Surface Characterization of Nano-Features Induced by a Low Temperature Oxidation Method

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Statement of Purpose: Titanium (Ti) is a preferred metal for orthopaedic implants due to its good mechanical properties and biological performance. However, fibrous tissue capsule formation around the implant can lead to implant failure, particularly in compromised patients [2]. Thus, better osseointegration between bone and implant is necessary. A recent surface nano-modification method using high temperature oxidation to generate combined nano- and micron surface roughness on Ti has shown improved osteoblast differentiation [3]. However, the temperature required to generate these nanostructures can alter the mechanical properties of the Ti implant [4]. The objective of this study was to characterize surface properties resulting from a new low temperature oxidation treatment to generate nanostructures on clinically-relevant Ti surfaces.

Methods: A low temperature oxidation (LTO) treatment was used to modify the surfaces of titanium (patent pending). Commercially pure titanium (cpTi-2) in the form of smooth (PT) or micro-rough (SLA) disks of 15 mm diameter and 1 mm height were exposed to the low-temperature oxidation treatment. After such modification, the disks were cleaned and characterized using x-ray photoelectron spectroscopy (XPS) for evaluation of surface chemical composition, contact angle analyses for surface wettability, scanning electron microscopy for morphology (SEM), and 3D laser confocal microscopy for micro-scale roughness. Due to the instrument's restricted z-limit, atomic force microscopy (AFM) for evaluating nanoscale roughness could only be performed on PT samples. Morphometric studies from SEM images were used to determine the average nanostructure diameter. Modified surfaces were autoclaved or gamma irradiated to allow for analyses of surface differences resulting from sterilization. All data were compared using the Student's t-test with Bonferroni correction or 1 way ANOVA, with statistical significance noted at $p<0.05$.

Results: The low-temperature oxidation (LTO) induced nanostructures were approximately 20 nanometers in diameter on PT and SLA surfaces. Compared to controls, the carbon content decreased on SLA LTO-modified surfaces, but was not noticeably different on PT surfaces. The contact angle was reduced on all LTO modified surfaces, with the modified SLA surfaces becoming completely hydrophilic (i.e., with a contact angle of less than 10 degrees). A time course study of SLA samples stored in saline revealed no change of the contact angle even after 83 days, while samples stored dry had increased contact angles after day 28. The effect of sterilization also changed characteristics of the surfaces. Autoclave sterilization increased carbon contamination and the contact angle when compared to non-sterilized controls. Carbon contamination was also higher on gamma-irradiated samples, but this was attributed to aging during transport to the sterilization facility, which took up to 50 days, and not to the irradiation itself. Time course studies showed that gamma radiation did not change surface characteristics when samples were stored in saline solution instead of storage in air. Surface micro-roughness of all LTO-modified samples significantly increased relative to non-modified controls.

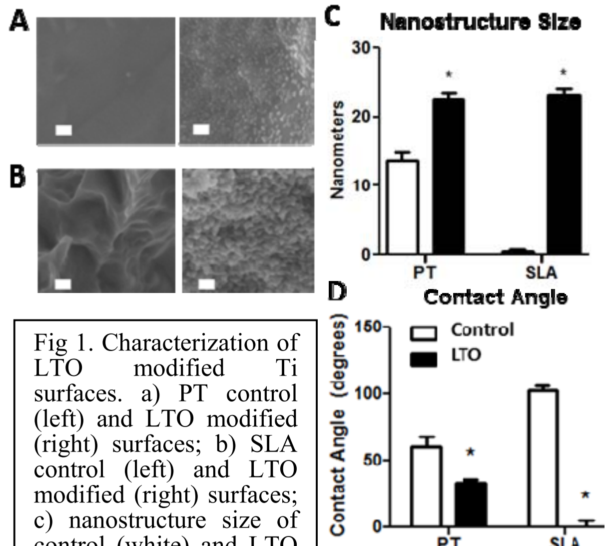

Fig 1. Characterization of LTO modified Ti surfaces. a) PT control (left) and LTO modified (right) surfaces; b) SLA control (left) and LTO modified (right) surfaces; c) nanostructure size of control (white) and LTO (black) surfaces, d) contact angle of control (white) and LTO (black) surfaces. Unpaired t-test, $p<0.05$. *v. control. Scale bars = 100 µm.

Conclusions: A novel LTO surface treatment was used to create nanostructures, increase wettability, and decrease carbon levels on clinically relevant Ti disks. Sterilization has been shown to affect surface chemistry and wettability on microstructured Ti [5], but the effects on nanostructured Ti are unknown. Gamma irradiation of LTO surfaces stored in saline preserved a low contact angle, but dry storage of disks altered surface properties. The combination of nano- and micro-surface roughness has been shown to increase osteoblast differentiation and inhibit cell proliferation. These data explore in depth the properties of nanostructured surfaces that may improve osseointegration of implants, and the storage and sterilization methods that preserve these properties. Further work will investigate cell responses to these topographies.

References:

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