UCM Universidad Complutense de Madrid

Electrochemical response in biological media of Plasma Electrolytic Oxidation treated Additively Manufactured Ti6Al4V alloy

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Characterization, Corrosion and Degradation of Advanced Materials

CCRM group

1st Corrosion and Materials Degradation Web Conference

17-19 May 2021

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OUTLINE

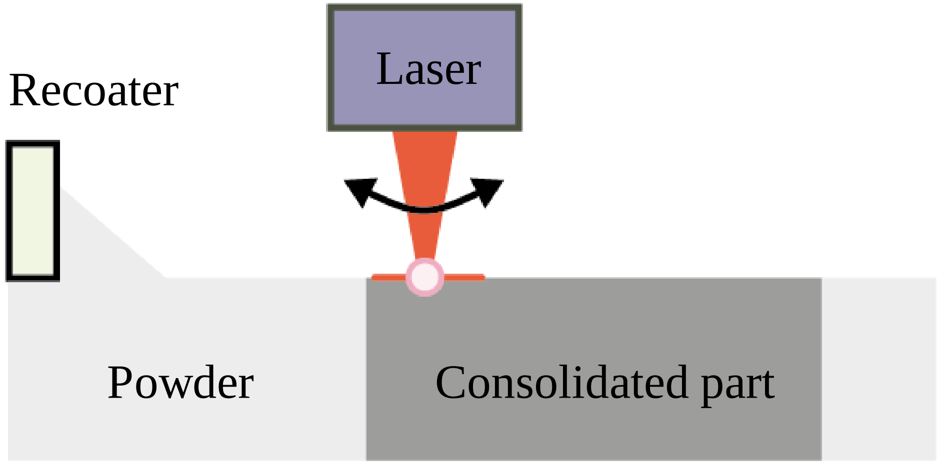
1. Introduction and objectives
2. Materials and experimental procedure
3. Results
4. Conclusions

Additive Manufacturing

- Cost efficient on-site 3D manufacturing
  - Dense parts
  - Mesh scaffolds
  - Patient-customized articles
- Biomedical-grade metallic materials: **Ti alloys**

Laser Powder Bed Fusion

- i.e. Direct Metal Laser Sintering – DMLS
- Layer-by-layer manufacturing
- Metallic powder melting
- High power laser as heating source



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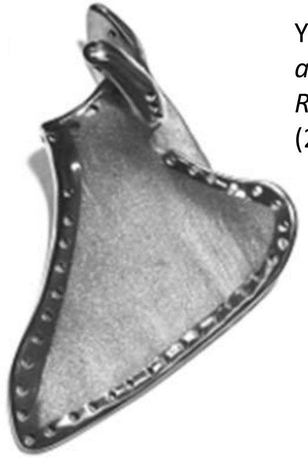

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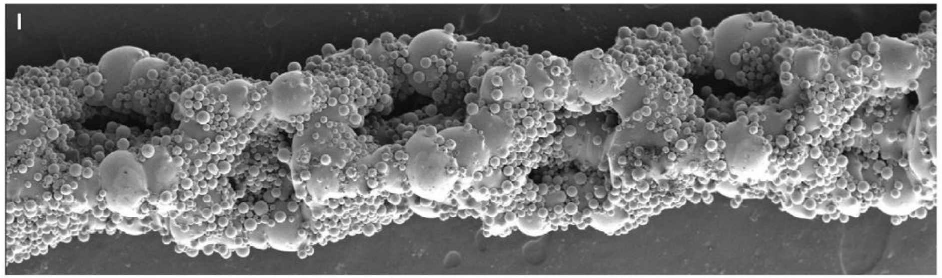

I.A.J. van Hengel *et al. Acta Biomaterialia* (2020)

M Ahmadi *et al* 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **293** 012009

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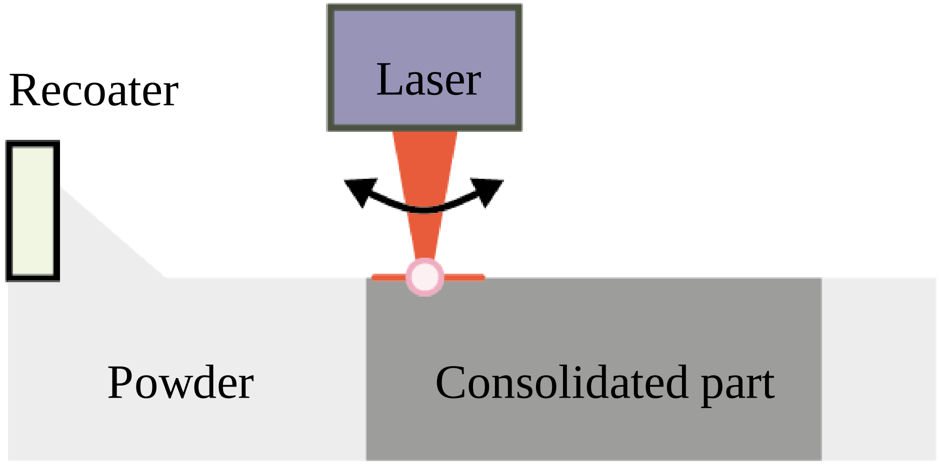

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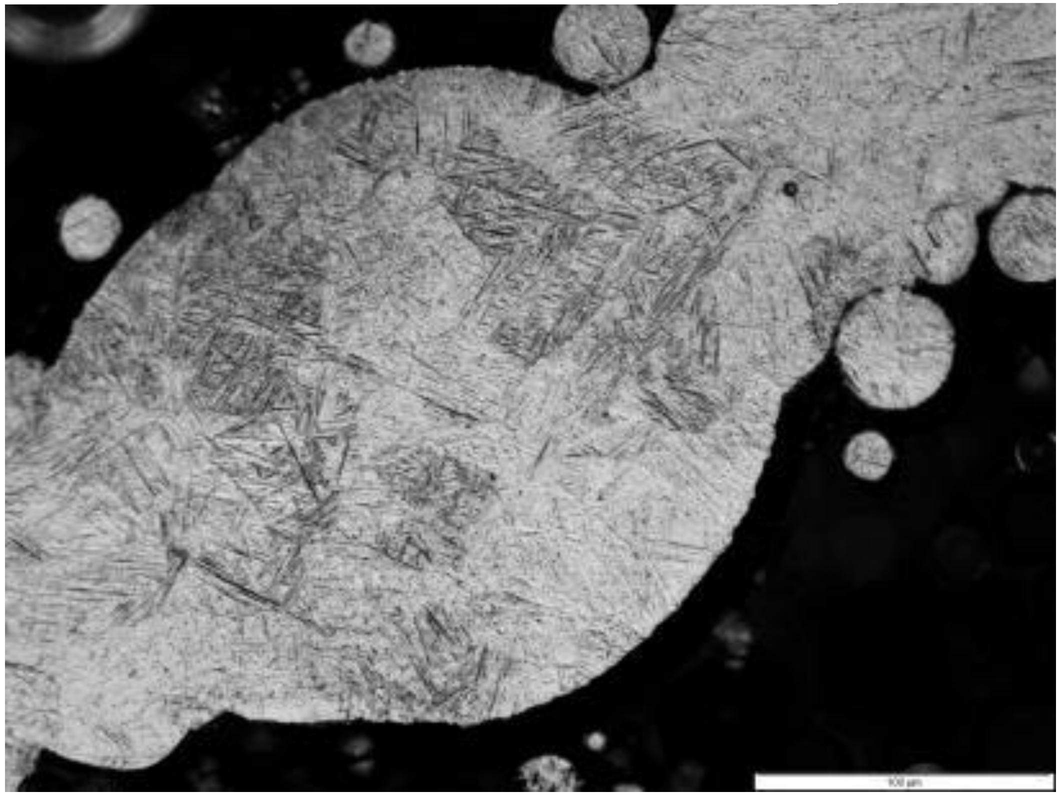
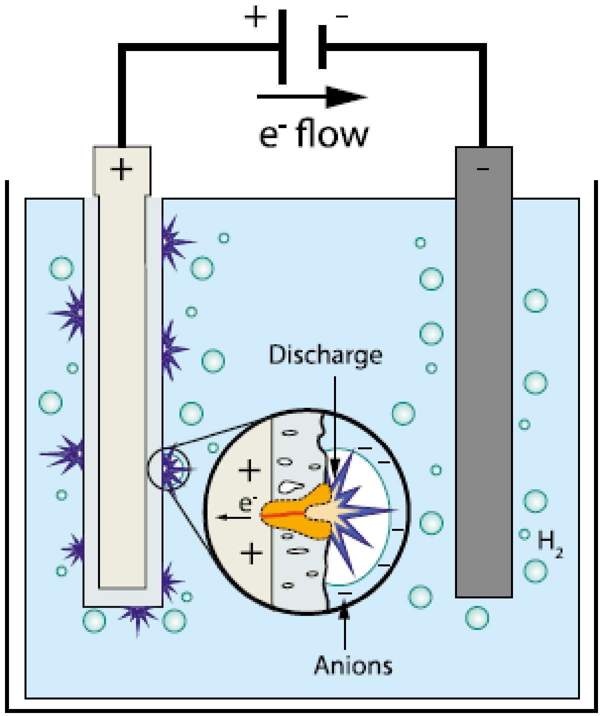


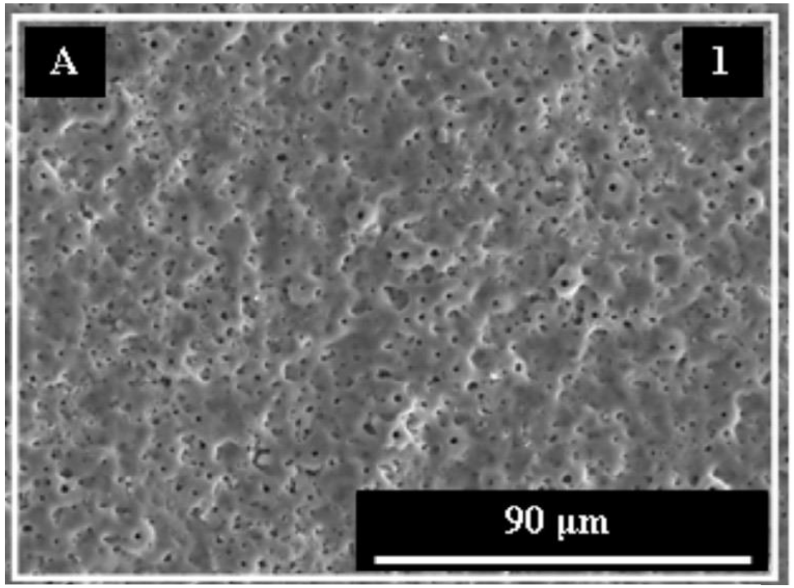
As-built microstructure
acicular α’

Different mechanical and electrochemical behaviour to traditionally manufactured alloy

Surface treatment – Plasma Electrolytic Oxidation - PEO

- Electrochemical high voltage surface modification technique
- Rough and porous ceramic coatings
- Dielectric breakdown → Micro discharges
- Enhanced corrosion protection, adhesion and wear resistance
- Enhanced adhesion and wear resistance
- **Osteoconductive coatings: Ca- and P-containing**





A. Santos-Coquillat et. al., *Applied Surface Science*, 2020
https://doi.org/10.1016/j.surfcoat.2020.126317

Objectives of the study

- Fabrication of Ca- and P-containing thin (~ 10 μm thickness) PEO coatings on a Ti6Al4V alloy manufactured via Direct Metal Laser Sintering (DMLS)
- Study the electrochemical behaviour of bare and PEO treated AM alloy in a modified α-MEM solution

Materials

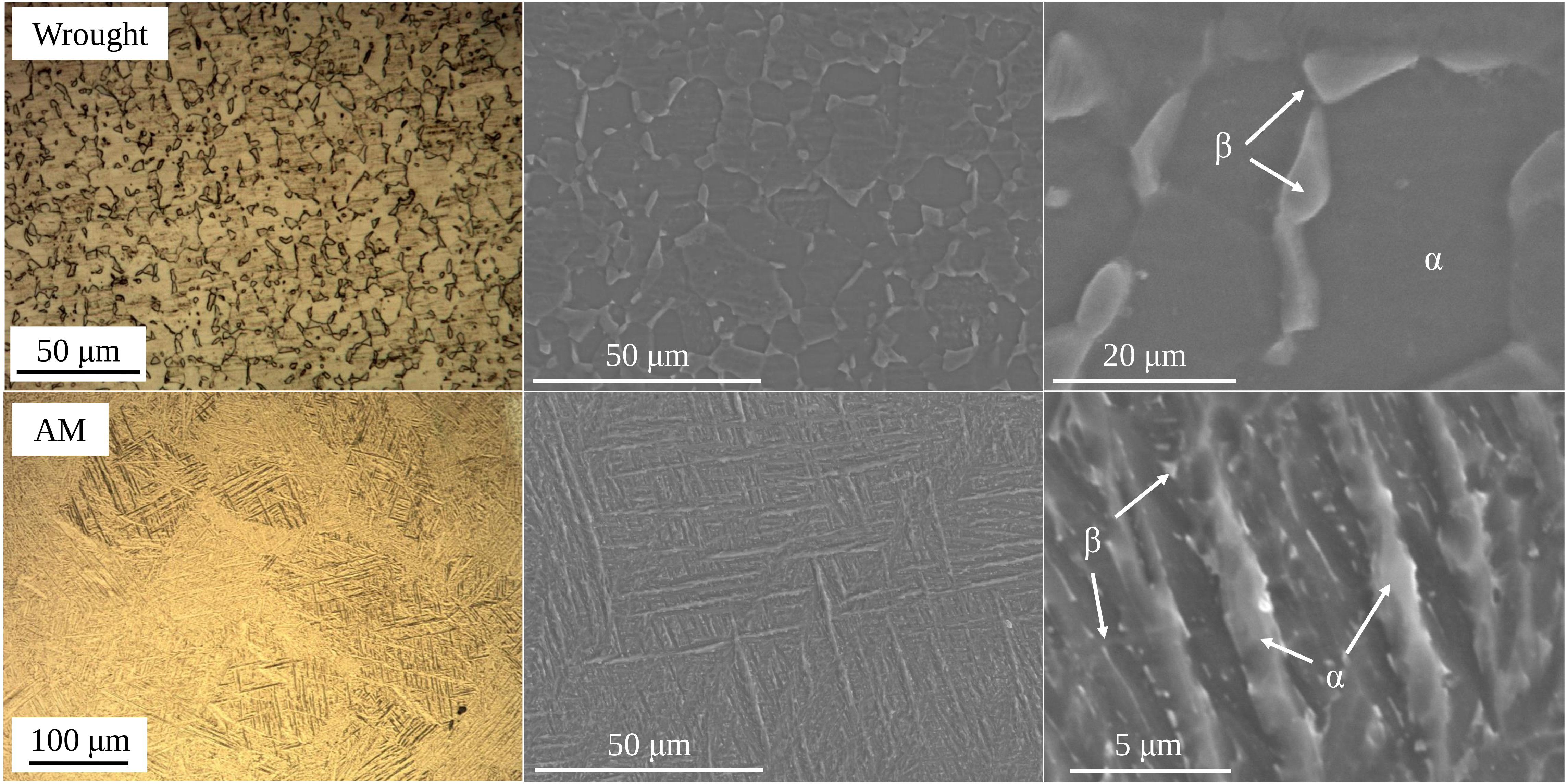
- Wrought mill-annealed Ti6Al4V - **Wrought**
- DMLS Ti6Al4V - **AM**
  - Stress relief heat treatment: 650 ºC – 3 h
  - + Thermal treatment ($\alpha' \rightarrow \alpha+\beta$): 750 ºC – 3.5 h, furnace cooling
  - Orientation studied: **XY**

Potentiodynamic polarisation

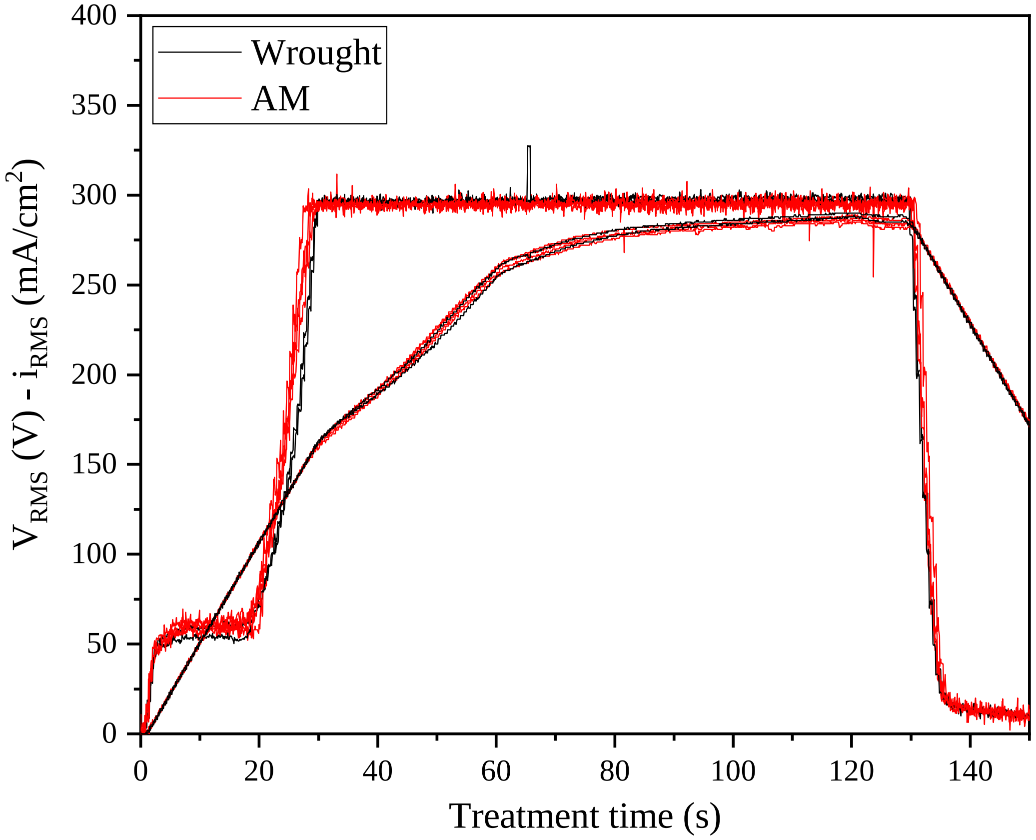
- Modified α-MEM:
  - NaCl – 6.8 g/L
  - $CaCl_2$ – 0.2 g/L
  - $MgSO_4$ – 0.098 g/L
  - KCl – 0.4 g/L
  - $NaHCO_3$ – 2.2 g/L
  - Na_2HPO_4 – 0.122 g/L
  - pH: 7.4-7.6

Plasma Electrolytic Oxidation

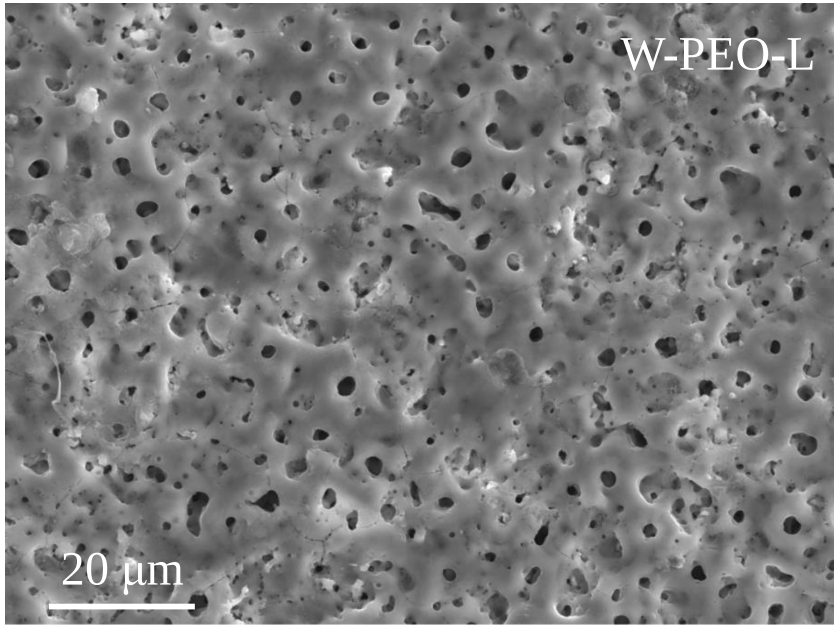
- Electrolyte:
  - $C_6H_{10}CaO \cdot 5H_2O$ – 0.05 M
  - $NaH_2PO_4 \cdot 2H_2O$ – 0.055 M
  - NaOH – 0.025 M
  - $Na_2(EDTA) \cdot 2H_2O$ – 0.15 M
- Voltage signal:
  - +490V/-30V – V_{RMS} = 347 V
  - 300 Hz
  - Maximum current density: 0.3 A/cm^2
- Treatment time:
  - 120 s
  - Onset of sparking: Visible sparking + 5 s


Wrought
50 μm
50 μm
β
α
20 μm
AM
100 μm
50 μm
β
α
5 μm

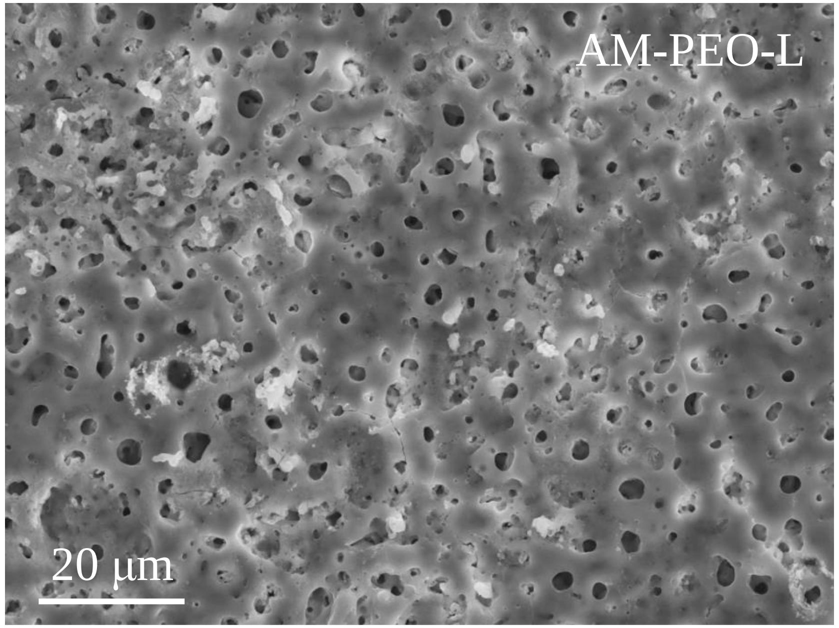
3. Results: 120 s PEO coatings



Thickness = 11.20 ± 0.64 µm
Sa = 0.68 ± 0.09 µm

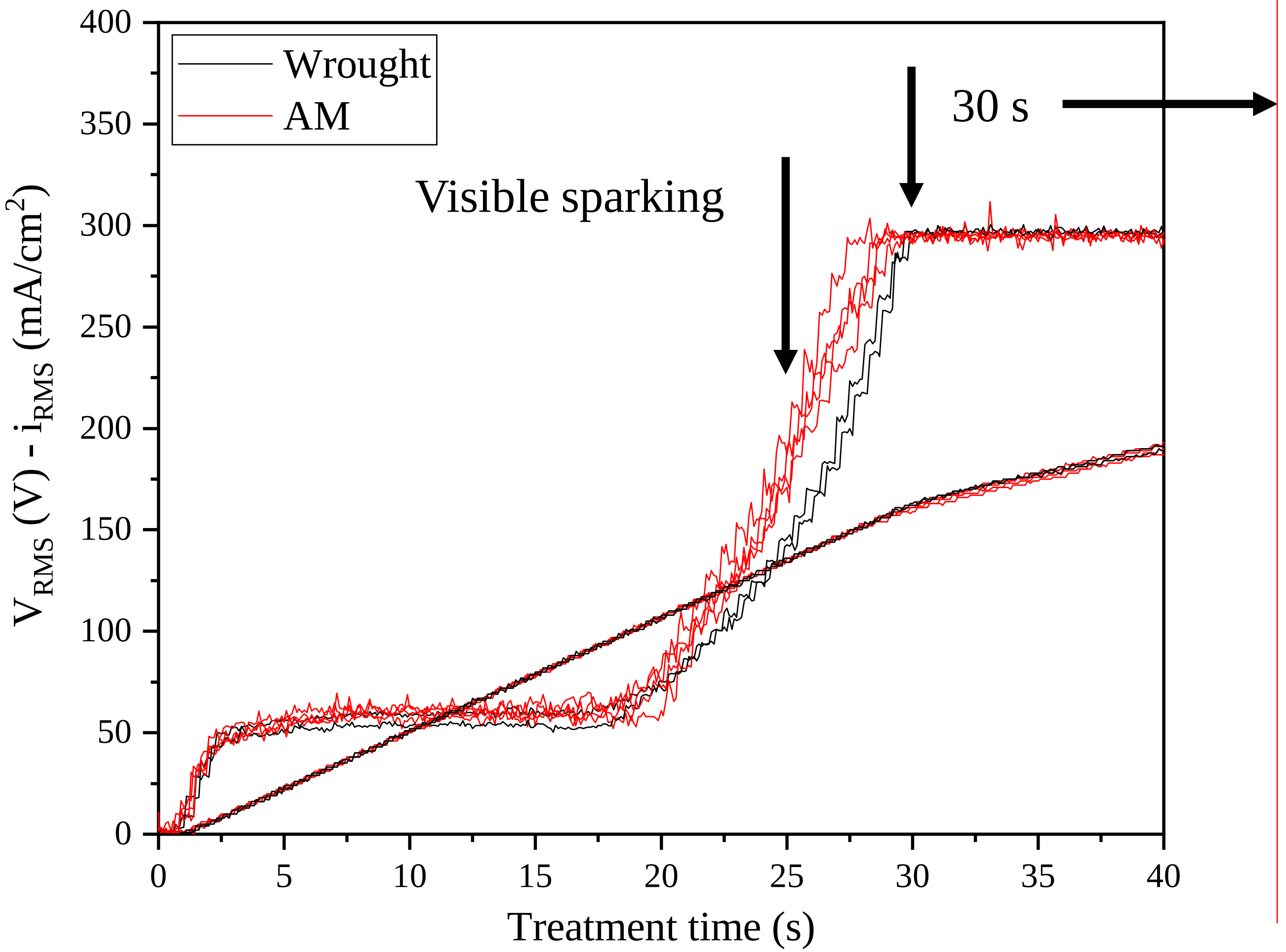


Thickness = 11.82 ± 0.85 µm
Sa = 0.68 ± 0.09 µm

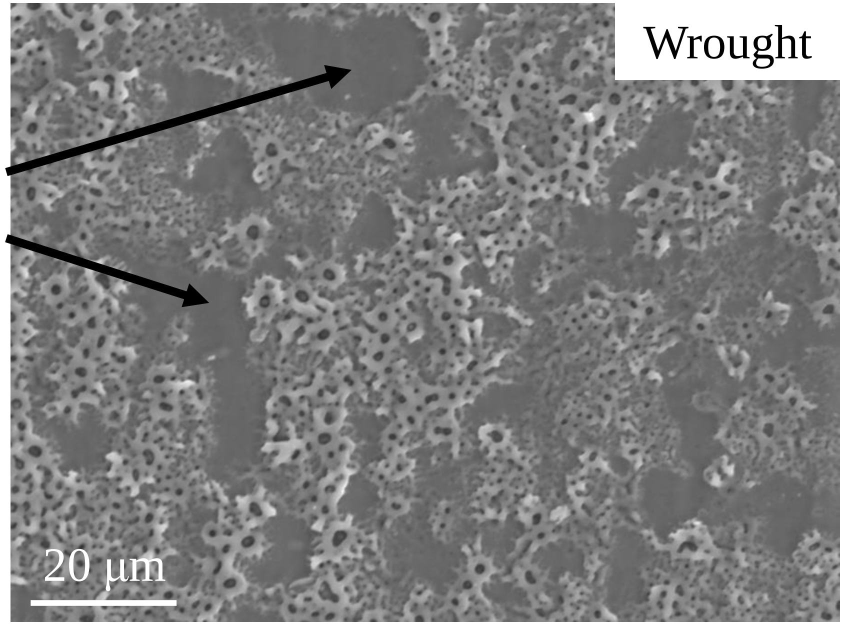


Sample	O (at. %)	Na	Al	P	Ca	Ti	V	Ca/P
W-PEO	68.85	0.70	1.21	7.07	8.33	13.4	0.44	1.18
AM-PEO	68.47	0.87	1.16	7.17	8.80	13.17	0.37	1.23

3. Results: Onset of sparking

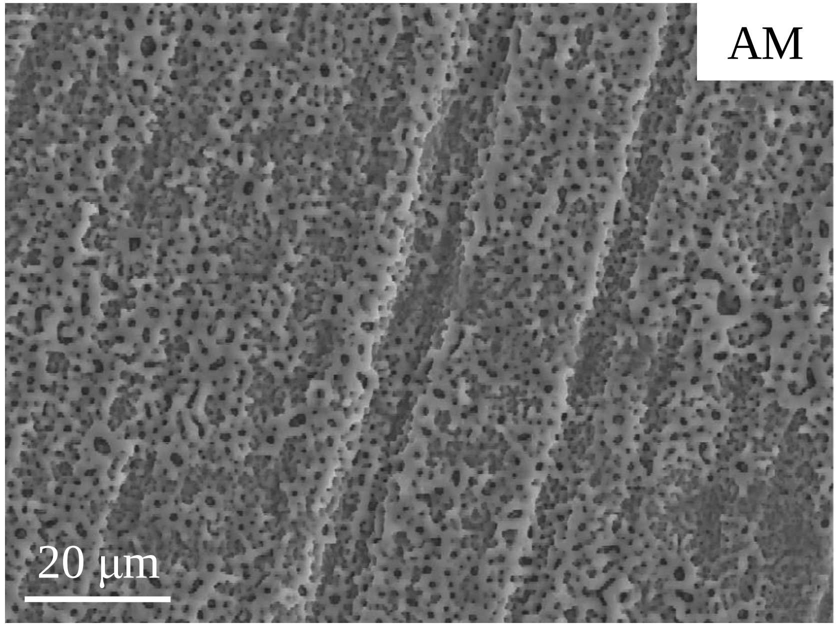


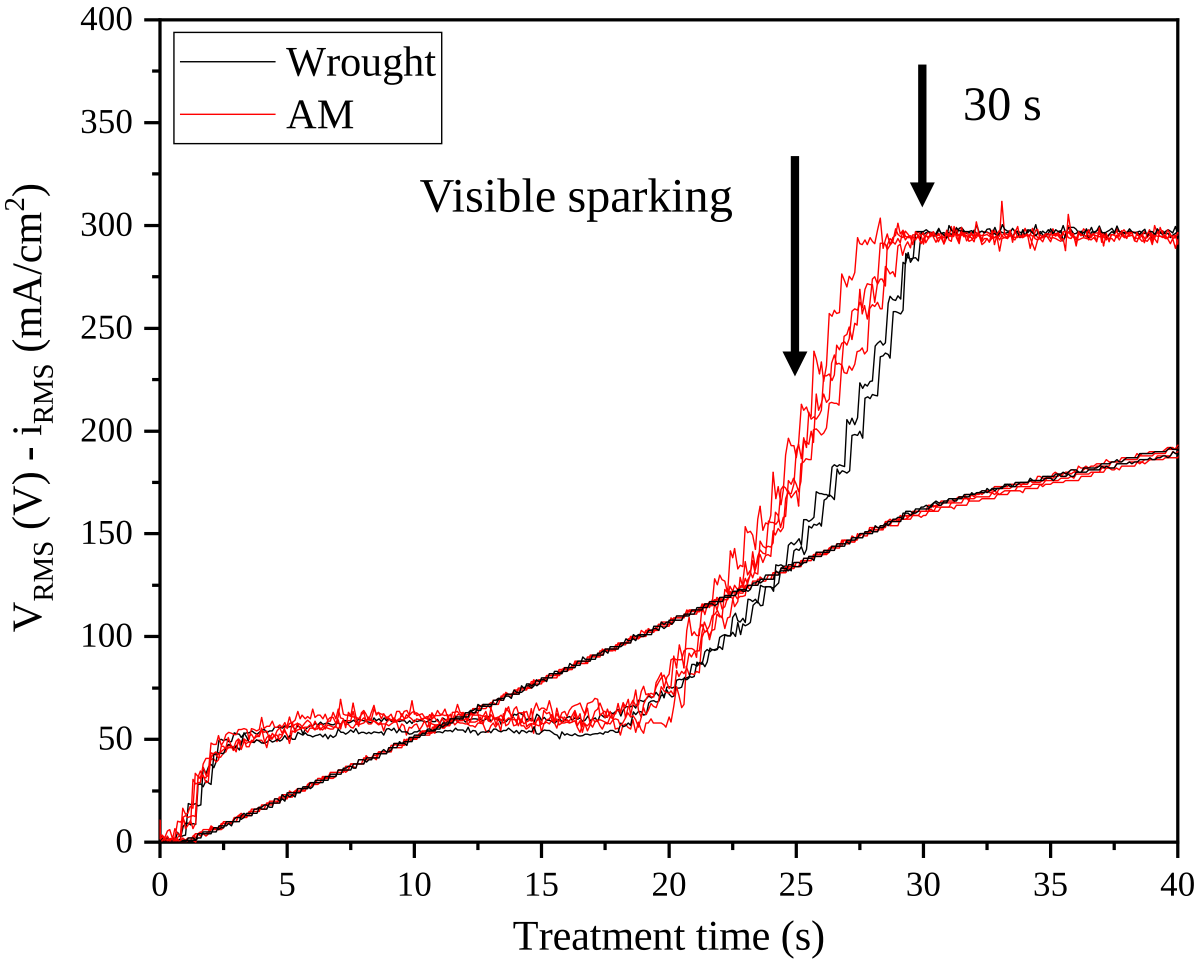
Incomplete
PEO growth



Homogeneous
PEO growth

Thickness
1.35 ± 0.4 μm





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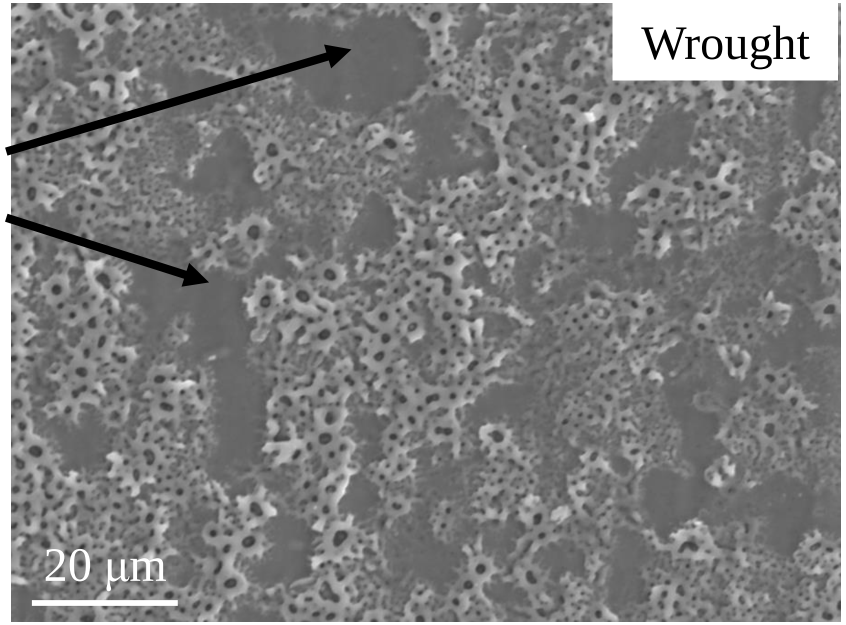
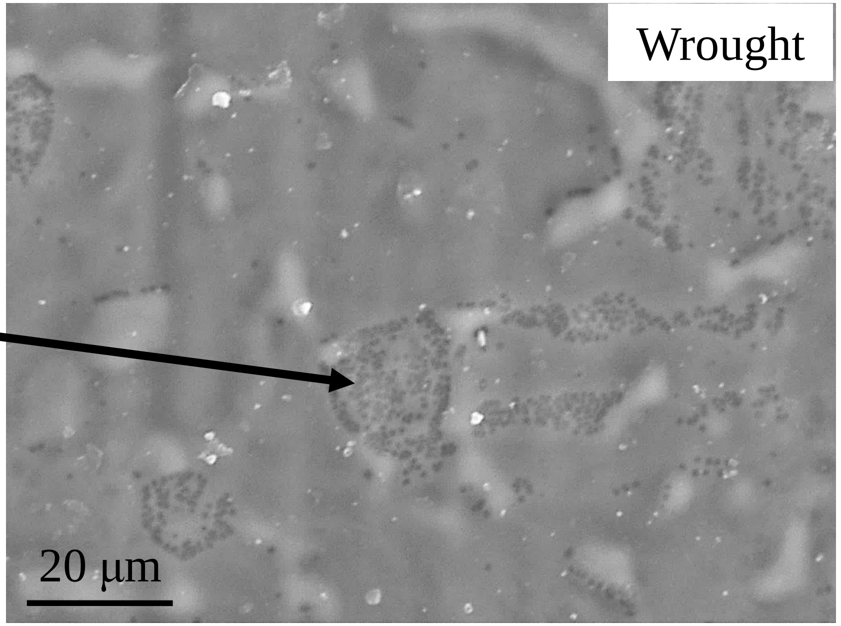
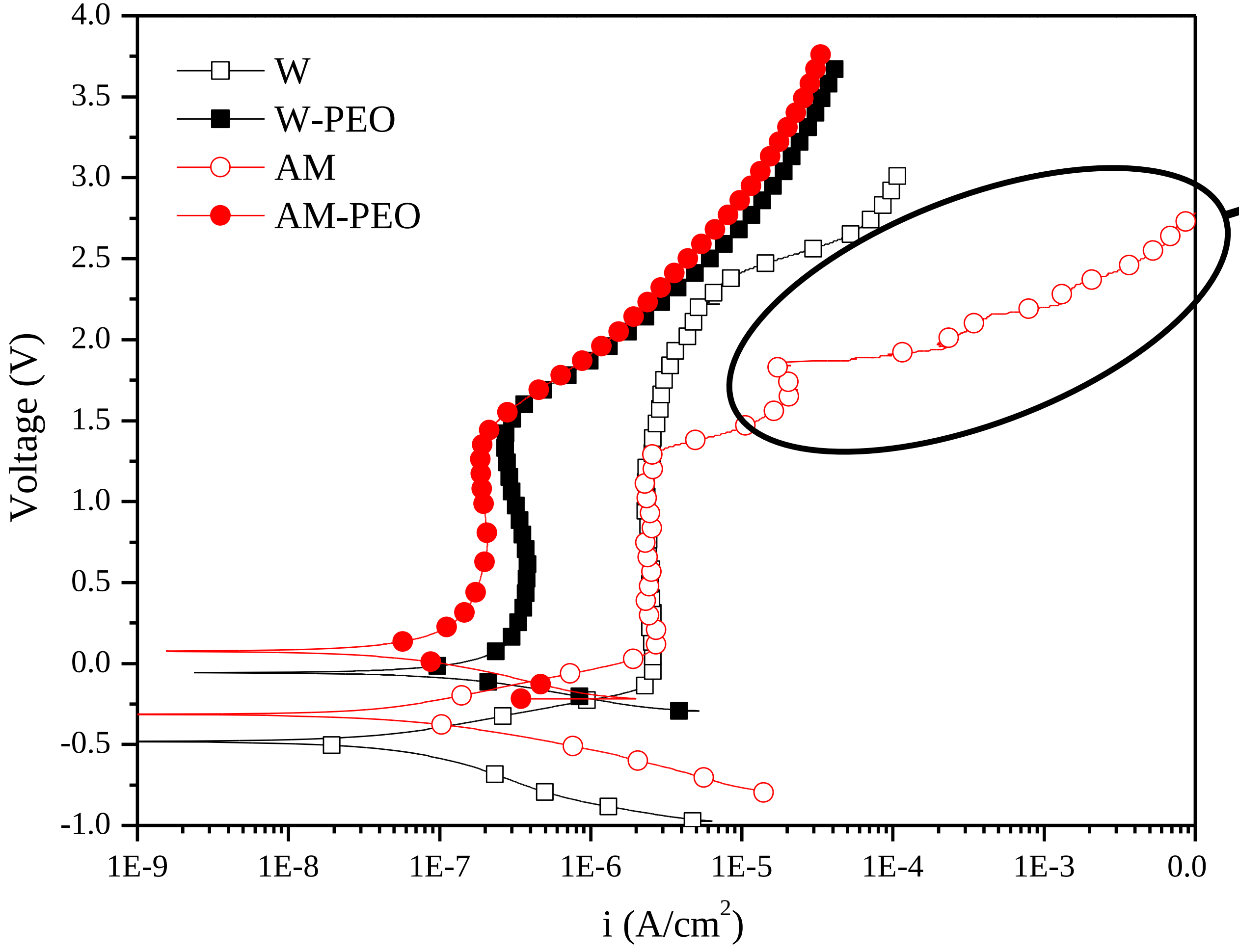


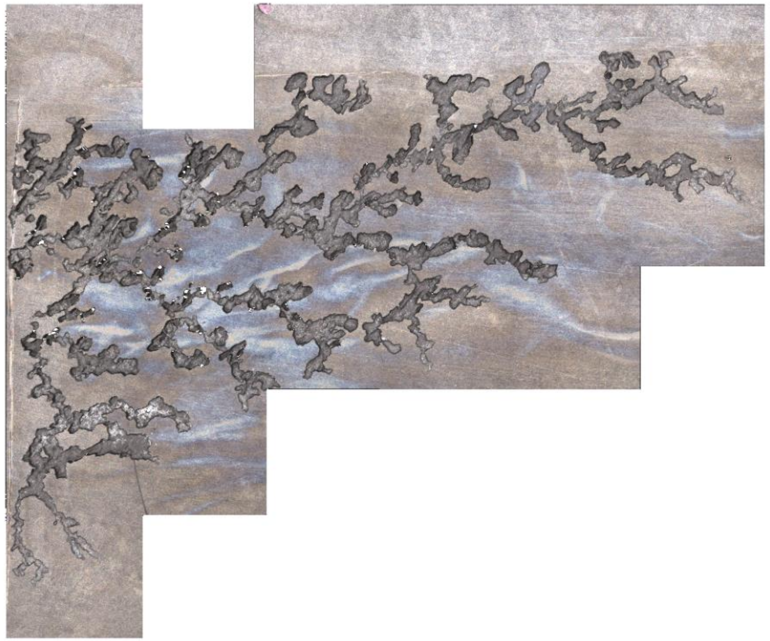
α basal-like planes

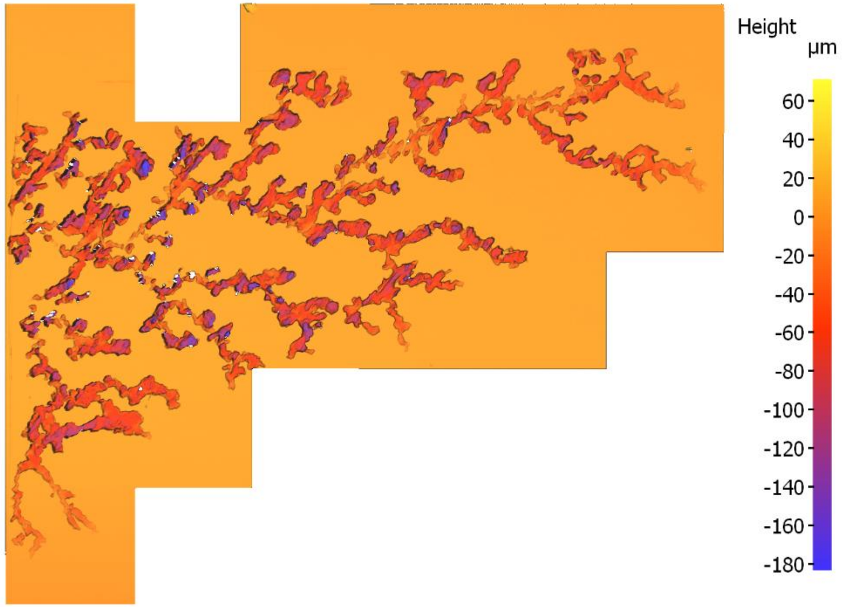
E. Matykina et al. *Thin Solid Films* 516 (2008) 2296–2305



3. Results: Potentiodynamic polarization







- The 120 s PEO coating on the Ti6Al4V AM alloy were similar in morphology and composition compared to the one fabricated on the conventional alloy
- Full PEO regime was reached earlier on the AM alloy
- Bare AM alloys presented severe pitting during electrochemical
- PEO coatings improved the corrosión resistance of both wrought and AM alloys