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Effect of sintering temperature on the crystallinity and crystallite size of hydroxyapatite-reduced graphene oxide bioactive coatings

Lara, N.(1); Komorizono, A.A.(1); Bernardi, M.I.B.(1);
(1) IFSC-USP;

Hydroxyapatite (HA) is the most promising calcium phosphate-based biomaterial for applications on hard tissue engineering and coatings for cementless bone implants, due to its composition and structure similar to the mineral phase of bone. In addition to having excellent biocompatibility, bioactivity and osteoconductivity, it can be doped with antimicrobial nanoparticles that can prevent infections in the implanted region. To evaluate the effect of sintering temperature on the crystallinity and crystallite size of HA coatings doped with antibacterial reduced graphene oxide (rGO) powders, commercially pure titanium (Ti-cp) plates were cut to 2 x 15 x 15 mm, polished with 400-grit metallographic sandpaper, ultrasonic cleaned in acetone, isopropyl alcohol and distilled water and dried at room temperature. The plates were activated by acidic treatment in 70% nitric acid solution for 3 minutes, to improve the coatings adhesion. A suspension (0.64 g/ml) of powdered pure HA (Alfa Aesar, USA) in ethylene glycol was heated for 30 min at 120 °C, cooled to room temperature, doped with rGO nanopowders in proportions of 0, 1 and 2% by mass and homogenized by ultrasound. The deposition on the Ti-cp samples was made by the spin coating process in 2 layers, at 2000 rpm, with a pre-sintering at 100 °C for 10 minutes between each spin. The sintering process was done in a muffle furnace at ambient atmosphere for 1 hour, with a heating speed of 3 °C/min, at four different temperatures: 600, 700, 800 and 900 °C. Structural characterization by X-ray diffraction (XRD) was performed using an Ultima IV diffractometer (Rigaku, Japan) with Cu-K(alpha) radiation (wavelength = 1.5418 Å) and LiF monochromator (100) in the conventional theta-2theta configuration. The coatings degree of crystallinity increased with the sintering temperature, ranging from around 85% at 600 °C to 94% at 900 °C for all compositions. The average crystallite size also increased with temperature, ranging from 50 to 150 nm. The rGO content did not affect crystallinity, but the crystallite size was up to 20% smaller with the addition of 2% rGO, due to the effect of heterogeneous nucleation. These results show that, in addition to the antimicrobial effect and the increase in strength by composite reinforcement, rGO allows the refinement of HA grains without affecting its crystallinity.