Impact of the Cu substrate surface preparation on Nb coatings for SRF

THP041

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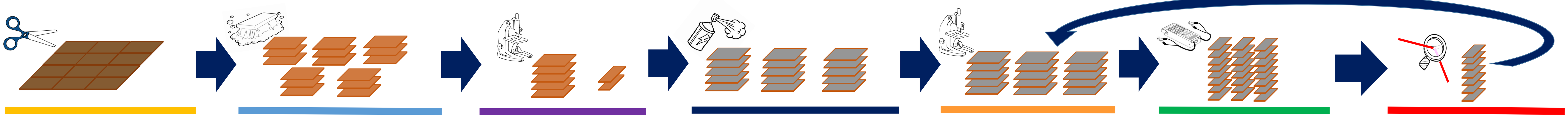
Abstract

Nowadays, one of the main issues of the superconducting thin film resonant cavities is the Cu surface preparation. A better understanding of the impact of copper surface preparation on the morphological, superconductive (SC) and RF properties of the coating, is mandatory in order to improve the performances of superconducting cavities by coating techniques. ARIES H2020 collaboration includes a specific work package (WP15) to study the influence of Cu surface polishing on the SRF performances of Nb coatings that involves a team of 8 research groups from 7 different countries. In the present work, a comparison of 4 different polishing processes for Cu (Tumbling, EP, SUBU, EP+SUBU) is presented through the evaluation of the SC and morphological properties of Nb thin film coated on Cu planar samples and QPR samples, polished with different procedures. Effects of laser annealing on Nb thin films have also been studied.

Workflow of the experiment



SAMPLE PRODUCTION
- 50 samples cutted from a single OFE copper sheet

5 DIFFERENT POLISHING TREATMENTS TESTED
- Chemical Polishing
- Electro Polishing
- EP+Chemical Polishing
- Tumbling
- Laser Polishing

MORPHOLOGICAL CHARACTERIZATION
- Visual inspection
- Reflectivity
- SEM
- EDS
- Profilometer
- AFM

THIN FILM COATING IN 3 DIFFERENT FACILITIES
- Magnetron Sputtering
- 3 different set-ups
- Similar parameters

MORPHOLOGICAL CHARACTERIZATION
- SEM
- EDS
- XRD
- AFM
- Profilometer

PHYSICAL PROPERTIES CHARACTERIZATION
- PPMS
- Exoelectrons
- *QPR*
- *AC-magnetometer*
- *Radiofrequency cathode*

POST-TREATMENT
- Laser irradiation

STANDARD POLISHING PROTOCOL USED

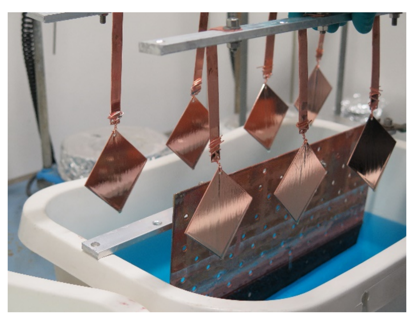
#	Treatment	Solution	Time
1	Degreasing	NGL 1740 ultrasounds	5 min
2	Activation	H_3NO_3S, 5 g/l	3 min
3	*Polishing*	*Treatment depending*	-
4	Passivation	H_3NO_3S, 20 g/l	1 min
5	Rinsing	Demineralized water	1 min
6	Spraying	Ethyl alcohol	-
7	Drying	Nitrogen gas	2 min
8	Packing	Wafer boxes under N_2	-

CHEMICAL POLISHING: SUBU5

- SOLUTION:
  - Sulfamic Acid (5g/l)
  - Hydrogen Peroxide 32% (50ml/l)
  - N-butanol 99% (50ml/l)
  - Ammonium Citrate (1g/l)
- TEMPERATURE: 72°C
- AGITATION: yes
- ETCHING RATE: 0,5-0,6 µm/h
- TOTAL THICKNESS REMOVED: 40 µm

ELECTROPOLISHING

- SOLUTION:
  - Phosphoric Acid 85%
  - N-butanol 99%
  - Ratio H_3PO_4:C_4H_{10} 3:2
- TEMPERATURE: room temperature
- AGITATION: no
- ETCHING RATE: 0,5-0,6 µm/h
- TOTAL THICKNESS REMOVED: 40 µm



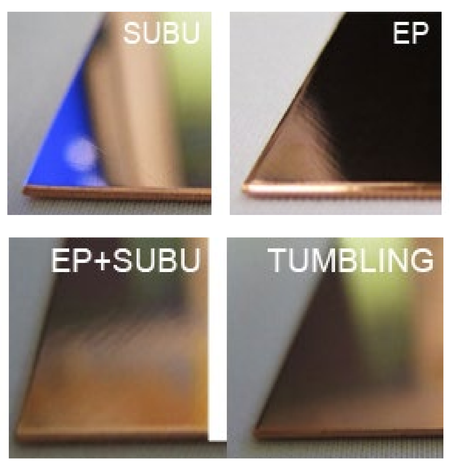
EP + SUBU

- TOTAL THICKNESS REMOVED WITH EP: 35 µm
- TOTAL THICKNESS REMOVED WITH SUBU: 5 µm

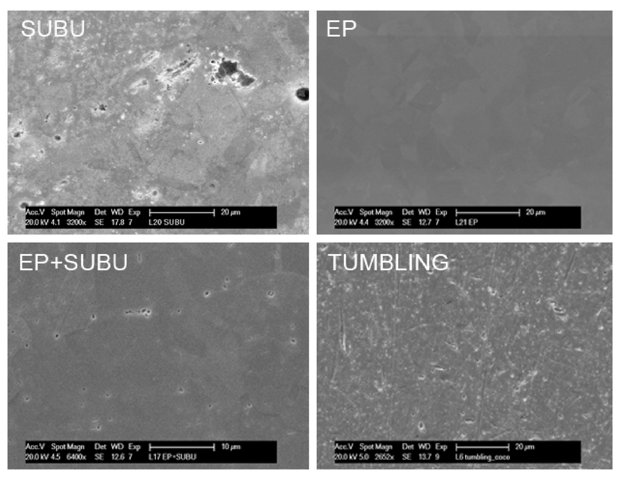
TUMBLING

- Tumbling with a 3 dimensional motion
- 2 MEDIA USED:
  1. alumina embedded media and Roadastel30 bath (WET TUMBLING)
  2. coconut powders (DRY TUMBLING)
- TOTAL THICKNESS REMOVED: 2 µm

VISUAL INSPECTION



SEM MICROGRAPHS OF Cu SAMPLES

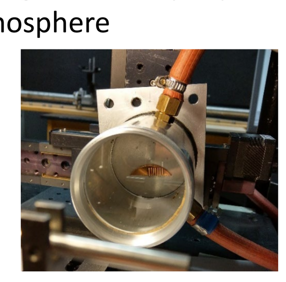


ROUGHNESS OF Cu POLISHED SAMPLES

Treatment	Ra [nm] *(along diagonal)*	Reflectivity [%]
Initial surface	130 ± 30	-
SUBU5	48 ± 7	65 ± 0,3
EP	225 ± 80 *(86 ± 14)*	64± 0,3
EP+SUBU5	115 ± 80 *(59 ± 9)*	66 ± 0,3
Tumbling	44 ± 7	52 ± 0,3

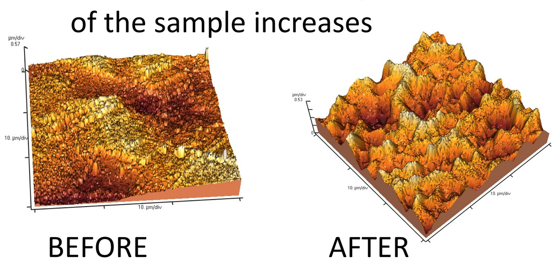
LASER POLISHING

- Pulsed Nd:YAG laser
- λ= 523 nm
- τ =4 ns
- Intensity: up to 1.6 GW/cm2
- Scanning mode, Step: 5µm
- Ar atmosphere



LASER POLISHING

- Irradiation by laser leads to ablation the smallest crystals. As a result surface roughness Ra of the sample increases

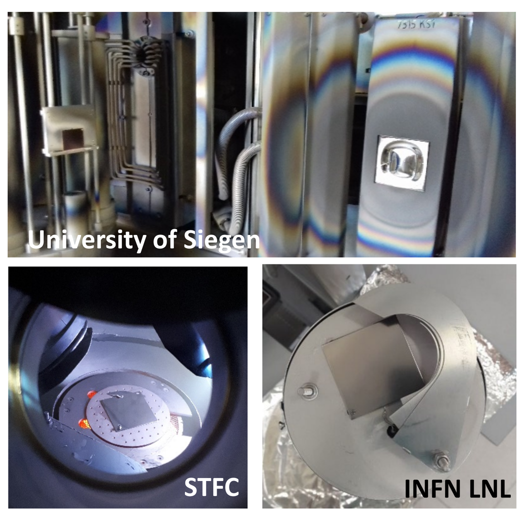


BEFORE IRRADIATION Ra = 68 nm

AFTER IRRADIATION Ra = 272 nm

Nb THIN FILM COATING

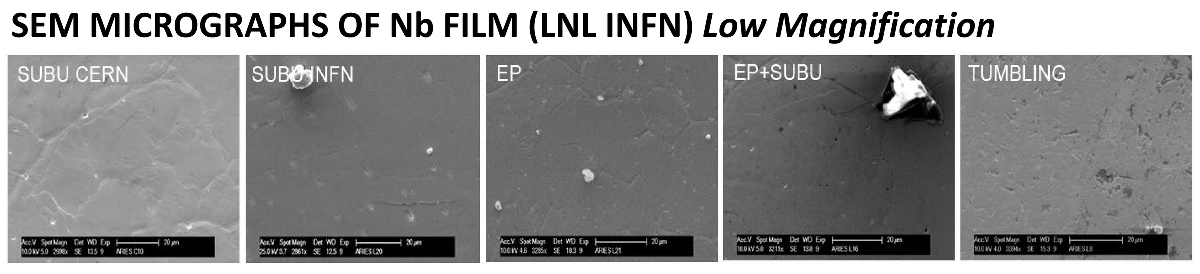
Parameter	STFC	U.Siegen	INFN
Base P @ 650 °C (mbar)	$<10^{-9}$	$1.22x10^{-5}$	$< 9x10^{-8}$
Deposition T	650 °C	650 °C	650 °C
Current density (mA/cm^2)	22	15	27
Target power (W)	≈ 400	≈ 400	≈ 750
Discharge gas	Kr	Ar	Ar
Disch. P (mbar)	$1.5x10^{-3}$	$1.5x10^{-2}$	$5x10^{-3}$
Substrate rotation	4 rpm	n/a	No
Deposition rate (nm/min)	20	150	150
Thickness	10 µm	3 µm	3 µm



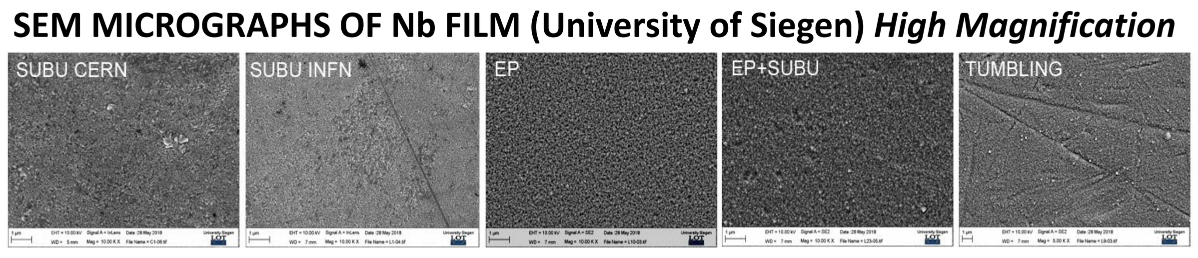
ROUGHNESS OF Nb FILM (Siegen samples)

Treatment	AFM - Roughness (Ra)
SUBU5 (CERN)	21 ± 12.1 nm
SUBU5 (INFN)	6.3 ± 1.2 nm
EP	11.5 ± 0.7 nm
EP + SUBU5	14.2 ± 2.4 nm
Tumbling	18.3 ± 1.5 nm

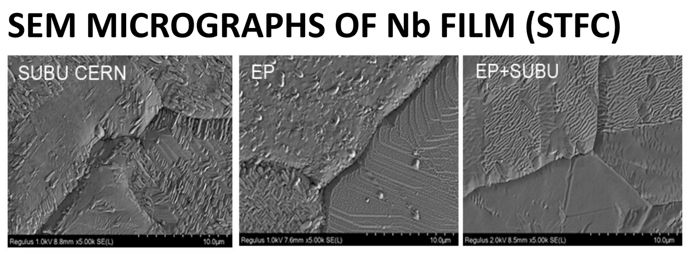
SEM MICROGRAPHS OF Nb FILM (LNL INFN) *Low Magnification*



SEM MICROGRAPHS OF Nb FILM (University of Siegen) *High Magnification*

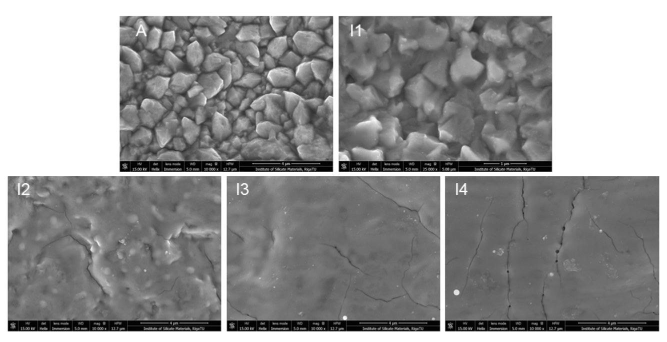


SEM MICROGRAPHS OF Nb FILM (STFC)



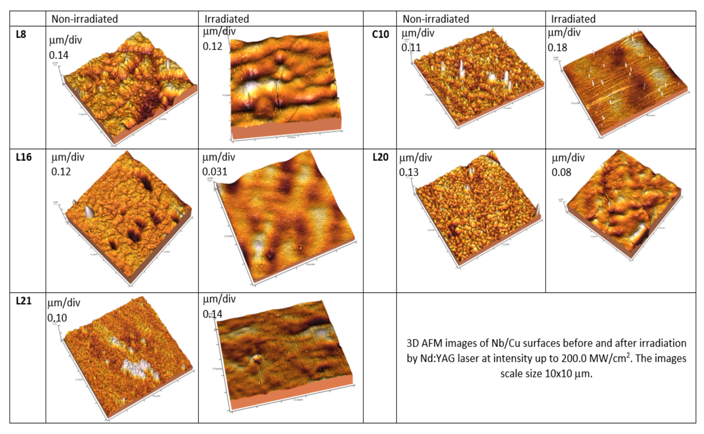
On STFC samples pitting is not visible because the Nb film is thicker than INFN and Siegen samples. (10 µm instead of 3 µm) Low deposition rate could help to mitigate substrate imperfections

LASER POST TREATMENT



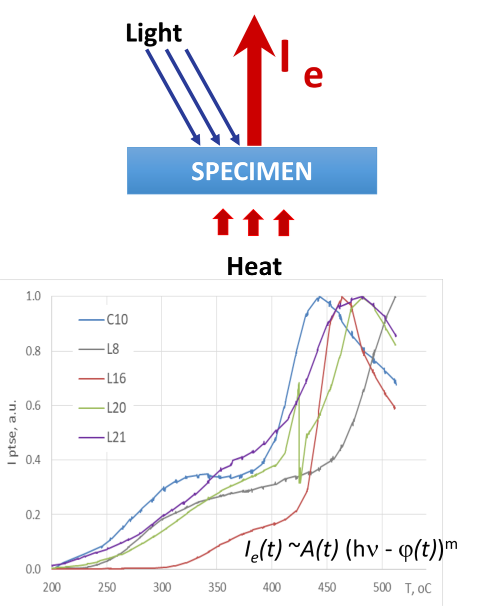
SEM images of Nb/Cu sample before irradiation (A) and after irradiation by Nd:YAG laser with intensities: I1 = 140 MW/cm²; I2 = 170 MW/cm²; I3 = 253 MW/cm²; I4 = 320 MW/cm².

Treatment	Ra Non irradiated	Ra Irradiated
SUBU5 (CERN)	9 nm	1 nm
SUBU5 (INFN)	9 nm	7 nm
EP	12 nm	5 nm
EP + SUBU5	13 nm	3 nm
Tumbling	25 nm	13 nm



Before the irradiation, Nb/Cu surface consists of nano-crystals with sizes ranging from 300 nm to 2 µm. After the irradiation, nanocrystals become smaller and surface becomes smoother due to its melting with increasing of laser intensity. Evaporation takes place at the intensity 320 MW/cm². However, cracks appear on the irradiated surface at 253 MW/cm² and higher intensities. XRD patterns of non-irradiated and irradiated Nb/Cu sample has shown the increase of crystallite size from 25 nm to 31 nm.

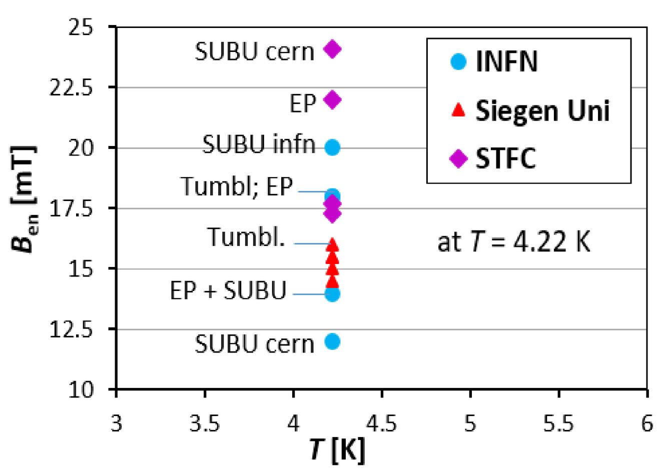
PHOTO-TERMOSTIMULATED EXOEMISSION



The interpretation of the results requires additional research to understand the nature of the introduced defects and physics of TSE/PTSE in Nb

FIRST FLUX ENTRY FIELD

Treatment	B_{en} Non irradiated	B_{en} Irradiated
SUBU5 (CERN)	12 mT	17.0 mT
SUBU5 (INFN)	20 mT	23.7 mT
EP	18 mT	18.8 mT
EP + SUBU5	14 mT	15.5 mT
Tumbling	18 mT	19.1 mT



CONCLUSIONS

Five surface treatments techniques: SUBU, EP, EP+SUBU, Tumbling and Laser Polishing, prepared in 3 different Institutions on an identical planar substrate was investigated. Based on the morphological characterization, the main conclusion is that the most promising procedure is EP, as a pitting free technique. SUBU provides the lowest roughness, but pitting is more dangerous from RF point of view. Superconducting Properties of Nb films shows a slight difference between deposition facilities rather than different surface preparation. RF test are mandatory for a better comprehension of the substrate effect on the final properties of Nb film coating. RF measurements at Helmholtz-Zentrum Berlin (HZB) on Quadrupole Resonator (QPR)'s sample are already planned. Laser post-treatment first results are very promising. Laser radiation show the capability to reduce dramatically the surface roughness and to increase the field of first entry in all the samples treated. The mechanism is still under investigation.

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871