Bioactive layers grown by MAO on nitrogen plasma treated titanium with improved tribo-mechanical features

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Abstract

The micro-arc oxidation (MAO) is a widely investigated method to become titanium surfaces bioactive. The obtained product are rough and porous oxide layers with few micrometers in thickness, which can also contain other suitable elements for osseointegration such calcium and phosphorous. The implant's mechanical compatibility with bone is also improved because of the matching elastic moduli, which prevents bone resorption. However, a previous research showed that the adhesion between layer and substrate is inadequate for use in bone implants, detaching with tangential loads as low as 20 gf. The previous treatment of Ti surfaces by ion nitriding can eventually enhance adhesion of such bioactive layers by increasing the load bearing capacity of the substrate. We selected c.p. Ti samples which were plasma nitrided by glow discharge in different conditions of temperature (673 K and 873 K) and time (1 h and 3 h), with known structural and tribo-mechanical conditions. The MAO process was performed using Ca and P-based electrolyte in galvanostatic mode with current density 150 mA/cm2. The surfaces were characterized by grazing incidence X-ray diffraction (GIXRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), Fourier-transform infrared spectroscopy (FTIR), instrumented indentation and nanoscratch tests. Analytical methods were employed to calculate the effective layer's mechanical properties, that is, subtracting the roughness effects and the substrate contribution on the measured values. In this work, by reference sample we mean the one only oxidized by MAO. Porous titania layers were identified on the reference and the previously nitrided samples, which thickness decreased as the nitriding conditions became more intense (from 673K/1h to 873K/3h). The coexistence of the crystalline phase TiO2 anatase, produced by MAO, and nitride phases [Ti2N and solid solutionTi(N)] were verified in all conditions. Also identified in all samples were smaller amounts of calcium and phosphorous with Ca/P _˜1.6. On surfaces previously nitrided in 673 K, the indentation imprints presented less or none lateral cracks than the reference sample; elastic modulus were 38 GPa, similar to the reference; and the critical loads for scratch tests were 40% higher than reference. On surfaces previously nitrided in 873 K, the thin anodic layers presented hardness and elastic modulus profiles lower than the reference ones, and these layers produced a third-body interaction during scratching. The results are indicative that ion nitriding and MAO can be synergically applied on Ti surfaces for implant designs with proper bioactive and tribo-mechanical properties.

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