PROGRESS REPORT FOR AINGRA07009P

PROJECT TITLE
Characterisation and analysis of sol-gel derived hydroxyapatite and zirconia nano-coating interfaces with anodised titanium substrates

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INSTITUTION AND DEPARTMENT
Chemistry, Materials and Forensic Science, University of Technology Sydney

SCIENTIFIC OBJECTIVES
The major objective of the research was the investigation of the interface interactions of zirconia and hydroxyapatite nanocoatings on titanium substrates.

PROGRESS REPORT and RESEARCH OUTCOMES

Zirconia sol-gel-derived ceramic coatings have a variety of uses, due to their ease of production and ability to coat complex shapes. The sol-gel’s nanocrystalline grain structure results in improved mechanical properties of the zirconia coating, which further aids their use in a variety of applications from thermal barrier coating to improved tribological properties on titanium substrates. Stabilised zirconia thin films were spin coated on anodised titanium substrates. The titanium was anodised in a dilute H₃PO₄/H₂SO₄ solution before spin coating with the zirconia sol gel. These films were then studied using secondary ion mass spectrometry (SIMS), to depth profile the elemental species through to the titanium substrate. In conjunction, scanning electron microscopy (SEM) and X-ray mapping were used to examine the craters formed by SIMS to gain an understanding of the diffusion gradient existing with the anodised titanium substrate and zirconia thin film.

The titanium alloy samples were anodized in a dilute phosphoric and sulphuric solution. The samples were anodized at 75V for 30 minutes based on an earlier work. Photo catalysis treatment consists of the treatment of the anatase layer with a UV wavelength of approximately 380nm for 1 hour. Exposing the catalyst to UV generates an excited state on the surface, which is able to initiate subsequent processes like redox reactions and molecular transformations.

These samples were then coated with alkoxide-derived zirconia. These coatings were applied by sol-gel spin coating methods, using techniques and protocols developed in an earlier work and were examined with Secondary ion mass spectroscopy. The samples were then imaged with a LEO SUPRA55VP, scanning electron microscope (SEM). SIMS measurements were performed using a CAMECA IMS 5f secondary ion mass spectrometer. A Cs⁺ primary ion beam was used for depth profiling by rastering an area of 250 x 250µm² on the surface of the sample with a net impact energy of 3keV. To eliminate any edge effects the actual analysis area was dictated by aperture settings which restricted the measurement of positive secondary ions to a 100µm diameter circular area within the rastered region. The measurement of MCs⁺ molecular secondary ions, where M denotes the element of interest, was used for all samples as it is now well accepted that the MCs⁺ technique can greatly reduce the contribution of matrix effects on the SIMS results. The depth of the SIMS craters were measured with a Alpha-step stylus profilometer to determine the average sputter rate of the analysis.
The zirconia coatings on both the anodised and Ti6Al4V samples demonstrated good adhesion and were successfully applied by the sol gel technique. The SIMS results also showed the zirconia diffused a significant degree into the anodised TiO$_2$ layer. Further work into the interface needs to be undertaken to see if a diffusion gradient can be x-ray mapped to further validate the SIMS results.

**Figure 1:** Ti6Al4V Alloy with zirconia coating showing SIMS sputtered area at low magnification.

**Figure 2:** Anodised Ti6Al4V alloy with zirconia coating showing floor of SIMS sputtered area at high magnification.

**Figure 3:** SIMS depth profile of anodised Ti6Al4V sample (75V) showing the elements existing in this sample through to a depth of 500nm.
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**PUBLICATIONS / REPORTS arising as a result of your work.**

Roest R; Atanacio A J; Latella B A; Wuhrer R; Ben-Nissan B: An investigation of sol gel coated zirconia thin films on anodised titanium substrate by secondary ion mass spectrometry and scanning electron microscopy. Materials Forum Vol 1 2007 160-163

**PhD STUDENTS**

Mr Richard Roest will be submitting his thesis in early June 2008.