dissolve nitride layer by EP-acid soak?

Surface after EP-acid soaking for 4 h



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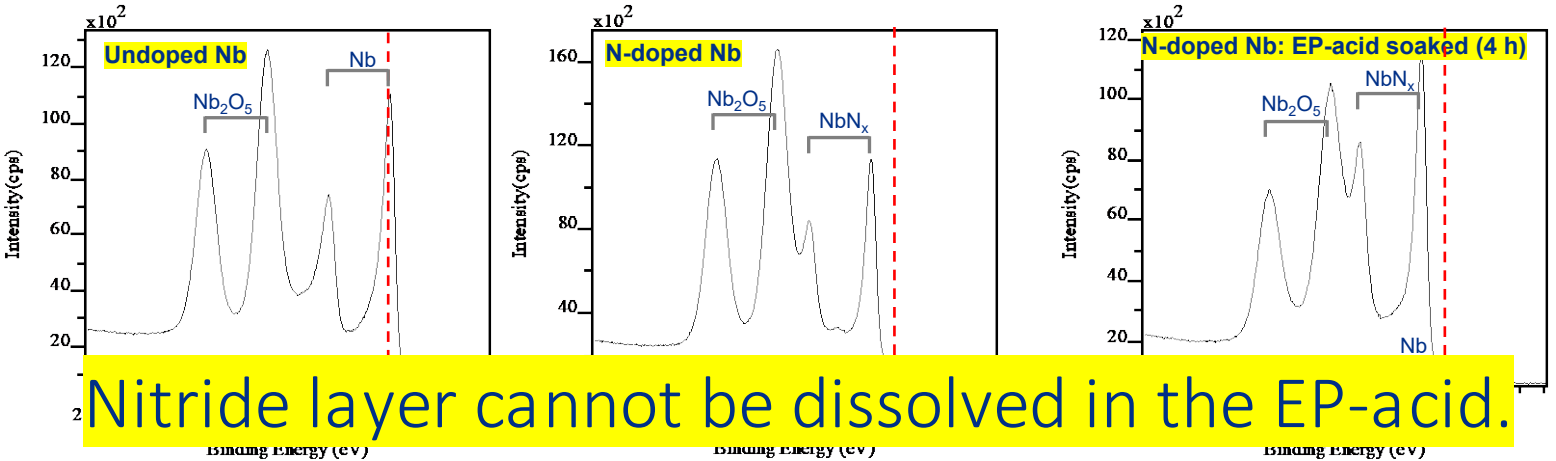

XPS analysis confirmed that nitride layer cannot be dissolved in several hours.

Nitride layer cannot be dissolved in the EP-acid.

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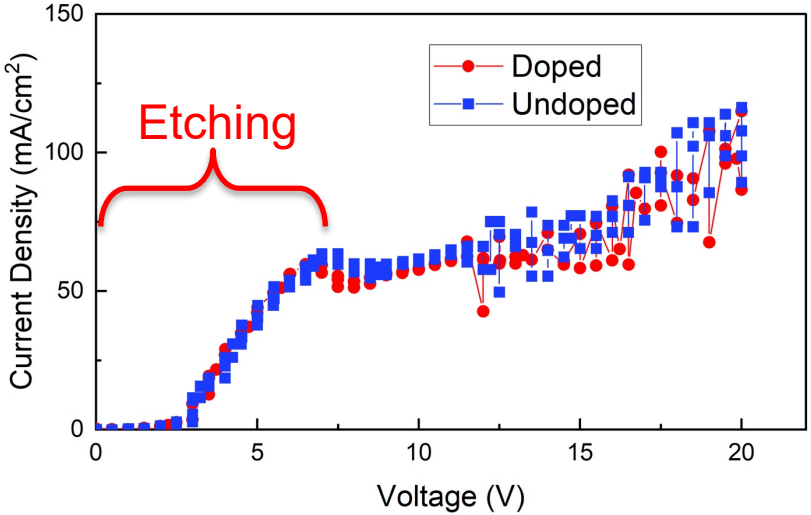
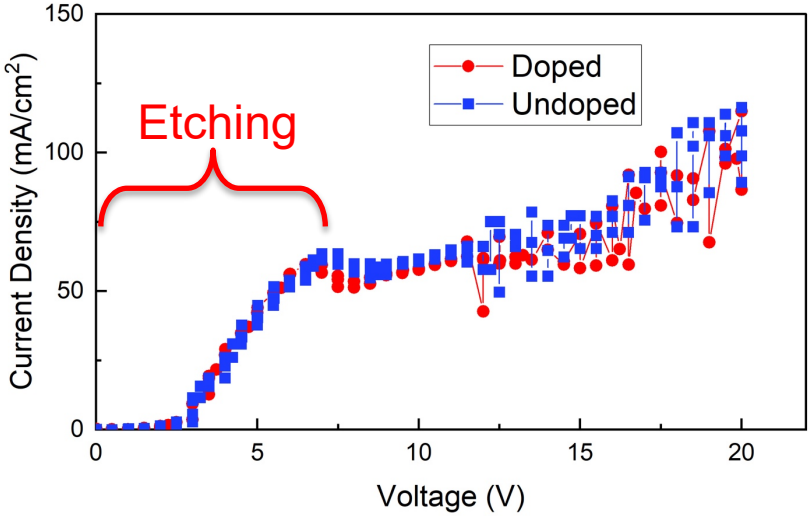
Effect of low voltage / I-V measurement

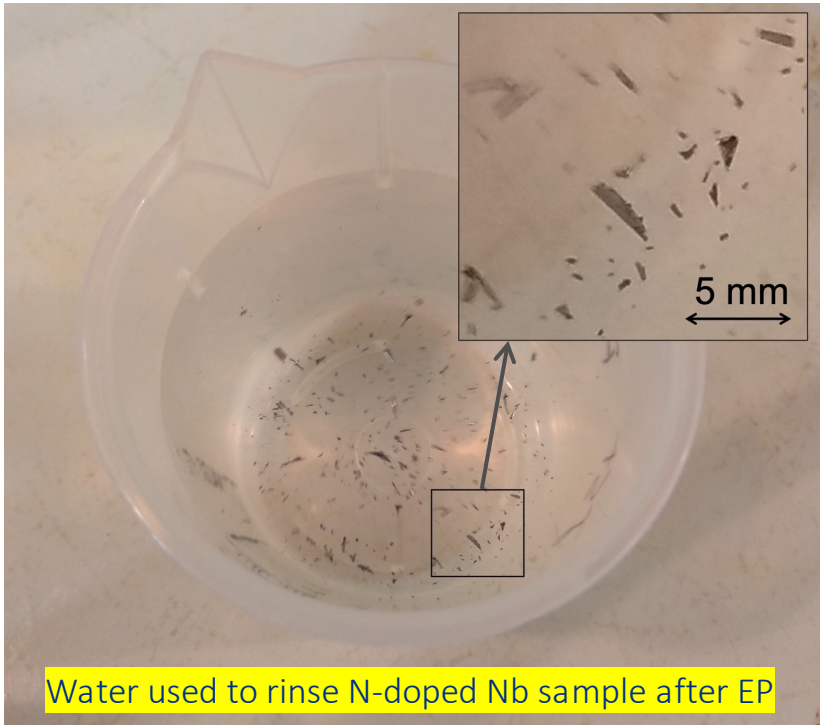


- A dark-gray film removed from the surface in an I-V test or low voltage etching.

Effect of low voltage / I-V measurement

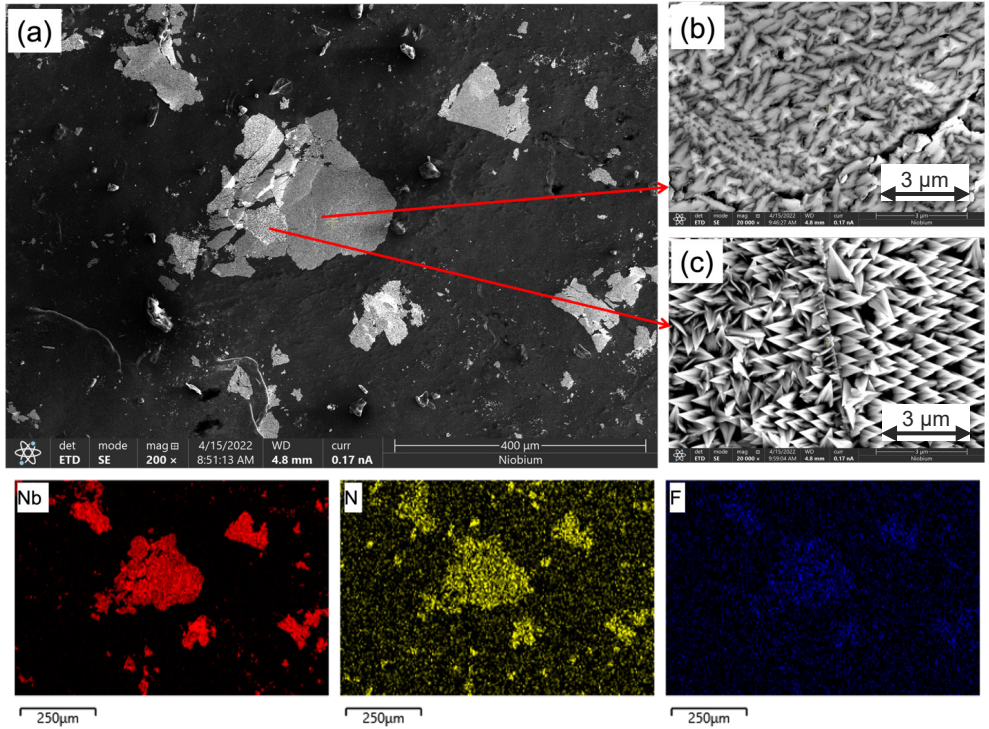


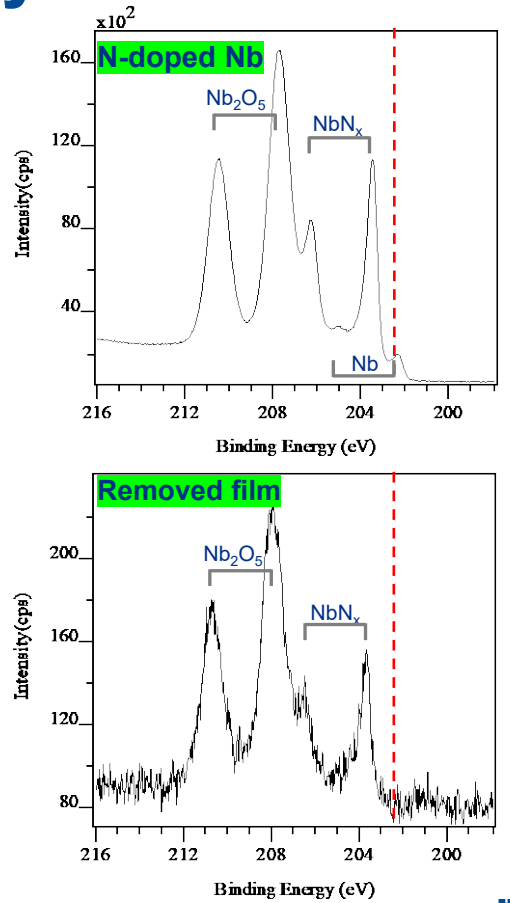
- A dark-gray film removed from the surface in an I-V test or low voltage etching.



Thickness of removed film ~ a few hundred nm

Microscopic and chemical study of removed film





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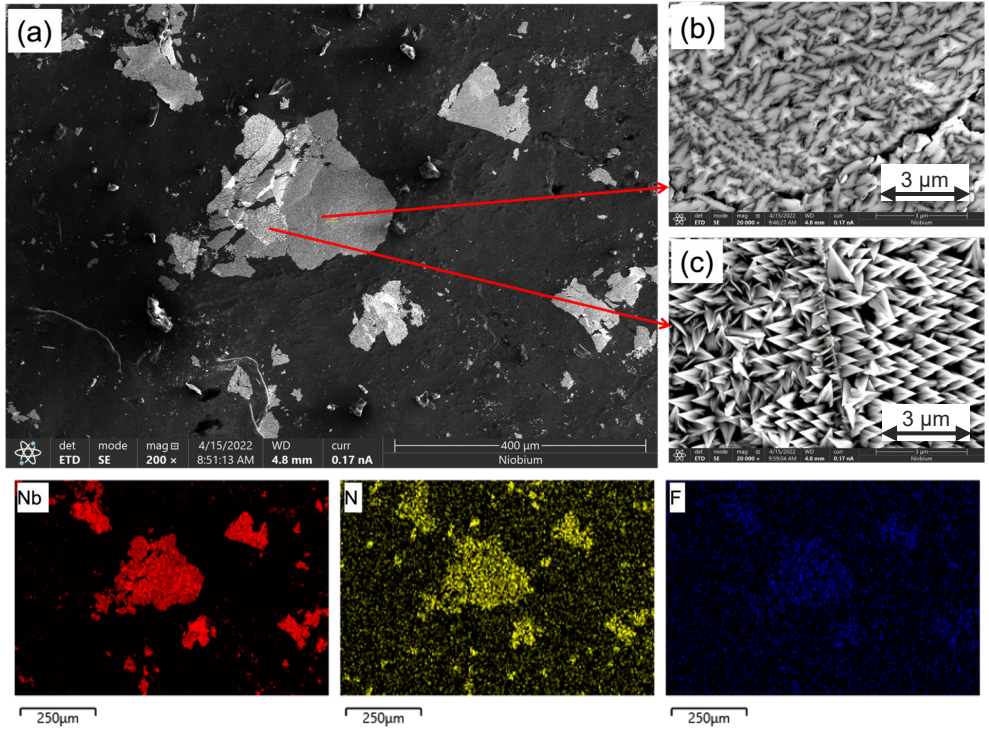
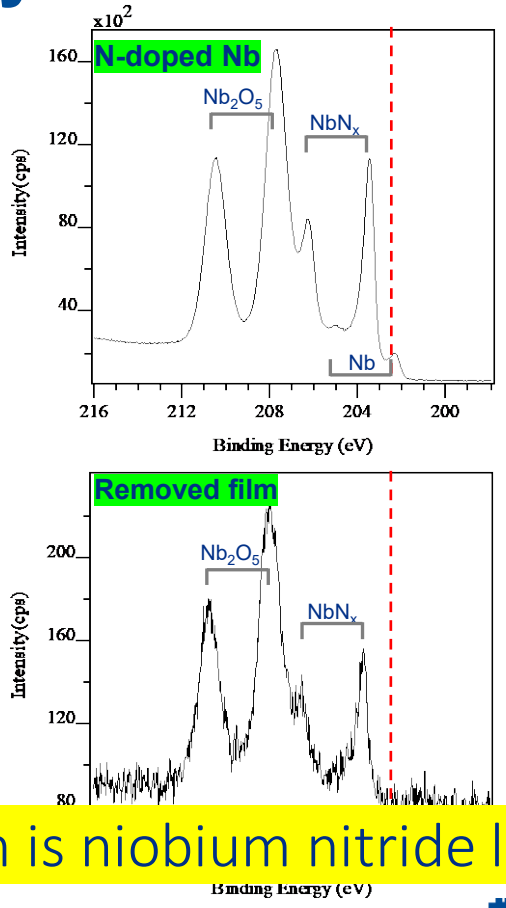
Microscopic and chemical study of removed film





Analysis confirmed that the removed film is niobium nitride layer.

Fermilab

EP recipe for N-doped surface (Two-step EP)

Post-doping EP recipe:

EP at 14-18 V for 5-10 μm

New Recipe:

Step-1

Low voltage etching (~0.3 μm) or a proper I-V

Step-2

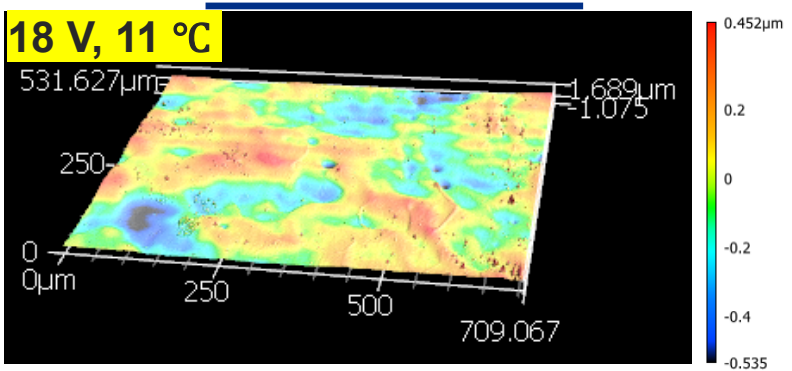
EP at ~18 V for rest removal

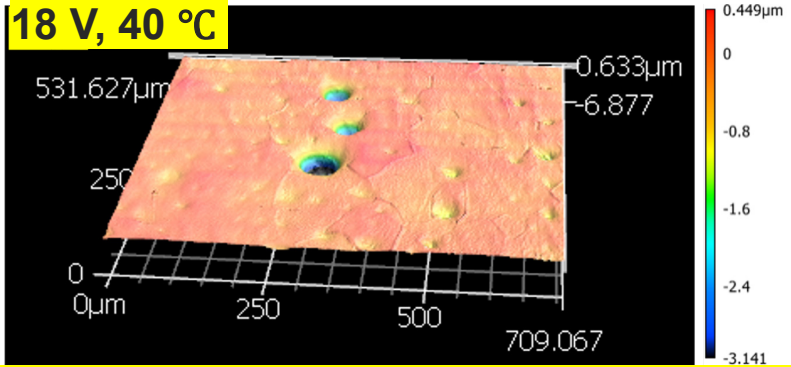
Step 1: To peel-off the top nitride layer

Step 2: To meet target removal thickness

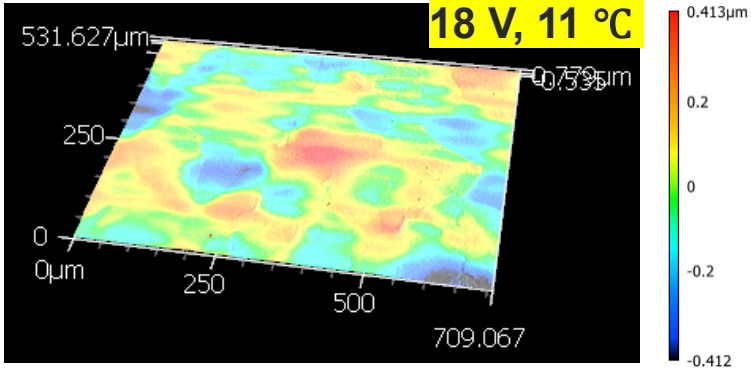
Surface after two-step EP

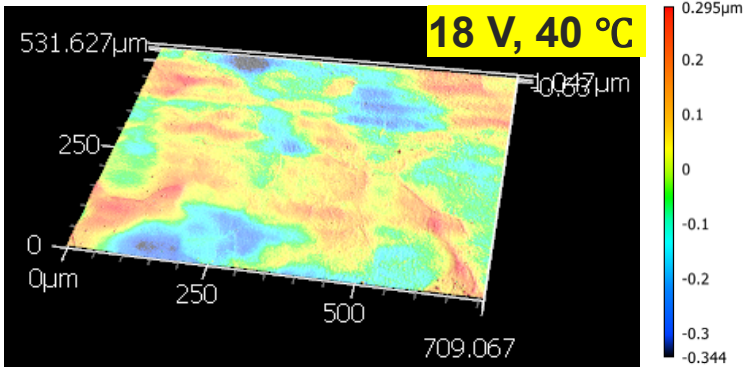
EP without I-V





Two step (I-V & EP)





Two-step EP eliminates the risk of pitting on an N-doped surface.

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Summary

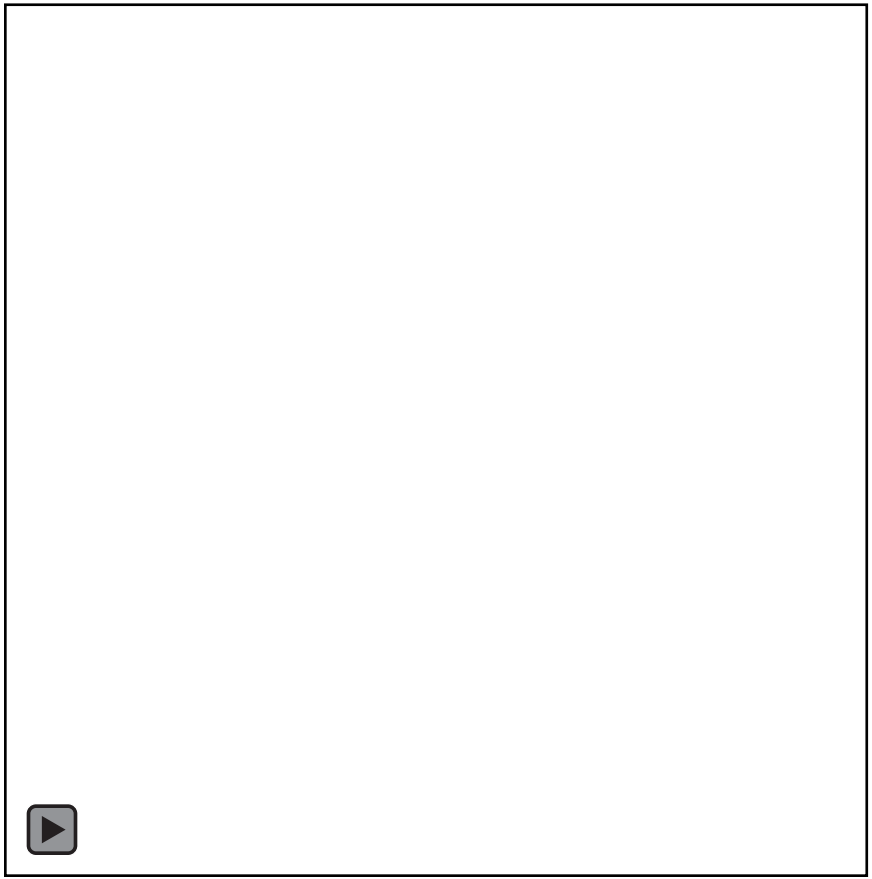
- The presence of a niobium nitride layer on the surface poses a risk of pit formation.
- The number of pits increases with increase in the surface temperature and voltage.
- Low-temperature EP reduce the risk of pit formation and surface roughness.
- A proper I-V scan can peel-off the nitride layer before EP is performed.
- The two-step EP (I-V and EP) could eliminate the pit formation caused by the nitride layer.

Next task:

- The two-step EP will be applied to multiple cavities to confirm the impact on the performance and yield.

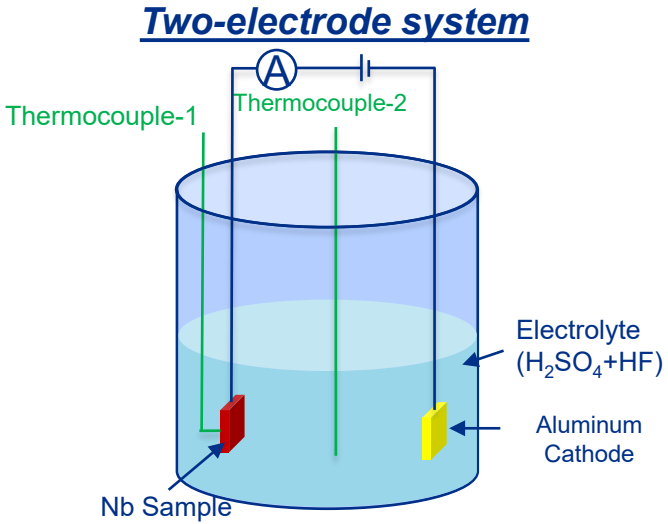
Thank You

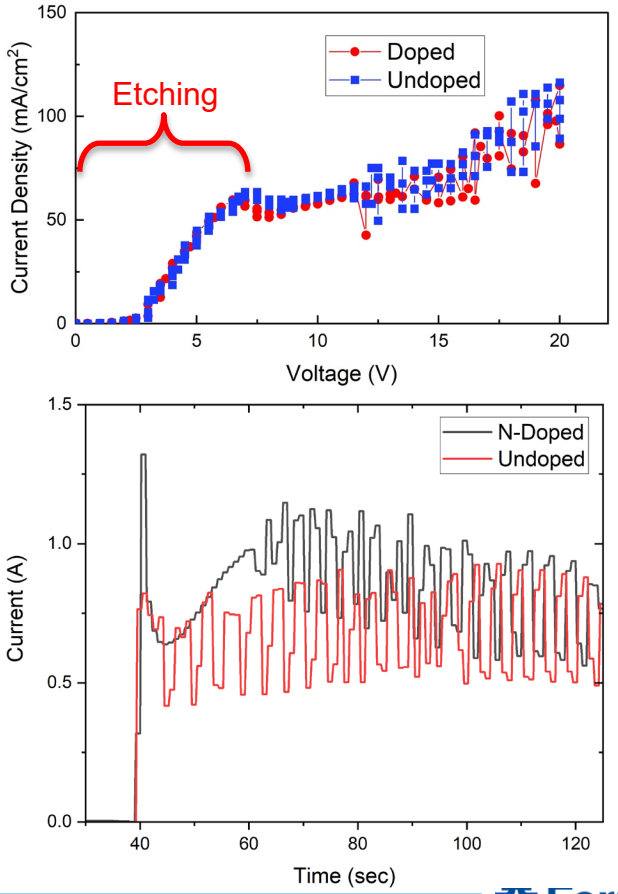
Thank You



Backup slides

Polarization curve and current profile





- N-doped and undoped samples were electropolished.
- Polarization curves were identical.
- EP current drawn from the N-doped surface was initially higher.