Junior EUROMAT
July 21st, 2022

Improving the interface between orthopaedic implants and bone – a comparison between different surface treatments

F. Melo-Fonseca[1,2,3], M. Gasik[4,5], Inês Mendes Pinto[3,6], S. Madeira[1,2], F. S. Silva[1,2], G. Miranda[7]

[1]Center for MicroElectroMechanical Systems (CMEMS-UMinho), University of Minho, Portugal
[2]LABBELS –Associate Laboratory, Braga, Guimarães, Portugal
[3]International Iberian Nanotechnology Laboratory (INL), Braga, Portugal
[4]School of Chemical Engineering, Aalto University Foundation, Espoo, Finland
[5]Seqvera Ltd., Helsinki, Finland
[6]i3S - Instituto de Investigação e Inovação em Saúde, Universidade do Porto, Portugal
[7]CICECO, Aveiro Institute of Materials, Department of Materials and Ceramic Engineering, University of Aveiro, Portugal

Universidade do Minho

INL INTERNATIONAL IBERIAN NANOTECHNOLOGY LABORATORY

MIT Portugal

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS

The team

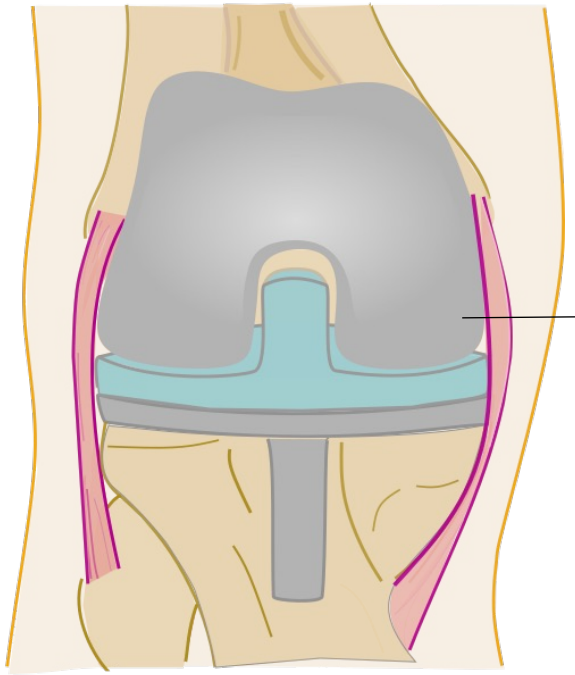
Research unit: Center for MicroElectromechanical Systems (CMEMS-UMinho)

Research unit: CICECO-Instituto de Materiais de Aveiro (CICECO/UA)

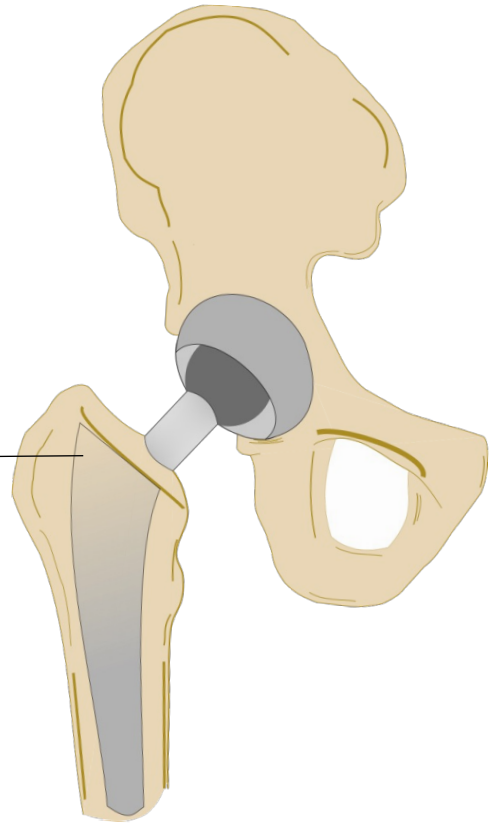
Contents

Contents

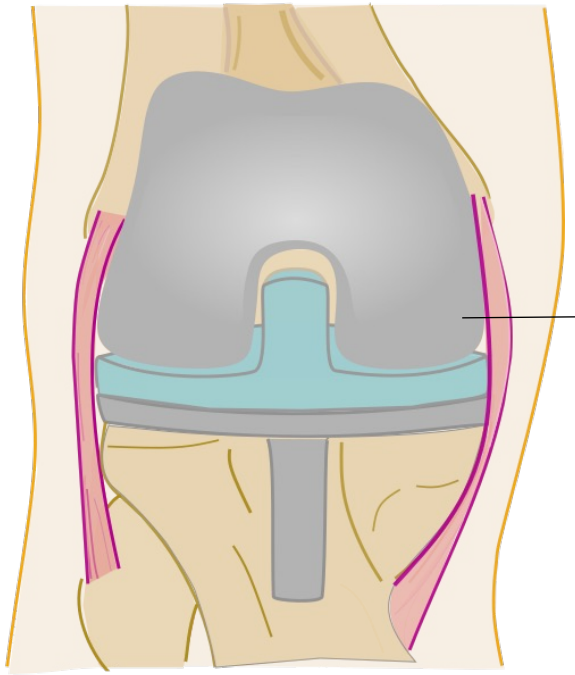
Motivation



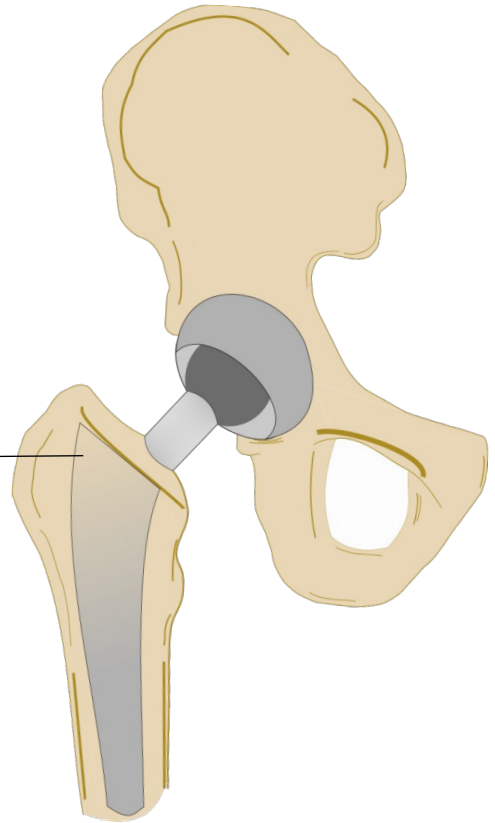
Titanium-based material



Motivation

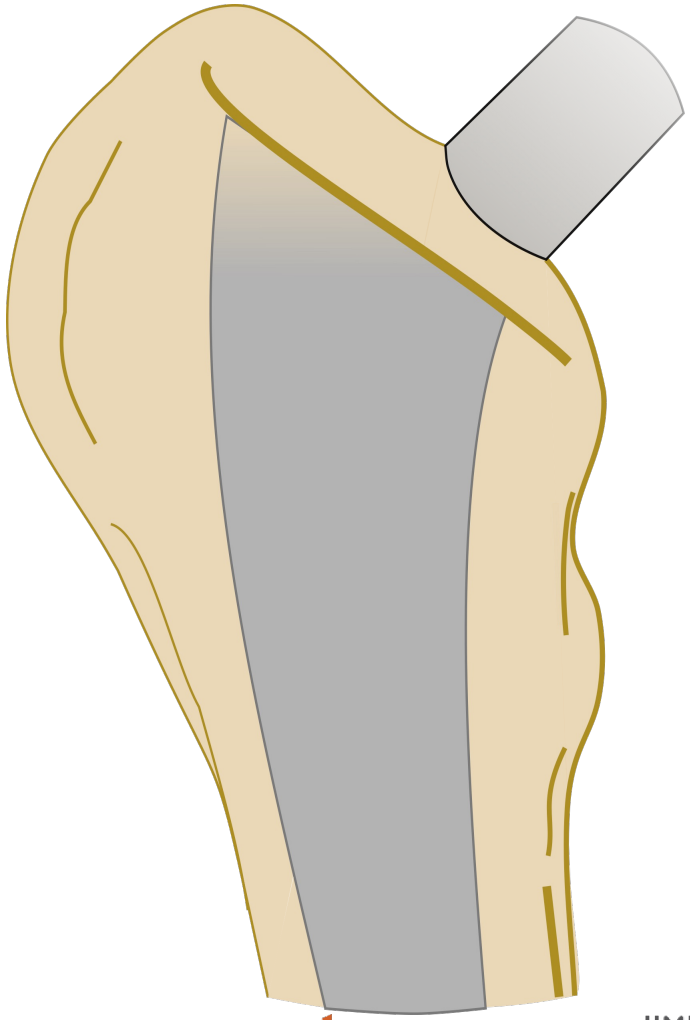


Insufficient bioactivity



Compromised osseointegration

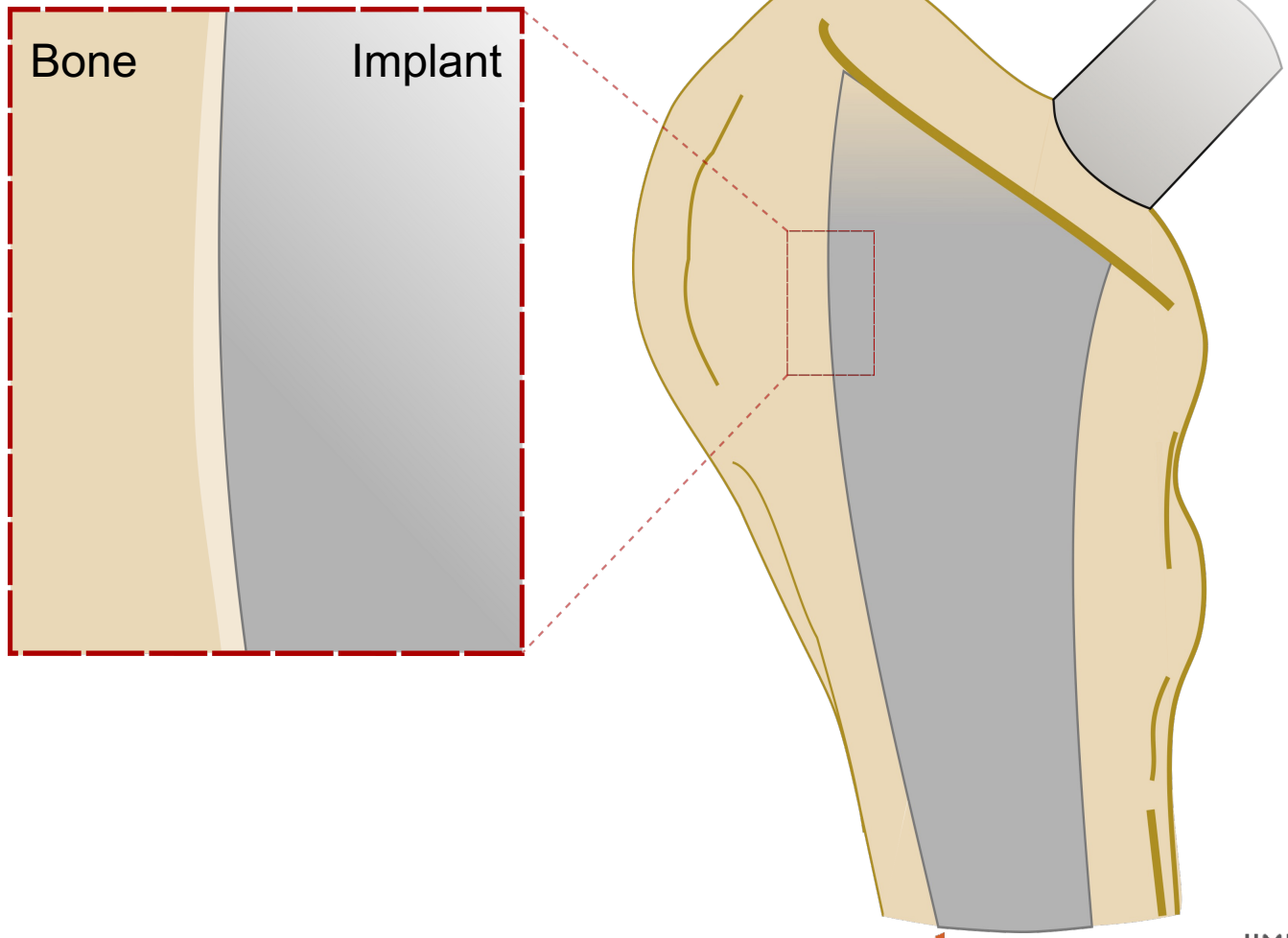
Motivation



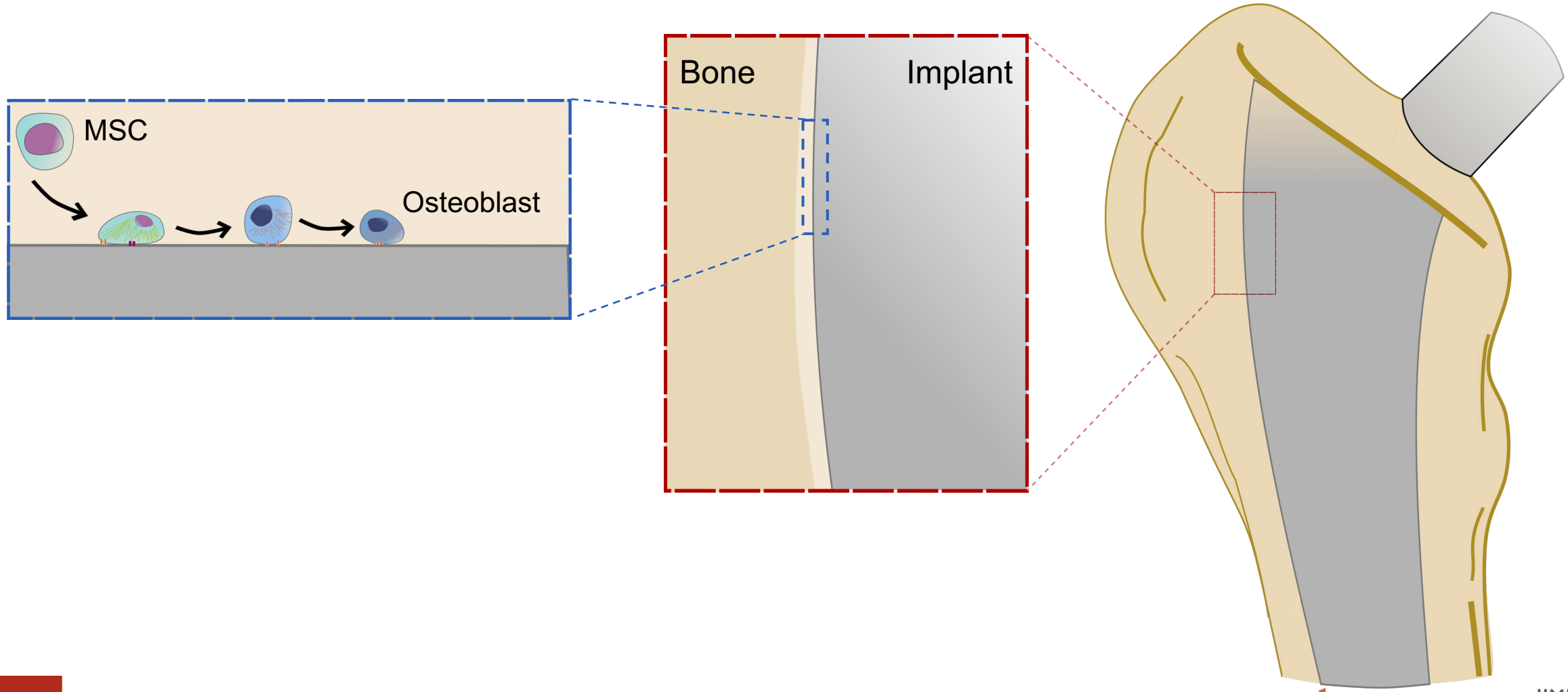
Motivation

Improvement of implant-bone interface:

- Surface chemical composition
- Surface energy
- Roughness
- Topography



Motivation

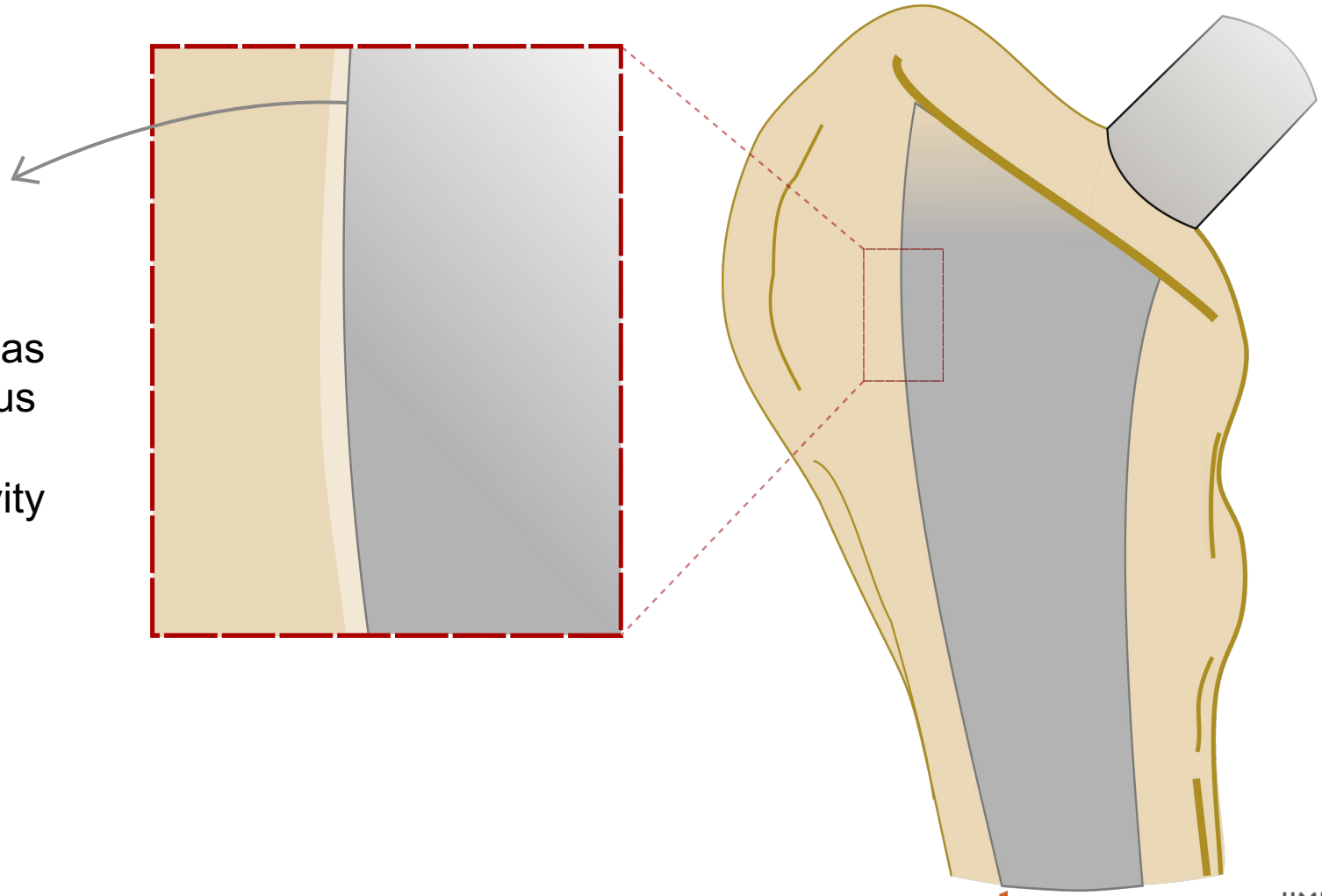


Motivation

TiO_2 layer

- When naturally formed, this layer has a tickness of 3-7 nm and is amorphous

- Ticker TiO_2 layer improves bioactivity and mechanical properties



Motivation

This oxide layer may be achieved by **surface modification techniques**

Pre-treatments:

- Mechanical polishing
- Chemical: alcohol cleaning vs acidic pre-treatment

Surface treatments:

- Anodic oxidation
- Hydrothermal treatment
- Anodic oxidation + hydrothermal treatment

Motivation

Compare surface treatments and investigate whether a simpler treatment would be effective to improve surface properties of titanium implants

Contents

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)

1. Pre-treatments:

- Non-polished vs **polished** → **mirror finishing**

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)

1. Pre-treatments:

- Non-polished vs polished
- Alcohol cleaning vs acidic pre-treatment

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)

1. Pre-treatments:

- Non-polished vs polished
- **Alcohol cleaning** vs acidic pre-treatment

↓

Cleaned with isopropyl alcohol

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)

1. Pre-treatments:

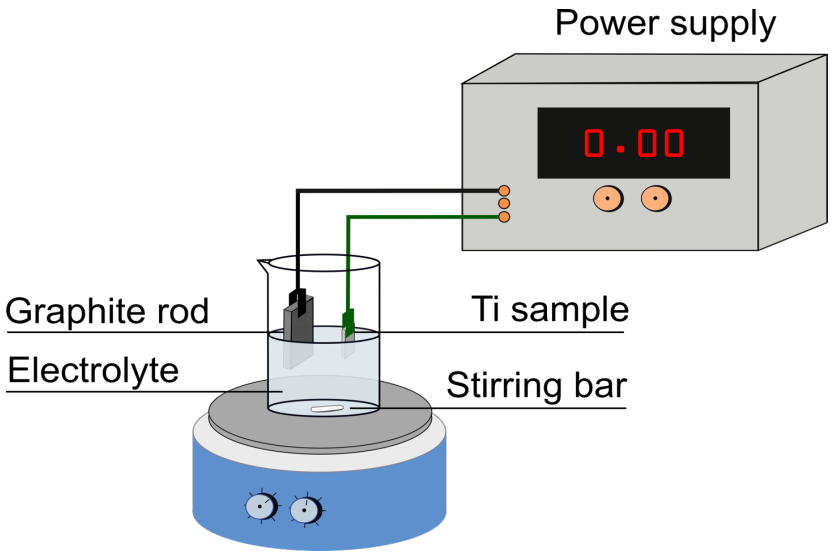
- Non-polished vs polished
- Alcohol cleaning vs **acidic pre-treatment**

10 M HCl (30 min) followed by ultrasonic rinsing with acetone (30 min)

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)



Electrolyte: 0.4 M H_3PO_4

Constante voltage of 120 V for 1 min

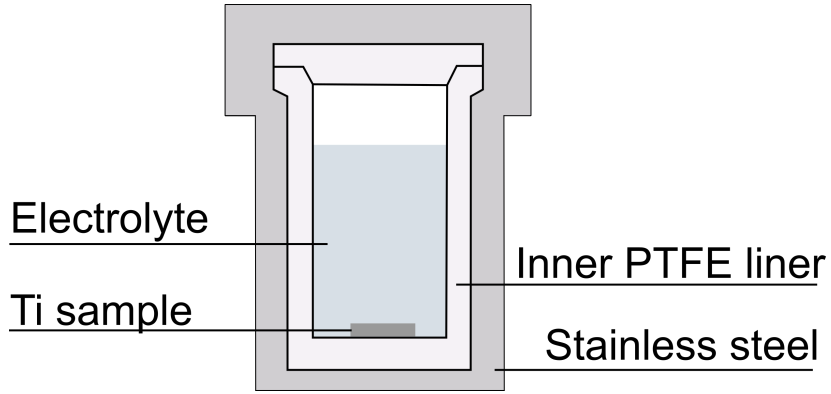
2. Surface treatment:

- **Anodic oxidation (AO)**

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)



Electrolyte: distilled water

180 ºC for 180 min

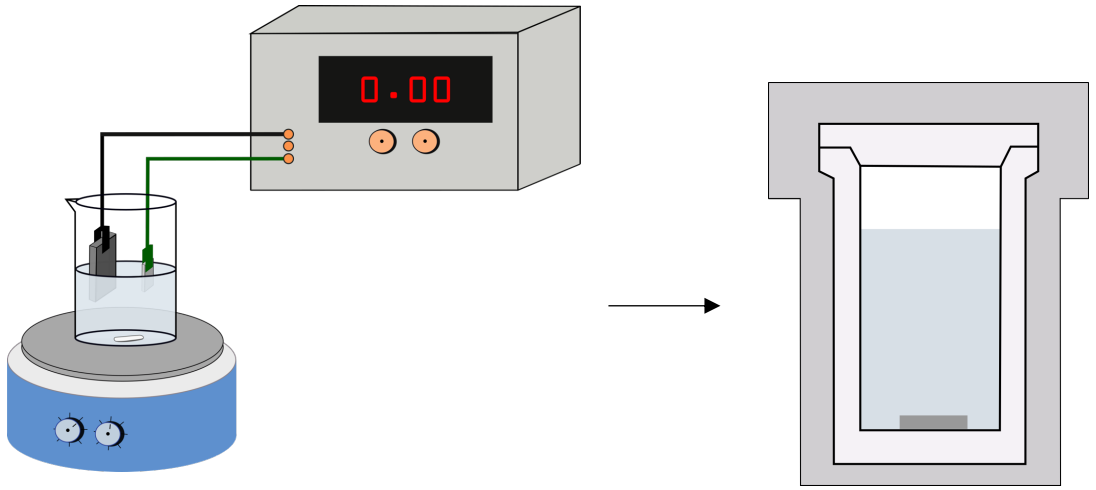
2. Surface treatment:

- Anodic oxidation (AO)
- **Hydrothermal treatment (Hydro)**

Experimental

Commercially pure Ti (Ti grade 2)

Ti6Al4V (Ti grade 5)



2. Surface treatment:

- Anodic oxidation (AO)
- Hydrothermal treatment (Hydro)
- **Anodic oxidation + hydrothermal treatment (AO + hydro)**

Experimental

Commercially pure Ti (Ti grade 2)

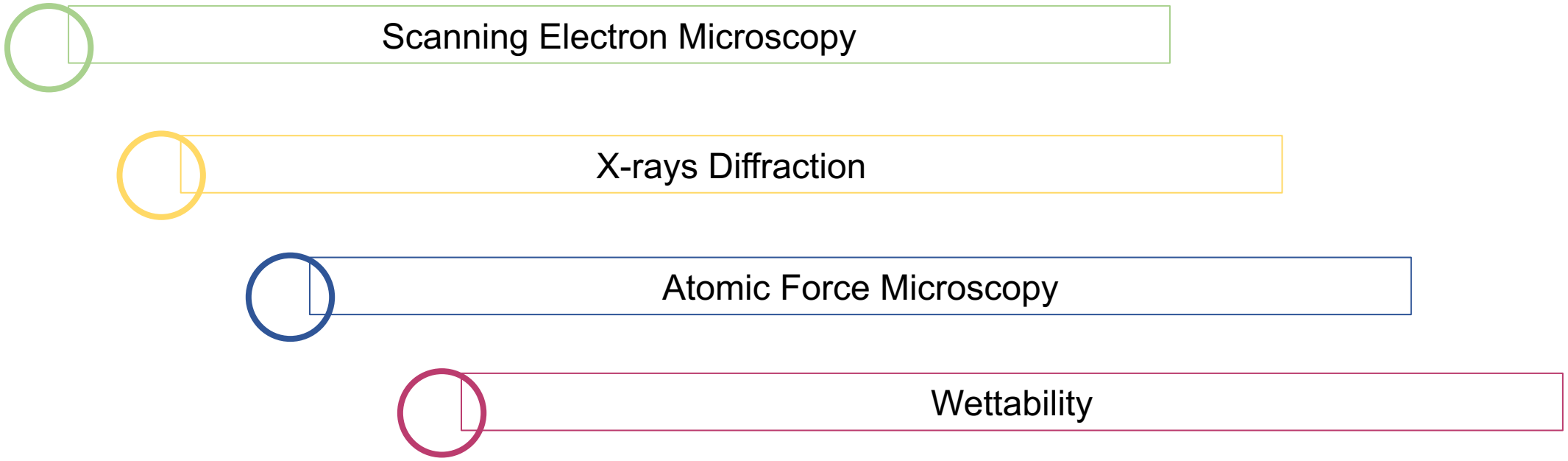
Ti6Al4V (Ti grade 5)

3. Sterilization and storage

- 125 ºC for 15 min
- 5X PBS for 24 h

Experimental

Characterization:



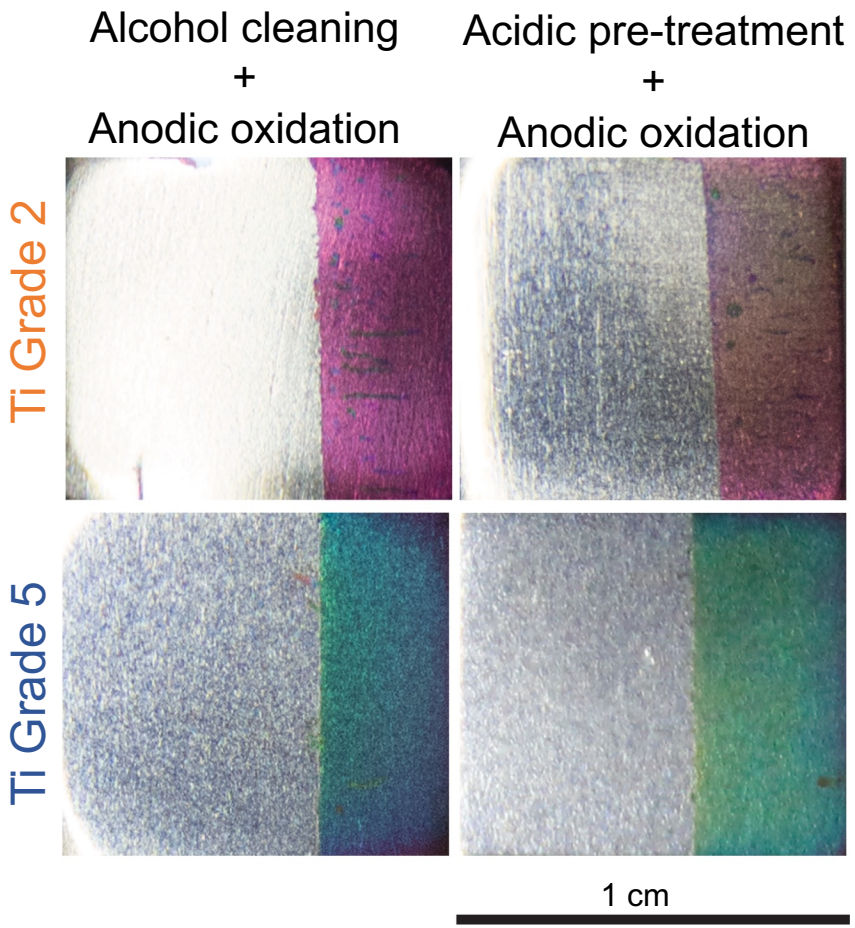
Experimental

1. Ti grade 2 and Ti grade 5 were subjected to different modification techniques
2. TiO_2 layer was characterized in-depth
3. Osseointegration potential was assessed by a preliminary cellular assay

Contents

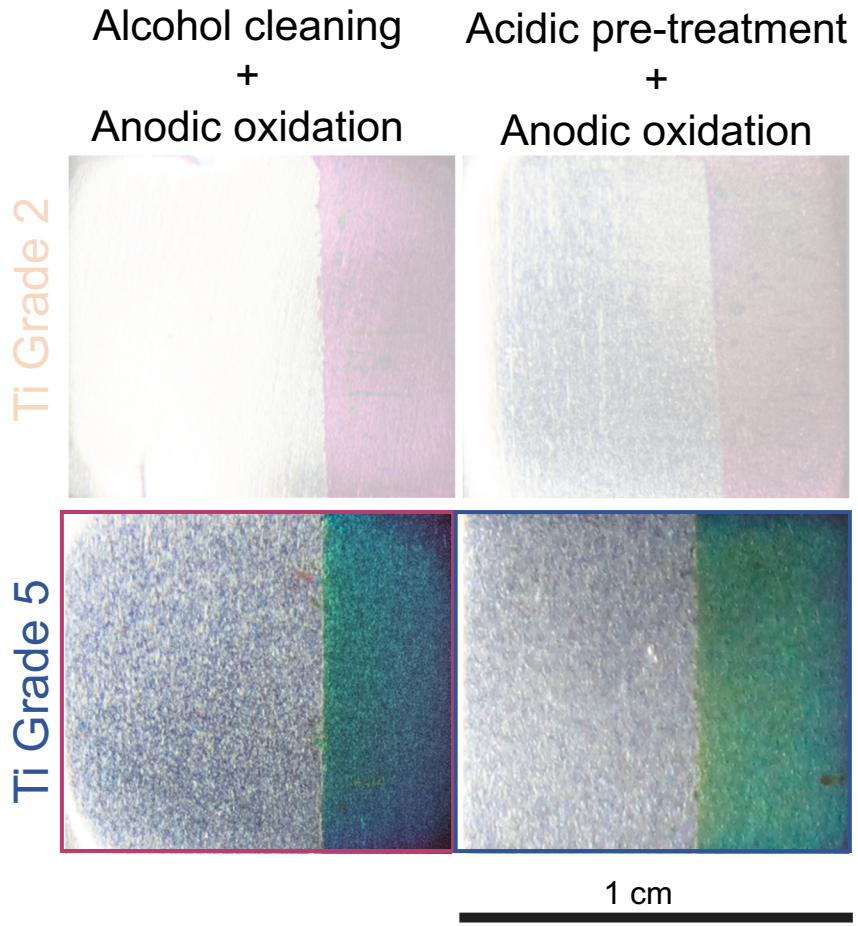
Results

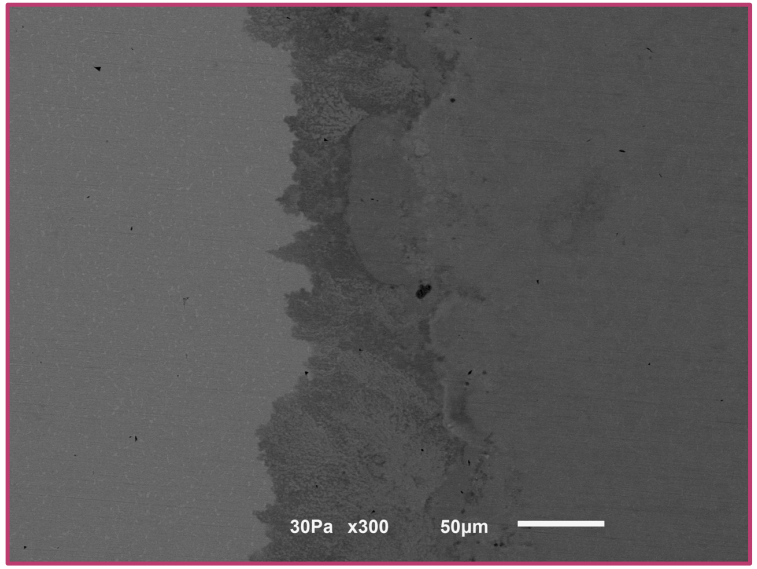
Morphology

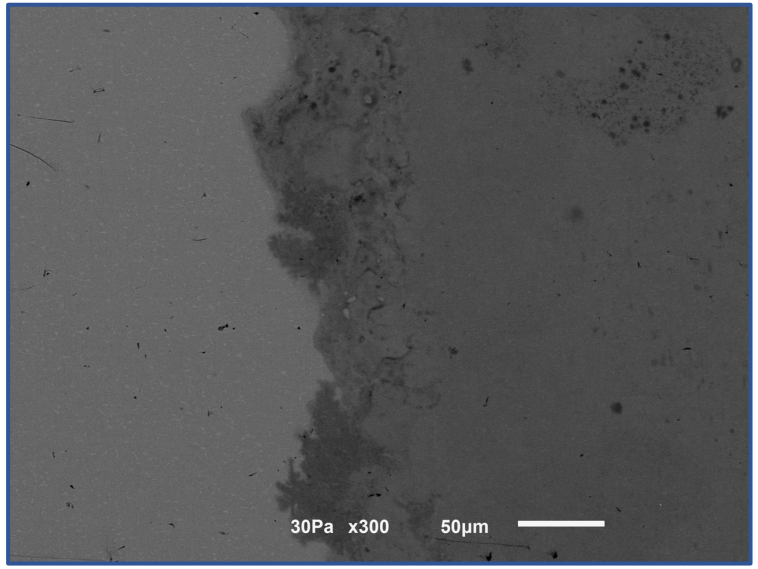


Results

Morphology



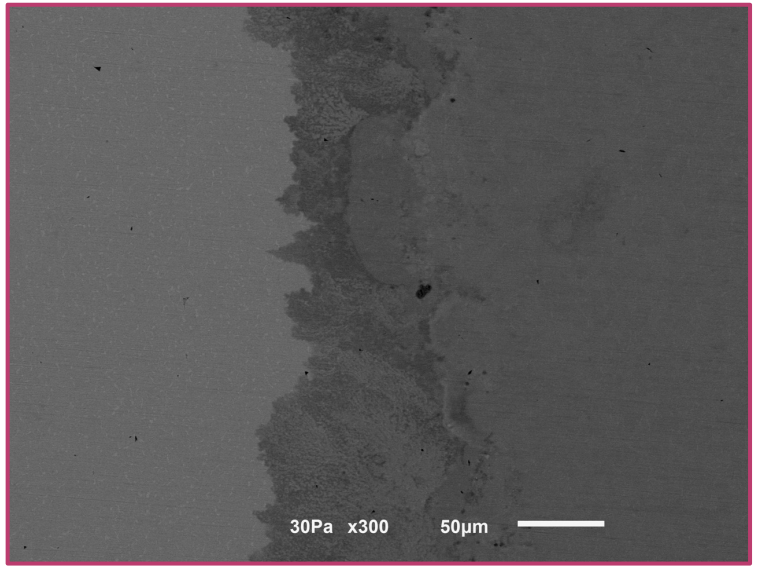


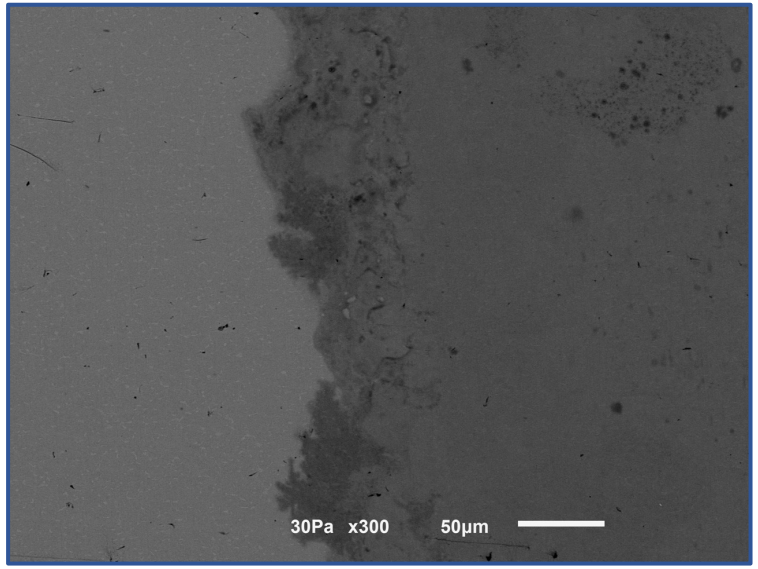


Results

Morphology

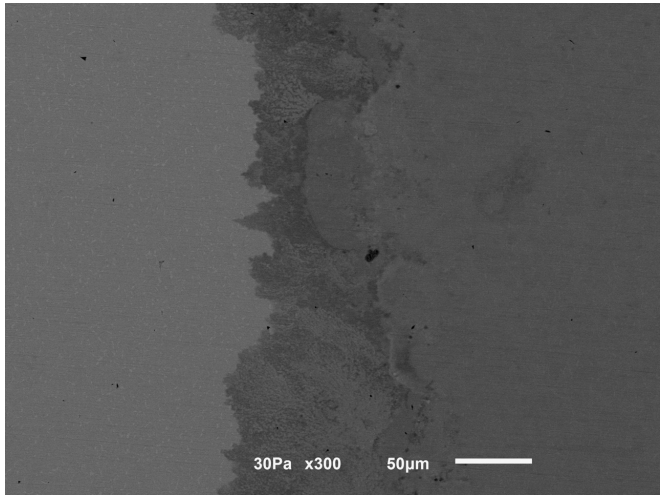
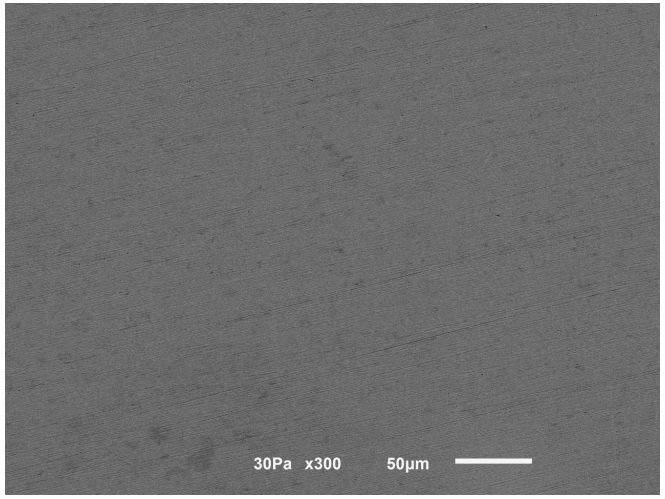
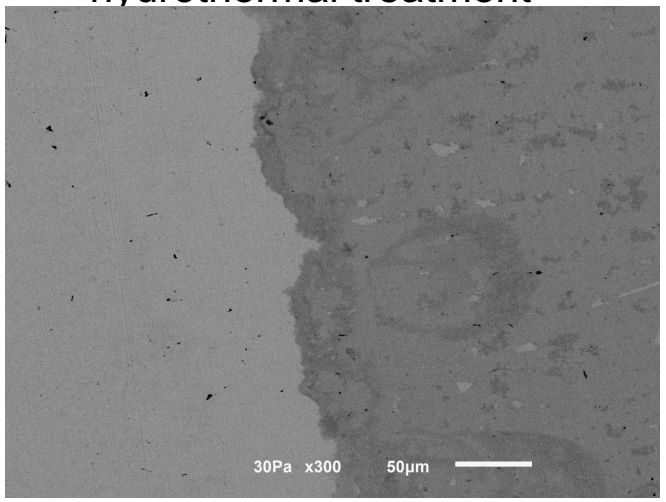
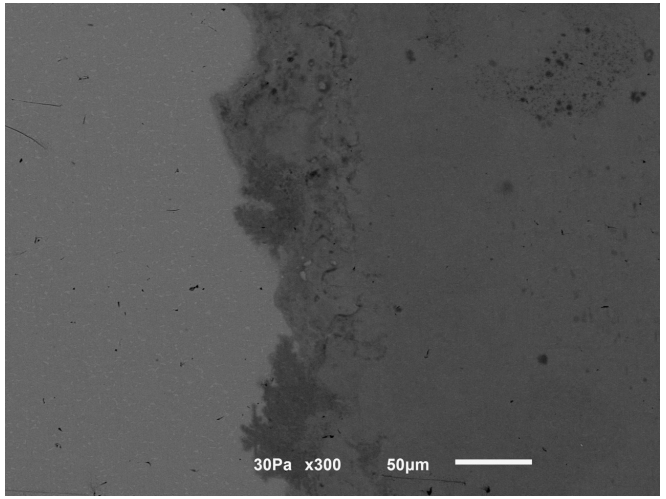
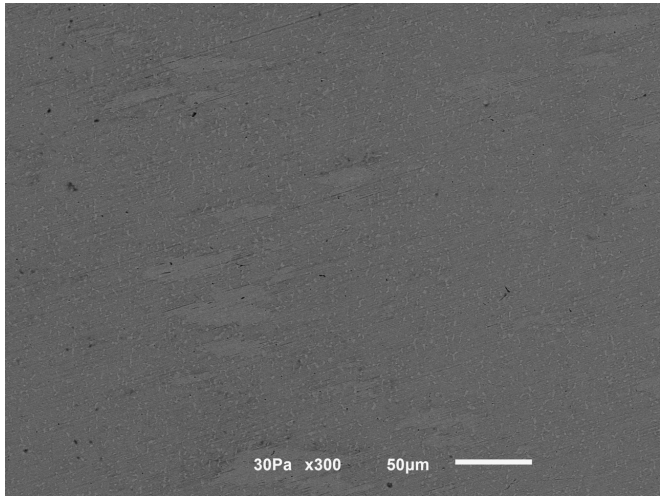
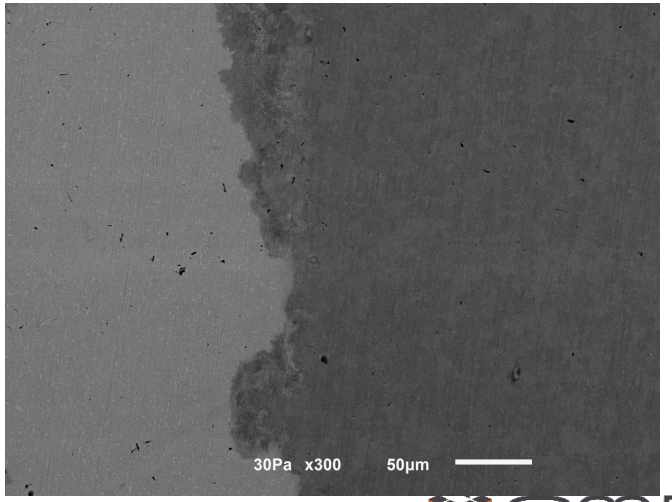
Both chemical pre-treatments are effective in **samples cleaning** and did not alter the surface morphology





Results

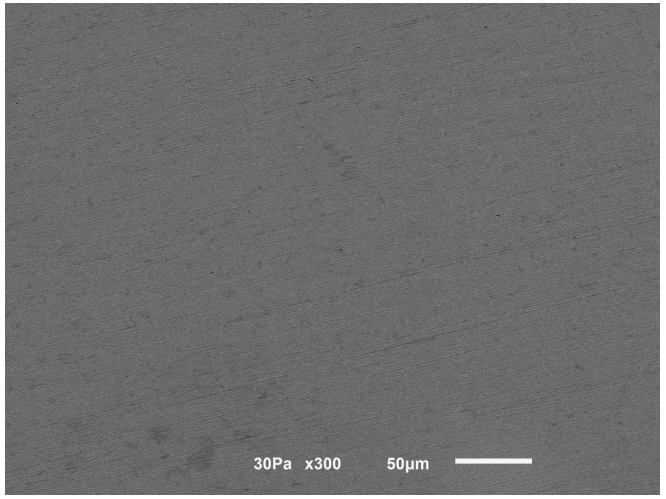
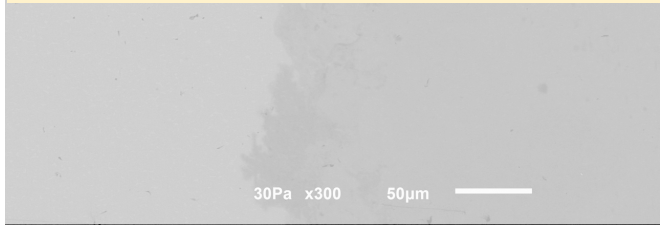
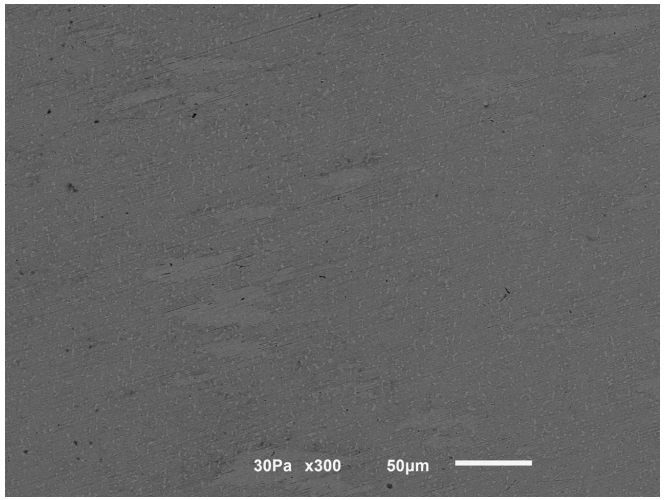
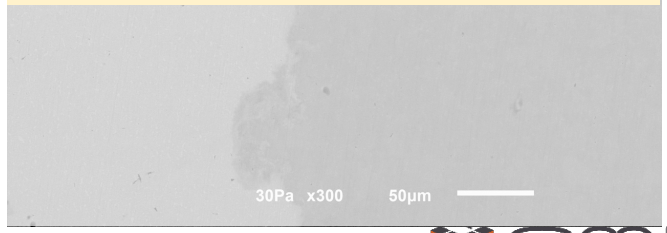
Morphology

	Anodic oxidation	Hydrothermal treatment	Anodic oxidation + hydrothermal treatment
Ti Grade 2			
Ti Grade 5			

Ti-based samples subjected to the acidic pre-treatment

Results

Morphology

	Anodic oxidation	Hydrothermal treatment	Anodic oxidation + hydrothermal treatment
Ti Grade 2	Oxide film presents the same surface morphology compared to the non-treated side		The oxide film did not present a sufficient thickness able to ascertain a clear transition
Ti Grade 5			

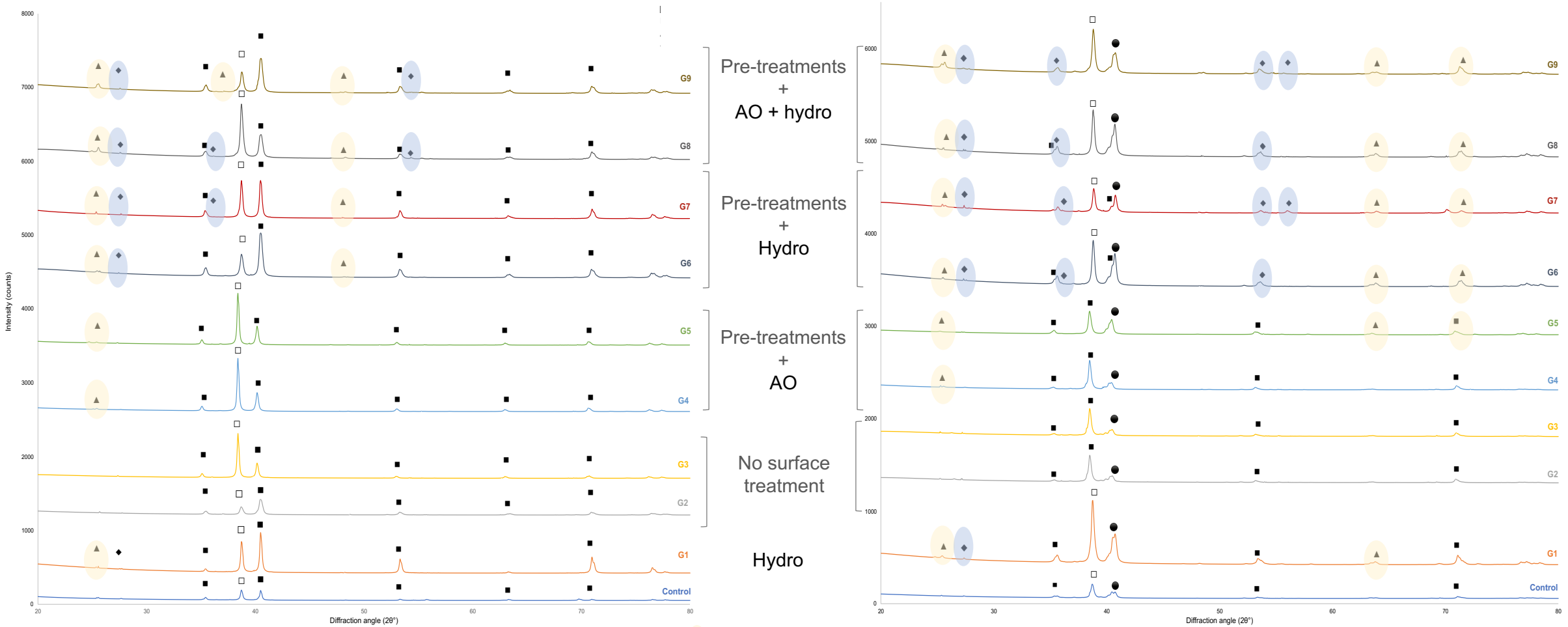
Ti-based samples subjected to the acidic pre-treatment

Results

Phase composition

Ti Grade 2

Ti Grade 5



Results

Phase composition

Anatase was found for **all** treated samples,

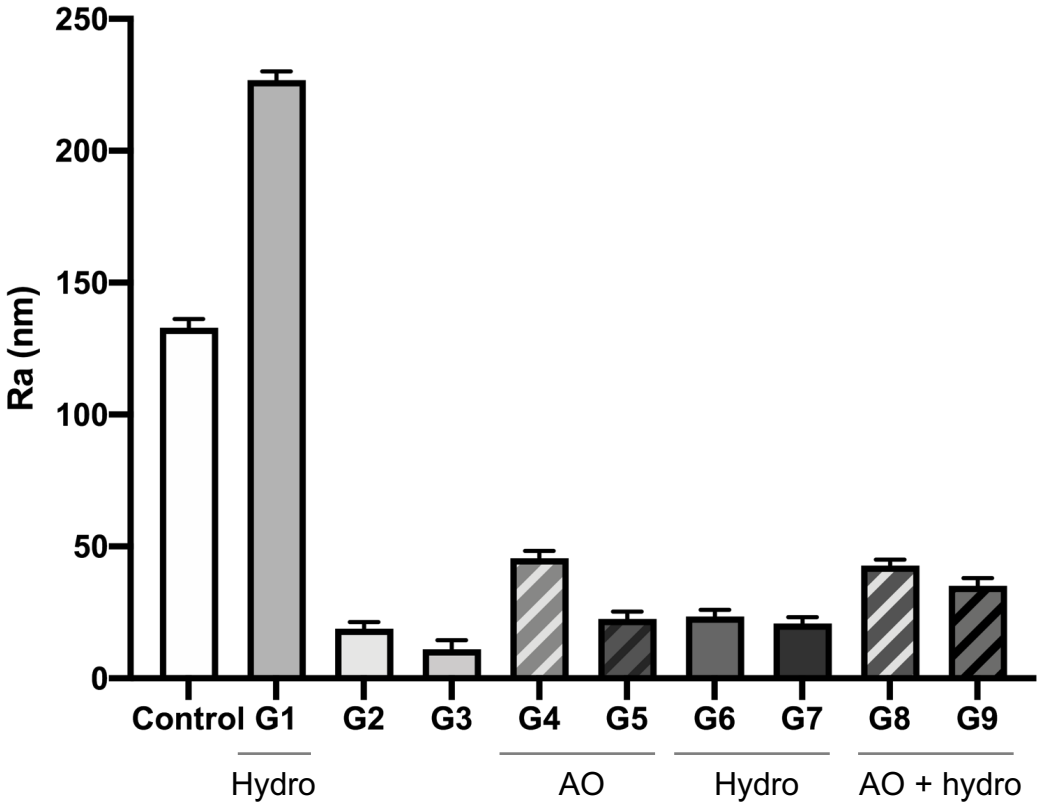
whereas **rutile** was only obtained for the **hydrothermal treatment**

The presence of **both crystalline phases** is preferable then an amorphous or plain anatase structure

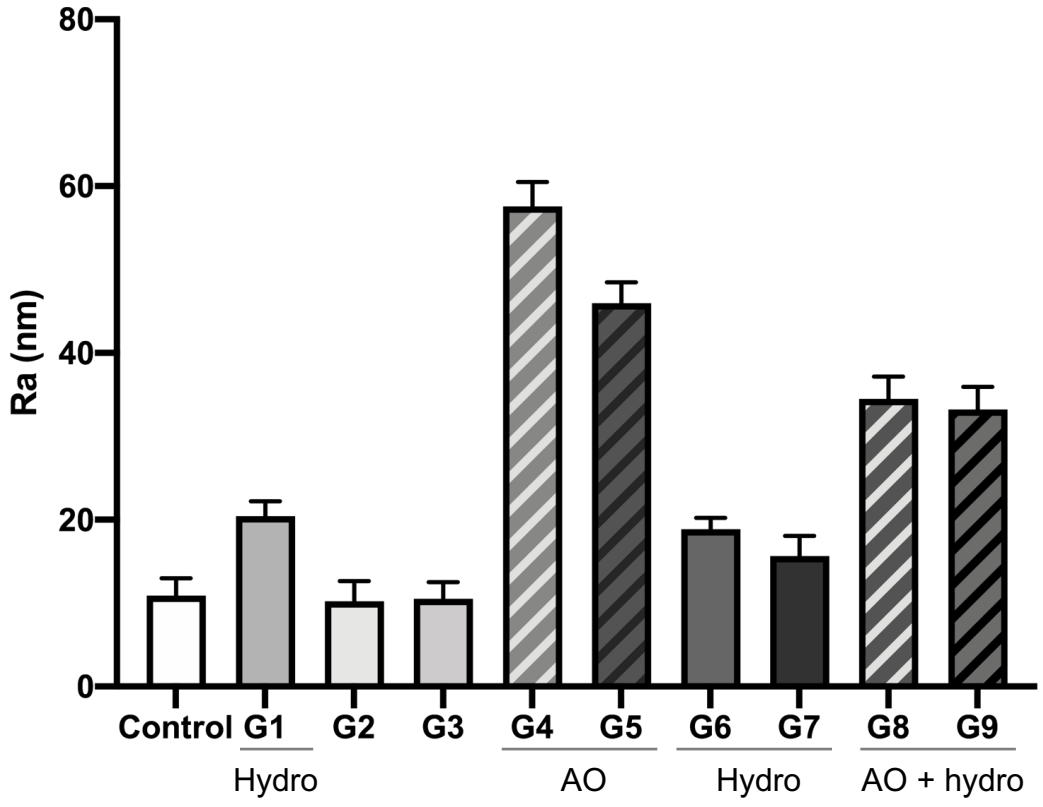
Results

Roughness

Ti Grade 2

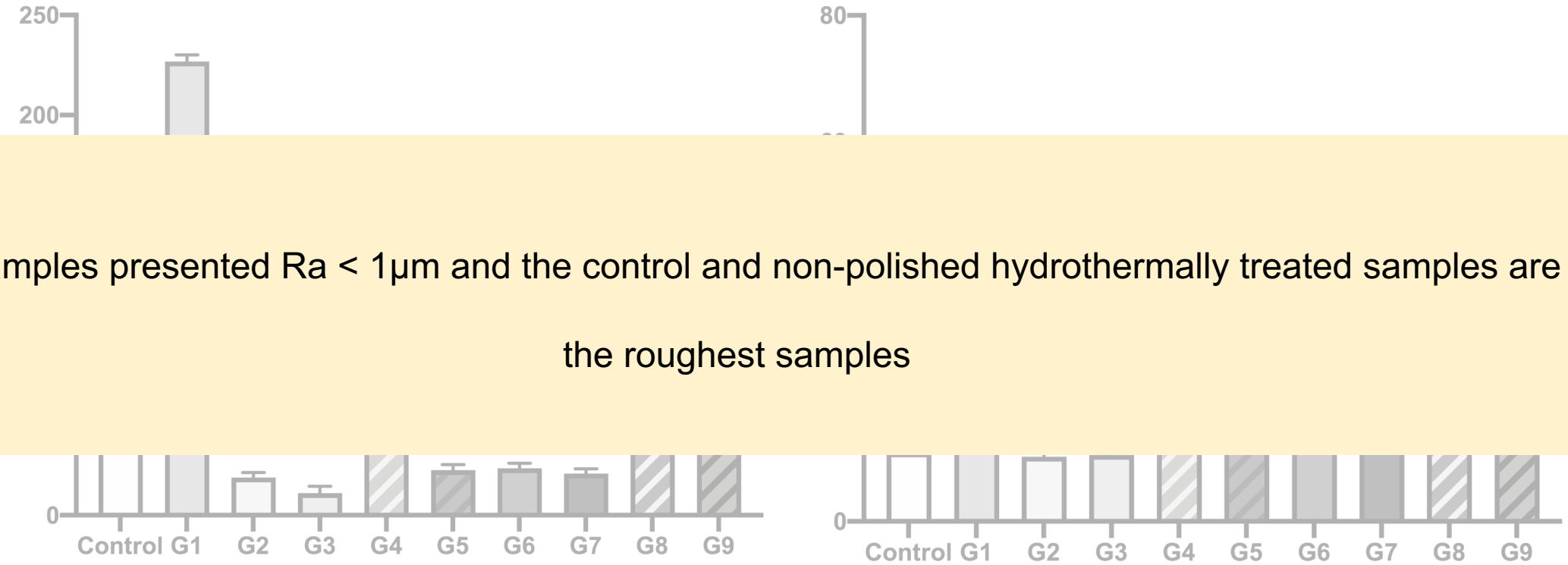


Ti Grade 5



Results

Roughness

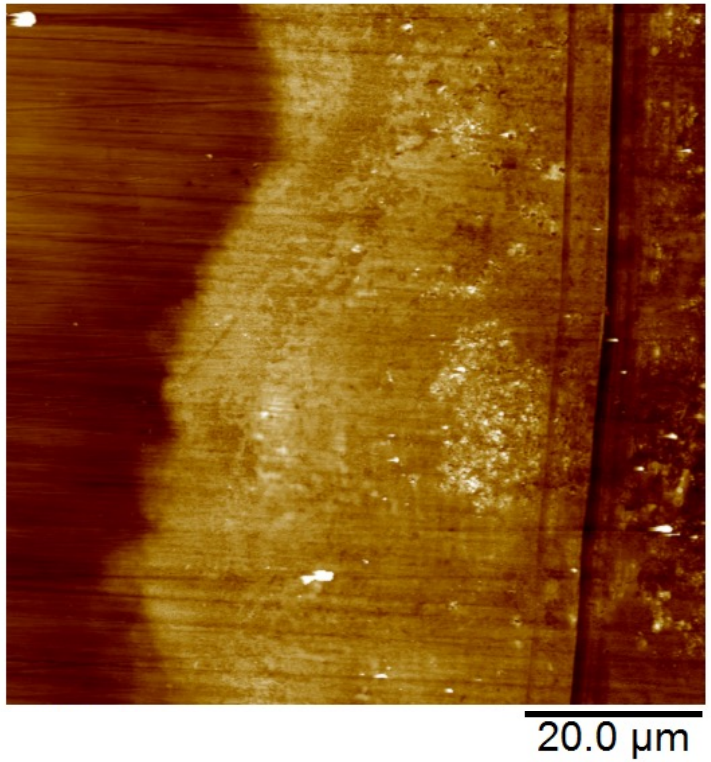


All samples presented Ra < 1µm and the control and non-polished hydrothermally treated samples are the roughest samples

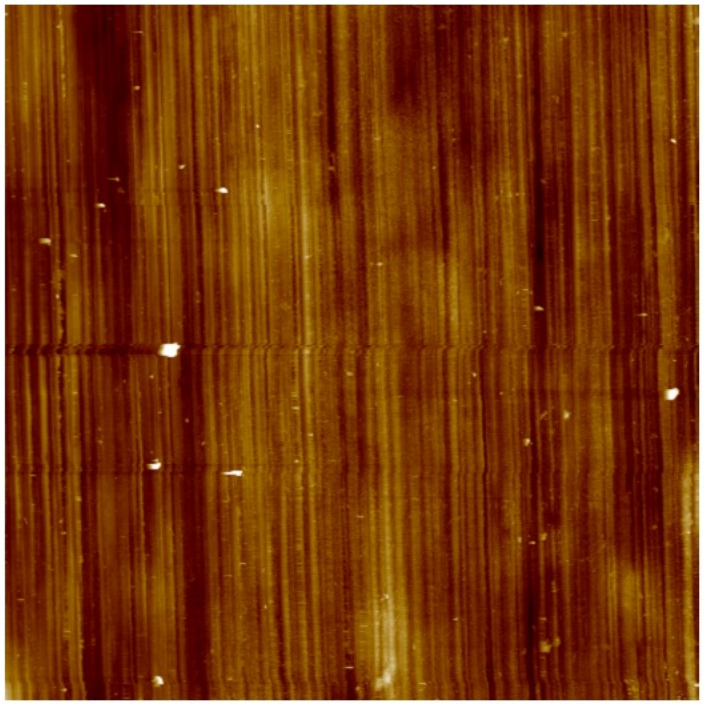
Results

TiO_2 thickness

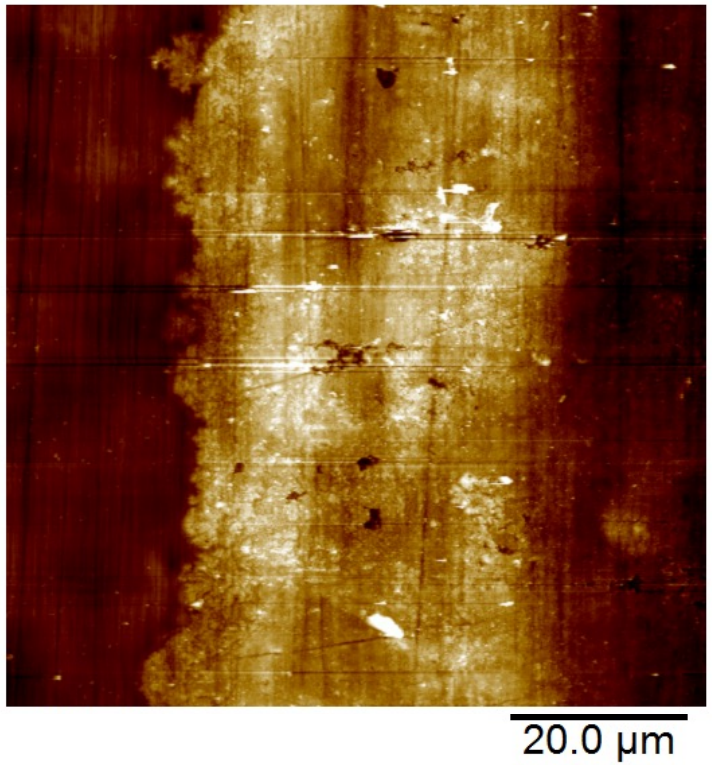
Anodic oxidation

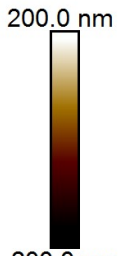


Hydrothermal treatment



Anodic oxidation + hydrothermal treatment

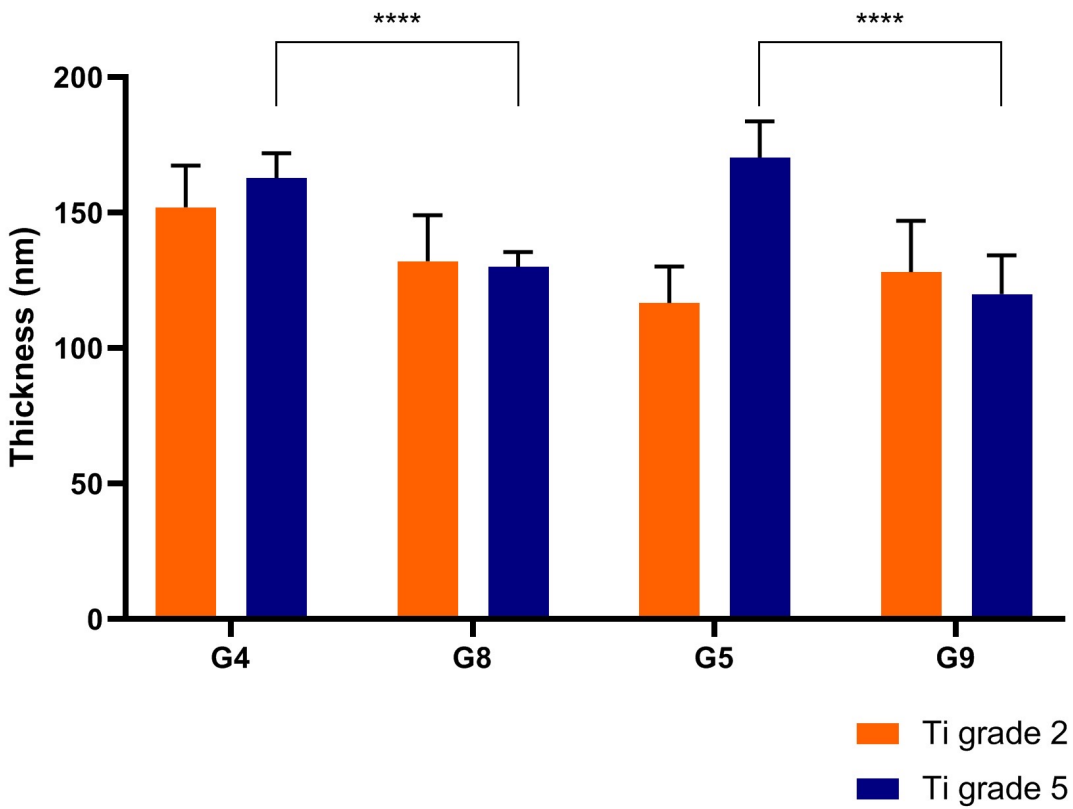




Ti grade 2 samples subjected to the acidic pre-treatment

Results

TiO_2 thickness



G4: Alcohol treatment + anodic oxidation
G5: Acidic pre-treatment + anodic oxidation
G8: Alcohol treatment + anodic oxidation + hydrothermal treatment
G9: Acidic pre-treatment + anodic oxidation + hydrothermal treatment

Anodic oxidation and anodic oxidation + hydrothermal treatment produced an oxide layer with a thickness > 100 nm

Results

Wettability

Pre-treatment		No pre-treatment	Mechanical polishing and chemical pre-treatment			
Surface treatment		Hydro	No surface treatment	AO	Hydro	AO + hydro
Ti Grade 2	Anatase	+		+	+	++++
	Rutile	+			+++	++
	Wettabiliy (0h)	++	+	++	+	++
	Wettability (24h in 5XPBS)	++	+	++	++	+++
Ti Grade 5	Anatase	+++		++	+	++++
	Rutile	+			++	++
	Wettabiliy (0h)	+	+	++	+	+
	Wettability (24h in 5XPBS)	++	++	++	+++	+++

Results

Overview

The presence of both anatase and rutile is preferable for cellular outcomes and confers bioactivity

Micro-rough surfaces are more prone to cell anchorage than smooth surfaces

Moderate-hydrophilic surfaces promote cellular and protein adhesion

Results

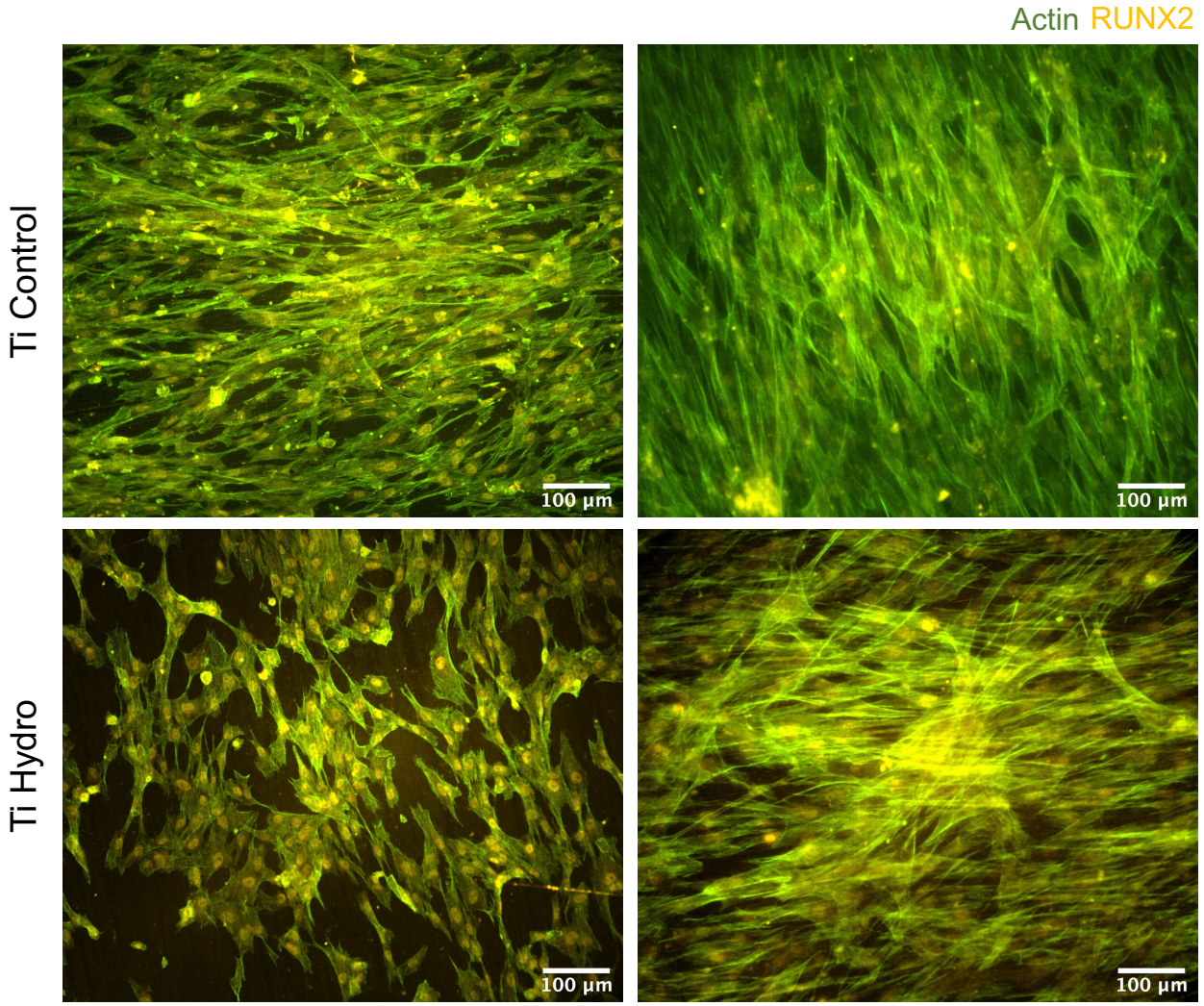
Overview

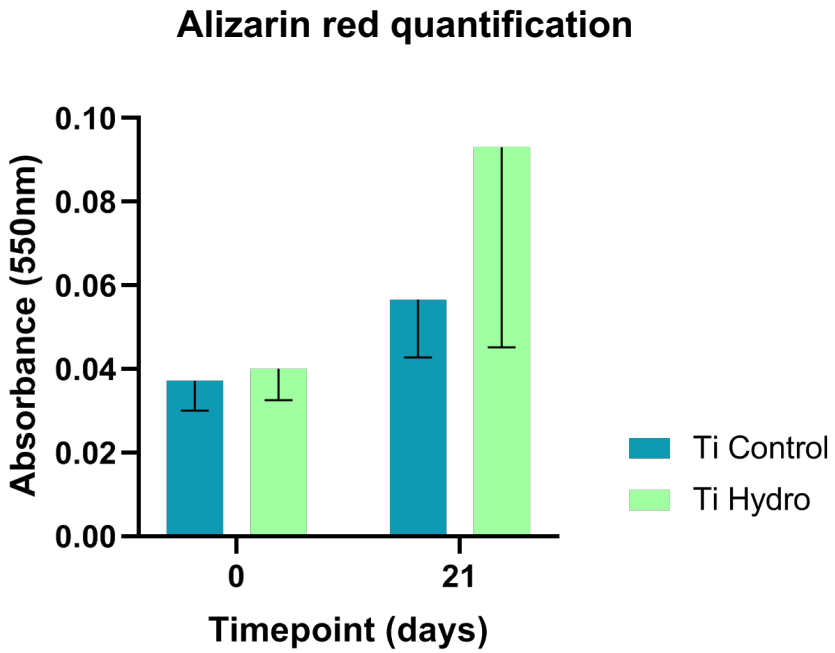
Alcohol cleaning and **hydrothermal treatment** followed by immersion for 24 hours in 5X PBS is an effective and simple surface modification treatment capable of creating a moderate-hydrophilic and bioactive surface

Results

BM-hMSC adhesion

Bone marrow derived human Mesenchymal Stem Cells (BM-hMSC) on Ti grade 5





Contents

Conclusions

No major differences were observed between **pre-treatments** (alcohol cleaning vs acidic pre-treatment) considering surface crystallinity, roughness and wettability

TiO_2 layer obtained by AO, Hydro and AO + hydro presented different characteristics regarding its crystallinity, roughness, thickness and wettability

Alcohol cleaning followed by hydrothermal treatment is a simple methodology that results in a bioactive oxide layer which properties are capable of enhancing bone-implant interface

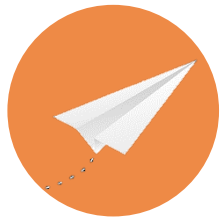
Contents

Acknowledgments

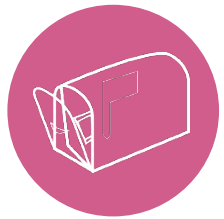
This work was supported by FCT (Fundação para a Ciência e a Tecnologia) through the grant SFRH/BD/141056/2018 and the projects PTDC/EME-EME/1442/2020, POCI-01-0145-FEDER-030498, UIDB/04436/2019 and UIDP/04436/2020. In addition, this work was developed within the scope of the project CICECO-Aveiro Institute of Materials, UIDB/50011/2020, UIDP/50011/2020 & LA/P/0006/2020, financed by national funds through the FCT/MEC (PIDDAC).

Thank you for your attention!

Francisca Melo-Fonseca
PhD student in Leaders for Technical Industries (MIT-Portugal)



francisca.fonseca@inl.int
franciscarmelofonseca@gmail.com



Universidade do Minho, Campus de Azurém,
4800-058 Guimarães, Portugal

+351 253 510 732

Junior EUROMAT
July 21st, 2022

Improving the interface between orthopaedic implants and bone – a comparison between different surface treatments

F. Melo-Fonseca[1,2,3], M. Gasik[4,5], Inês Mendes Pinto[3,6], S. Madeira[1,2], F. S. Silva[1,2], G. Miranda[7]

[1]Center for MicroElectroMechanical Systems (CMEMS-UMinho), University of Minho, Portugal
[2]LABBELS –Associate Laboratory, Braga, Guimarães, Portugal
[3]International Iberian Nanotechnology Laboratory (INL), Braga, Portugal
[4]School of Chemical Engineering, Aalto University Foundation, Espoo, Finland
[5]Seqvera Ltd., Helsinki, Finland
[6]i3S - Instituto de Investigação e Inovação em Saúde, Universidade do Porto, Portugal
[7]CICECO, Aveiro Institute of Materials, Department of Materials and Ceramic Engineering, University of Aveiro, Portugal

Universidade do Minho

INL INTERNATIONAL IBERIAN NANOTECHNOLOGY LABORATORY

MIT Portugal

UMINHO CMEMS CENTER FOR MICROELECTROMECHANICAL SYSTEMS