

Abstract

Titanium has been widely used as a bioimplant material due to its excellent biocompatibility that relates to the nature of the titanium surface (Bauer S., 2013). It is known that surface nanostructuring further enhances Ti biocompatibility (Eliasa C.N., 2008). Chemical Mechanical Polishing (CMP) is a process commonly applied in microelectronics manufacturing for interlayer planarization and it has recently been introduced as a nano-structuring technique for implant surfaces (Zantye P.B., 2004). Titanium surface modification by CMP enables both chemical and mechanical modifications simultaneously. In this study, titanium plates CMP'ed with an alumina based slurry and H₂O₂ oxidizer were characterized for the growth and structure of the nano-scale oxide layer and biocompatibility through cell growth analyses. In order to be able to implement the CMP process on 3D structures such as dental and orthopedic implants, a 3-D CMP process is being developed.

Objectives

Control the bio-compatibility and infection resistance of implantable materials through control of surface roughness induced by Chemical Mechanical Planarization (CMP)

- Reduce surface contamination through micro-scale material removal rates and creating a nano/micro surface roughness (Basim, G.B., 2002).
- Create a self protective surface oxide on titanium that can prevent further contamination and promote bio-activity (Chathapuram V.S,2003), (Variola, F.,2008), (Jouanny, I., 2010)
- Design a 3-D CMP process to apply CMP on 3-D implant materials.

Experimental

Materials:

- Titanium foils : 1 mm thickness and 99.6% purity (Ti000430) from Goodfellow Cambridge Limited
- CMP : 70 N downforce, Suba IV subpad stacked under a polytex buff pad and abrasive paper
- Slurry: 5% wt Al₂O₃ , 0.05µm size
- Oxidizer : 3 %wtH₂O₂

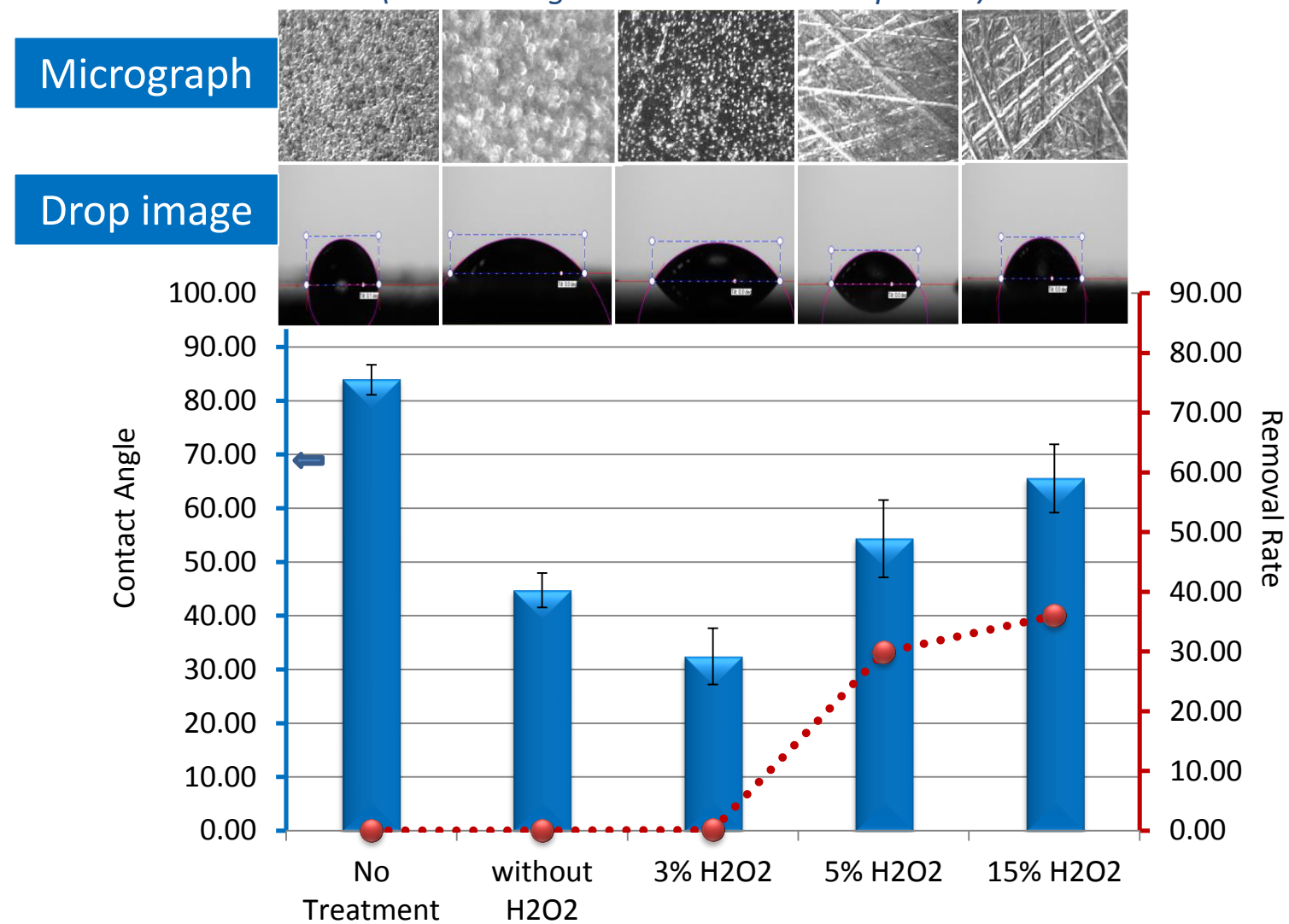
Methods:

- Surface characterization:** Surface wettability measured by Contact Angle with sessile drop method by body serum. AFM(Atomic Force Microscopy) used to obtain surface morphology and roughness value. XRD, EDX and XPS applied to achieve surface combination difference with various chemicals.
- Biological tests:** Bacterial growth test applied 7 day as first step of biological test. Cytotoxicity test, aimed detection of the biological activity of samples after CMP. Additionally cell adhesion test applied to see surface combination and roughness effect.

Results

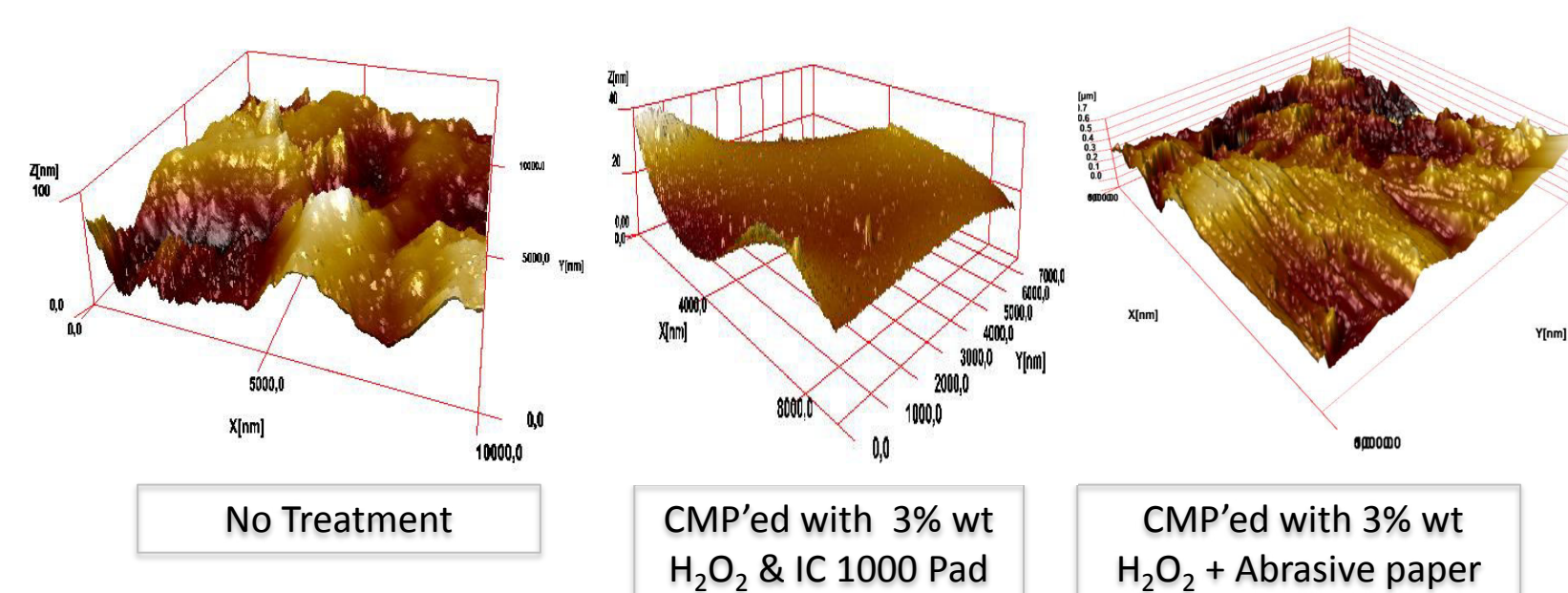
Surface Characterization

Optical micrographs of Ti samples pre and post CMP
(contact angle & removal rate responses)



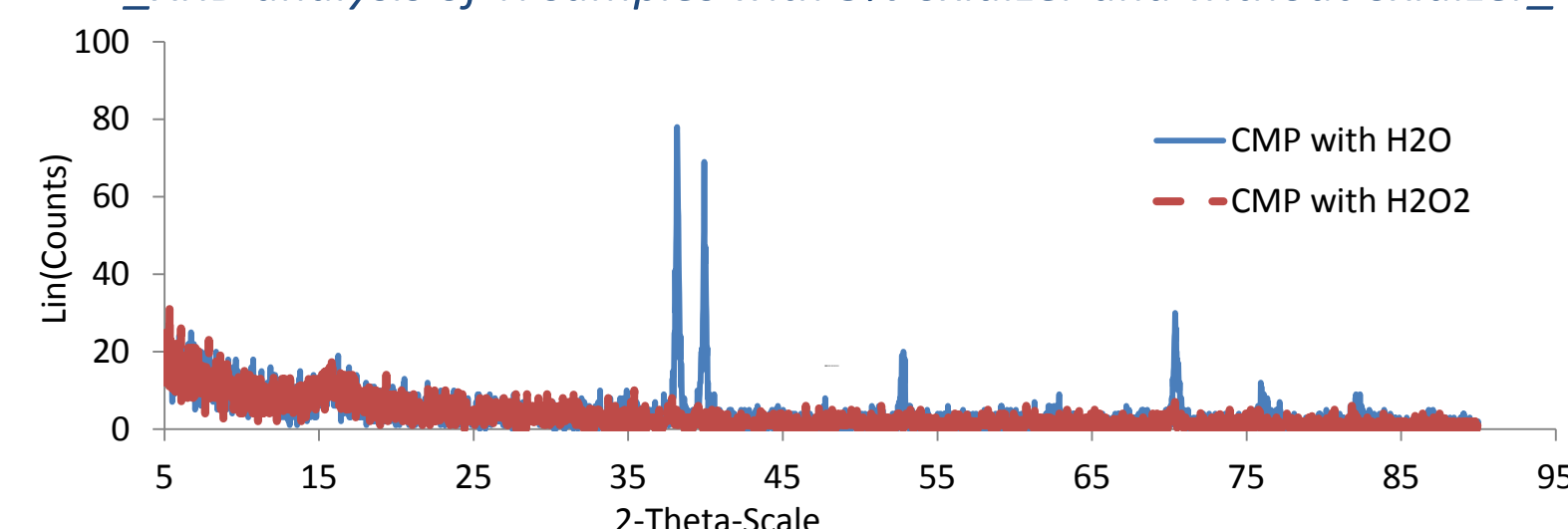
- It can be seen that the increased roughness through porosity or induced scratching results in higher contact angle indicating a more hydrophobic and less wettability on the surface.

Titanium samples surface characterization by AFM images

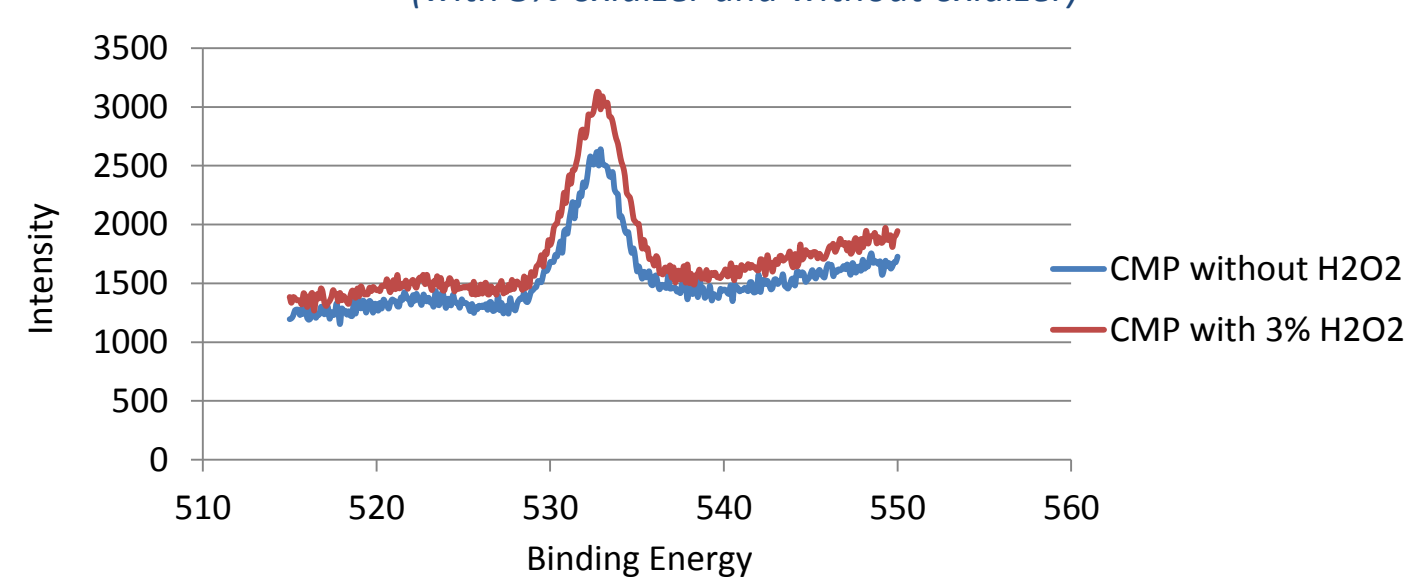


- Surface roughness reduced significantly after CMP and abrasive paper induced microstructure on the Ti samples with CMP.

XRD analysis of Ti samples with 3% oxidizer and without oxidizer



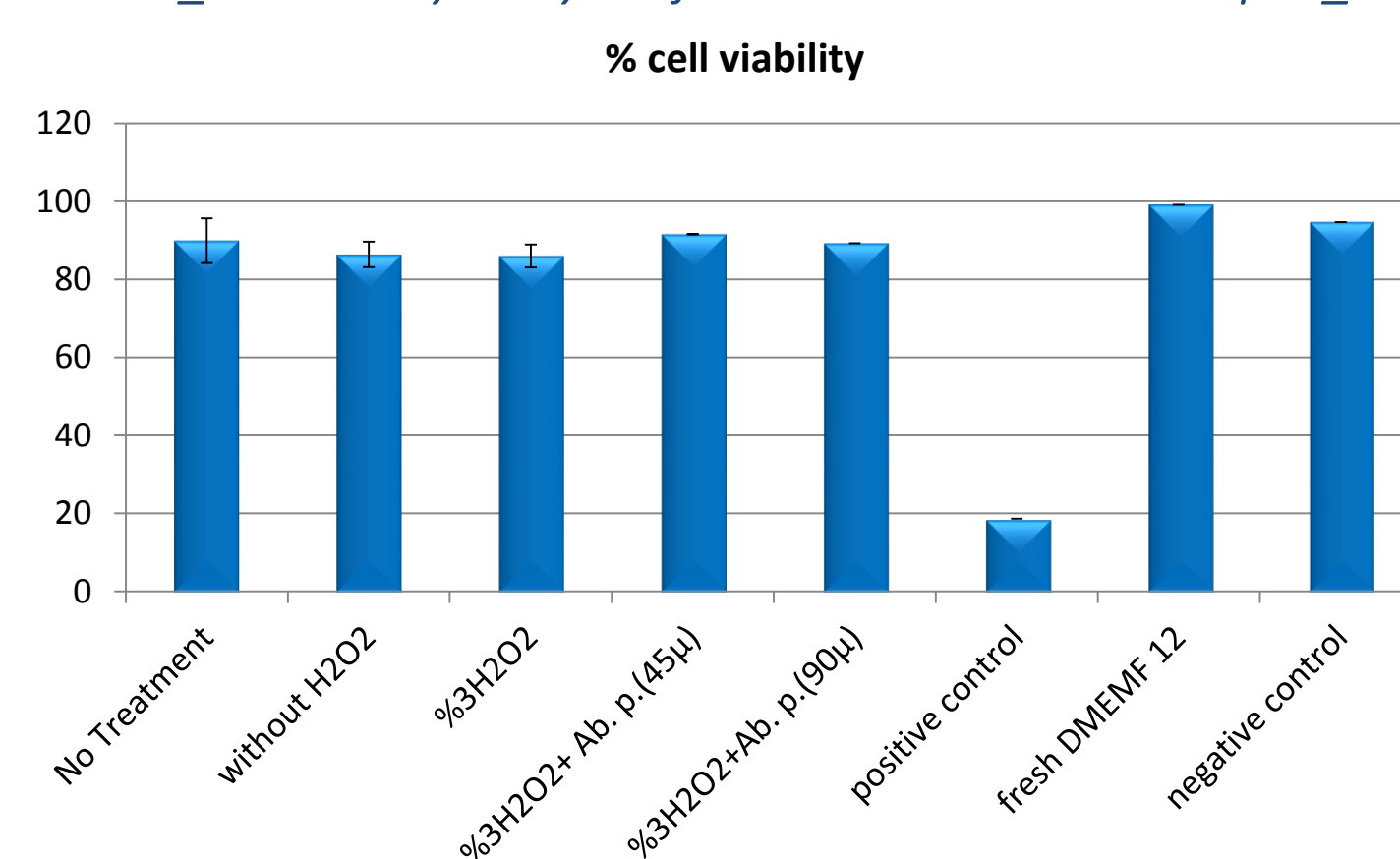
XPS analysis of Ti samples at O1s region
(with 3% oxidizer and without oxidizer)



- CMP without oxidizer exposes titanium surface. Addition of oxidizer forms thin oxide layer on titanium and hence hiding the Ti peaks on XRD.
- XPS analysis of the samples from figure also show different intensities between the oxidized and non-oxidized samples at the 1s region for Oxygen element.

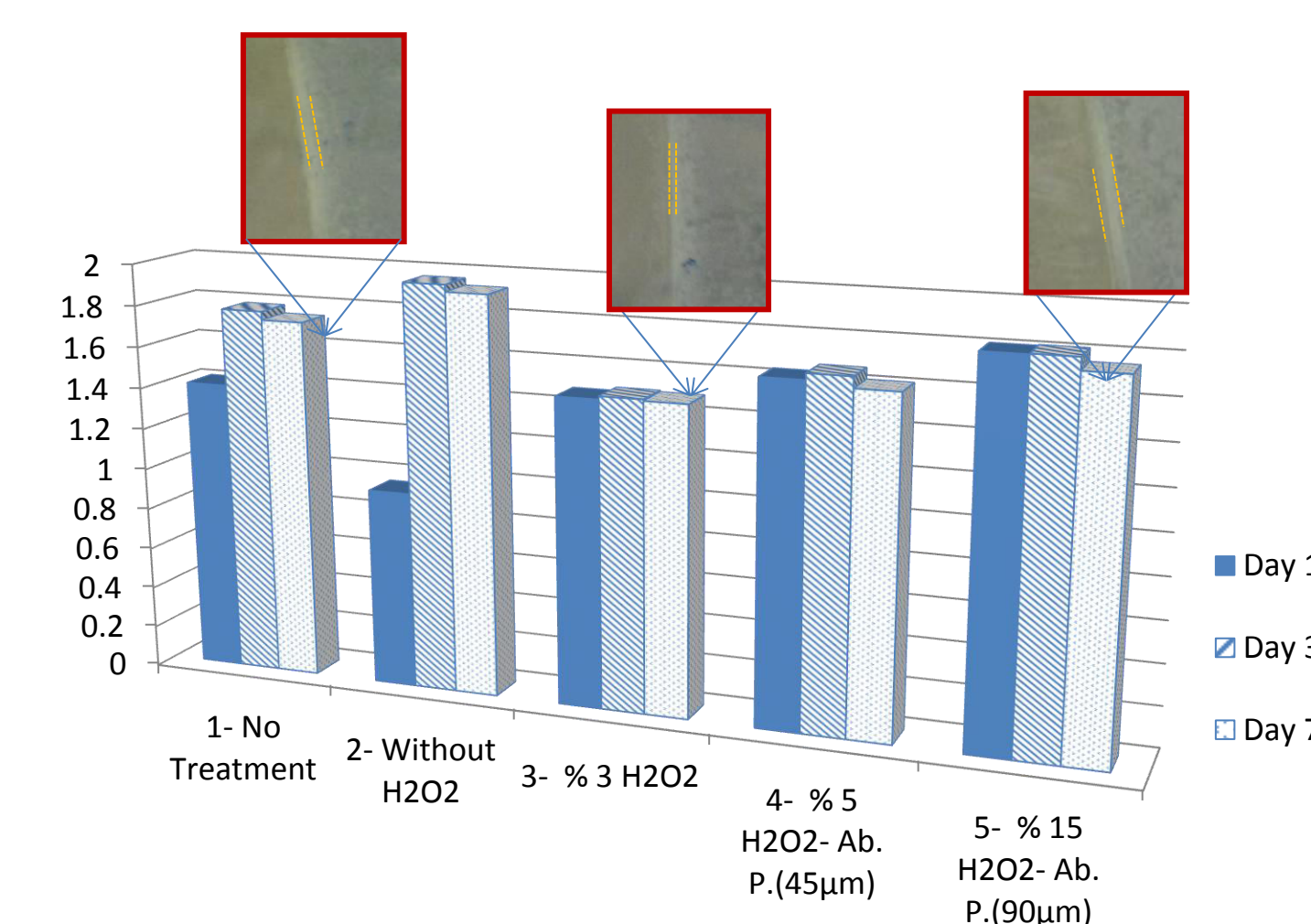
Biological Evaluations

Citotoxicity analysis of the CMP'ed titanium samples



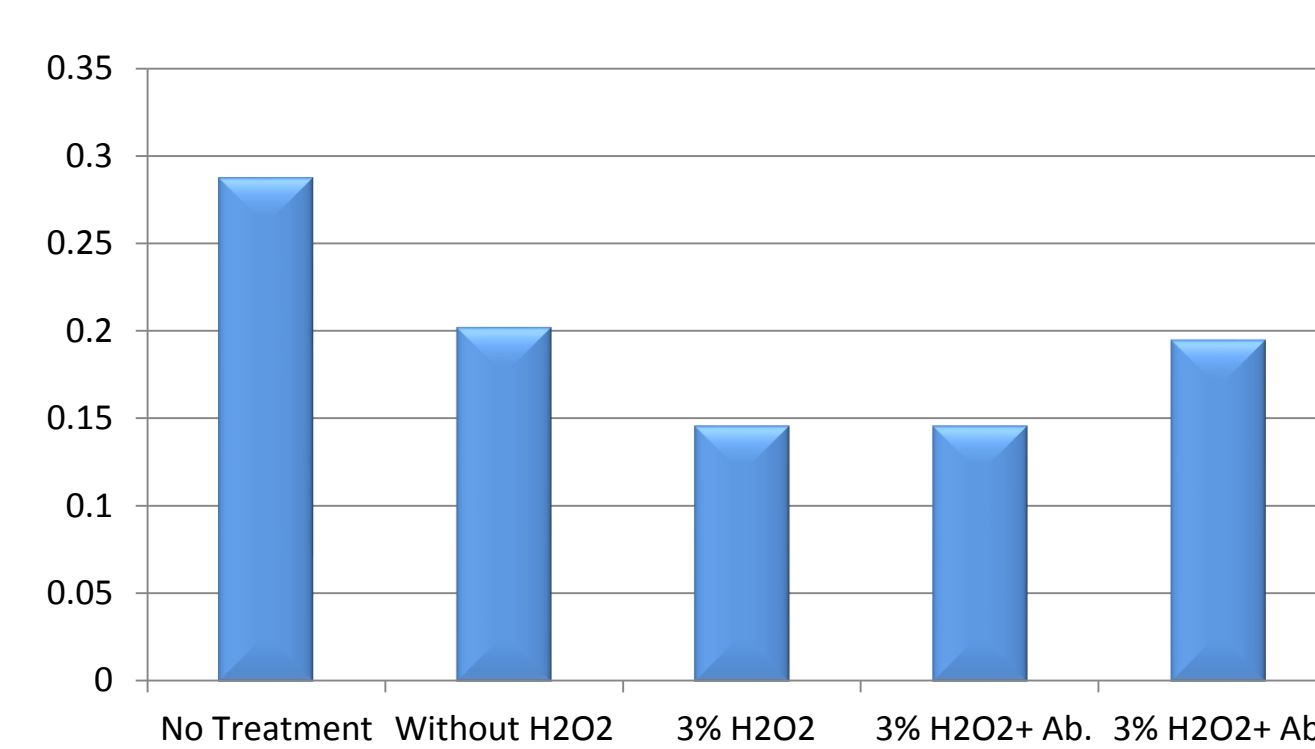
- Citotoxicity analyses conducted on the CMP'ed samples did not show an adverse effect on the titanium surfaces.

Bacteria growth analysis on the titanium surfaces with & w/o CMP



- Bacteria growth tends to increase with increasing roughness but is consistent on the CMP'ed samples.
- Particularly the sample processes through proper CMP process with the smoothest surface resulted in the least amount of bacteria growth around the titanium plate.

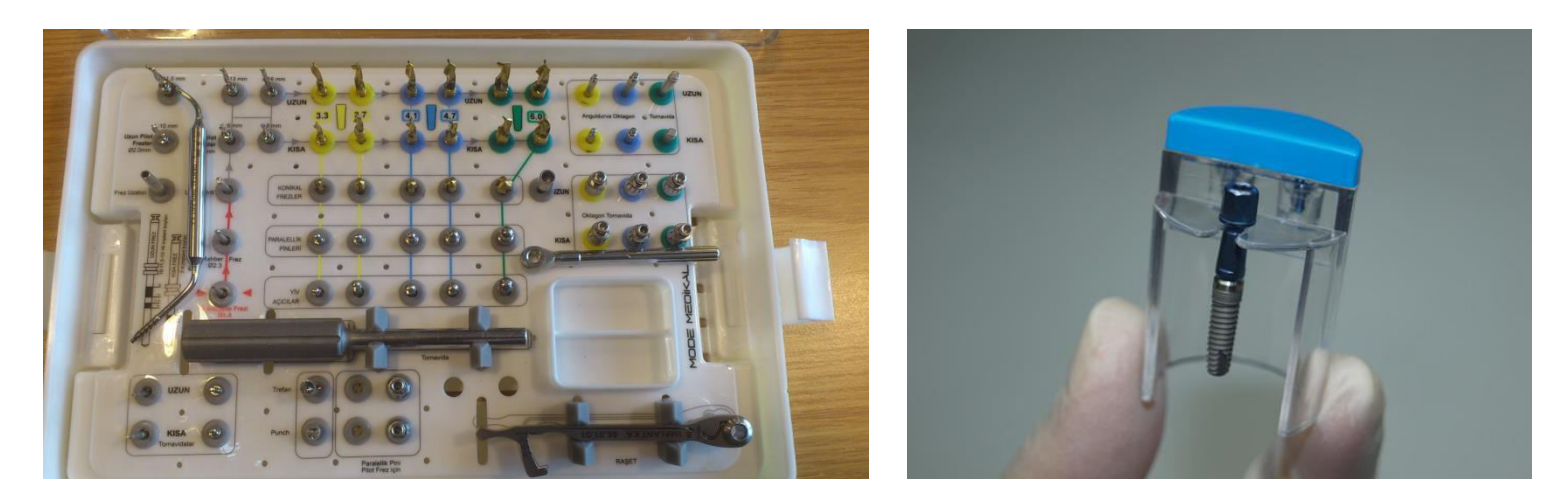
Cell attachment results after five days with L929



- The experimental model used in-vitro studies are the interaction of the L929 type cell with the Ti implant material enabling the prediction of the cell's interaction with the implant in the organism.
- Cells cultured on the standard CMP'ed samples showed less cell growth than the other samples.

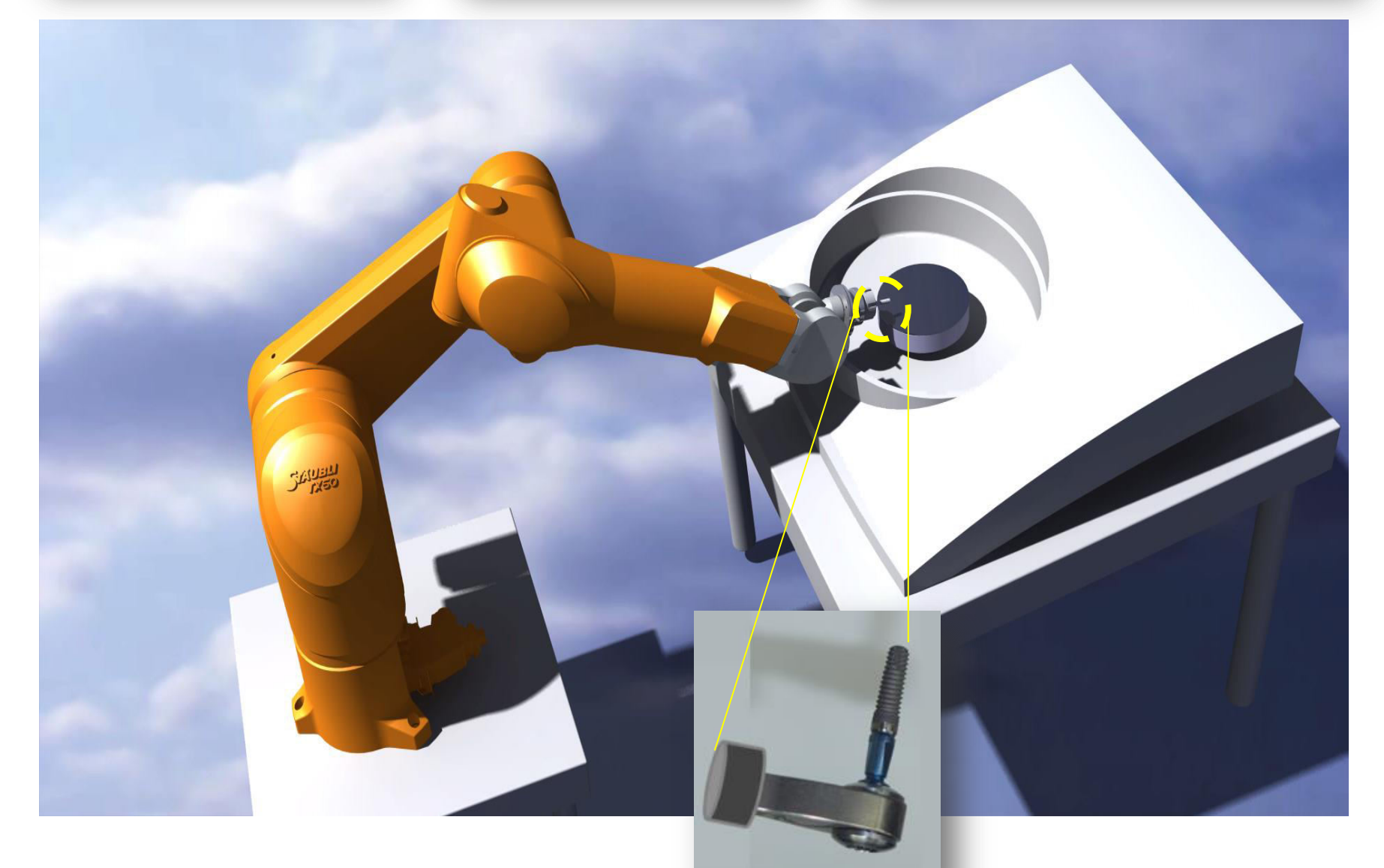
3D CMP Development

Dental implant kits and a sterilized implant sample



- 3-D CMP process needs to be developed for the proper structuring of implants.
- CMP machines are designed to planarize 2D structures.
- In order to achieve CMP of 3D surfaces, the number of degrees of freedom (DoF) in the system needs to be increased.

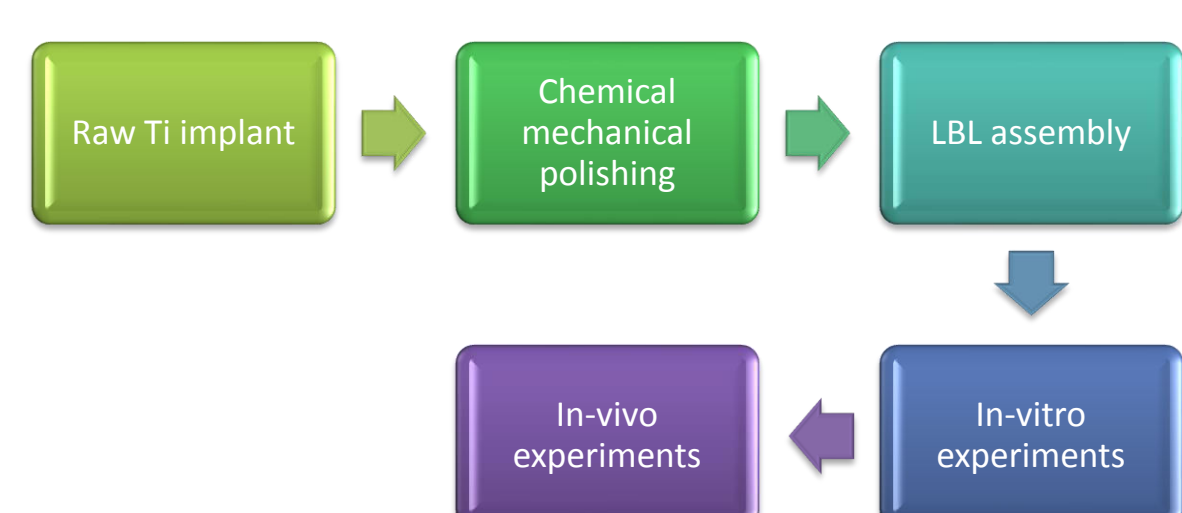
3-D CMP design alternatives with a robotic arm and various polish pads



- One way to accomplish this is to use a 6-DoF Robotic Arm. Robotic Arm is a mechanical arm that has similar functions of a human arm. It consists of joints, which give robotic arm the capability of rotary and linear motion.
- A 6-DoF robot can easily reach any point within its workspace and in any desired orientation.
- A 6-DoF Robotic Arm and standard CMP machine can be used as an alternative. A holder mechanism for dental implant on robotic arm with a force-torque sensor is proposed.
- The pad on CMP is soft and easily gets the shape of the implant. When pad is turned with a constant RPM value, dental implant is polished. Chemicals are fed through the process using the systems on general CMP machine.

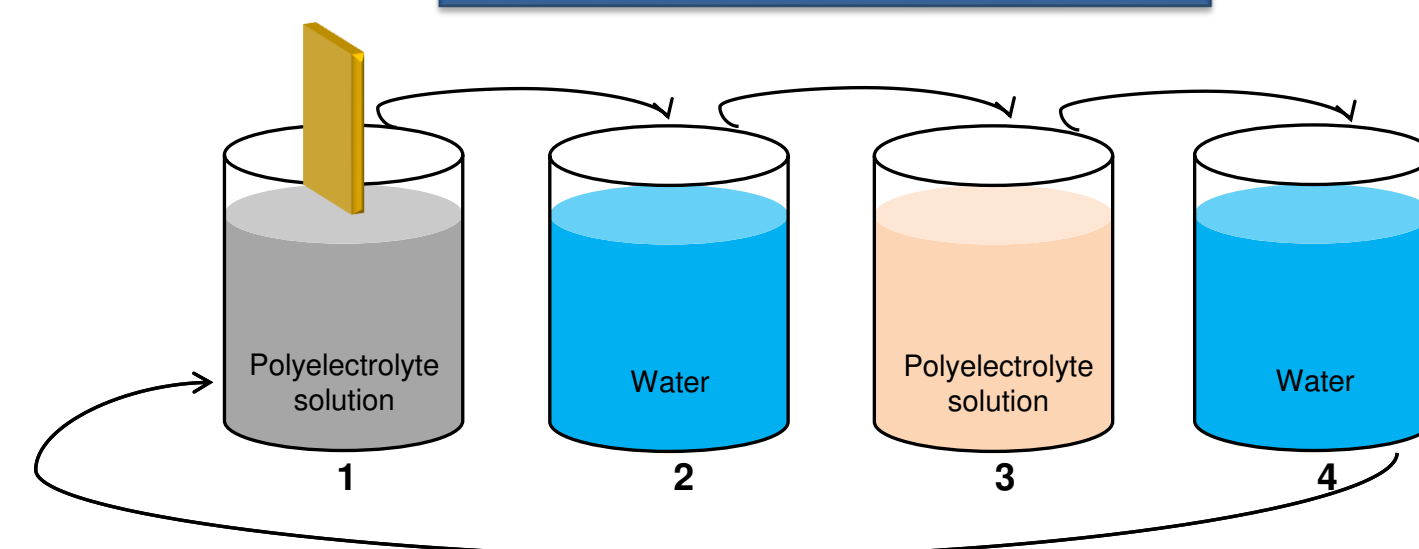
Future Work

Our Approach



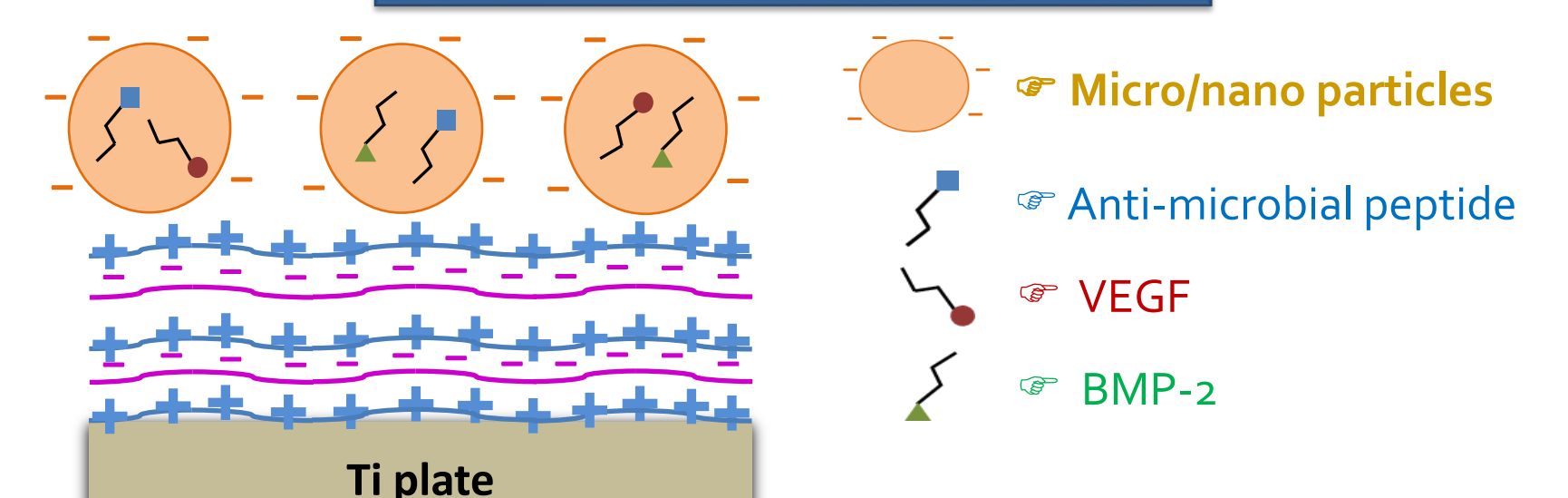
- After CMP process, the surface of Ti implant forms a self protective oxide layer.

LBL Assembly Process



- Following CMP, positively and negatively charged polyelectrolytes will be coated on Ti alternatively via layer-by-layer (LBL) process. Finally, micro/nano particles including bio-molecules will be assembled on the surface.

LBL Assembled Biomolecules



- After the implant is located in the body, the PLGA micro/nano particles dissolve at a certain rate. Consequently, controllable release of bio-molecules can be achieved.

Summary

- CMP process has been shown to be an alternative technique to induce microstructure or smoothness on the titanium surfaces to control surface morphology.
- CMP also results in the growth of a self protective oxide on the implant surface simultaneously with surface alteration. It is suspected to be the reason for the constant bacteria growth on the CMP'ed samples in the presence of an oxidizer.
- 3D CMP design is needed for the implant polishing, as a part of this study a new design with robotic arm is proposed.

References

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