Cristian **Pira**

Plasma Electrolytic Polishing as a promising treatment replacement of Electropolishing in the Cu and Nb substrate preparation for SRF

SRF 2021

July 1, 2021 **THOTEV06**

Work supported by the INFN CSNV experiment TEFEN

Introduction and motivation

Is there a better option than EP?

Introduction and motivation

Is there a better option to EP?

Nb SRF cavity polishing requires hydrofluoric acid (HF)

- ➔HF is an extremely dangerous and poisoning acid
- ➔Serious workers hazard risks
- ➔Expensive procurement and disposal
- ➔Expensive infrastructure for safe handling

Introduction and motivation

Is there a better option to EP?

- Nb SRF cavity polishing requires hydrofluoric acid (HF)
- EP without HF has been explored with different levels of success

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Plasma Electrolytic Polishing (PEP)

Outline

PEP introduction

PEP - EP comparison

Results on Nb

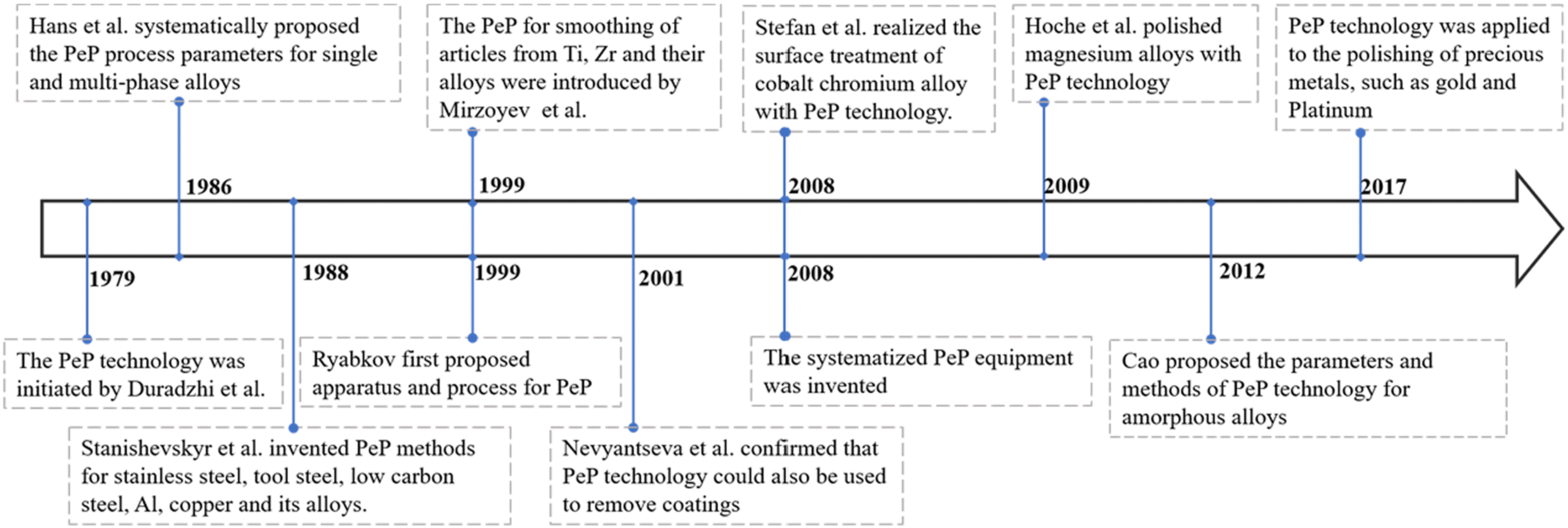
Results on Cu

Scalability

Conclusion

PEP introduction

Hystorical overview



Huang, Y. et al., Int J Adv Manuf Technol 114, 2021

PEP introduction

Applications

In principle any metal can be treated

Commonly polished, available recipes

Stainless steel alloys
Brass, Bronze
Cr, Ni, Co, Mo, W, Zn alloys

Not well studied, challenging applications

Nb
Cu and its alloys
V, Ti and its alloys

Henning Zeidler et al. / Procedia CIRP 49 (2016)

Klaus Nestler et al. / Procedia CIRP 42 (2016)

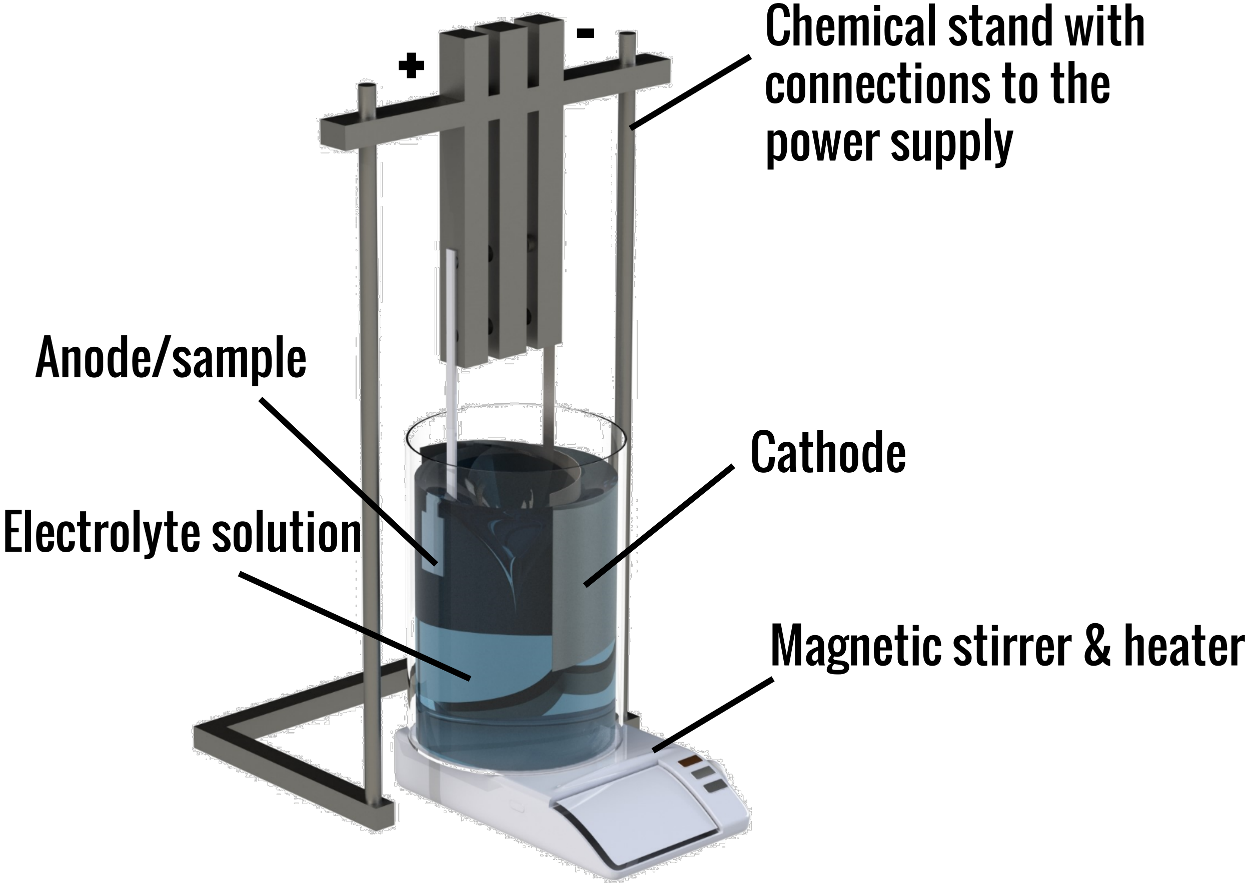
Yu. G. Aliakseyeu et al., Nauka I Tekhnika, 2018

PEP introduction

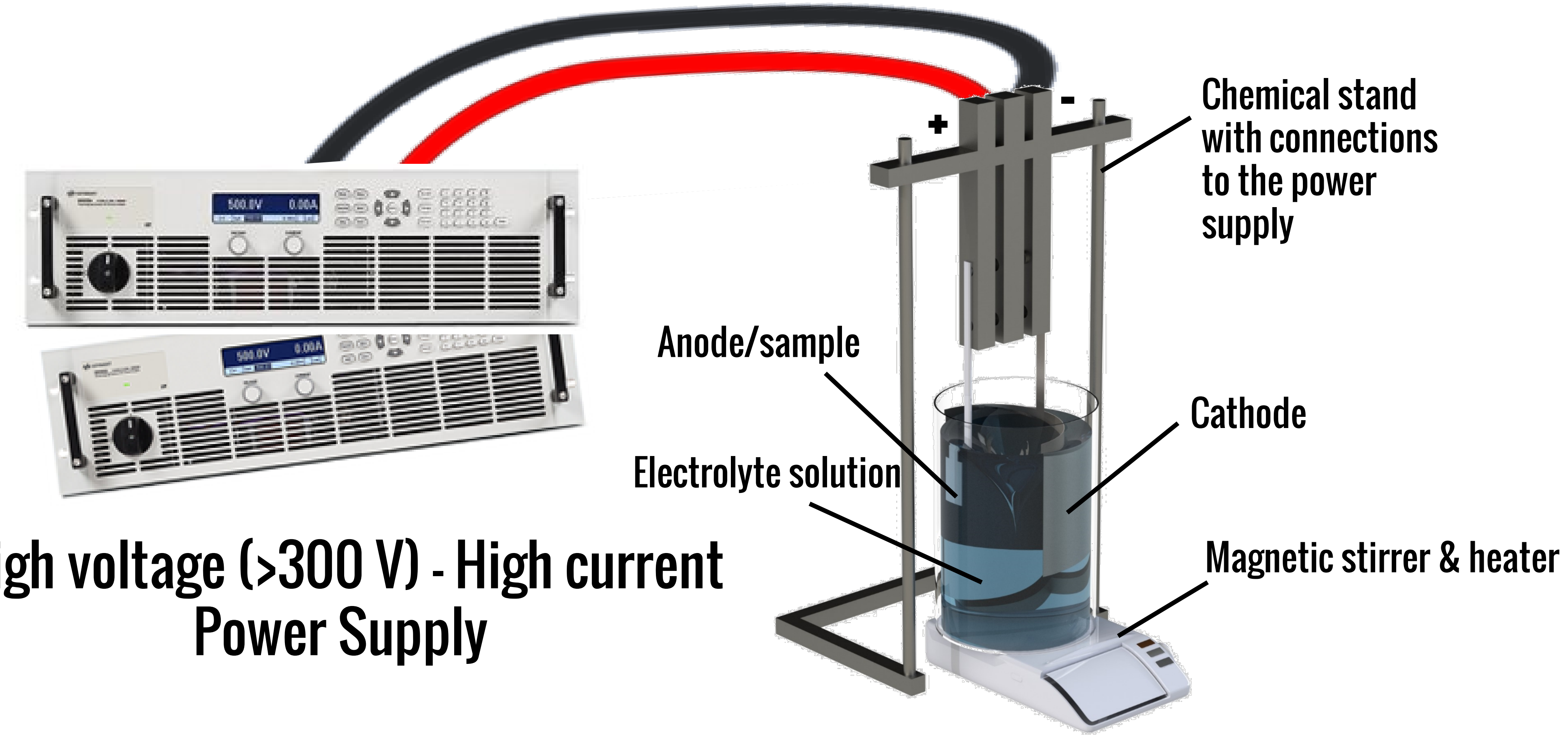
Plasma Electrolytic Polishing set-up

Identical EP set-up

Except for…



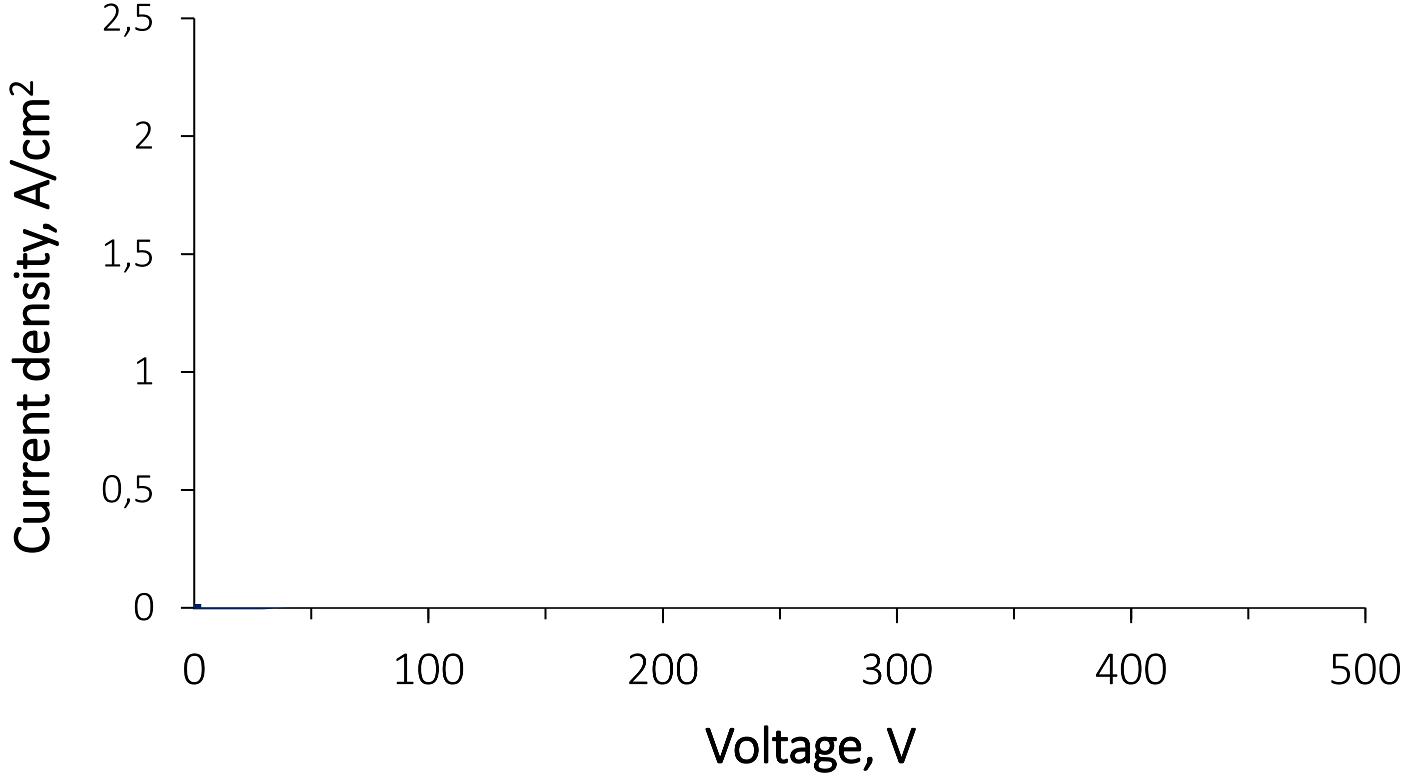
Plasma Electrolytic Polishing set-up

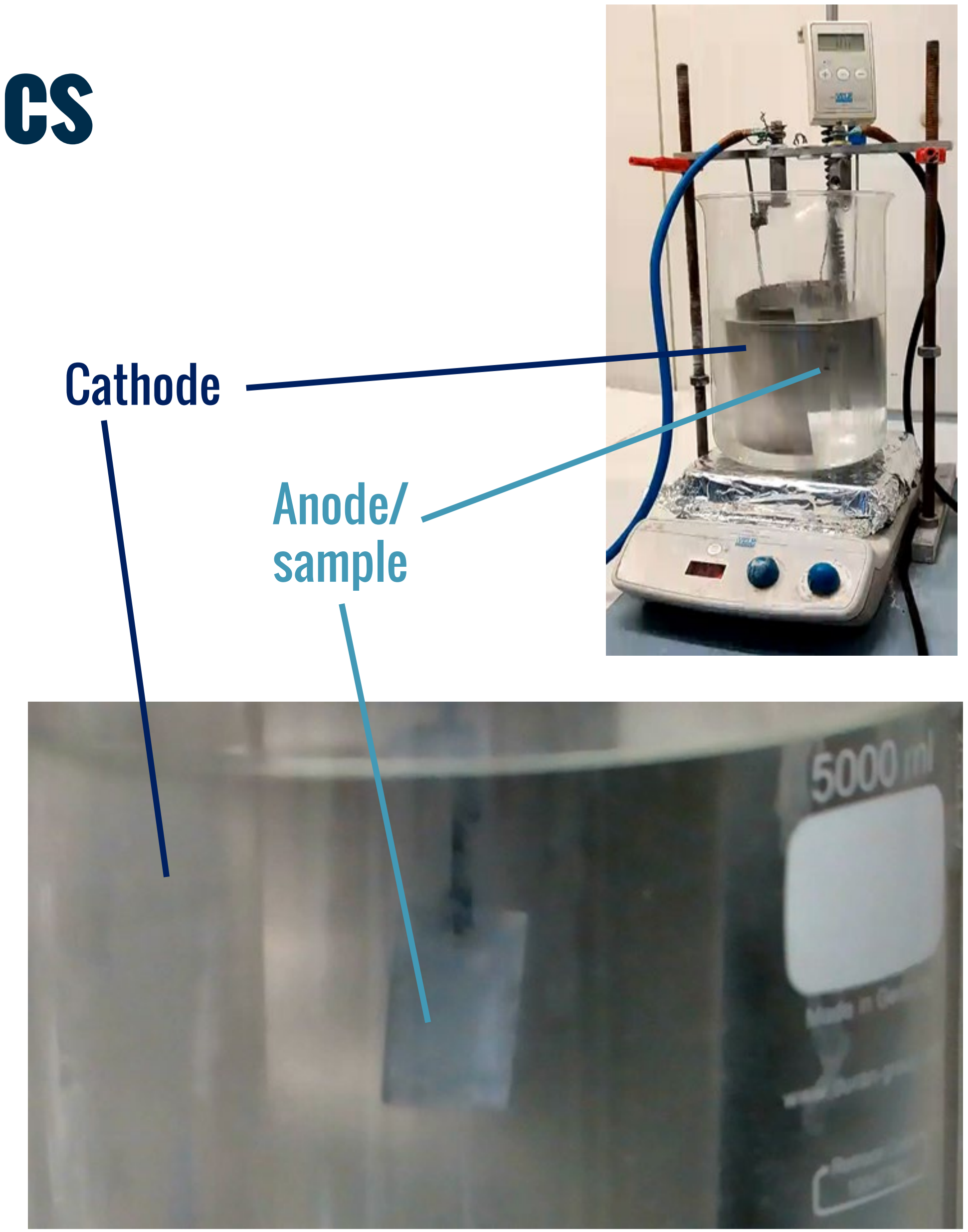


High voltage (>300 V) - High current Power Supply

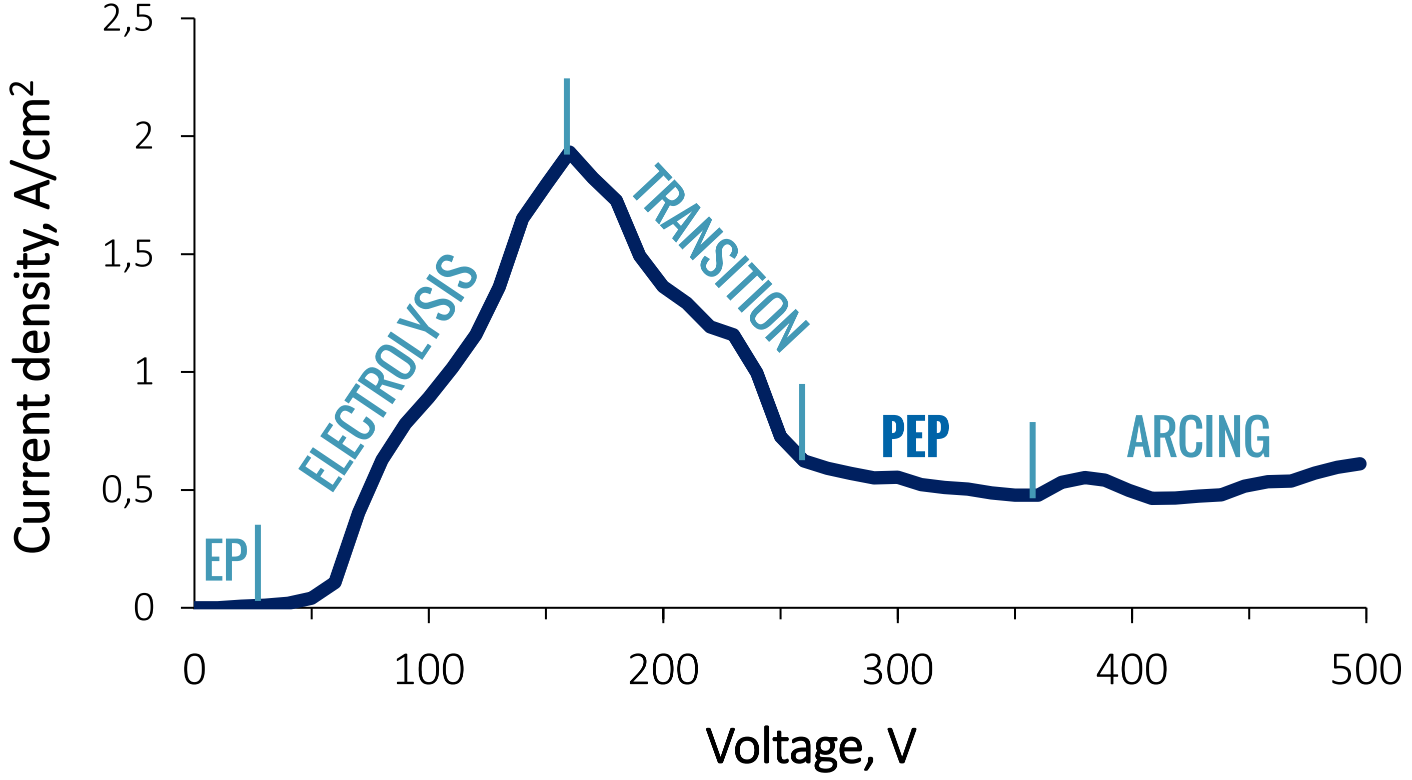
PEP introduction

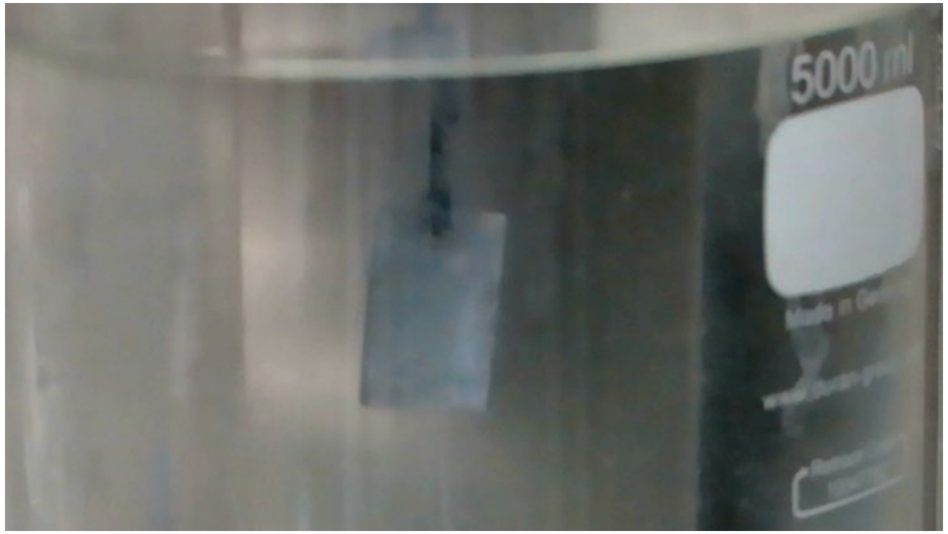
Current-voltage characteristics





Current-voltage characteristic





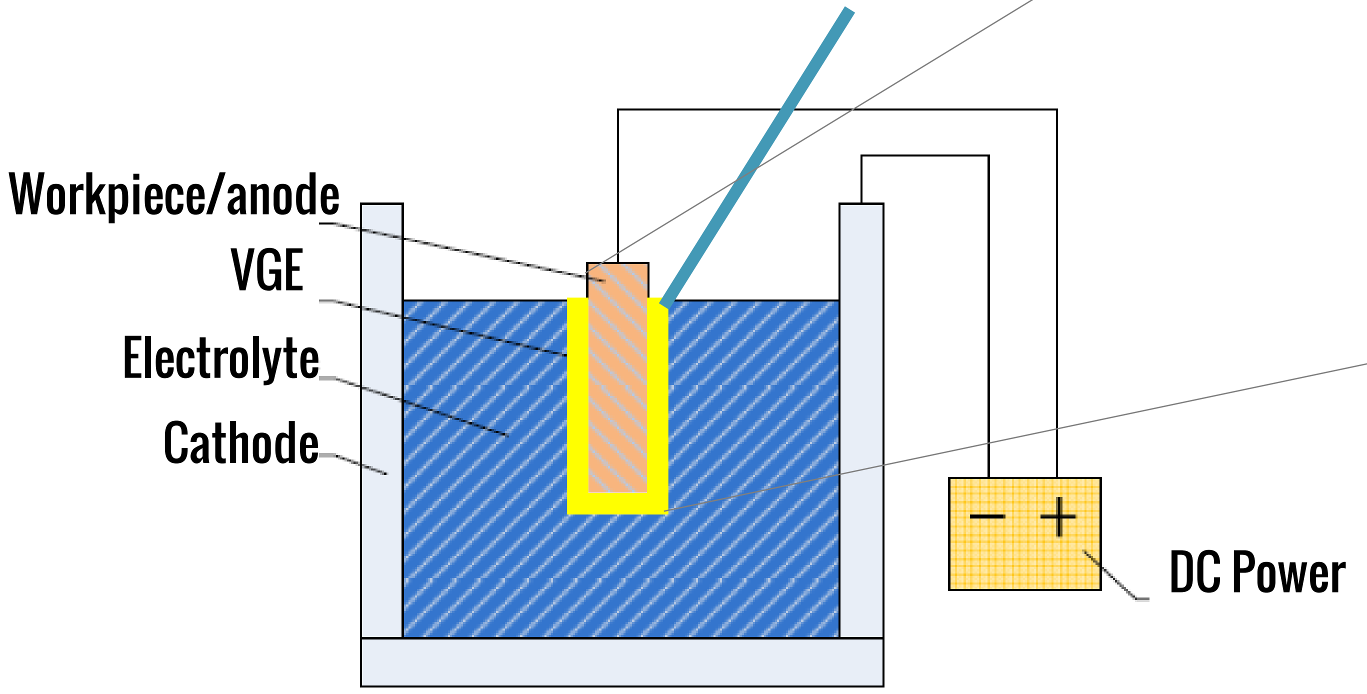
PEP Mechanism

Not fully understood yet

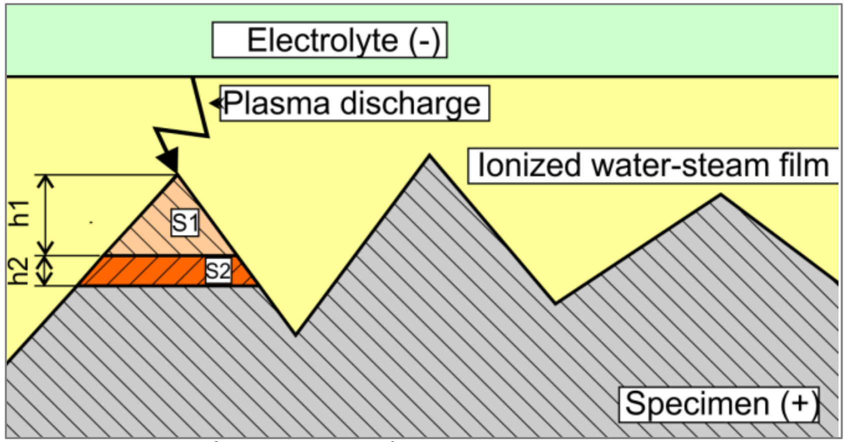
Different models proposed

PEP Mechanism

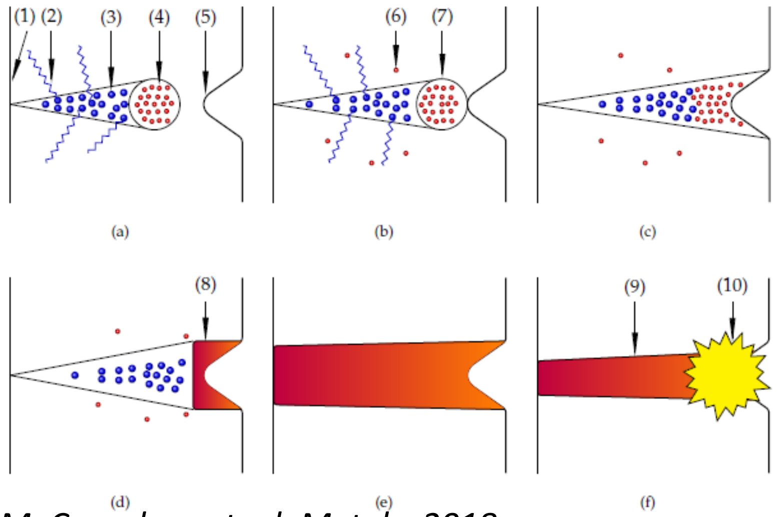
Formation of a Vapor Gas Envelope (VGE) also called Ionized Water-Steam film



J. Wang et al., AMR, 2012



Vana, D et. al, Int. J. Mod. Eng. Res. 2013



M. Cornelsen et. al, Metals, 2018

PEP - EP comparison

Risk assessment comparison

BCP 1:1:2	EP Nb 1:9	PEP Nb
Quantity of chemicals (w. %)		
79 %	93 %	~5%
Acids, harsh chemicals		
3 Acids: $HF:HNO_3:H_3PO_4$	2 Acids: $HF:H_2SO_4$	No acids! Only salts

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Risk $\propto$ Probability $\propto$ Process Time (Time to remove 200 μm)		
~4 h	~6-10 h	~1 h

PEP - EP comparison

Comparison EP VS PEP

Parameters	Conventional EP	Plasma Electrolytic Polishing
Pre-treatment	In some cases mechanical pre-treatment	Not required
Roughness	0.2-0.1 μm	Achievable up to 0.01 μm
Cathode	Should follow the shape of the piece	Shape is not important
Electrolytes	Harsh electrolytes, concentrated acids	Greener, diluted water based salts (1-10%)
Time to remove 100 μm	3 h - 7 h	15-30 min
Voltage	2-60 V	260-360 V
Temperature	Room temperature	> 65 °C
Current density	0,01-0,04 A/cm^2	0,2-0,6 A/cm^2

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Results on Nb

Results on Cu

Trials and optimization

Almost 30 different solutions tested

No satisfying results with solutions in **literature** for Nb and Cu

1 original Solution for Nb and 2 for Cu have been optimized
(currently under patenting evaluation)

Parameters studied:
voltage, conductivity, working temperature, cathode shape

Processes parameters comparison

Process / parameters	BCP (1:1:2)	EP (1:9)
Solution composition	$HF:HNO_3:H_3PO_4$	$HF:H_2SO_4$
Voltage	-	18 V
Current density	-	0.025 A/cm^2
Power density	-	0.45 W/cm^2
Removing rate	1 µm/min (15°C)	0.3 µm/min (30°C)

Results on Nb

Processes parameters comparison

Process / parameters	BCP (1:1:2)	EP (1:9)	PEP
Solution composition	$HF:HNO_3:H_3PO_4$	$HF:H_2SO_4$	**Diluted salts**
Voltage	-	18 V	**300 V**
Current density	-	0.025 A/cm^2	**0.4-0.6 A/cm^2**
Power density	-	0.45 W/cm^2	**~150 W/cm^2**
Removing rate	1 µm/min (15°C)	0.3 µm/min (30°C)	**3.5 µm/min (78°C)**

PEP

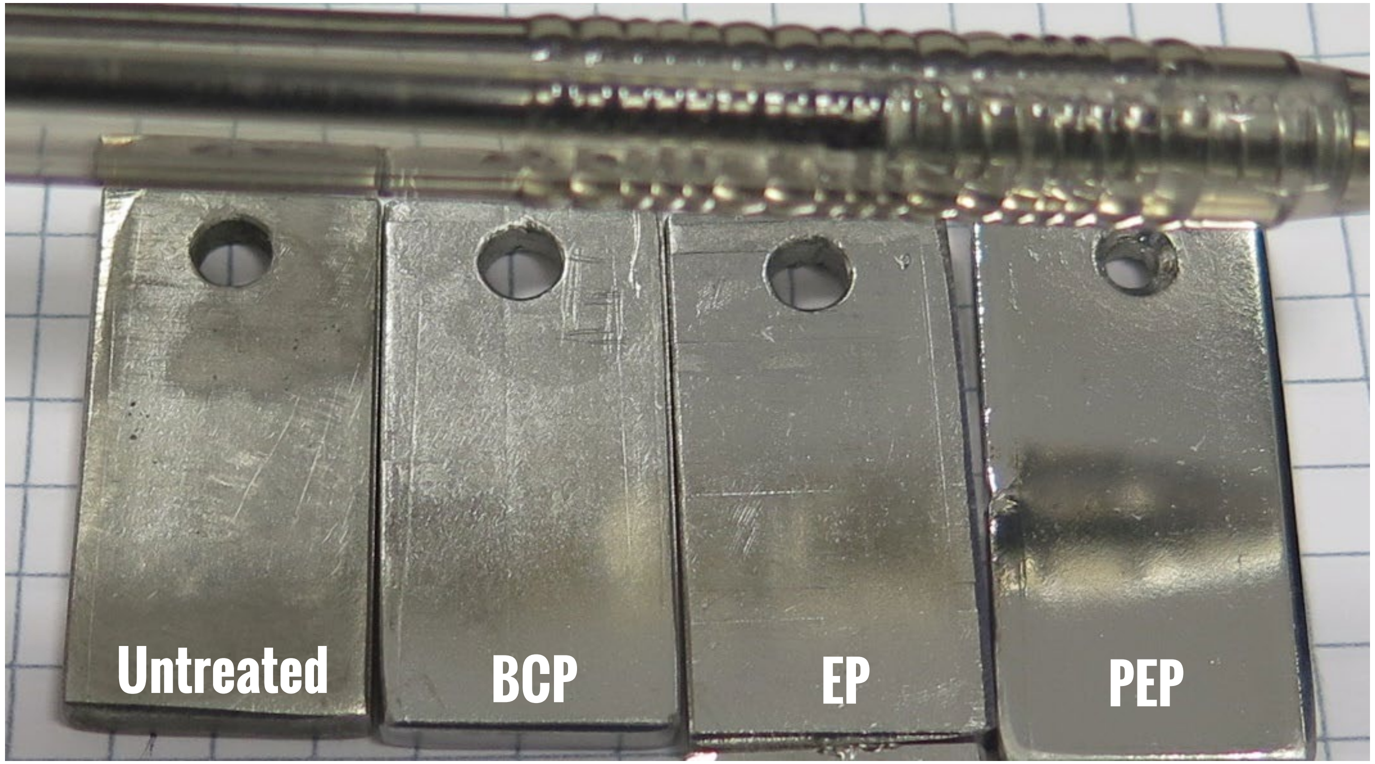
3.5 times faster than BCP

PEP

>10 times faster than EP

Fast polishing test

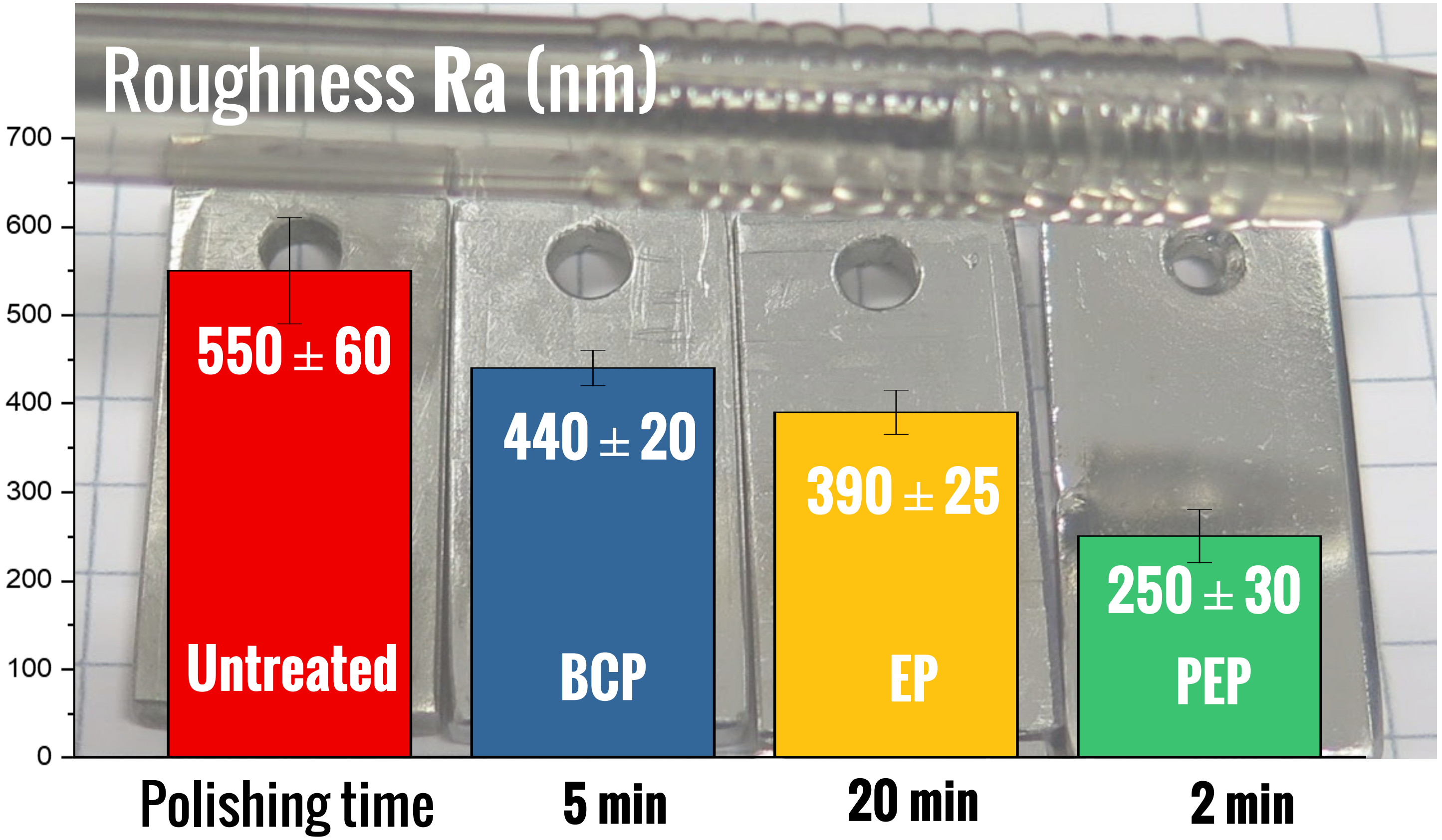
$6.5 \pm 0.5\ \mu m$ removed



Results on Nb

Fast polishing test

6.5 ± 0.5 μm removed



Results on Nb

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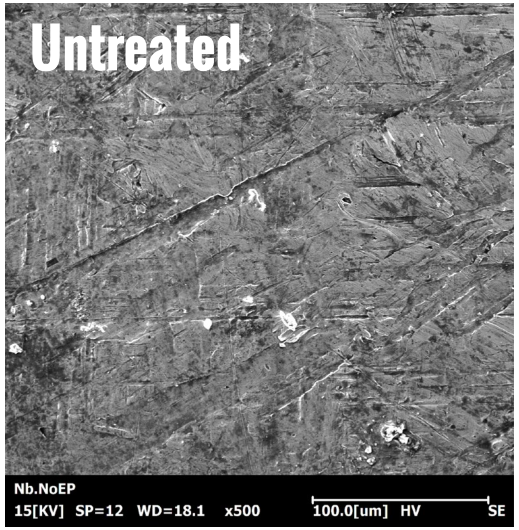

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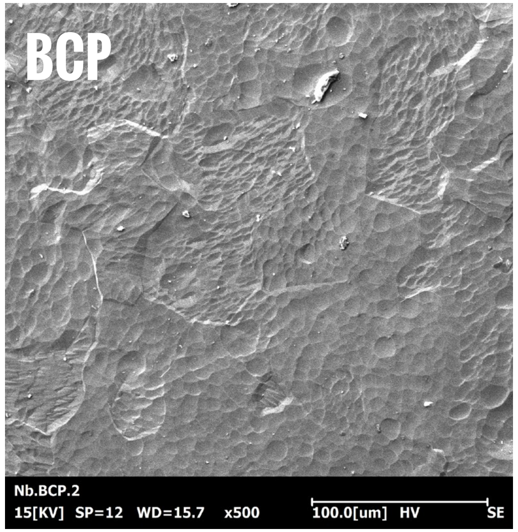
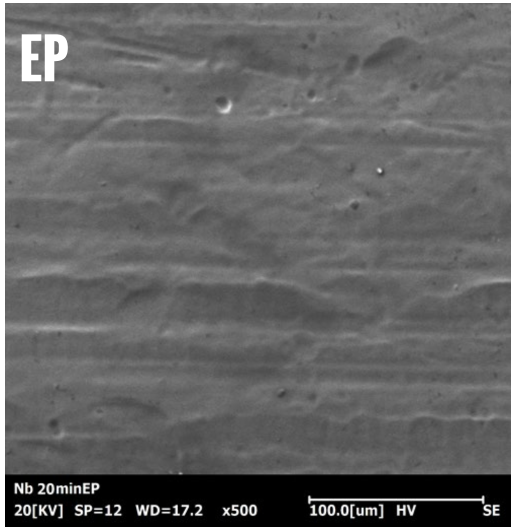
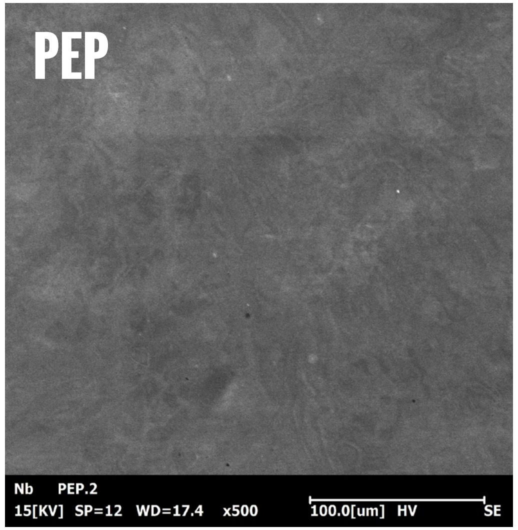
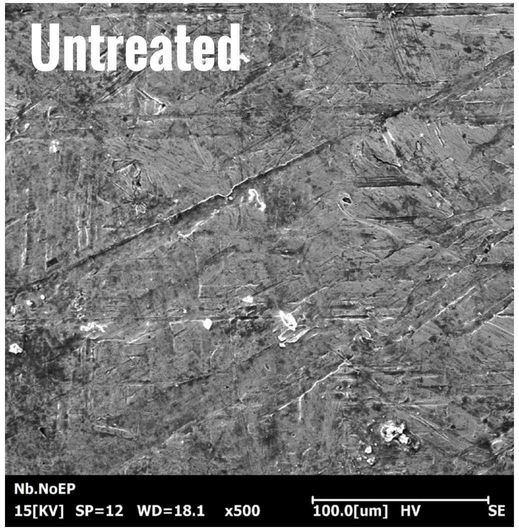




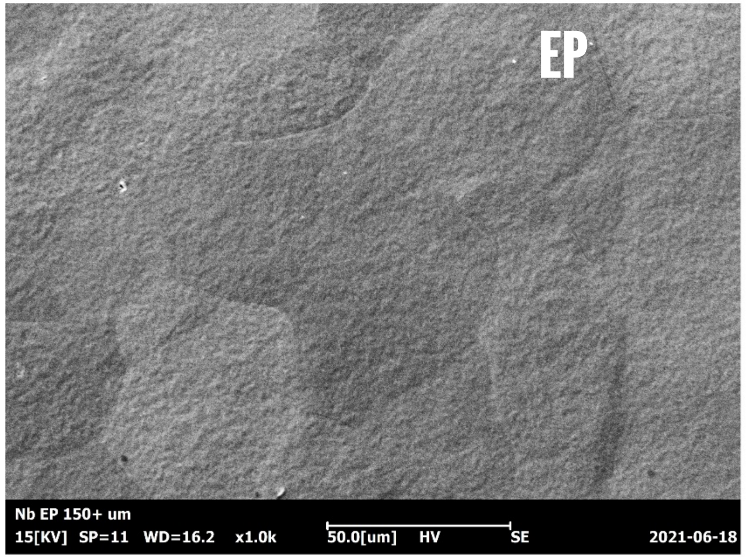


Polishing time | 5 min | 20 min | 2 min

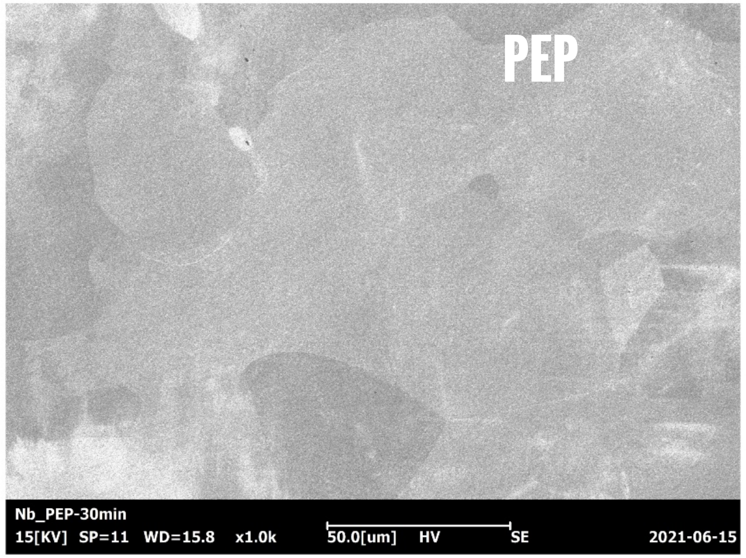
Long polishing test



~ 150 μm removed

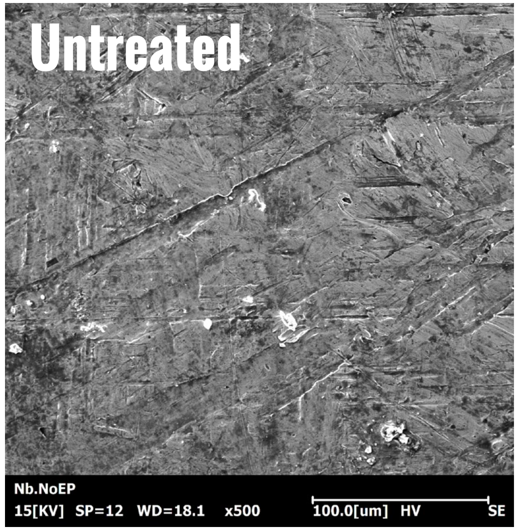


150μm ~ 5 h



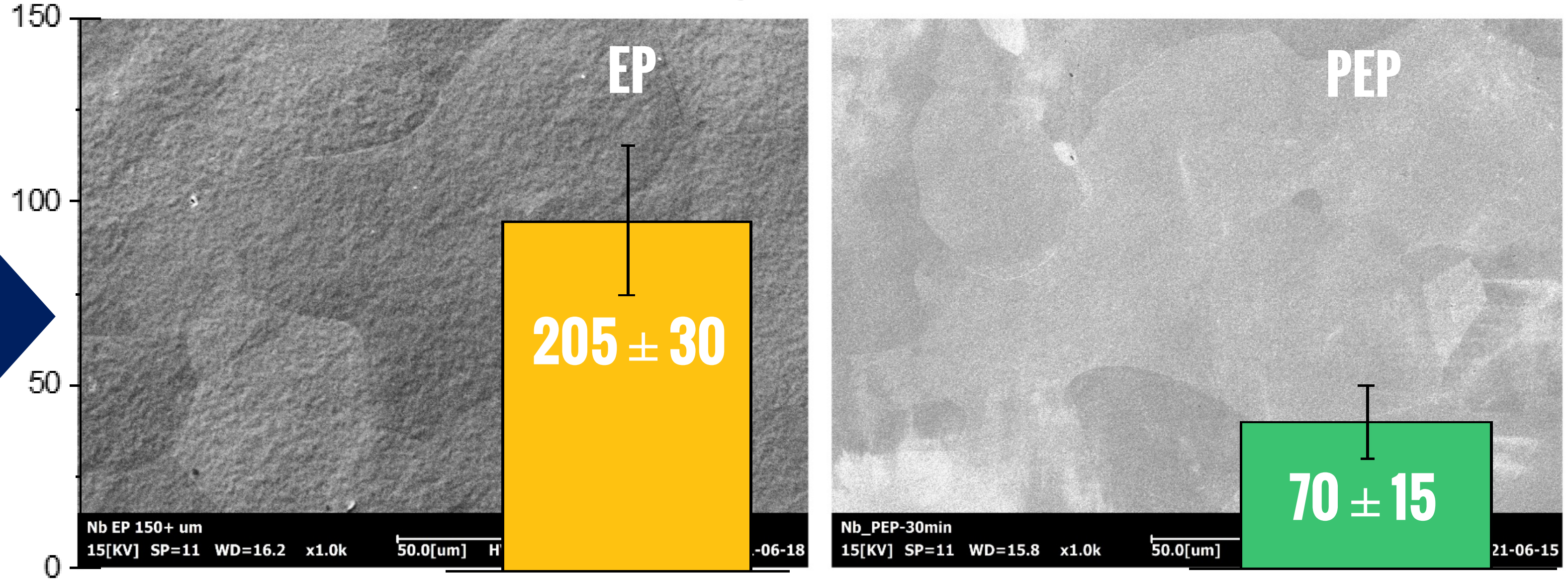
150μm ~ 40 min

Long polishing test



~ 150 μm removed

Roughness **Ra** (nm)



150μm ~ 5 h

150μm ~ 40 min

Results on Cu

Processes parameters comparison

Process / parameters	SUBU5	EP (3:2)
Solution composition	Sulfamic acid 5 g/l; NH_4-citrate 1 g/l Butanol 50 ml/l; H_2O_2 50 ml/l	85 % H_3PO_4 : n-Butanol
Voltage	-	2-6 V
Current density	-	0,01 - 0,03 A/cm^2
Power draw	-	0,12 W/cm^2
Removing rate	1,5 μm/min (70 $\pm$ 2°C)	0,15-0,5 μm/min (25°C)

Results on Cu

Processes parameters comparison

Process / parameters	SUBU5	EP (3:2)	PEP
Solution composition	Sulfamic acid 5 g/l; NH_4-citrate 1 g/l Butanol 50 ml/l; H_2O_2 50 ml/l	85 % H_3PO_4 : n-Butanol	Diluted salts
Voltage	-	2-6 V	300 V
Current density	-	0,01 - 0,03 A/cm²	0,35-0,45 A/cm2
Power draw	-	0,12 W/cm²	135 W/cm2
Removing rate	1,5 μm/min (70 ± 2 °C)	0,15-0,5 μm/min (25 °C)	3-10 μm/min (70-80 °C)

PEP

~ 3 times faster than SUBU5

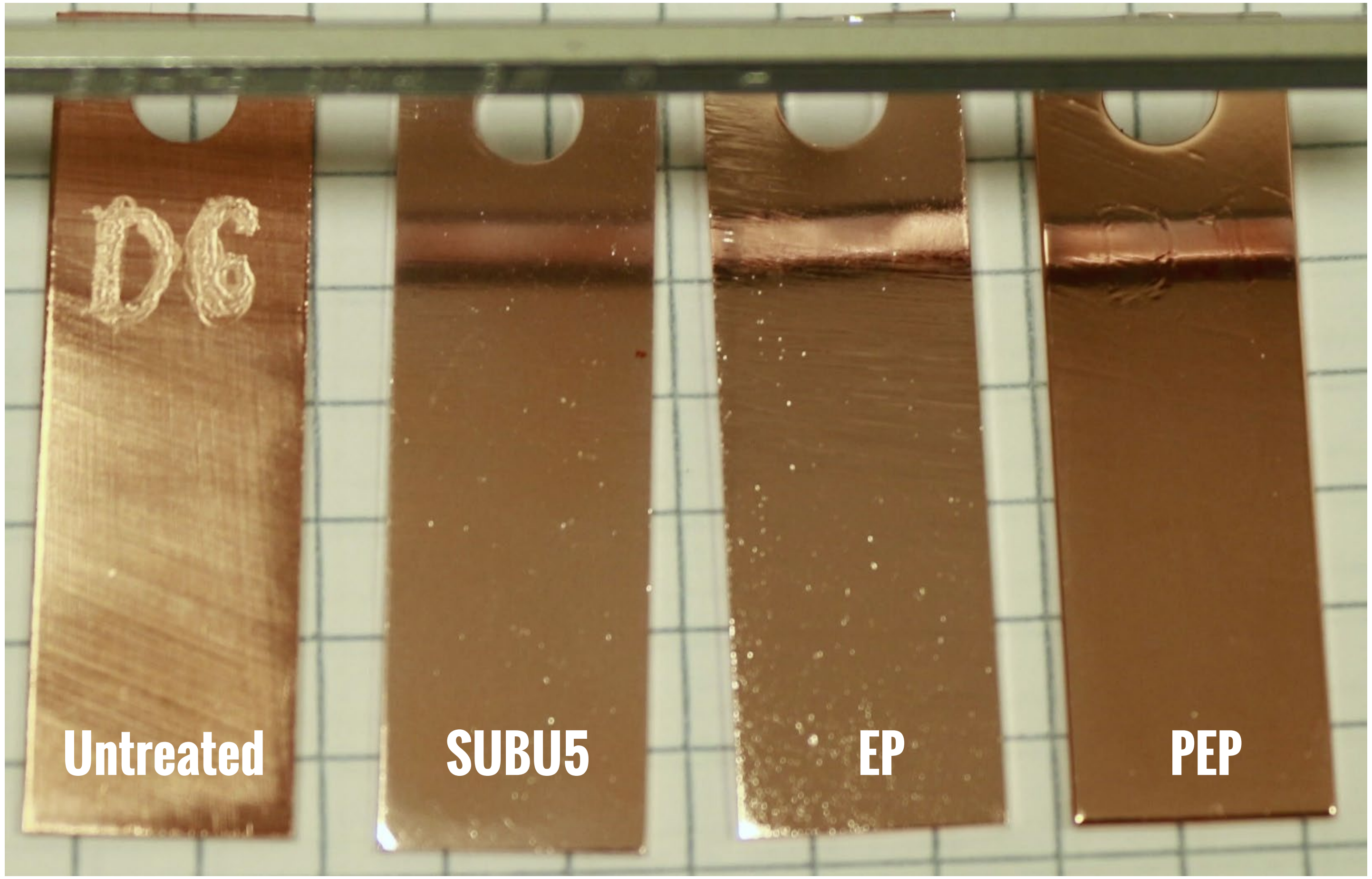
PEP

10-20 times faster than EP

Results on Cu

Fast polishing test

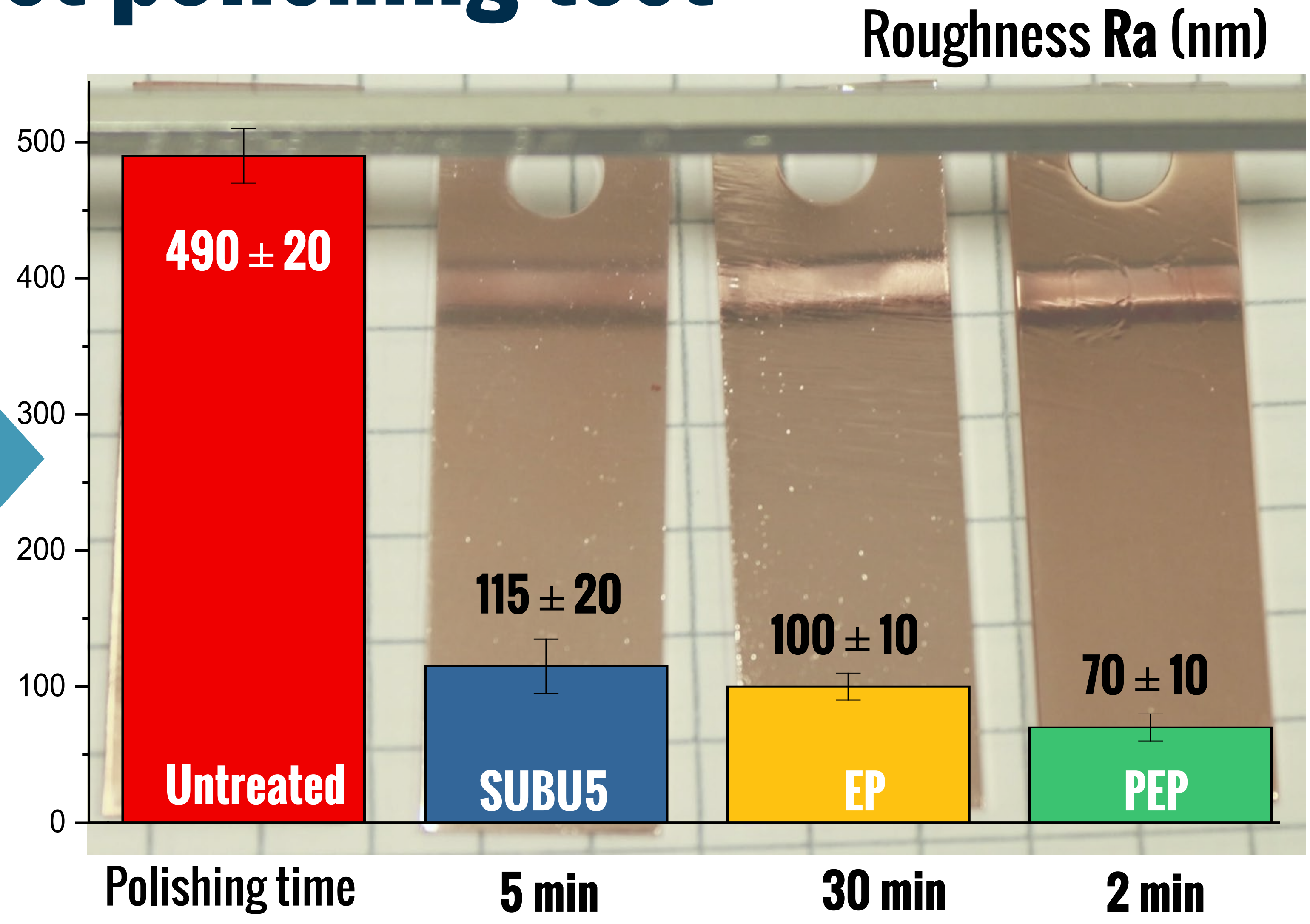
$8 \pm 0.5\ \mu m$ removed



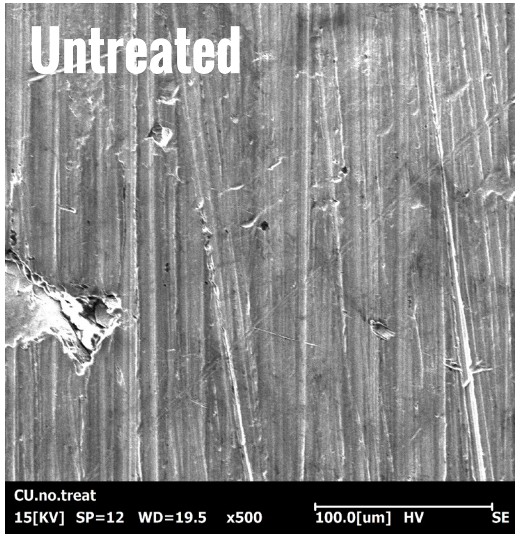
Results on Cu

Fast polishing test

8 ± 0.5 μm removed



Fast polishing test



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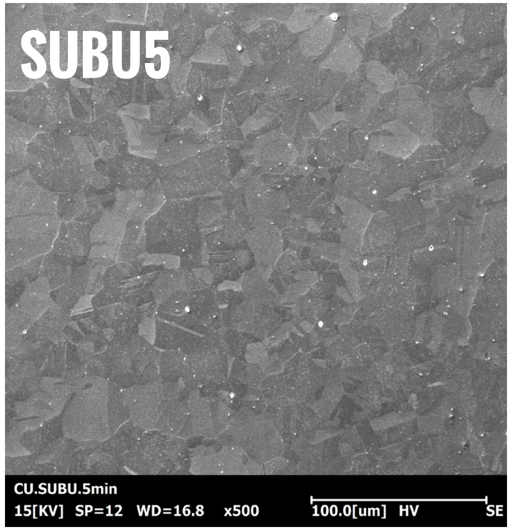
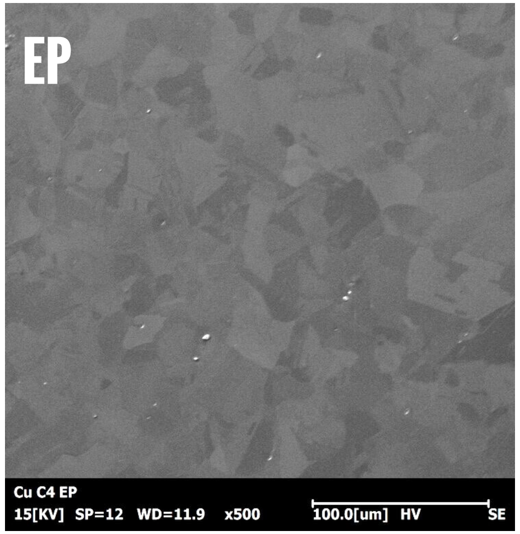
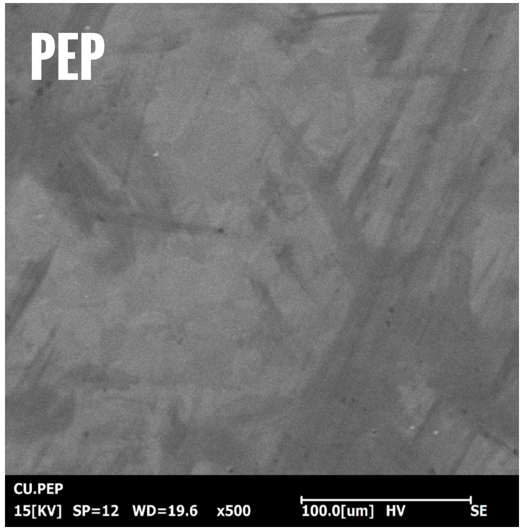
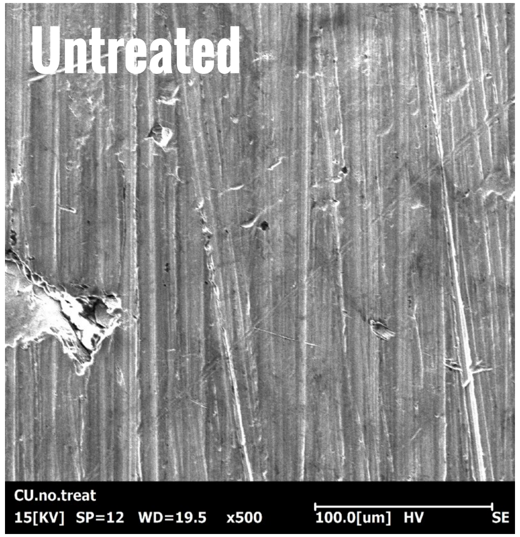






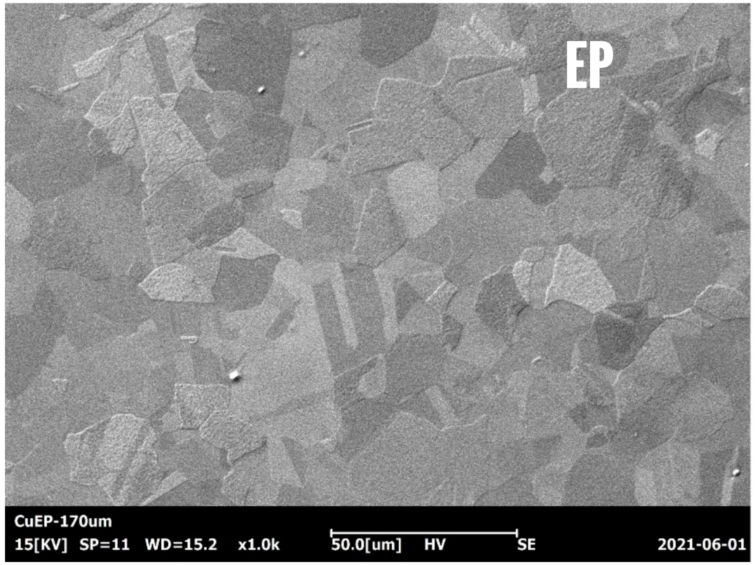
Polishing time	5 min	30 min	2 min

Long polishing test

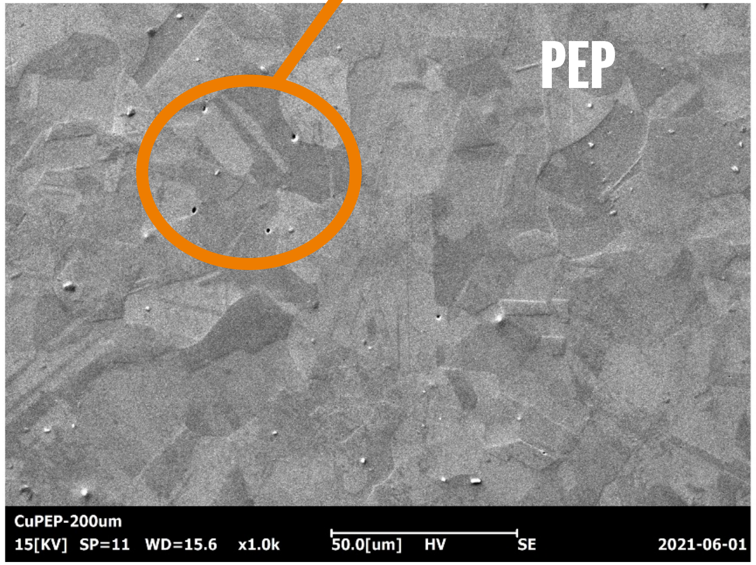


>170 µm removed

Pitting on a small portion

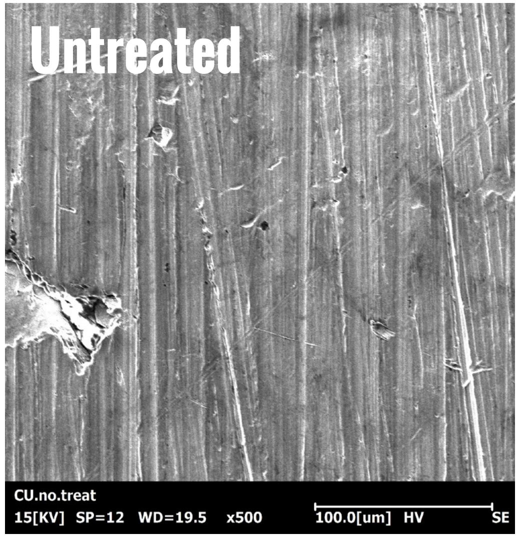


170µm ~ 5 h



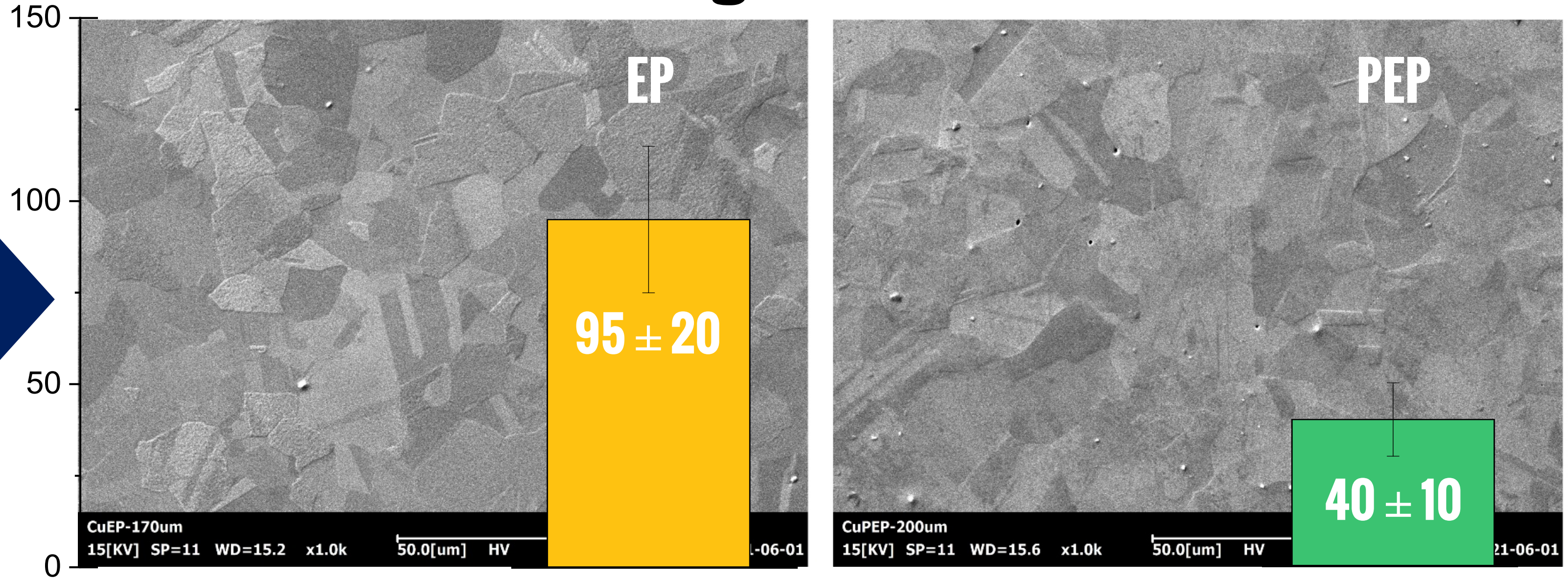
200µm ~ 40 min

Long polishing test



>170 μm removed

Roughness Ra (nm)



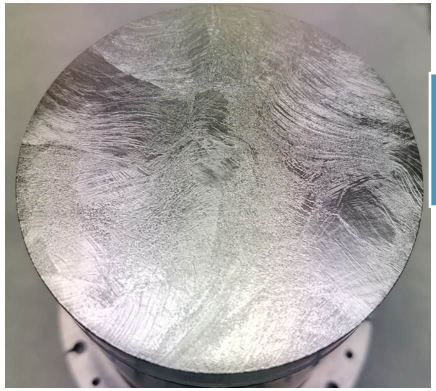
170μm ~ 5 h

200μm ~ 40 min

Real samples treatment

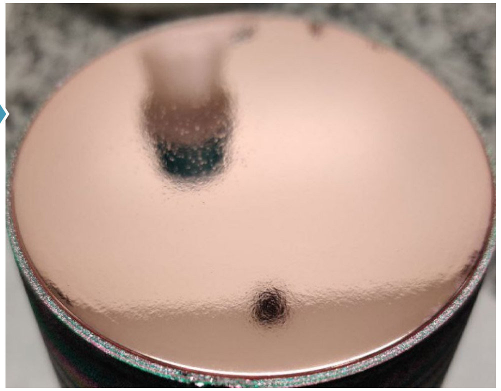
PEP tested on HZB Quadrupole Resonators

(not tested yet)



100 μm
60 min

30 μm
4 min



Eduard Chyhyrynets

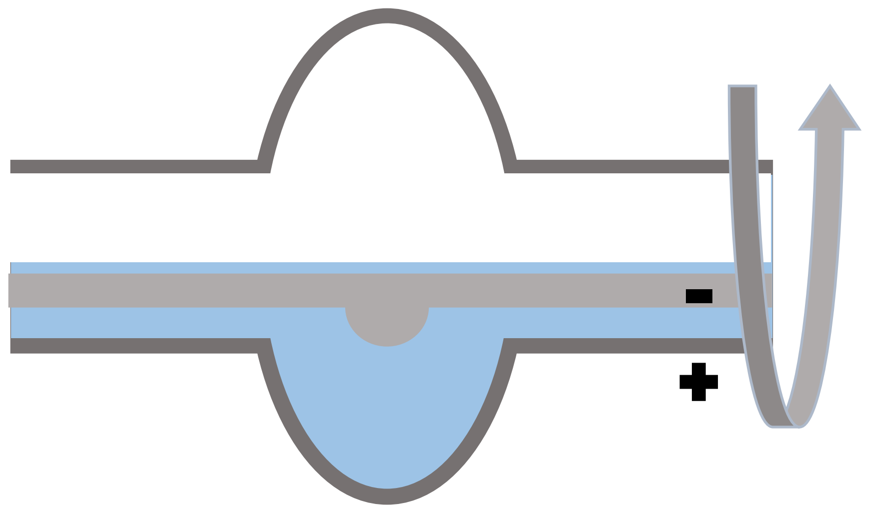
SUPTEV002 **Application of Plasma Electrolytic Polishing onto SRF Substrates**

SRF 2021
Student Poster Session

Scalability

Scalability to large areas is very challenging:

- High power demand
- Large amounts of gases produced



Single cell 1.3 GHz in horizontal configuration

~ 20 kW in PEP regime

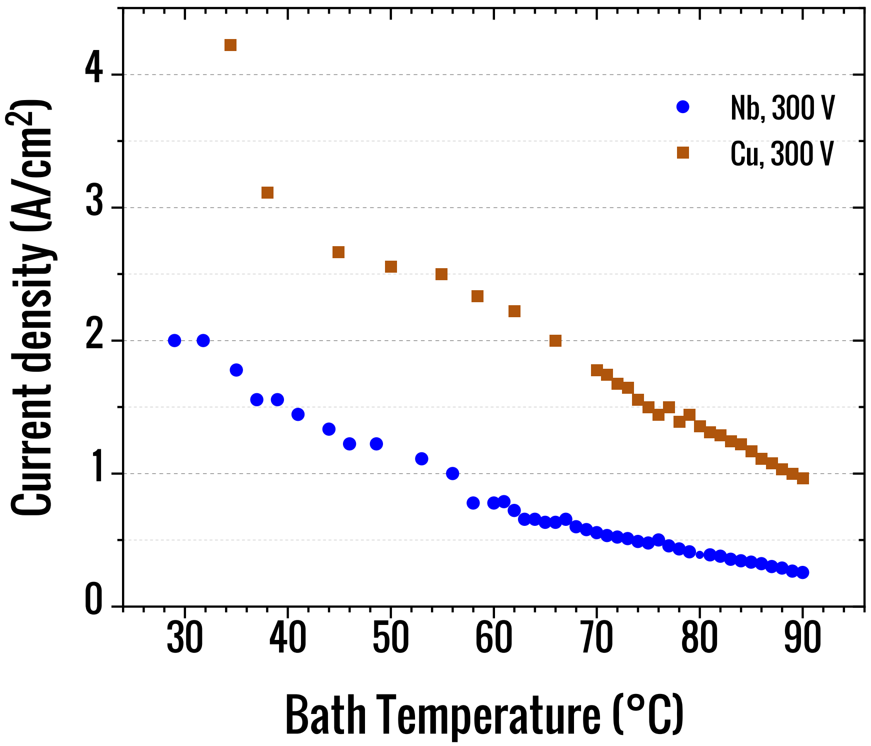
Scalability

Scalability to large areas is very challenging:

- High power demand
- Large amounts of gases produced

Possible solutions to explore:

- Pulsed PEP
- Increase bath temperature
- Lowering voltage after triggering the plasma



Scalability

Scalability to large areas is very challenging:

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Possible solutions to explore:

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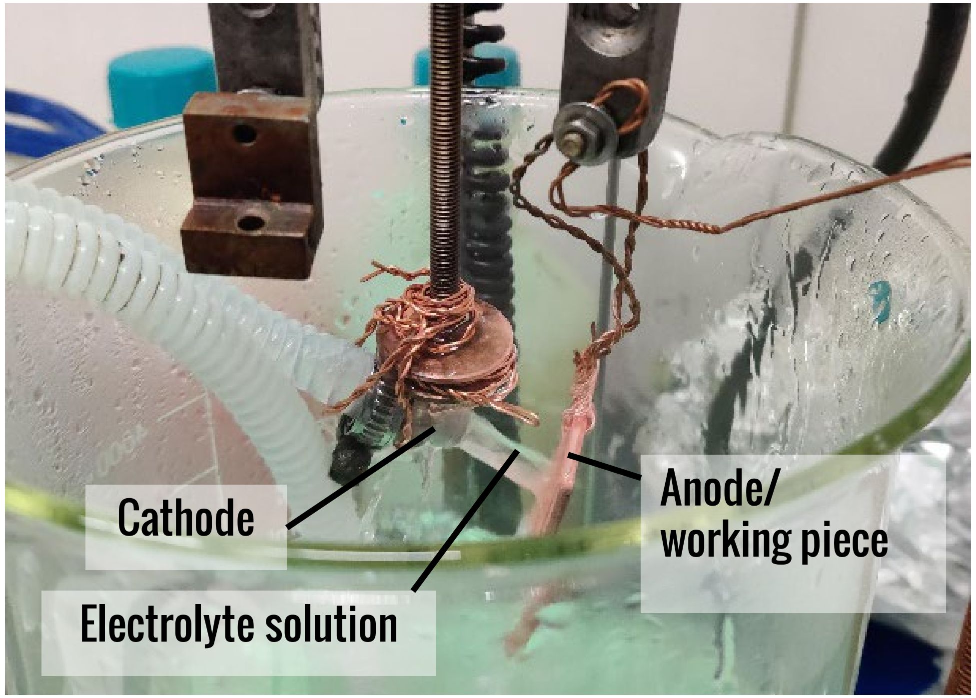
- Increase bath temperature
- Lowering voltage after triggering the plasma
- **Jet-polishing**

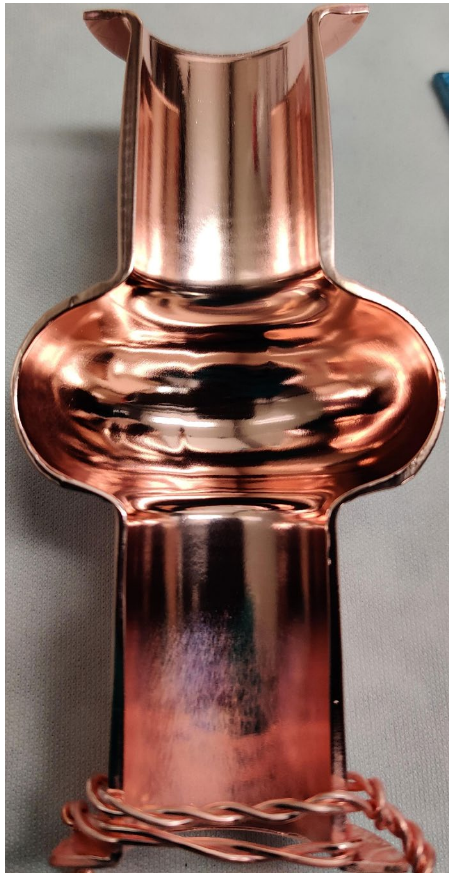
Jet-Plasma Electrolytic Polishing

Local polishing for large area treatments



Jet-PEP set-up

Half 6 GHz Untreated



Half 6 GHz JET-PEP polished

Conclusions

Polishing via PEP of Nb and Cu possible

- Smoother surface than EP
- Safer and more eco-friendly than EP
- 10 times faster than EP
- Polishing of large areas challenging → Jet-PEP possible

cristian.pira@lnl.infn.it

Special thanks to
Roberta Caforio, Vanessa Garcia, Fabrizio Stivanello
and **Eduard Chyhyrynets**

Thank you for the attention!