



**RESEARCH
UNIVERSITY**

EXCELLENCE INITIATIVE

Ministry of Science
and Higher Education



Silesian
University
of Technology



**Electrochemistry
Group**

***HYBRID BIODEGRADABLE COATING FOR ONE-WIRE PERIPHERAL NITINOL
STENT FOR PREVENTION OF RESTENOSIS AND PLAQUE FORMATION***

HYBBISTENT

Wojciech Simka

Project meeting / workshop

Institute of Atomic Physics and Spectroscopy, University of Latvia

12.04.2024

CONSORTIUM

COORDINATOR: Fraunhofer Institute for Ceramic Technologies and Systems IKTS (Germany)

PARTNERS: MAT PlasMATEc (Germany)
University of Latvia (Latvia)
Silesian University of Technology - SUT (Poland) – [formly from 20.10.2023](#)

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**Silesian University
of Technology**

Hybrid biodegradable coating for one-wire peripheral nitinol stent for prevention of restenosis and plaque formation „Hybbistent”



M-era.Net

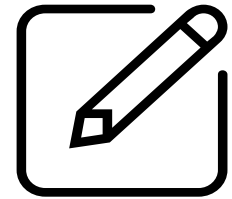
NiTi STENTS ANODIZATION

SPECIFIC OBJECTIVES (form project description)

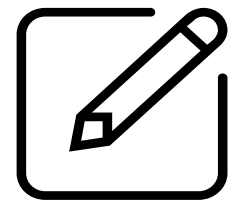
3 **Deposition of the PEO inner coating** – will be based on development of a PEO solution containing ethylenediamine tetraacetic acid (EDTA), calcium hydroxide ($\text{Ca}(\text{OH})_2$), potassium dihydrogen phosphate (KH_2PO_4) and calcium formate ($\text{Ca}(\text{HCOO})_2$). AS/DC electrical mode will be used for deposition process. Alternatively, anodic oxidation from spark discharge will be applied. The NPs will be added to electrolytes enabling later their integration into the layer structure. The concentrations of the NPs in the solvent and in the layer will be correlated. The temperature for the deposition process will be adjusted. The technology will enable also the integration of drugs for example used in drug-eluting stents and provides the potential of extended application. The composition of electrolyte bath during the PEO deposition processes will be adjusted to form chemical groups like COOH , CO , CN_3 , OH etc. helping to achieve a good adhesion of the PLGA layer to the PEO layer.



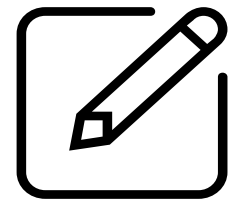
MILESTONES



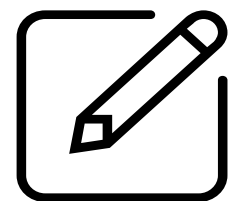
Development of the deposition modes for PEO treatment of the nitinol stents



Protocols for nanoparticle incorporation into PEO coating are developed.



Optimization of the PEO technology depending on results of the stent characterization.



Manufacturing of the coated one-wire peripheral nitinol stents for further coating with PLGA and further characterization.

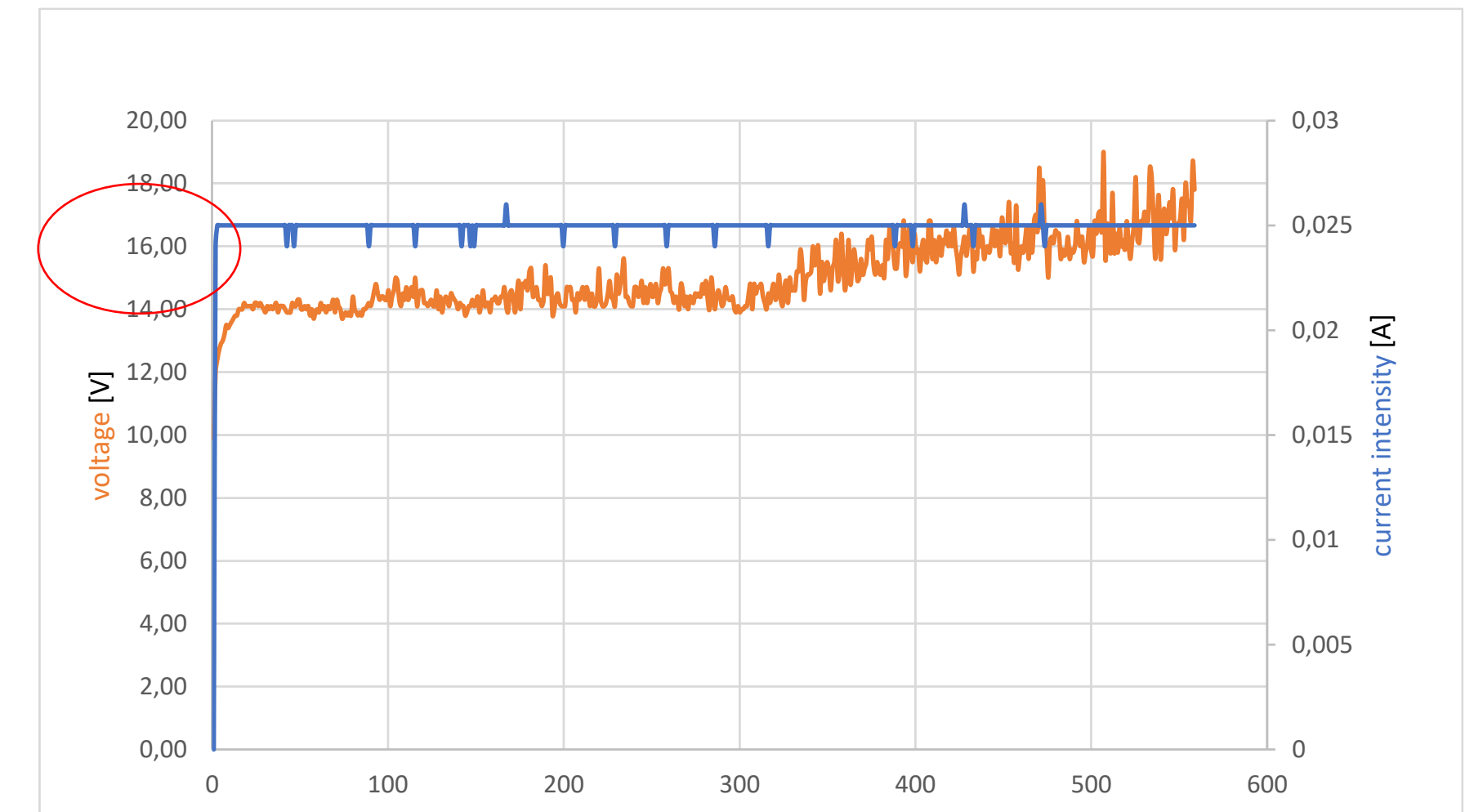


NiTi ANODIZATION

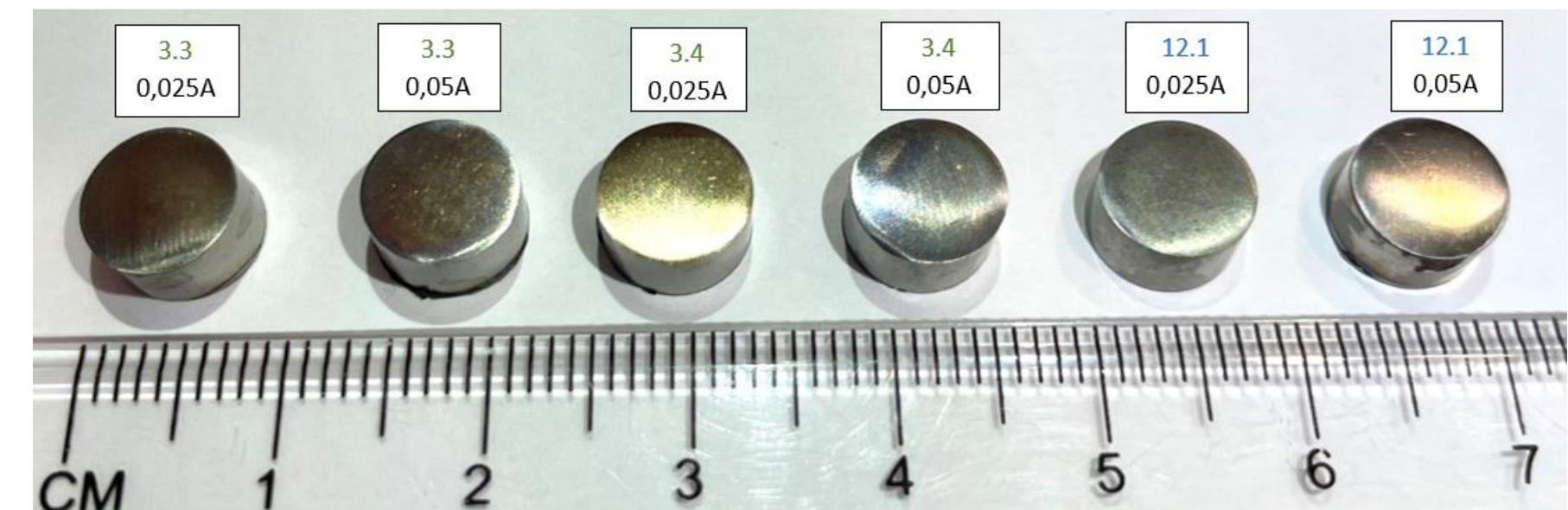
Oxygen evolution reaction occur (on Ni), not formation of oxides !!!

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Compound	Description	Concentration, mol/dm ³	Current density (intensity)	Voltage, V
Na ₂ SiO ₃	AF 3.1	0.0003	50 mA/cm ² (0.025A) 100 mA/ cm ² (0.05A)	Up to 500
	AF 3.2	0.0015		
	AF 3.3	0.009		
	AF 3.4	0.05		
NaH ₂ PO ₄	AF 11.1	0.05	50 mA/cm ² (0.025A) 100 mA/ cm ² (0.05A)	Up to 500
Na ₂ HPO ₄	AF 11.2	0.05	50 mA/cm ² (0.025A) 100 mA/ cm ² (0.05A)	Up to 500
Na ₃ PO ₄	AF 11.2	0.05	50 mA/cm ² (0.025A) 100 mA/ cm ² (0.05A)	Up to 500



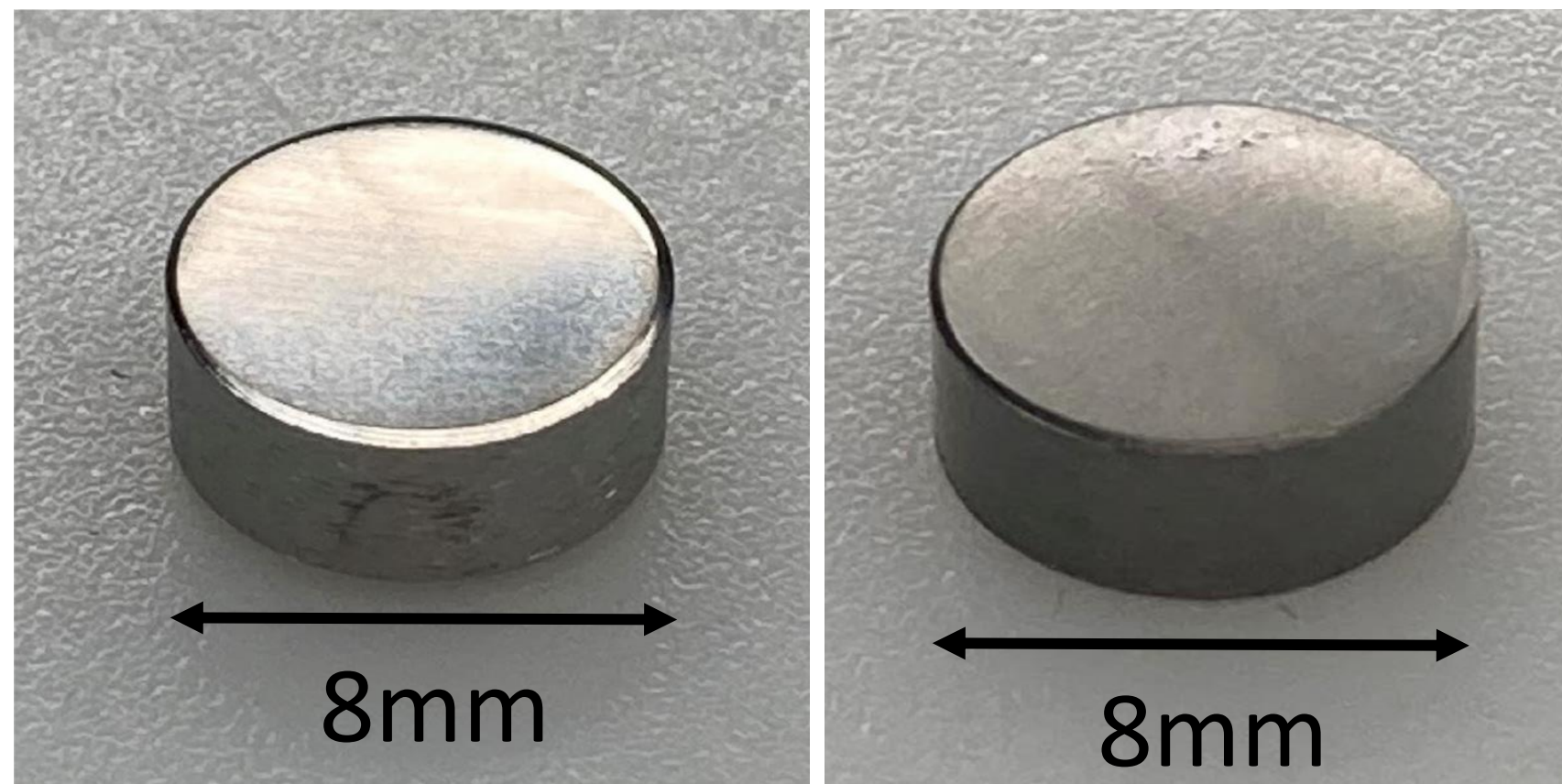
$$(U, I) = f(t)$$



NiTi ANODIZATION

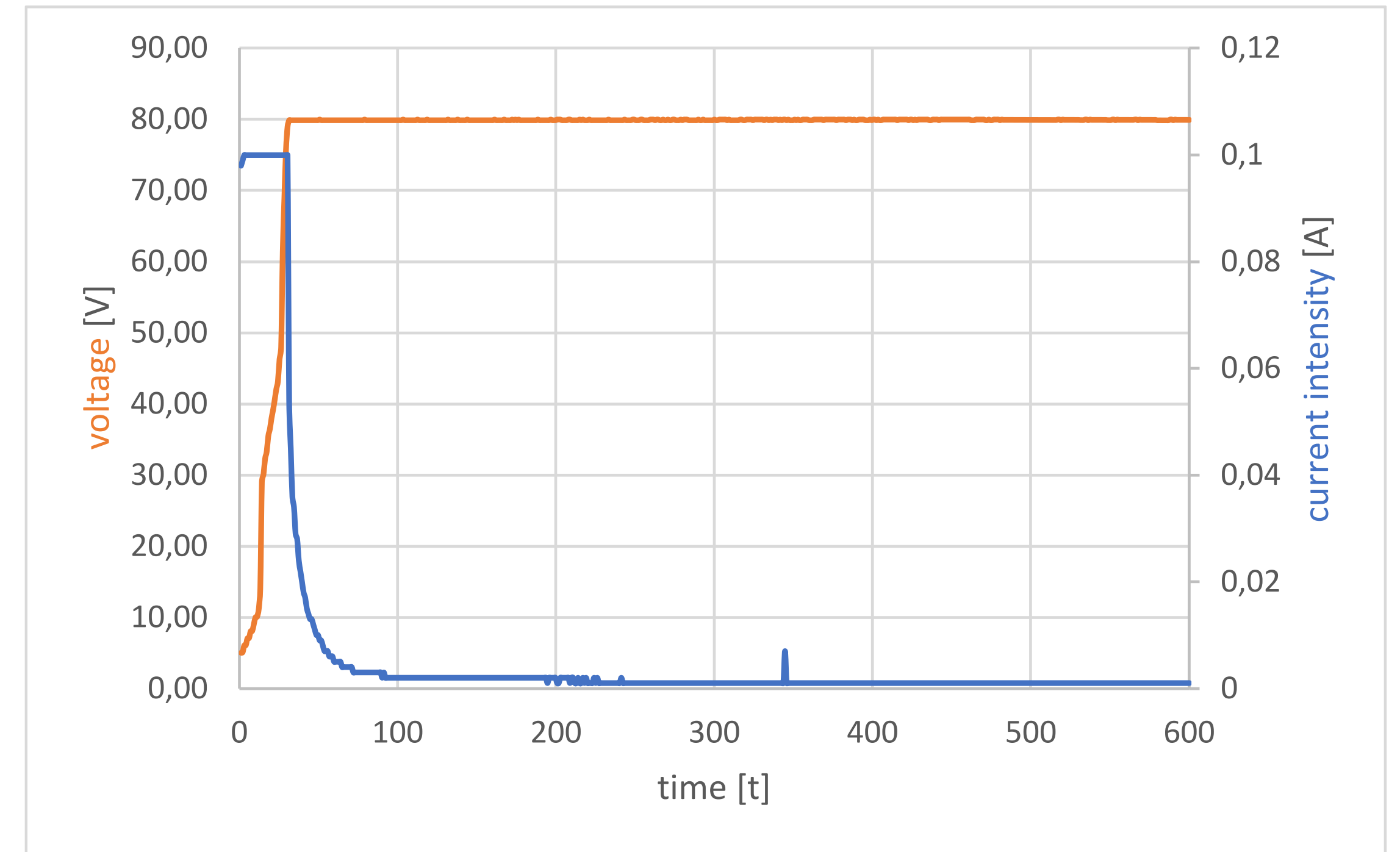
Solution	ND concentration, g/l	Current density (intensity), nA/ cm ²	Voltage, V
75% H ₃ PO ₄ (ethylene glycol)	5	400 (0.2A)	70, 80
75% H ₃ PO ₄ (ethylene glycol)	10	400 (0.2A)	80, 90, 100, 110, 120

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NiTi after grinding

NiTi after PEO, 70 V

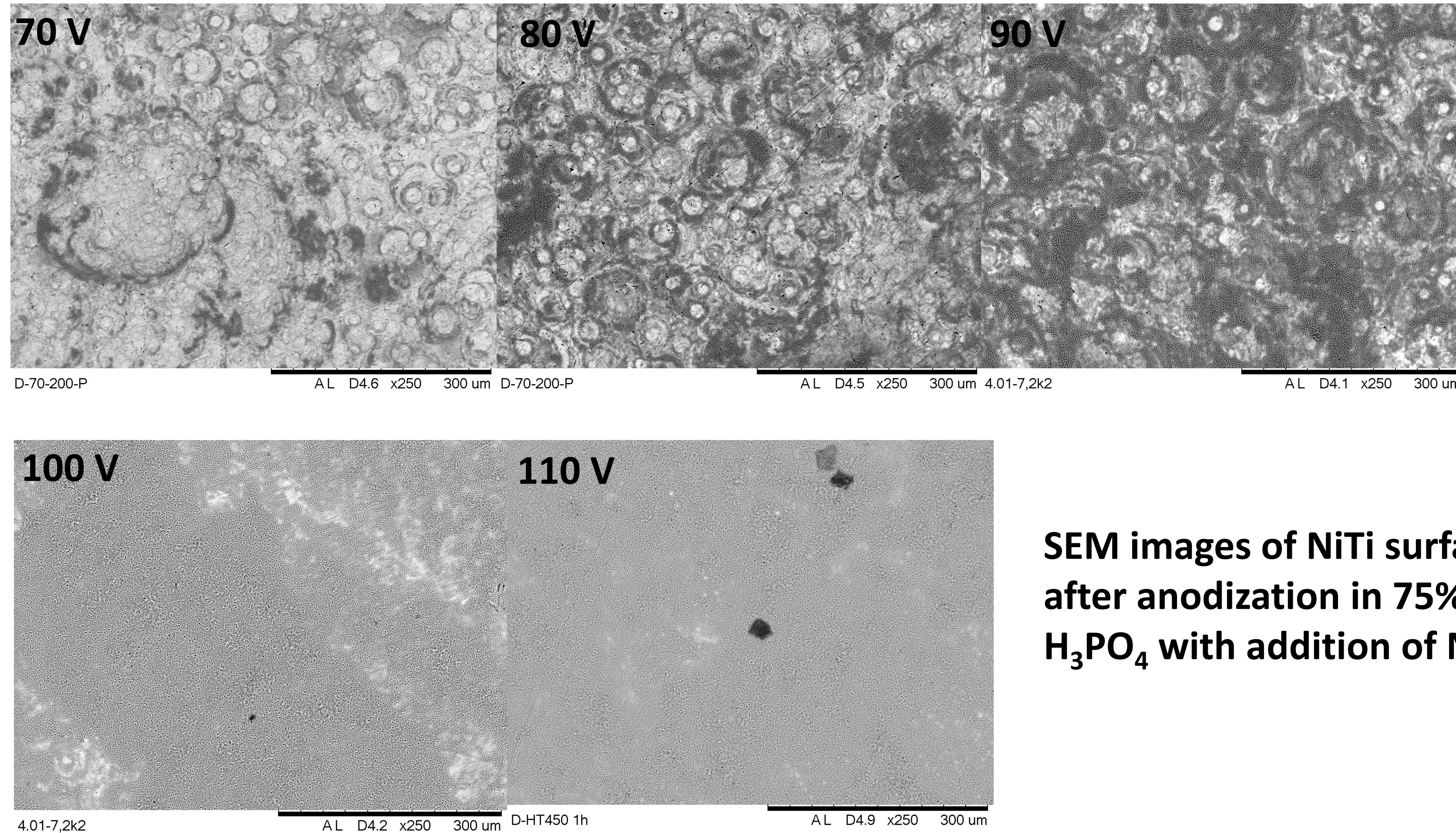


$$(U, I) = f(t)$$



NiTi ANODIZATION

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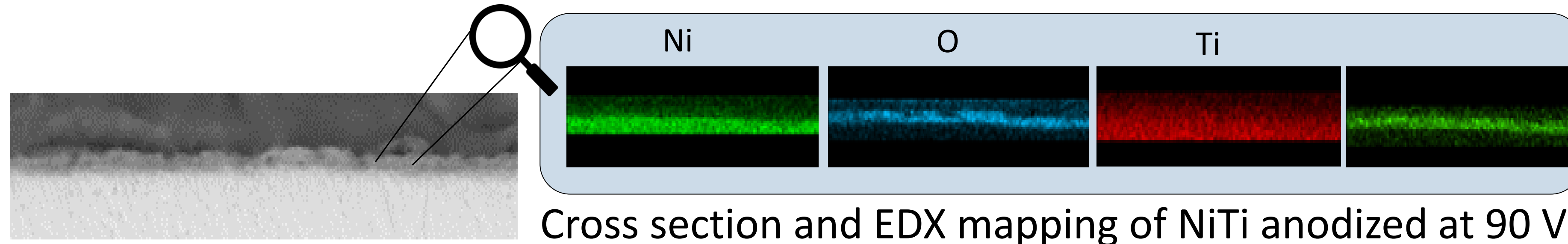


SEM images of NiTi surface after anodization in 75% H_3PO_4 with addition of ND



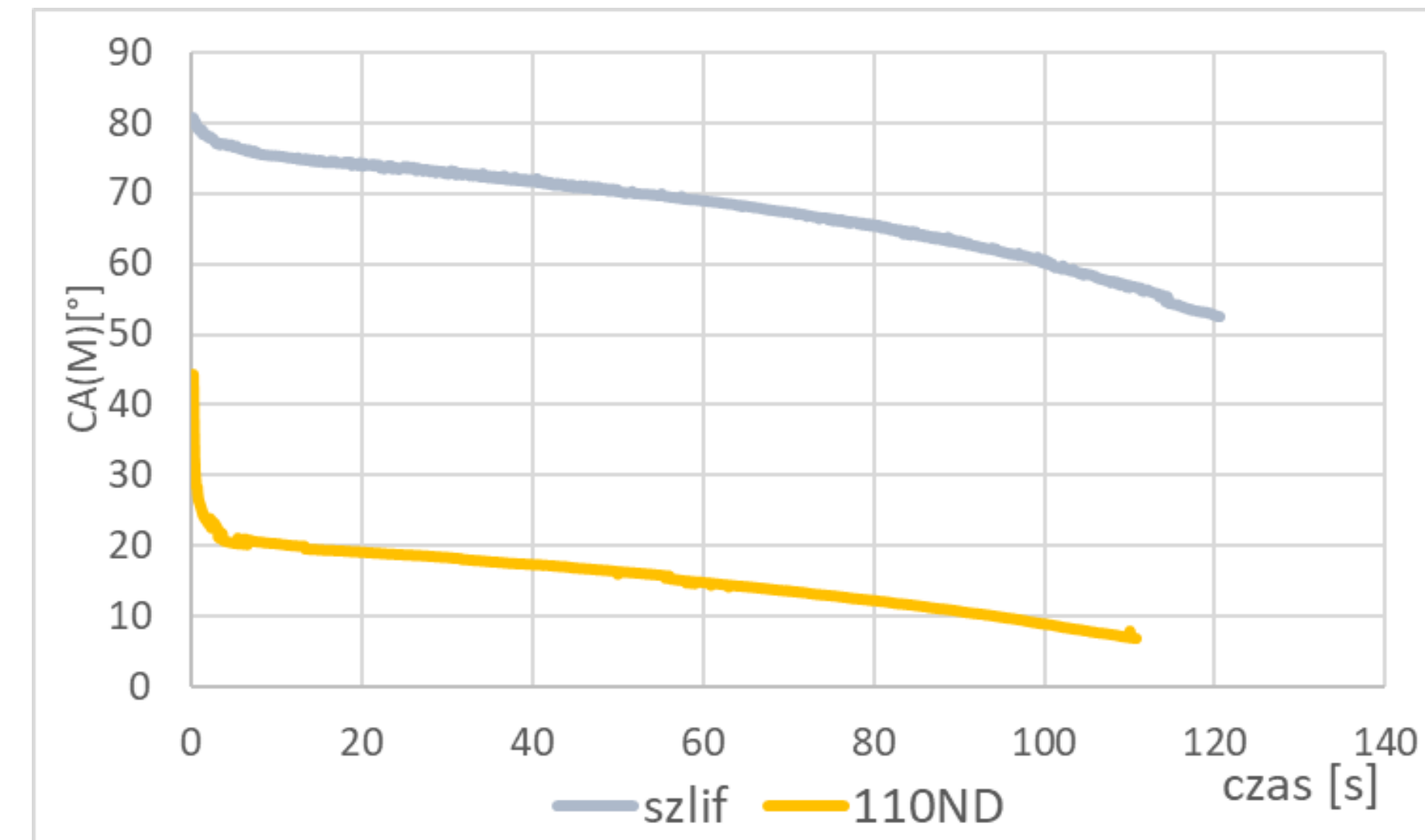
NiTi ANODIZATION

SUCCESS 😊



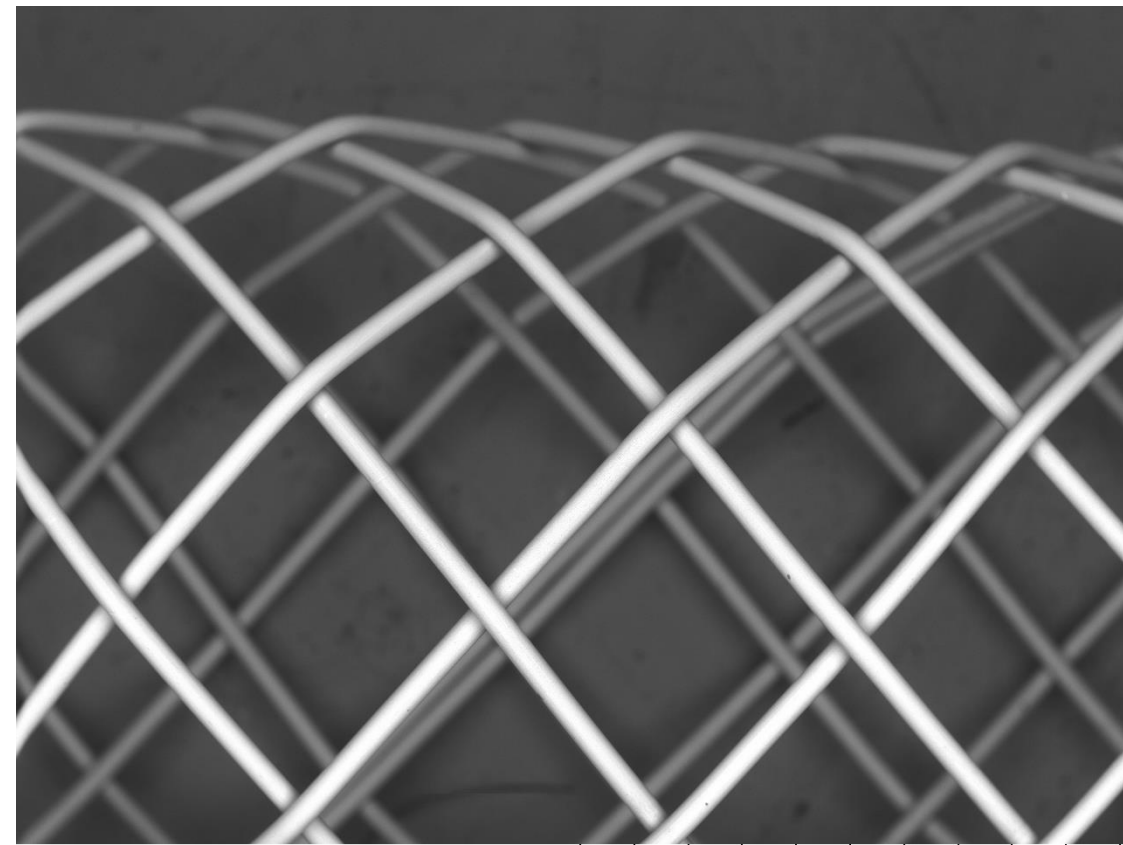
8

sample label	contact angle, °	image	roughness, μm		coating thickness, μm
			Ra	Rz	
grinded	80.83 (±1.58)		0.21 (±0.02)	1.52 (±0.25)	-
90	45.56 (±1.78)		0.30 (±0.06)	1.93 (±0.06)	13.8 (±1.9)
100	45.02 (±2.23)		0.31 (±0.03)	2.40 (±0.08)	16.6 (±1.6)
110	45.41 (±1.55)		0.50 (±0.08)	3.99 (±0.32)	17.8 (±1.2)
90ND	37.87 (±1.45)		0.24 (±0.05)	1.75 (±0.06)	17.4 (±1.5)
100ND	39.87 (±1.23)		0.33 (±0.05)	2.07 (±0.09)	19.2 (±1.9)
110ND	38.25 (±1.40)		0.34 (±0.07)	2.40 (±0.05)	20.3 (±1.8)

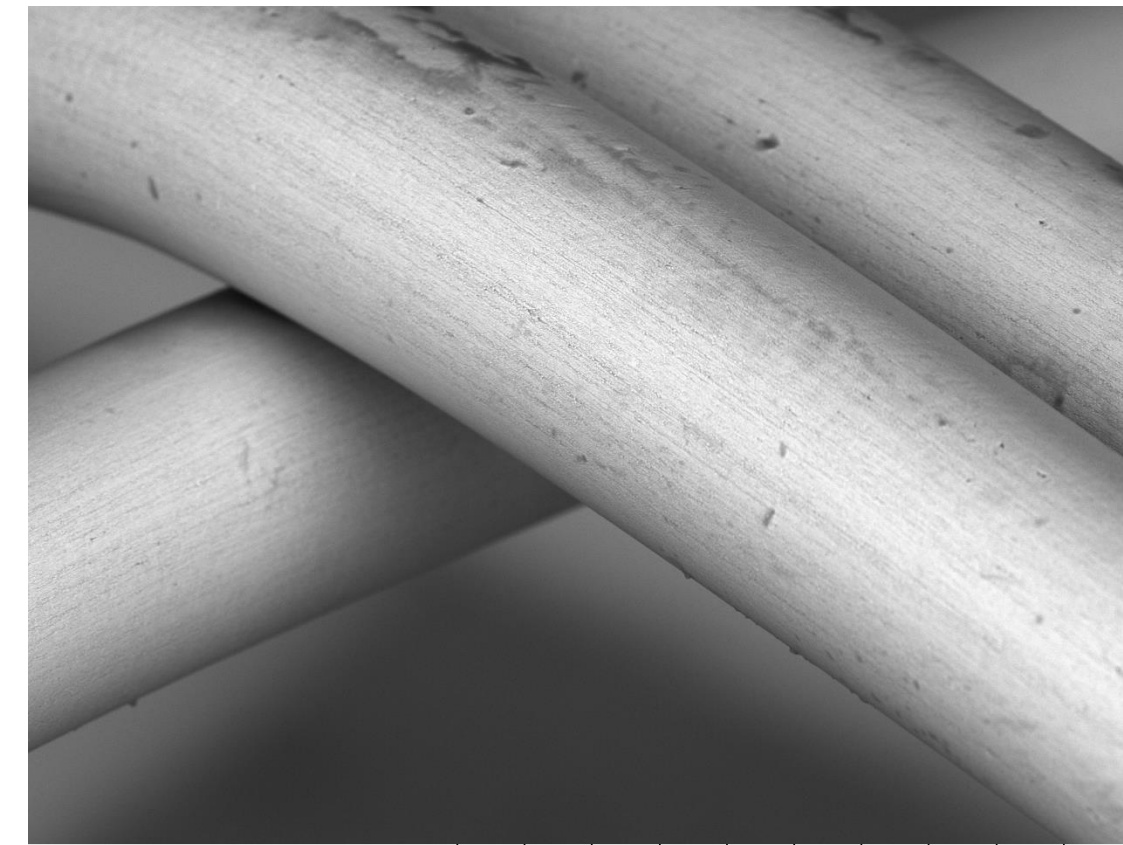


NiTi ANODIZATION

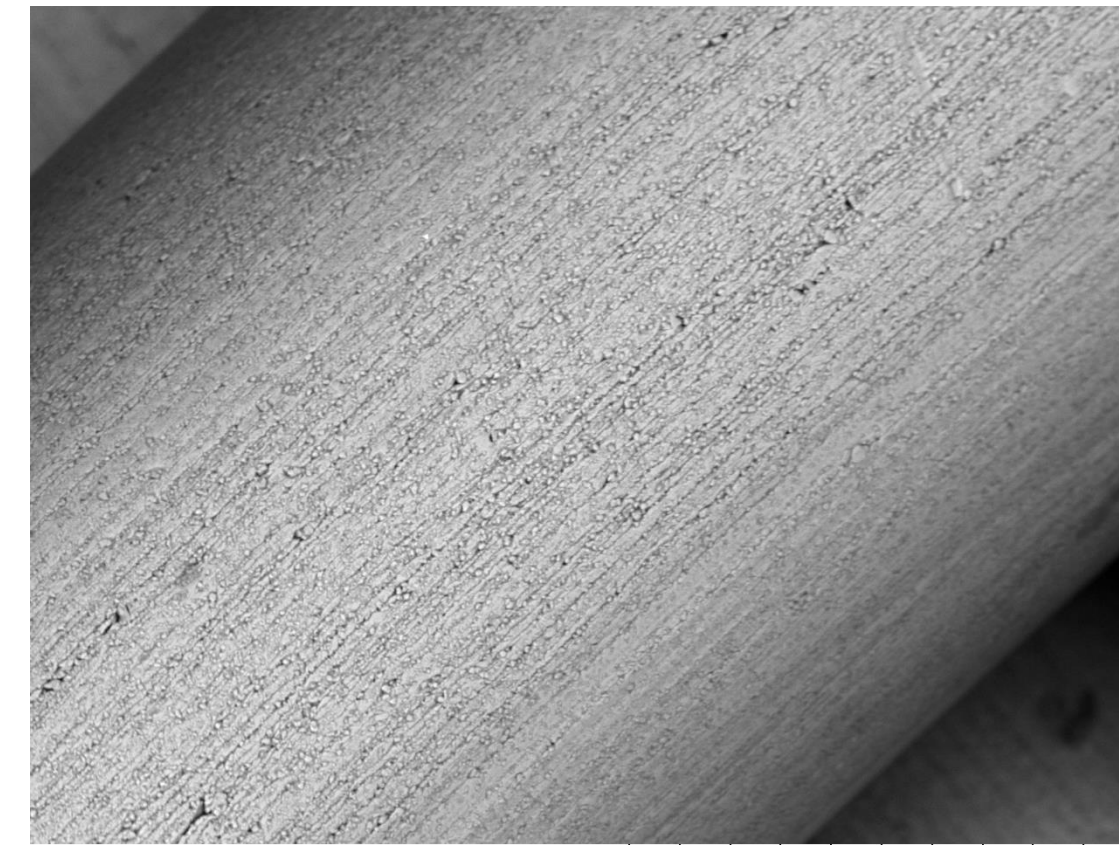
9



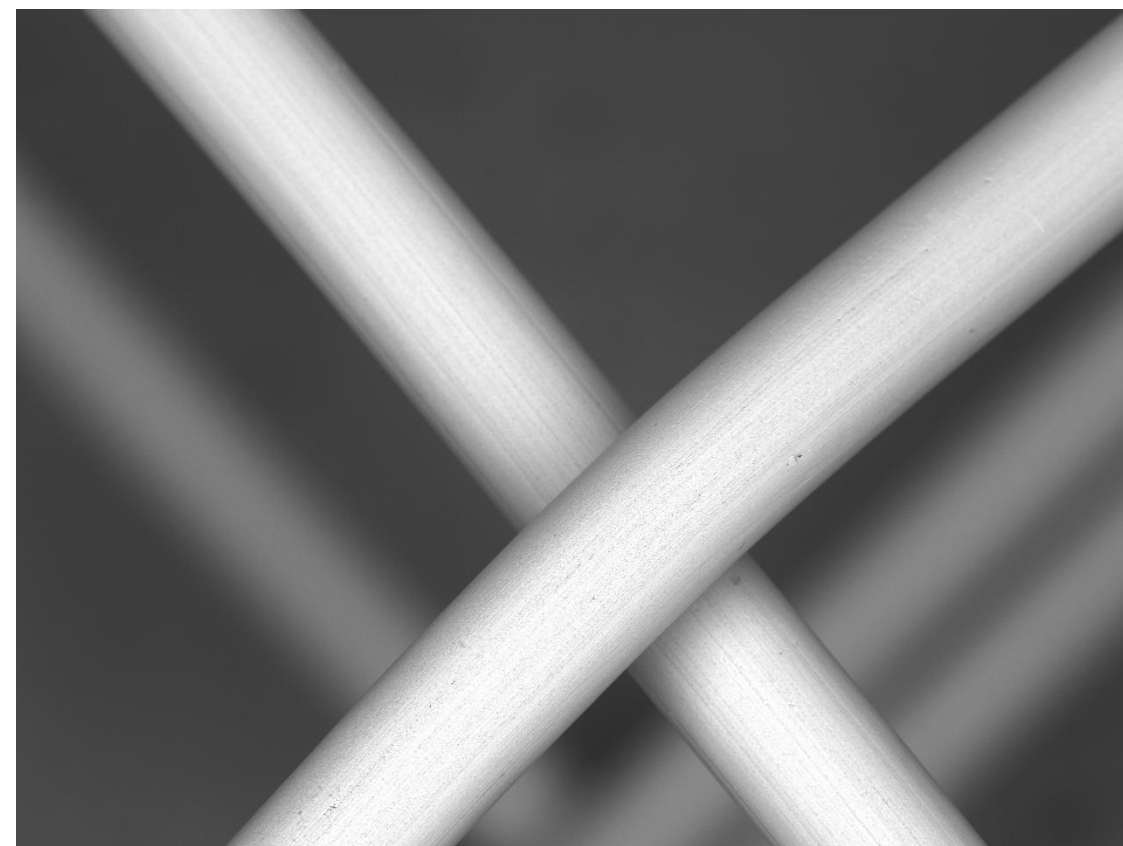
AZ91-F7 AL D5.8 x40 2 mm



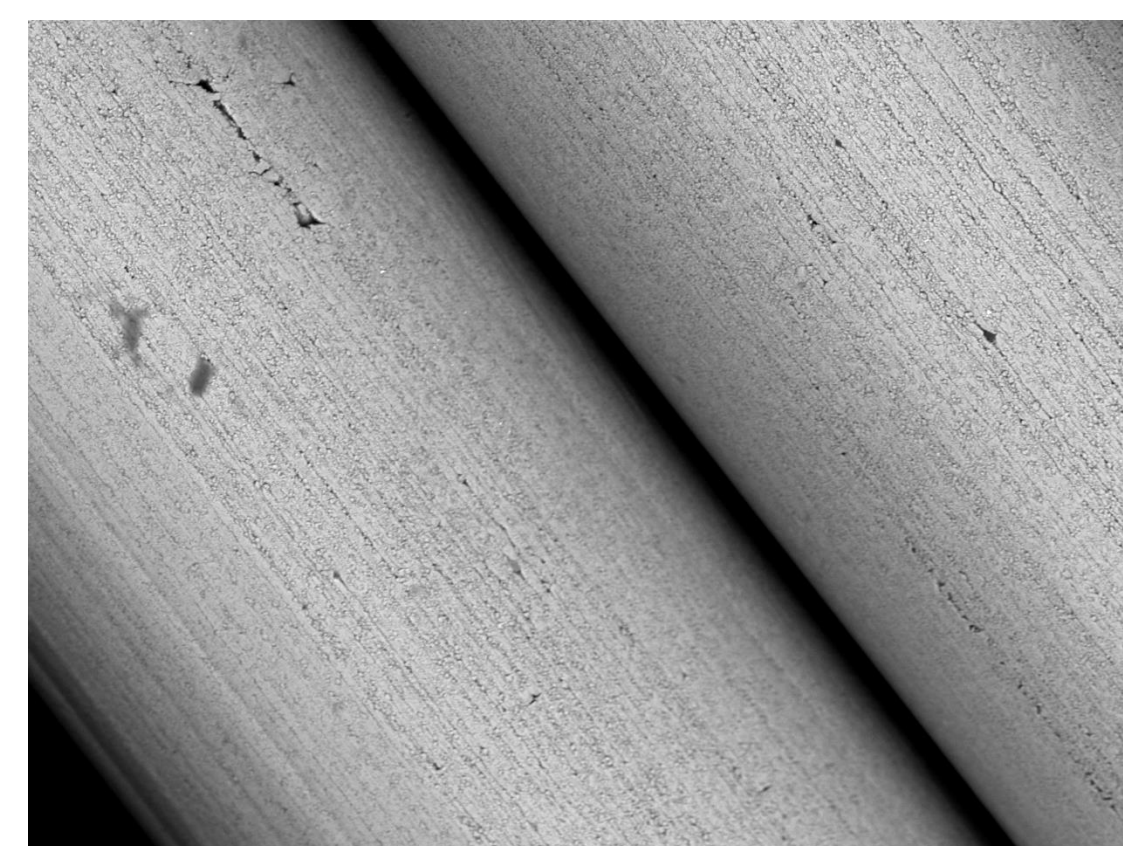
AL D6.1 x500 200 um



AZ91-F7 AL D5.8 x1.5k 50 um



AZ91-F7 AL D5.8 x250 300 um



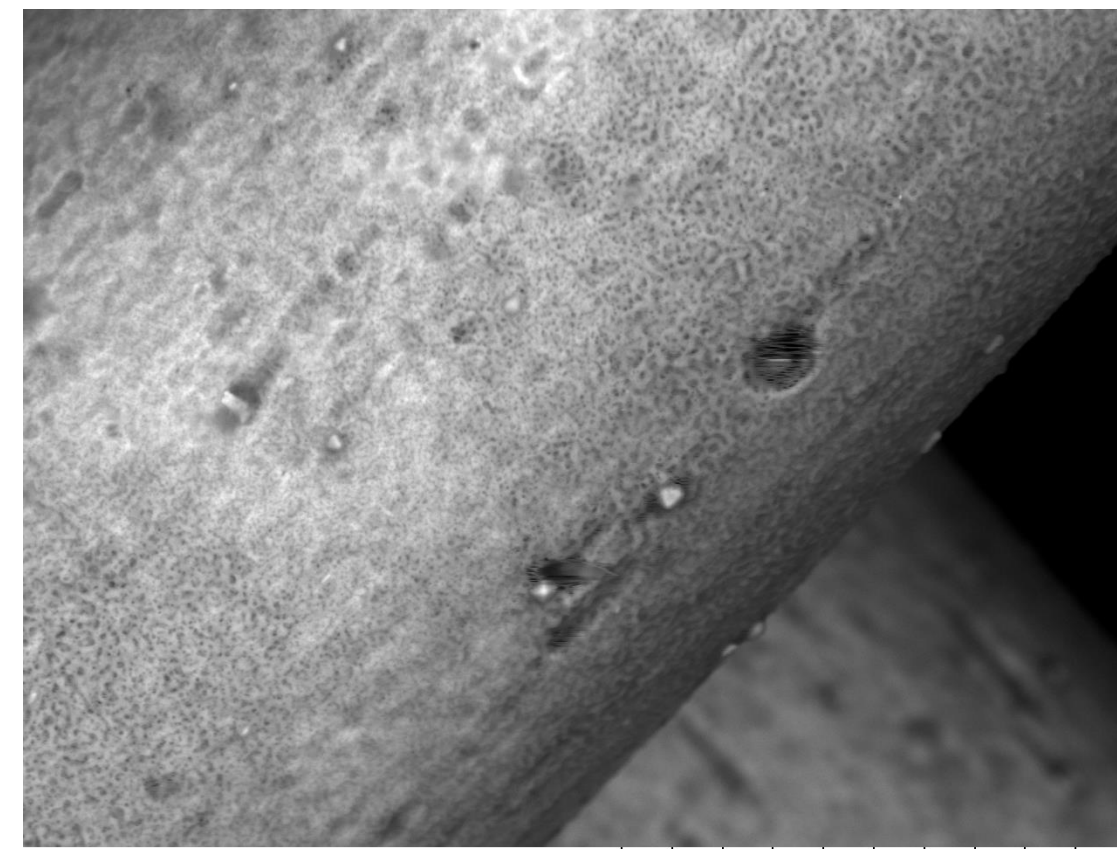
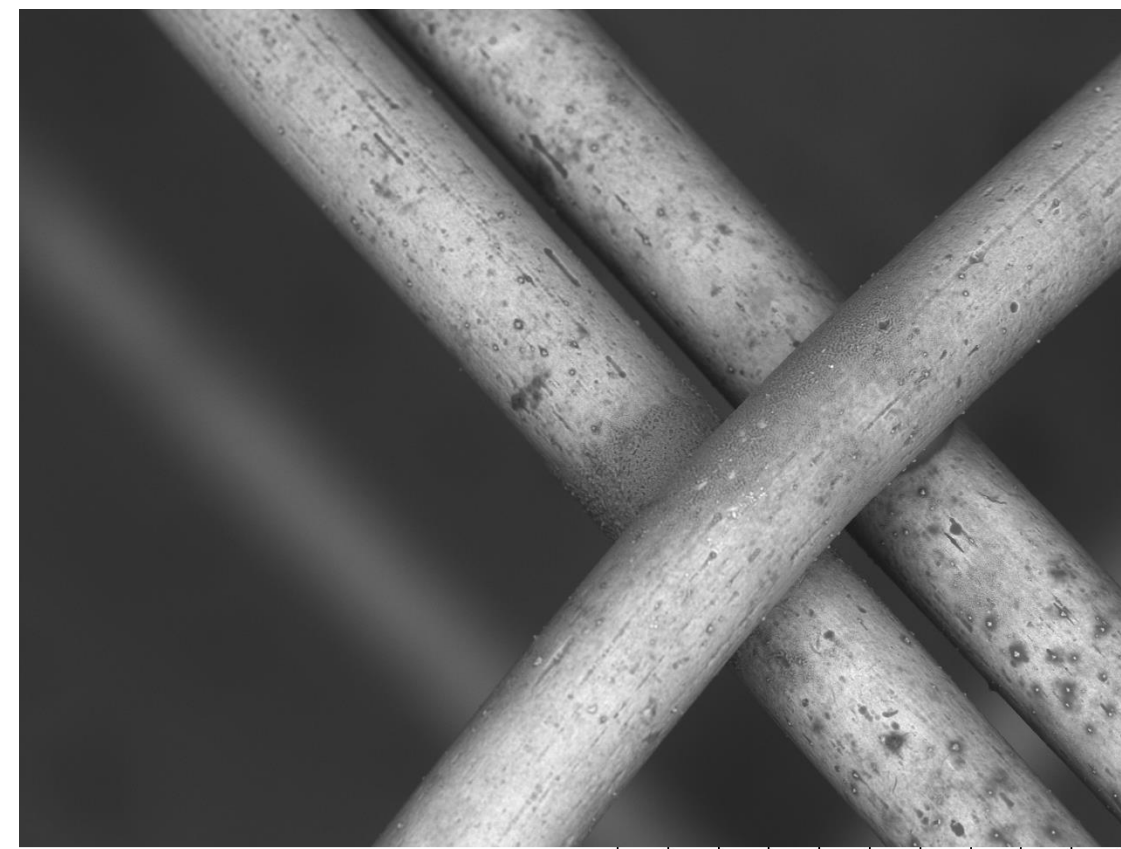
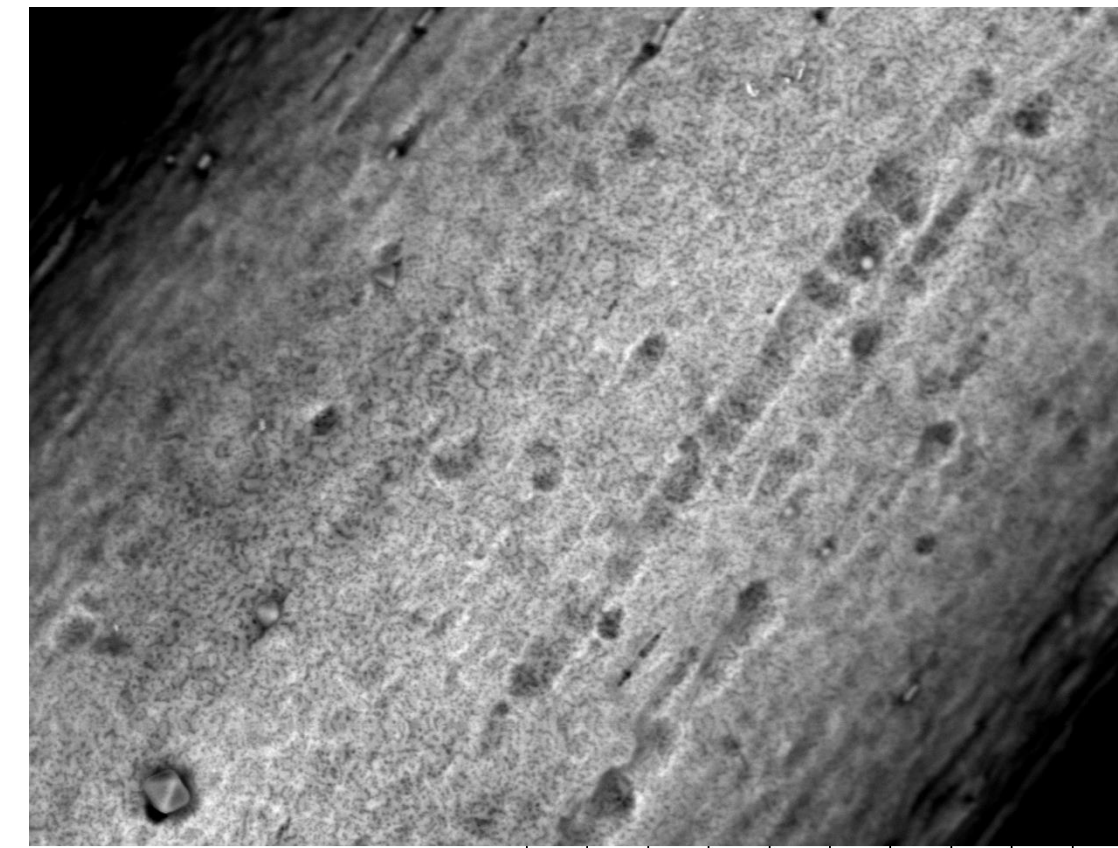
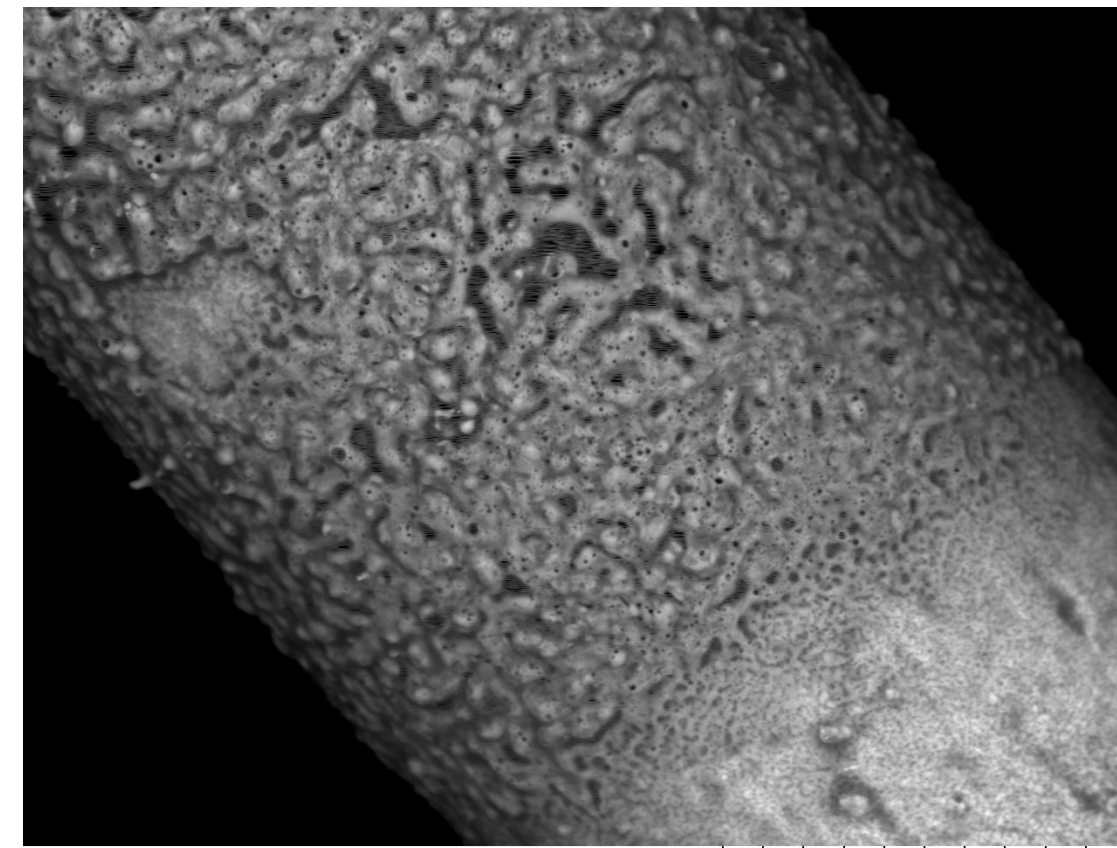
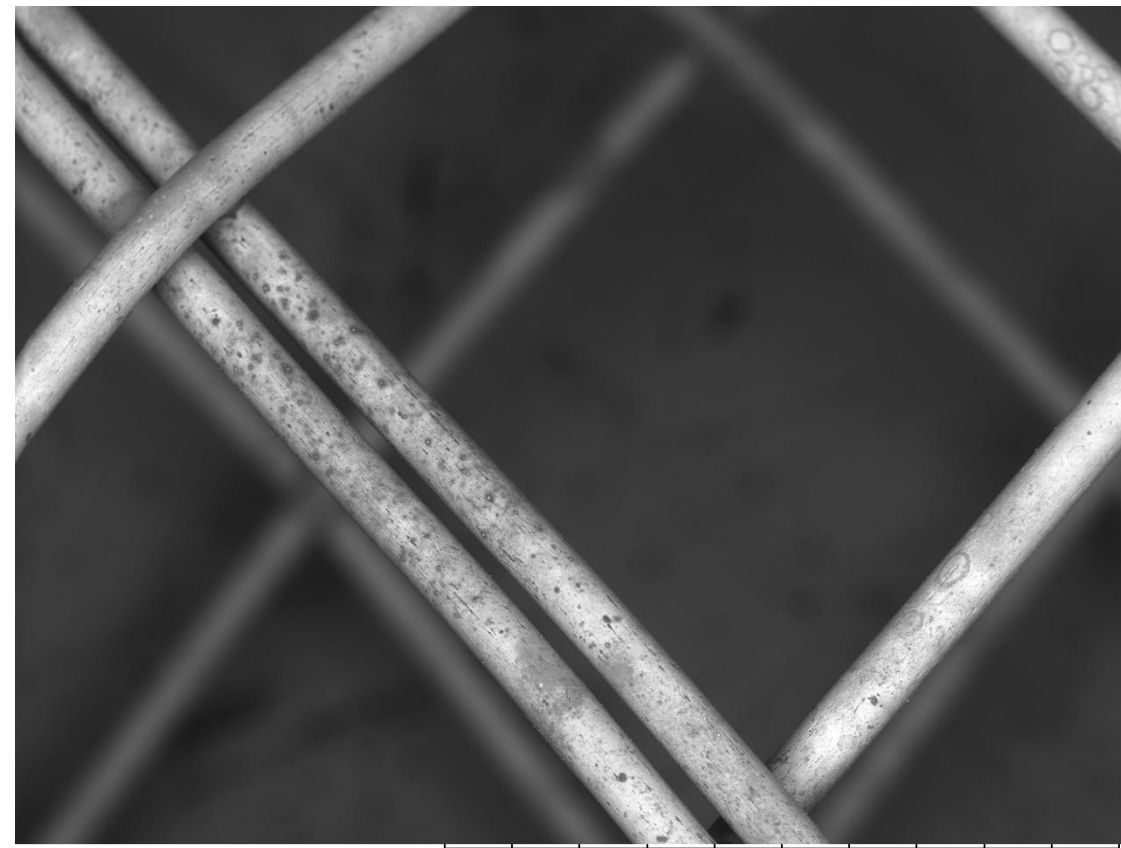
AZ91-F7 AL D5.7 x1.0k 100 um

SEM images of stent before PEO



NiTi ANODIZATION

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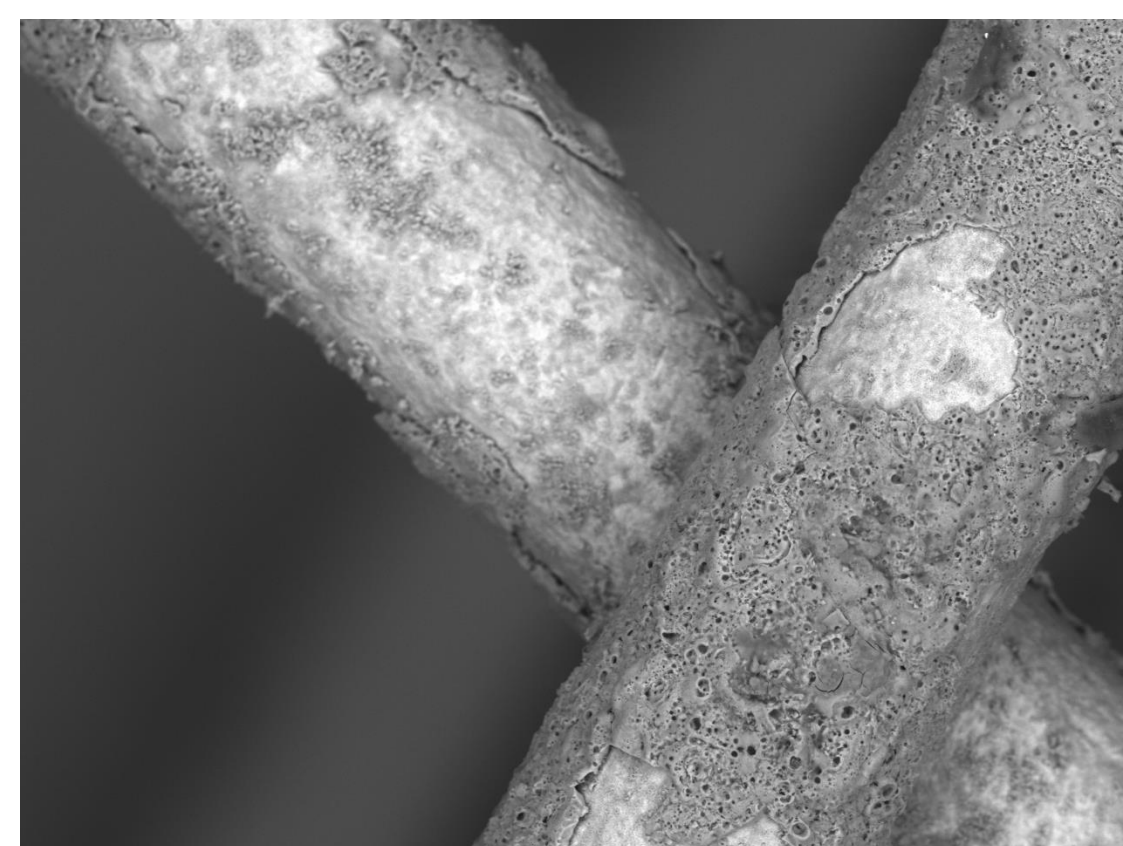
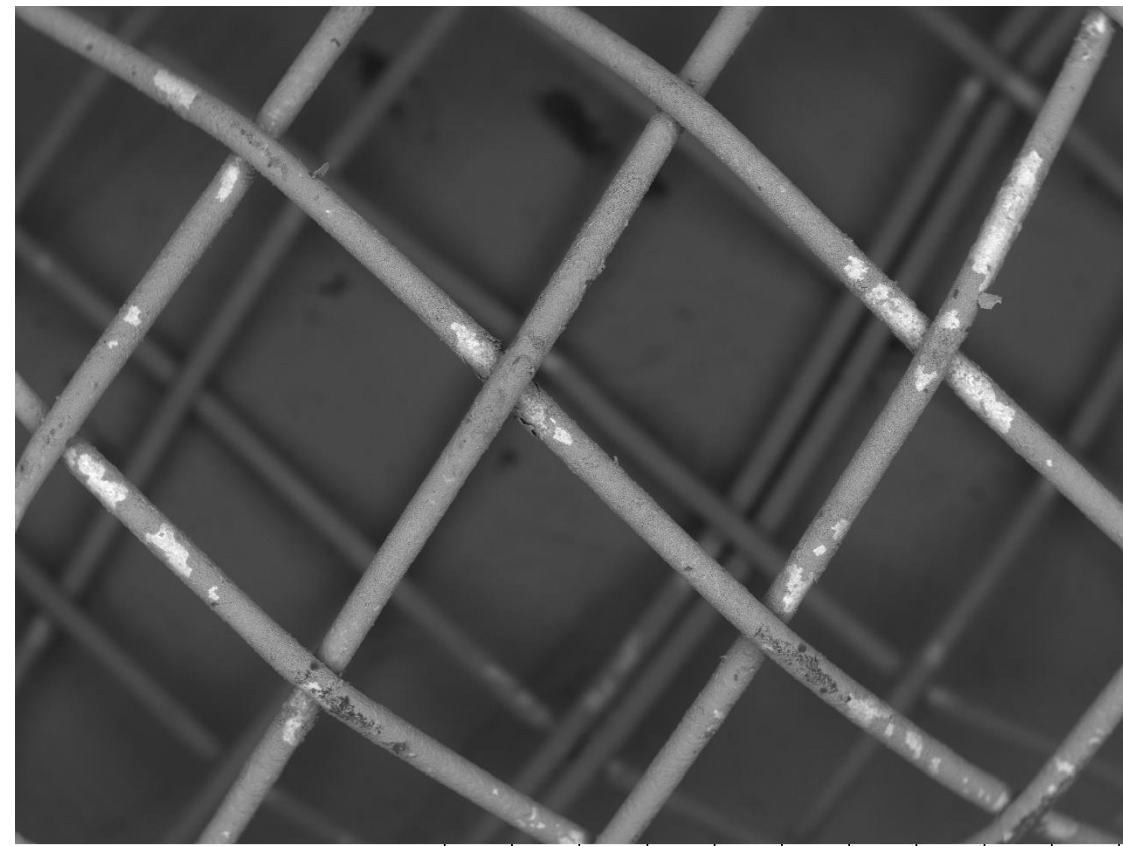
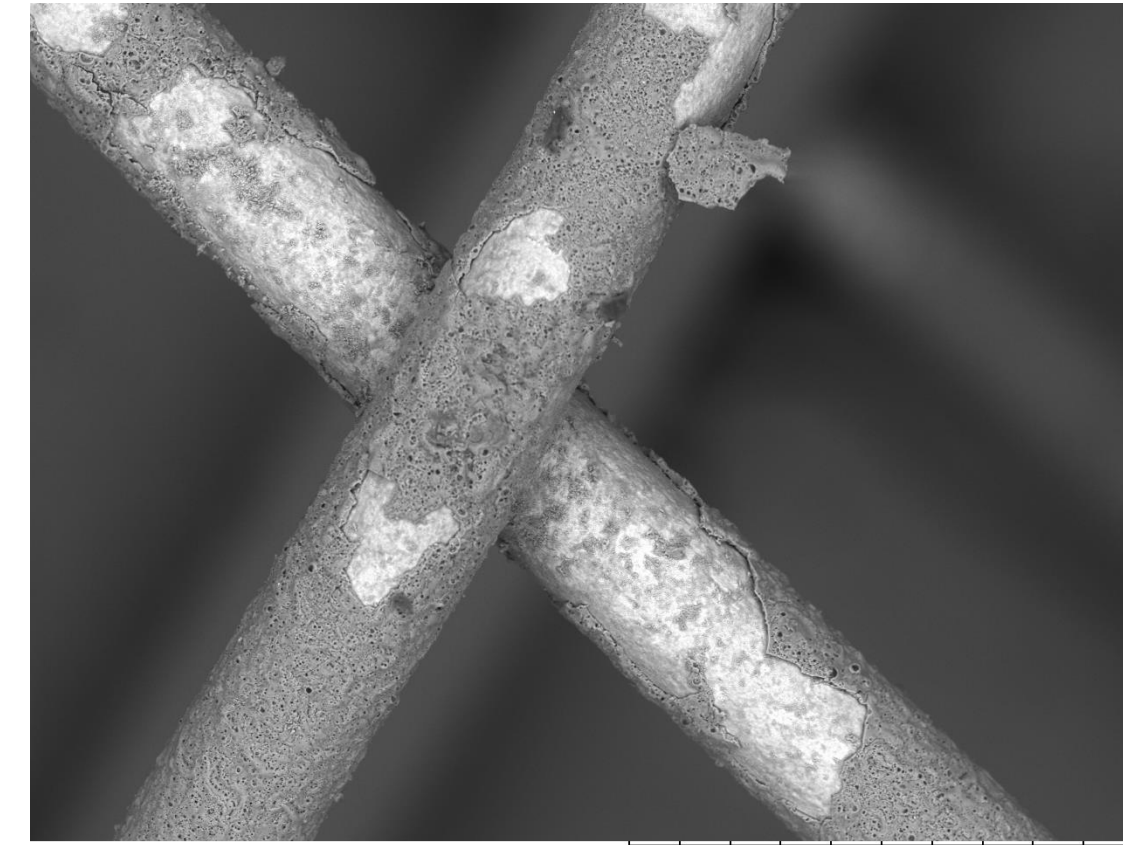
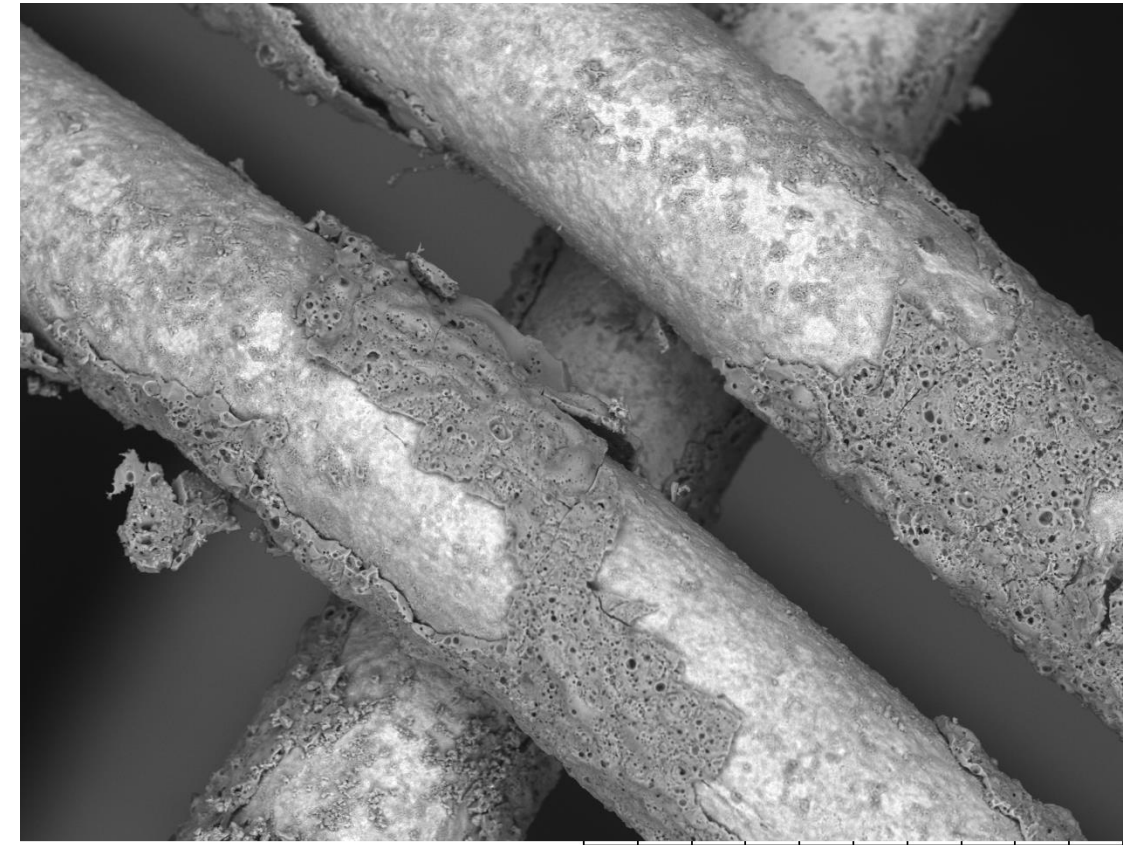
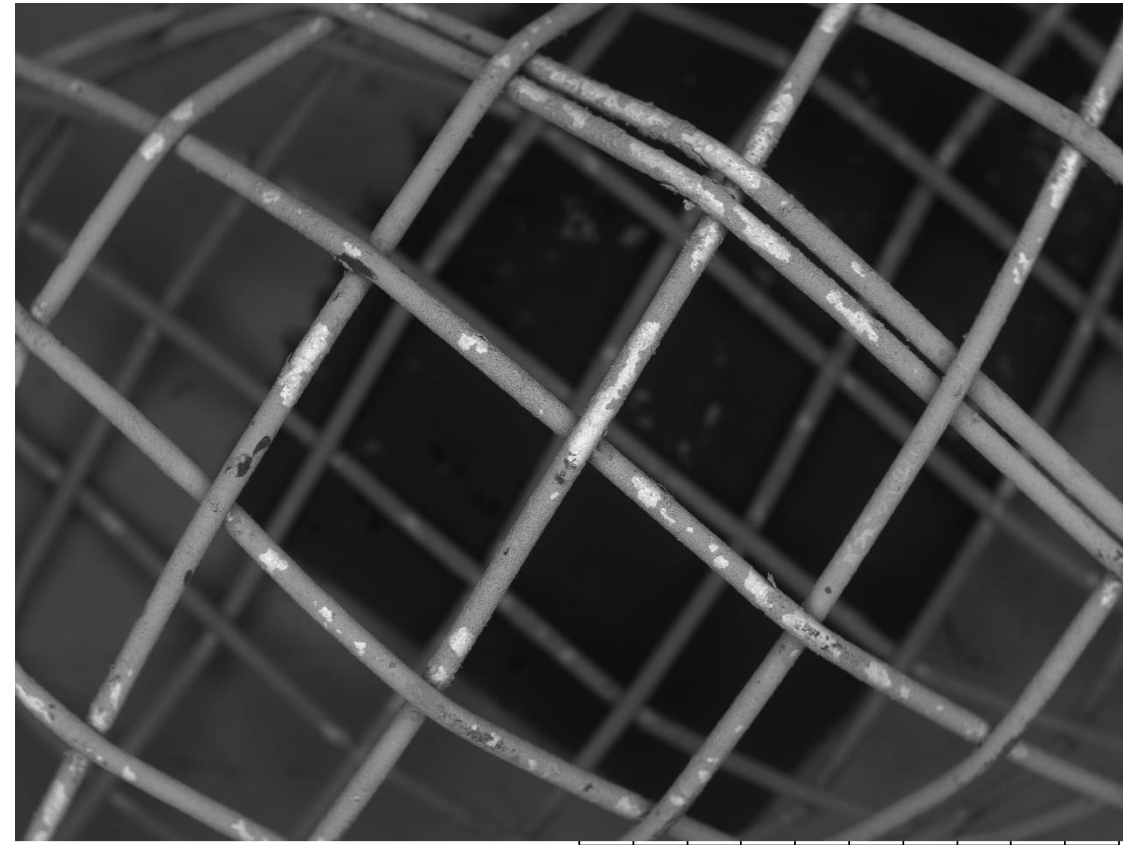
SEM images of stent after PEO

SUCCESS ???



NiTi ANODIZATION

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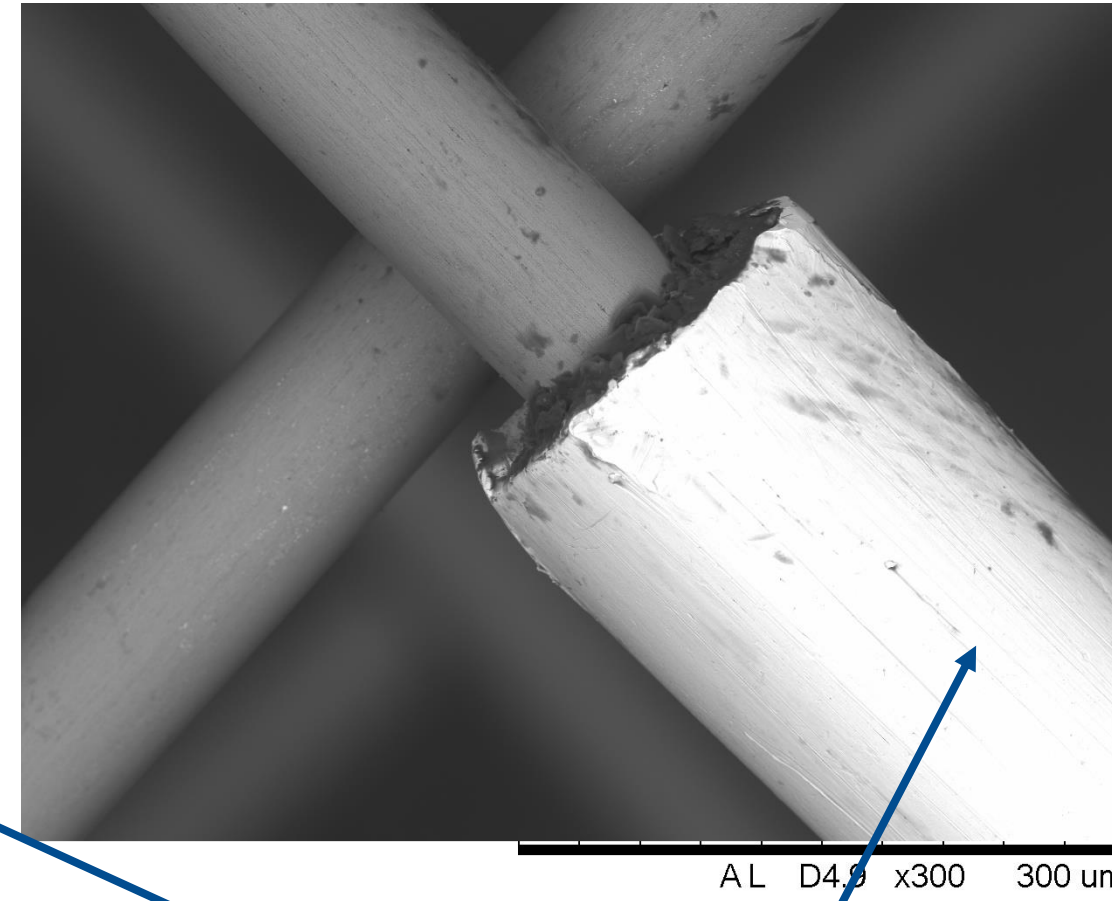
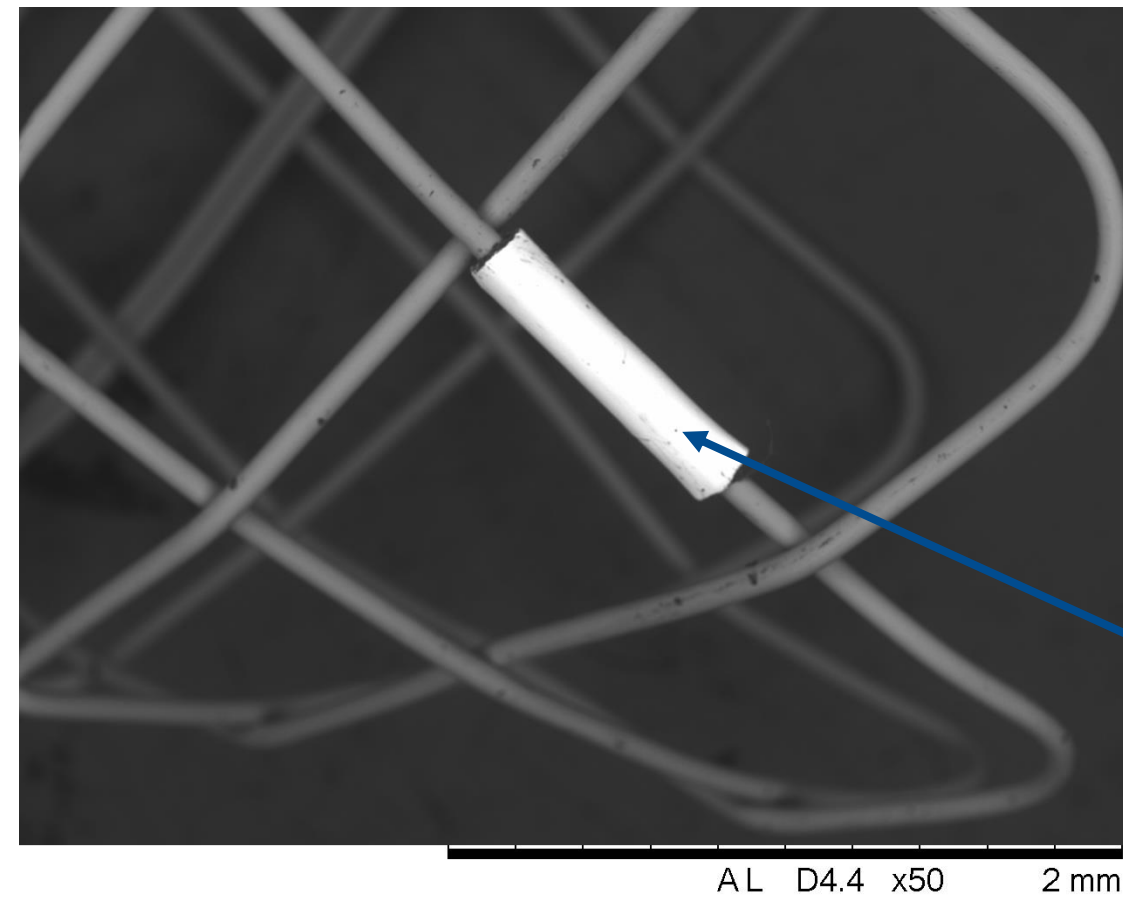
SEM images of stent after PEO and deformation

FAILED 😞

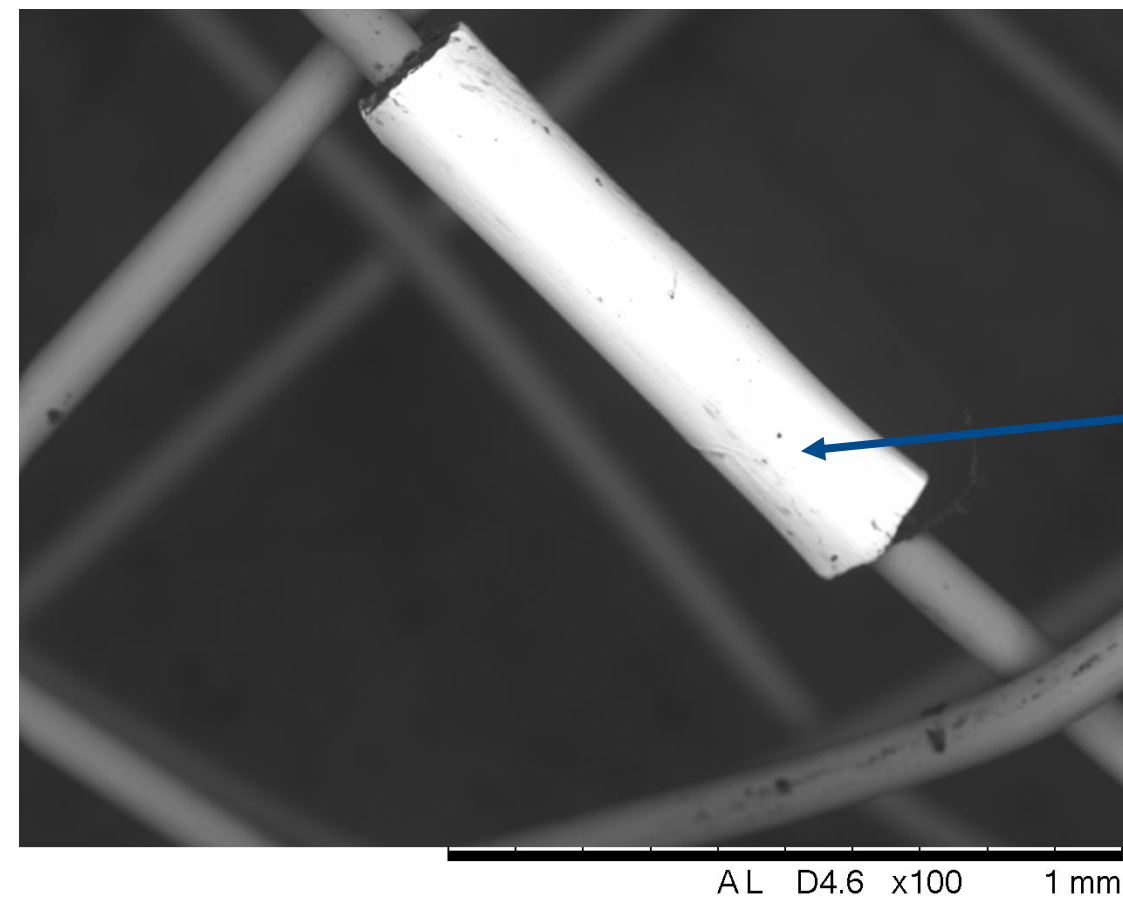


NiTi ANODIZATION

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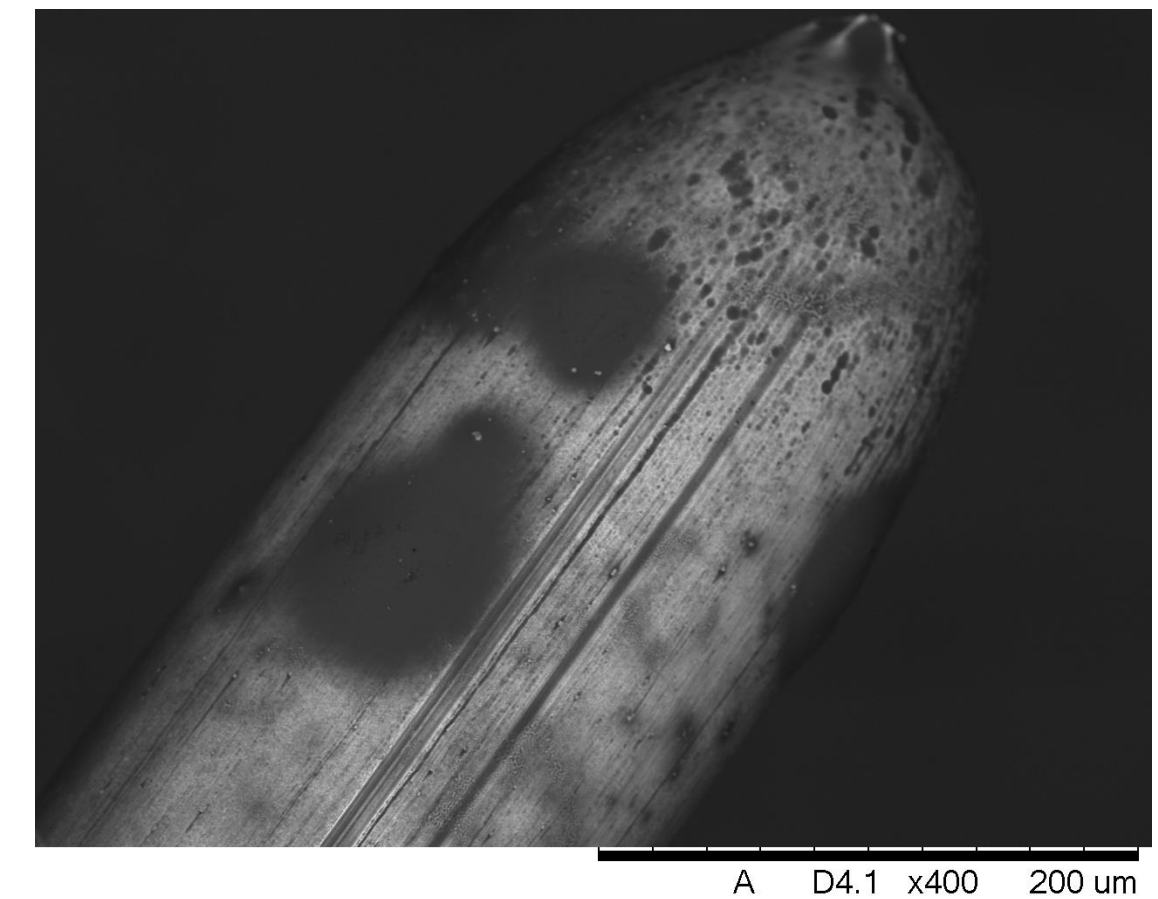


SEM images of stent after PEO

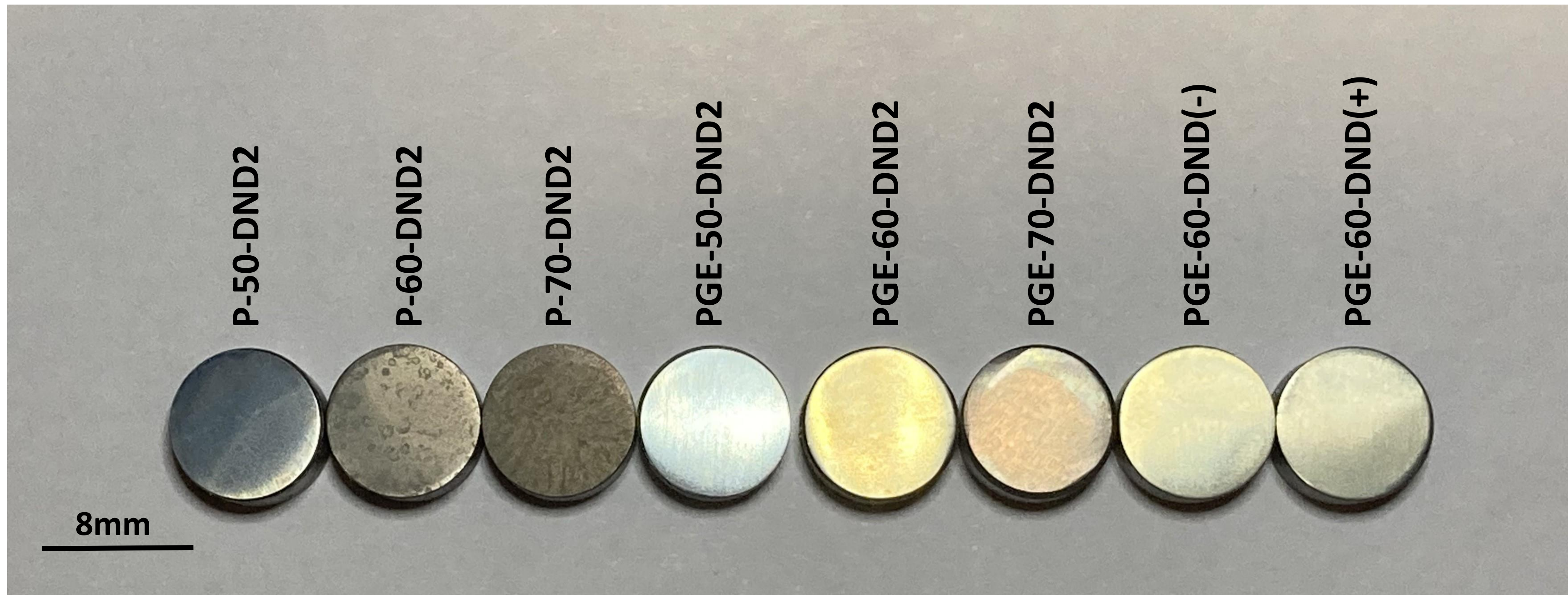


SEM images of Pt marker

Pt = O₂ evolution or electrolyte degradation



NiTi PEO PROCESS-NANODIAMONDS



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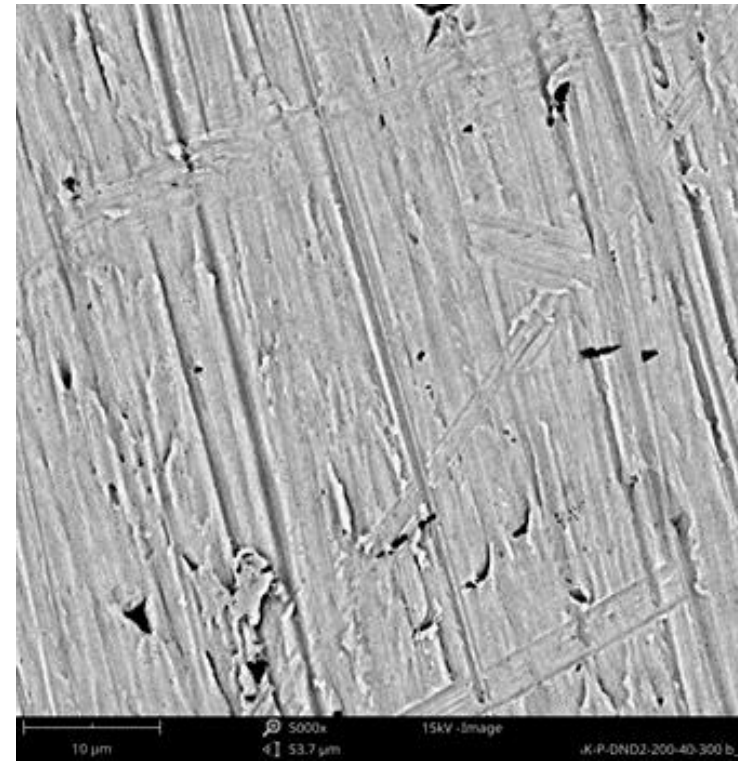


NiTi PEO PROCESS-NANODIAMONDS

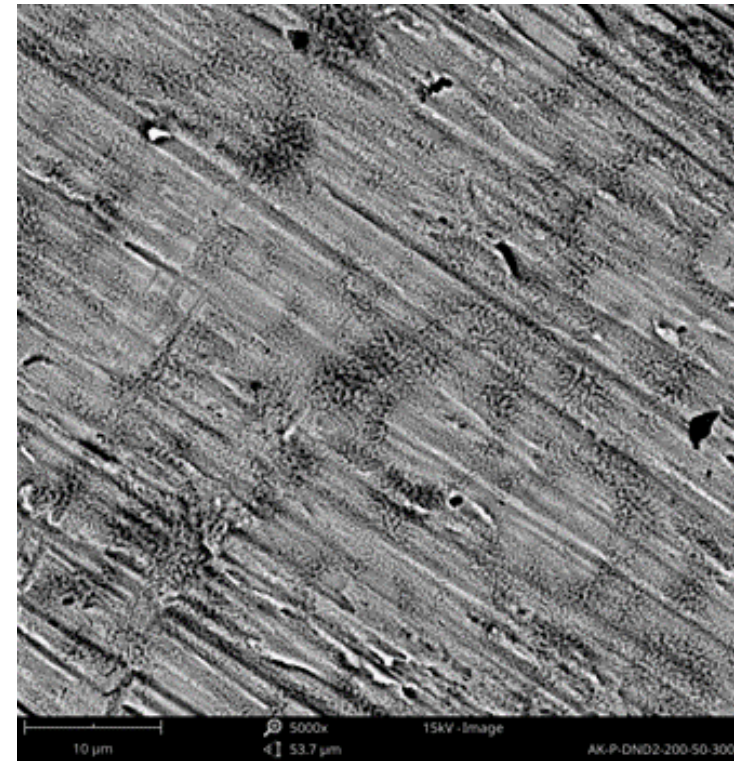
14

P-DND2-200

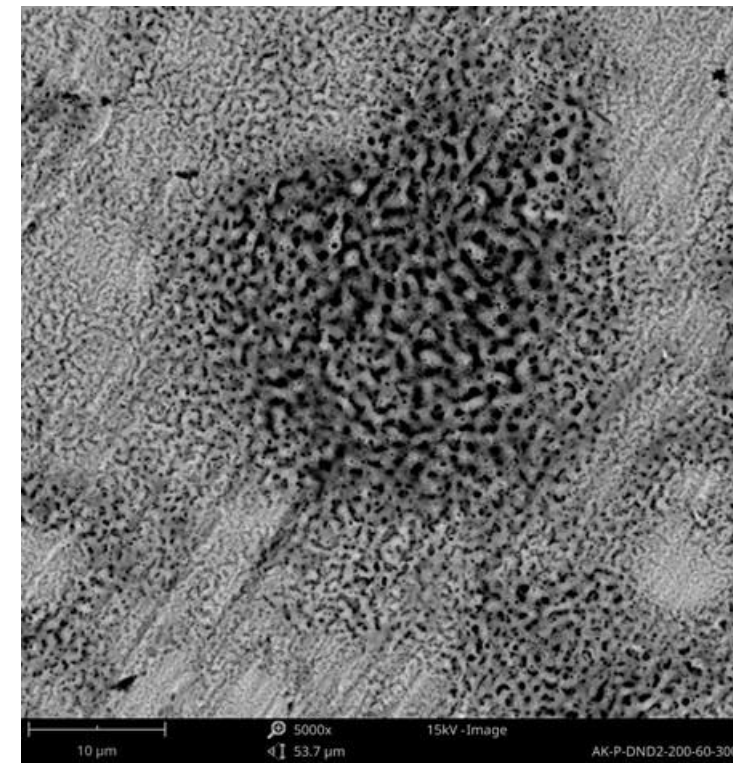
40V



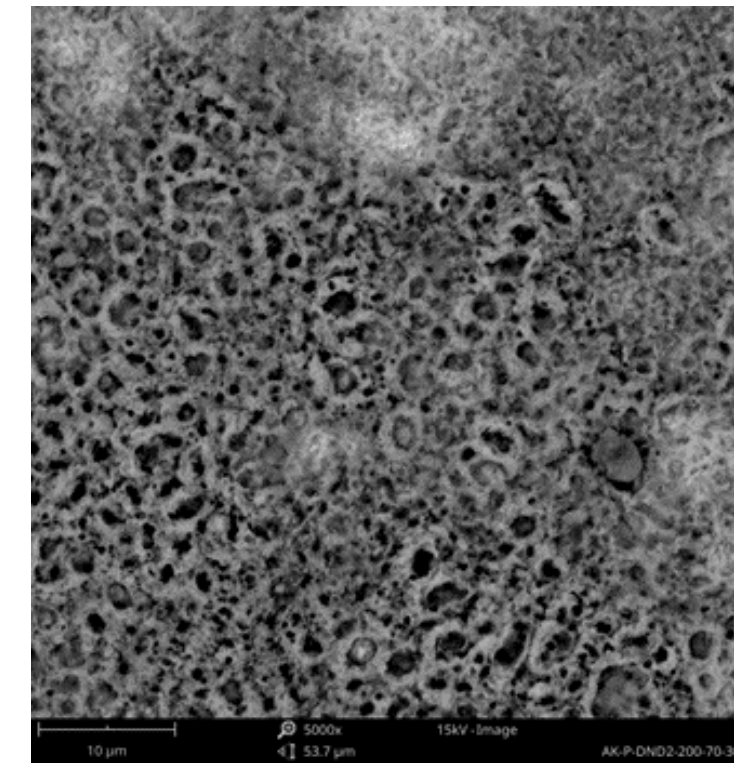
50V



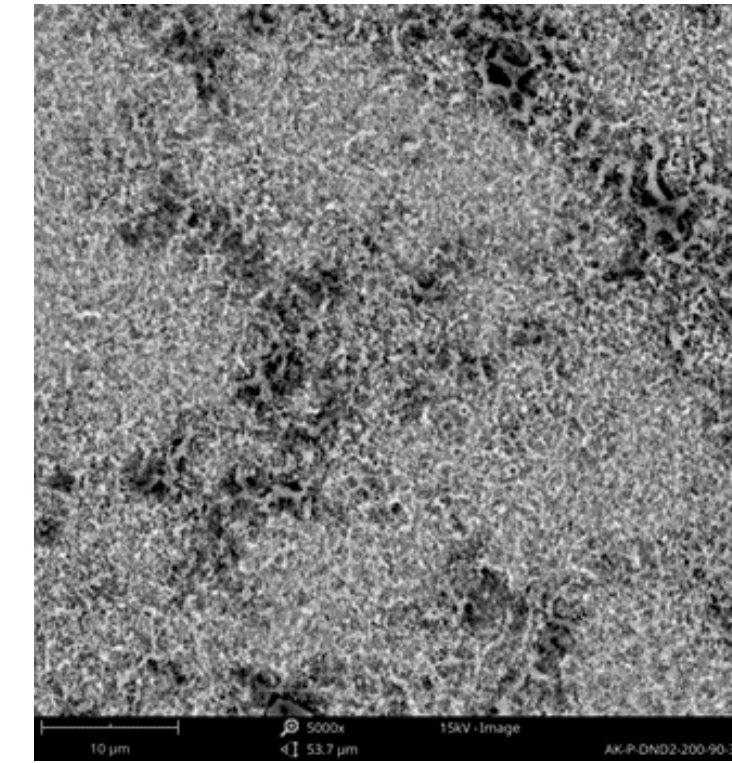
60V



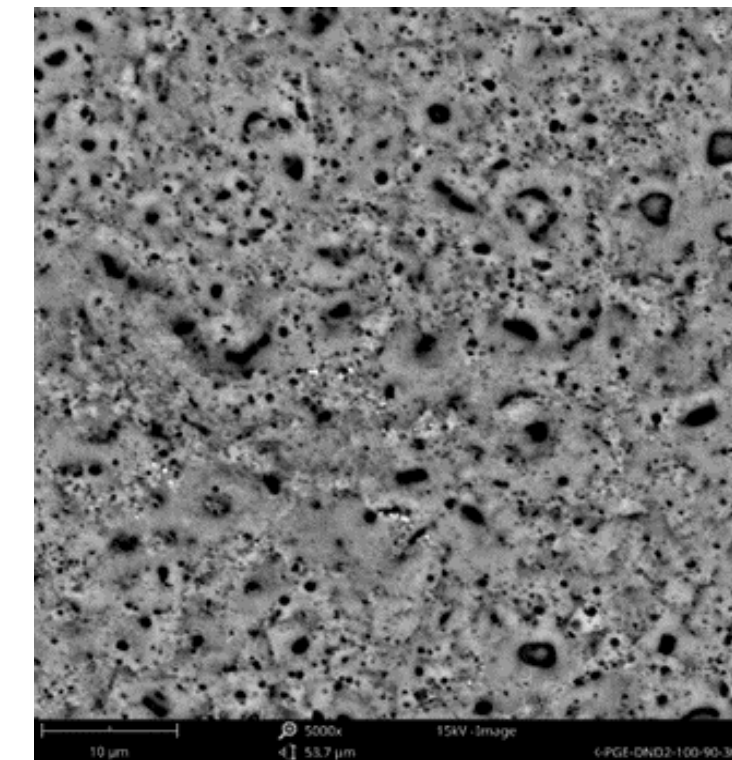
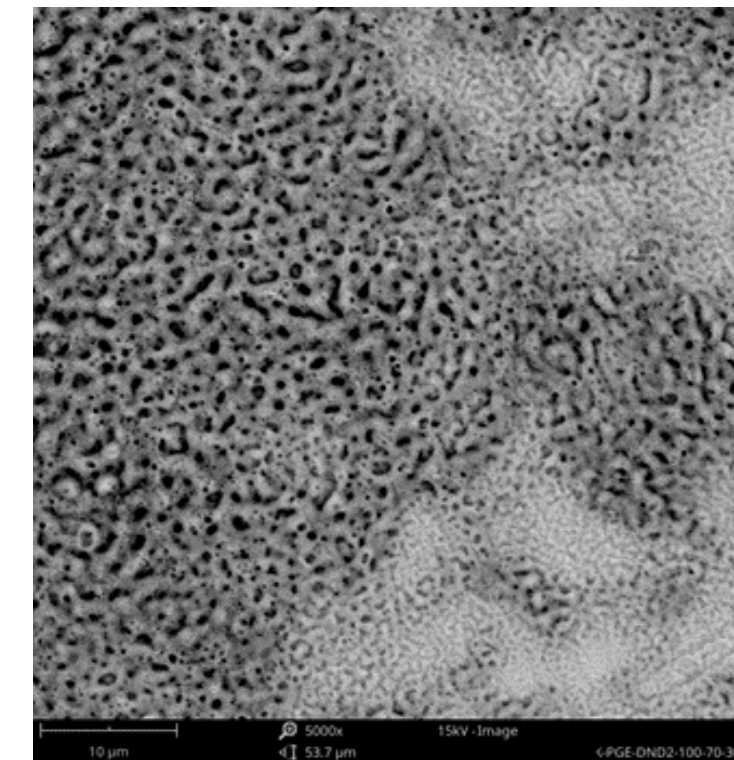
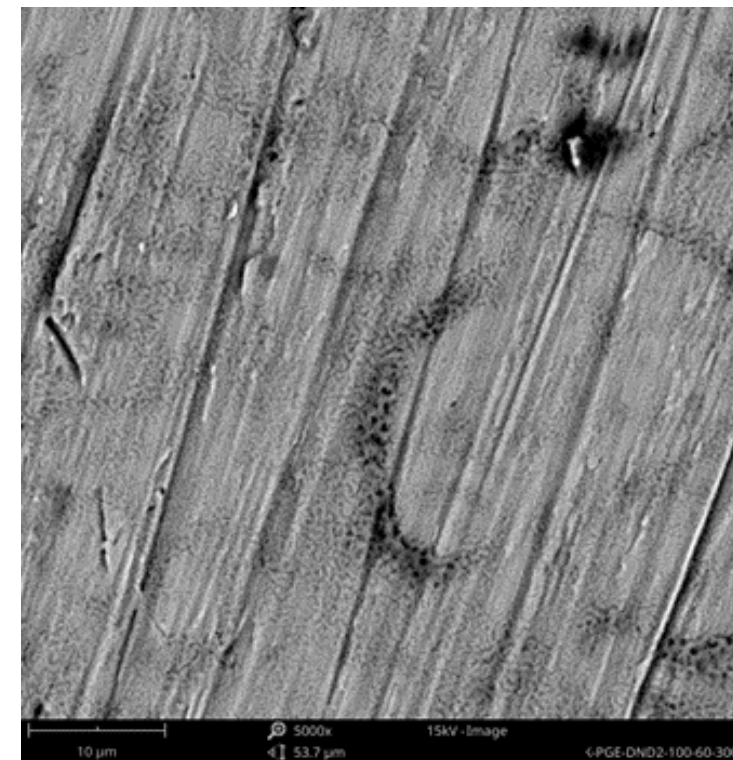
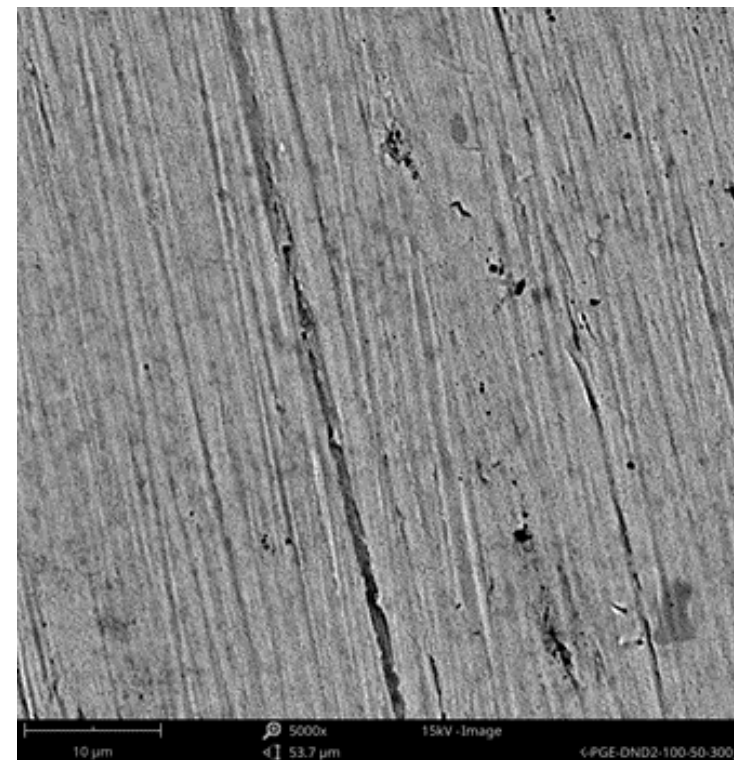
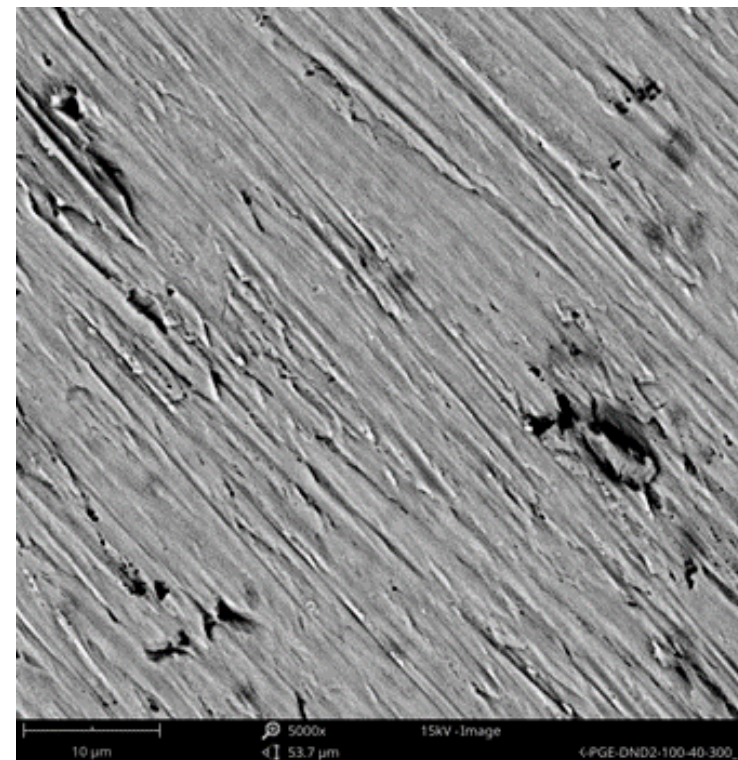
70V



90V



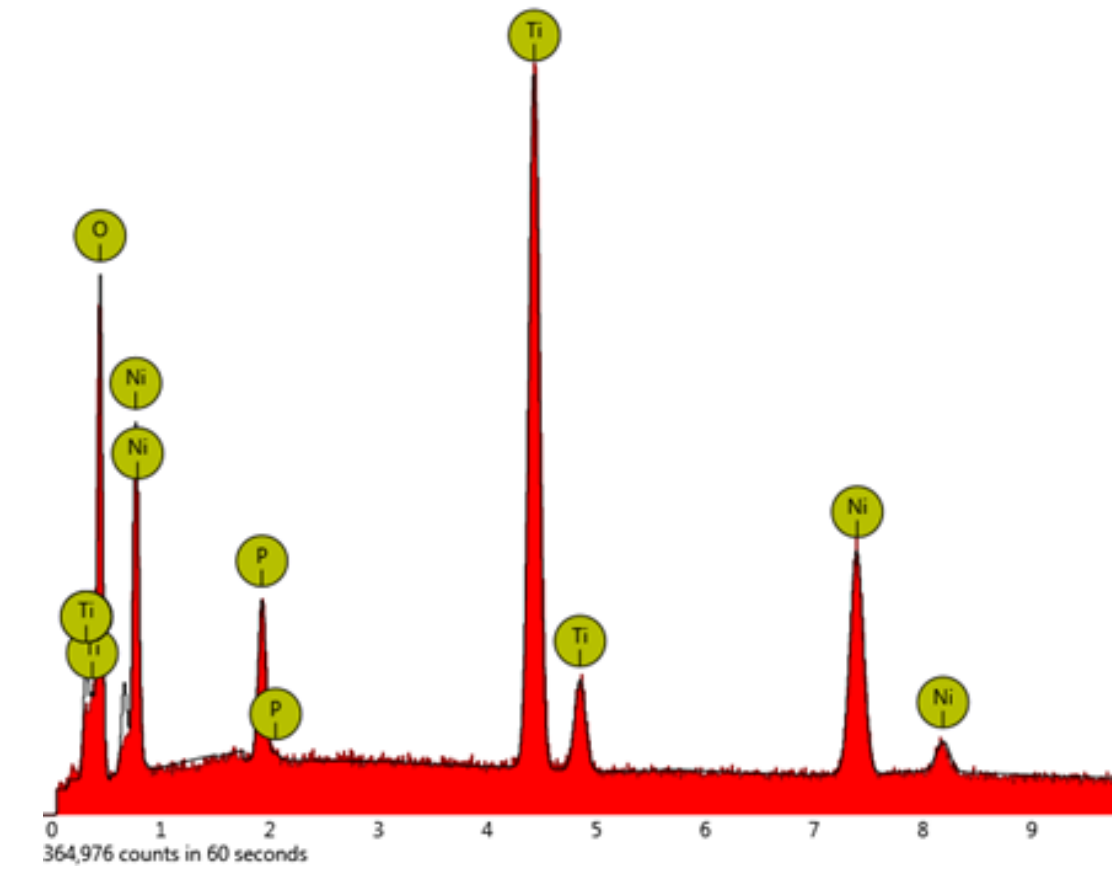
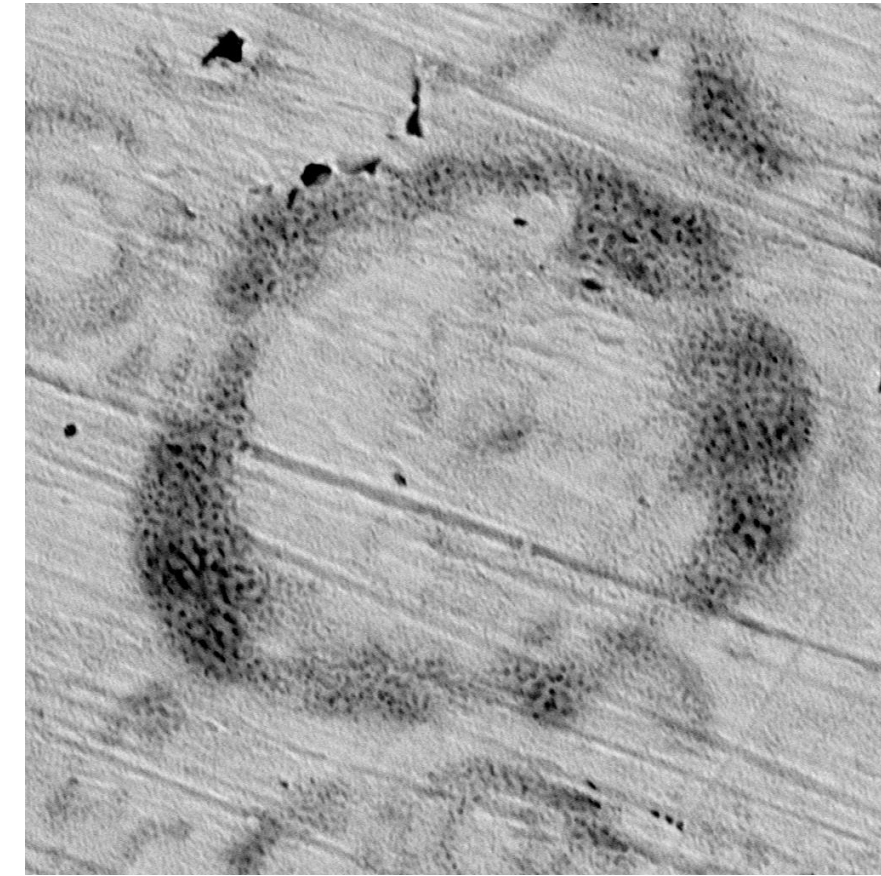
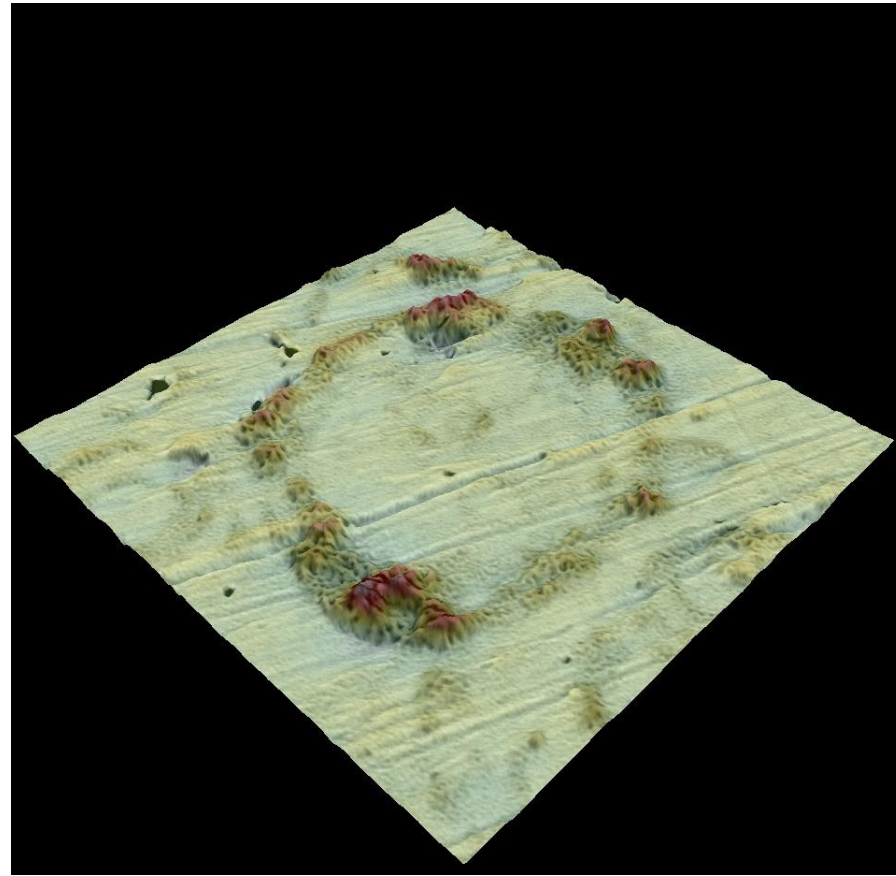
PGE-DND2-100



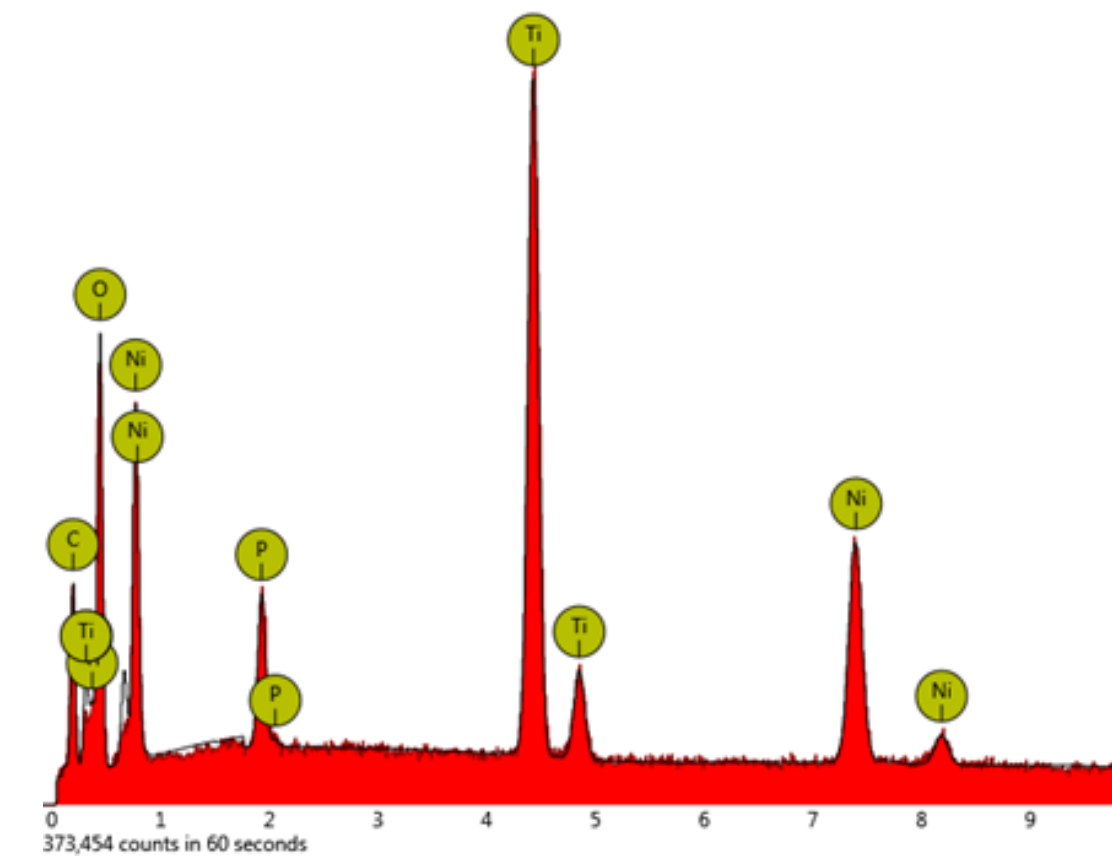
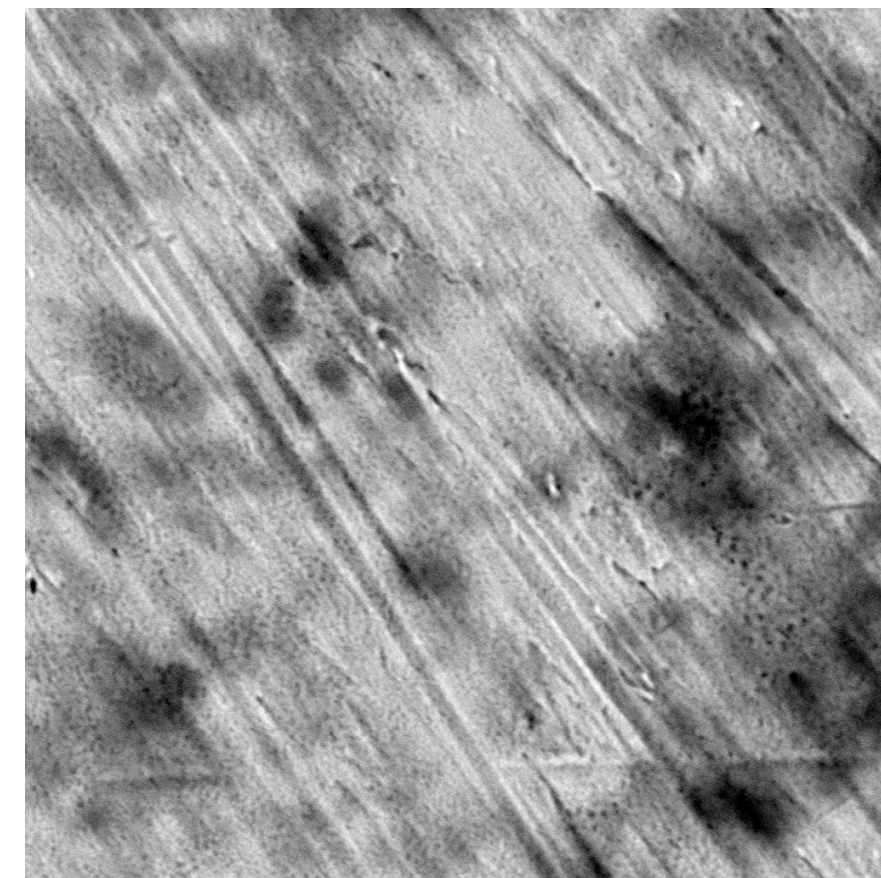
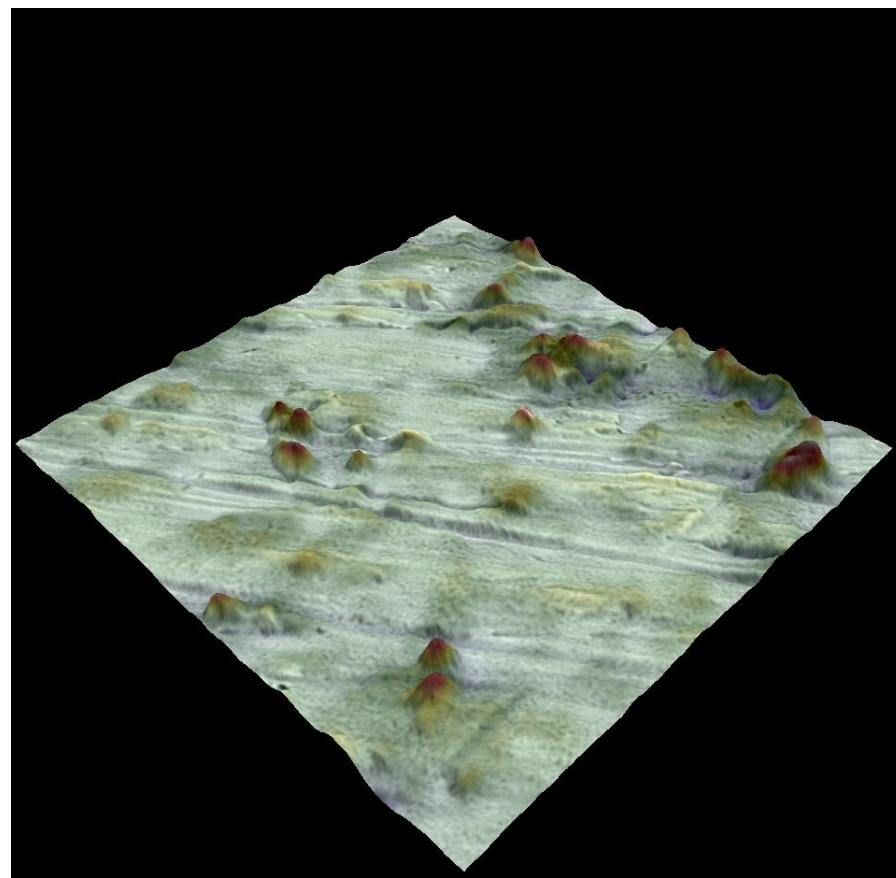
NiTi PEO PROCESS-NANODIAMONDS

15

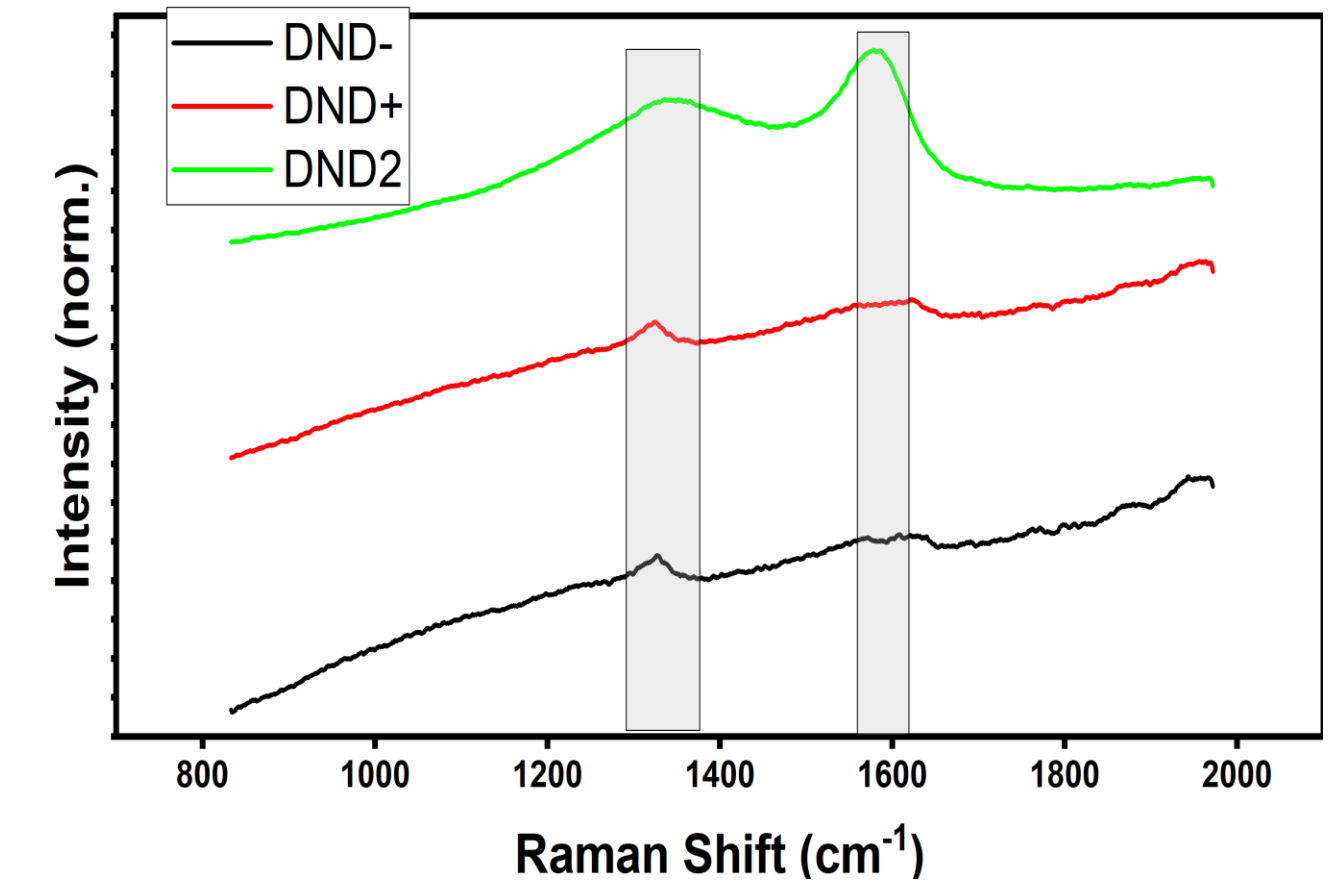
AK-PGE-DND(-) 200-60-300



AK-PGE-DND(+) 200-60-300



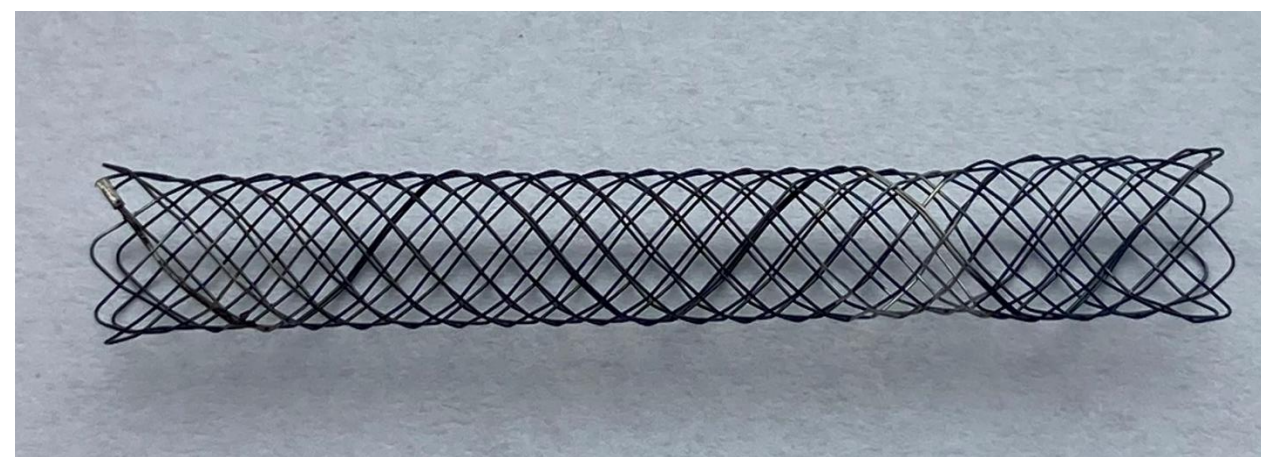
Nanodiamonds



NiTi PEO PROCESS-NANODIAMONDS



Sample holder



PGE-DND(+)-200-50V-
0,25A(2min)-1,25A(10s)

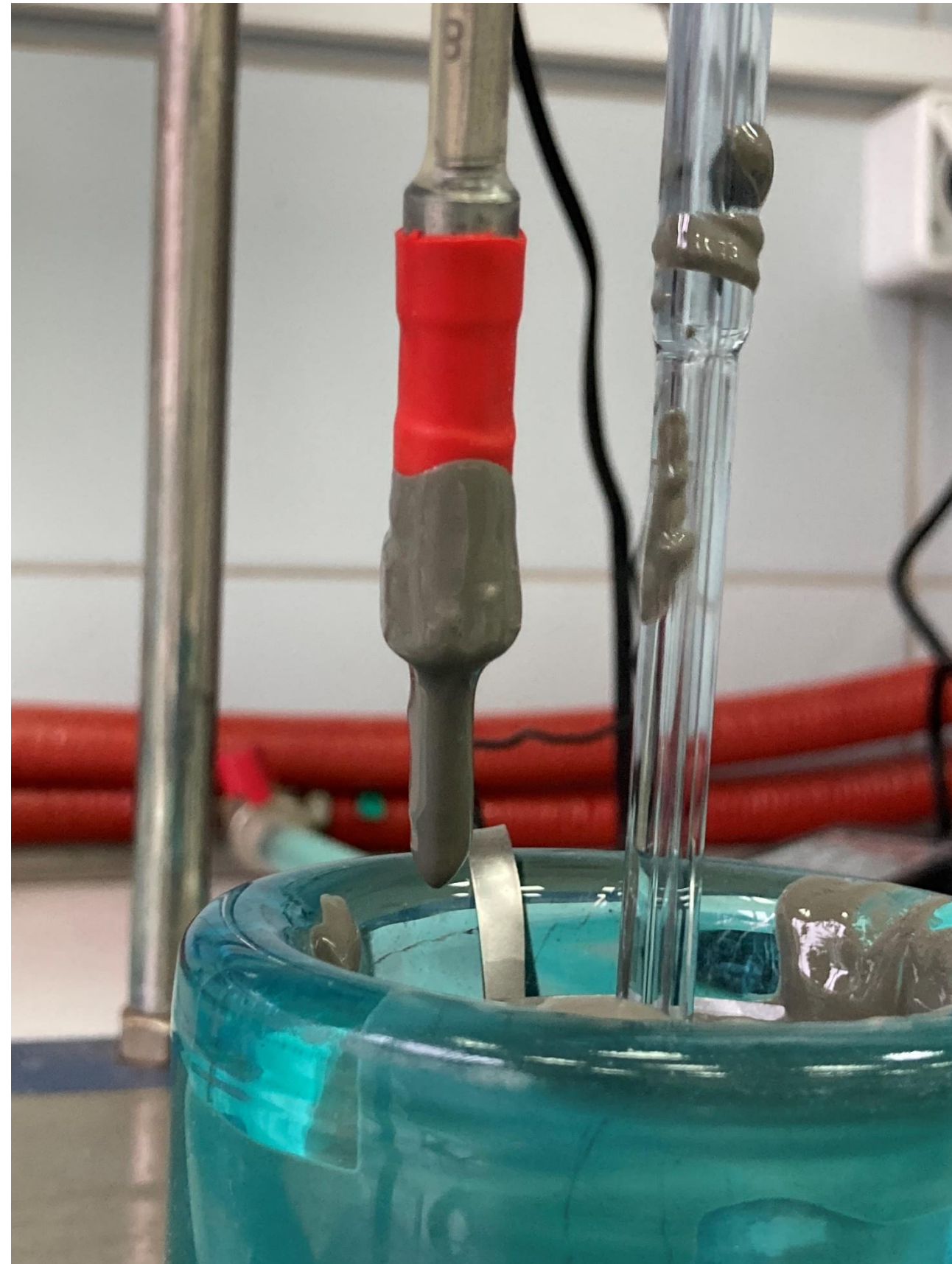


PGE-DND(+)-200-60V-
0,25A(2min)-1,25A(10s)

NiTi PEO PROCESS-NANODIAMONDS

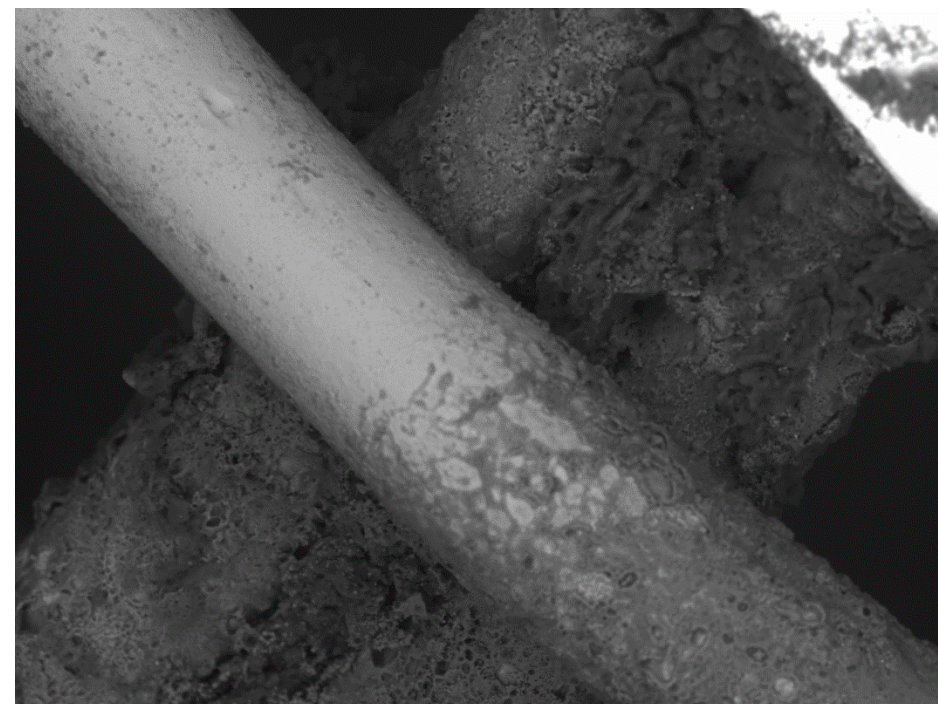
FAILED 😞

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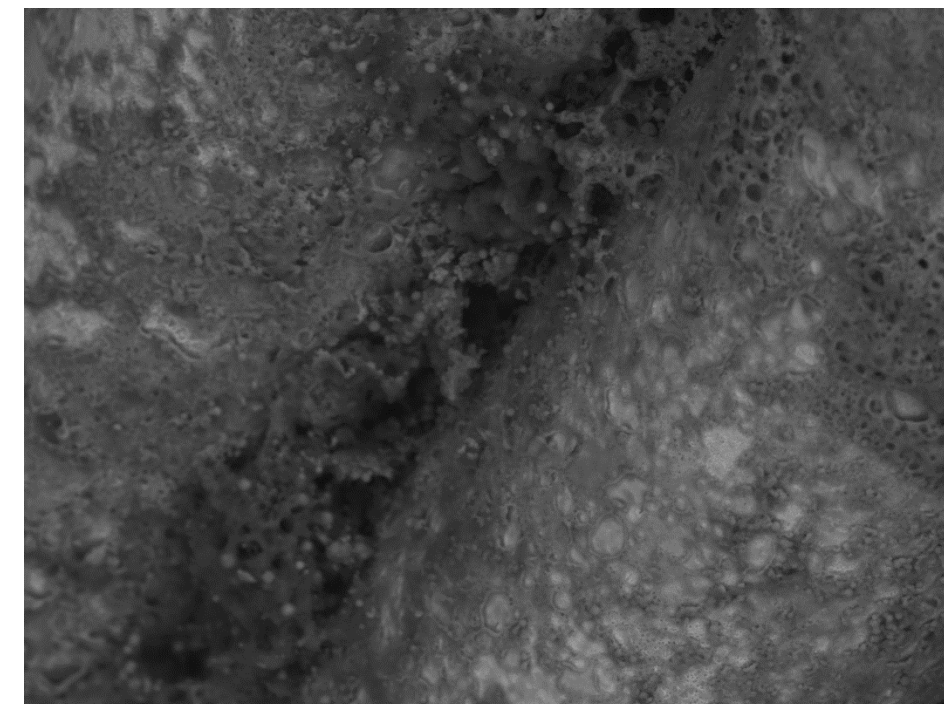


NiTi PEO PROCESS-NANODIAMONDS

FAILED ☹️



AL D5.3 x500 200 um

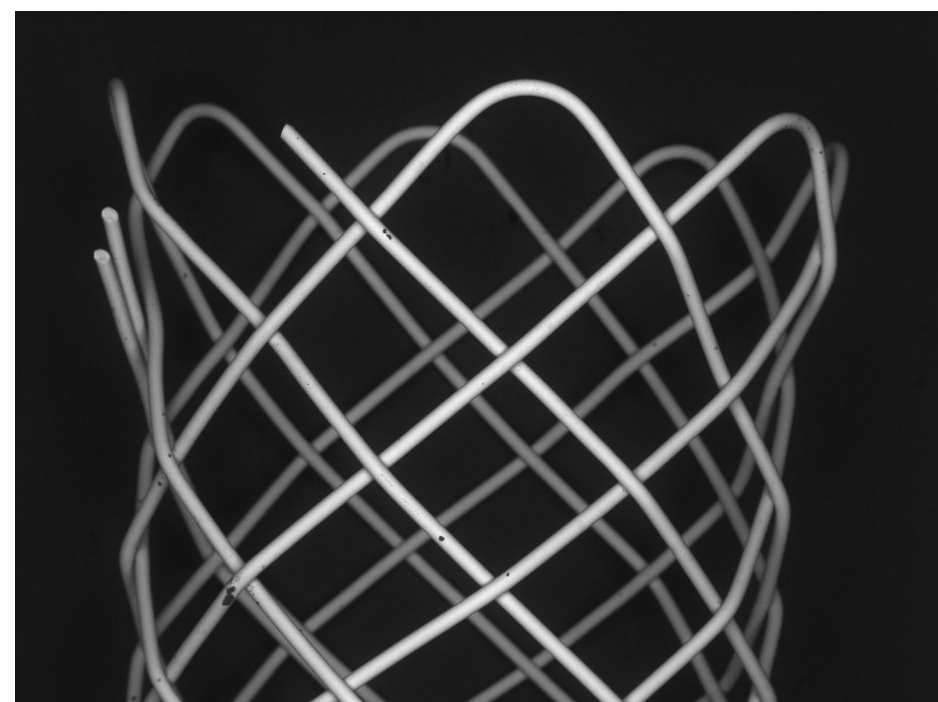


AL D5.8 x1.0k 100 um



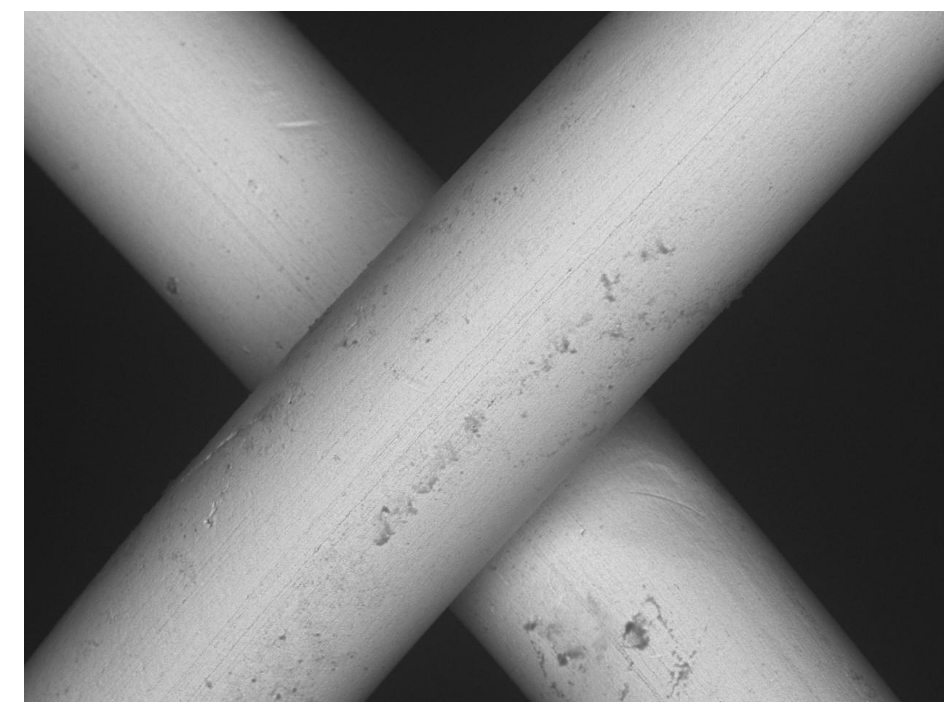
AL D6.6 x100 1 mm

**PGE-DND(+)-200-60V-
(0.25-2min-1.25-10s)**



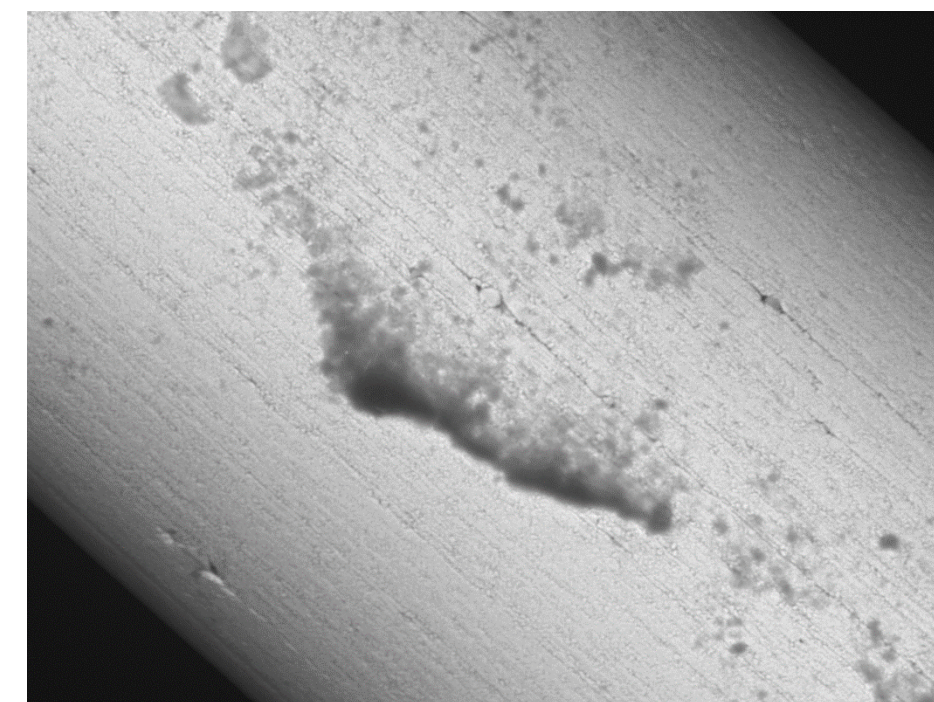
60V 0,75A

AL D14.8 x30 2 mm



60V 0,75A

AL D15.1 x500 200 um



60V 0,75A

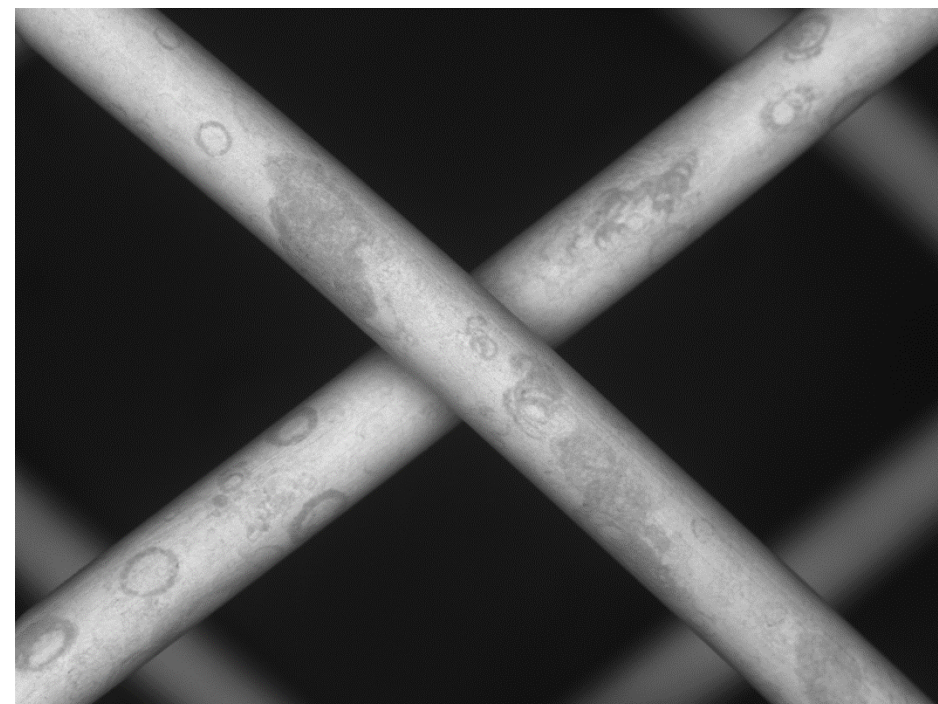
AL D14.9 x1.5k 50 um

**PGE-DND2-200-60V-
075A-5min**

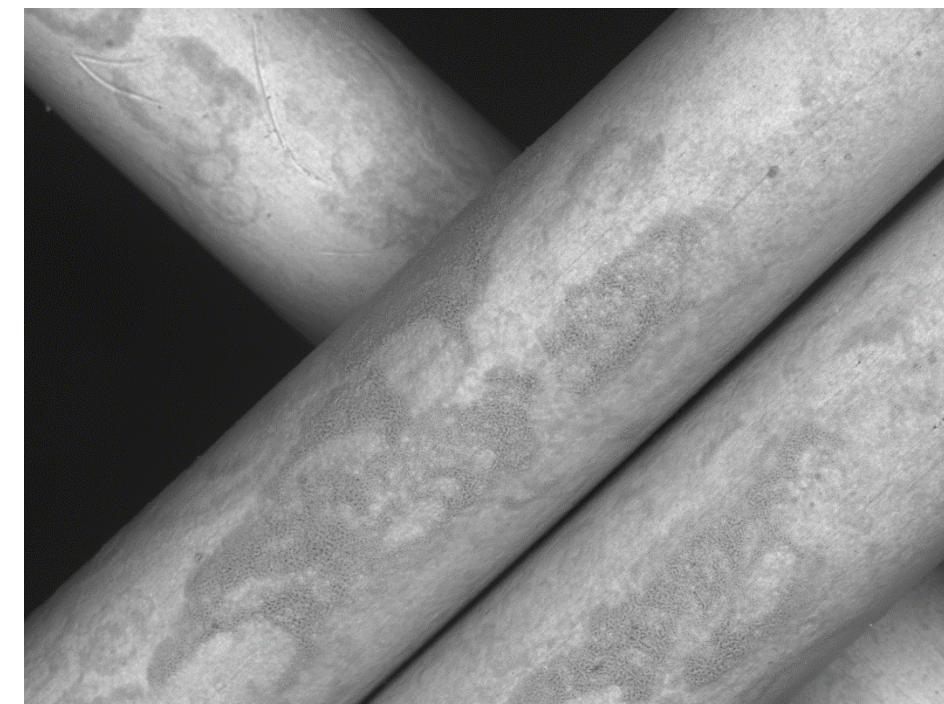
NiTi PEO PROCESS-NANODIAMONDS

SUCCESS ???

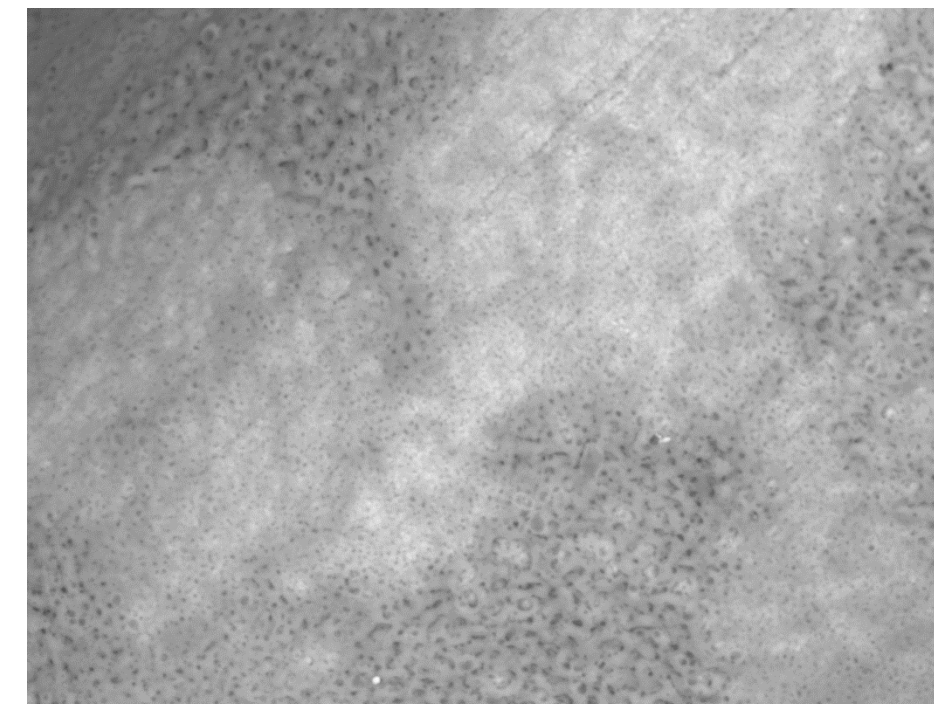
19



AL D8.5 x200 500 um

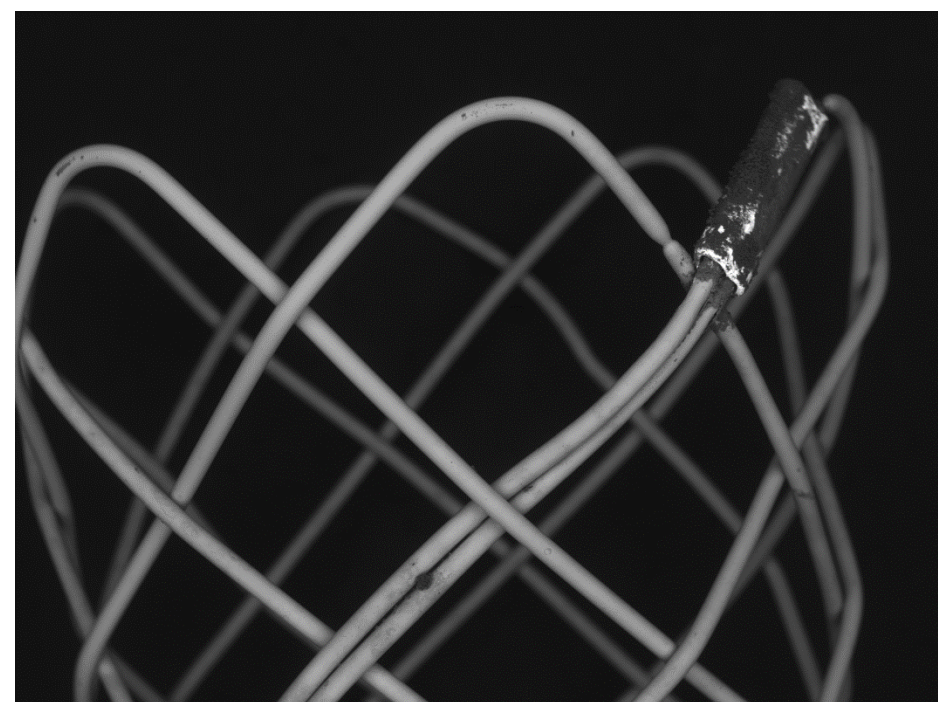


AL D8.5 x500 200 um

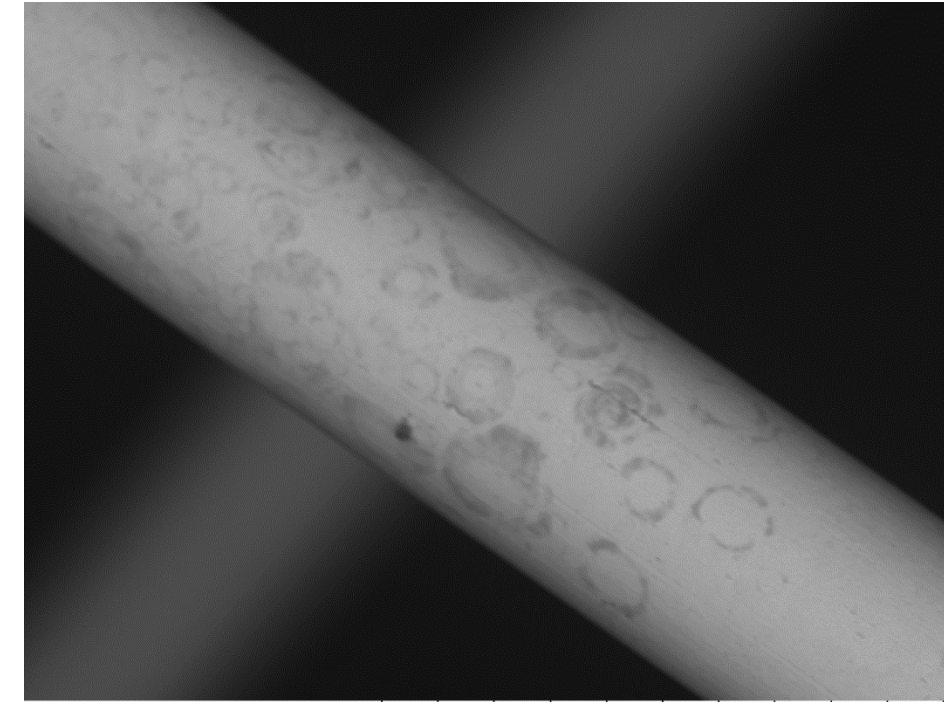


AL D8.5 x2.5k 30 um

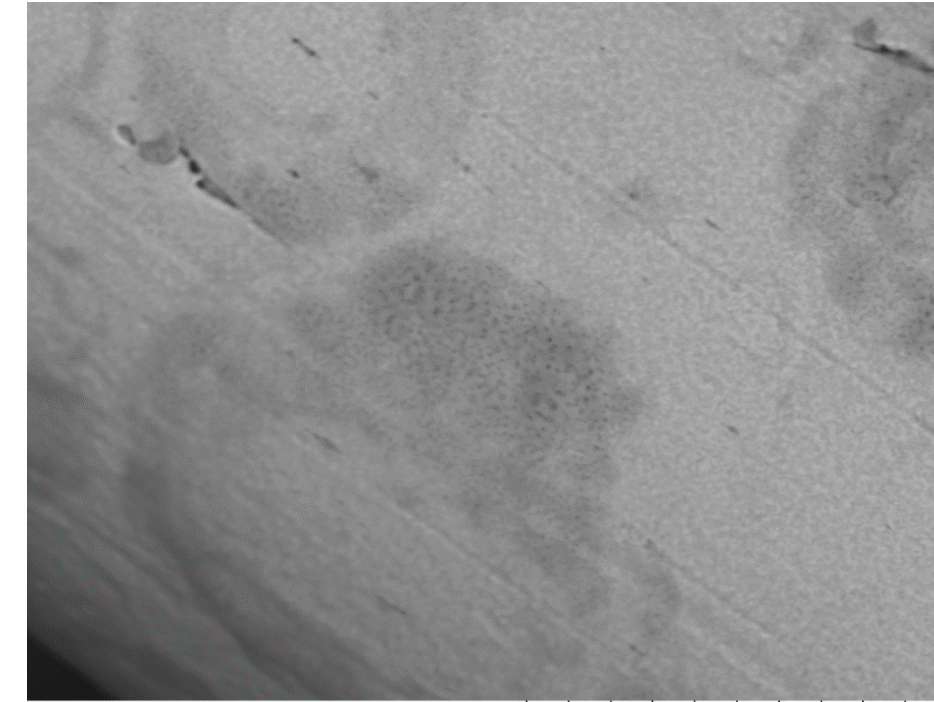
**P-DND2-200-60V-
(0.25-2min-1.25-10s)**



AL D5.3 x40 2 mm



AL D5.2 x500 200 um



AL D5.3 x2.5k 30 um

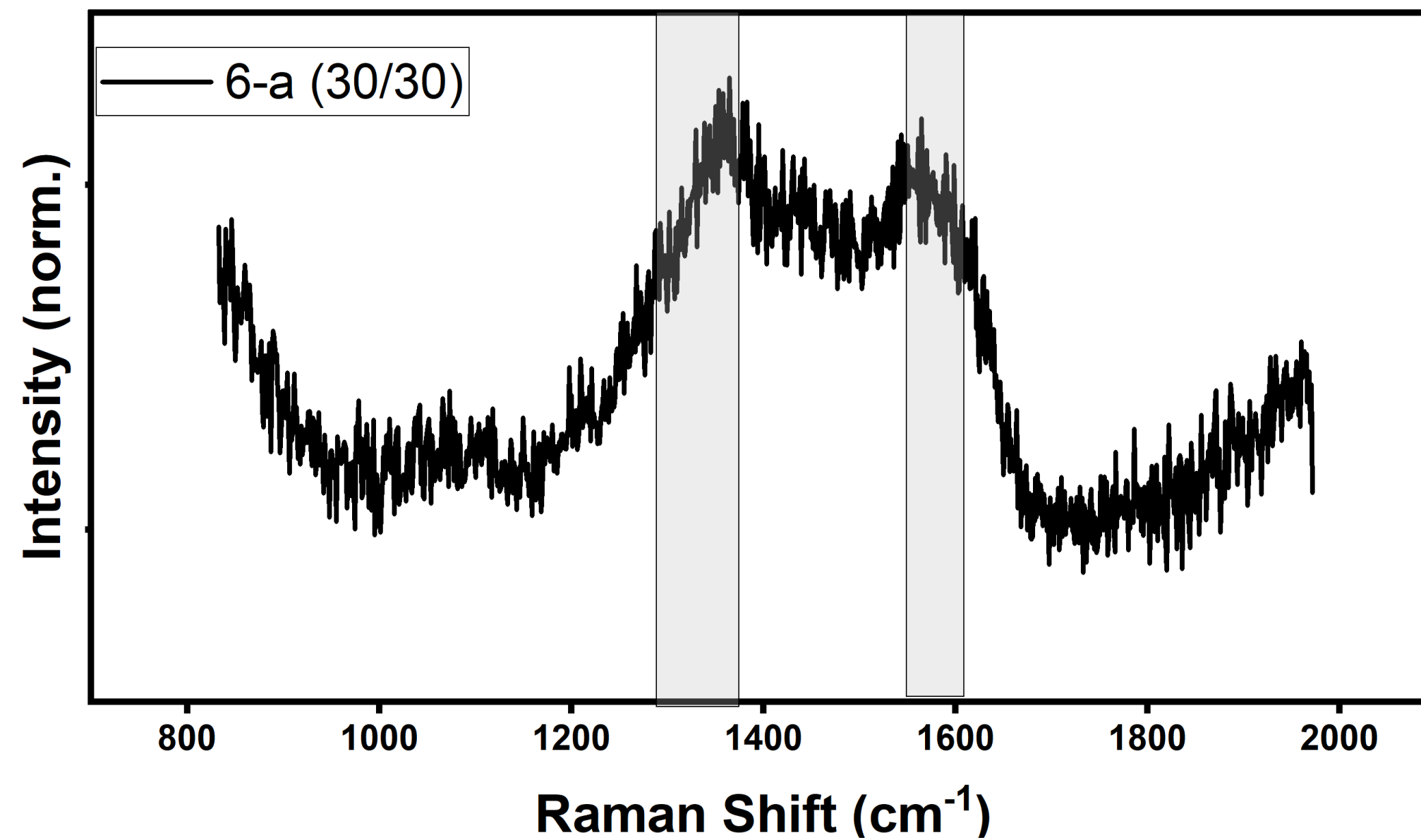
**PGE-DND(-)-200-60V-
(0.25A-2min-1.25A-10s)**



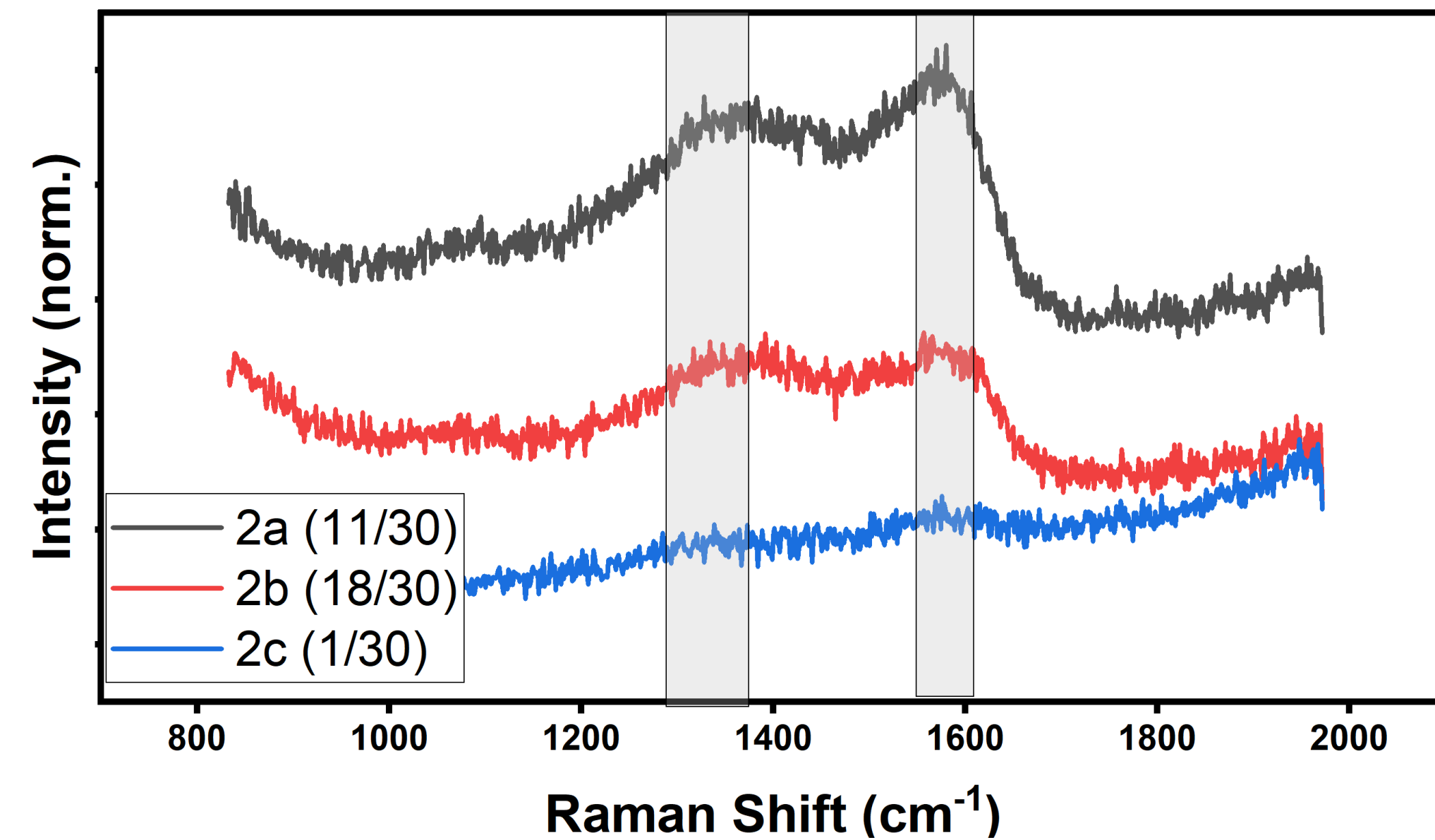
NiTi PEO PROCESS-NANODIAMONDS

SUCCESS 😊

PGE DND2-200-60 0.25 A - 2 min, 1.25 A - 10 s

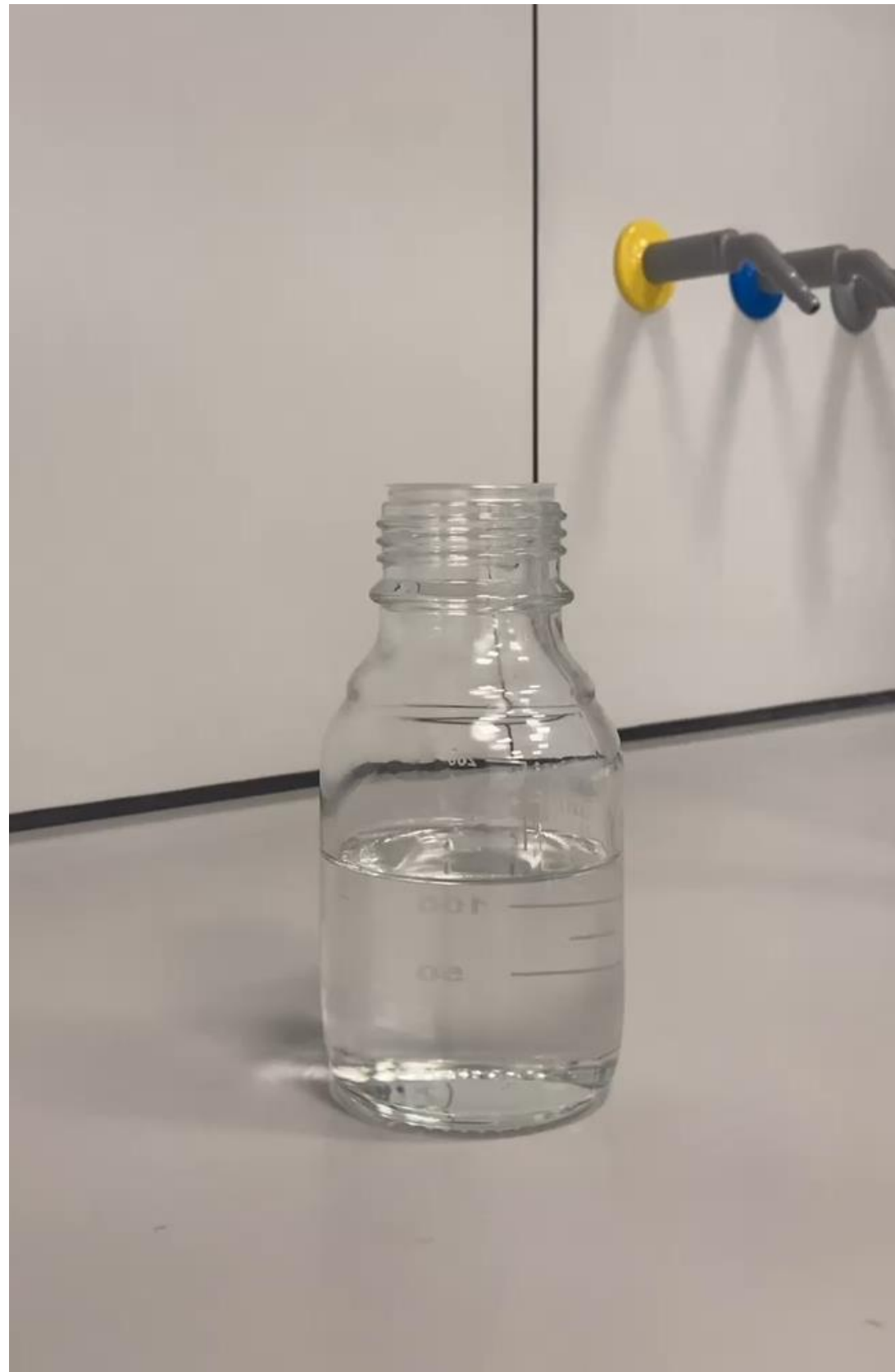


PGE DND2-200-50 0.25 A - 2 min, 1.45A - 10s



NiTi PEO PROCESS- Fe@C

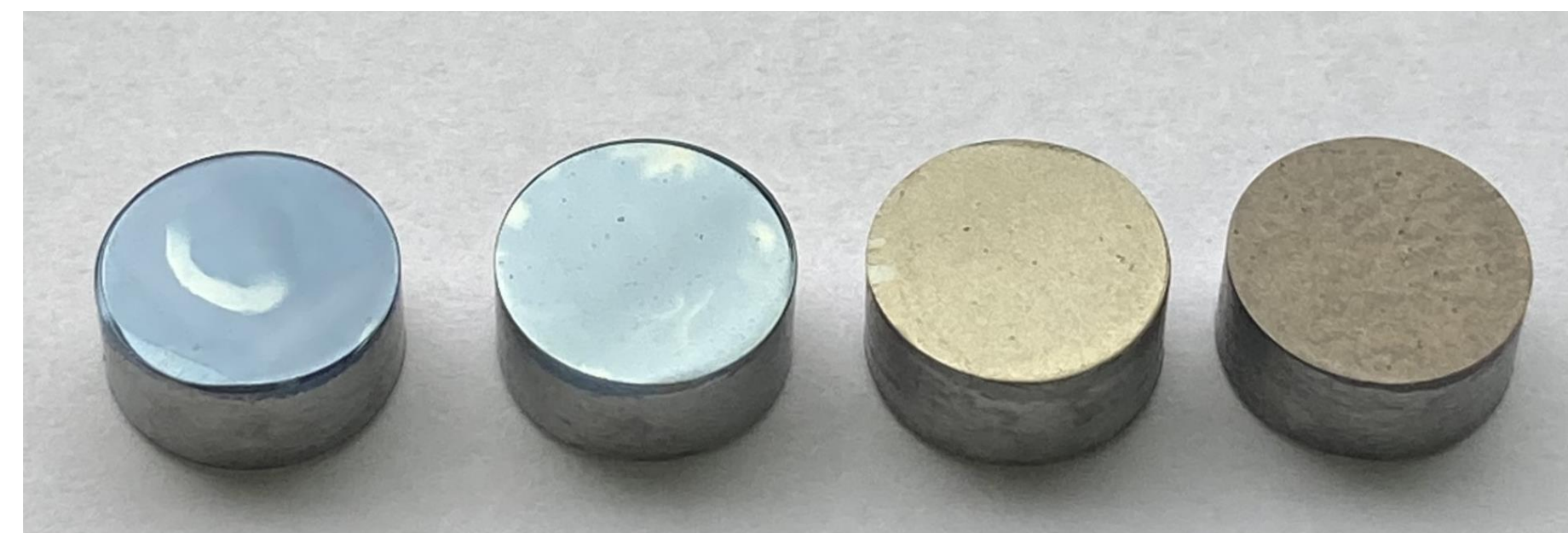
21



AK-P-Fe-50-(40-70)-200-5min



AK-P-Fe-50-(40-70)-300-5min



AK-PGE-Fe-100-(40-70)-200-5min



CONTACT

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