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Surface preparation of samples prior to thin film deposition

ARIES 1st Annual Meeting, 22-25 May 2018, Riga

Enzo Palmieri

03/11/1962 - 16/03/2018

Outline

- Introduction to Surface treatments in SRF cavities
- ARIES surface polishing treatments set up
- Surface Characterizations

Introduction

Importance of Surface Treatments in SRF

- Used to remove the surface damaged layer, due to cavity fabrication methods
- To get high gradient $E_{acc} > 30$ MV/m → surface roughness < 2 μm

Saito K., Proceedings of the 2003 Workshop on RF Superconductivity, Paris, France

Importance of Surface Treatments in SRF

- On Nb-Cu cavities the thin film is not chemical treated
- Different polishing treatments of Cu are used
- Morphology of Cu surface is replicated by the Nb film
- Direct correlation between Cu surface preparation and Nb films SC properties

Importance of Surface Treatments in SRF

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- Different polishing treatments of Cu are used
- Morphology of Cu surface is replicated by the Nb film
- **Direct correlation between Cu surface preparation and Nb films SC properties**

Cu Treatments in Accelerator Cavities

- **LEP** → Chemical Polishing (SUBU5)

Benvenuti et al., Proceedings of the 1987 Workshop on RF Superconductivity, Argonne National Laboratories, Illinois, USA

- **ALPI** → Tumbling + ElectroPolishing + SUBU5

Palmieri et al., Proceedings of the 1997 Workshop on RF Superconductivity, Abano Terme (Padova), Italy

- **LHC** → ElectroPolishing + SUBU5

Calatroni et al., Proceedings of the 1999 Workshop on RF Superconductivity, La Fonda Hotel, Santa Fe, New Mexico, USA

- **HIE Isolde** → SUBU5

Venturini et al., Proceedings of the 2003 Workshop on RF Superconductivity, Paris, France

ARIES Surface polishing (WP 15.2)

4 Treatments investigated

1. **SUBU** (25 samples at CERN + 5 samples at INFN)
2. **Electropolishing (EP)** (8 samples at INFN)
3. **EP+ SUBU** (4 samples at INFN)
4. **Tumbling** (6 samples at INFN)

All treatments remove 40 microns except the tumbling

Copper Samples

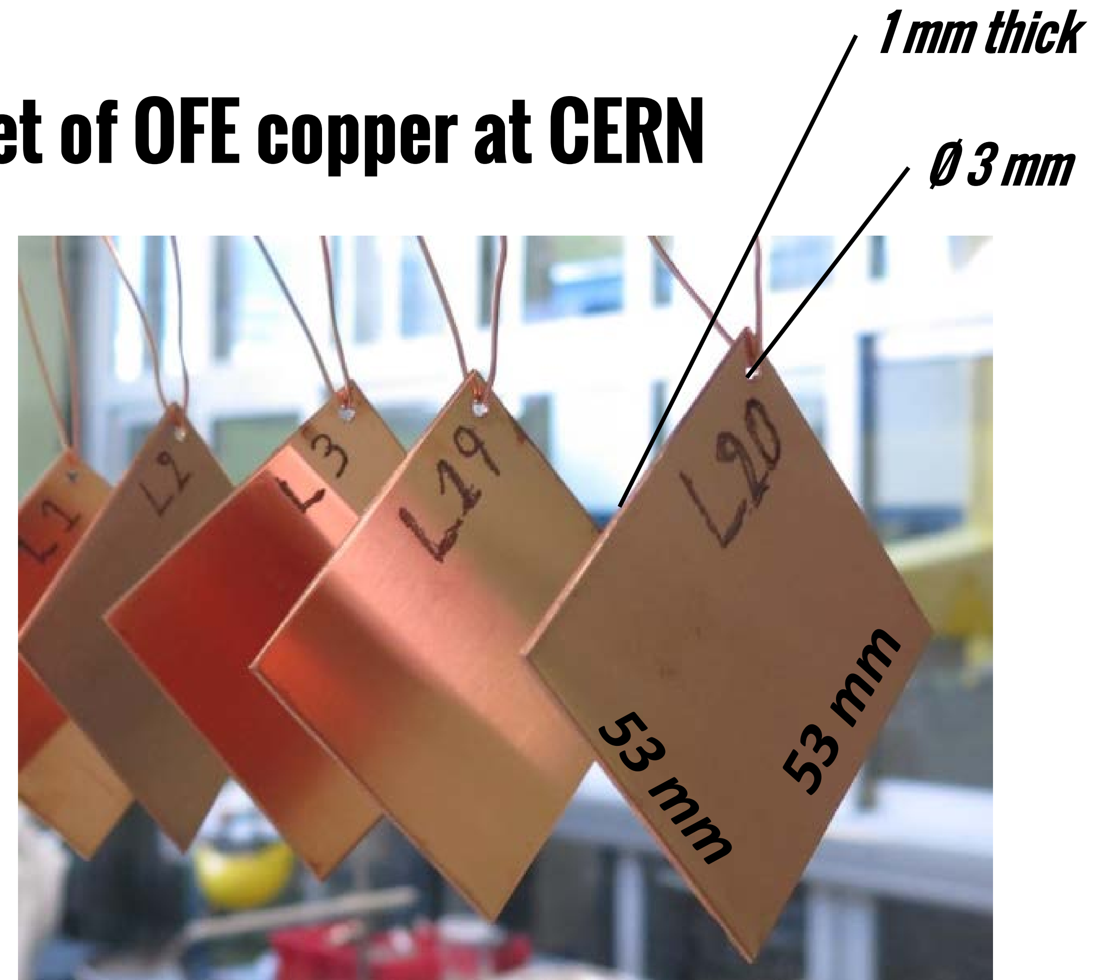
50 samples produced from the same sheet of OFE copper at CERN

Dimensions:

- 53 x 53 mm x 1 mm thick
- 3 mm hole at one corner (≈ 2,5 mm from edge)

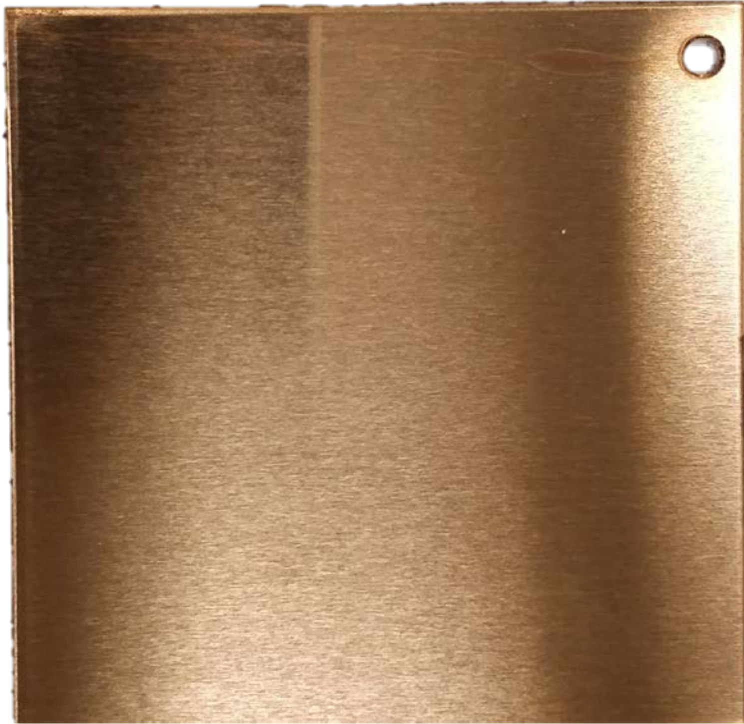
Marked on the back side

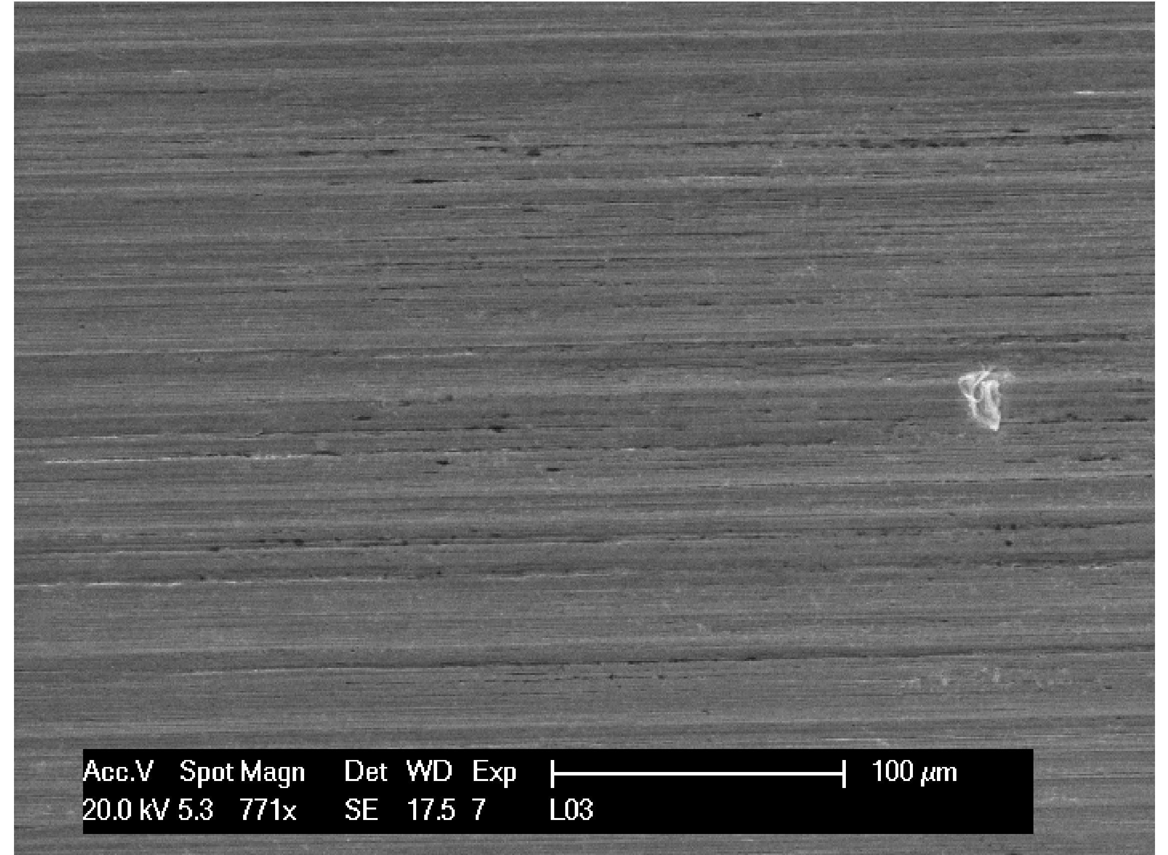
- CERN Samples: **C XX** (XX = 01-25)
- LNL Samples: **L XX** (XX = 01-25)



Initial Surface

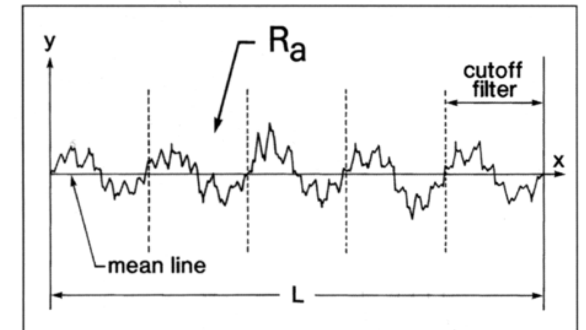
- Lamination texture on the surface





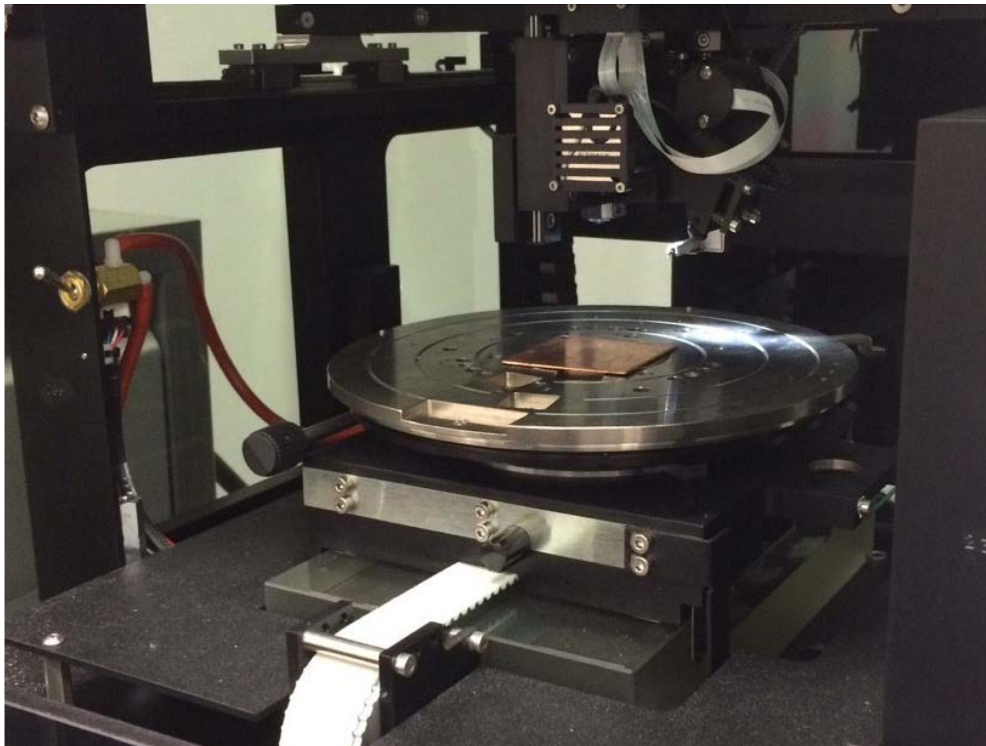
Characterization of the initial surface

- Roughness **Ra = 130 ± 30 nm** (1 mm scan length)



R_a is the arithmetic average deviation from the mean line within the assessment length (L).

$$R_a = \frac{1}{L} \int_{x=0}^{x=L} |y|\, dx$$

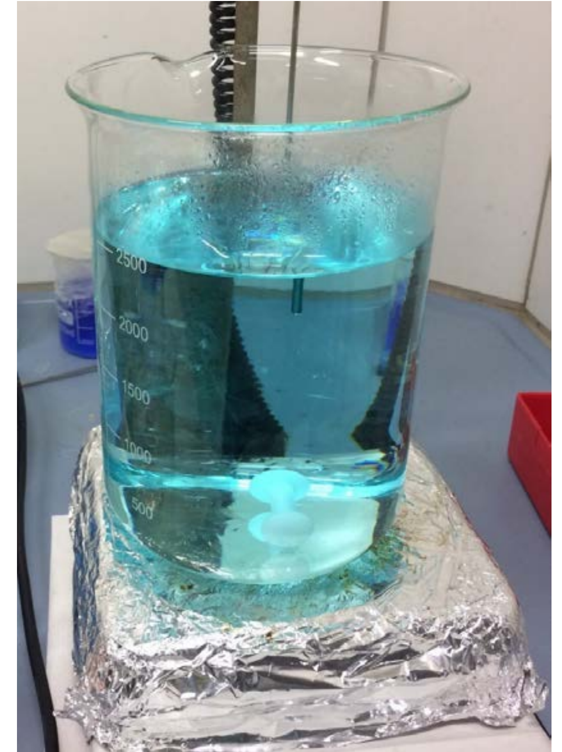


Evaluated with a Veeco Dektat 8 Profilometer

Set Up of the 4 polishing treatments investigated

1. Chemical Polishing: SUBU5

- Developed for LEP2 at CERN
- Working temperature = 72 ± 2 °C
- Etching rate: 350-600 nm/min
  (depend on the surface/solution ratio and aging)
- SUBU5 composition
  - sulfamic acid: 5g/l
  - hydrogen peroxide 32%: 50ml/l
  - n-butanol 99%: 50ml/l
  - ammonium citrate: 1g/l



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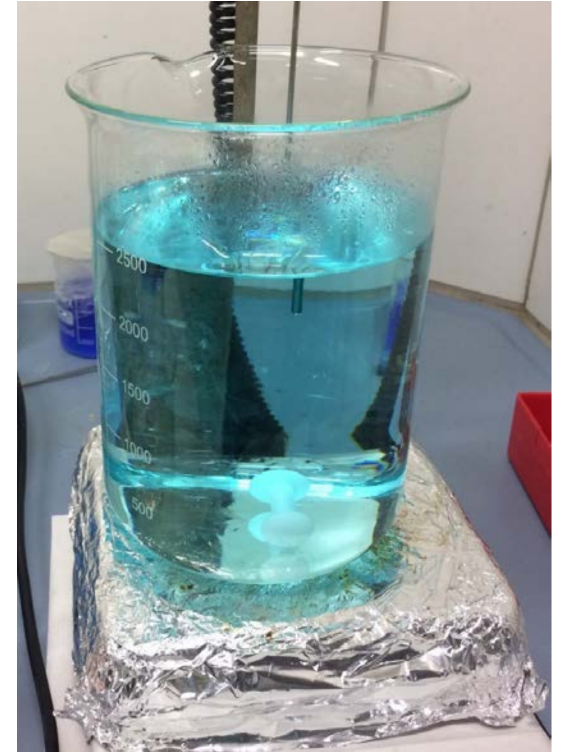
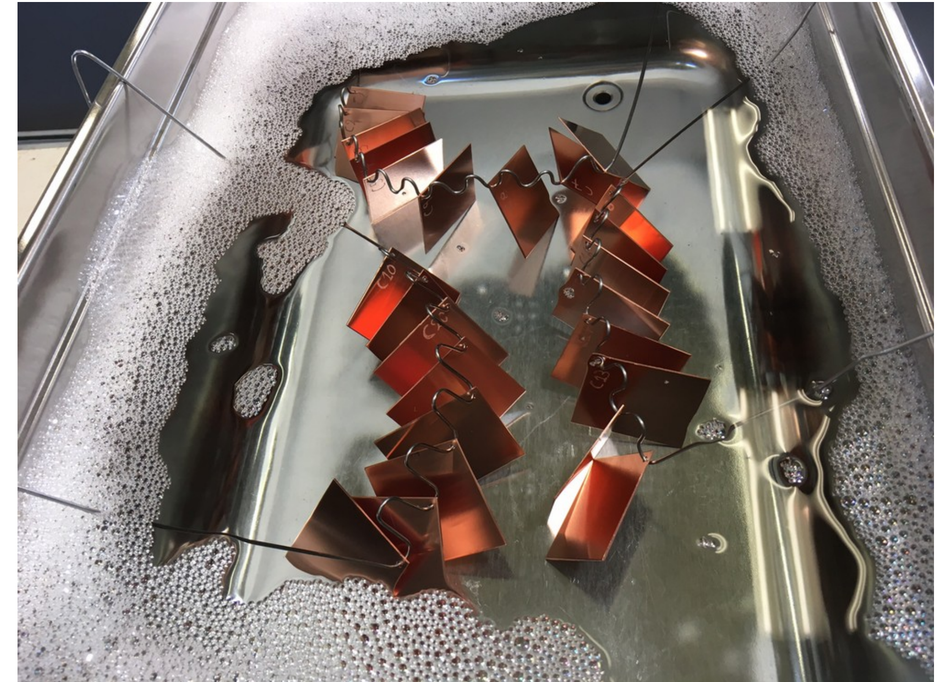
- Developed for LEP2 at CERN
- Working temperature = 72 ± 2 °C
- Etching rate: 350-600 nm/min
  (depend on the surface/solution ratio and aging)
- SUBU5 composition
  - sulfamic acid: 5g/l → corrodes copper forming copper sulfamate
  - hydrogen peroxide 32%: 50ml/l → dissolves the copper
  - n-butanol 99%: 50ml/l → moderator of the reaction (limits the pitting process on the surface)
  - ammonium citrate: 1g/l → limits the hydroxide and oxide formation



1. SUBU5 Cleaning Procedure

- **Degreasing:** NGL 1740 bath 2 hours → 3' ultra-sonic ON at start and again 3’ min ultra-sonic ON before end



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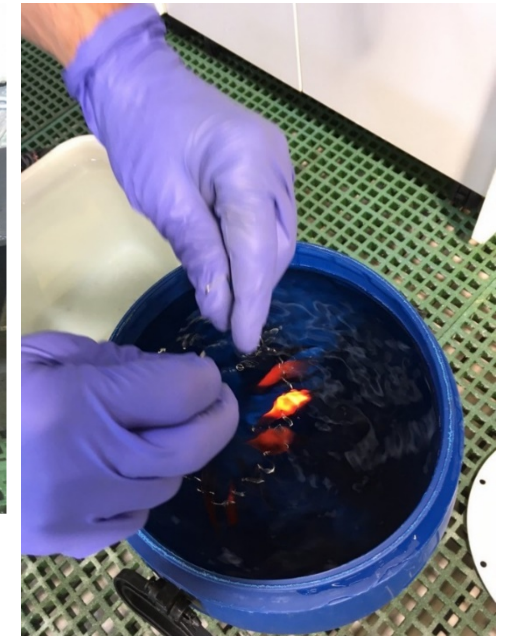
- **Passivation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 1'
- **Rinsing with water:** demineralized water for about 30s



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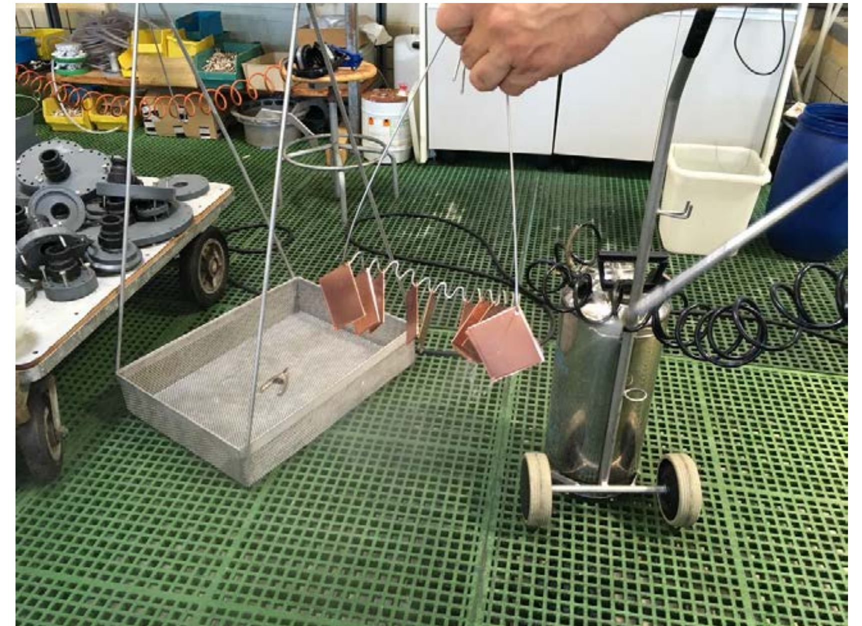
- **Spaying with alcohol:** ethyl alcohol to enhance drying
- **Drying with N_2**



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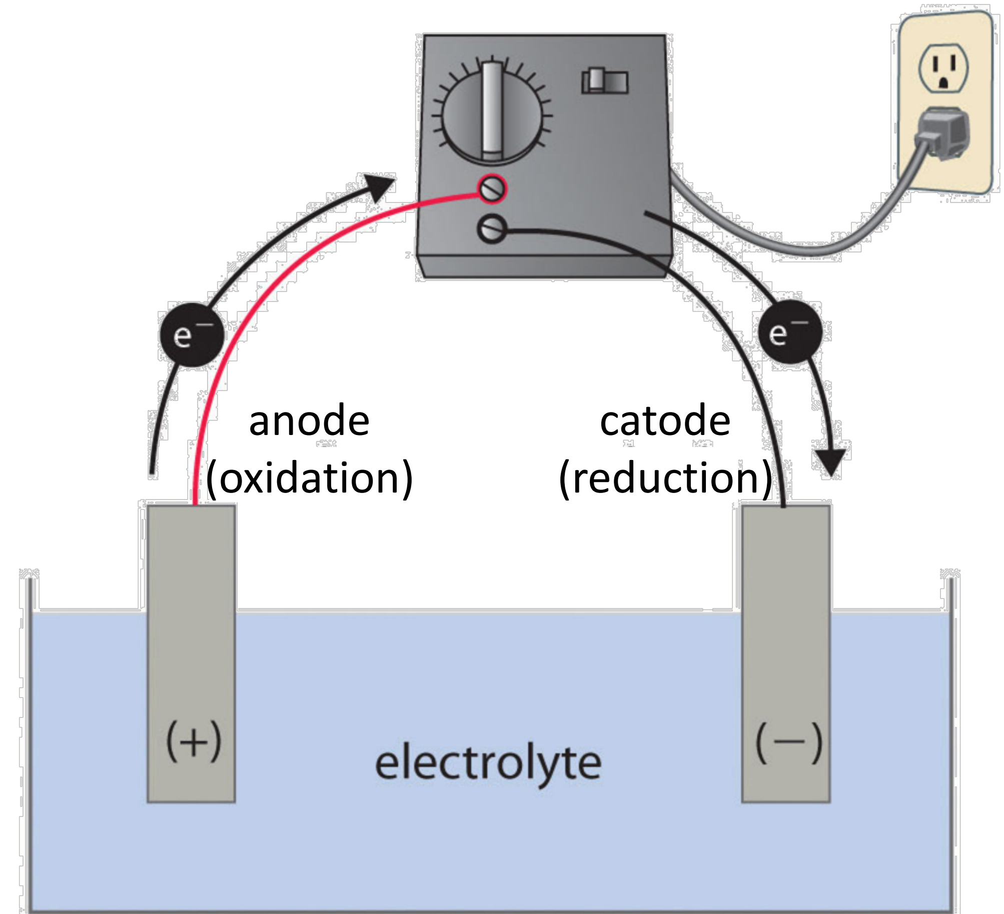
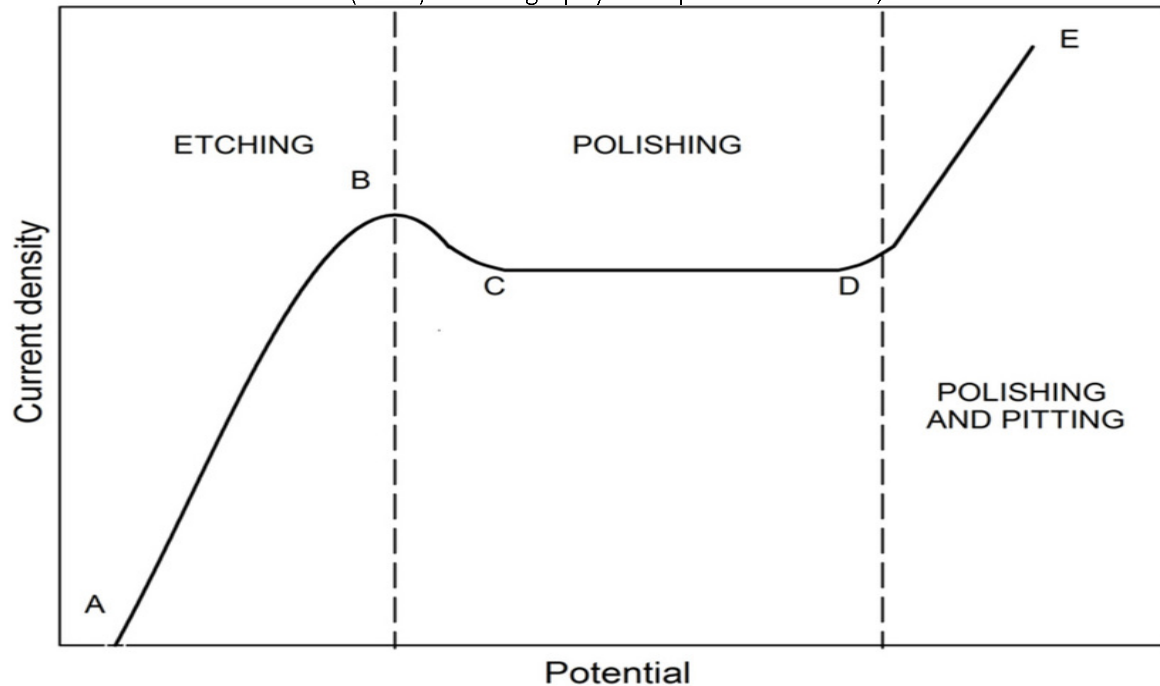
2. Electropolishing, generalities

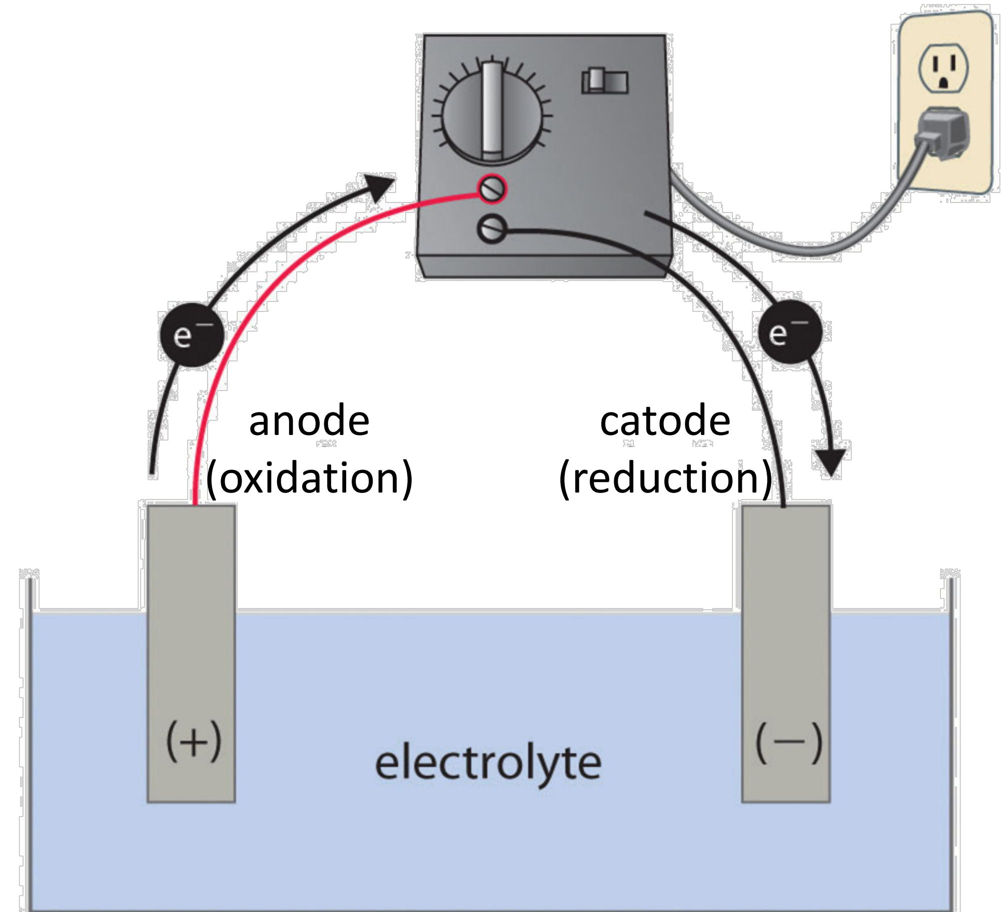
- Process inverse of electroplating
- Sample is connected to + terminal of a DC Power Supply
- Redox reactions occur
- Current flow beetween two electrodes
- The sample works as the anode (oxidised reaction)
- A viscous electrolyte is used to moderate the reaction



2. Electropolishing

Vander Voort G.F. (1999). Metalography Principles and Practice, ASM International



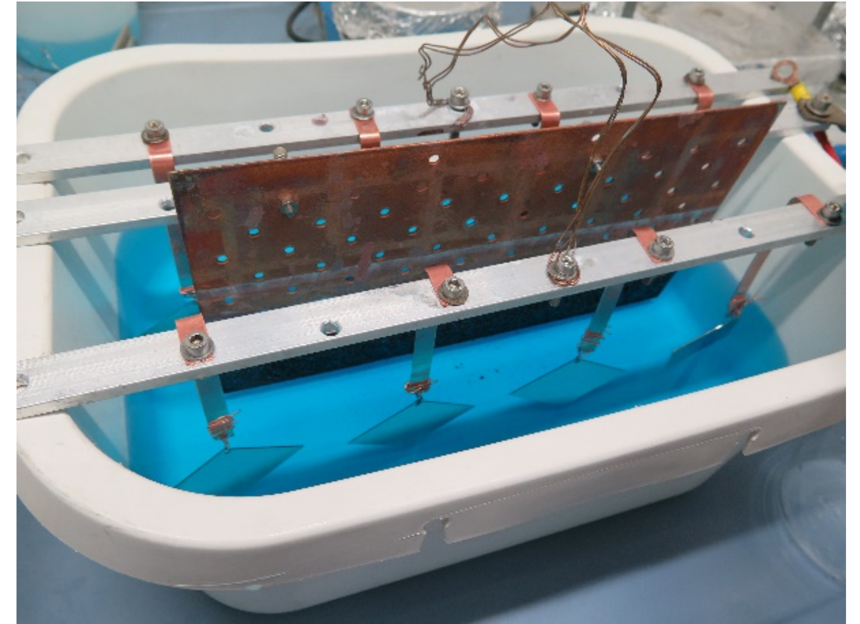


- Key parameter: I-V characteristic curve

- A polishing effect is observed between C and D

2. ARIES Samples EP Set up and Parameters

- Solution: Phosphoric acid (85%) : Buthanol (99%), ratio 3:2
- Anode: 8 samples at time
- Cathode material: OFHC copper
- Temperature: Room Temperature
- No agitation (could create anisotropy in the treatment)
- Process time ≈ 5 hours (2 + 3)
- Etching rate ≈ 130 nm/min (Could be higher)



2. EP Cleaning Procedure

- **Degreasing:** NGL 1740 bath 2 hours → 3' ultra-sonic ON at start and again 3' min ultra-sonic ON before end
- **Activation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 3'
- **Polishing:** 5 h EP without bath agitation
- **Passivation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 1'
- **Rinsing with water:** demineralized water for about 30s
- **Spaying with alcohol:** ethyl alcohol to enhance drying
- **Drying with N_2.**
- **Packing** in wafer box and then in plastic bag under N_2

3. EP+SUBU Cleaning Procedure

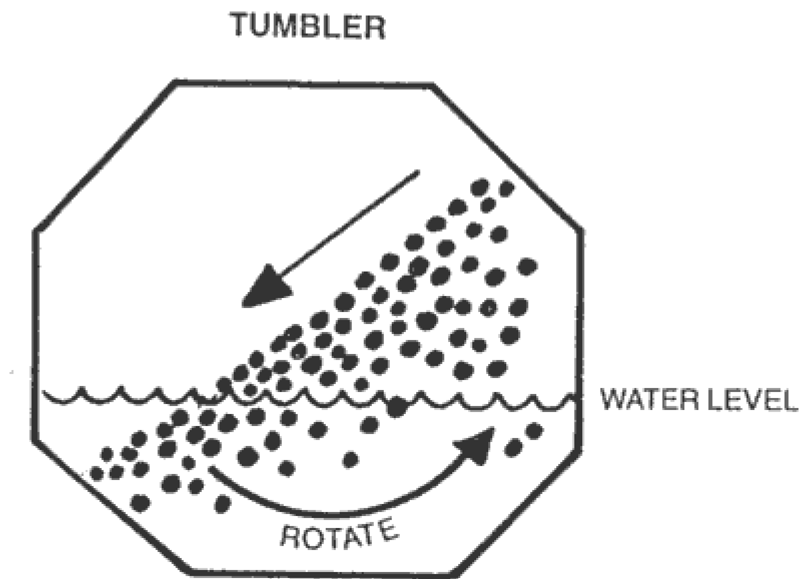
- EP prevent pitting
- SUBU remove EP texture
- SUBU also acts on the masked areas during the EP (eg the electrical contacts)

3. EP+SUBU Cleaning Procedure

- **Degreasing:** NGL 1740 bath 2 hours → 3' ultra-sonic ON at start and again 3’ min ultra-sonic ON before end
- **Activation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 3'
- **Polishing:** 5 h EP without bath agitation
- **Rinsing with water:** demineralized water for about 1 min
- **Polishing:** 8' SUBU5 with bath agitation
- **Passivation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 1'
- **Rinsing with water:** demineralized water for about 30s
- **Spaying with alcohol:** ethyl alcohol to enhance drying
- **Drying with N_2.**
- **Packing** in wafer box and then in plastic bag under N_2

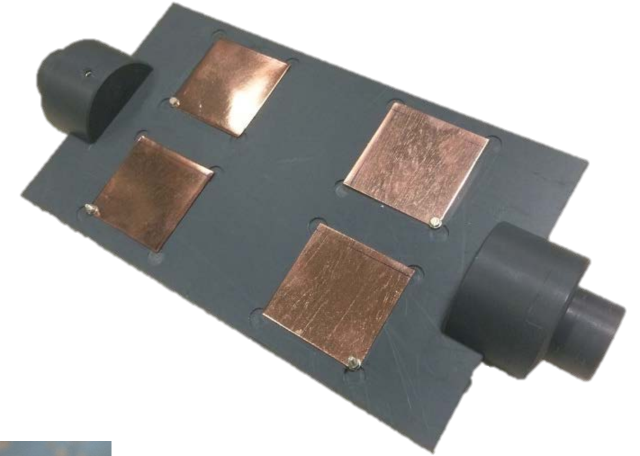
4. Tumbling, generalities

- Mechanical polishing used to prepare the cavity before chemistry
- In ARIES is used to evaluate the effect of mechanical treatment on Nb SC film
- A barrel, filled whith the samples, is then rotated
- Could be added media, water, or other lubricants



4. ARIES Samples Tumbling Set Up

- Samples keeped in a sample holder to prevent bending



- Two different media used:

1. Alumina embedded in ureic resin

2. Coconut powders

4. ARIES Samples Tumbling Set Up

3. Tumbling Procedure

- **Degreasing:** NGL 1740 bath 2 hours → 3' ultra-sonic ON at start and again 3' min ultra-sonic ON before end
- **Rinsing with water:** demineralized water for about 1 min
- **Polishing:** 20 h Tumbling with Alumina embedded media and Roadastel30 bath
- **Rinsing with water:** demineralized water for about 1 min
- **Polishing:** 20 h Tumbling with Coconut powders media
- **Degreasing:** Rodastel bath 2 hours → 3' ultra-sonic ON at start and again 3' min ultra-sonic ON before end
- **Rinsing with water:** demineralized water for about 1 min
- **Passivation:** sulfamic acid (H_3NO_3S, 5 g/l) for about 1'
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- **Spaying with alcohol:** ethyl alcohol to enhance drying
- **Drying with N_2.**
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Surface Characterization

Surface Characterizations

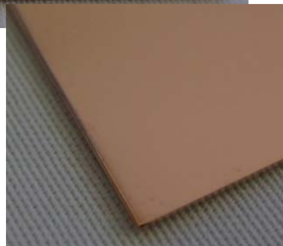
1. Optical Inspection
2. Reflectivity
3. SEM
4. EDS
5. Roughness

Optical inspection

Initial surface

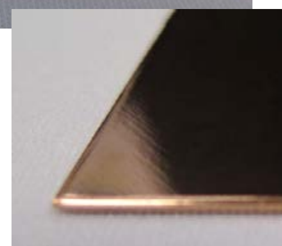
- Lamination texture

SUBU



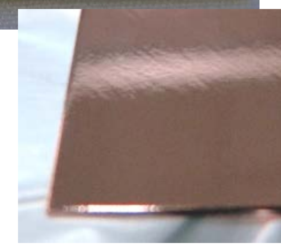
- Mirror like surface
- Reflectivity 65 ± 1 %

EP



- Mirror like surface
- Texture due to oxigen evolution
- Reflectivity 64 ± 1 %

EP + SUBU

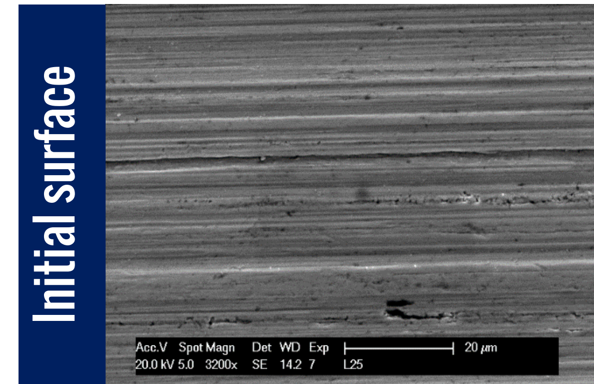


- Mirror like surface
- Texture due to oxigen evolution reduceded by SUBU
- Reflectivity 66 ± 1 %

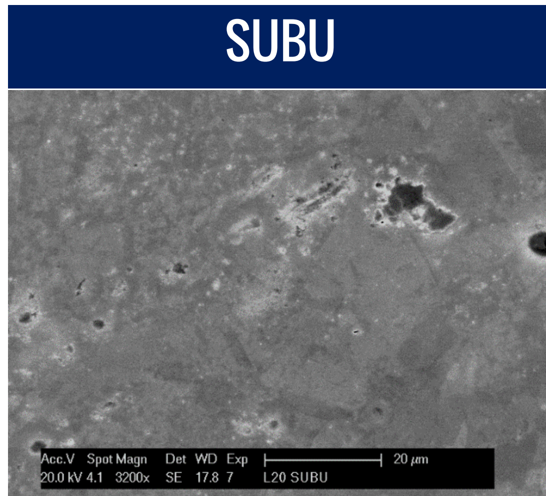
Tumbling

- Shining surface
- Small visible scratches on surface
- Reflectivity 52 ± 1 %

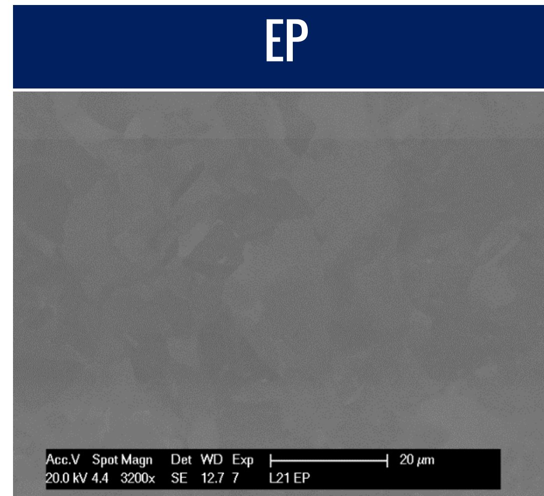
SEM Characterization



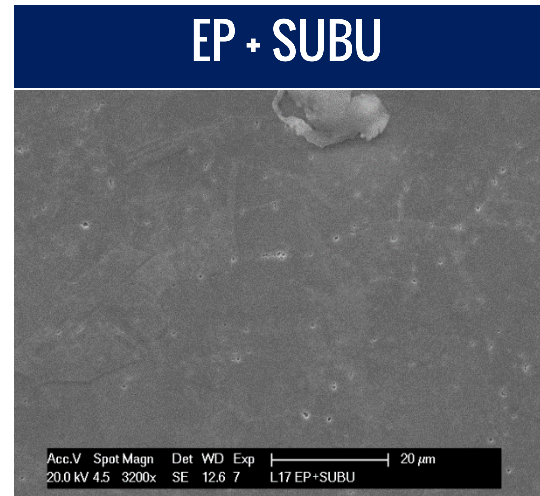
- Lamination texture



- Presence of pitting



- No pitting



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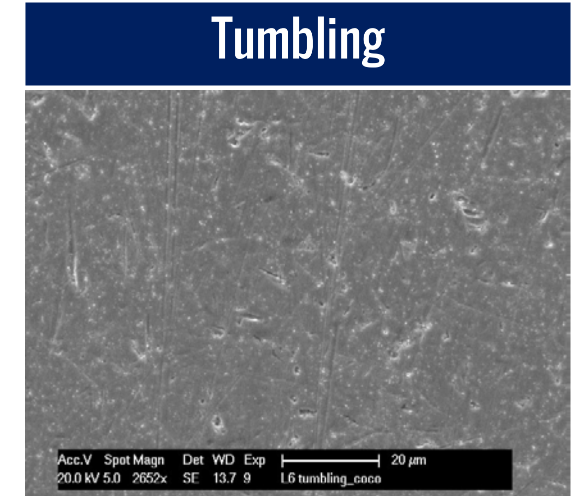
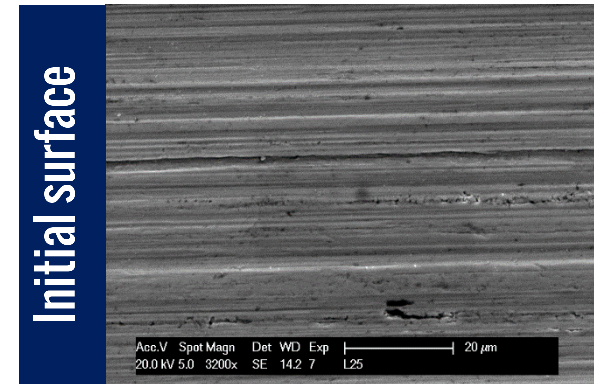
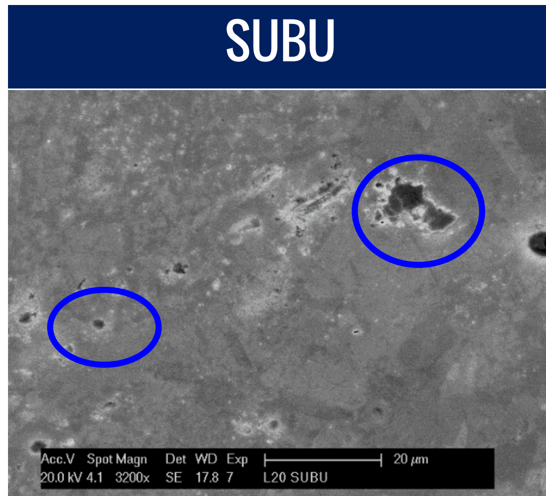


- Presence of scratches
- Possible inclusion of media

SEM Characterization



- Lamination texture



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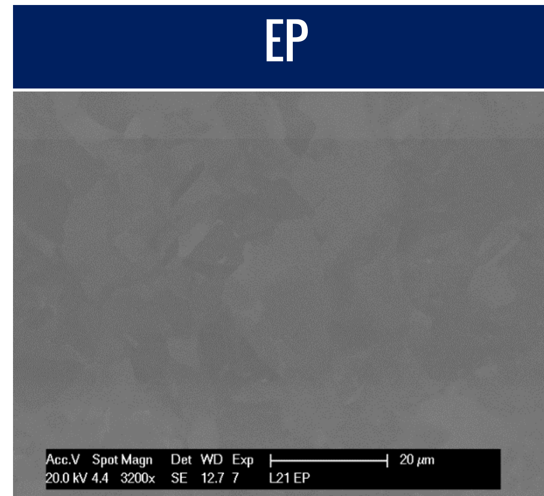
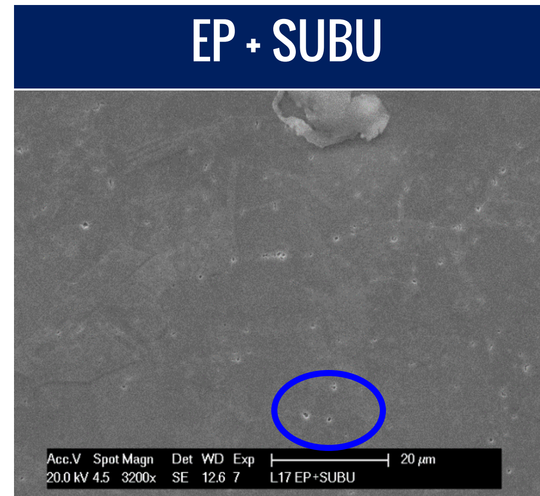


- No pitting



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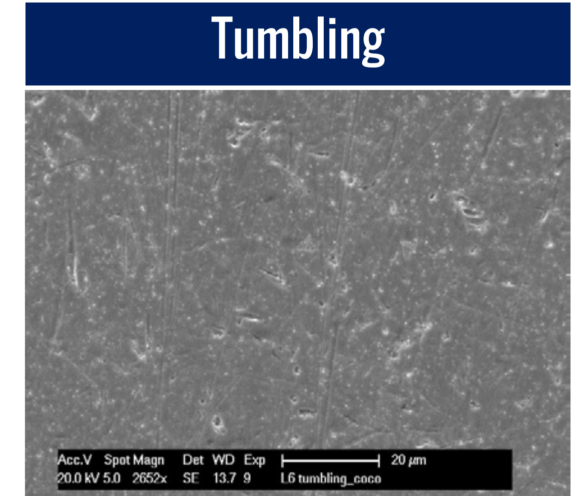

- Presence of scratches
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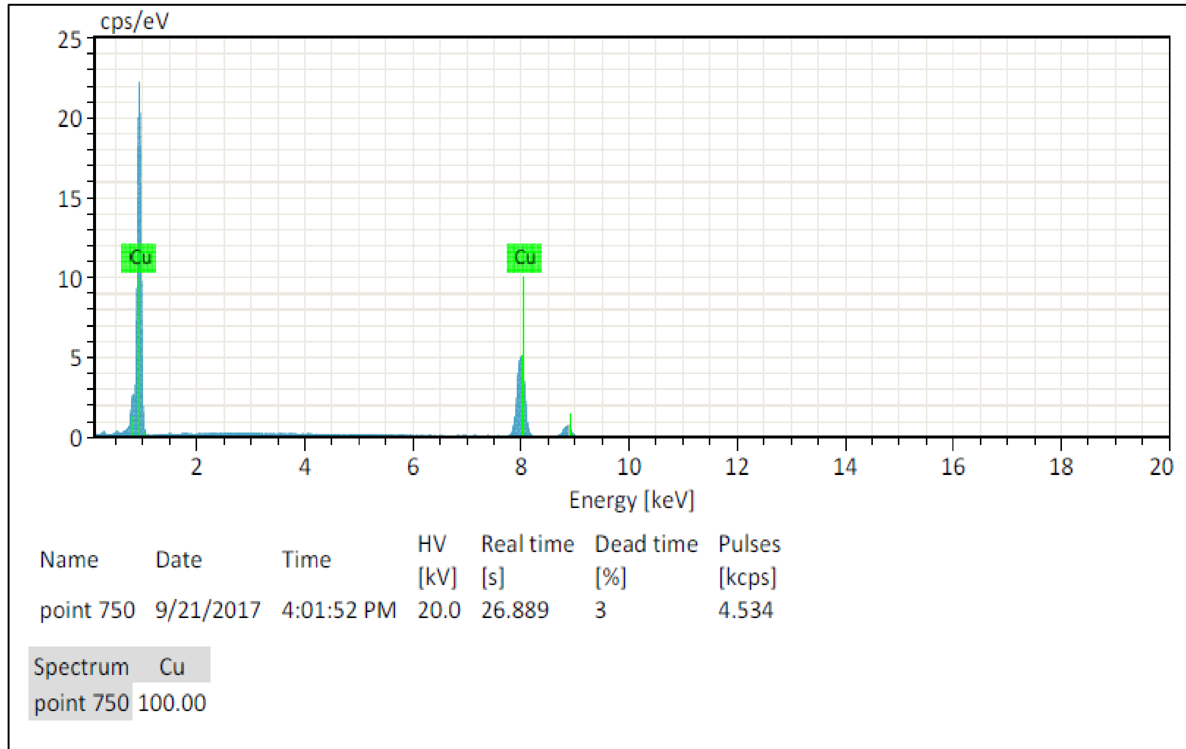
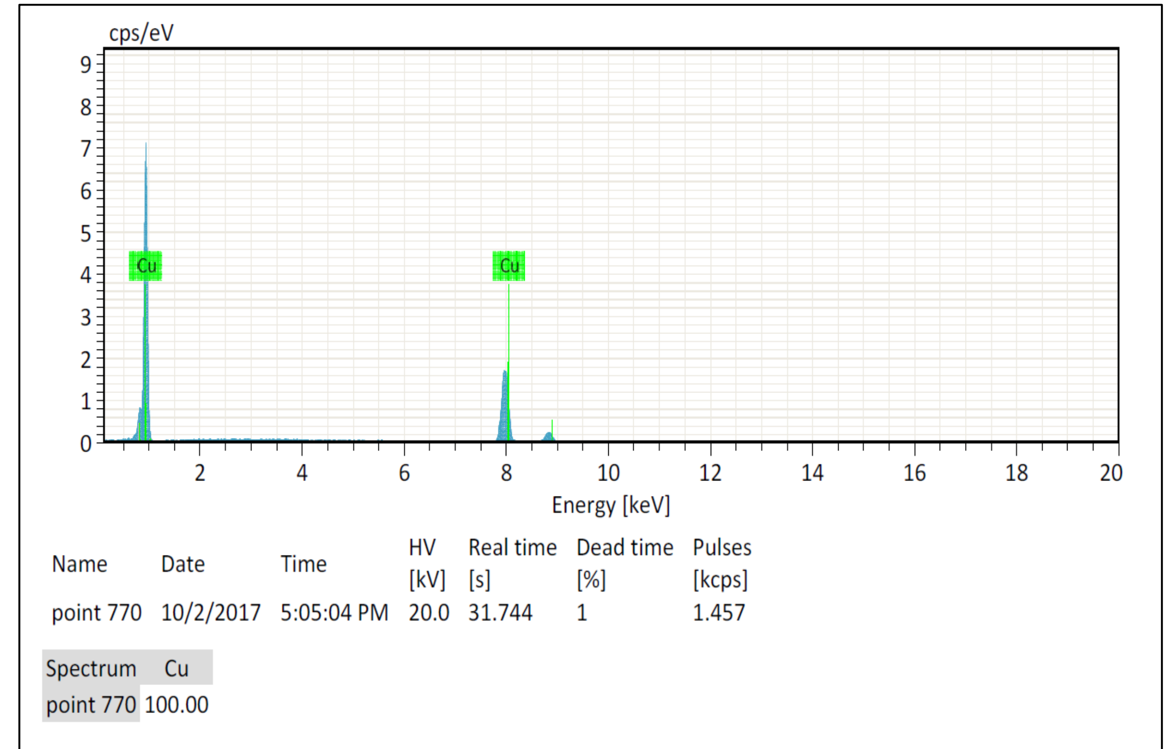
Energy Dispersive Spectroscopy



UNTREATED SAMPLE



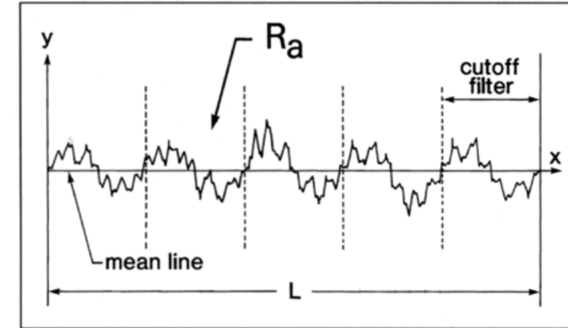
POLISHED SAMPLE

- No visible contaminations are revealed with EDS technique

Roughness

Polishing Treatment	Ra
Initial surface	*127 ± 26 nm*
SUBU5	48 ± 7 nm
EP	225 ± 80 nm
EP+SUBU5	115 ± 80 nm
Tumbling	48 ± 13 nm

Scan length of 1 mm



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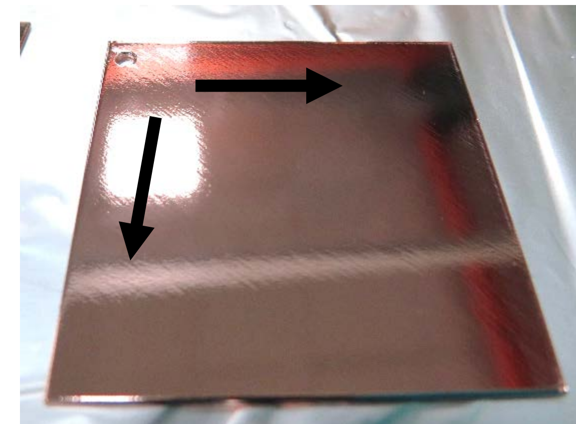
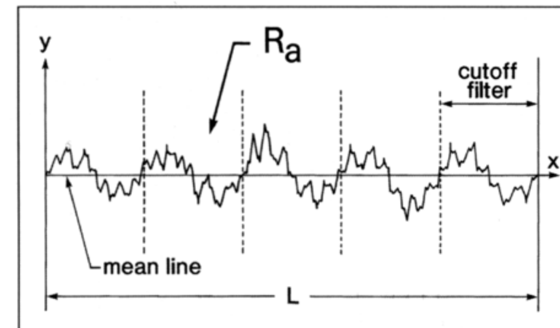


Roughness

Polishing Treatment	Ra	Ra diagonal
Initial surface	*127 ± 26 nm*	
SUBU5	48 ± 7 nm	
EP	225 ± 80 nm	86 ± 14 nm
EP+SUBU5	115 ± 80 nm	59 ± 9 nm
Tumbling	48 ± 13 nm	

Scan length of 1 mm



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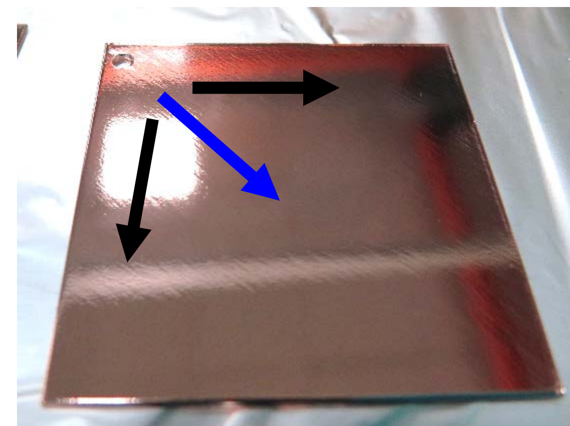

Conclusions

- Surface characterizations show that SUBU5 reduces roughness more than the other treatments
- SUBU5 produces pitting on the surface, also if used just for the etching of 5 microns (EP+SUBU)
- EP treated surface does not present pitting, but roughness is influenced by the dynamic of the process
- Tumbling reduces surface roughness at the same values of SUBU5
- Tumbling introduces small scratches on the surface and possible inclusions
- WP15 will focus more on EP (pitting free) and SUBU5 (lowest roughness without scratches) polishing treatments
- SC characterizations are necessary to evaluate the effect of polishing treatment on Nb thin film

Thank you for your attention

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