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DEVELOPMENT AND CHARACTERIZATION OF COATINGS ON SELECTED METALS AND ALLOYS OBTAINED BY PLASMA ELECTROLYTIC OXIDATION

8th GD Day 2016

OUTLINE

1. INTRODUCTION
2. PROBLEM FORMULATING
3. PEO – SET UP
4. METHODIC
5. SEM, EDS, XPS, GDOES RESULTS
6. OTHER SCIENTIFIC INTERESTS

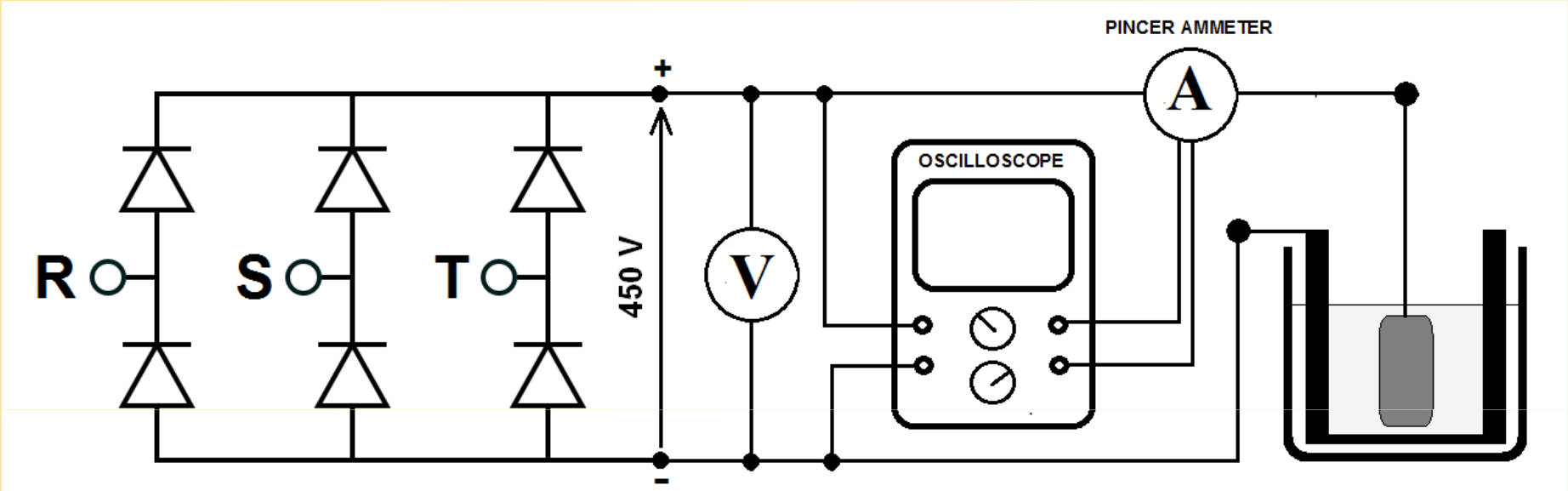
INTRODUCTION

To form the micro-coatings within micro- and nano- pores, the Plasma Electrolytic Oxidation (PEO) mainly is used. The technic is widely used by companies, such as Keronite (UK), Magoxide-Coat (Germany) and Microplasmic (USA), which are active in commercial development of PEO technology. At the beginning that method was used mainly for treatment of aluminum and its alloys. In later years, PEO was applied to oxidation of magnesium and its alloys, and titanium, and its alloys as well as niobium, zirconium and tantalum.

PROBLEM FORMULATING

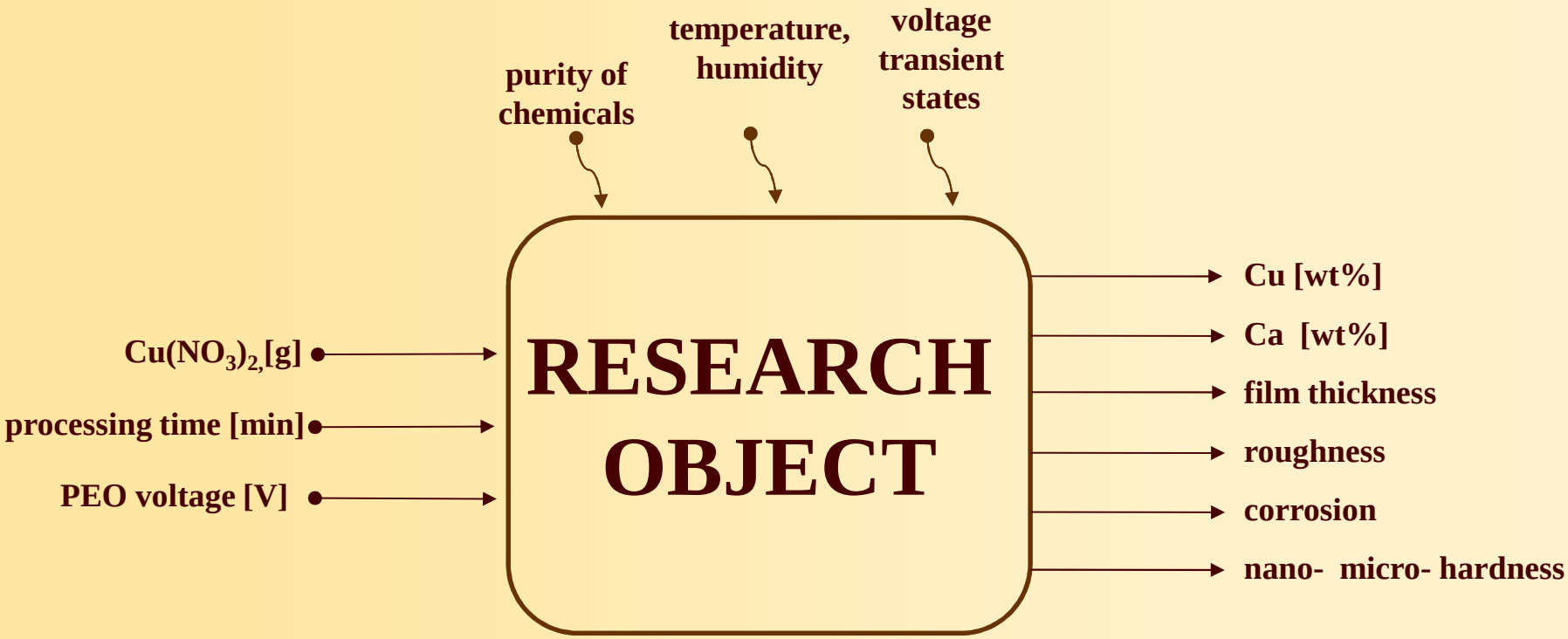
1. **Is it possible to obtain a porous coating enriched in copper on titanium and its alloys, as well as on niobium and tantalum by Plasma Electrolytic Oxidation in an electrolyte based on concentrated phosphoric acid H_3PO_4 and copper nitrate $Cu(NO_3)_2$?**
2. **What is the effective range of $Cu(NO_3)_2$ content in H_3PO_4 to obtain the formation of a porous coating enriched in copper of required concentration?**
3. **Is the copper equally distributed in the PEO coatings in whole volume?**
4. **Do we need the copper to be equally distributed in the first sublayer or should it go deeper into the 2nd and/or 3rd one?**

PEO - SET UP


PINCER AMMETER
A
OSCILLOSCOPE
+
R
S
T
450 V
V
-


GW INSTEK OSCILLOSCOPE GOS-620 20MHz
HORIZONTAL
TRIGGER
TIME/DIV
POSITION
SWP. VAR.
LEVEL
SLOPE
VERTICAL
VOLTS/DIV
MODE
CH1
CH2

DEFINING OF OBJECT

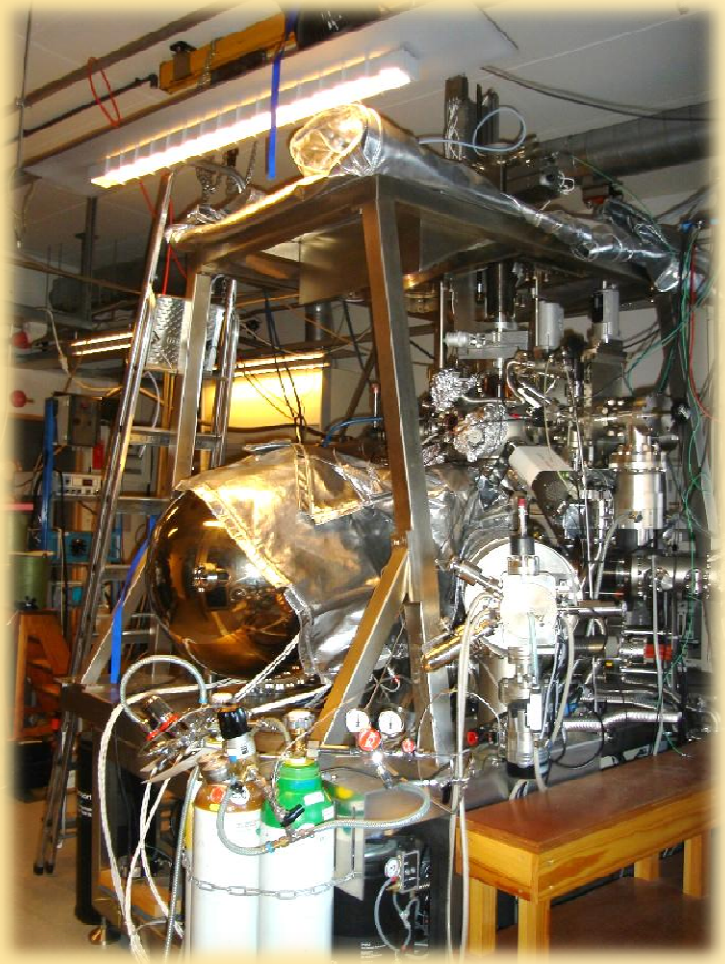


water content in the electrolyte

cathode – AISI 316L

samples preparation by mechanical grinding

METHOD



SCIENCE SES 2002

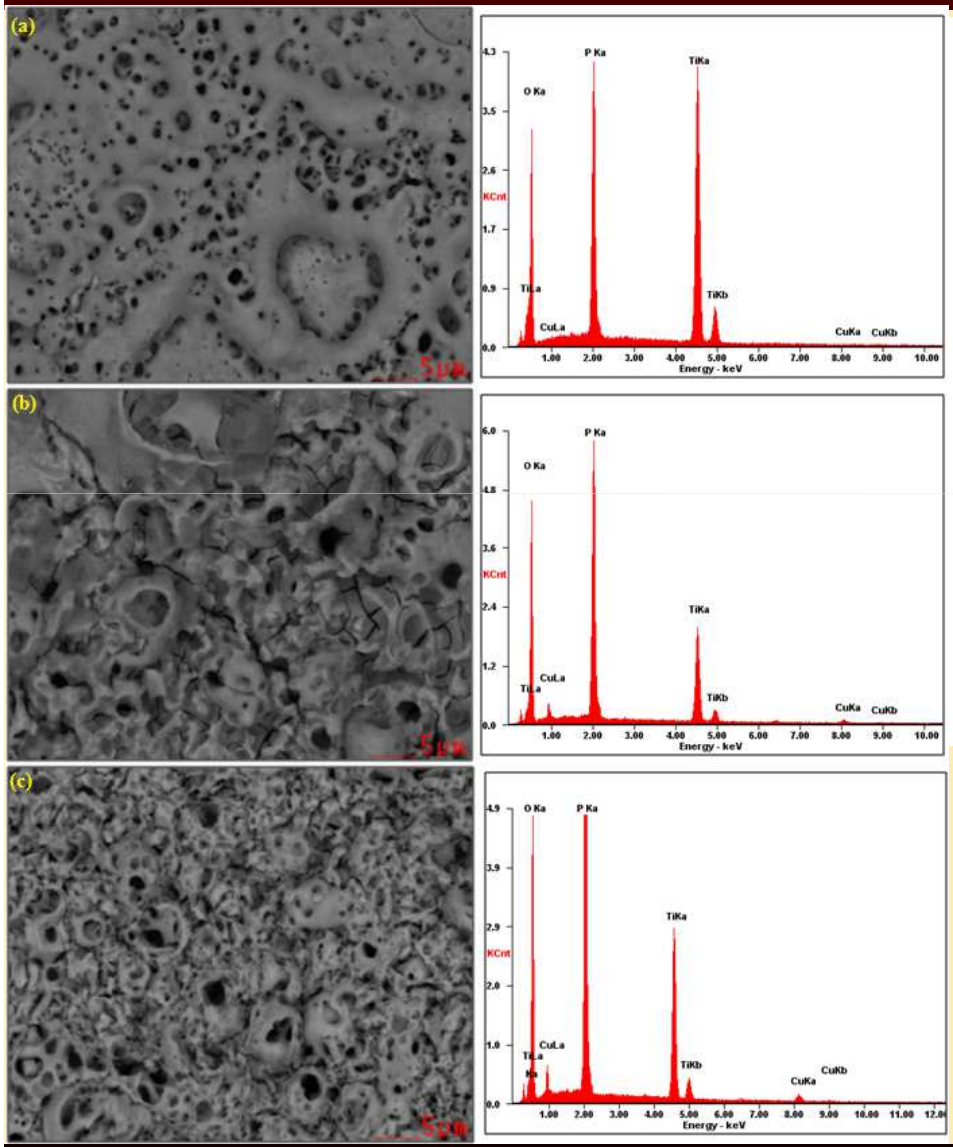
HORIBA - PROFILER 2

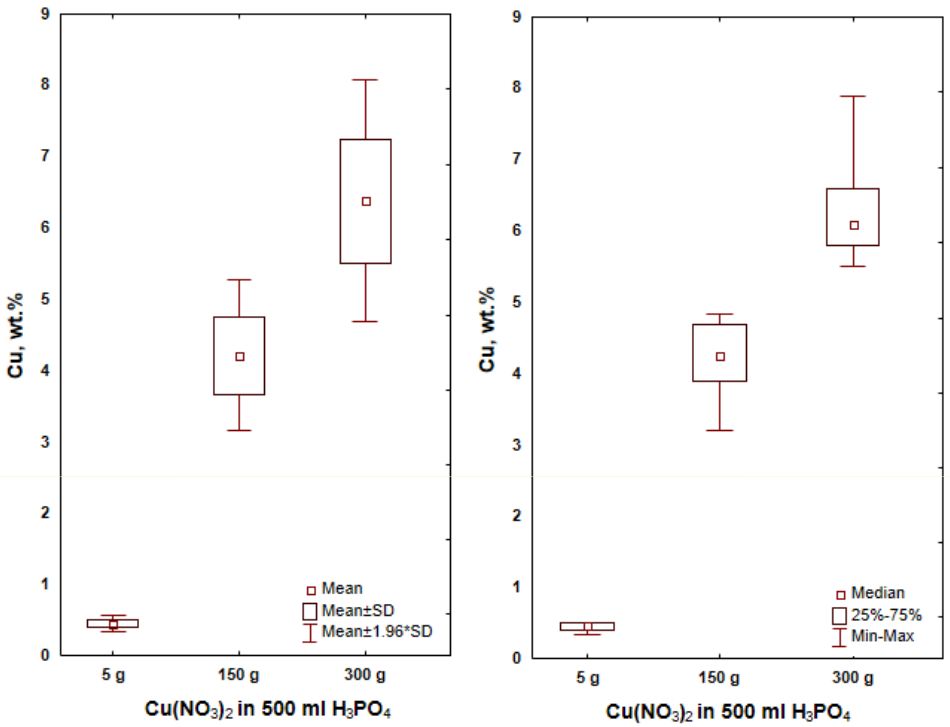
METHOD

HOMMEL TESTER T800 SYSTEM
HOMMELWERKE GMBH

BRUKER-AXS
D8 ADVANCE

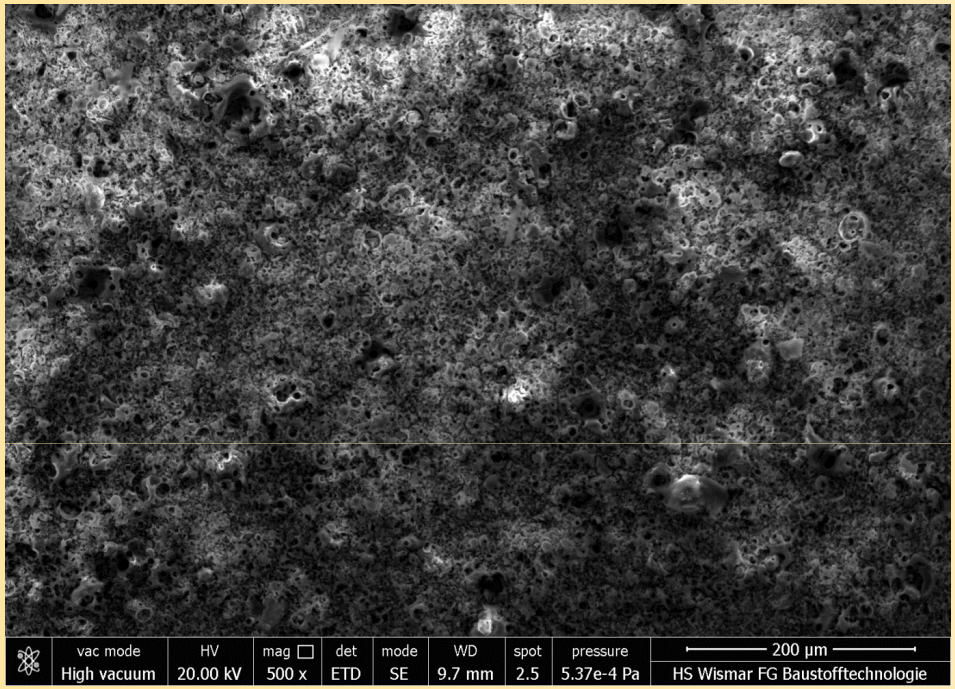
PEO - TITANIUM - SEM & EDS

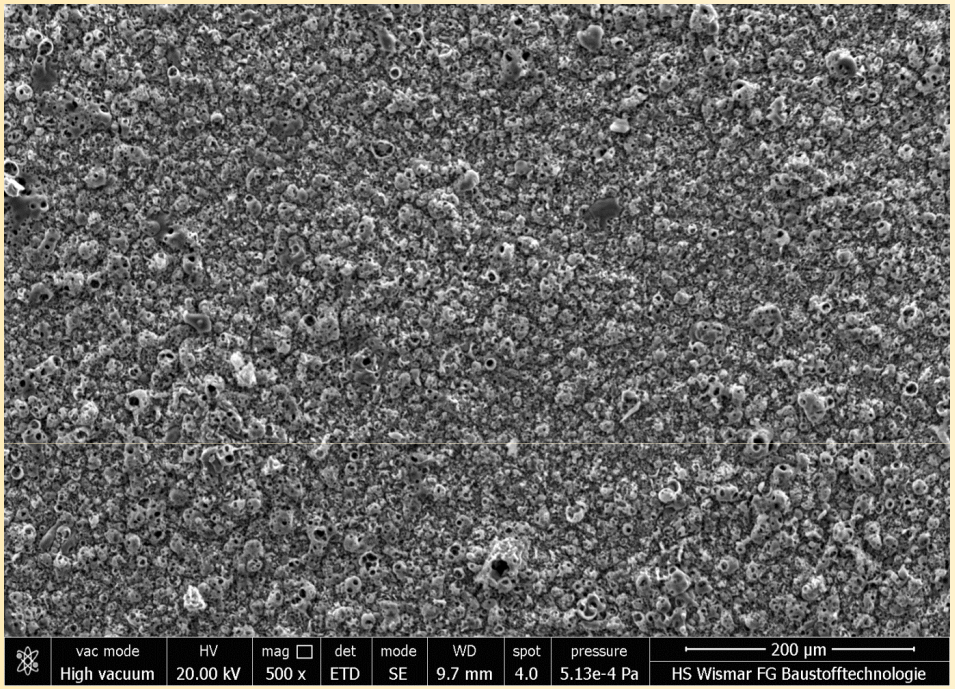




Cu, wt%	5 g $Cu(NO_3)_2$	150 g $Cu(NO_3)_2$	300 g $Cu(NO_3)_2$
Average	0.45	4.22	6.38
Variance	0.004	0.29	0.75
Standard deviation	0.06	0.54	0.86
Median	0.46	4.27	6.10
Maximum	0.51	4.85	7.9
Minimum	0.33	3.21	5.52

TITANIUM - SEM RESULTS

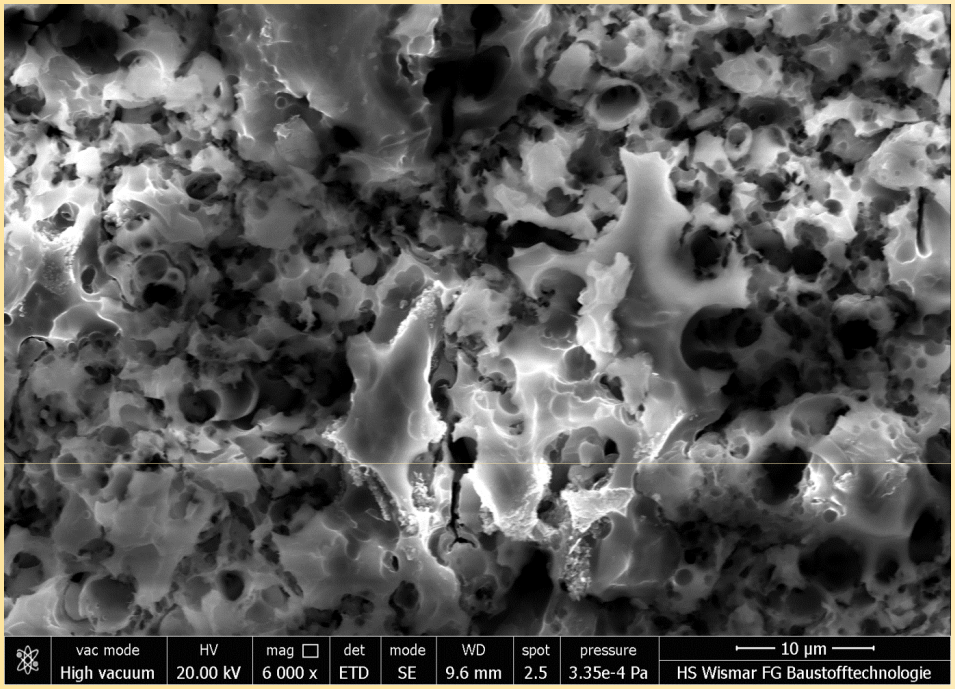


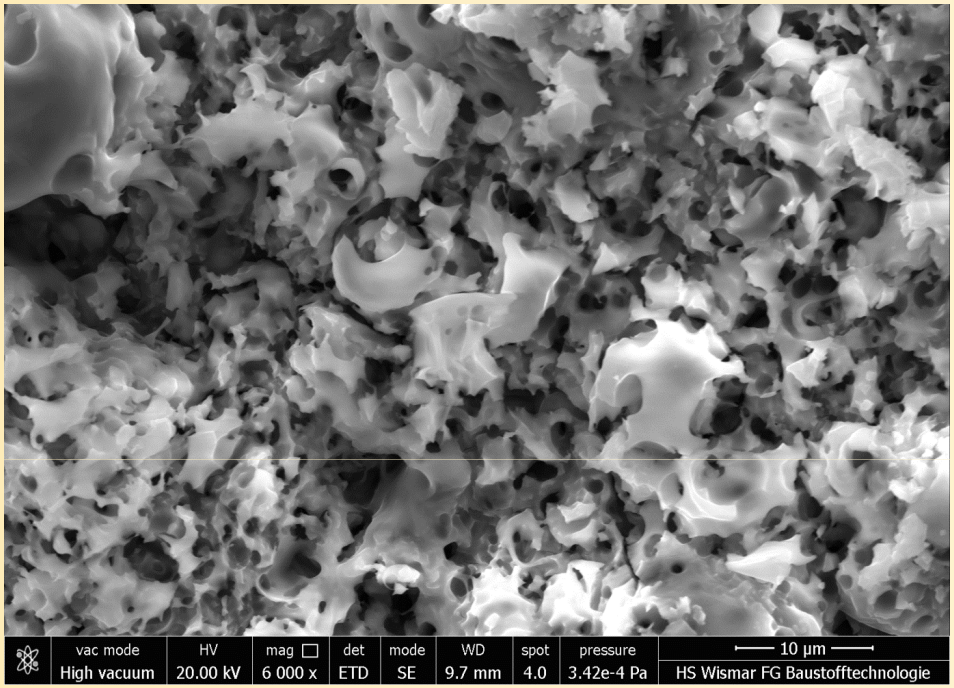


300 g $Cu(NO_3)_2$ in 1 L H_3PO_4 **600 g $Cu(NO_3)_2$ in 1 L H_3PO_4**

SEM pictures of porous coatings on titanium obtained by PEO at voltage of 450 V in electrolyte containing H_3PO_4 and $Cu(NO_3)_2$

TITANIUM - SEM RESULTS

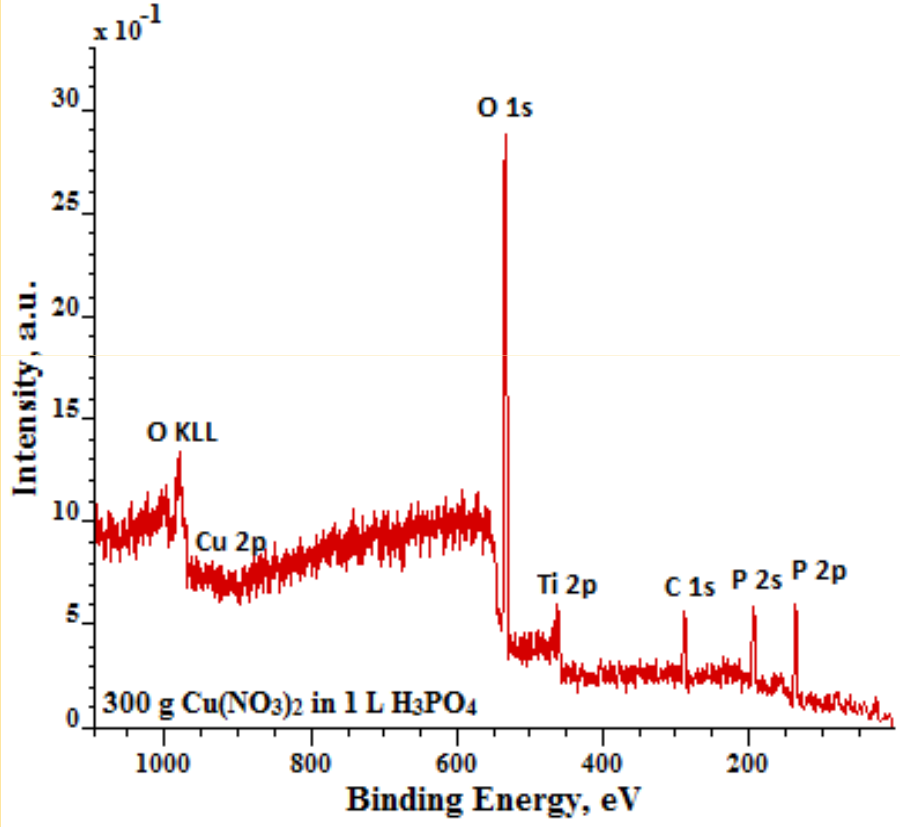


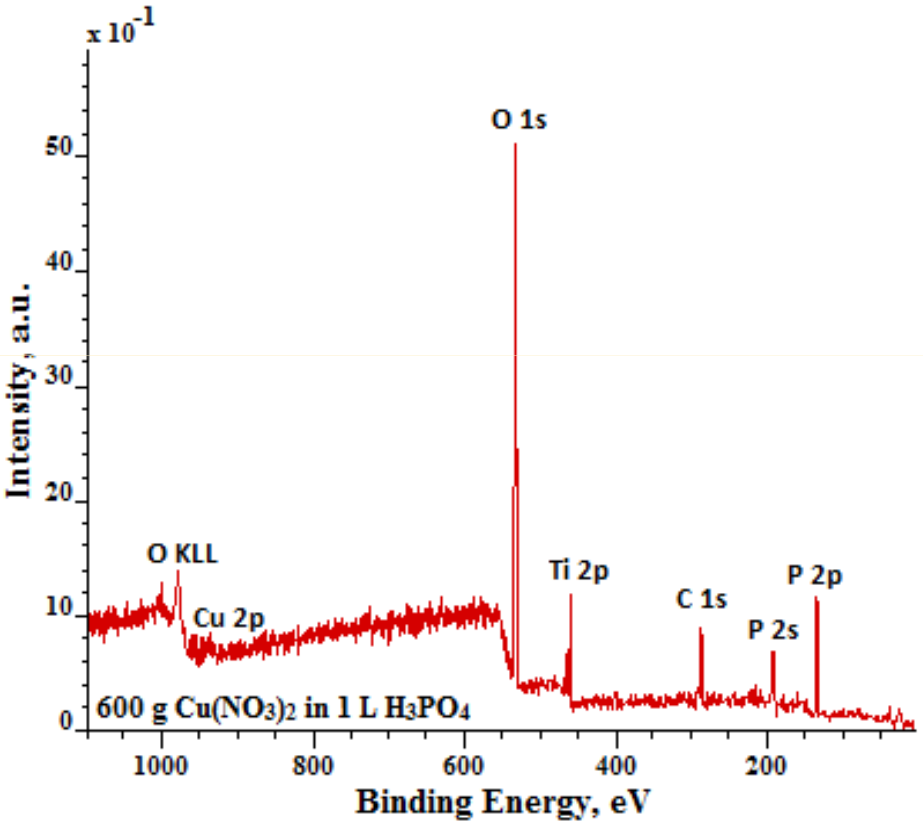


300 g $Cu(NO_3)_2$ in 1 L H_3PO_4 **600 g $Cu(NO_3)_2$ in 1 L H_3PO_4**

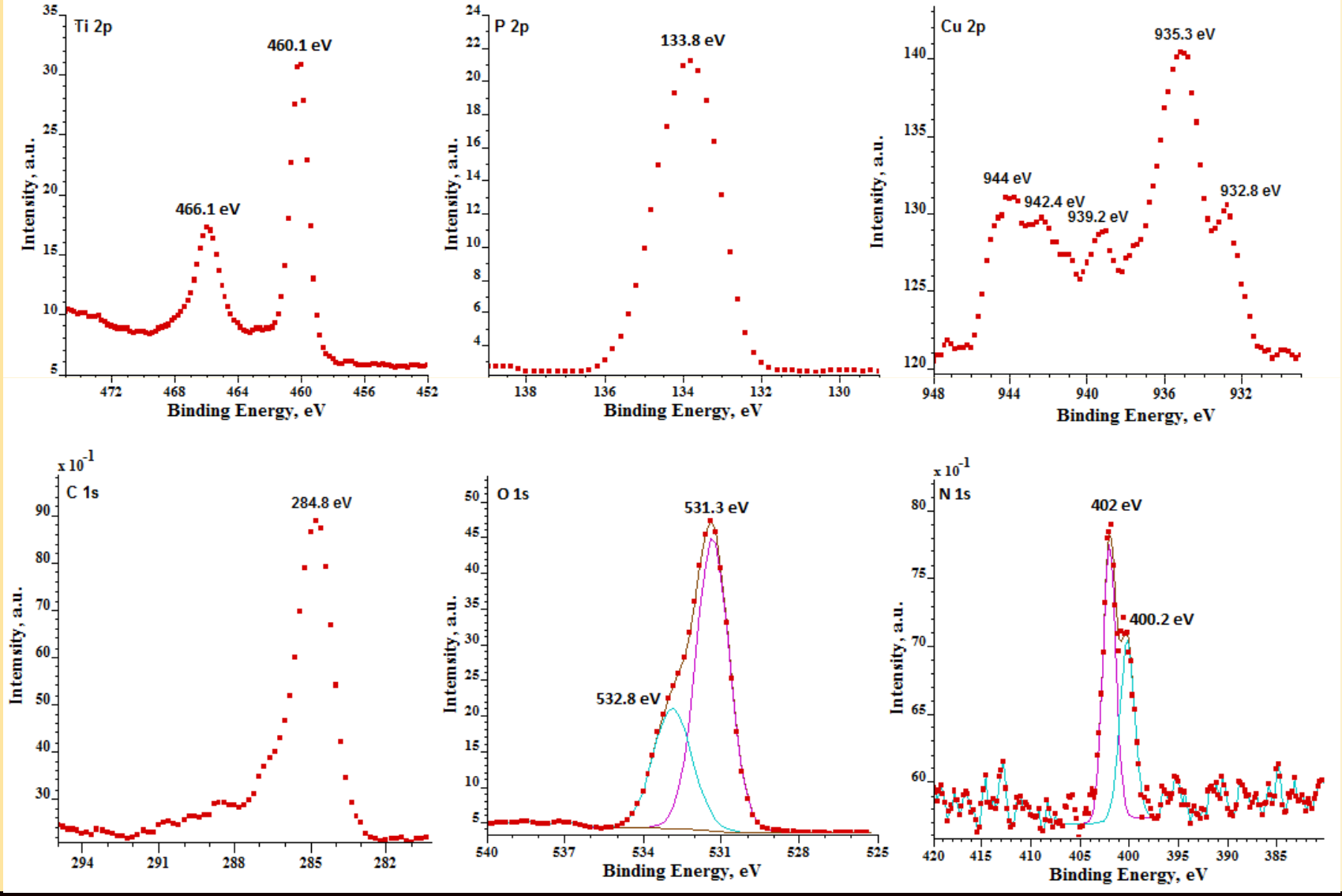
SEM pictures of porous coatings on titanium obtained by PEO at voltage of 450 V in electrolyte containing H_3PO_4 and $Cu(NO_3)_2$

TITANIUM - XPS RESULTS

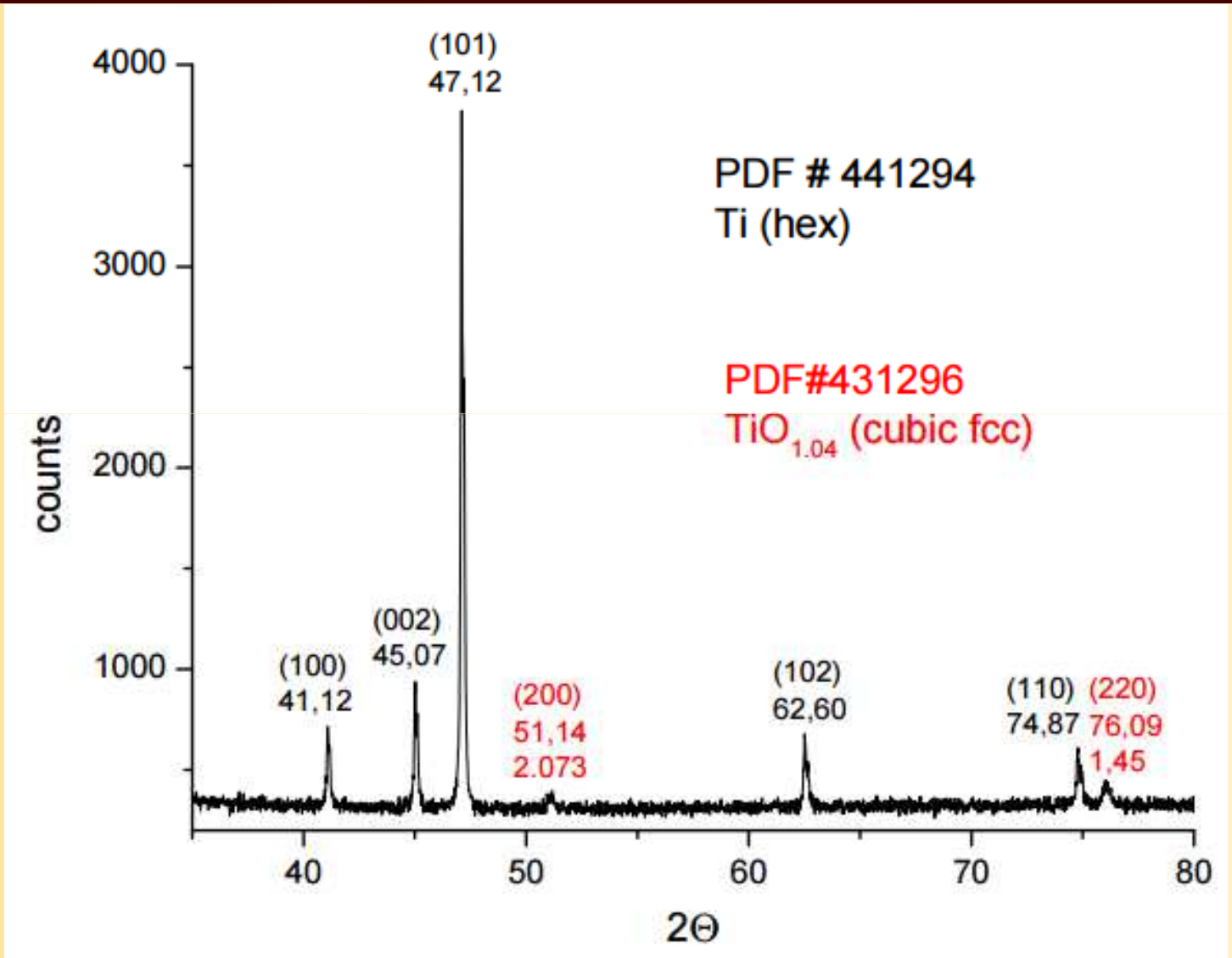




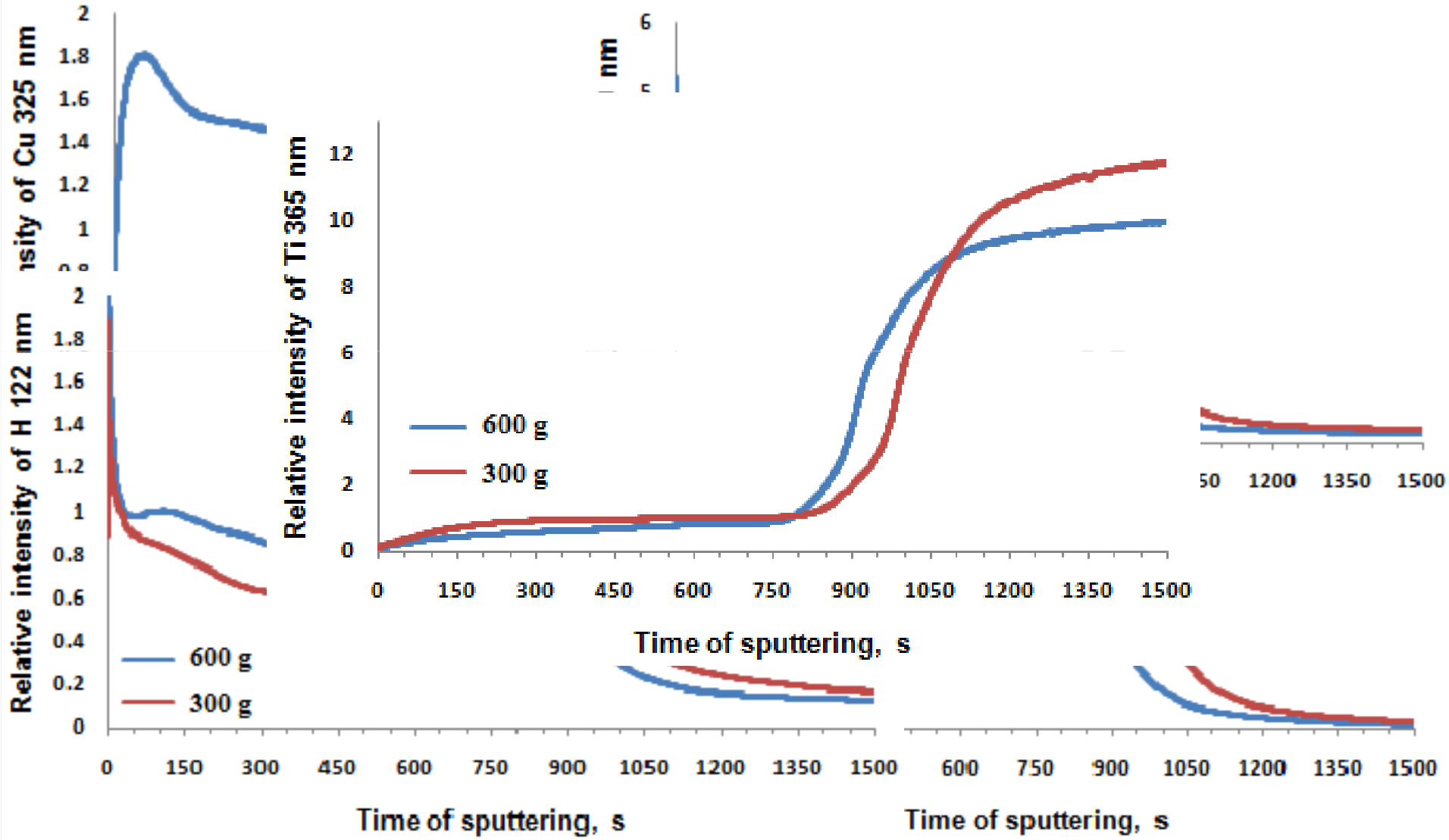
TITANIUM - XPS RESULTS



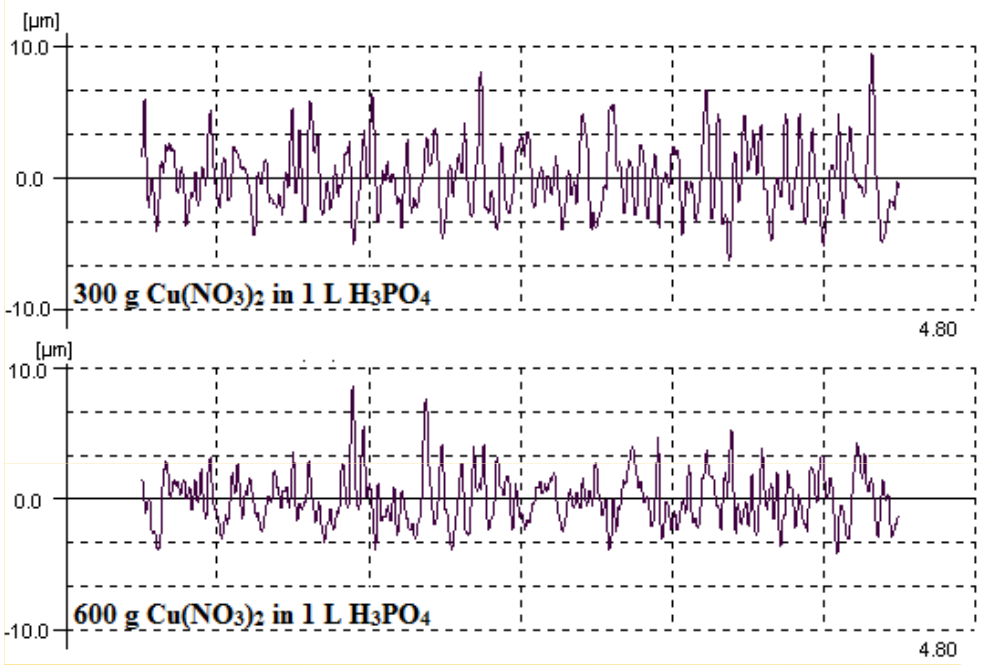
TITANIUM - XRD RESLTS

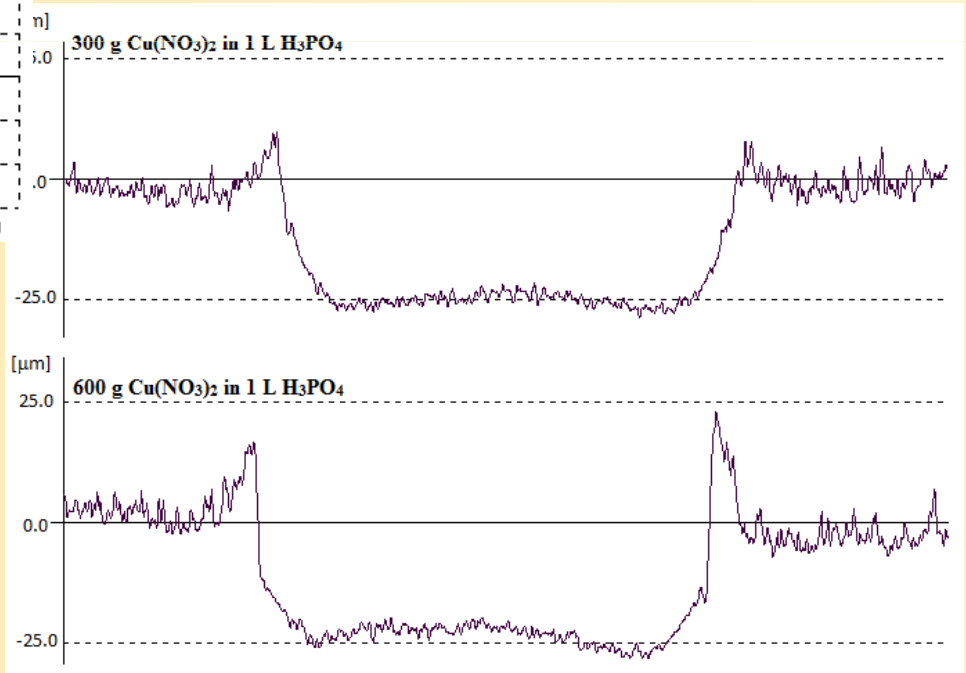


TITANIUM - GDOES RESULTS

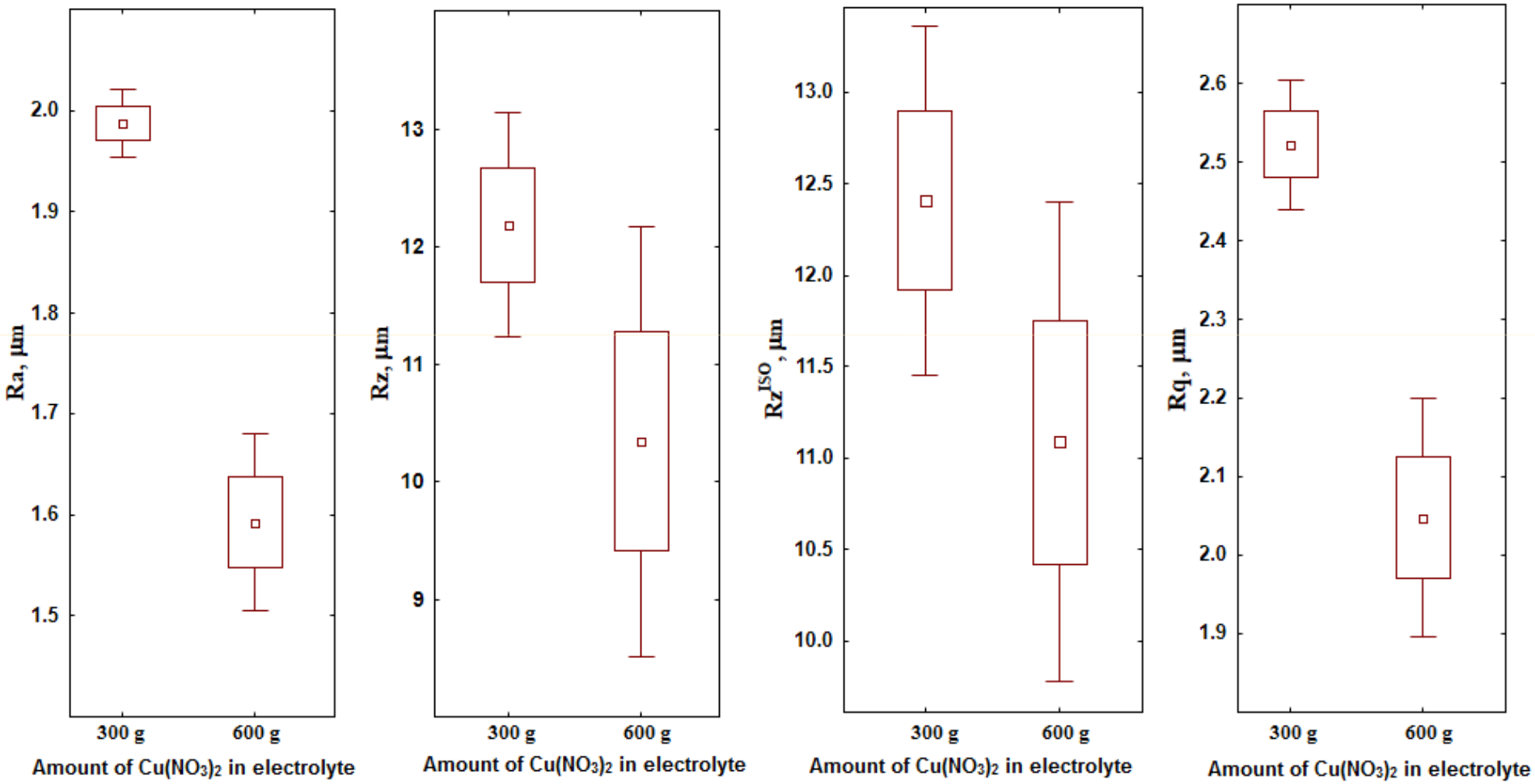


TITANIUM - 2D PROFILES

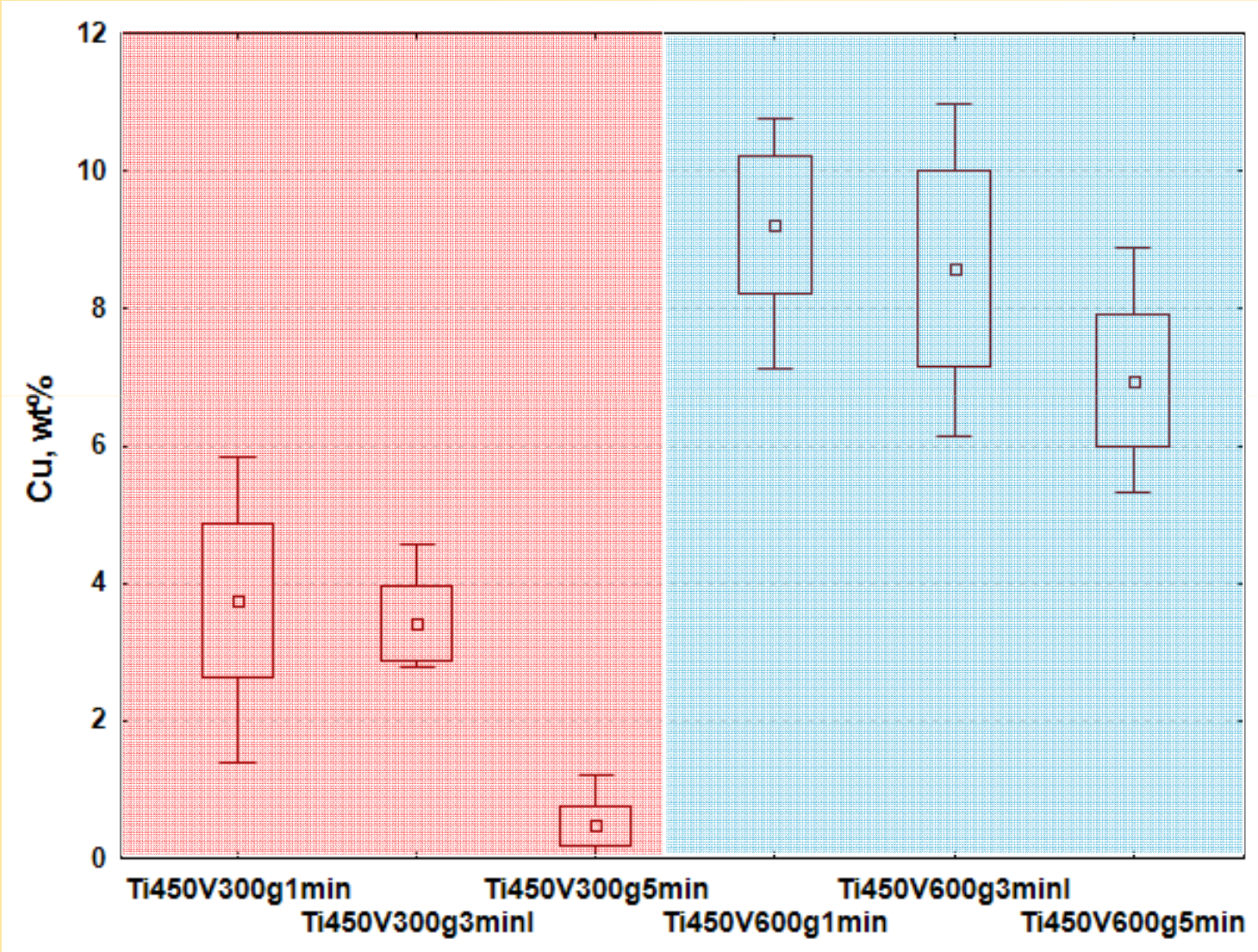




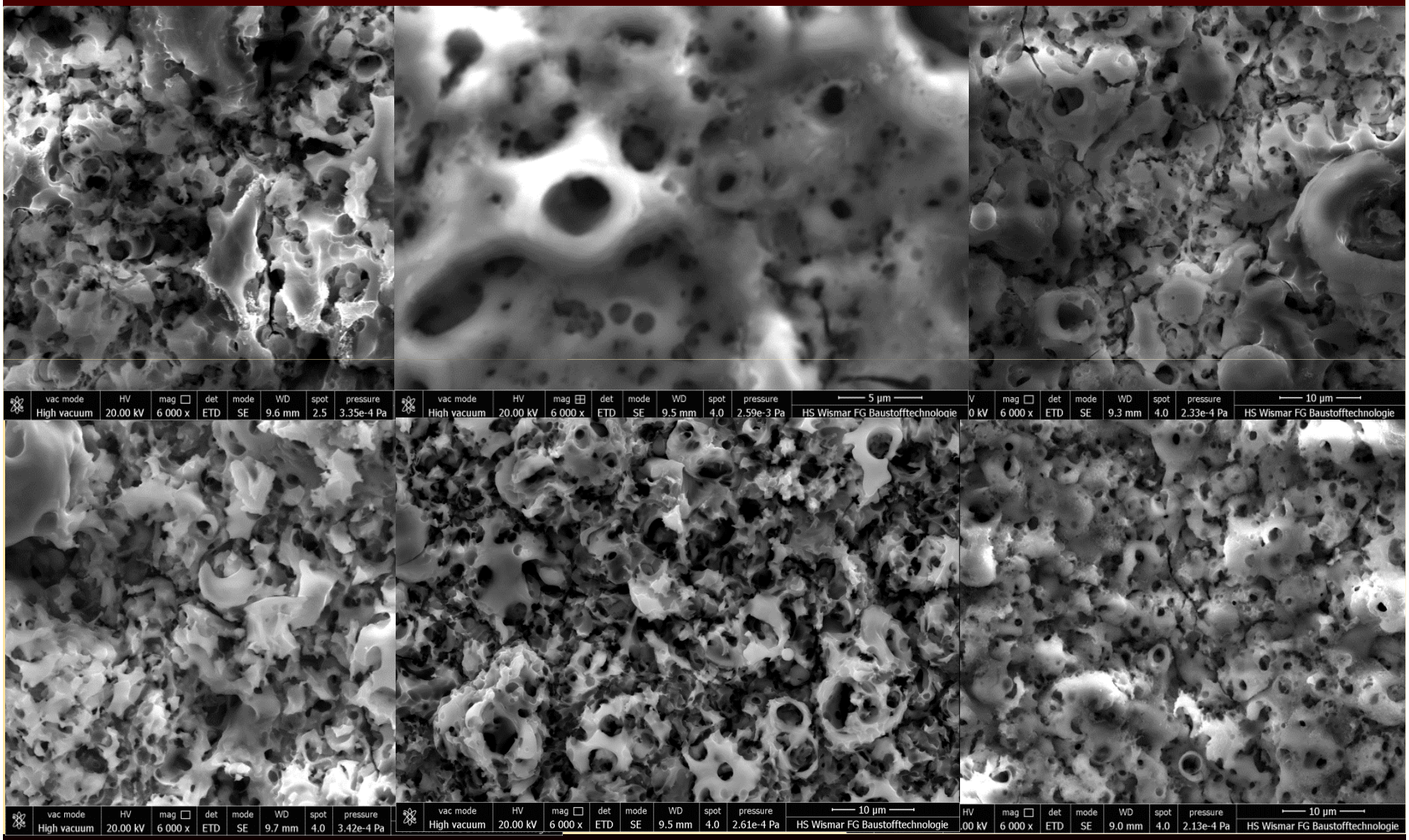
ROUGHNESS RESULTS



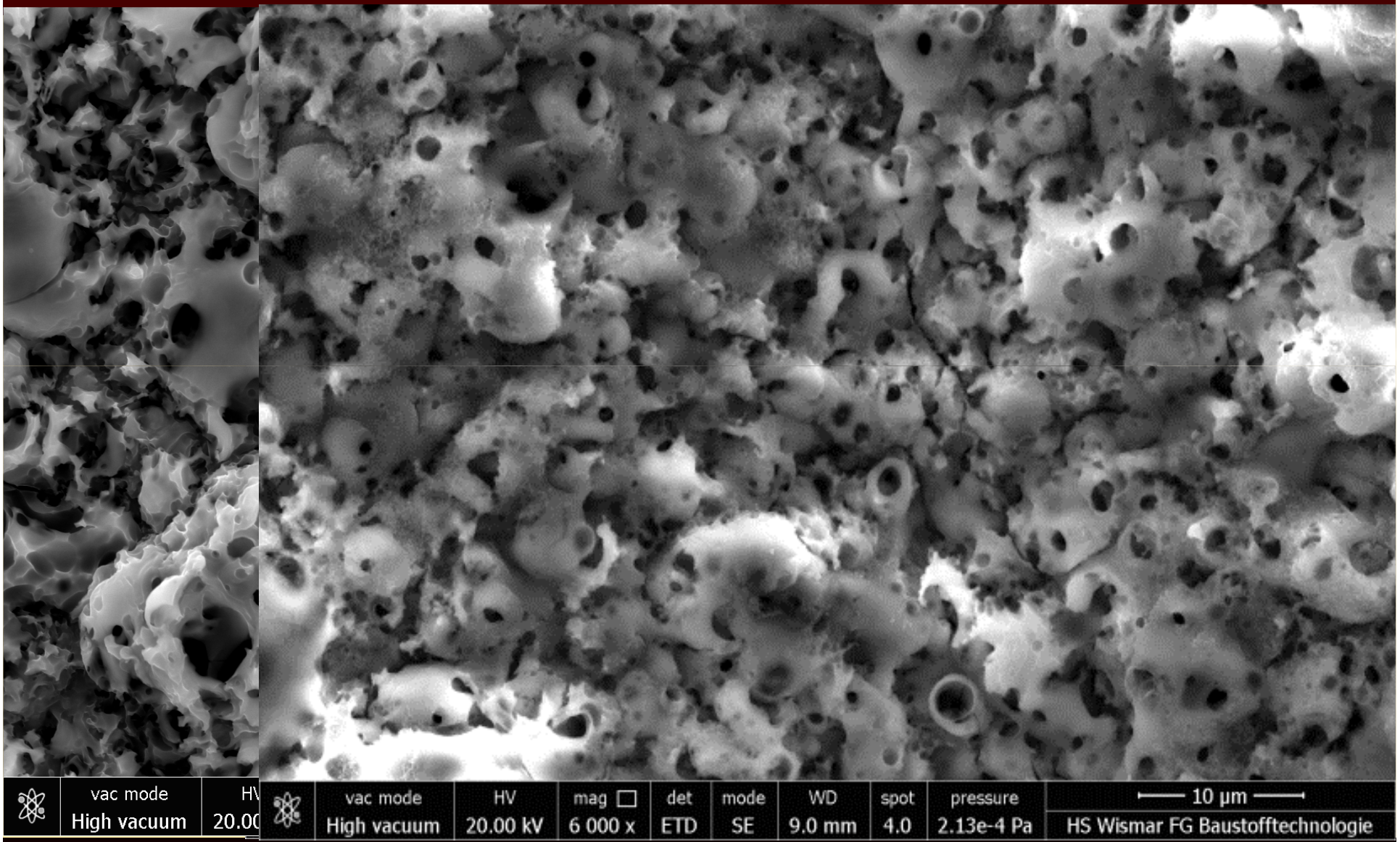
TITANIUM - RESULTS



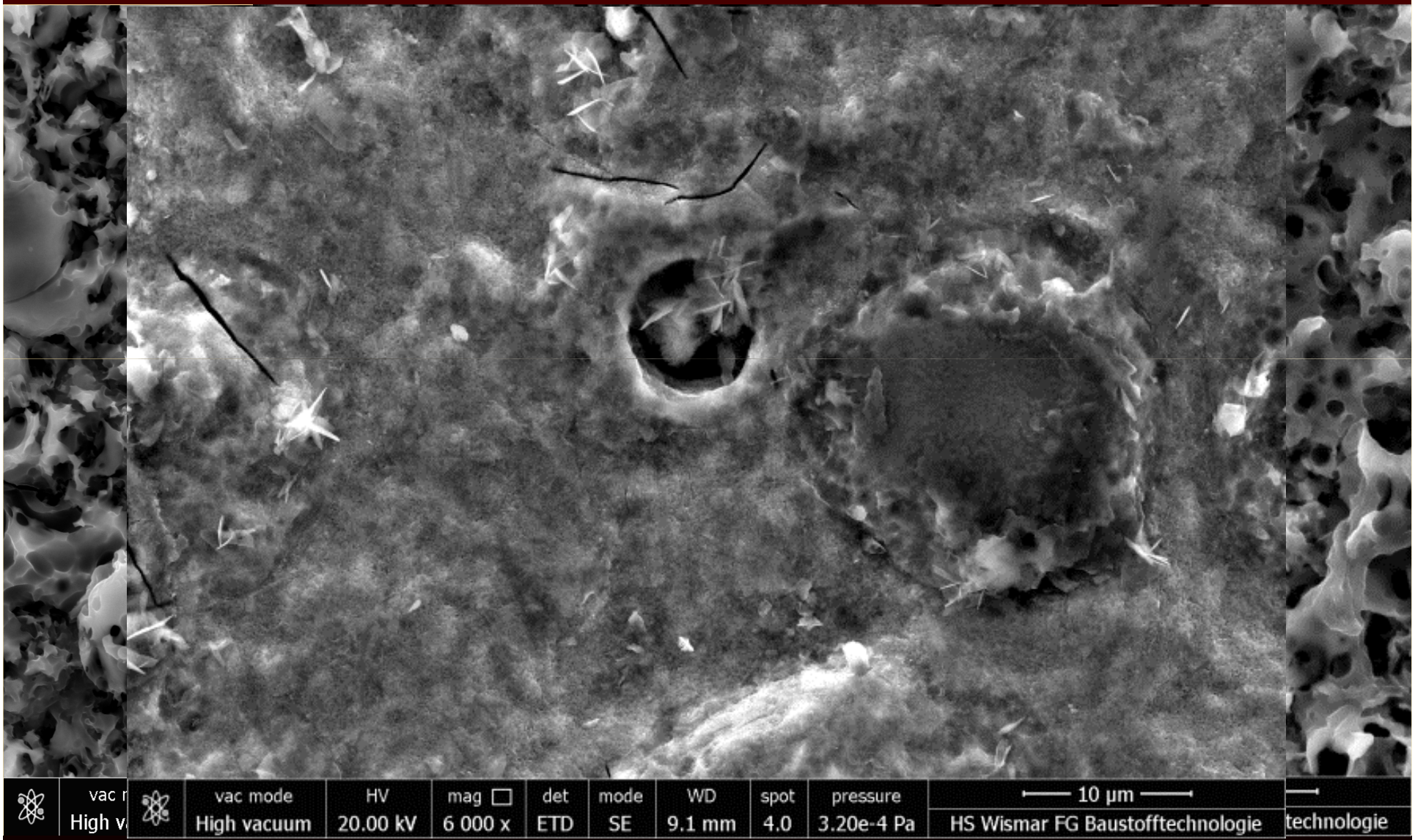
SEM - RESULTS


vac mode High vacuum | HV 20.00 kV | mag 6 000 x | det ETD | mode SE | WD 9.6 mm | spot 2.5 | pressure 3.35e-4 Pa
vac mode High vacuum | HV 20.00 kV | mag 6 000 x | det ETD | mode SE | WD 9.5 mm | spot 4.0 | pressure 2.59e-3 Pa | 5 µm | HS Wismar FG Baustofftechnologie
mag 6 000 x | det ETD | mode SE | WD 9.3 mm | spot 4.0 | pressure 2.33e-4 Pa | 10 µm | HS Wismar FG Baustofftechnologie
vac mode High vacuum | HV 20.00 kV | mag 6 000 x | det ETD | mode SE | WD 9.7 mm | spot 4.0 | pressure 3.42e-4 Pa
vac mode High vacuum | HV 20.00 kV | mag 6 000 x | det ETD | mode SE | WD 9.5 mm | spot 4.0 | pressure 2.61e-4 Pa | 10 µm | HS Wismar FG Baustofftechnologie
mag 6 000 x | det ETD | mode SE | WD 9.0 mm | spot 4.0 | pressure 2.13e-4 Pa | 10 µm | HS Wismar FG Baustofftechnologie

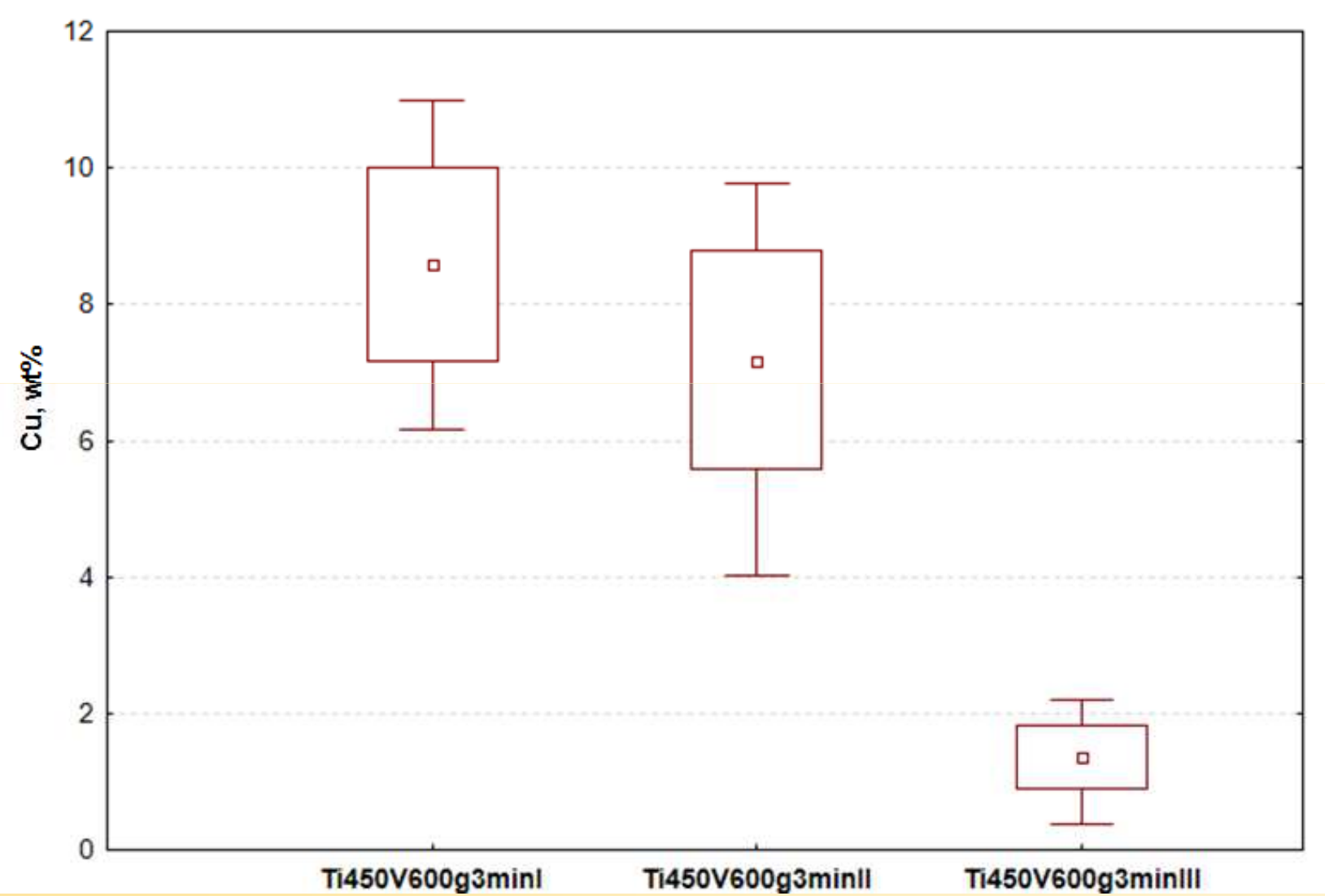
TITANIUM SEM RESULTS



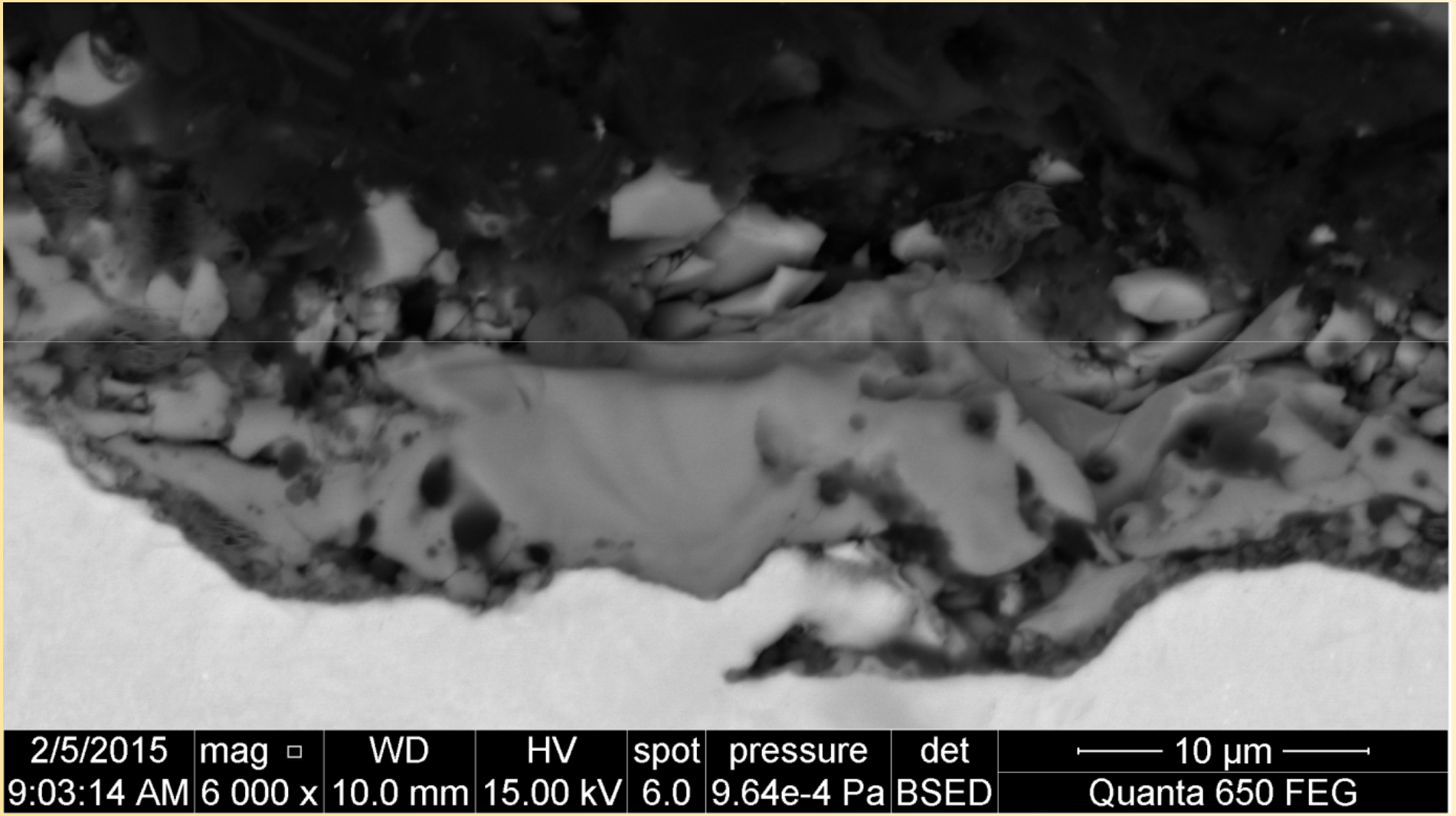
TITANIUM - SEM RESULTS



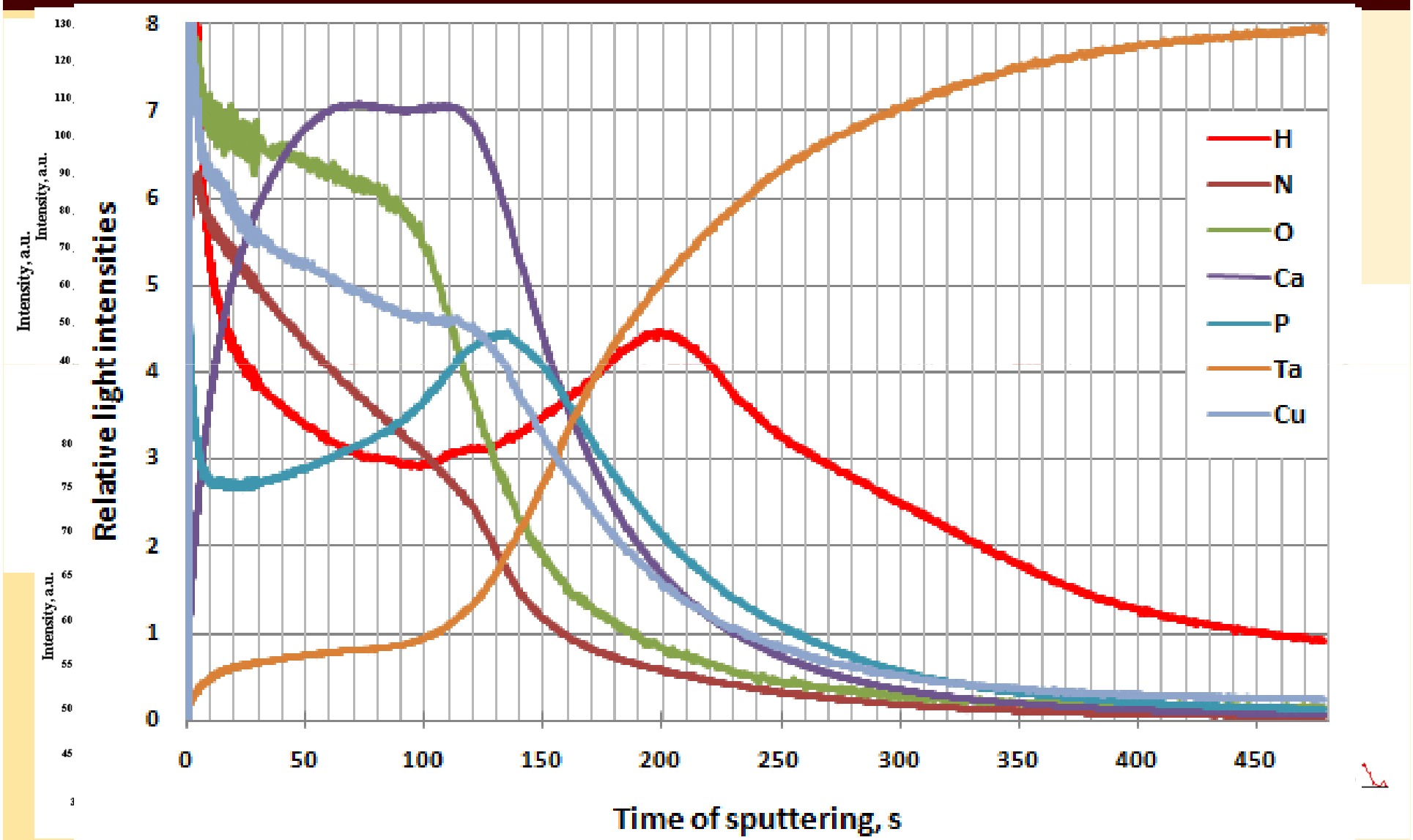
TITANIUM RESULTS



TITANIUM - SEM RESULTS

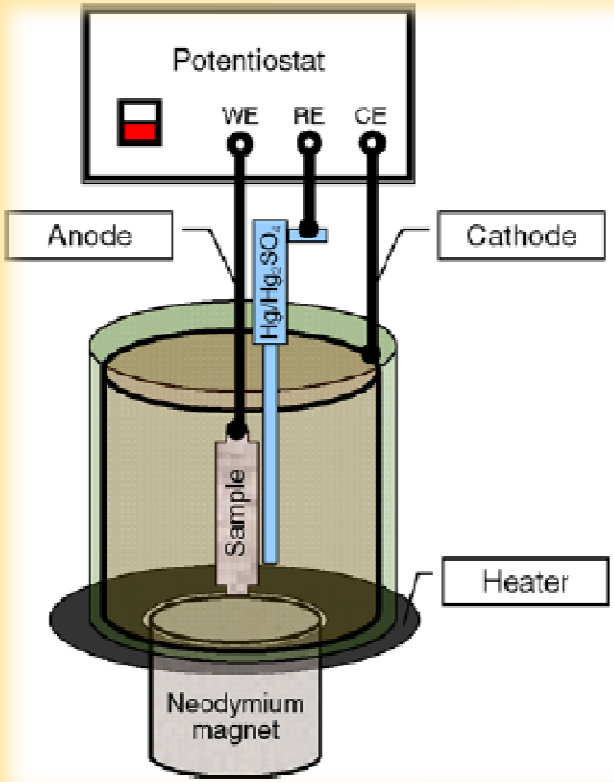


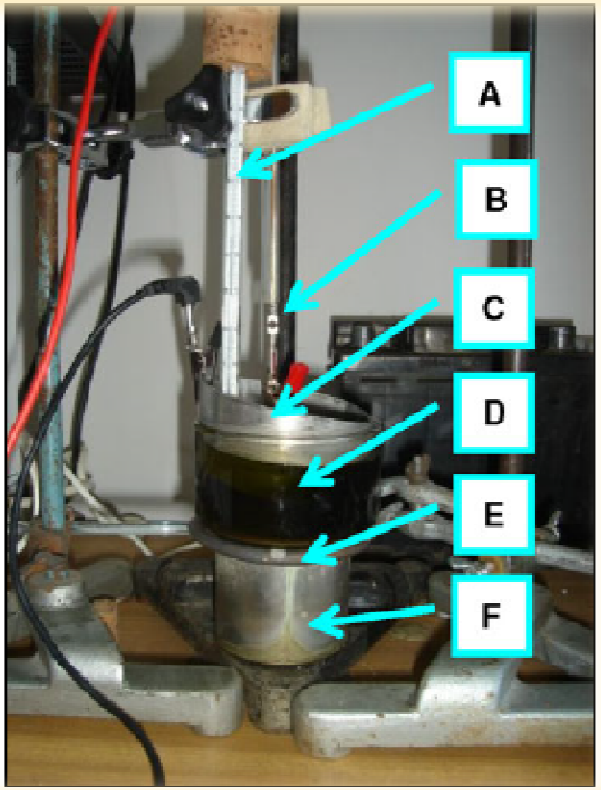
PEO ON TANTALUM

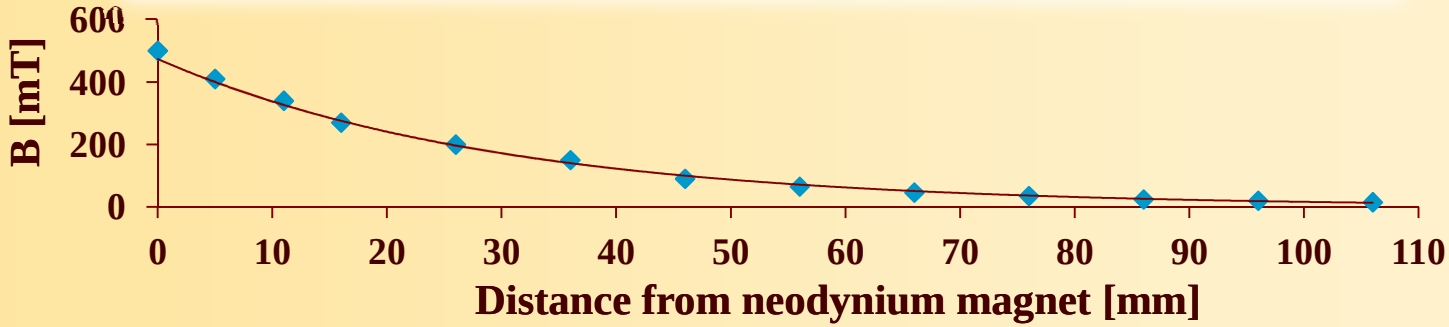


OTHER SCIENTIFIC INTERESTS

SET UP FOR MEP





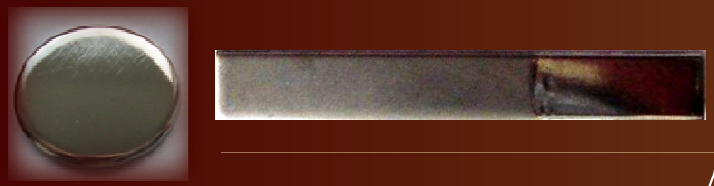


ELECTROPOLISHING

SAMPLES

STEEL

Cylinder: ϕ = 10 [mm], h = 1 [mm]
Cuboid: 5 X 30 X 1 [mm]



NITINOL
dental drills

TITANIUM

Cylinder : ϕ = 0.8 [mm], h = 70 [mm]
Cuboid : 10 x 2 x 50 [mm]

ELECTROLYTE

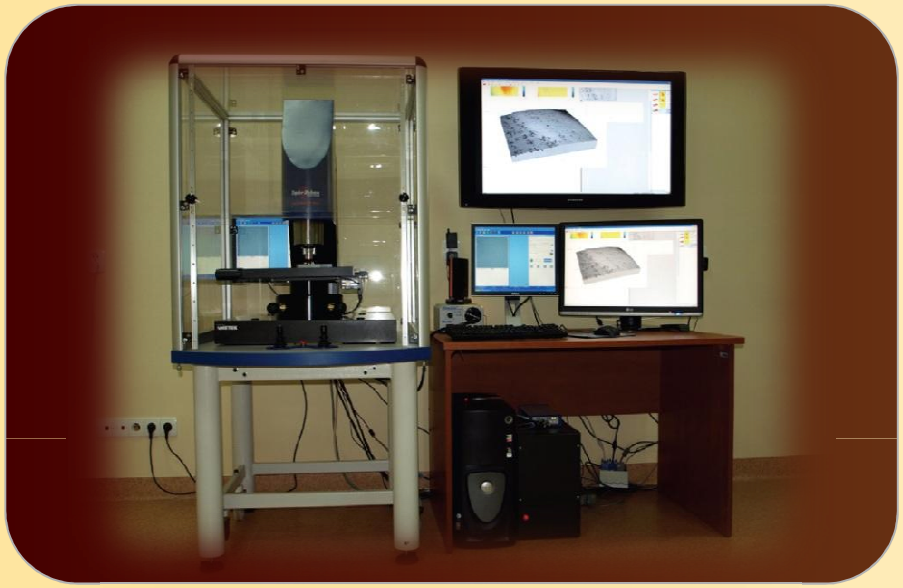
STEEL

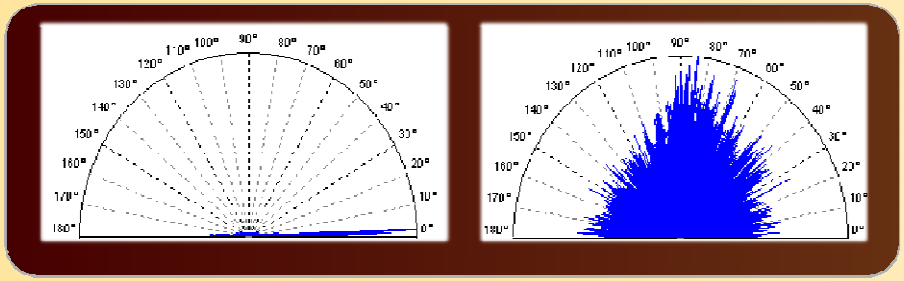
$H_2SO_4 : H_3PO_4$ = 2 : 3 (vol.)
10% of water in electrolyte (vol.)
Conductivity : 5,6 S/cm^2
Temperature: 65 ± 5°C
Density: 1,685 g/cm^3
Viscosity : 215 mPa·s
pH = -1,15

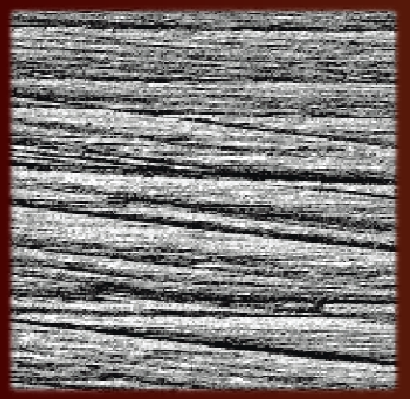
TITANIUM AND ITS ALLOYS

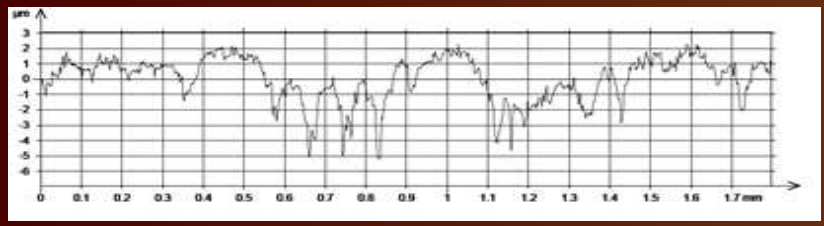
10 % H_2SO_4 + 5 % HF + 85% CH_3OH

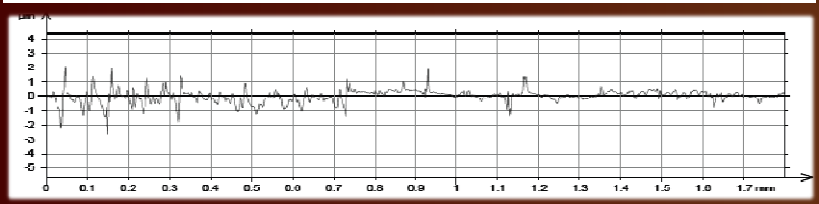
ROUGHNESS OF THE SURFACES









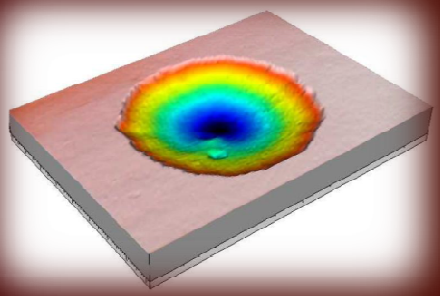


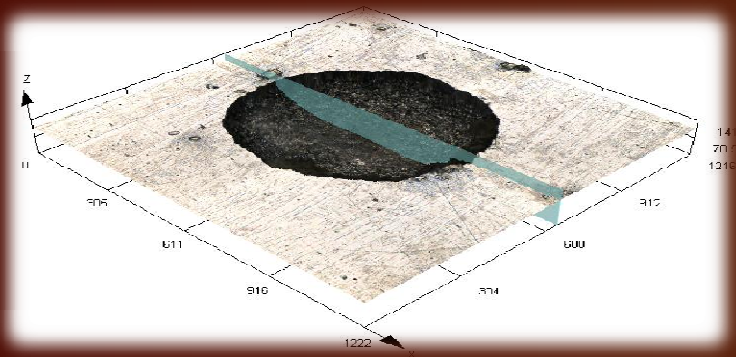
TALYSURF CCI 6000

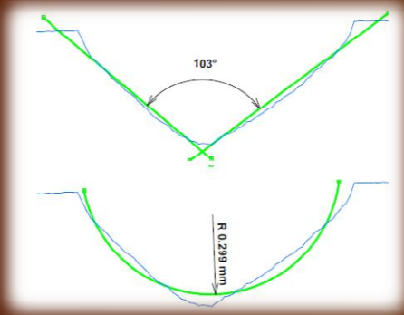
Sa = (1,63 – 3,02)·Ra ; Sa = 0,854·Sq

PITS GEOMETRY

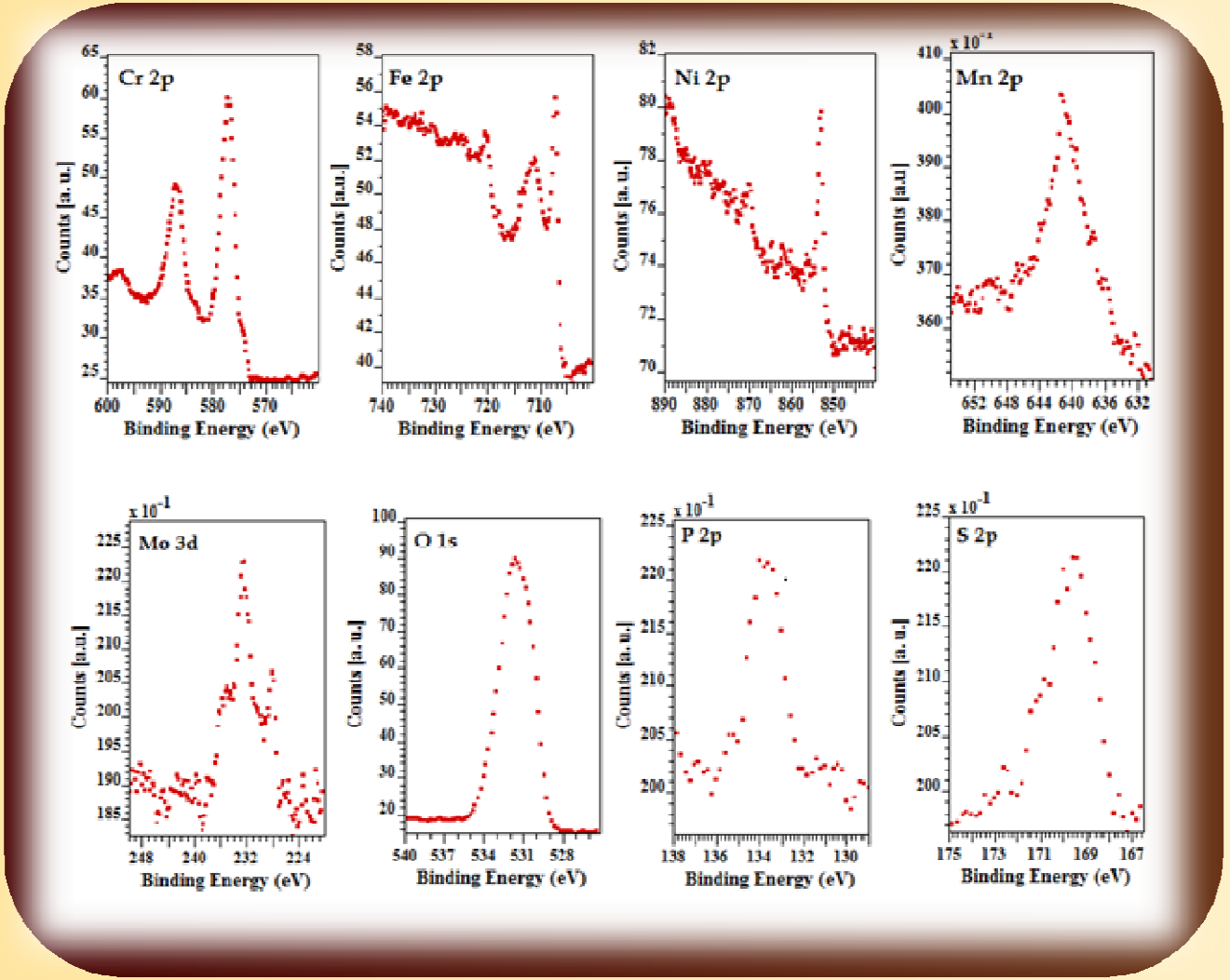
OLYMPUSLEXT OLS 4000

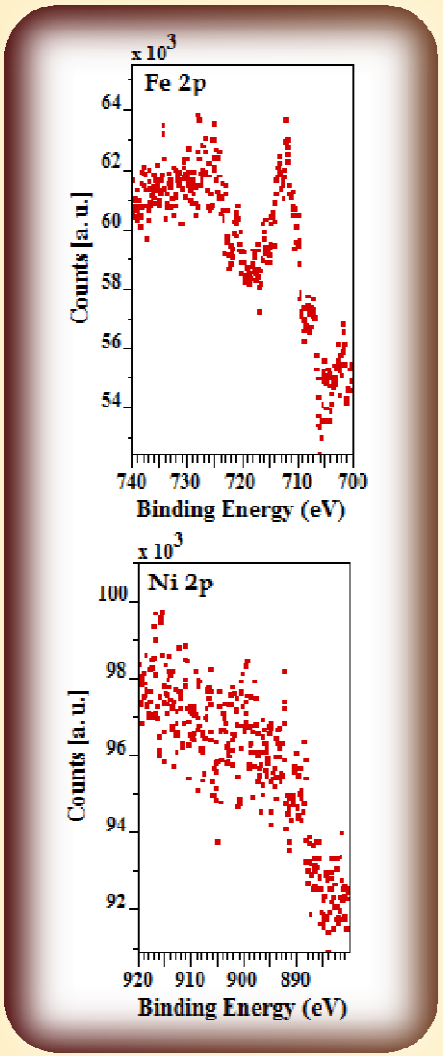




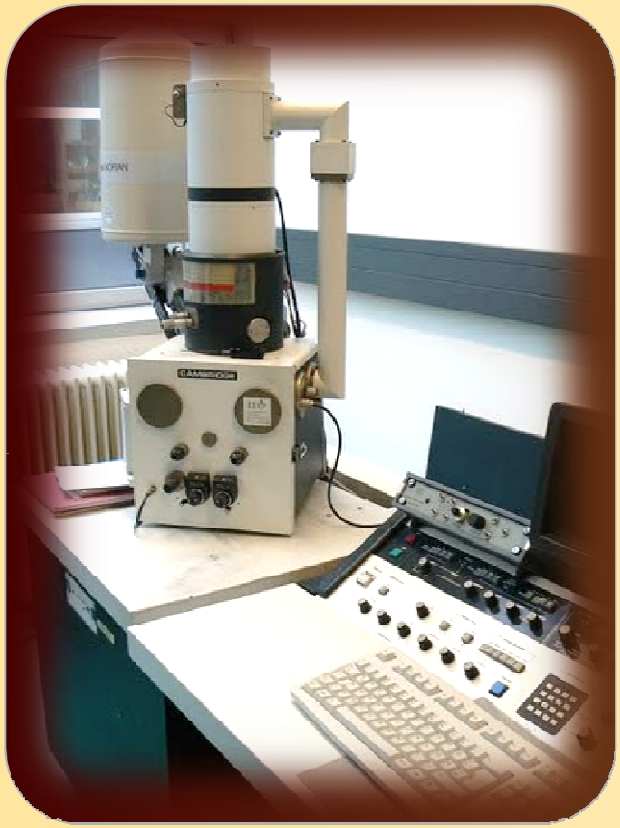


AISI 316L - XPS RESULTS



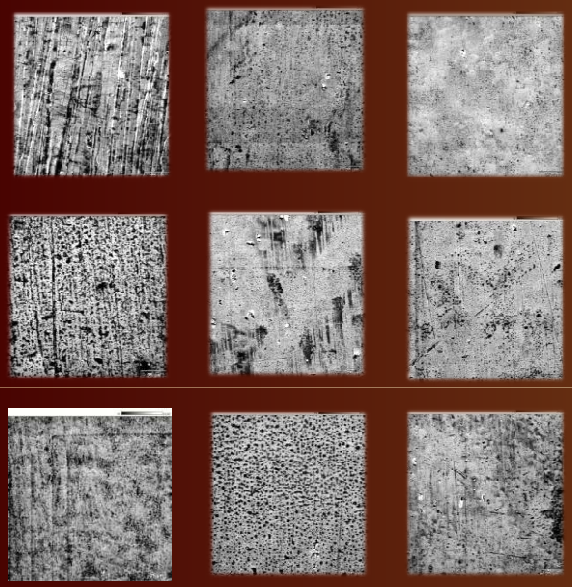


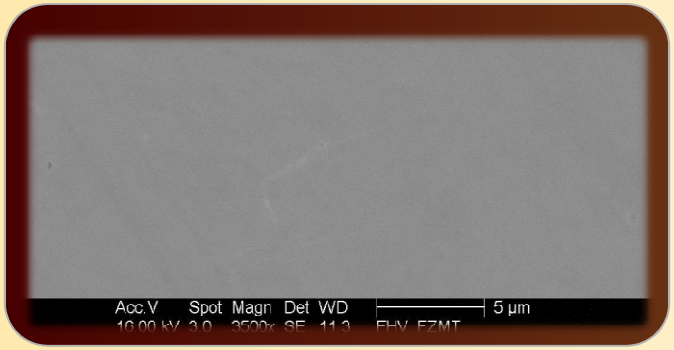
SEM/EDX



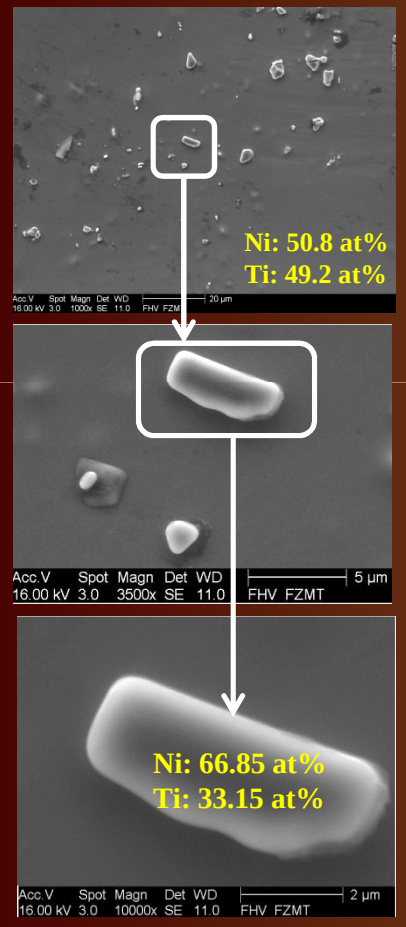
CAMBRIDGE S200

AISI 316L SS





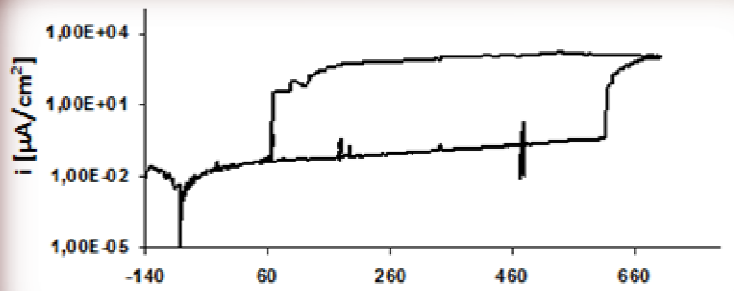
NITINOL



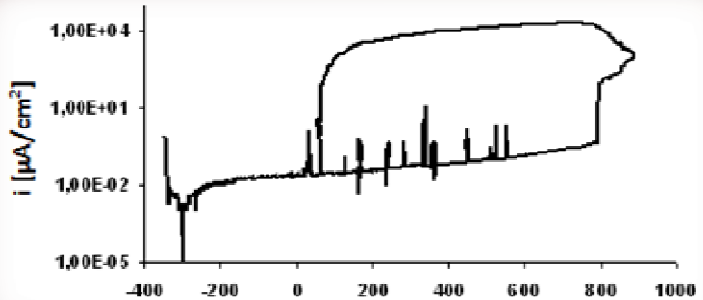
HOCHSCHULE NEUBRANDENURG (GERMANY)

PITTING CORROSION – AISI 316L SS

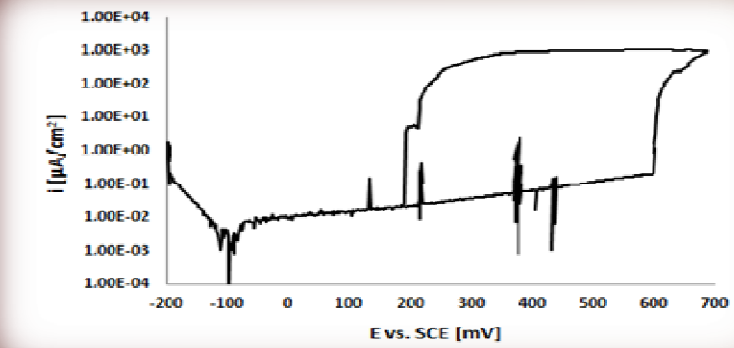
EP - 50 A/dm²



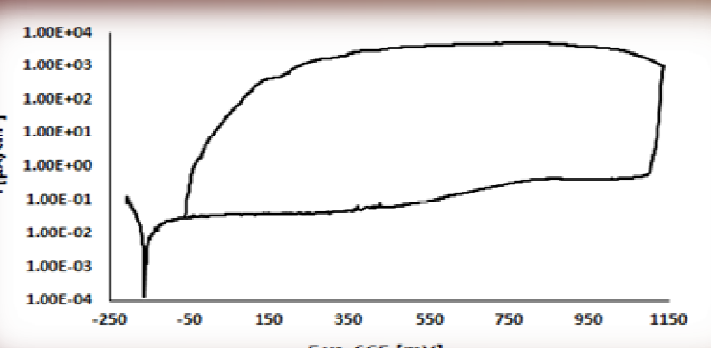
MEP - 200 A/dm²



EP - 130 A/dm²



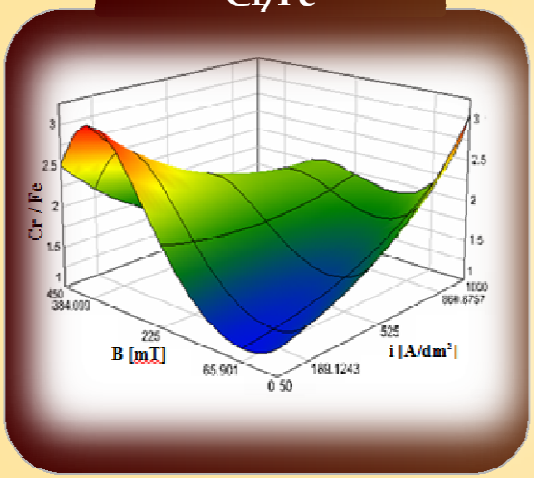
MEP - 130 A/dm²



Rokosz K., Hryniewicz T., Raaen S., Characterization of Passive Film Formed on AISI 316L Stainless Steel after Magnetoelectropolishing in a Broad Range of Polarization Parameters, Steel Research International, Vol. 83, Online ISSN: 1869-344X, 2012, 1-9,.

SURFACE LAYERS AFTER MEP

Cr/Fe

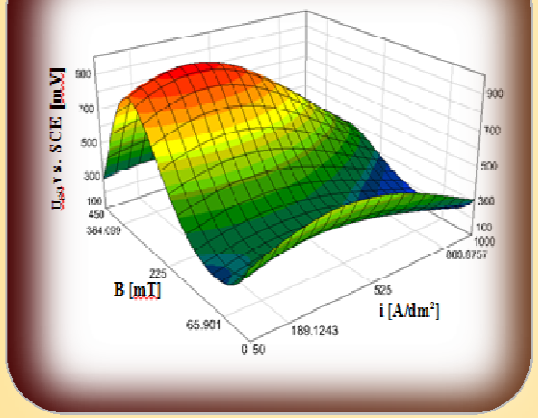


$$Epit(B,i) = \alpha(B,i) \pm 372{,}017 \cdot \sqrt{\beta(B,i)}$$

$$0 \quad mT \le B \le 450 \quad mT$$

$$50 \quad \frac{A}{dm^2} \le i \le 1000 \quad \frac{A}{dm^2}$$

CORROSION

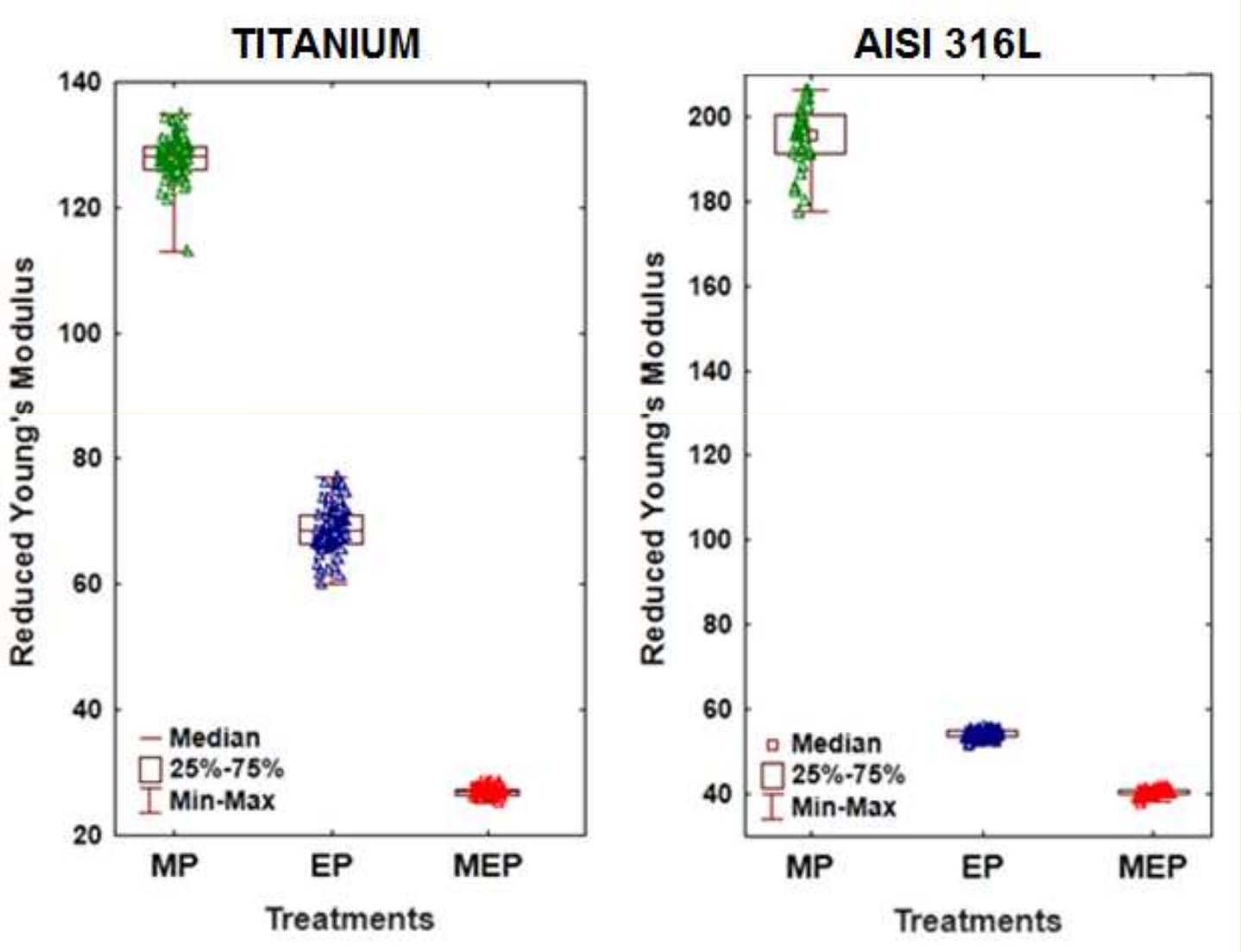


$$\alpha(B,i) = 4717 - 3{,}96 \cdot B - 6{,}178 \cdot i - \frac{417}{10^5} B \cdot i + \frac{2}{10^3} B^2 - \frac{228}{10^5} i^2 - \frac{349}{10^{11}} B^2 i^2 - \frac{7{,}9 \cdot 10^5}{B+i}$$

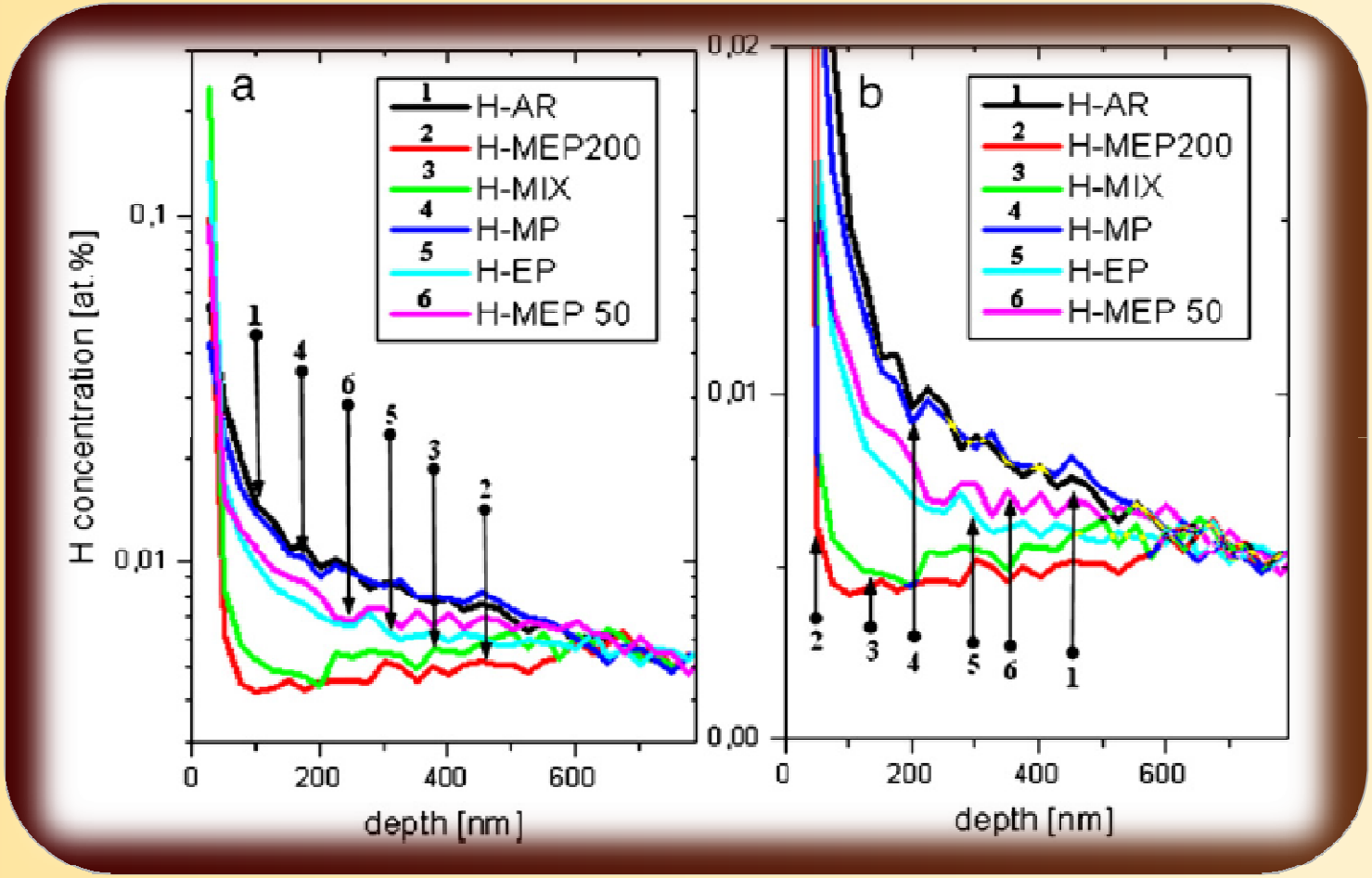
$$\beta(B,i) = 54{,}5 - 0{,}218 \cdot B - 18{,}5 \cdot 10^{-2} \cdot i + 6{,}58 \cdot 10^{-4} \cdot B \cdot i + 3{,}94 \cdot 10^{-4} \cdot B^2 + 2{,}47 \cdot 10^{-4} \cdot i^2 +$$
$$- 3{,}49 \cdot 10^{-10} \cdot (B \cdot i)^2 - 9 \cdot 10^{-7} \cdot B^2 \cdot i - 6{,}71 \cdot 10^{-7} \cdot B \cdot i^2 - 4{,}08 \cdot 10^{-7} \cdot B^3 - 15.36 \cdot$$
$$\cdot 10^{-8} \cdot i^3 + 2{,}29 \cdot 10^{-10} \cdot B^4 + 3{,}84 \cdot 10^{-11} \cdot i^4 + 2{,}26 \cdot 10^{-10} \cdot B \cdot i^3 + 4{,}76 \cdot 10^{-13} \cdot B^2 \cdot$$
$$\cdot i^3 - 2{,}34 \cdot 10^{-16} \cdot B^2 \cdot i^4 + 4{,}58 \cdot 10^{-10} \cdot B^3 \cdot i + 7{,}02 \cdot 10^{-13} \cdot B^3 \cdot i^2 - 11{,}08 \cdot 10^{-16} \cdot B^3$$
$$\cdot i^3 - 6{,}22 \cdot 10^{-16} \cdot B^4 \cdot i^2 + 8{,}57 \cdot 10^{-22} \cdot (B \cdot i)^4 - \frac{17060}{B+i} + \frac{32{,}4 \cdot B}{B+i} + \frac{28{,}4 \cdot i}{B+i} - 4{,}02 \cdot 10^{-2} \cdot$$
$$\cdot \frac{B \cdot i}{B+i} - 18{,}42 \cdot 10^{-3} \cdot \frac{B^2}{B+i} - 13{,}02 \cdot 10^{-3} \cdot \frac{i^2}{B+i} + {,}34 \cdot 10^{-8} \cdot \frac{3(B \cdot i)^2}{B+i} + 137 \cdot 10^4 \frac{1}{(B+i)^2}$$

ROKOSZ K., POLEROWANIE ELEKTROCHEMICZNE STALI W POLU MAGNETYCZNYM, WYD. UCZ. POLITECHNIKI KOSZALIŃSKIEJ, KOSZALIN 2012.

NANOINDENTATION



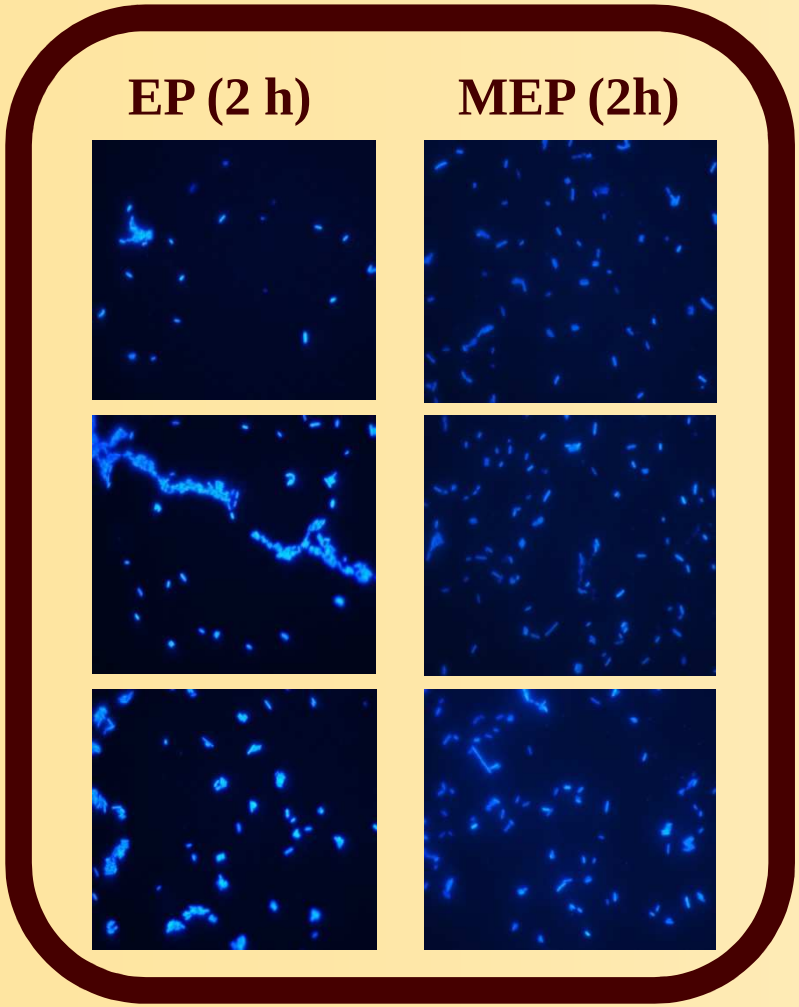
HYDROGENATION – AISI 316L SS

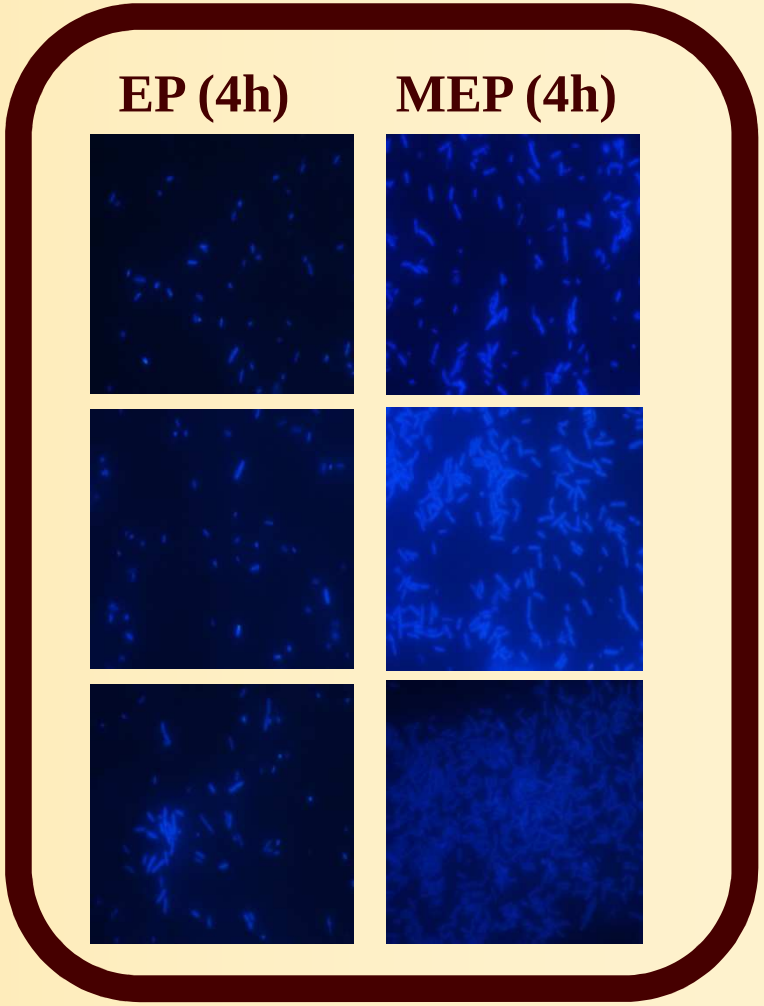


INSTYTUT TELE-I RADIOTECHNICZNY, LABORATORIUM BADAWCZO-POMIAROWE TECHNIKI PRÓŻNI

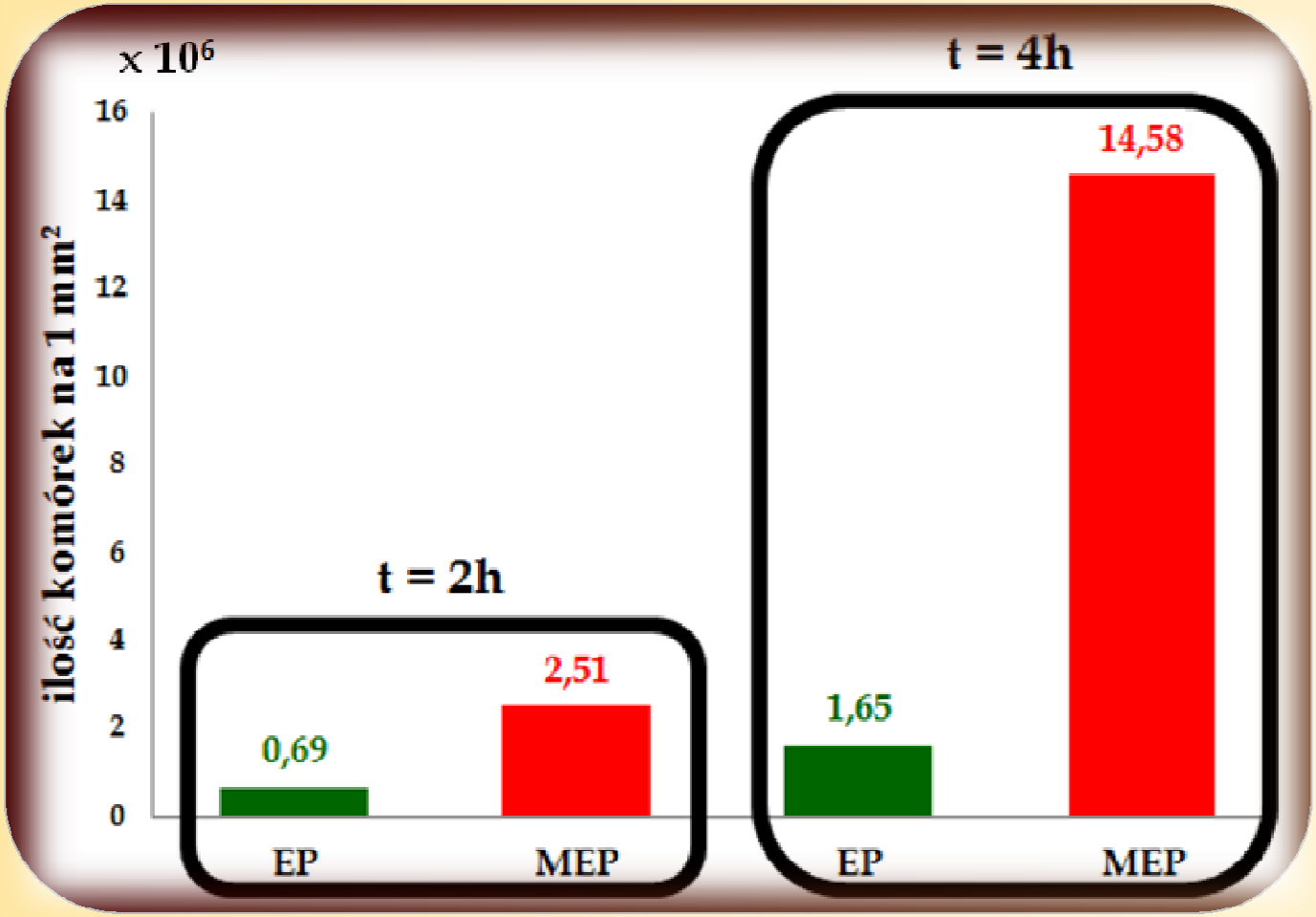
HRYNIEWICZ T., KONARSKI P., ROKOSZ K., ROKICKI R., SIMS ANALYSIS OF HYDROGEN CONTENT IN NEAR SURFACE LAYERS OF AISI 316L SS AFTER ELECTROLYTIC POLISHING UNDER DIFFERENT CONDITIONS, SURFACE & COATINGS TECHNOLOGY, 2011, 205, 4228–4236.

BIOLOGICAL STUDIES – AISI 316L

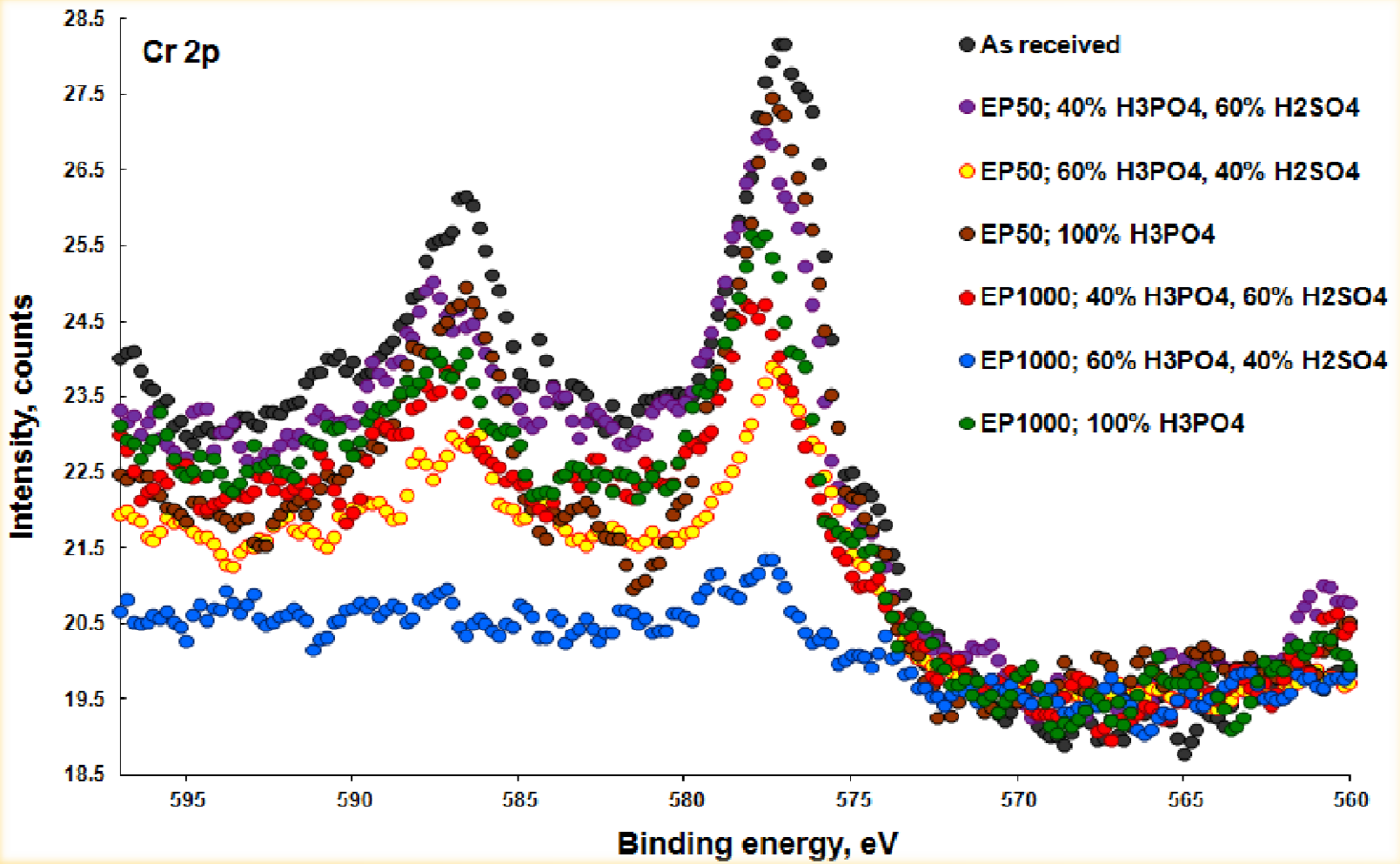




BIOLOGICAL STUDIES – AISI 316L



HIGH-CURRENT DENSITY ELECTROPOLISHING



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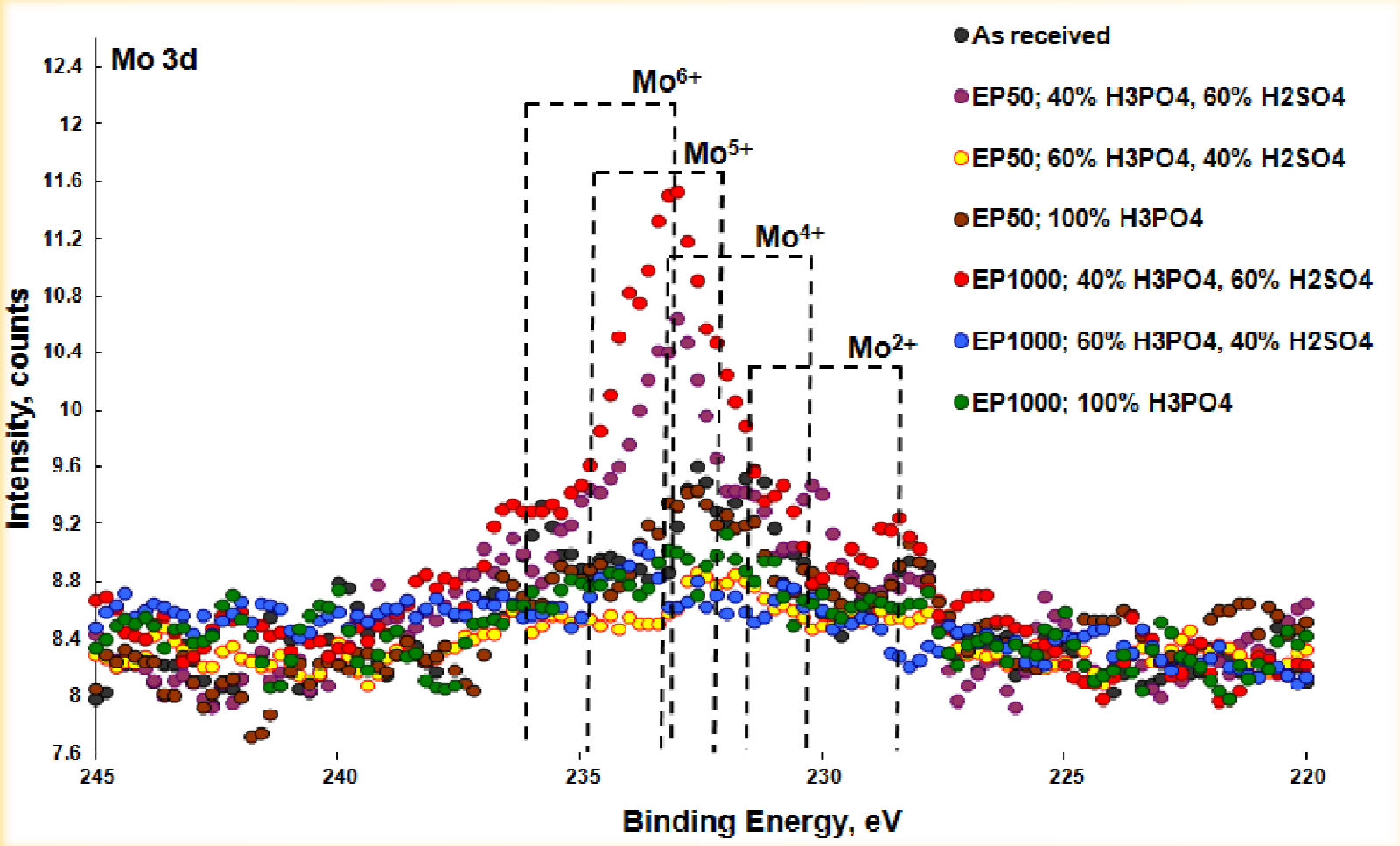

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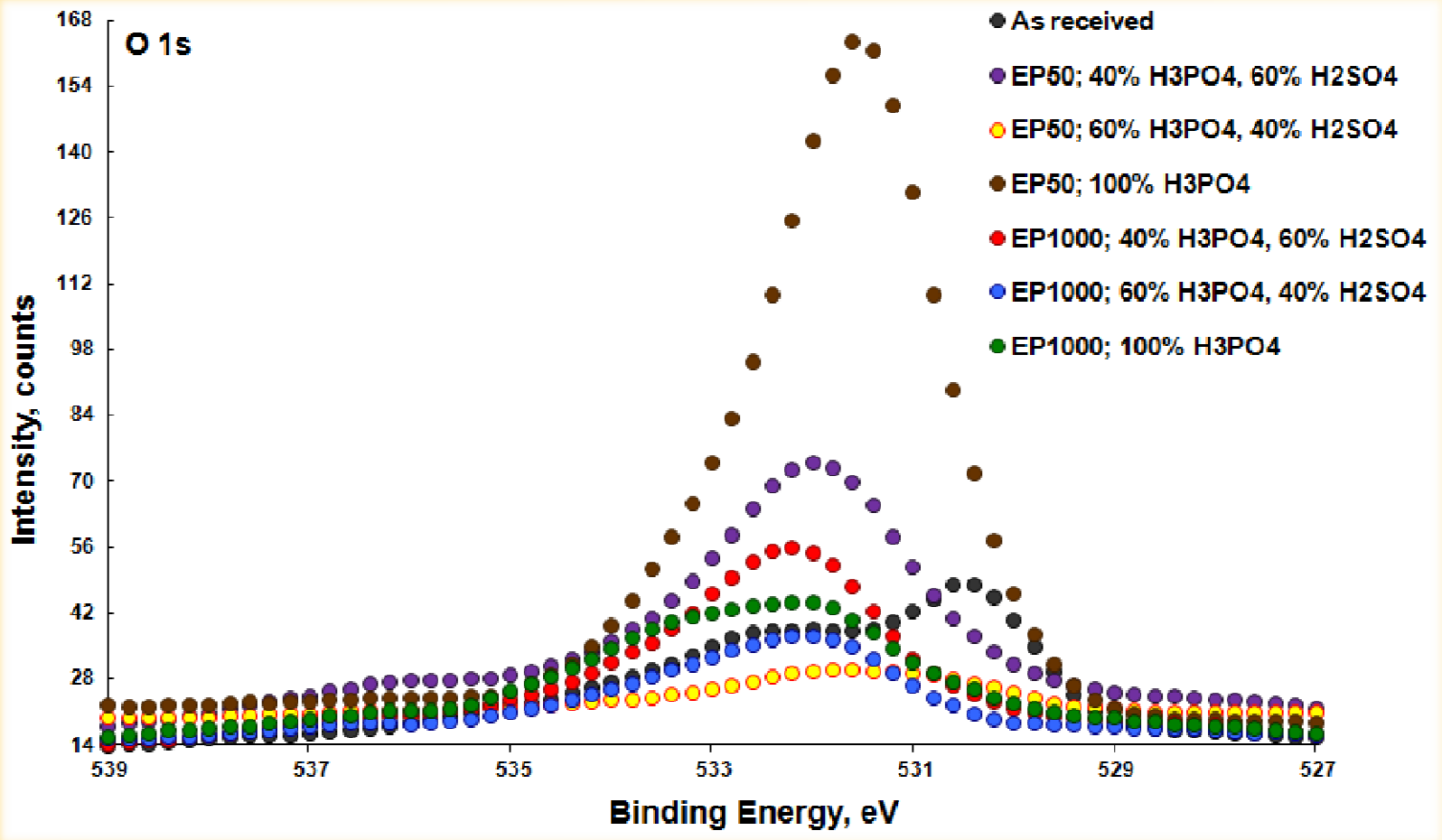

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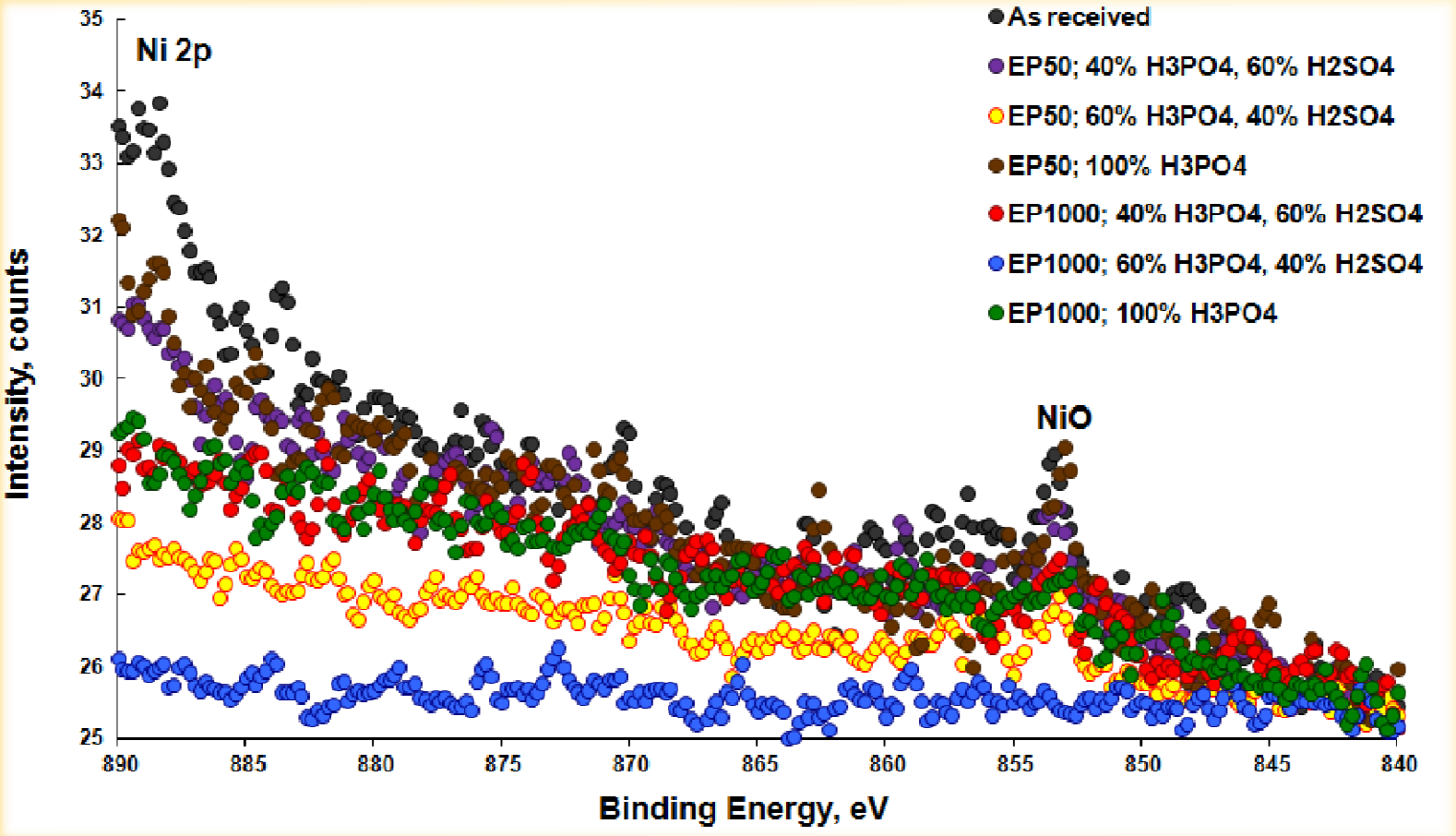

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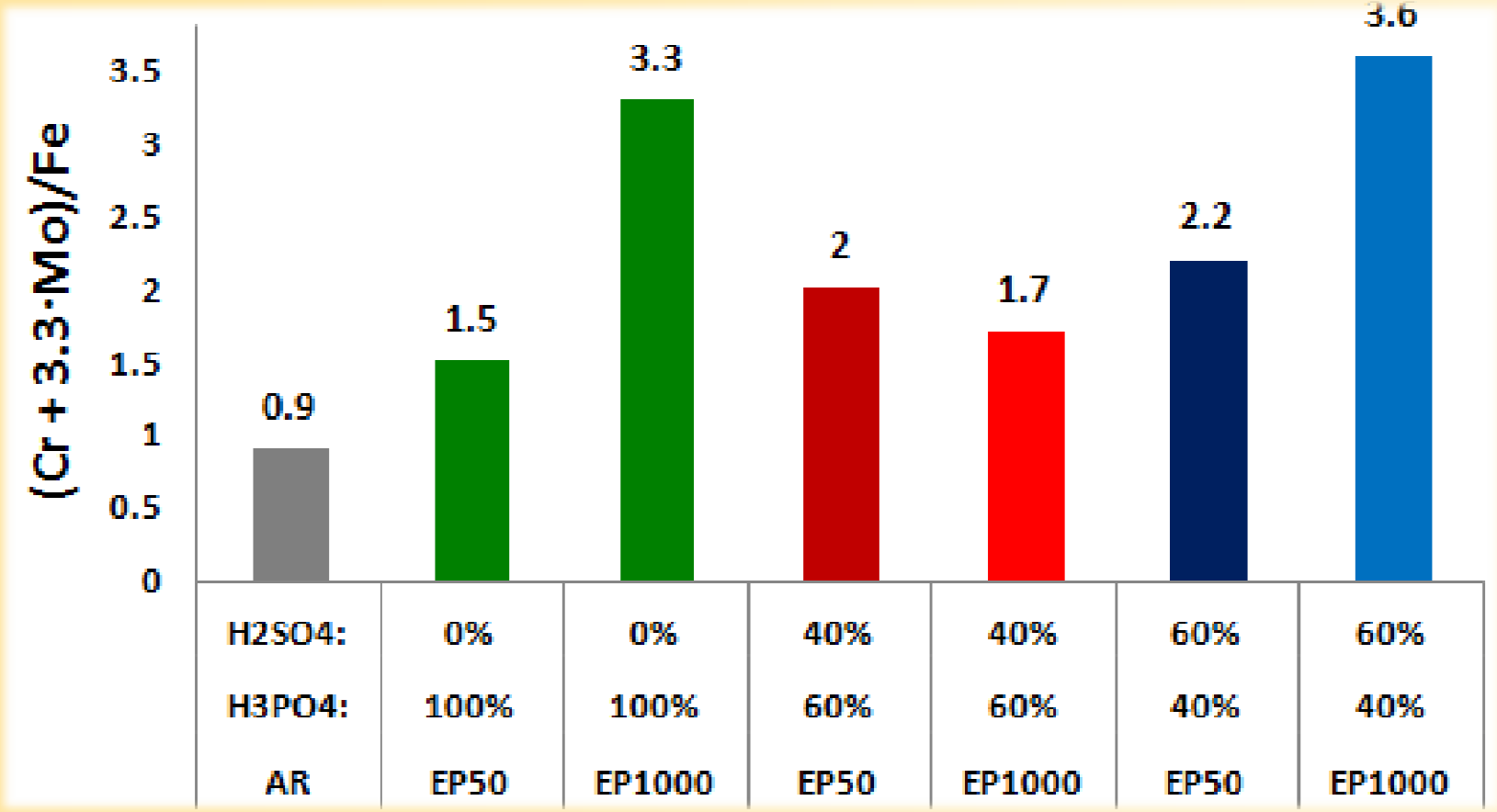

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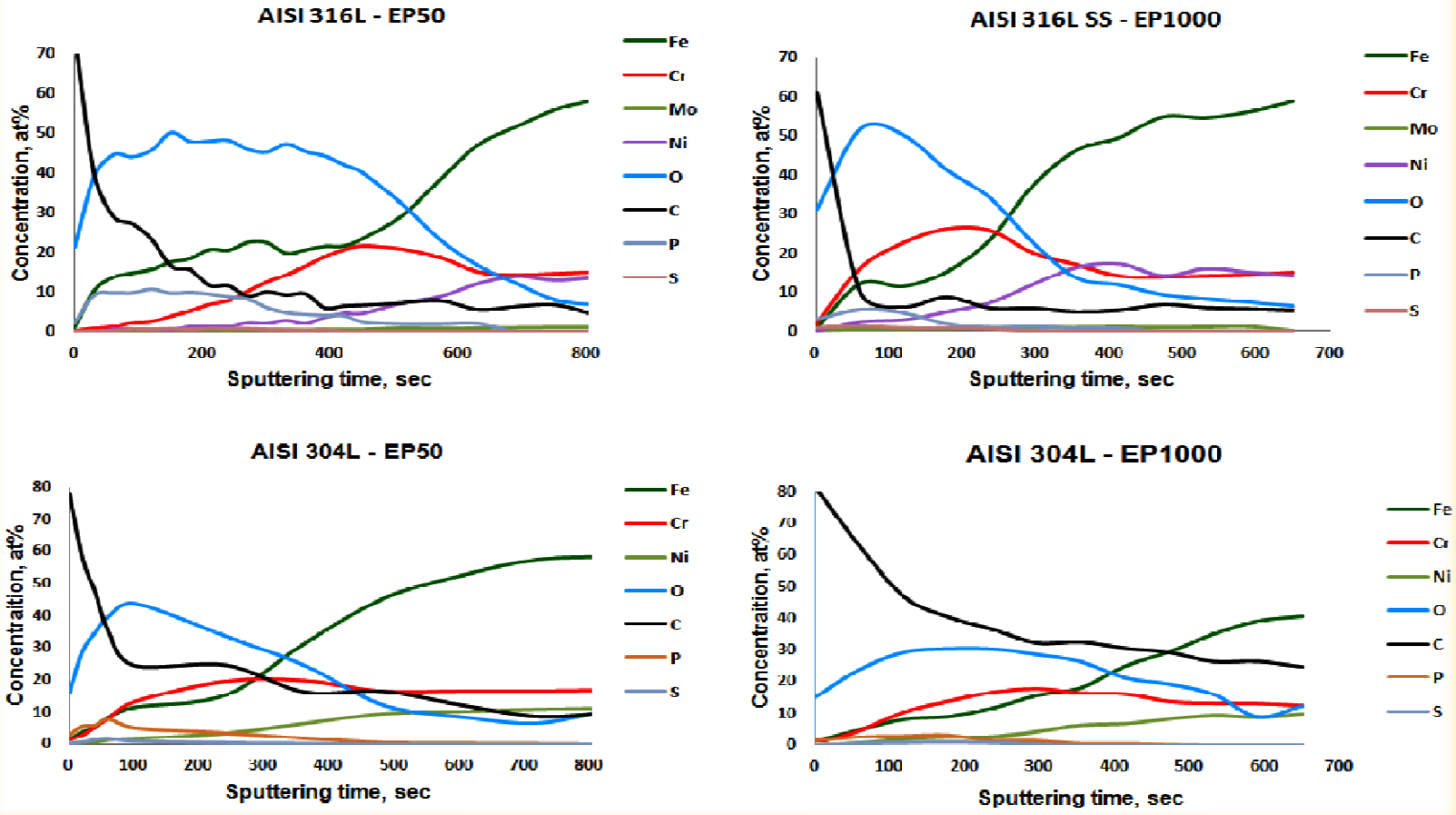

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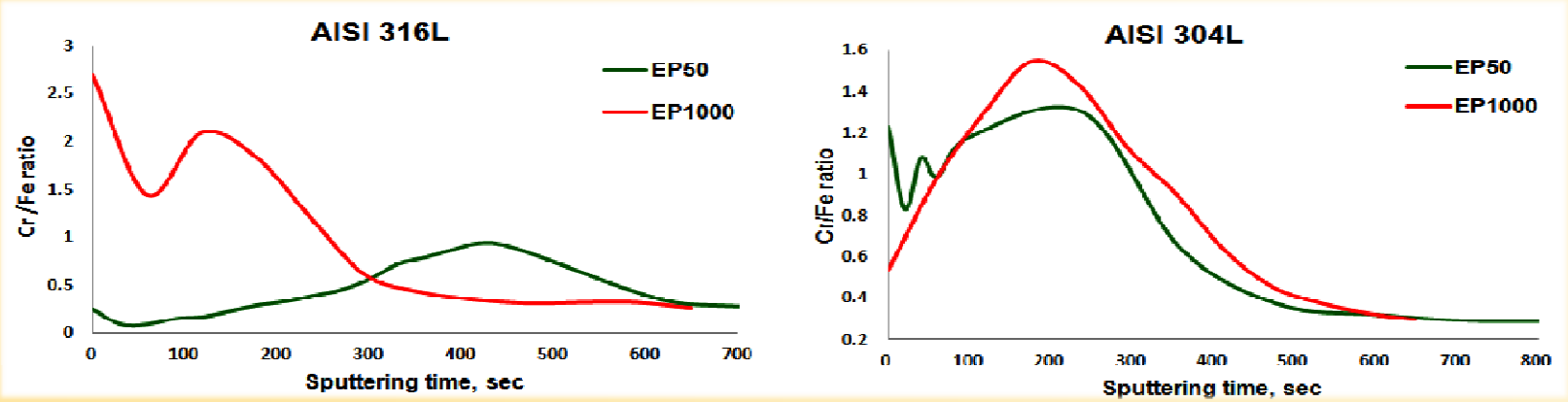

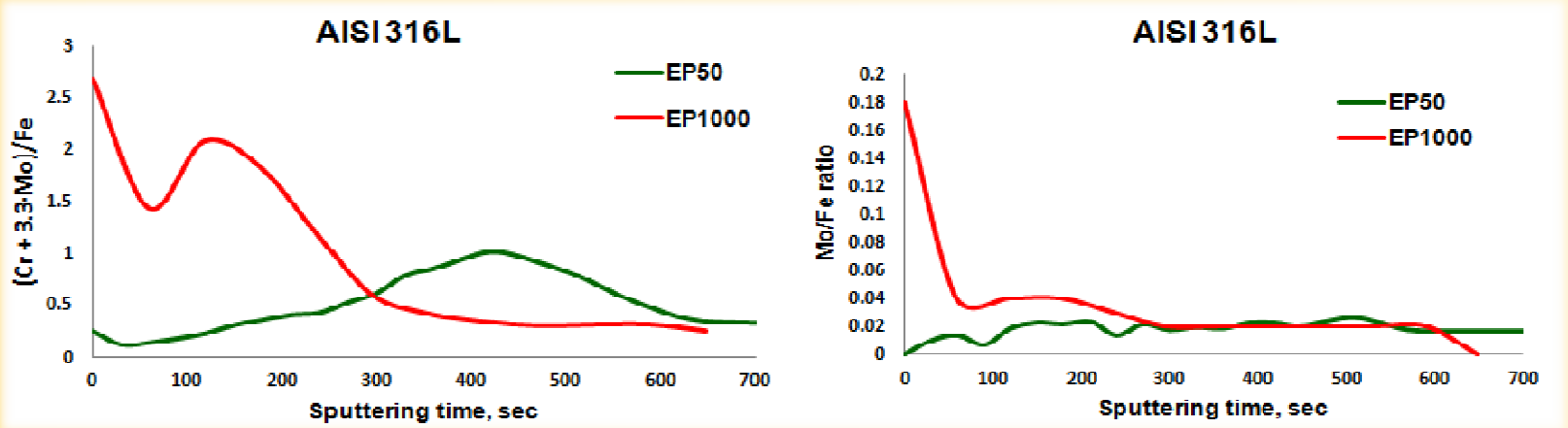

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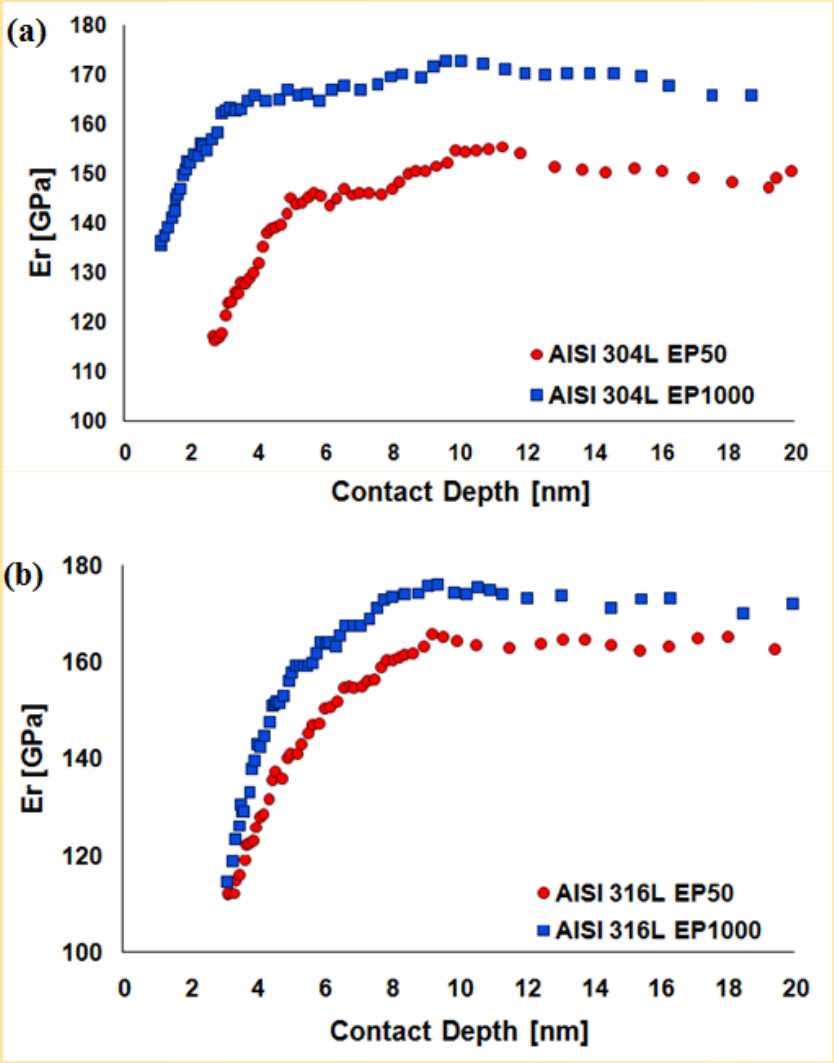
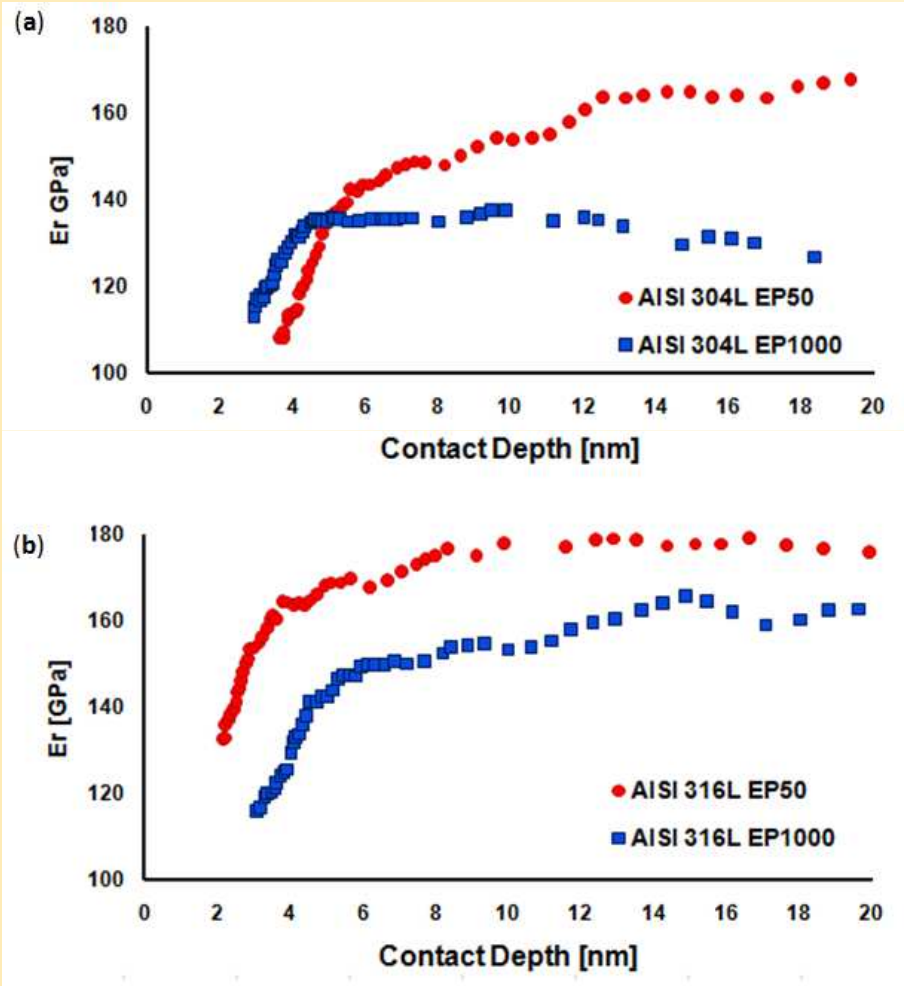
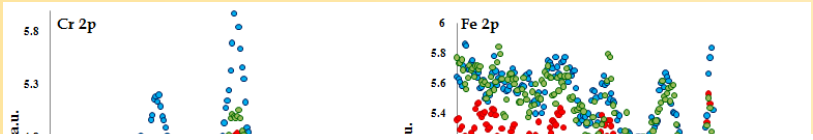
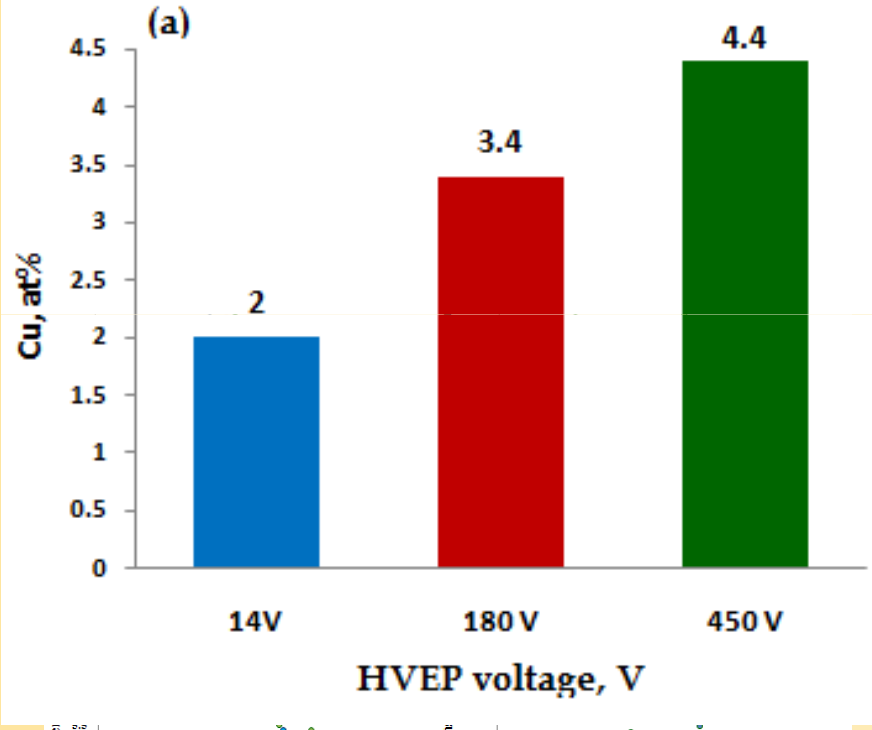
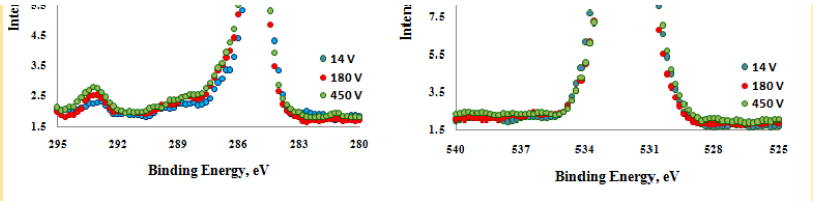


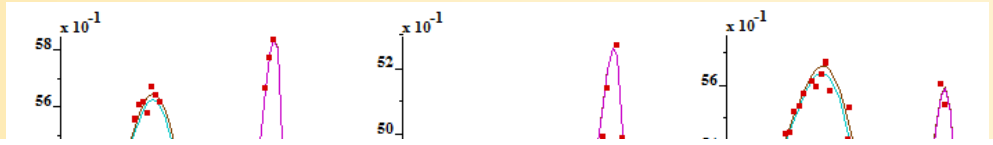


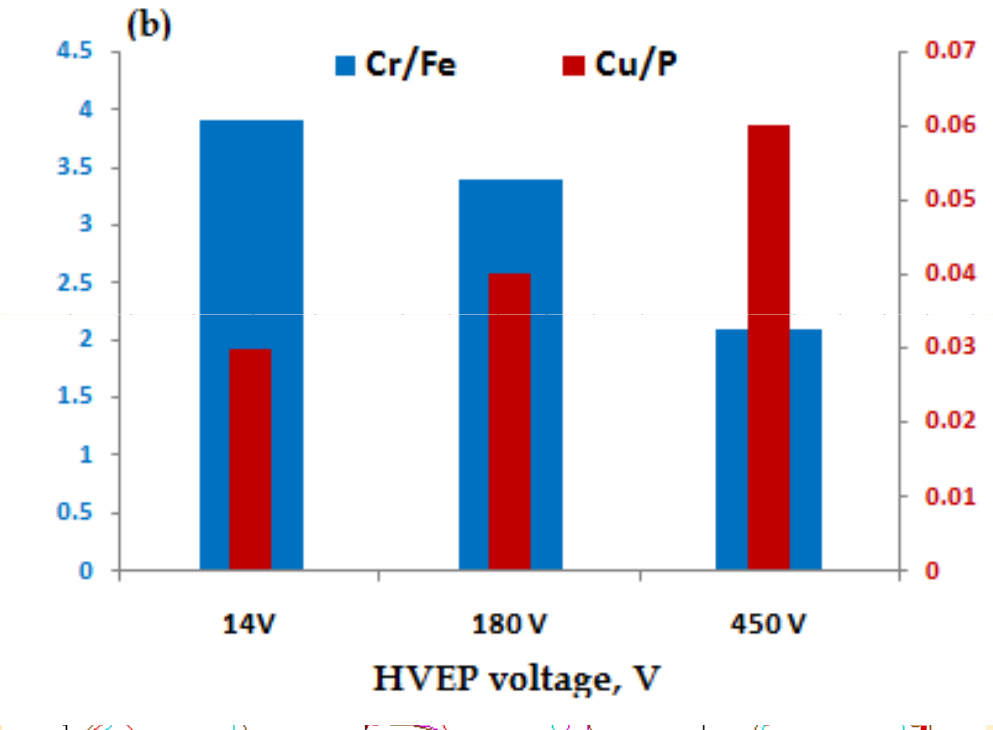
EP - AISI 304L SS

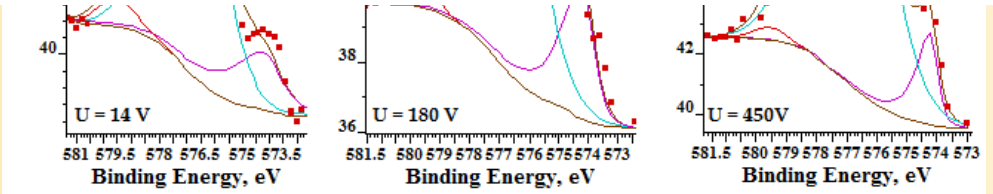












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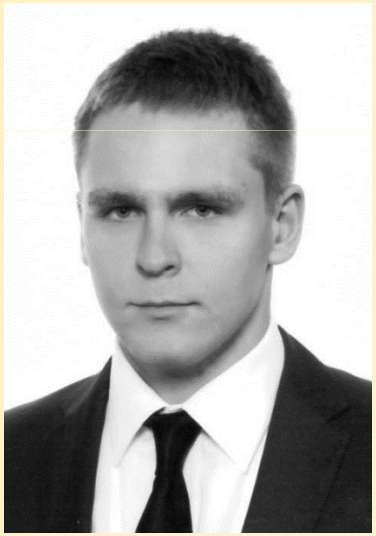
K. Rokosz

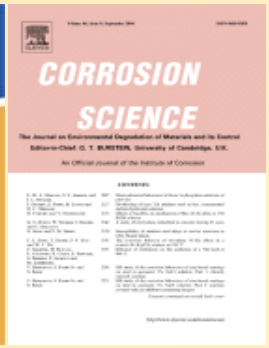
Ł. Dudek

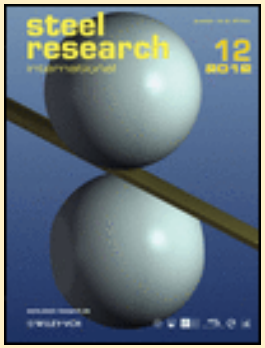
S. Rzadkiewicz

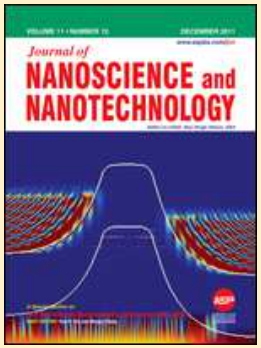


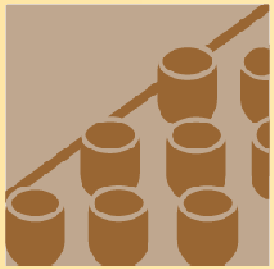
K. Pietrzak

PUBLICATIONS

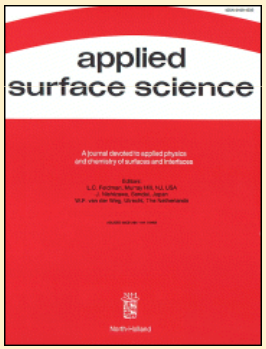


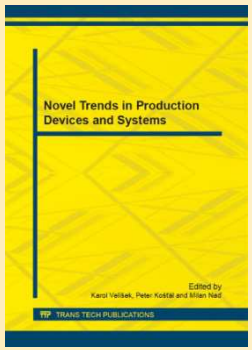


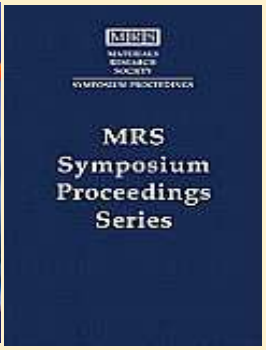




materials







Prof. nzw. dr hab. inż. Krzysztof Rokosz

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THANK YOU FOR ATTENTION