Strontium and Magnesium Oxide Doped Nanoscale Hydroxyapatite coatings on Titanium using rf Induction Plasma Spray for load bearing implants

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Introduction: Though hydroxyapatite (HA) coated implants are commercially available, its acceptance is still not wide spread due to challenges related to weak interfacial bonding between metal and ceramic phases, and low crystallinity of HA [1,2]. Moreover, developments in osteoconductivity and crystallinity of HA coatings are necessary to improve healing time and long term stability. The ***objective*** of this research is to test our ***hypothesis***, which is chemistry and microstructure in induction plasma sprayed HA coating on Ti can both (1) improve the interfacial strength and (2) promote intimate implant - tissue integration while improving bone remodeling around the implant. The ***rationale*** is that the incorporation of different metal ions in the HA coating will make the coating chemistry analogous to natural bone, and thereby improve the stability of the coating *in vivo*. In the present work, Sr and Mg doped HA coatings were prepared using a 30 kW inductively coupled radio frequency (RF) induction plasma spray equipped with supersonic plasma nozzle and *in vitro* biocompatibility along with mechanical properties were evaluated.

Methods: The optimized parameters of induction plasma spray were selected from previous optimization study and used to prepare the HA coating on Ti metal [3]. The coating was prepared at 25 kW plate power and 110 mm working distance. Initially, HA powder was doped with 1 wt% SrO (Sr-HA) and 1 wt% MgO (Mg-HA), calcined at 800 °C for 6h and then used for coating preparation. The coatings were cross sectioned, polished, etched and observed in FESEM to reveal the coating microstructure. The bond strength of the as sprayed HA coatings was evaluated using a standard tensile adhesion test (ASTM C633) set up, in which 5 replicates were used. *In vivo* performances of the HA coatings were assessed using a cortical defect model in rat femur. *In vitro* biocompatibility study using human fetal osteoblast cell line (hFOB) was used to evaluate the effect of metal ion doping on cell-material interactions and compared to that of HA control sample. The cell proliferation was measured using MTT assay.

Results: Figure 1 shows the XRD patterns of HA, Sr-HA, and Mg-HA coatings. HA (JCPDS # 09-0432) was the major phase in all the coatings along with minor amounts of α-TCP (JCPDS # 09-0348) and β-TCP (JCPDS # 09-0169). Neither Calcium oxide (CaO) nor TTCP phases were found in the XRD pattern of any of the coatings. The sharp and distinct HA peaks indicated that the coatings were highly crystalline in nature, with minimum phase decomposition. Microstructural analysis indicated coherency in the structure with an average grain size of 15-30 nm. Average adhesive bond strength of 17 MPa ensured sufficient mechanical strength of the coatings. The cell proliferation was determined by MTT assay and is shown in Figure 2. Cell proliferation was evident at all culture days. Significantly higher cell density was observed on Sr-HA compared to HA coatings at day 3 and 11. Presence of Sr in the coating also stimulated hFOB cell differentiation and alkaline phosphatase (ALP) expression when compared to either HA or Mg-HA coatings. Histological evaluation at the bone-implant interface was performed at 2 weeks to evaluate the biocompatibility and new bone formation which is shown in Figure 3. *In vivo* studies indicated osteoid formation on the HA coated implant surface after 2 weeks of implantation. Osteoid formation was scarce on the surface of the control uncoated Ti implant surface.

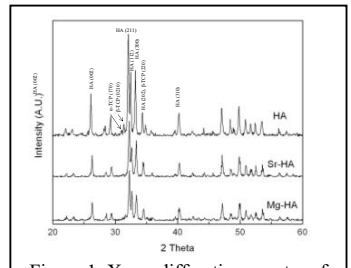

Figure 1. X-ray diffraction spectra of HA, Sr-HA and Mg-HA coatings.

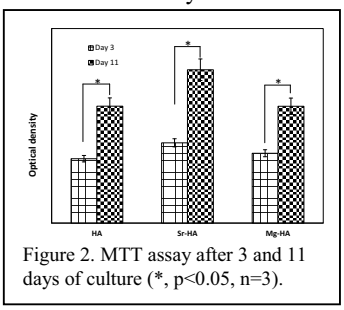

Figure 2. MTT assay after 3 and 11 days of culture (*, $p<0.05$, n=3).

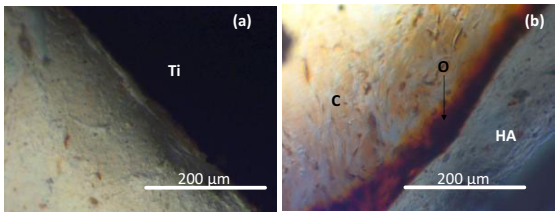

Figure3. Optical photomicrograph of a longitudinal section of (a) Uncoated Ti and (b) HA coated Ti, implanted in rat femur for 2 weeks showing (O) nonmineralized osteoid, and (C) collagen matrix (Goldner's Masson Trichrome stain).

Conclusions: Doped nano crystalline HA coating on Ti processed with rf induction plasma supersonic nozzle can produce coating with better interfacial mechanical property. They also show excellent osteointegration *in vivo*. Authors acknowledge financial support from National Institute of Health and W. M. Keck Foundation.

References: 1. Roy M. *Acta Biomaterialia* DOI: 10.1016/j.actbio.2010.09.016 (2010); **2.** Sun L. *J. Biomed. Mater. Res.* 62 (2002) 228-236; **3**. Roy M. *Surf. Coat. Technol.*, 2010, DOI:10.1016/j.surfcoat.2010.10.042.