



Electrochemistry
Group

Functionalization of hard tissue implants surface by plasma
electrooxidation

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Gliwice, Poland
Osteoplast R&D, Dębica, Poland

17-19.10.2018, Sumy, Ukraine

Anodic oxidation

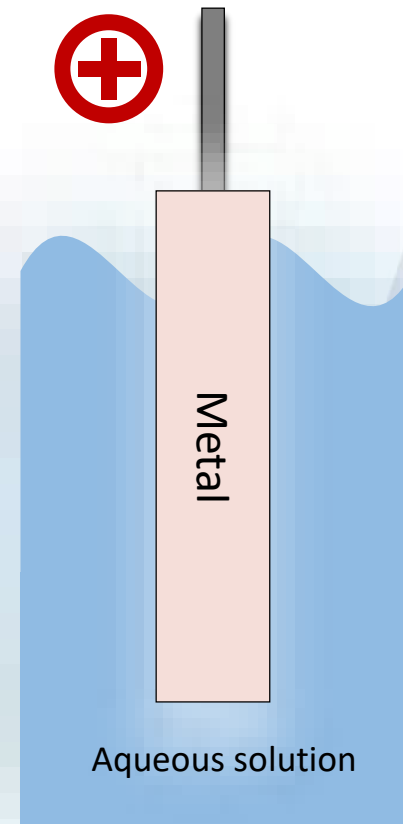


Anodic oxidation of metals and alloys

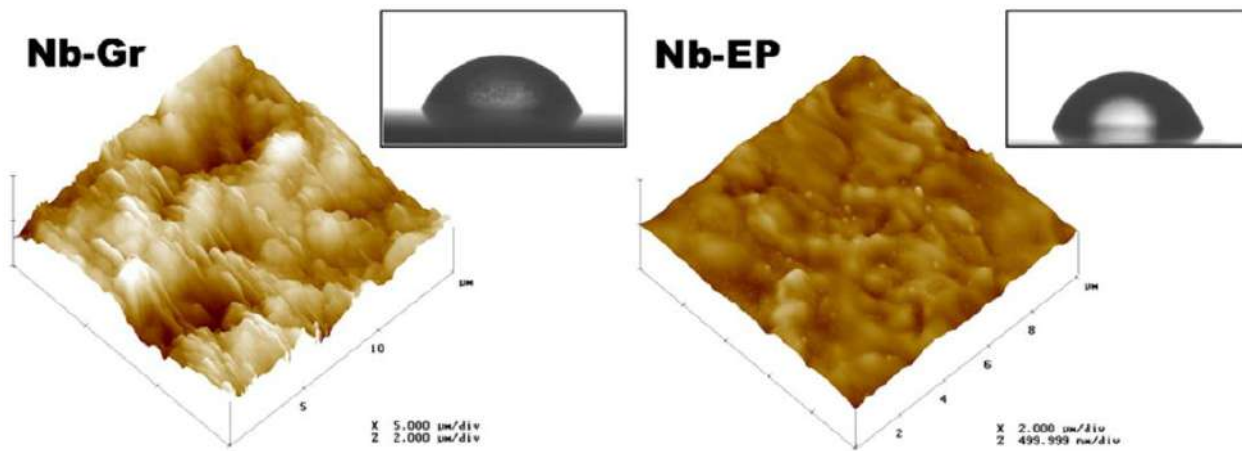
The process is based on **anodic polarisation** of the treated element

As a result of the process is

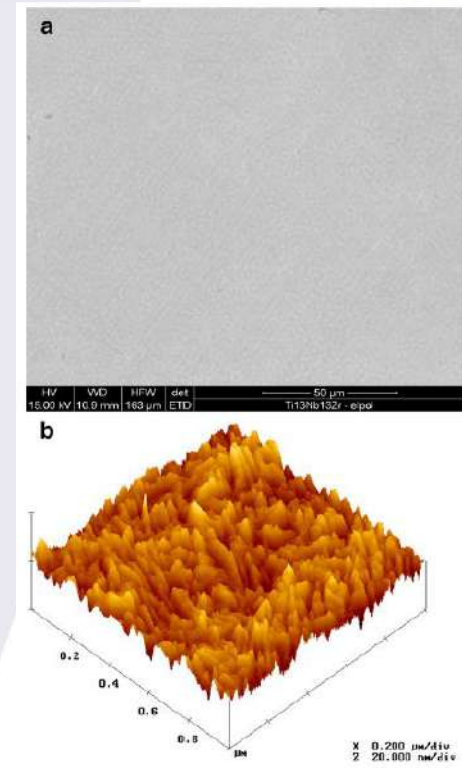
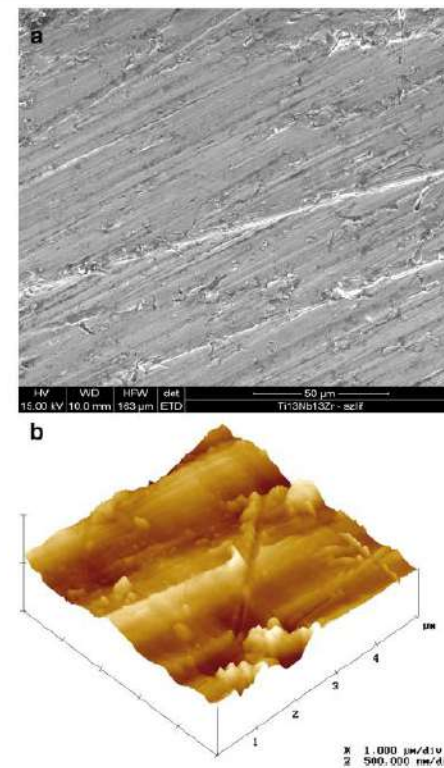
???



Electropolishing

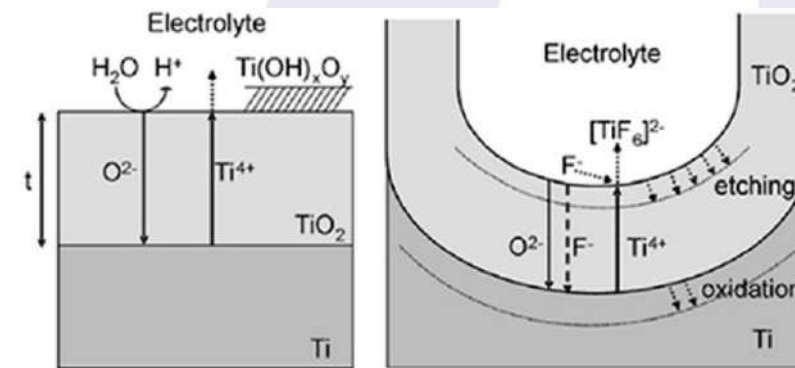
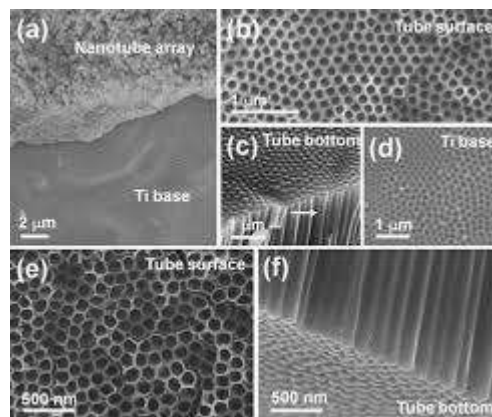
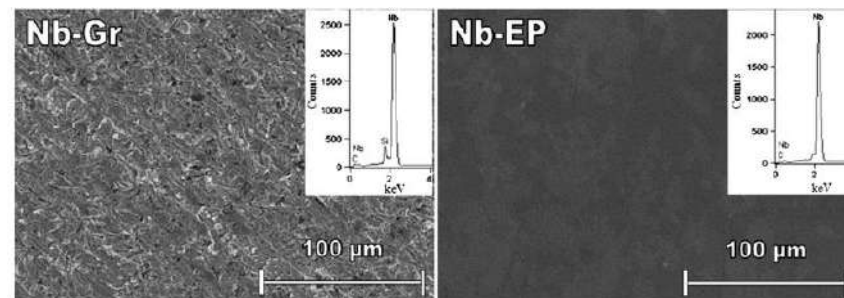
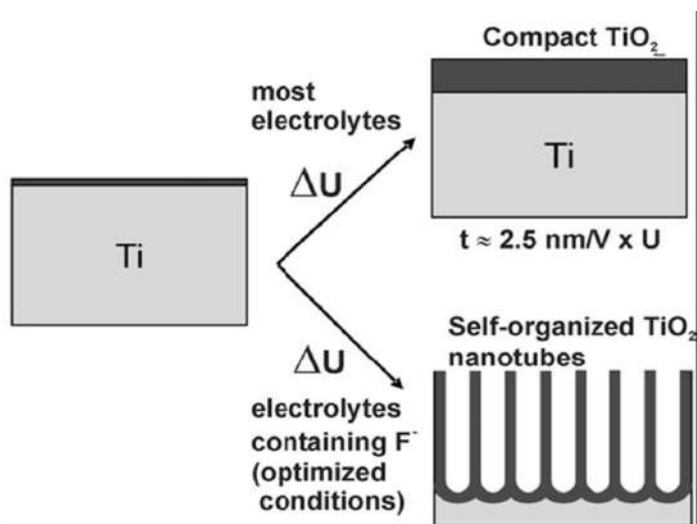


AFM images of Nb before and after electropolishing



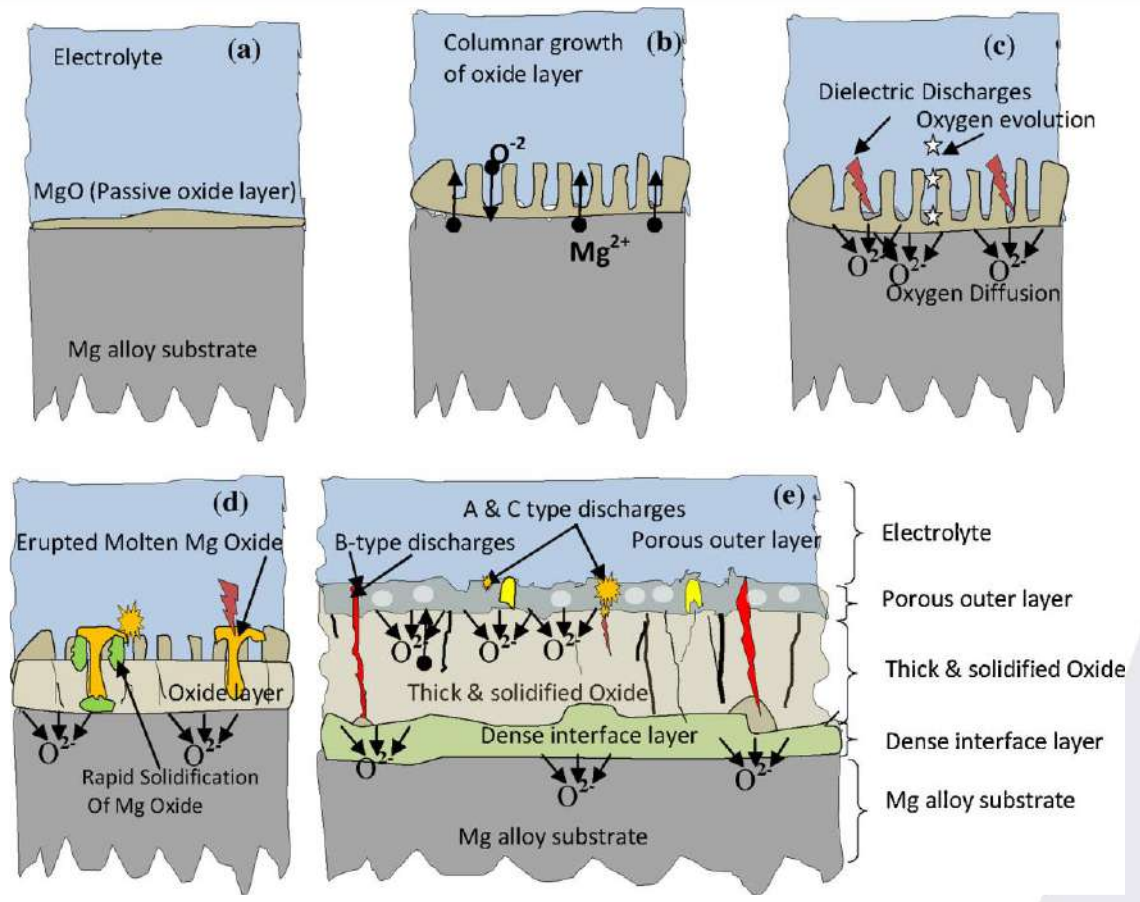
SEM (a) and AFM (B) images of Ti-13Nb-13Zr alloy before and after electropolishing

Anodic oxidation





Plasma electrolytic oxidation (PEO)



Plasma electrolytic oxidation (PEO)



Parameters:

- Voltage
- Current type (AC, DC, pulse)
- Current density
- Electrolyte chemical composition
- Temperature
- Time
- Pretreatment



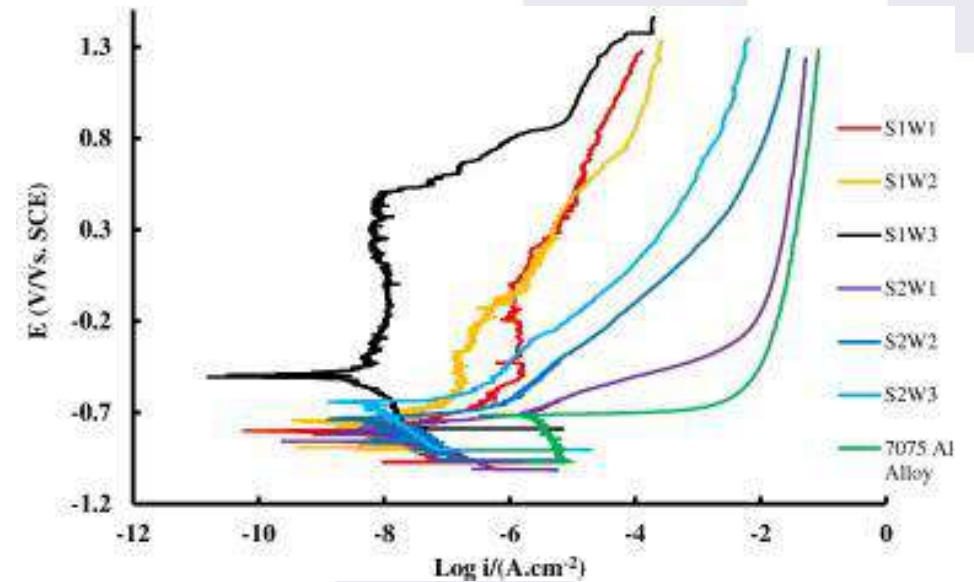
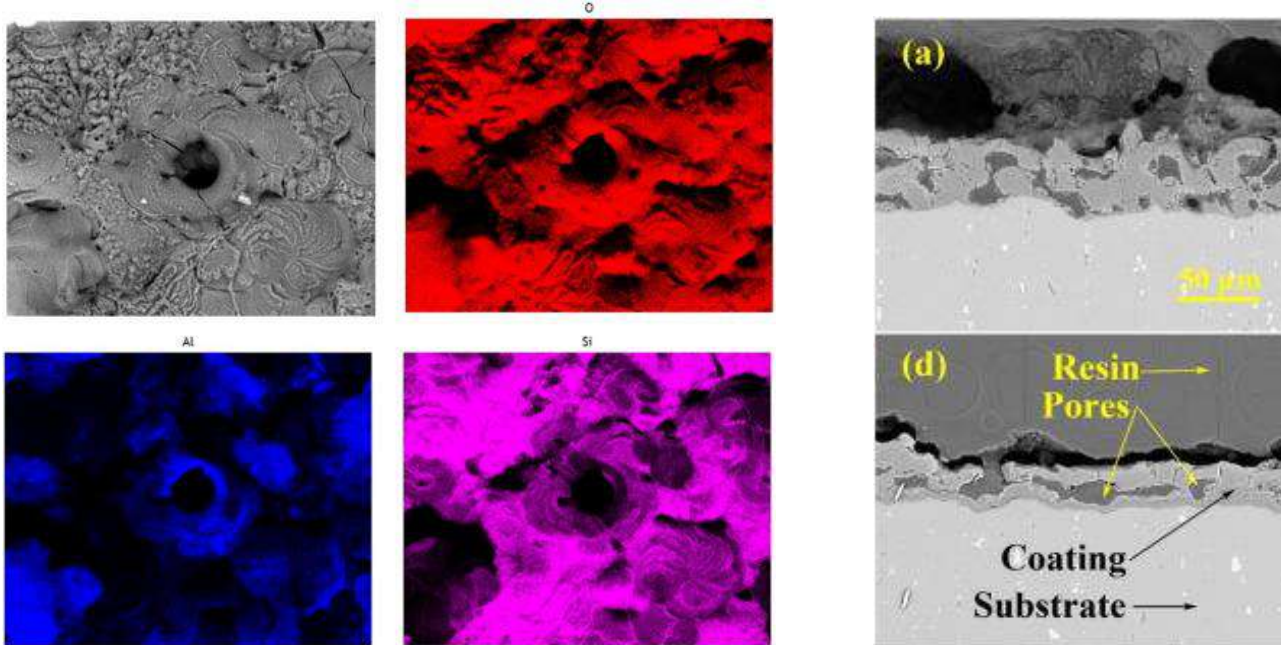
Different results:

- Morphology, topography
- Oxide layer thickness
- Chemical composition of oxide layer
- Mechanical properties
- Corrosion resistance
- Biological properties

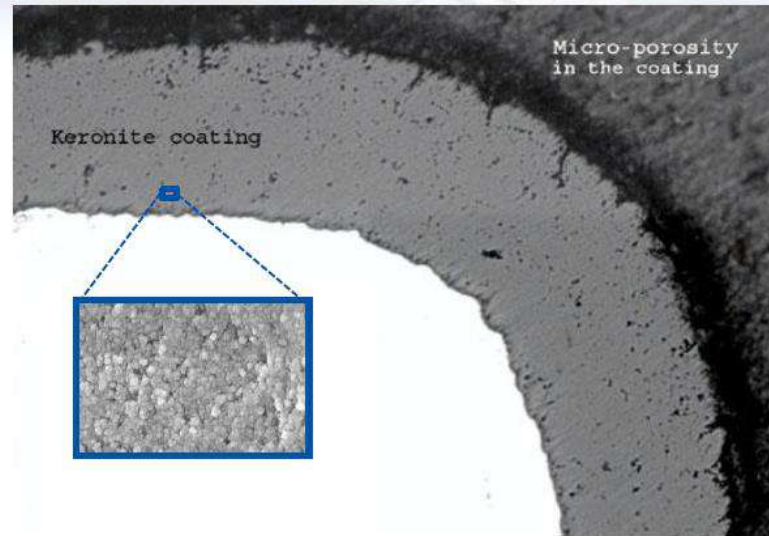
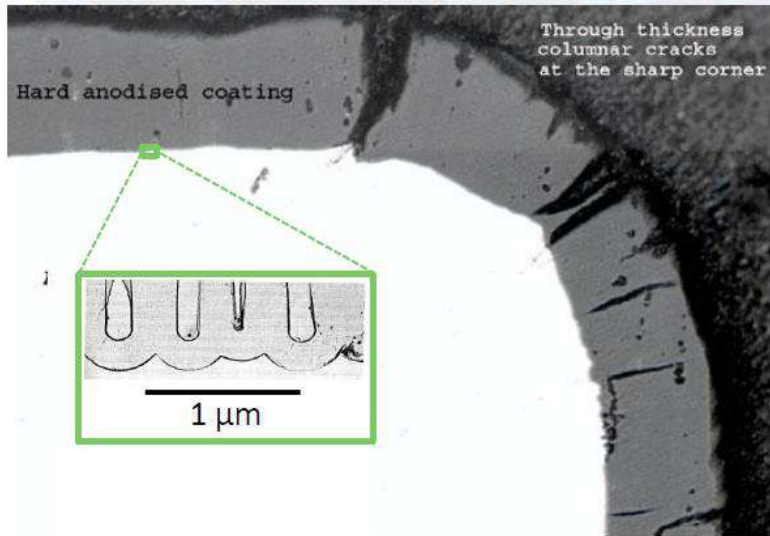
Corrosion and wear protection



PEO of Al in silicate solution



Corrosion and wear protection



KERONITE, UK

Chrome-free Mg corrosion and wear protection

Ti6Al4V wear protection

Aluminium wear protection

Thermal barrier protection, optical surfaces, high power dielectric insulation

Similar to anodising: Uniform coverage of complex shapes
Well-controlled, predictable growth
Non-columnar structure: Superior edge protection
Less susceptible to corrosion, wear
Lower fatigue debit

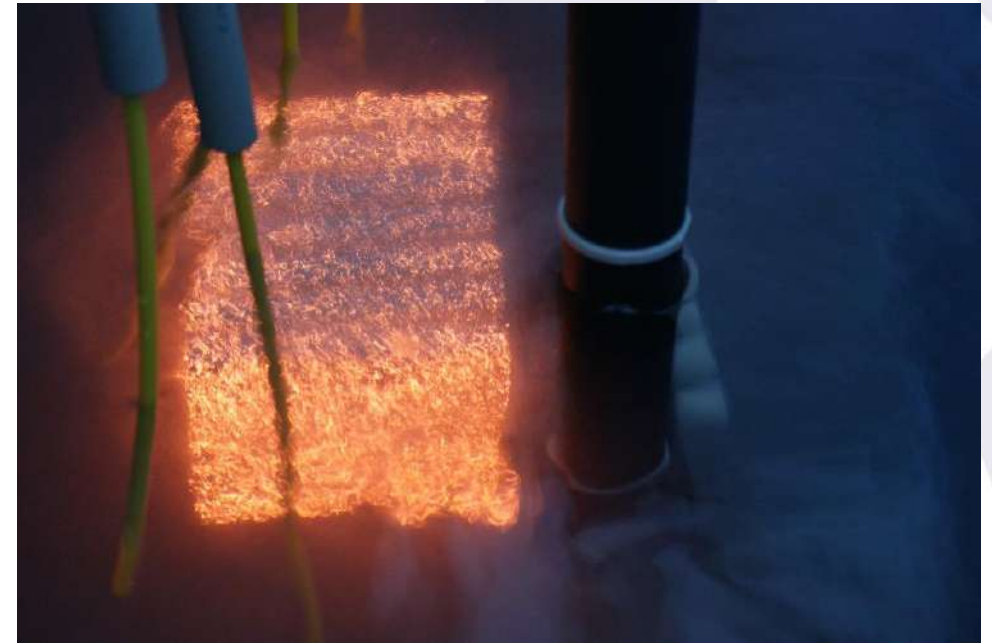


Corrosion and wear protection



Magnesium and aluminium alloys

in cooperation with **Pratt & Whitney, Rzeszów, Poland** / and Rzeszów University of Technology

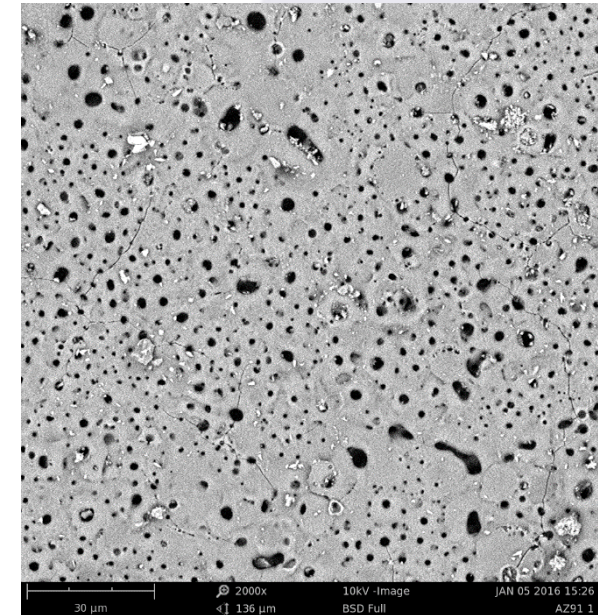


Corrosion and wear protection



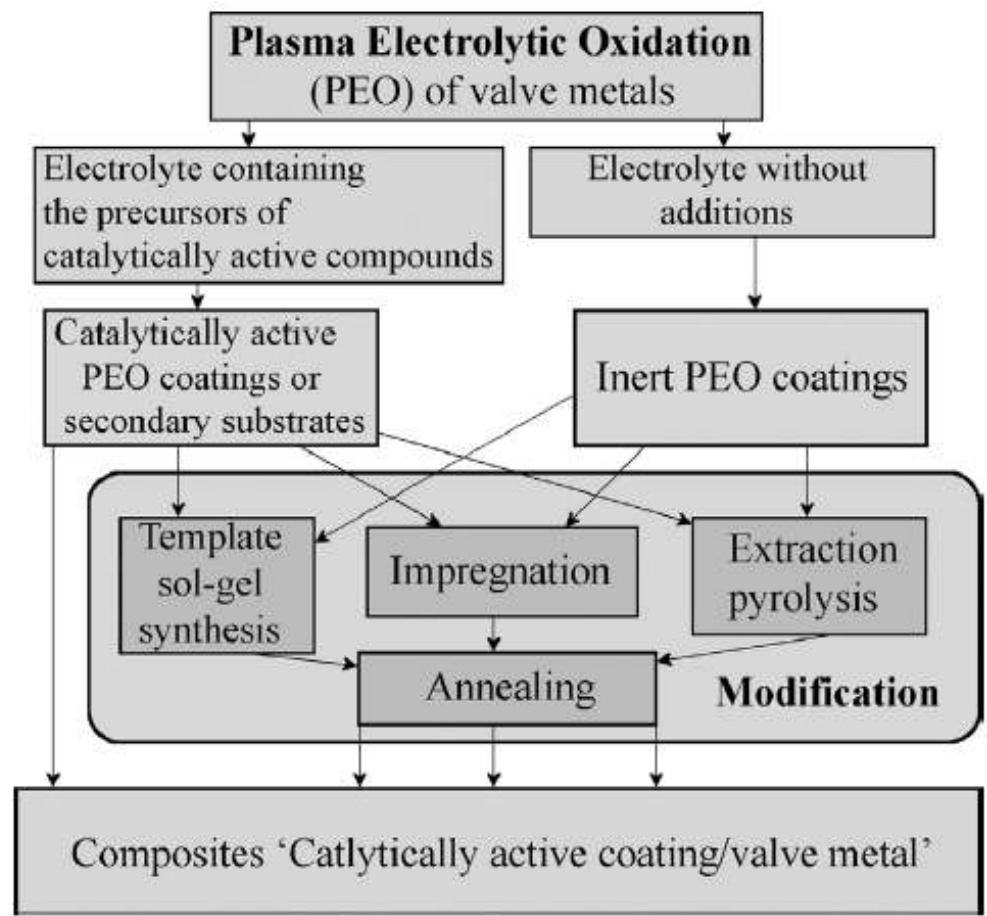
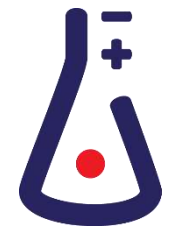
Magnesium and aluminium alloys

in cooperation with **Pratt & Whitney, Rzeszów, Poland** / and Rzeszów University of Technology



✓ **Improvement of corrosion properties**

Catalysis

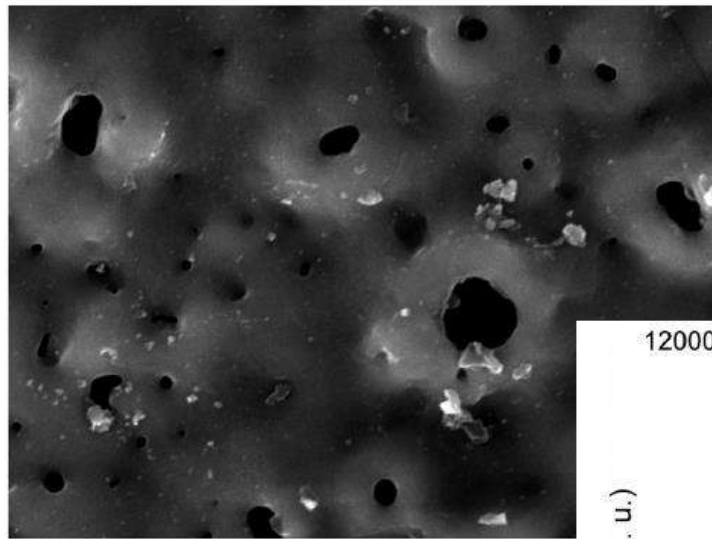


Catalysis

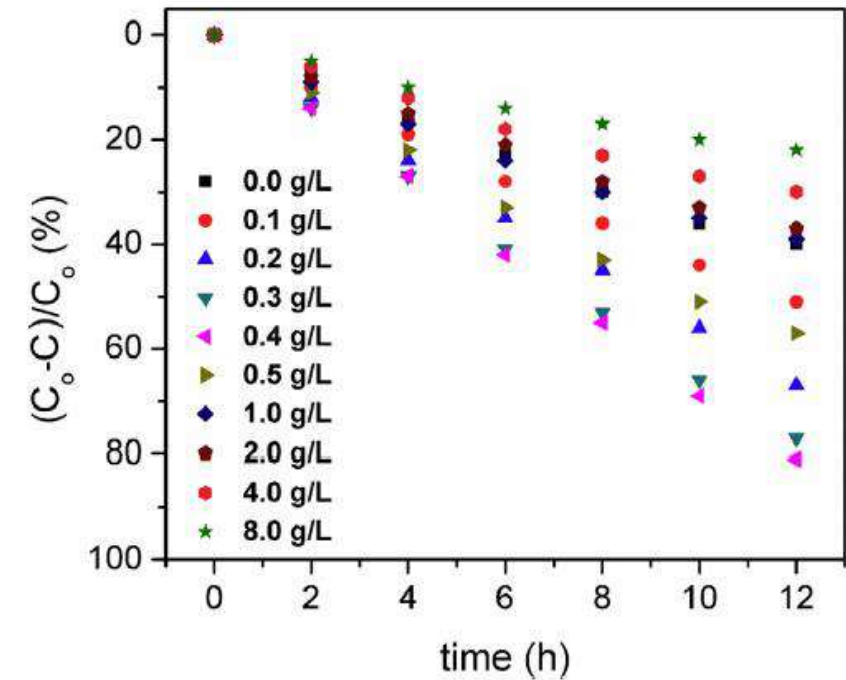
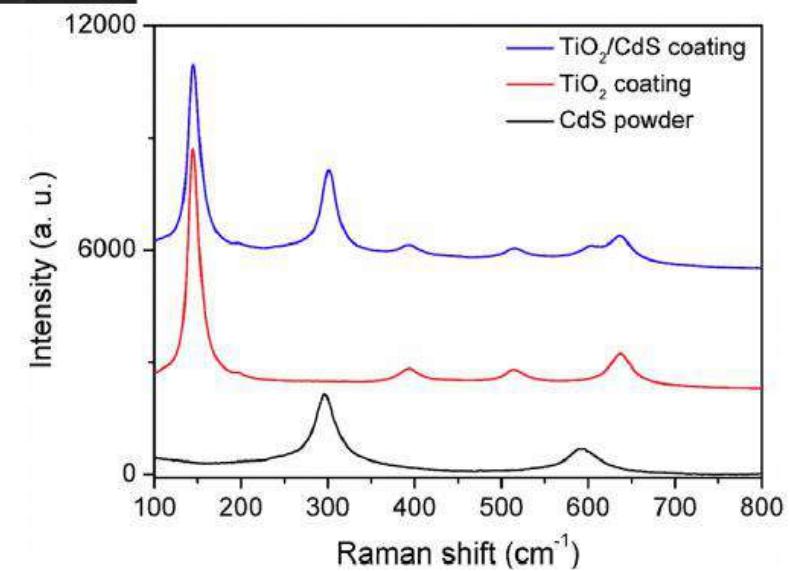


Ti/TiO₂-CdS photocatalyst

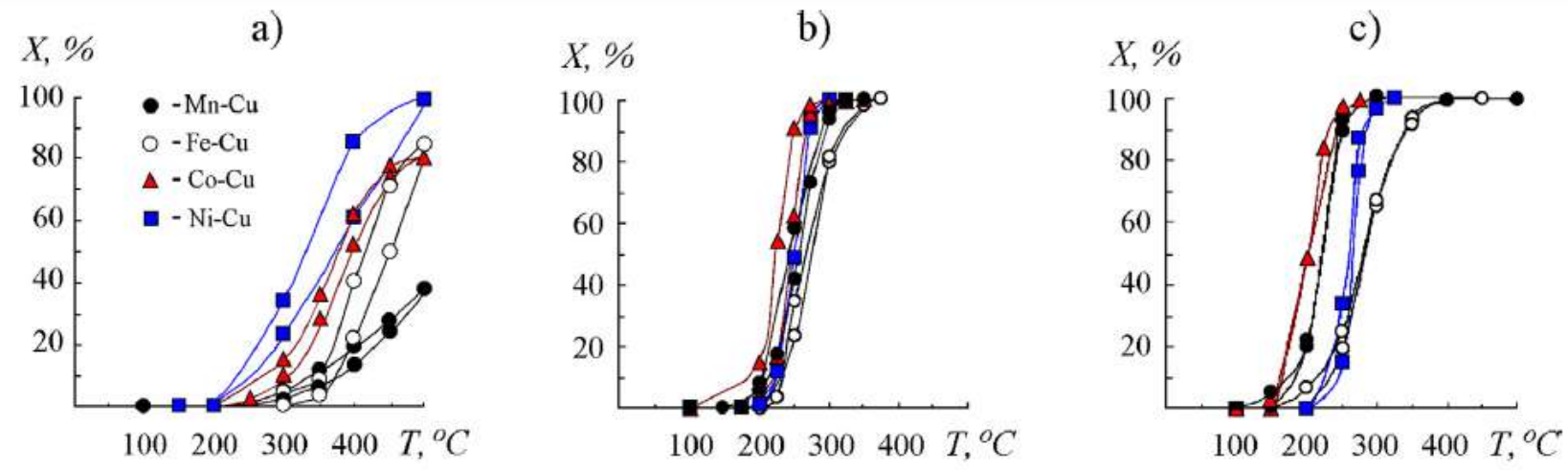
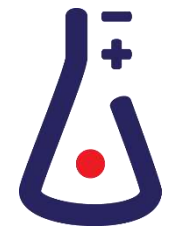
The photodegradation of aqueous methyl orange (MO)



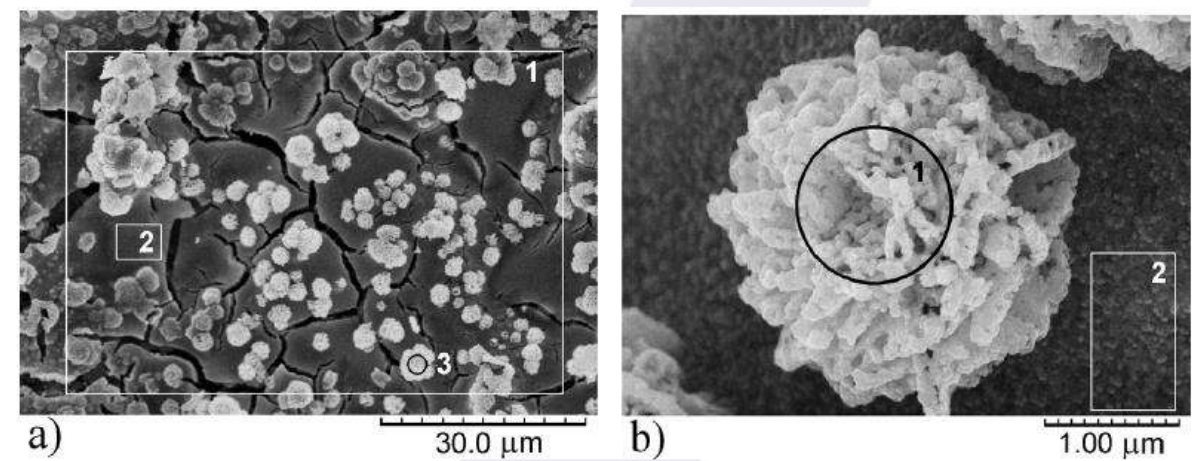
5 μm



Catalysis



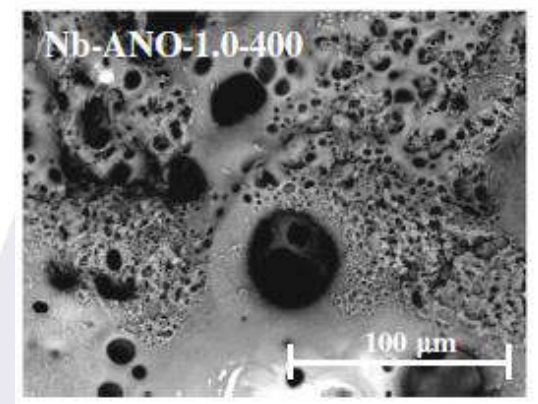
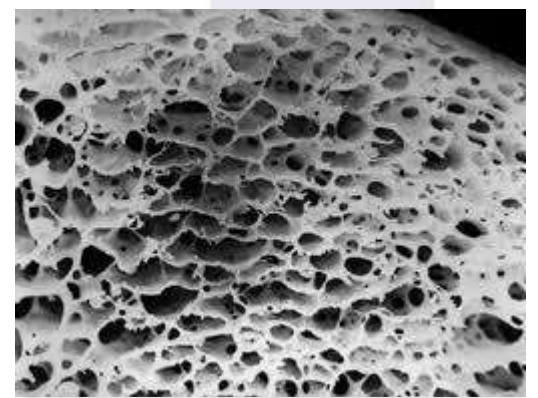
PEO catalyst for CO oxidation



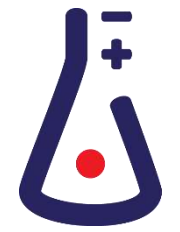
Biomaterials



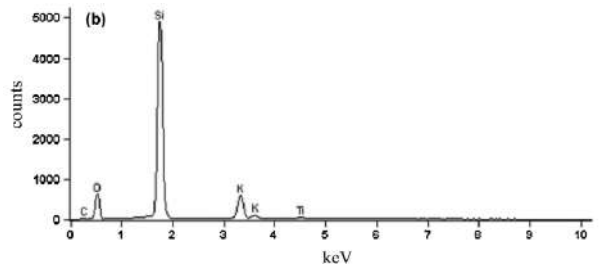
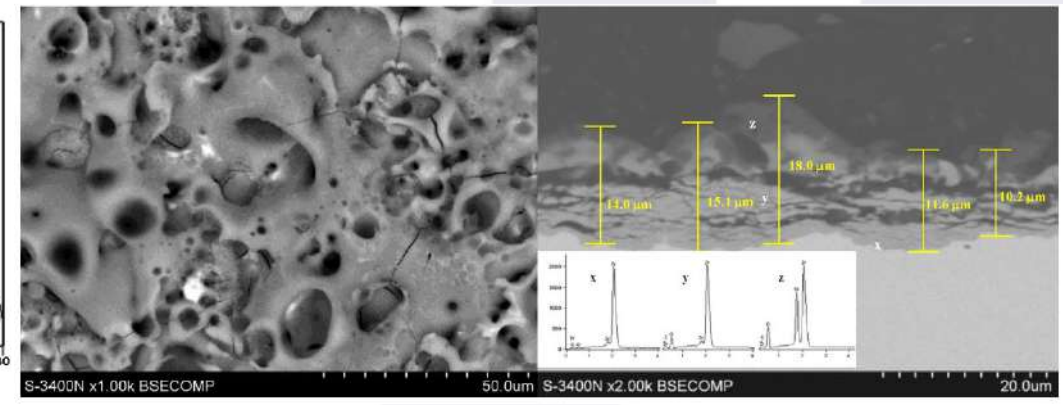
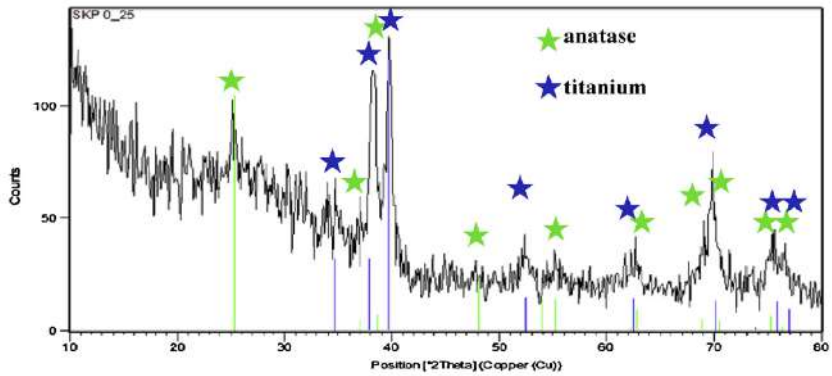
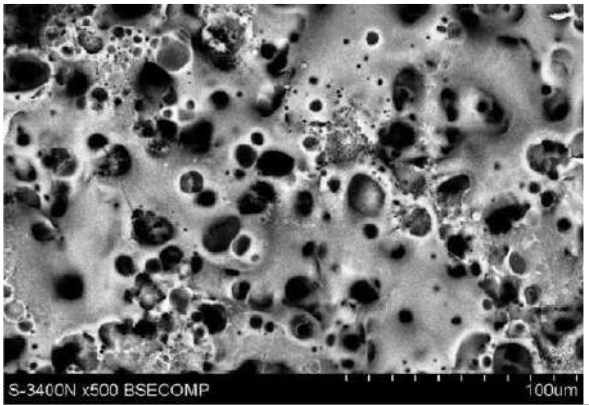
PEO ceramic coatings (in bone replacement)



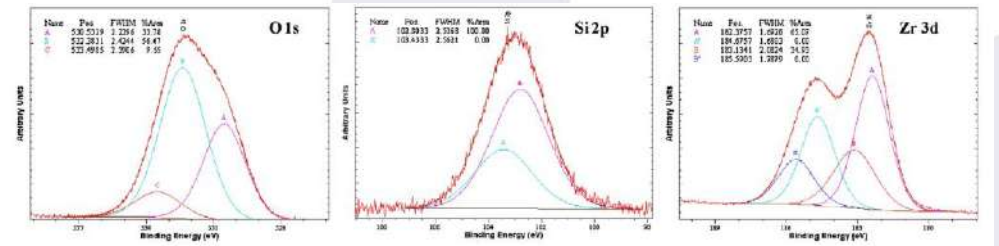
Biomaterials



Si addition



Ti-13Nb-13Zr alloy after PEO process in silicate solution; 400 V

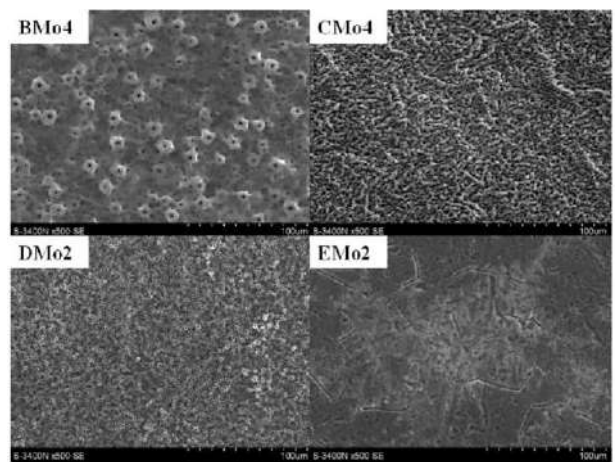


Zr after PEO process in silicate solution; 400 V

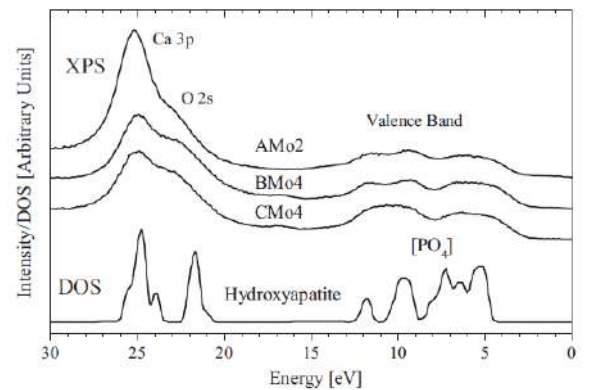
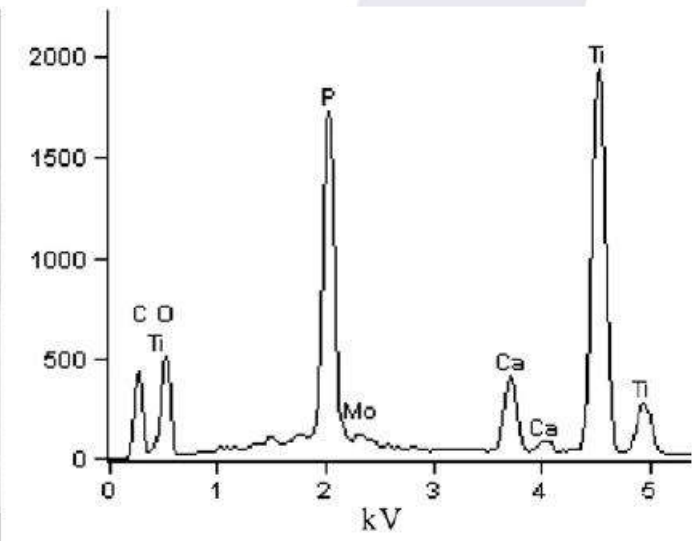
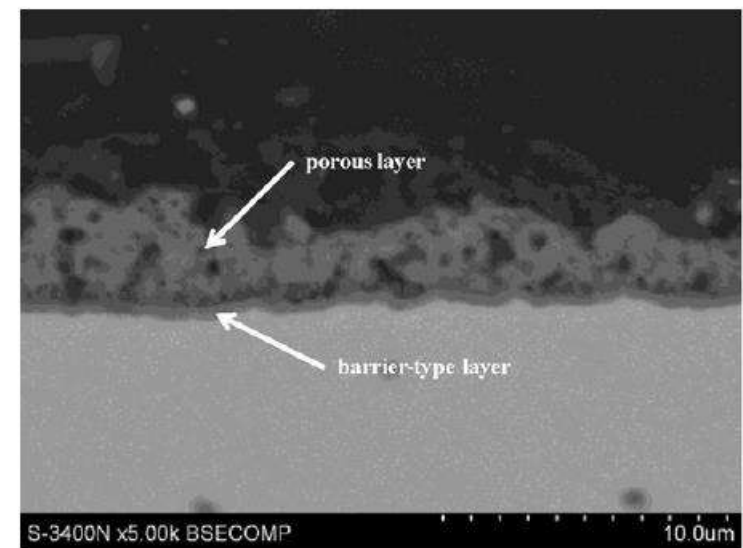
Biomaterials



Ca, P addition



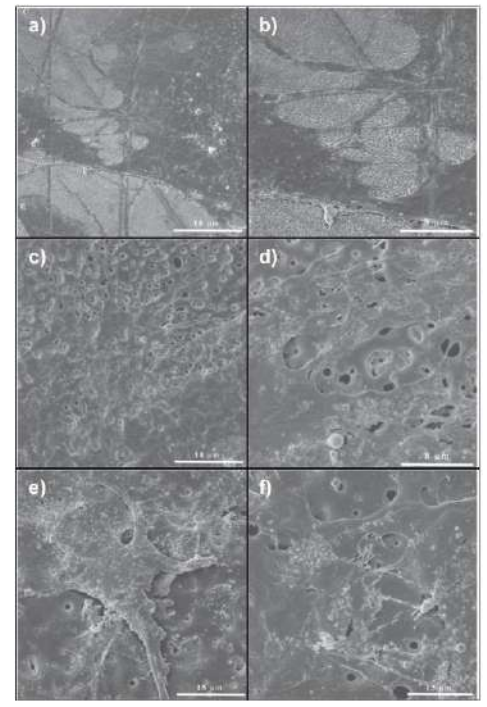
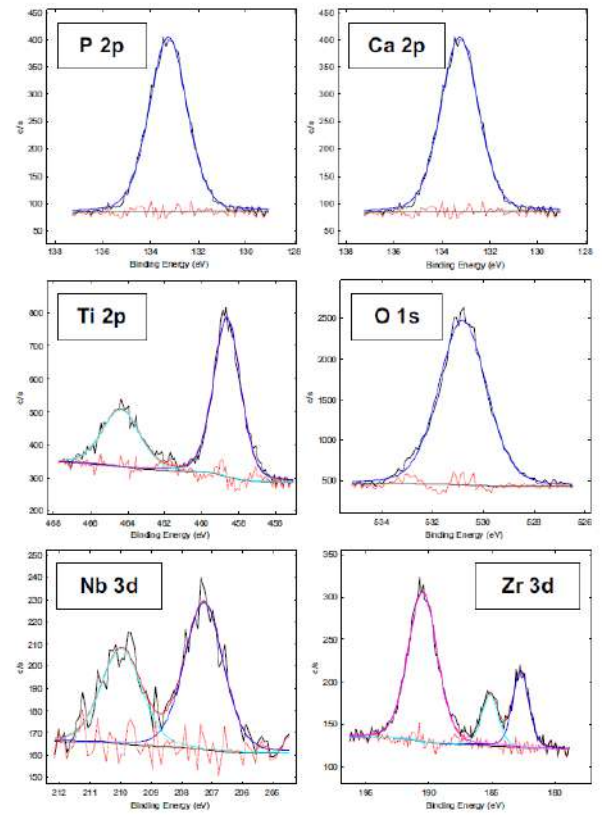
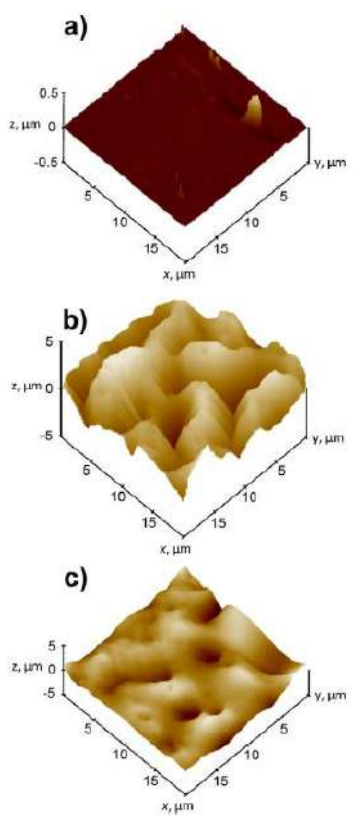
Ti-15 Mo alloy after PEO process in Ca-P solution; 200-400 V



Biomaterials



Ca, P addition

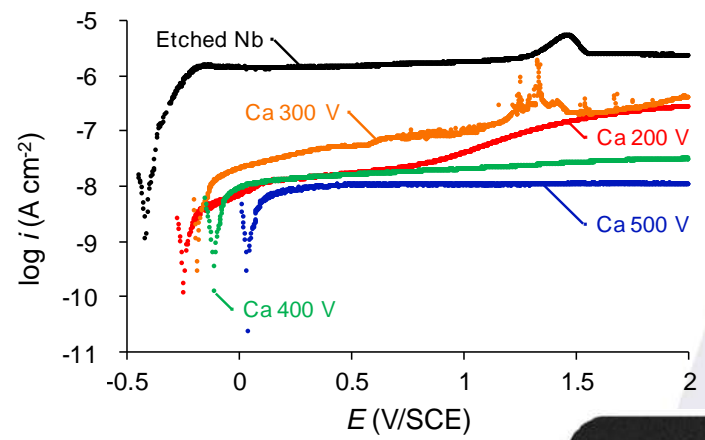
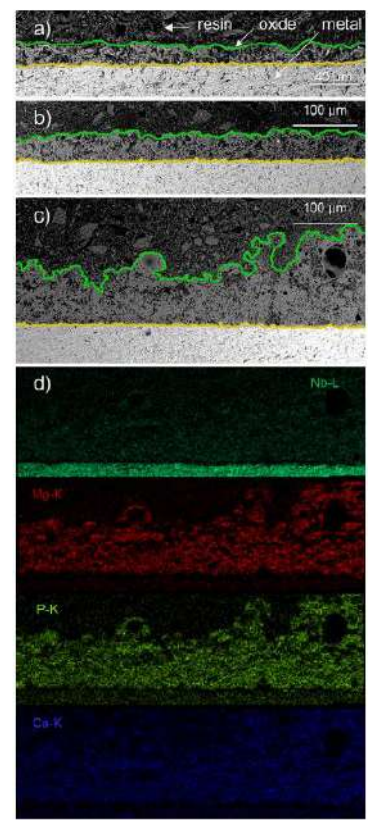
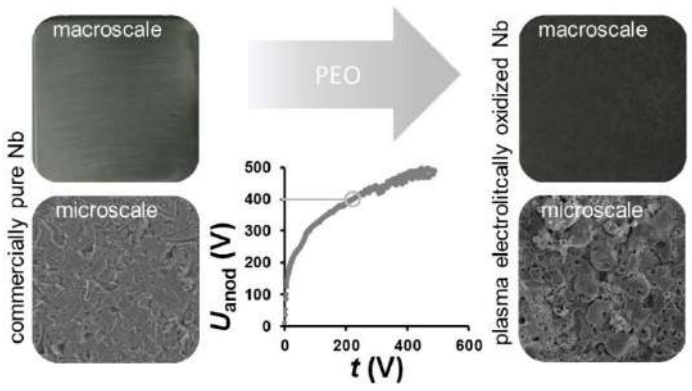


Ti-13Nb-13Zr alloy after PEO process in Ca-P solution



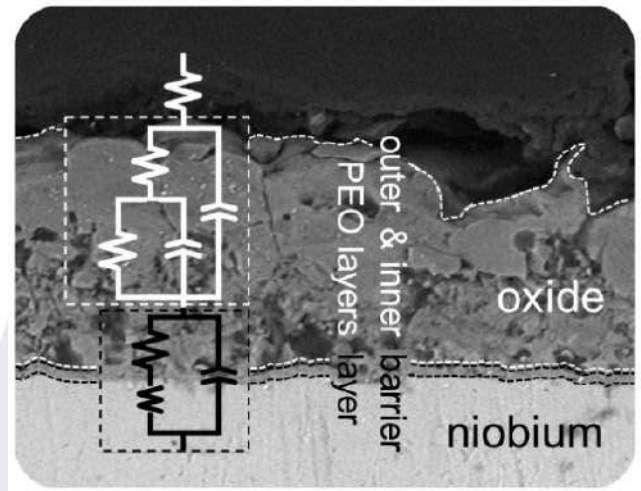
Plasma electrolytic oxidation (PEO)

Ca, Mg, P addition

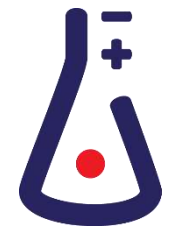


Polarization curves of pure Nb and Nb after PEO process in Ca-P solution,

Nb after PEO process in Ca-Mg-P solutions, cross-sections and EDX mapping



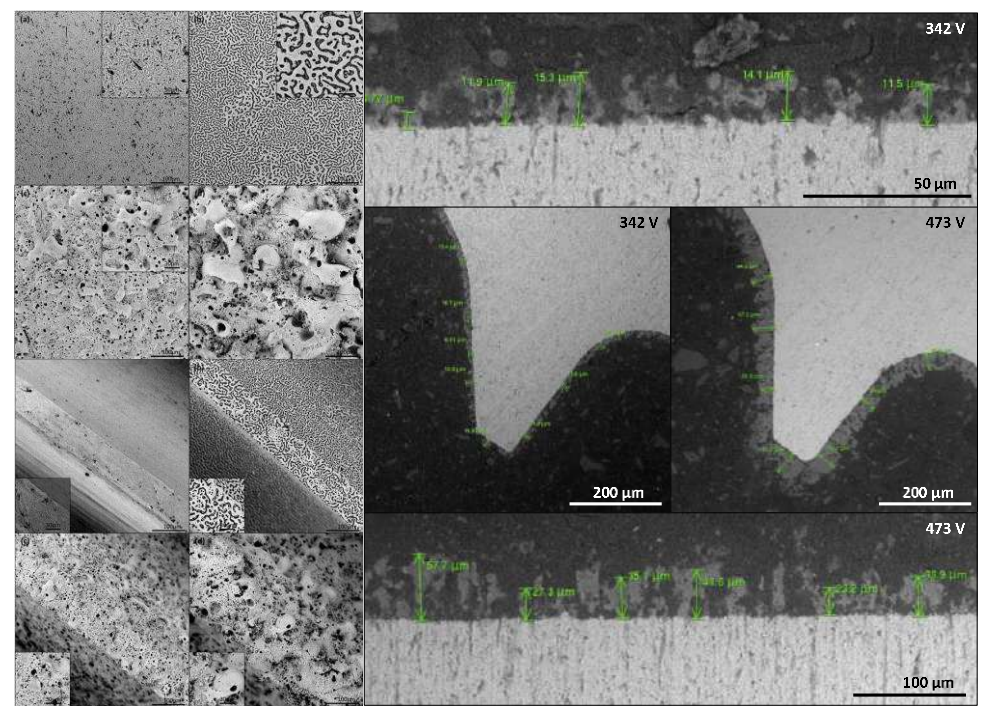
Biomaterials



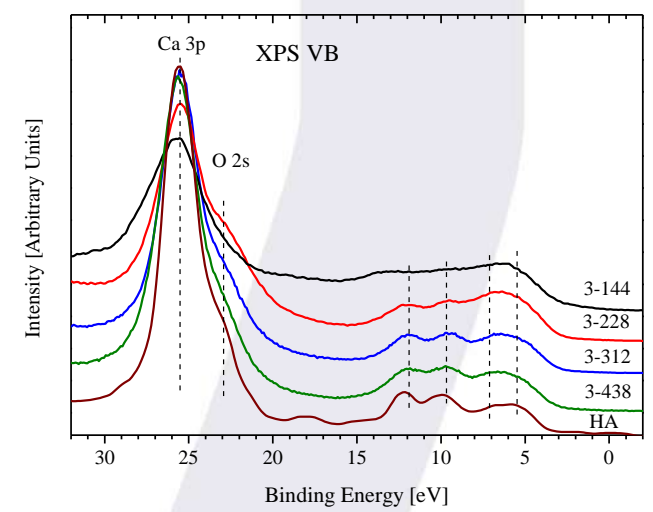
PEO of GumMetal dental implants



Macro view of GumMetal samples after PEO process



The SEM images of GumMetal samples after anodization at 128, 223, 342, and 473 V in Ca-P solution

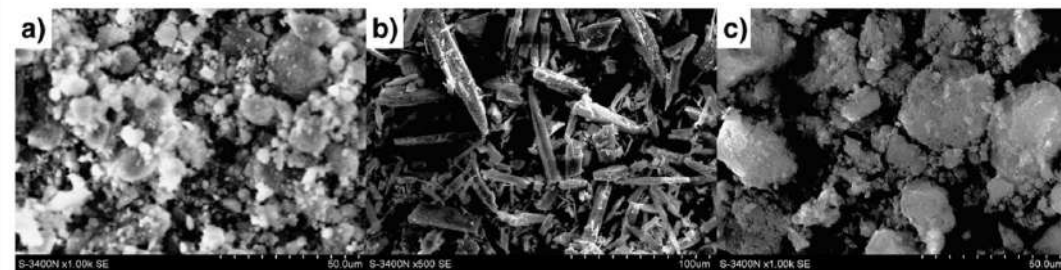


The XPS spectra of GumMetal samples at energy range of 0–30 eV

Biomaterials



bioactive particles addition



TCP

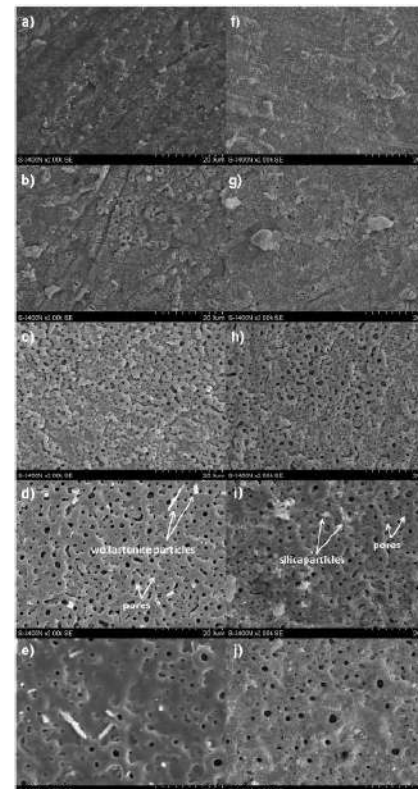
wollastonite

silica

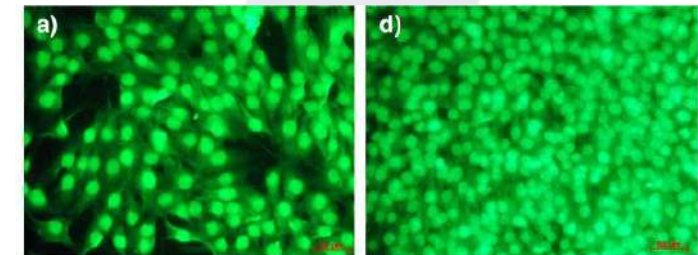
The roughness of samples and the layer thicknesses

Sample	Surface roughness [μm]				Thickness of oxide layer [μm]
	Ra	Rq	Rz	Rt	
Ti-15Mo	0.28	0.36	0.11	0.30	-
TM-200-TCP50	0.35	0.45	0.82	0.13	1.25-1.75
TM-250-TCP50	0.71	0.90	0.12	0.22	2.10-3.10
TM-300-TCP50	1.42	1.73	20.10	37.96	4.15-4.60
TM-350-TCP50	1.64	2.03	41.84	142.57	5.85-6.70
TM-300-W50	1.30	1.58	13.85	24.92	3.40-4.60
TM-300-W100	1.37	1.65	19.06	31.16	3.10-4.45
TM-300-W150	1.50	1.82	35.06	130.79	2.50-3.50
TM-300-S50	1.01	1.25	21.67	47.58	3.90-4.35
TM-300-S100	1.20	1.47	13.75	19.06	2.85-4.50
TM-300-S150	1.07	1.31	12.61	23.07	2.75-3.60

wollastonite silica

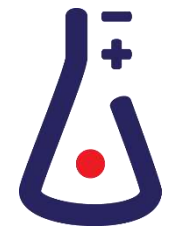


Ti-Mo alloy after PEO process in wollastonite and silica suspensions

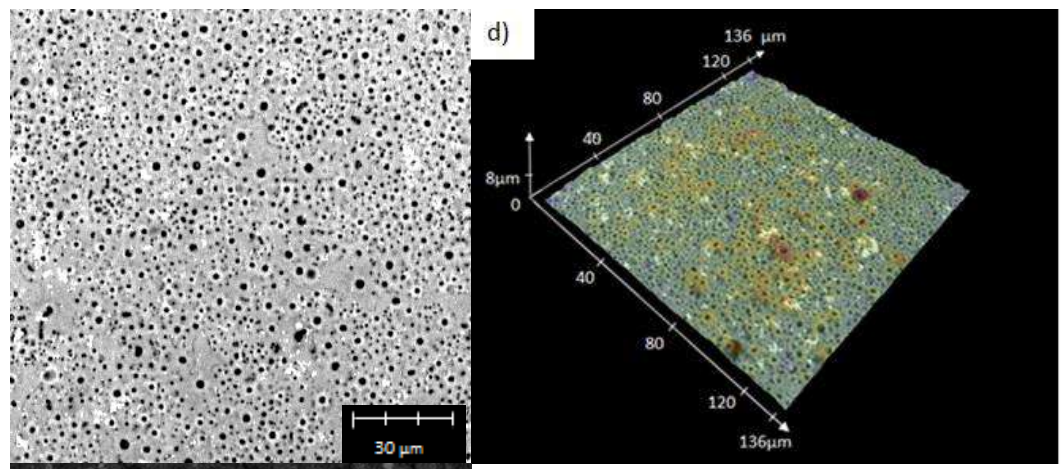


Fluorescence microscope images of MG-63 cells on 24 h (a) and 3 days (d) of culture on samples anodized in basic solution containing wollastonite

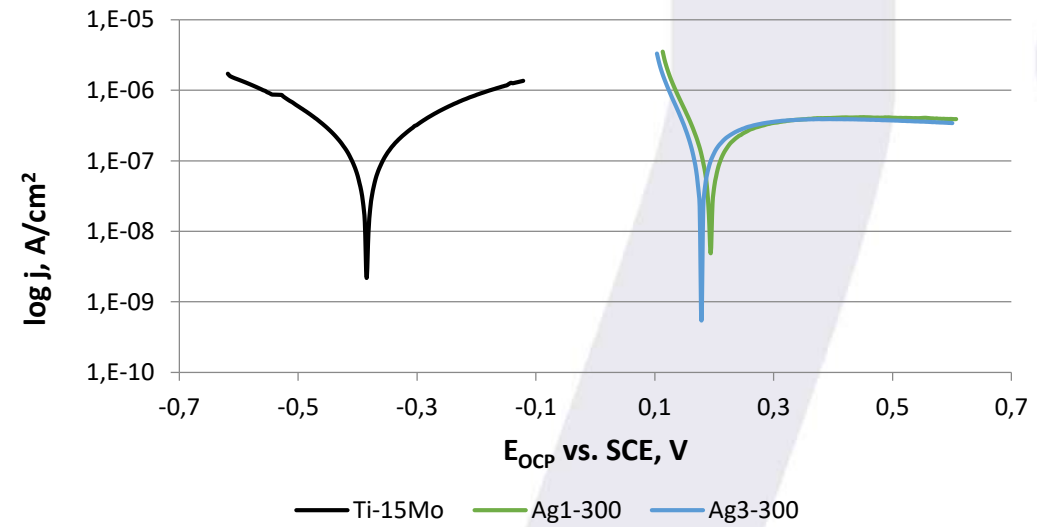
Biomaterials



Ag compounds addition



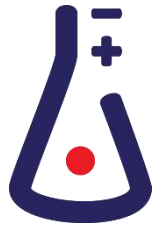
The SEM images of surface and cross section of anodized Ti-15Mo alloy as well as 3D reconstruction of its surface after PEO process



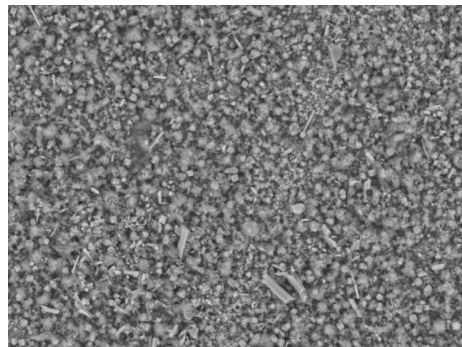
Polarization curves of Ti-15Mo alloy recorded in Ringers solution

Bacteriostatic investigations are in progress

Biomaterials

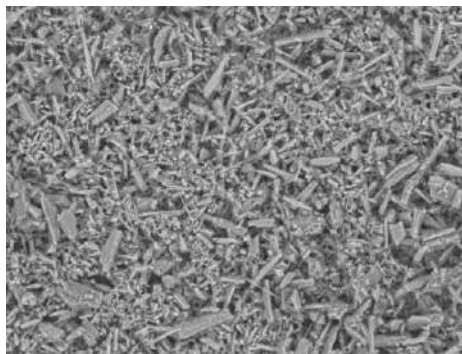


deposition of ceramic particles, e.g. wollastonite



2013/12/11 03:43 H D4.8 x1.0k 100 um

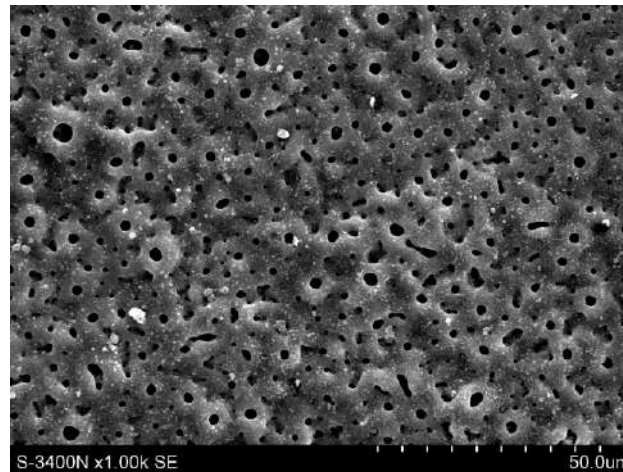
electrophoretic deposition



H D4.7 x1.0k 100 um

sol-gel process

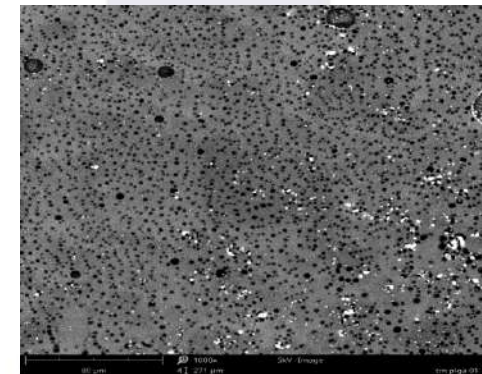
porous oxide layer



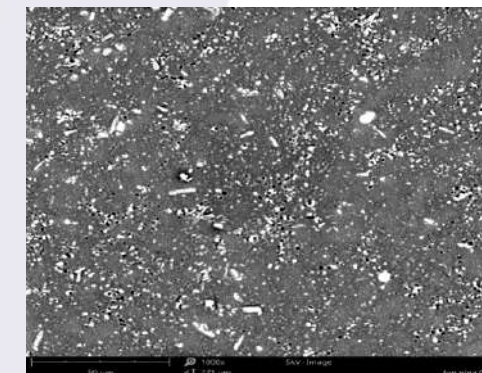
S-3400N x1.00k SE 50.0um

anodization of Ti alloys
in suspensions

deposition of biodegradable polymer



poly(D,L-lactide-co-glycolide) 50/50 (PLGA)



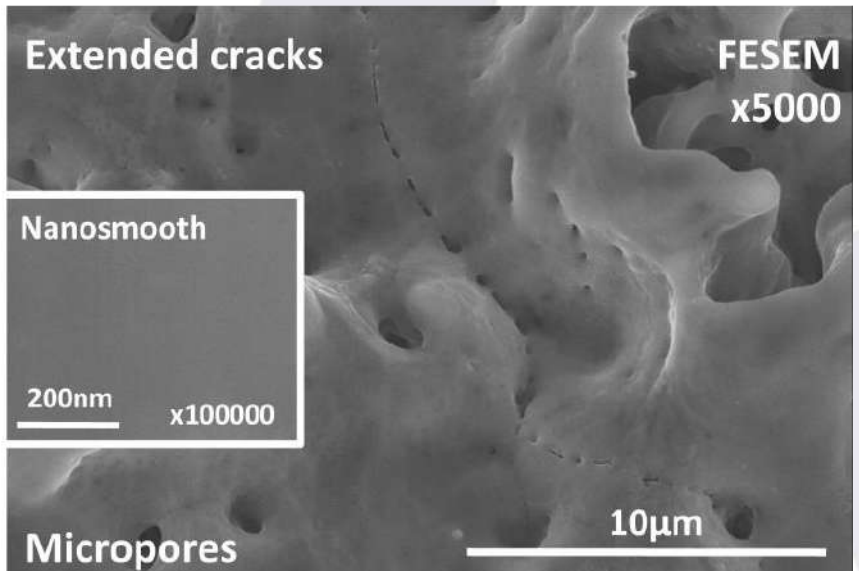
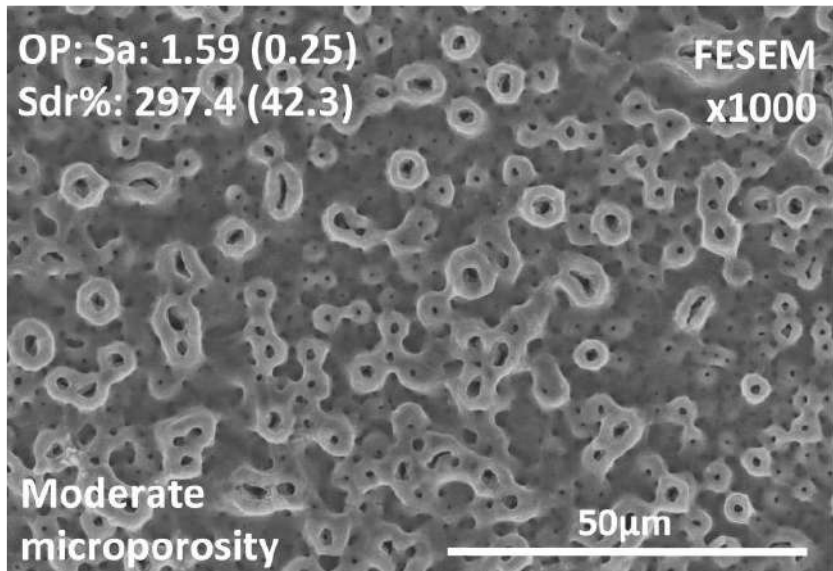
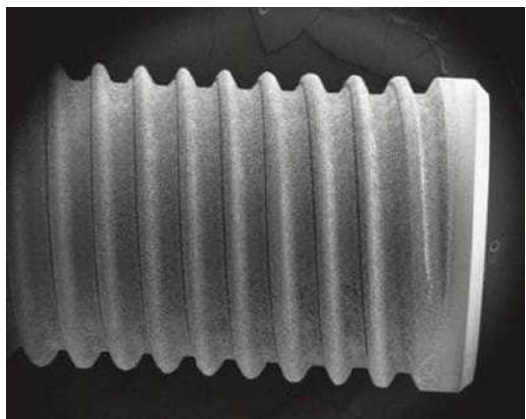
polycaprolactone (PCL)



TiUnite™ Surface by NobelBiocare, Sweden

TiUnite (Replace; Nobel Biocare, Gothenburg, Sweden)
 (Ref:32217; Batch:432892)
 XPS/ESCA Surface chemical composition (%)

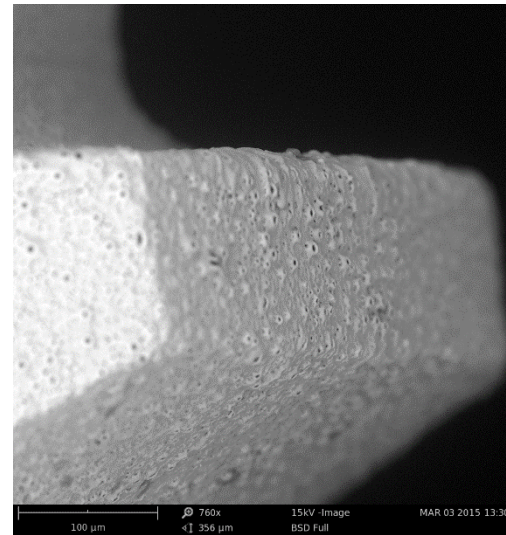
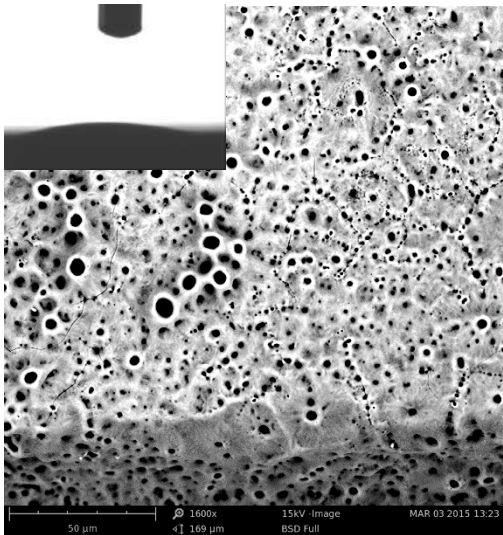
Ti	14.4 %	P	7.1 %
O	55.4 %	S	0.5 %
C	21.8 %	F	0.1 %
N	0.7 %		



Biomaterials

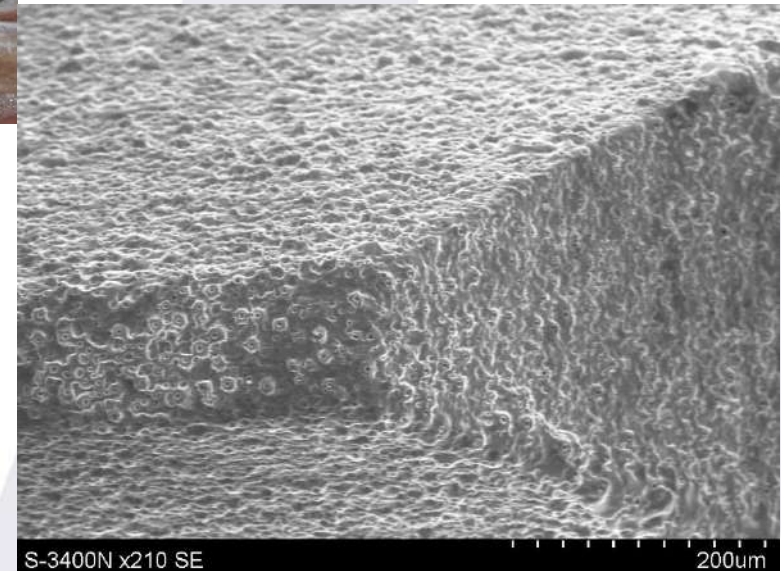


PEO of dental implants with Osteoplast R&D Company, Poland



Dental implants in pig mandible

The SEM images of Osteoplast R&D implant as well as a drop view on the implant surface



The surface SEM image of dental implant after test in pig mandible

Plasma electrolytic oxidation (PEO)



Main benefits:

- functionalization of biomaterials
- anticorrosive protection
- uniform and well adherent layers
- formation of (photo)catalysts
- anodization of complicated shapes

Disadvantages:

I don't have any idea 😊



Thank you for attention !!!

www.electrochemistry.polsl.pl

www.osteoplant.info



Narodowe Centrum
Badań i Rozwoju

Project no. **POIR.01.01.02-00-0022/16**, *Development of technology and launching the production of innovative dental implants with increased osteoinductive properties*