Multi-Scale Surface Modification on Ti-6Al-4V alloy by SPE process

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Statement of Purpose: Development of smaller, but reliable orthopedic implants has gained more attention to overcome obstacles such as limited space of narrow teeth like mandibular incisorsv.[1] However, mechanical strength and fracture toughness of pure Ti limit the possibility to narrow down the current implant designs, maintaining relaible long-term service after implantation. In this context, Ti alloy has become a promising substitute material for high performance Ti implants, but reduced biocompatibility of Ti alloys and cytotoxic metal ion leakage from alloys have been major concerns of alloys as a biomateral .[2] Therefore, in this study, we have introduced the combined SLA (Sand-blasted with large-grit and acid etching)-SPE (Selective plasma etching) surface treatment to Ti-6Al-4V alloy in order to create multi-scale surface roughness on the alloy surface for improved biocompatibility. Moreover, metal ion leakeage is expected to be inhibited by the formed Ta layer on Ti alloy during the SPE process.

Methods: The commercially available non-treated and SLA treated Ti-6Al-4V alloy discs were used for the experiments as references. SPE process was applied on both non-treated and SLA-treated alloy specimens using direct current (DC) magnetron sputter with a Ta target under ambient argon atmosphere at high negative bias of 800 V. Surface morphologies of various Ti alloy surfaces were observed by FE-SEM and Ta ion embedment was assessed by EDS analysis. The *in vitro* cell tests were carried out with pre-osteoblast cell line (MC3T3-E1). Cell attachment was observed by SEM after culturing for 3 hr and cell viability was evaluated by by MTS assay after culturing 5 d (n=3), respectively.

Results: Surface morphologies of Ti alloys via various surface modifications are shown in **Fig. 1**. The SLA-treated surface exhibits microscale roughness while SPE-treated surface is almost flat with nanoscale pores. The combined SLA-SPE process was found to produce the multi-scale roughned surface where the nanoscale pore structure (~ 50 nm in width) was created on the micro-roughned surface (**Fig. 1c, d**). Ta embedment into the SPE-treated surface was verified by EDS analysis (**Fig. 2**). Ta ions were uniformly distributed with the quantity of ~ atom % on the surface. Cell attachment morphologies on various Ti alloy surfaces exhibit that surface topography at nano and microscales gave the attachment sites to cells, where cells were well-spread with larger surface coverage as compared to non-treated surface(**Fig. 3**). Moreover, cells on the multi-scale roughened surface show combinational morphologies, dense cell integrins around the cell boundary with larger cell spreading area. Cell viability test also revealed that all specimens were biocompatible, but the level of cell proliferation on various Ti alloy surfaces wasn't significantly different (data not shown).

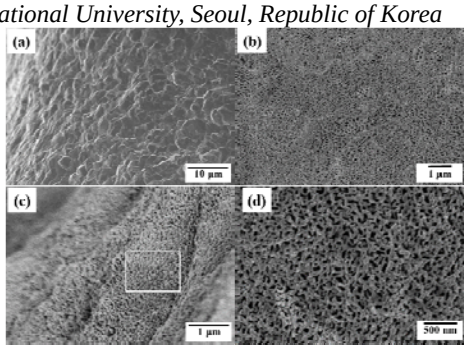

Figure 1. Surface morphologies of various Ti alloy surface: (a) SLA-treated surface, (b) SPE-treated surface and (c, d) SLA-SPE treated surface at different magnifications

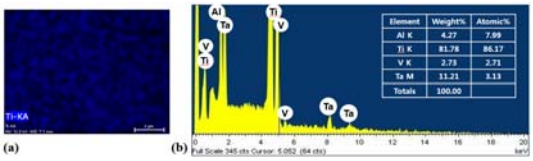

Figure 2. EDS analysis of SPE-treated Ti alloy surface: (a) Ta mapping image, and (b) corresponding ion spectrum.

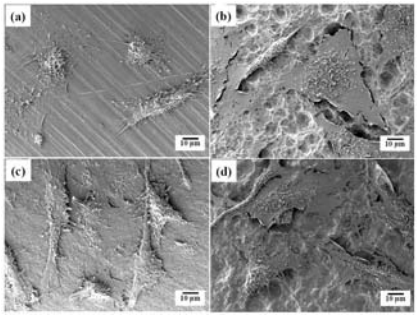

Figure 3. Cell morphologies of various Ti alloy surfaces after culturing for 3 hr: (a) Non-treated, (b) SLA-treated, (c) SPE-treated, (d) SLA/ SPE-treated

Conclusions: Multi-scale surface roughness on the surface of Ti alloys has been successfully created by the combined SLA and SPE process. Ta ions were also detected on the alloy surface via the SPE process, showing the uniform distribution. The short-term cell attachment test clearly shows that cell adhesion on the roughned surface is improved as the roughness is increased, indicating better cell speading with a well-developed integrin structure. Further *in vitro* and *in vivo* tests are expected to reveal the improved bioactivity of multi-scale roughened Ti alloy.

REFERENCES

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