Structural characterization of mesoporous titania containing silver nanoparticle inclusions for antibacterial implant applications

T. Thersleff1, K. Grandfield1, W. Xia1, K. Welch1, H. Engqvist1

1. Department of Engineering, Uppsala University, Uppsala, SE

thomas.thersleff@angstrom.uu.se

Mesoporous titania, drug delivery, biomedical interfaces

Titanium dioxide is a well-known biomaterial, ideal for use in implant coatings due to its biocompatibility and surface topography. It can also be fabricated with a mesoporous structure, exhibiting a pore size ranging between 5 – 50 nm in diameter [1]. This enables the coating to act as a reservoir for nanoparticles and drugs, while not significantly compromising its mechanical properties [2,3]. Recently, it was demonstrated that mesoporous titania can be used as a novel surface drug-delivery system when loaded with the antibiotic Cephalothin [4].

In this study, the ability of this mesoporous matrix to hold silver nanoparticles is investigated. Such a nanocomposite structure may be capable of acting as an antimicrobial surface, combining the antibacterial effects of ultraviolet irradiated titania with those of silver ions. The success of such nanocomposites depends, to a large extent, on the spatial distribution of silver nanoparticles within the mesoporous titania matrix. This study is designed to make a detailed assessment of the nanoparticle distribution and structure-property relationship of silver nanoparticle-doped mesoporous titania coatings.

Mesoporous titania films approximately 100 nm in thickness were deposited on bulk titanium using an evaporation induced self-assembly method [4]. Silver nanoparticles were introduced into the mesoporous coatings by soaking in a silver nitrate solution, followed by treatment with UV light (254 nm) to decompose the nitrate into silver nanoparticles or silver oxides. All coatings were initially characterized using x-ray diffraction in the Bragg-Brentano and grazing incidence geometries. The surface topology of the films was assessed in a Carl Zeiss 1550 scanning electron microscope operated at 3 kV in secondary electron mode. One doped and one undoped specimen were then prepared for structural and chemical analysis in a transmission electron microscope using the focused ion beam in-situ lift-out method followed by a low-voltage polish (FEI Strata DB235). The structure of the films was locally assessed using electron diffraction and high resolution imaging with both parallel and convergent beams and related to the x-ray data (JEOL 2000 FXII and FEI Tecnai F30 ST). The chemical distribution was investigated by generating real-space elemental maps using energy dispersive x-ray and electron spectroscopic imaging. The ability for Ag nanoparticles to penetrate these pores is further documented through the use of a 3D reconstruction using STEM Z-contrast electron tomography.

Figure 1 shows an SEM image of the undoped mesoporous titania surface, revealing an average pore diameter of 5.44 ± 1.30 nm and an average “roundness” of 1.31 ± 0.21, where “roundness” is determined by the P2A measurement:

\[ P2A = \frac{\text{Perimeter}^2}{4\pi \text{Area}} \]

Figure 2 shows a bright field overview of the doped mesoporous titania structure after preparation in the focused ion beam. Pores are visible in this viewing mode and an intermediate layer between the mesoporous titania and the polycrystalline titanium substrate is observed.

References


The authors are grateful for the support from BIOMATCELL.
Figure 1. Secondary electron micrograph of the surface of undoped mesoporous titania captured in a scanning electron microscope. The nanoporous surface structure can be clearly delineated.

Figure 2. Bright field TEM overview micrograph showing the mesoporous titania structure in cross-section. The image was acquired by illuminating the sample with a parallel beam and selecting the transmitted beam with a small objective aperture. An intermediate buffer layer appears between the titanium substrate and the mesoporous titania.