Novel Surface Modification of Titanium Implants in Hydrofluoric Acid:

Surface Characterisation and in Vivo Performance

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Introduction: Titanium is currently known as one of the most successful biomaterial for dental implants. Etching implant surface with a concentrated acid has already shown good results in term of osseointegration[1]. A method has been established using an ionic solution with low pH and a stable current to change the topography and chemistry of the titanium implant surface. The aim of this study was to measure the effect of the hydrofluoric acid (HF) at different concentrations on commercially pure (cp) titanium polished surfaces. Advanced surface analysis were performed followed by an *in vivo* study according to the animal model previously described[2].

Materials and Methods: Cp titanium coins were grinded and polished (Phoenix 4000, Buehler GmbH, Düsseldorf, Germany). The surface modification set-up consisted of two power supply (Protek Dual DC power, Korea) and (Xantrex XDL 56-4P, Burnaby, Canada), a data logger (NI DAQPad, National Instruments, Asker, Norway) and a magnetic stirrer with heating control (IKA-RET Control Visc C, VWR, Kaldbakken, Norway). Six coins at the time were attached to the electrode and submerged in 1L of 1M sodium chloride (NaCl) with 0.2M hydrochloric acid (HCl), pH 2, and three different concentrations of hydrofluoric acid (HF): 0.001 vol. %, 0.01 vol. % and 0.1 vol. % for 30 min., while a current of 1 mA/cm^2 was applied. The control batch was treated for 30 min. with the same solution but without any HF. The weight of each coin was measured before and after the procedure (Mettler AT261 Delta Range, 10^{-4}g of precision). The topography of the modified surfaces was measured using: an Atomic Force Microscope (AFM Asylum research, Santa Barbara U.S., in aqueous contact mode with an AC160 Olympus cantilever), a profilometer (Sensofar plμ2300, Spain) and a SEM (Philips XL 30 ESEM, FEI Electron Optics, Eindhoven, Netherlands). After the topographical characterization of the different groups of implants, an *in vivo* experiment for the in situ measurement of attachment strength of the implants to cortical bone was performed. Evaluation of the implants using the tensile test was completed 4 weeks after the surgery.

Results: As suspected, the 3 HF concentrations modified the surface properties: shaping distinctive topographies (Fig 1) with an increasing roughness, as well as implementing fluoride, oxide and hydride ions into the titanium. The tensile strength values of these modified implants obtained from the in vivo study were 0.66 ± 0.40N, 2.21 ± 0.9 N, 6.89 ± 2.1 and 2.19 ± 0.55 N respectively for the control and the treatments with 0.001, 0.01 and 0.1 vol. % HF. There was a significant difference ($p<0.05$) between the control and the two highest HF concentrations, and a high significance ($p<0.001$) between the control and the 0.01 % HF treated group (Fig. 2). Strong correlation could be calculated between: skewness (S_{sk}), kurtosis (S_{ku}), fluoride and the pull-out results after implantation. No correlation was found when comparing the average height deviation from the mean plan (S_a) and bone-to-implant strength.

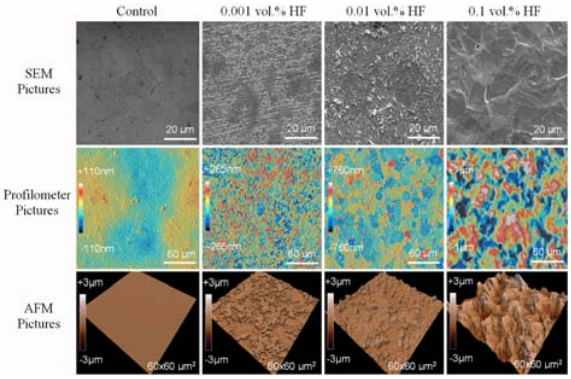

Fig. 1: Images of the treated surfaces

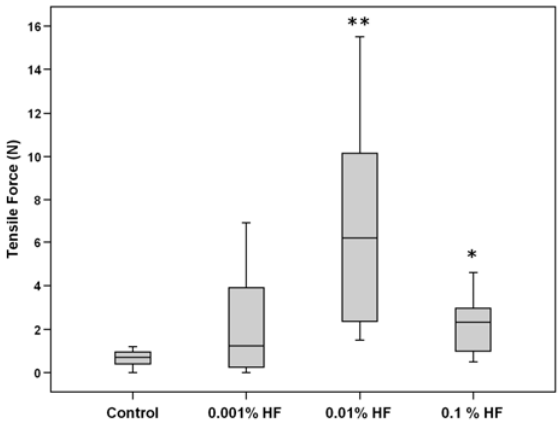

Fig. 2: Pull-out strength versus HF concentration

Conclusion: A novel technique to create particular surface topography with specific ion implementation has been established. Geometrical parameters such as S_{sk}, S_{ku} and S_{ci} have been proved to be strongly correlated to bone attachment. Implementation of fluoride ions into the titanium surface gave positive *in vivo* achievements. In addition it has been shown that regular implant surface roughness parameters such as S_a, S_q and S_t did not alone predict clinical success.

References:

1. Ellingsen JE et. al., Int. J Oral Maxillofac Implants, 2004:19:5: 659-66
2. Rønold HJ et. al., Biomaterials, 2002: 23: 2201-09