

Bioceramic Coatings Obtained by Physical and Chemical Techniques

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INTRODUCTION: Hydroxyapatite-based ceramics (HA: $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) are largely applied as biocompatible coatings on metallic components of prostheses for inducing osteoblasts apposition and subsequent re-growth. Nevertheless, the ceramics-metal interfaces are often the seat of residual stresses with amplitude primarily depending on the deposition technique and the coating conditions. HA layers are grown by pulsed laser deposition on either chemically-etched or mechanically-polished G4 titanium samples. Usually the layers are deposited in the presence of water vapours or in oxygen with partial pressures of several tens of Pa. In our first investigations, depositions were carried out in a vacuum of about 10^{-4} Pa in order to preserve the memory of the physicochemical state of the expelled material from the target under the laser beam impact. Thin films of HA were also produced by spin-coating sol-gel solution on chemically-etched Ti substrate. Calcium nitrate and triethyl phosphite were then used as precursors. We investigated the morphology, the structure and the composition of the deposited material by various techniques.

METHODS: All depositions have been performed with UV pulses generated by a KrF* laser source ($\lambda = 248$ nm and $\tau = 7$ ns), following the PLD technique. After deposition, the samples were allowed to cool down to ambient temperature inside the irradiation chamber. Some of the samples were heat treated at 400°C for 6 hours in an atmosphere enriched in water vapours in order to improve the HA crystallinity status and to restore the loss of OH groups from the HA molecule. These samples are further noted #1 and #2. The obtained sol-gel (#3) was spin-coated onto a chemically-etched Ti disk at a speed of 2000 rpm for 5 s in ambient atmosphere. The films were dried at 80°C for 30 min. Five consecutive depositions were performed according to this protocol prior to a final heat treatment to 500°C for 30 min in air.

RESULTS: Coatings have been analyzed using grazing incidence X-ray diffraction in order to get in-depth phase distribution. Depth penetration of Cu-K $_{\alpha}$ X-ray depends on the incidence angle chosen between 0.5° and 3° .

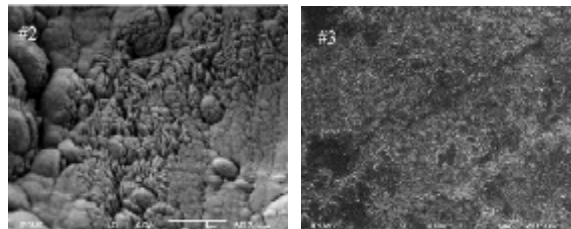


Fig. 2. SEM image of sample #1 and #3

DISCUSSION & CONCLUSIONS: PLD films exhibit an irregular morphology: it presents nanometric aggregates of different sizes (< 100 nm) and crystalline orientations, as well as droplets of different micrometer sizes ($0.1\text{--}3$ μm). Such particulates have been previously observed on the surface and in the bulk of HA PLD films. The HA films of the sol-gel sample shows a porous and coral-like structure. PLD coatings present nanometric particles and micrometric droplets. Post treatment in water vapour involves a recrystallization of the HA coating. The sample elaborated by sol-gel process shows a porous and coral-like structure. In both cases, the HA layers are well crystallised but the PLD deposited HA layer is expected to be more susceptible to the natural remodelling processes when it is implanted in a living body due to its better adhesion on titanium.

REFERENCES: ¹M. Iliescu, V. Nelea, J. Werckmann, I.N. Mihailescu (2004) *Surf. Coat. Technol.* 187, pp 131-140, ²H-W. Kim, J.C. Knowles, V. Salih, H-E. Kim (2004) *J. of Biom., Mater. Research Part B: Applied Biomaterials*, 71B, pp 66 - 76.

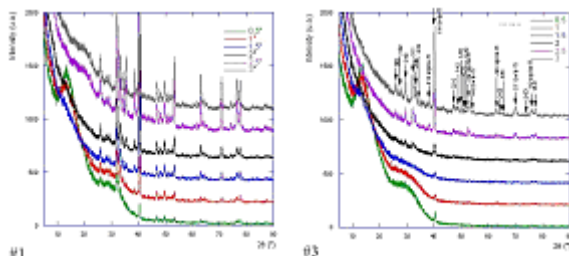


Fig. 1: X-ray diffraction phase analyses for samples #1 and #3