

Alternative polishing technique of Niobium for SRF applications

Oleksandr Hryhorenko¹, Claire Antoine², Takeshi Dohmae³, William Magnin⁴, David Longuevergne¹

¹ IJCLAB, Laboratoire de Physique des 2 Infinis Irène Joliot Curie, 15 Rue Georges Clemenceau, 91400 Orsay, France

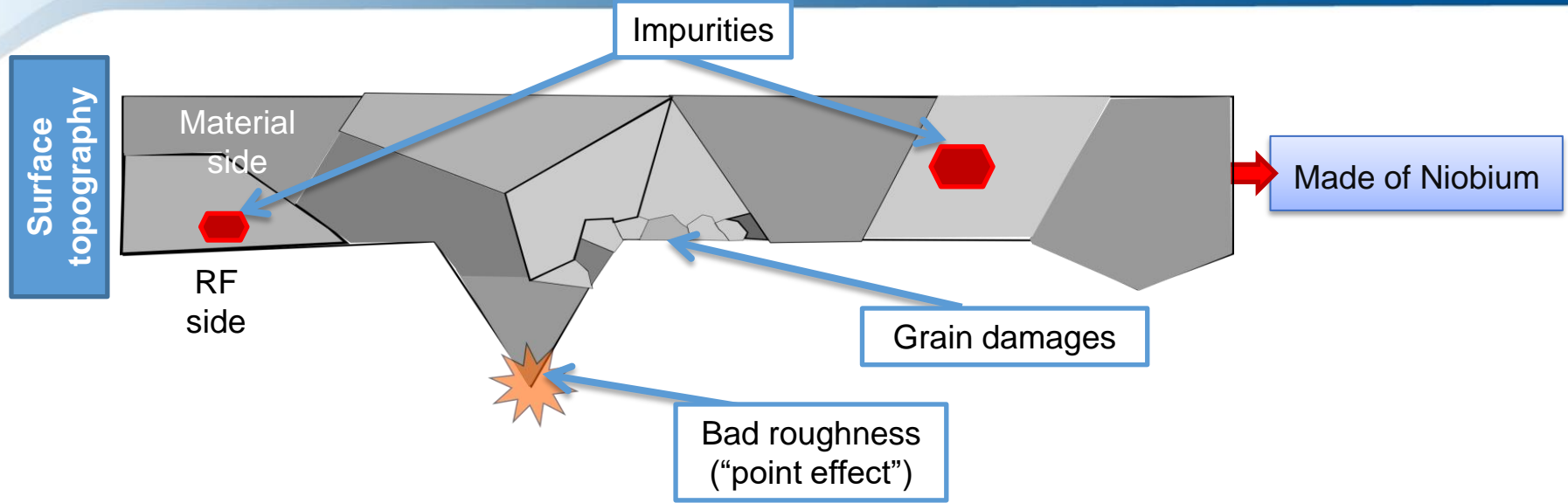
² Département des Accélérateurs, de Cryogénie et de Magnétisme, CEA/DRF/IRFU, 91191 Gif-sur-Yvette, France

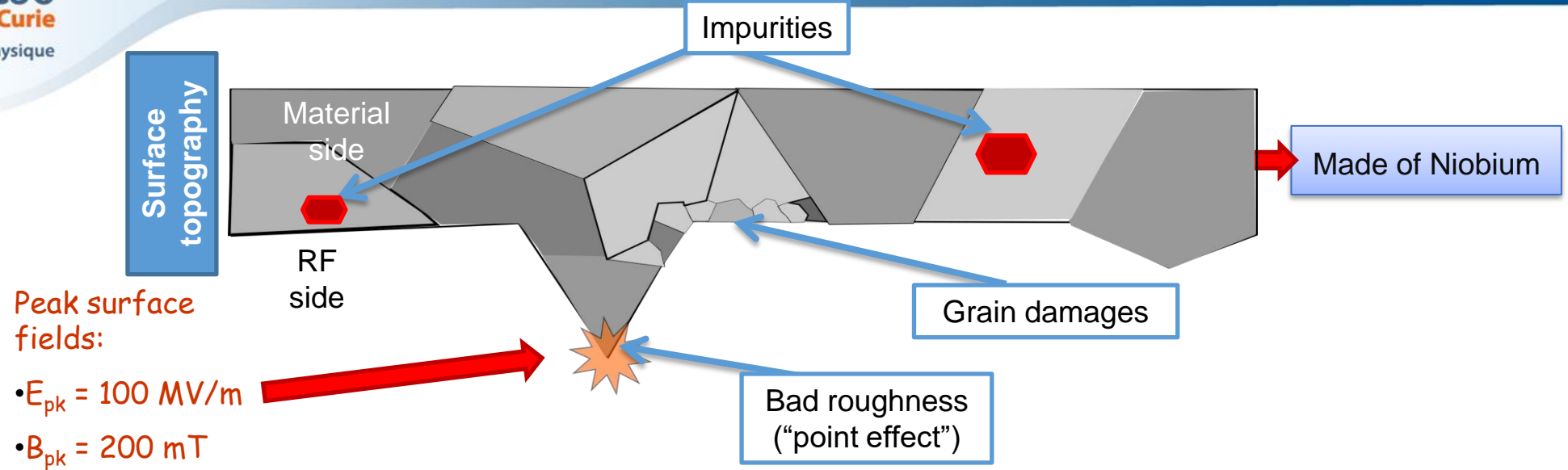
³ KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan

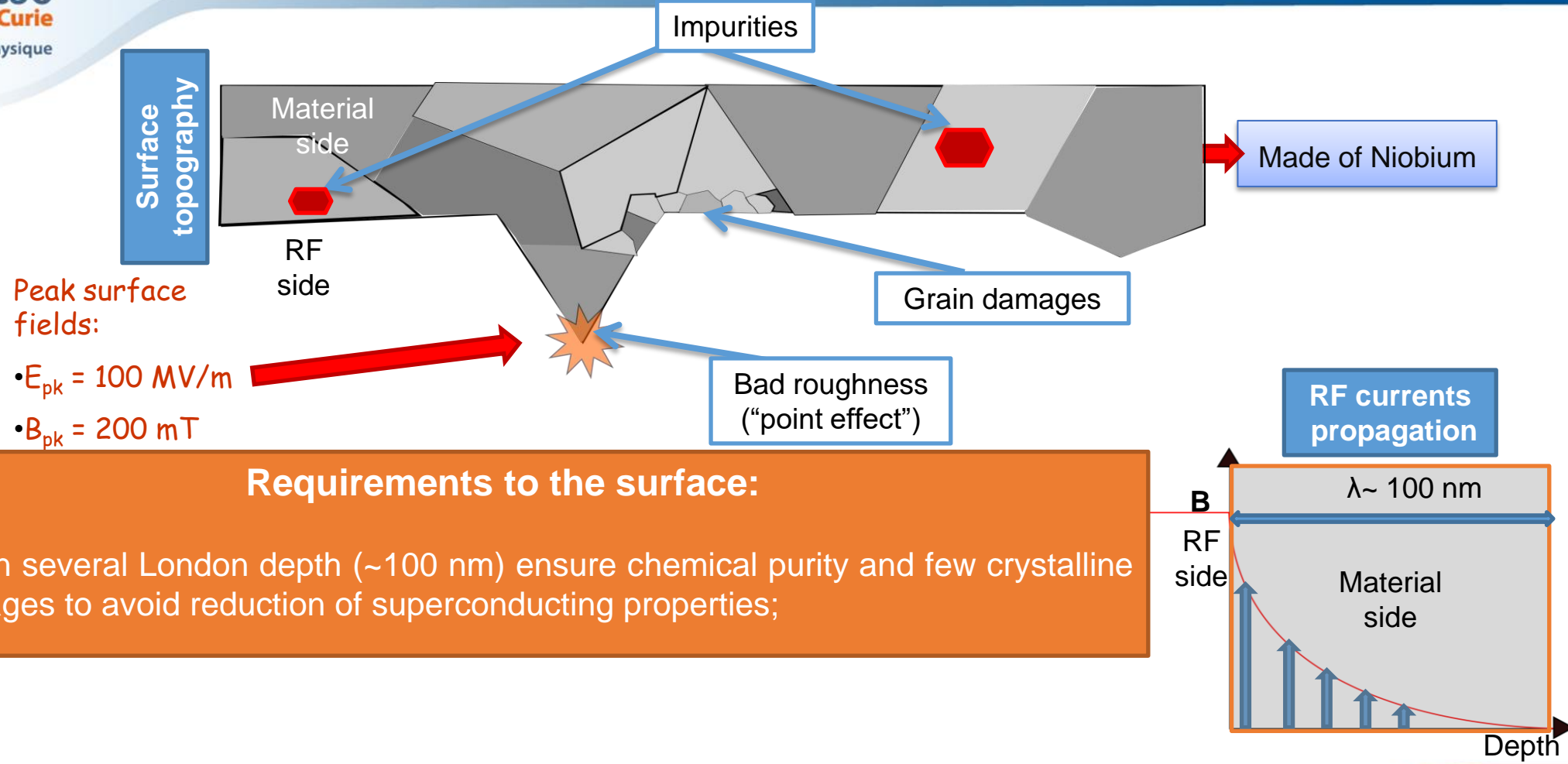
⁴ LAM PLAN, 7 Rue des Jardins, 74240 Gaillard, France

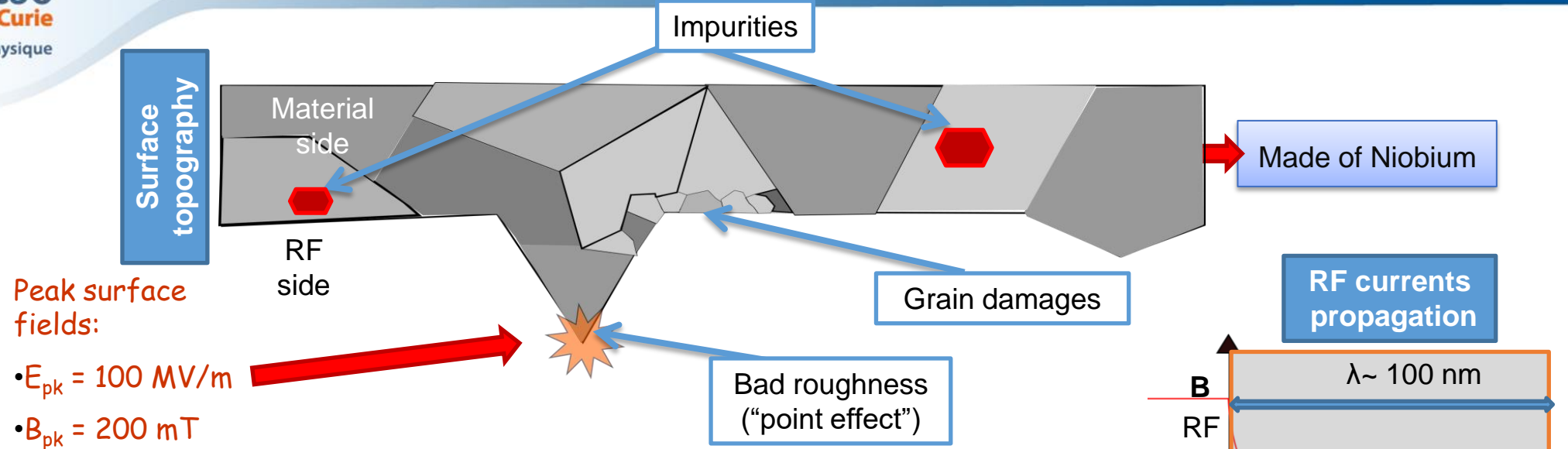
- Motivation for SRF applications (Why polish? Why alternative?)
- Alternative path for SRF cavity fabrication and surface processing
 - Requirements for Niobium
 - Requirements for industrialization
- Metallographic polishing as an alternative technique
 - R&D on the small samples
 - Surface characterization (roughness, EDS, EBSD...) & cryogenic RF test
 - Transfer of recipe to large disks
- Conclusion and perspective

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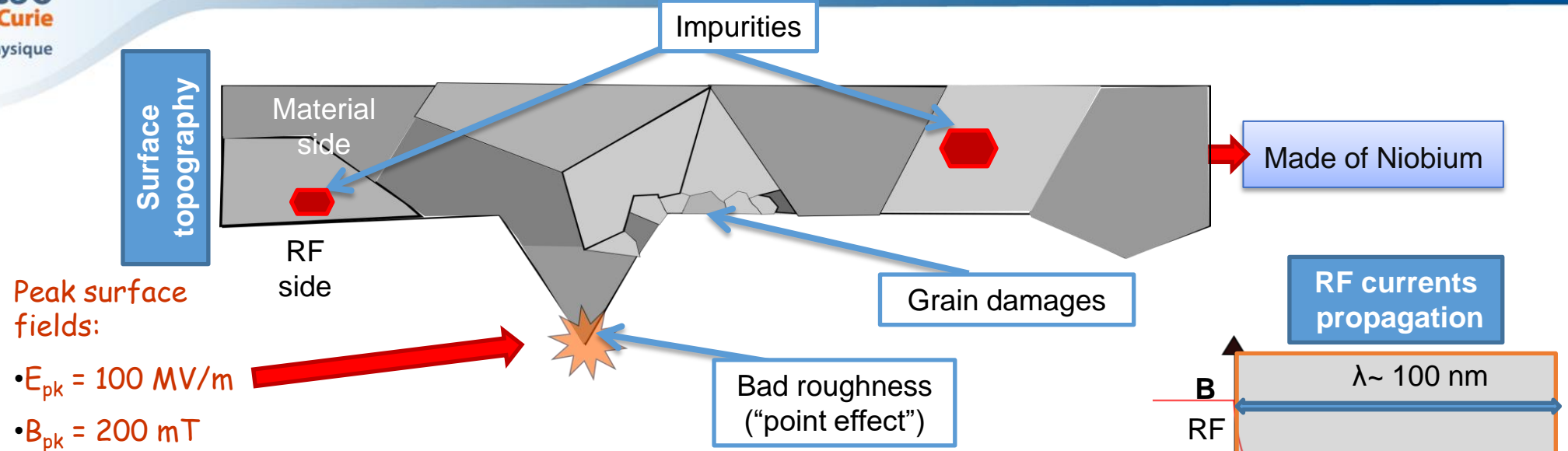






Requirements to the surface:

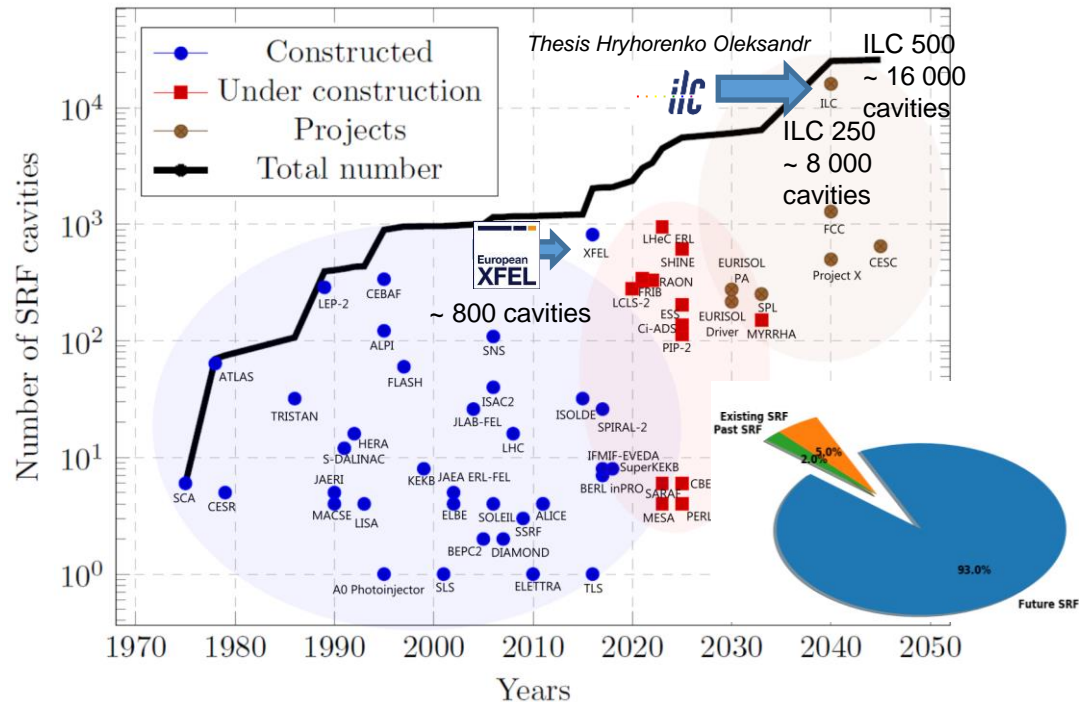
- Within several London depth ($\sim 100 \text{ nm}$) ensure chemical purity and few crystalline damages to avoid reduction of superconducting properties;
- Ensure roughness at least as good as with EP ($Sa \leq 0.1 \mu\text{m}$);



Requirements to the surface:

- Within several London depth (~100 nm) ensure chemical purity and few crystalline damages to avoid reduction of superconducting properties;
- Ensure roughness at least as good as with EP ($Sa \leq 0.1 \mu\text{m}$);
- Ensure and maintain high thermal conductivity of bulk Nb (for high gradient operation)

Possible reduction of the cost of cavity surface processing (replace bulk EP and BCP).

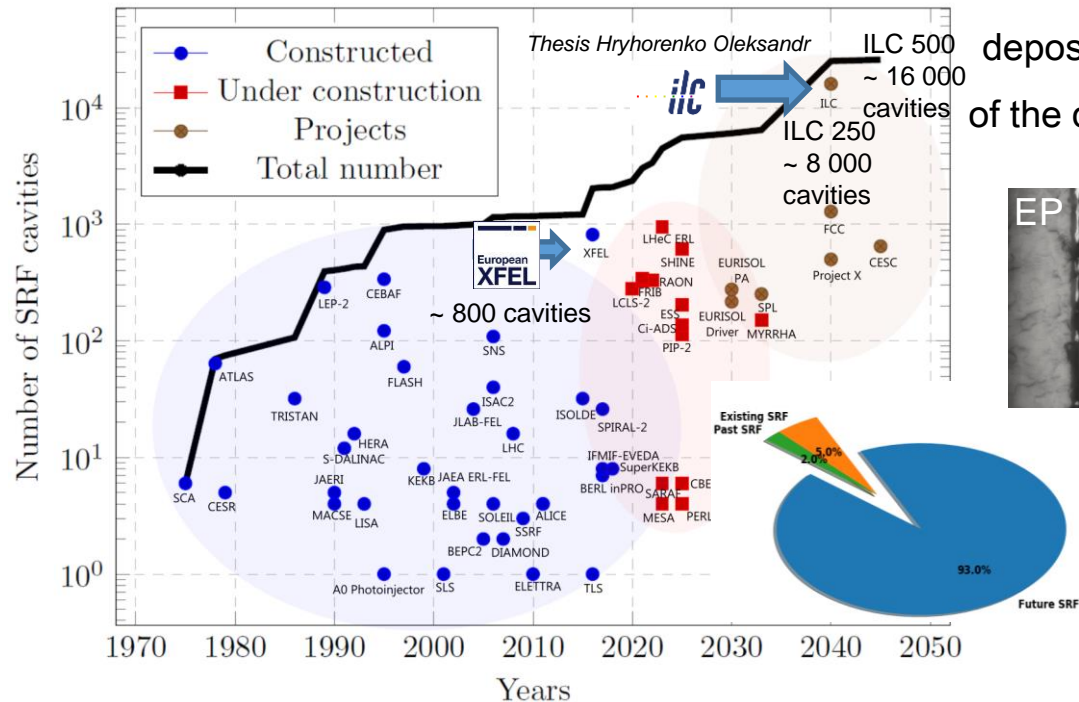


<https://tel.archives-ouvertes.fr/tel-02455975>

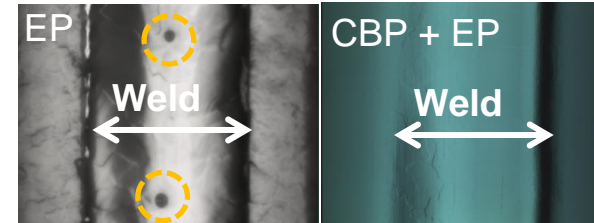
Laboratoire de Physique
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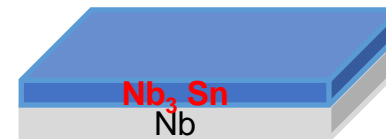
Achieve better surface roughness to improve the performance (removal of all type of defects, substrate preparation for thin film deposition) => possible reduction of the cost of accelerator operation



<https://tel.archives-ouvertes.fr/tel-02455975>



[A.D. Palczewski et al. "R&D Progress in SRF Surface Preparation With Centrifugal Barrel Polishing \(CBP\) for both Nb and Cu", in Proc. 16th Int. Conf. RF Superconductivity \(SRF'13\), Paris, France, Sep. 2013, paper TUIOB01, pp. 398-403.](#)



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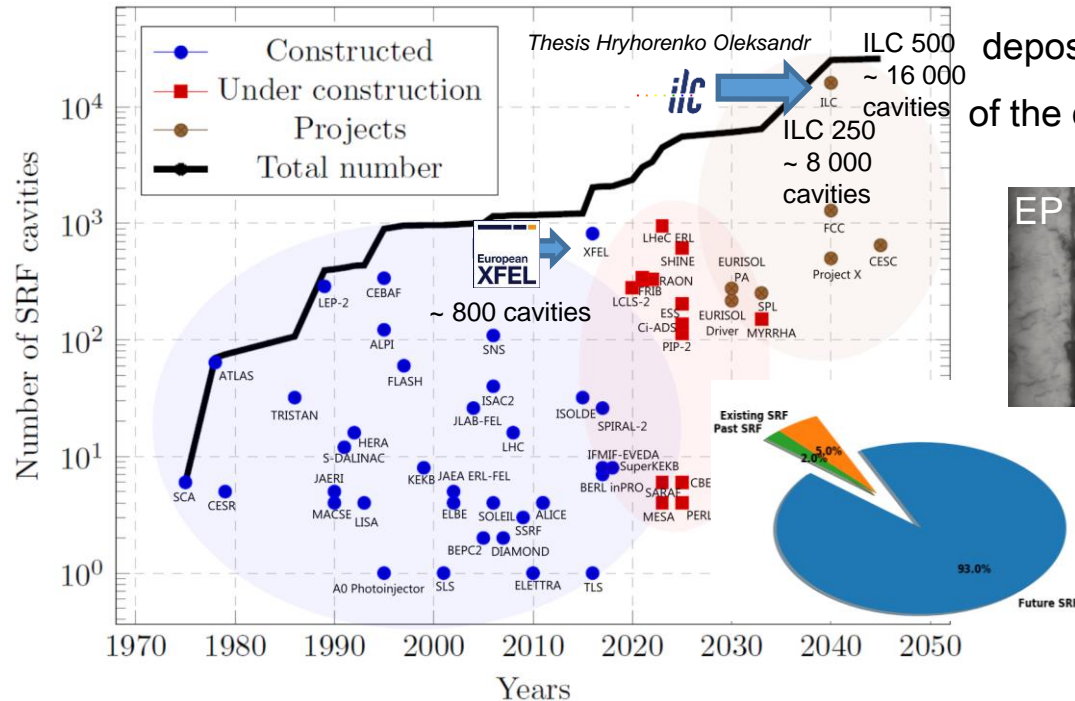
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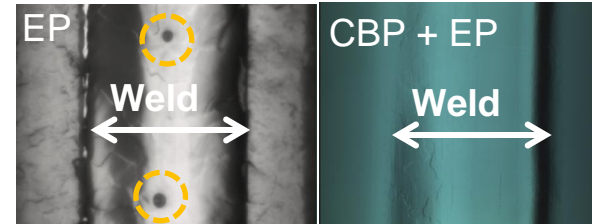
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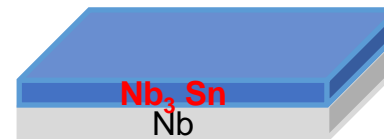
Improve environmental footprint and worker safety (remove or at least reduce the amount of used acids)



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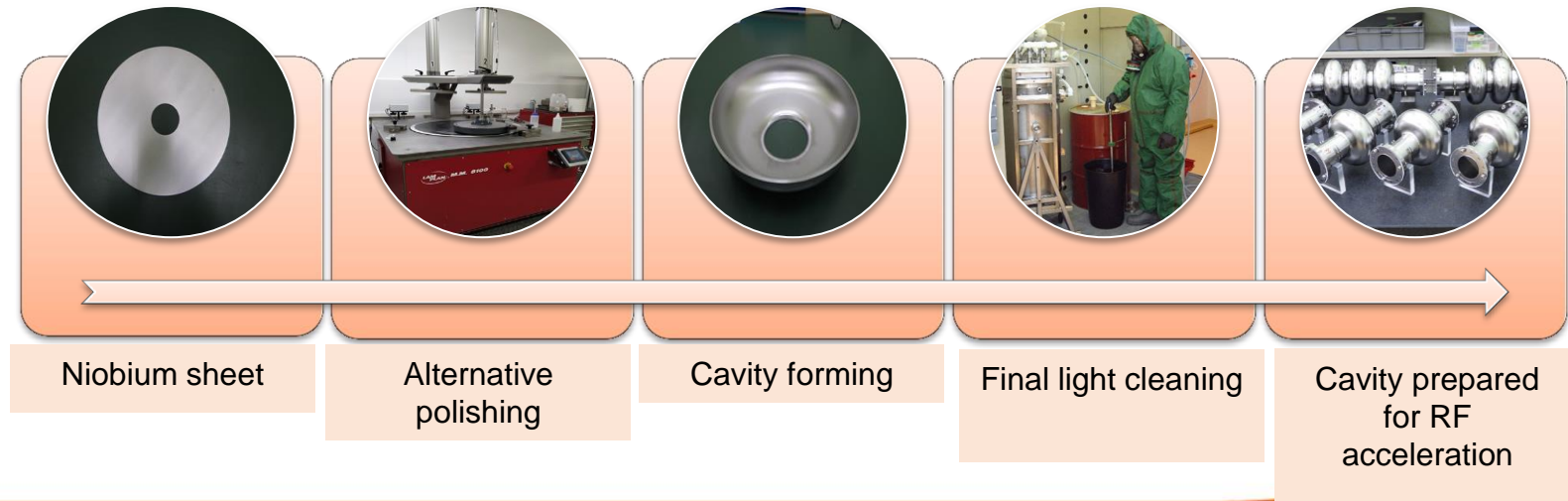
Standard path :




Standard path :

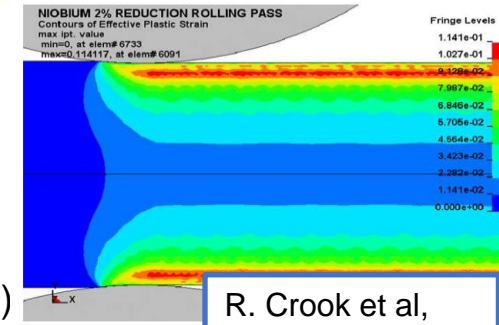


Alternative path :



Requirements for Niobium:

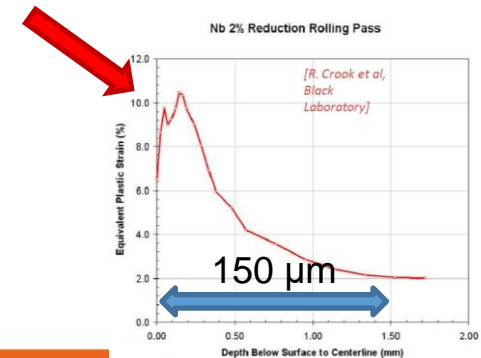
1. Remove the polluted & damaged layer induced by Nb sheet fabrication. 
2. Smooth and compataible roughness with EP & BCP (average surface roughness less than 1 μm)
3. Chemically clean (heavy elements < 0.01 wt%: Ta, W, Si, Fe; light elements < 0.001 wt%: O, N, H, C)
4. Minor crystallographic damages (stress as low as possible)



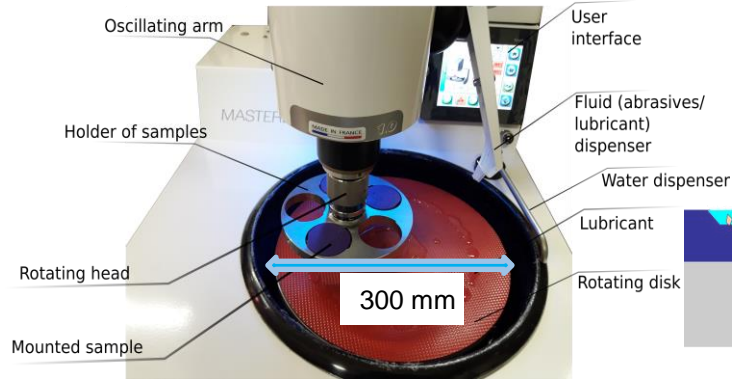
R. Crook et al,
Black Laboratory

Requirements for industrialization:

1. Time of treatment should be shorter than conventional polishing (~5 hours BCP, ~8 hours EP) => High removal rate ~μm/min.
2. Limit manipulations and process to 2 steps.



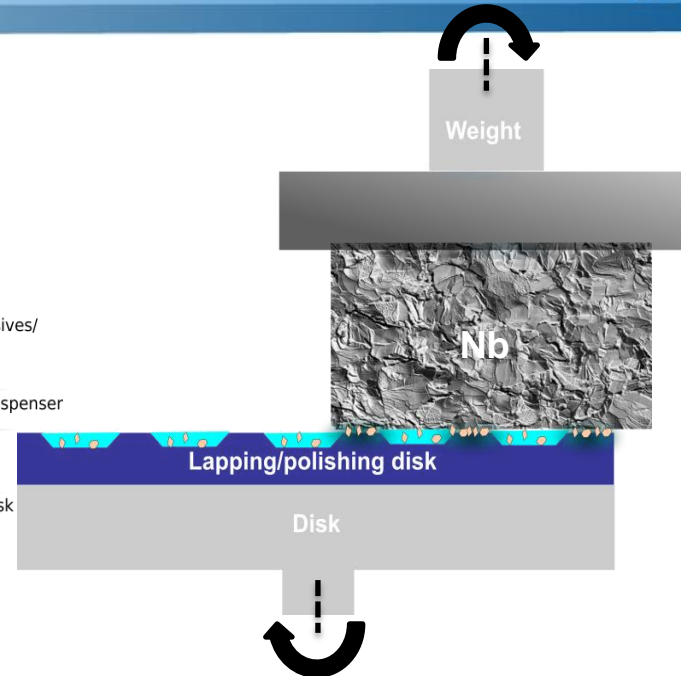
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Polishing protocol

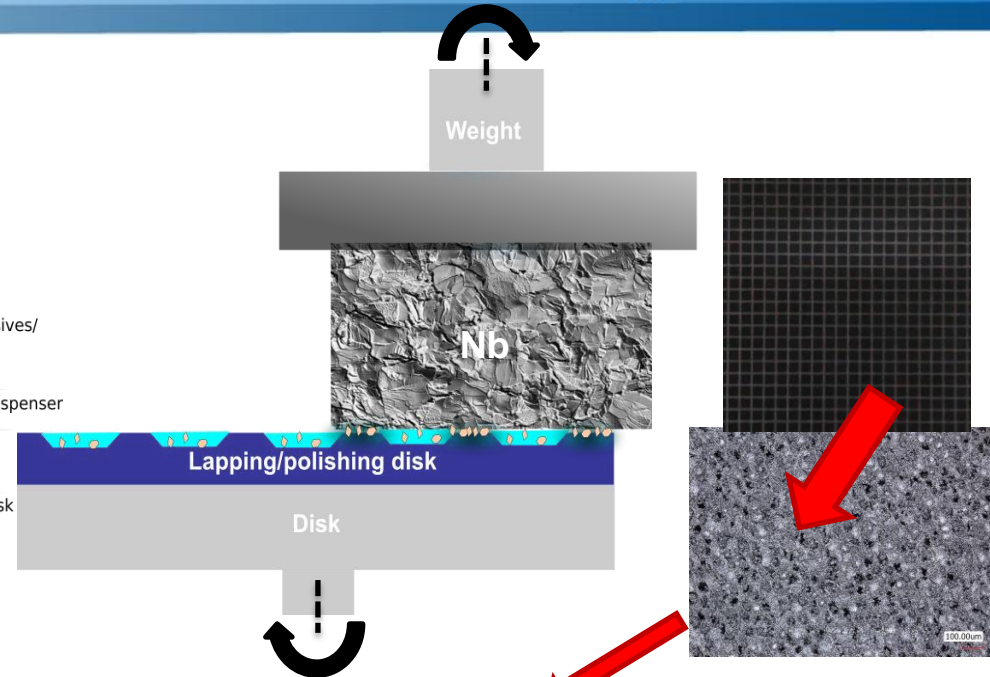
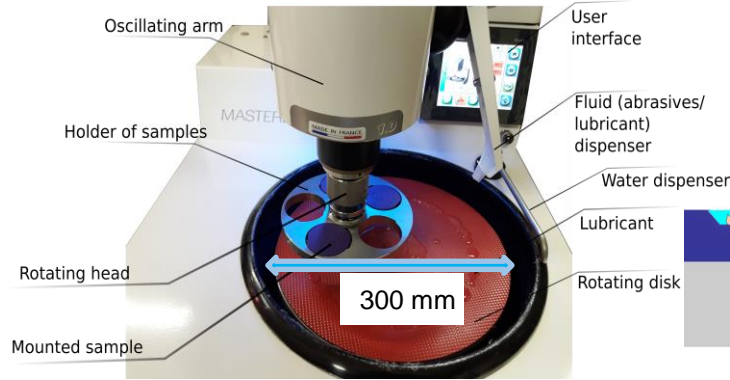
First step:

1. Coarse planarization
2. Remove the damaged layer due to the rolling of sheets (150 microns).



Second step:

1. Remove damages and pollution from previous step
2. Achieve the final surface requirement ($S_a \leq 0.1 \mu\text{m}$),
3. Limit pollution, minor crystalline damages



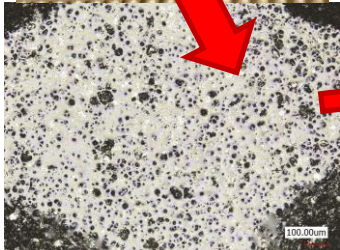
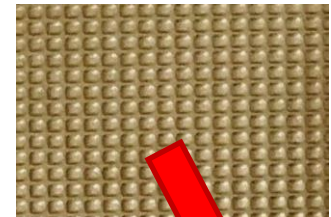
Polishing protocol

First step: RCD + diamonds of 3 μm

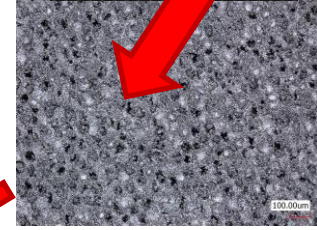
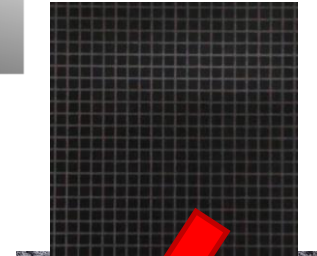
1. Coarse planarization
2. Remove the damaged layer due to the rolling of sheets (150 microns).

Second step: polyurethane cloth + SiO_2 (pH=10)

1. Remove damages and pollution from previous step
2. Achieve the final surface requirement ($S_a \leq 0.1 \mu\text{m}$),
3. Limit pollution, minor crystalline damages



Resin and Cu powder



Before:

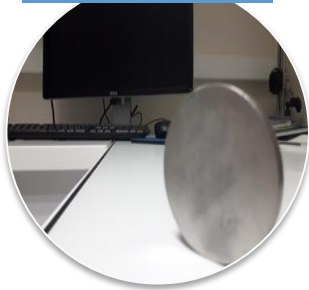
Roughness

Optimization of
polishing recipe



15x40 mm

RF test at
SLAC



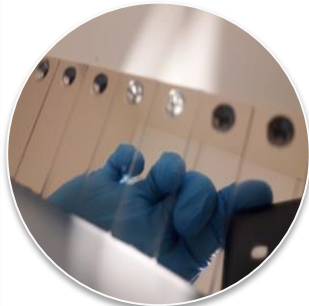
Ø 50 mm

Extended to
larger area



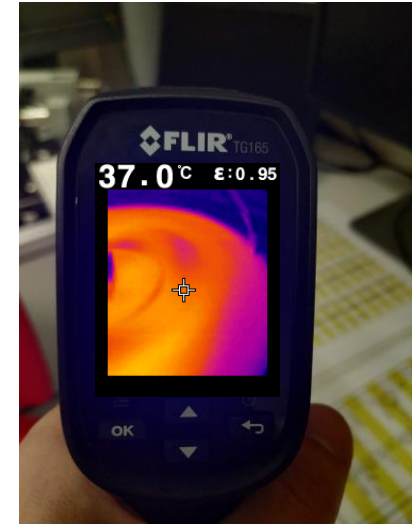
Ø 126 mm

After:



Size, mm

Temperature is fixed
below 40 degrees



Before:

Roughness

Optimization of
polishing recipe



15x40 mm

RF test at
SLAC



∅ 50 mm

Extended to
larger area

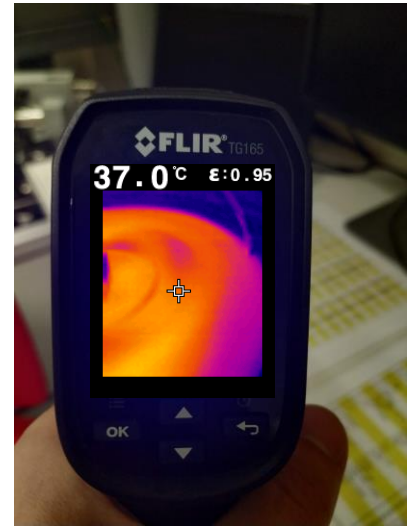


∅ 126 mm

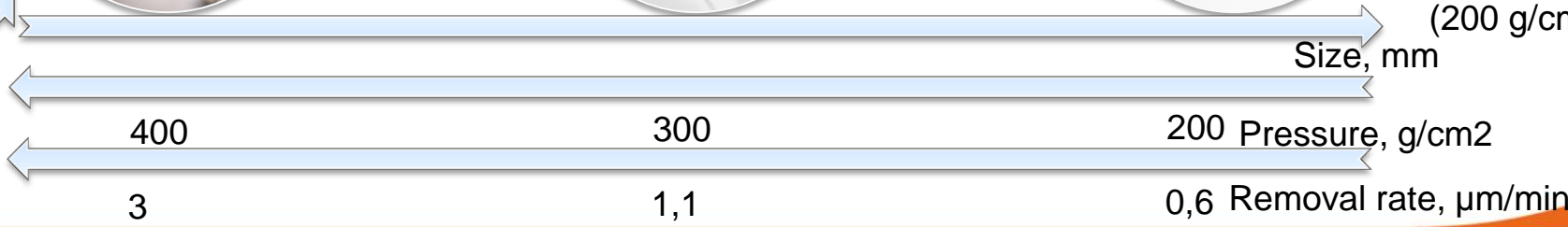
After:



Temperature is fixed
below 40 degrees



Pressure:
(200 g/cm² < P < 400 g/cm²)

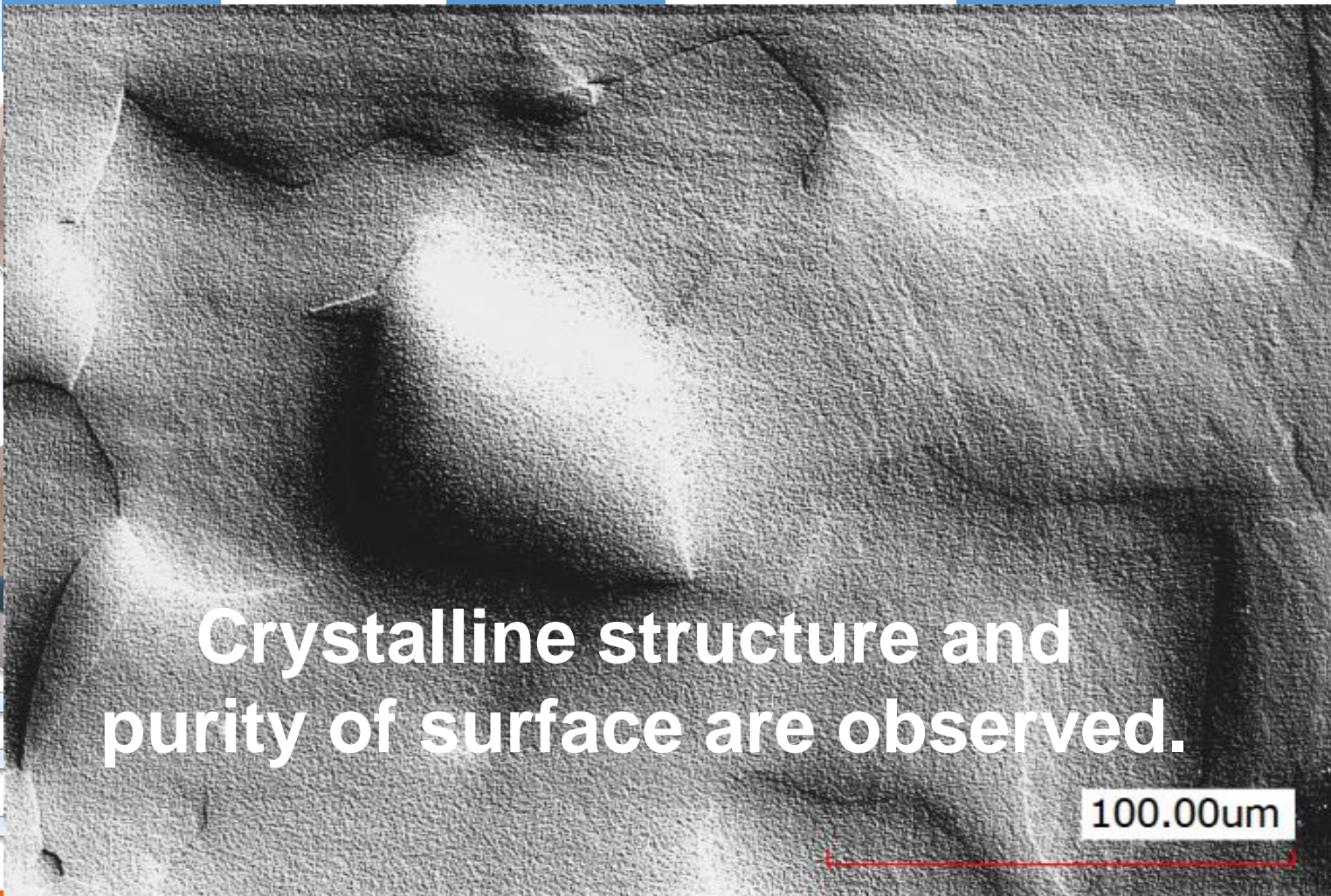


Removal rate:
EP – 0.3 μm/min
BCP – 0.5 μm/min

Before:

Roughness

After:



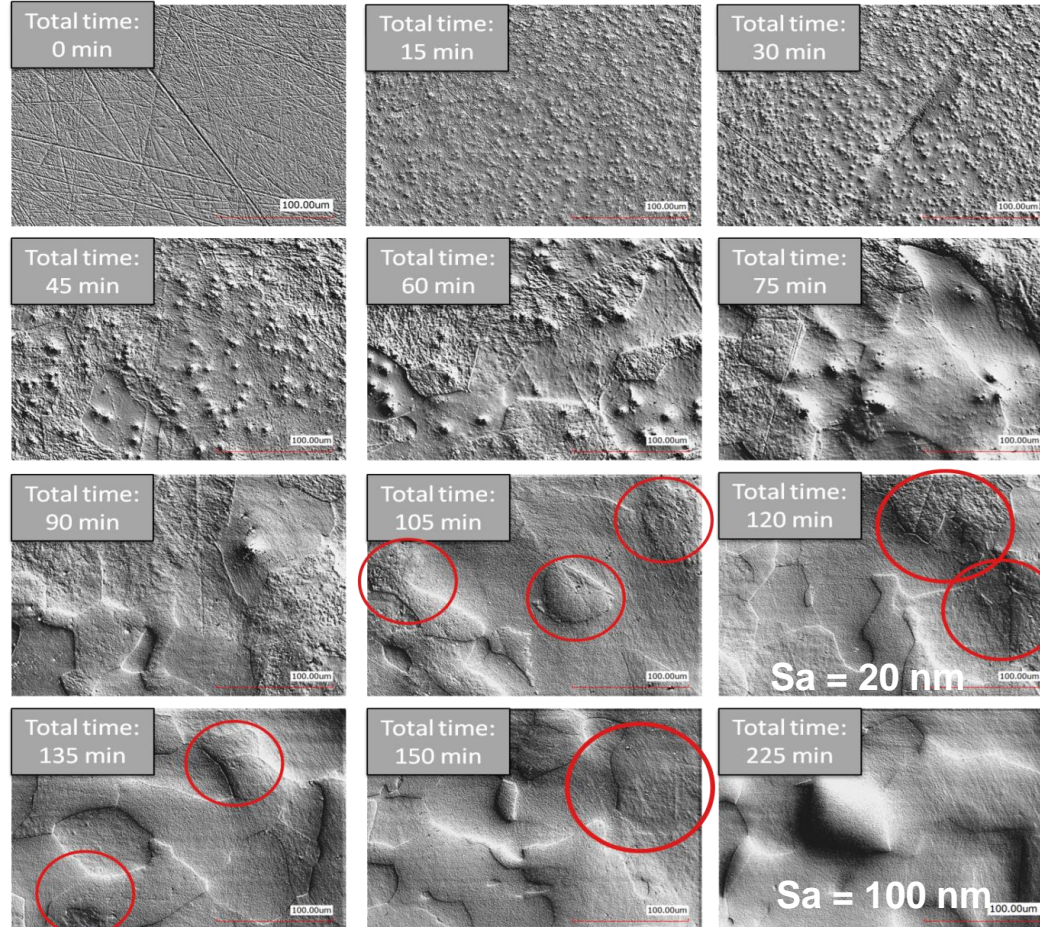
Temperature is fixed
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re:
($cm^2 < P < 400 \text{ g/cm}^2$)

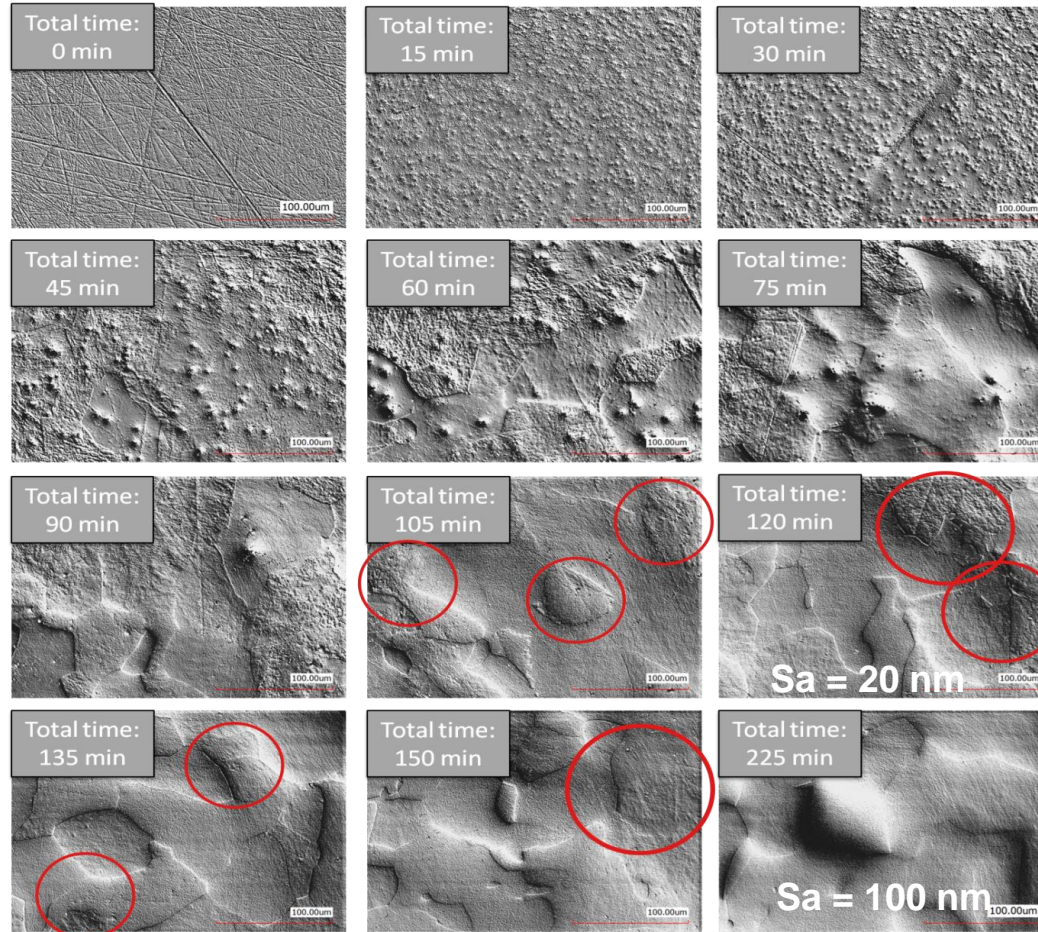
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EP – 0.3 $\mu\text{m/min}$
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Final polishing step

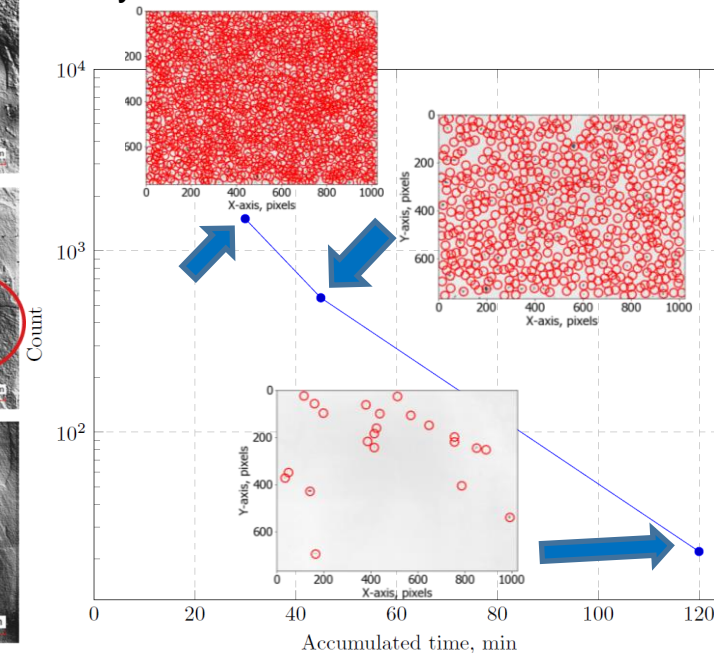


➤ Contamination is observed after lapping (diamond particles)

Final polishing step

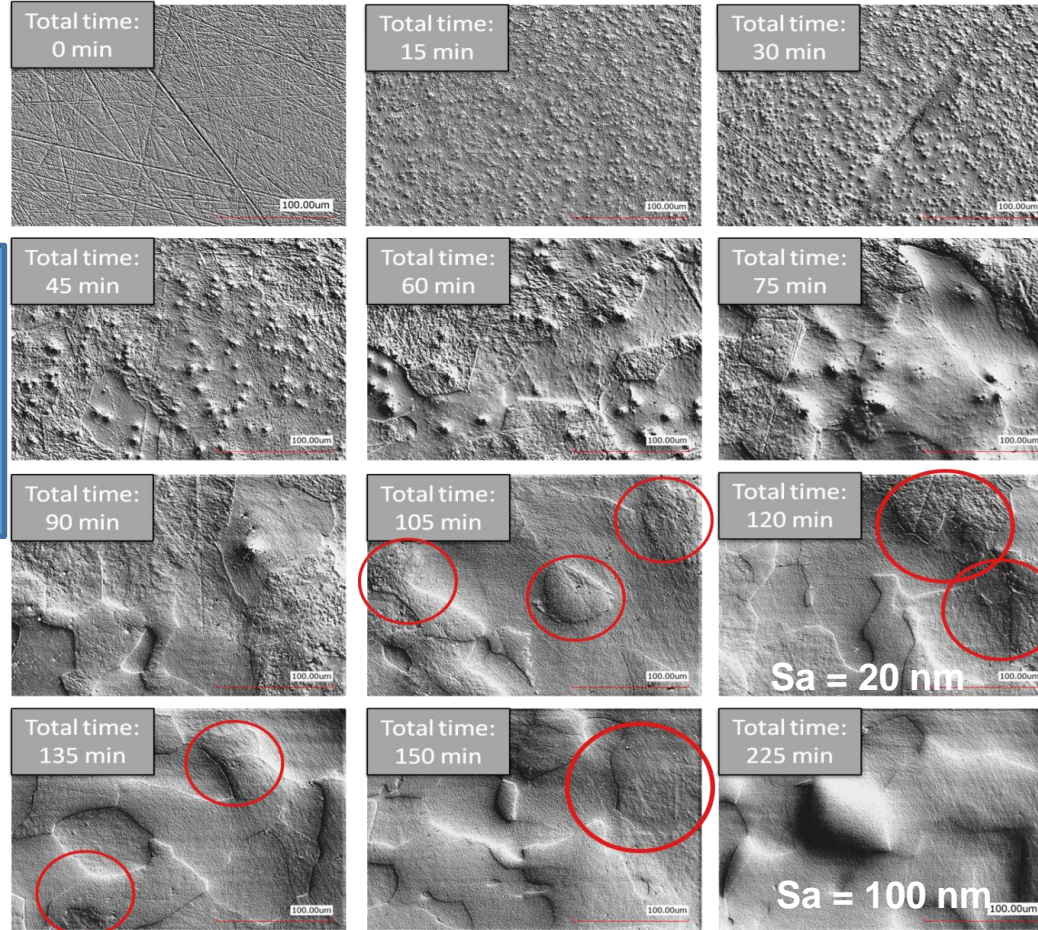
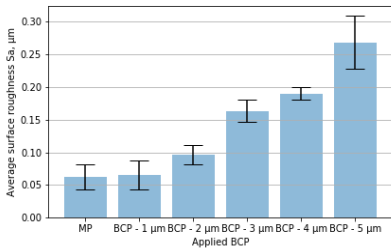


- Contamination is observed after lapping (diamond particles)
- Chemical-mechanical polishing is required (colloidal silica)
- Reappearance of surface crystalline structure

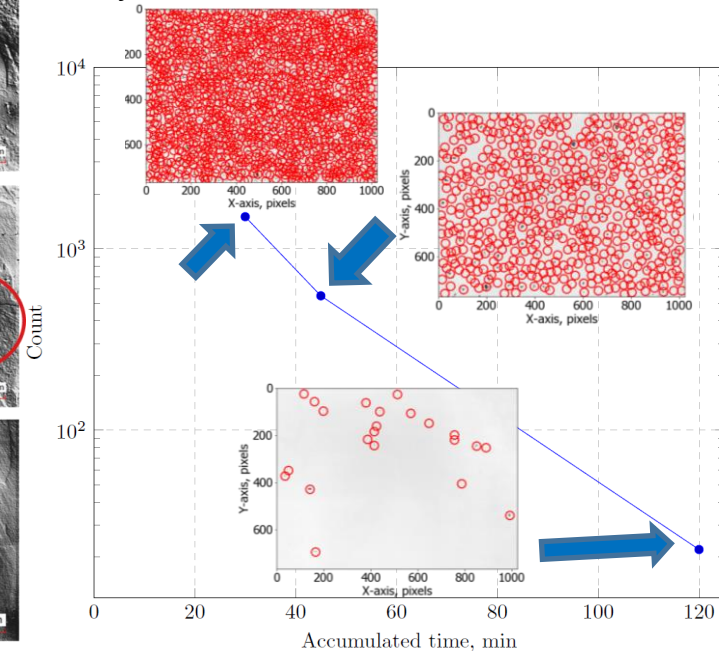


Final polishing step

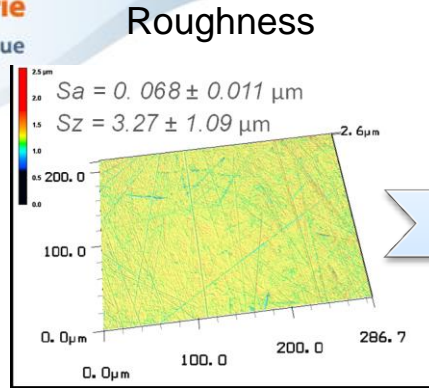
5 μm of BCP also efficient to remove the pollution, but not enough to remove the damages, also increases roughness by a factor of 5.



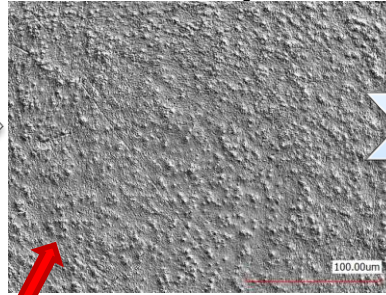
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After first step:

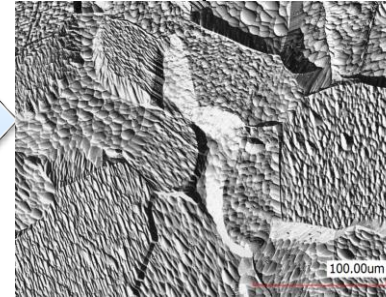


Pollution:
Revealed by CMP



Diamonds

Damages:
Revealed by BCP

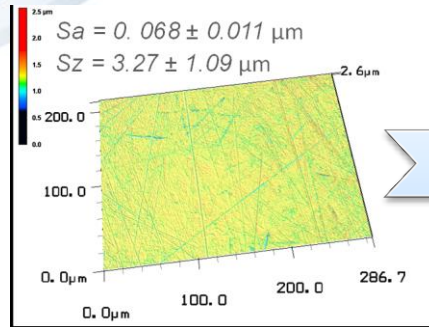


Final results:

Roughness

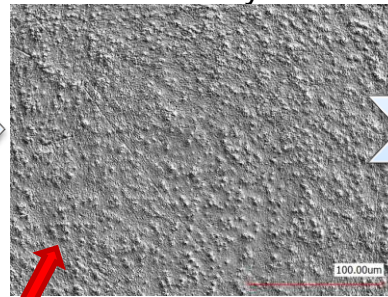
Pollution
Damages

Roughness



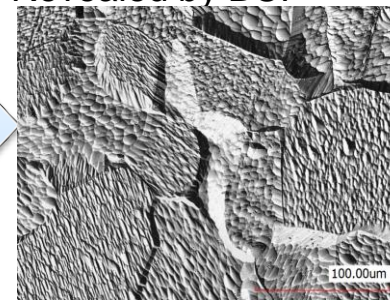
After first step:

Pollution: Revealed by CMP



Diamonds

Damages: Revealed by BCP

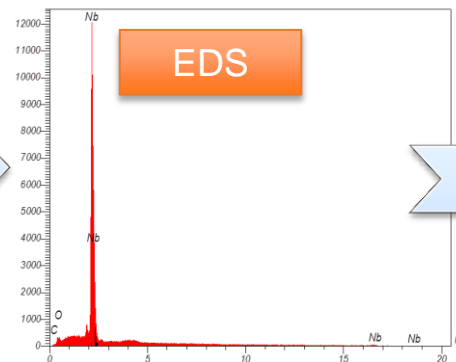
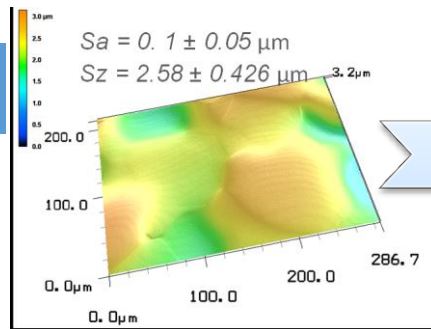


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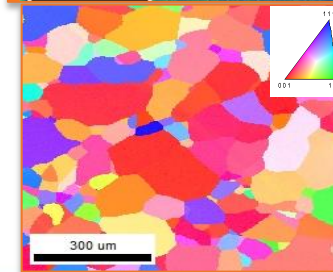
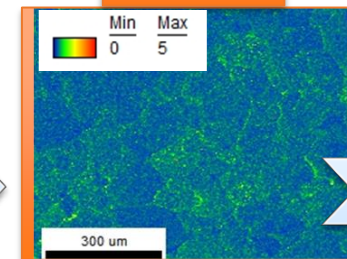
Roughness

Pollution
Damages

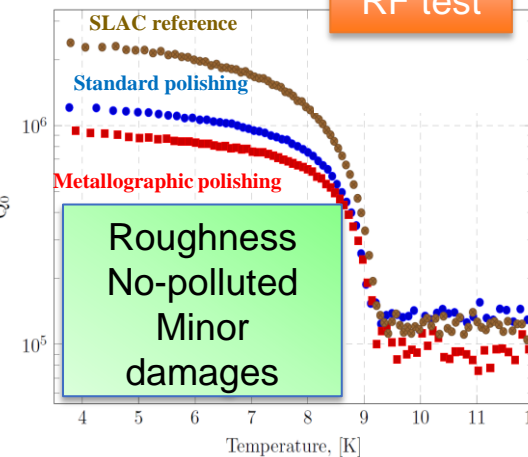
After second step:



EBSD

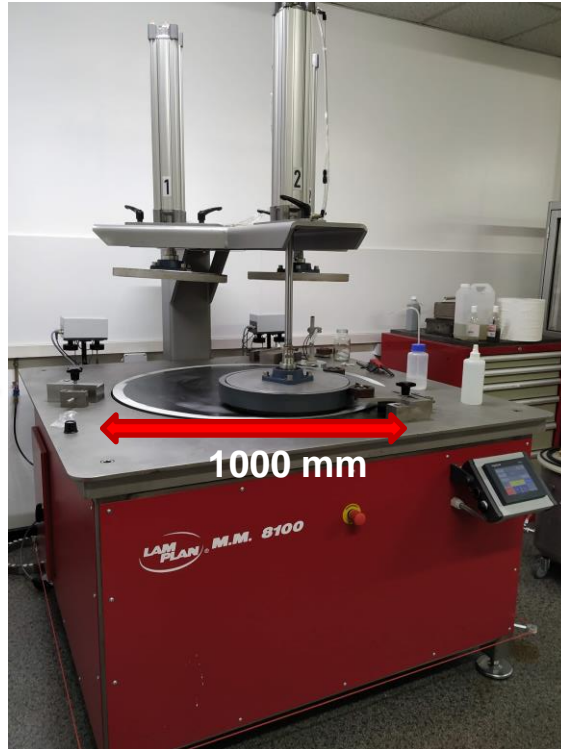
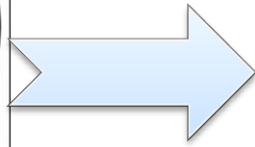
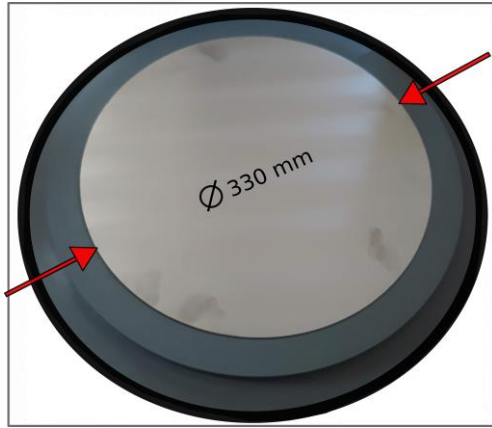


RF test



Thesis Hryhorenko Oleksandr: “Development & Optimization of Mechanical polishing for Superconducting Accelerating Cavities”

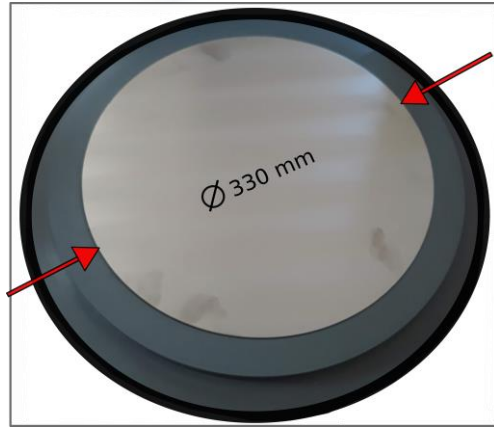
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Polishing on a lapping machine
at LAM PLAN company

<https://www.lamplan.com/fr/>





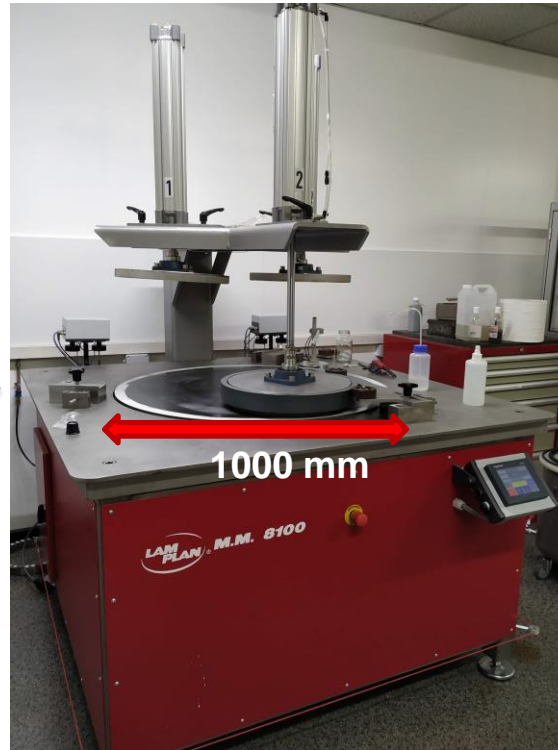
Polishing on a lapping machine
at LAM PLAN company

<https://www.lamplan.com/fr/>



Pressure: 150 g/cm²

Removal rate: 0.5 μm/min
EP – 0.3 μm/min
BCP – 0.5 μm/min

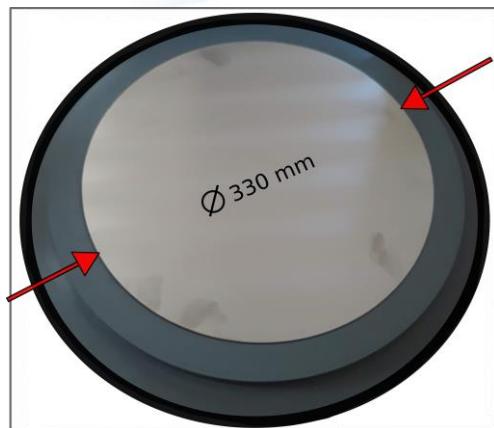


-Diamonds of 3 μm
replaced by 9 μm

**First
step:**

Pressure:
150 g/cm²





Polishing on a lapping machine
at LAM PLAN company

<https://www.lamplan.com/fr/>



-Diamonds of $3 \mu\text{m}$
replaced by $9 \mu\text{m}$

**First
step:**

Pressure:
 150 g/cm^2



- SiO_2 (pH=10)

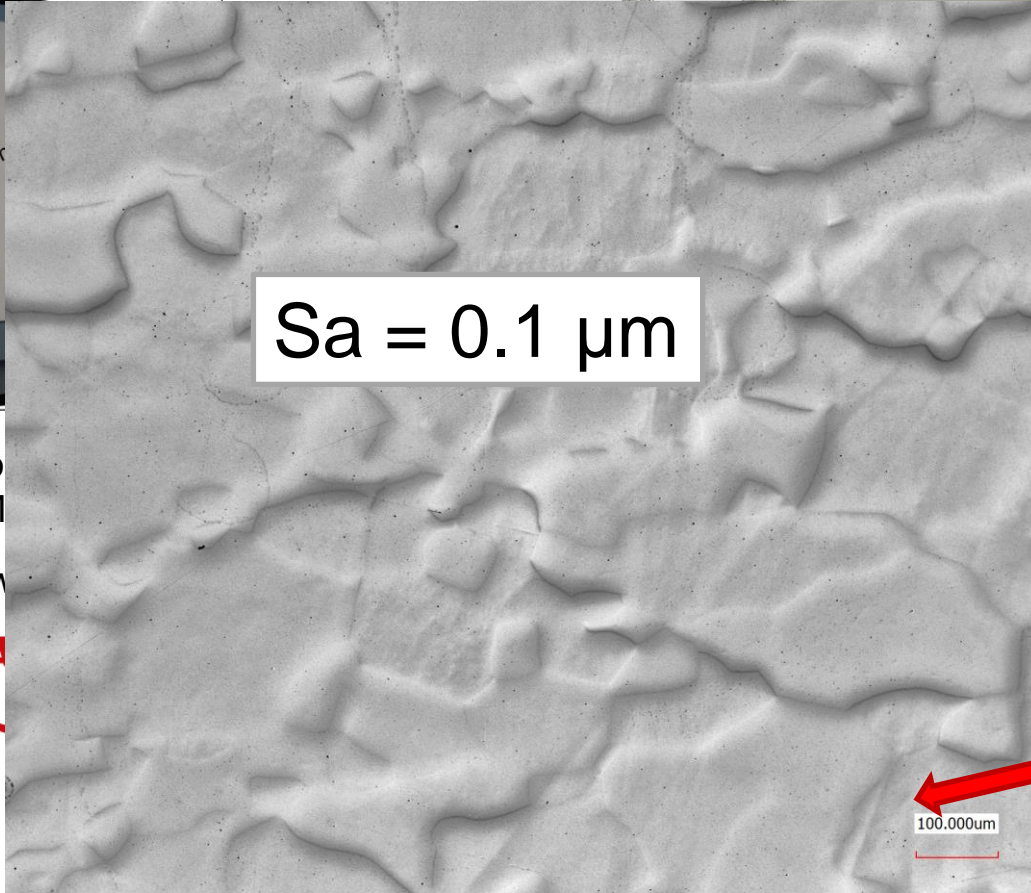
**Second
step:**

Pressure:
 150 g/cm^2





Polishing of
at LAM
<https://www>



-Diamonds of 3 μm
replaced by 9 μm

**First
step:**

Pressure:
150 g/cm²



- SiO₂ (pH=10)

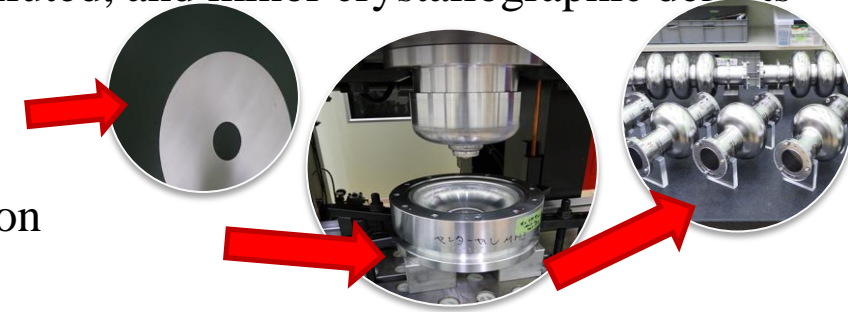
**Second
step:**

Pressure:
150 g/cm²



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- 2 steps metallographic polishing recipe has been developed compatible with SRF applications (at IJCLab)
- Surface characterizations show smooth ($20 < Sa < 100$ nm), non-polluted, and minor crystallographic defects
- Polishing procedure extended to the large sheets (at LAM PLAN)
- Forming of 1.3 GHz half-cells with the following cavity fabrication using the polished disks (KEK – FJPPL program)
- Characterization of surface pollution and crystalline damages after half cell forming (analysis of half cell cut-outs)
- Fabrication of a full single cell (KEK – FJPPL program)
- RF test measurements are mandatory to evaluate the effect of polishing treatment on Nb for SRF applications (at IJCLab & KEK).





**THANK YOU FOR YOUR
ATTENTION**

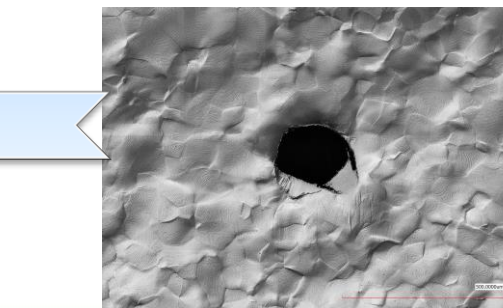
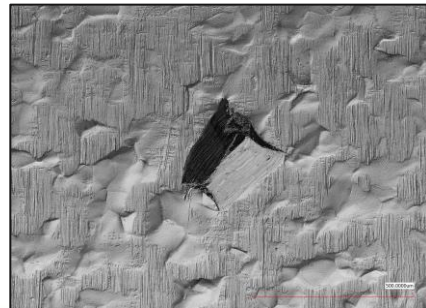
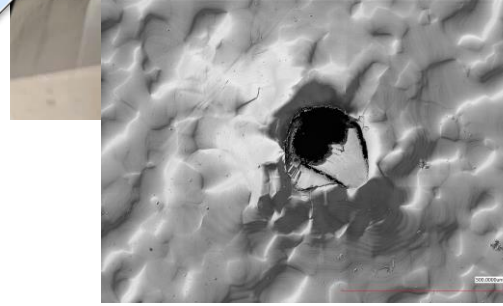
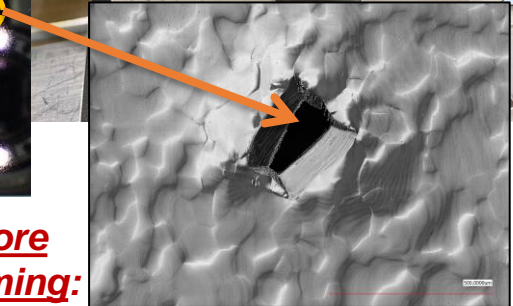
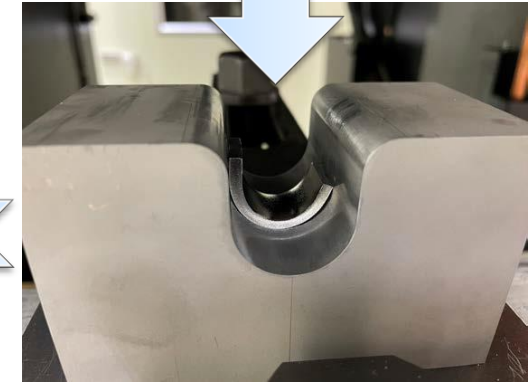
Back-up slides

Conventional

Alternative



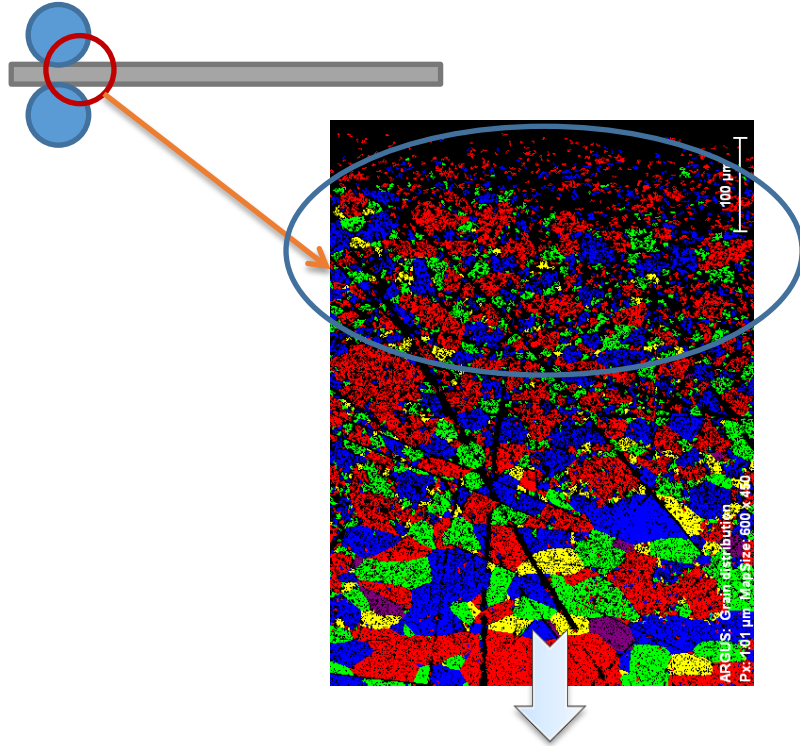
Urethane sheet ~ 30 um



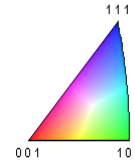
Before forming:

After forming:

Lamination

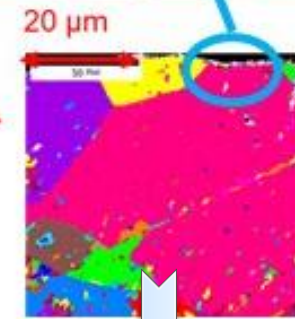
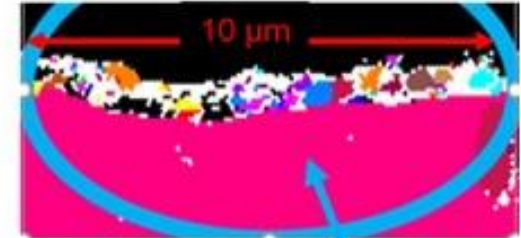


Damaged layer ~ 150 μm



Diffraction planes of electrons

Forming



Damaged layer ~ 1 μm