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TEM and STEM analysis on heat-treated and in vitro plasma-sprayed hydroxyapatite/Ti-6Al-4V composite coatings

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Abstract

A cogent understanding of the microstructure, and indeed nano-structure, of hydroxyapatite (HA) and the interface between Ti-6Al-4V and HA is crucial to its appropriateness as a biomaterials. This paper reports the analysis of plasma-sprayed HA/Ti-6Al-4V composites by transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) to elucidate the intricate nature of the materials following plasma spray processing and in vitro evaluation. The novel Ti-6Al-4V/HA composite coating, with approximately 48 wt% HA, had demonstrated attractive tensile adhesion strength ($\sim$28 MPa) and improved Young's modulus ($\sim$55 GPa). Experimental results demonstrated that amorphous calcium phosphate and fine HA grains were formed during rapid splat solidification in the as-sprayed composite coatings. Small Ti-6Al-4V grains were observed adjacent to the amorphous calcium phosphate. The coatings were further heat treated at 600°C for 6 h, and significant crystallisation of the amorphous calcium phosphate phase took place. However, complete crystallisation was not achieved at this temperature, as the coatings invariably contained a small amount of amorphous calcium phosphate phase in some local regions. After immersion in simulated body fluid for 2 weeks and 10 weeks, TEM and STEM confirmed that the interfaces inside the coating maintained good microstructural integrity.

Keywords: Hydroxyapatite; Ti-6Al-4V alloy; Composite; Plasma spraying; Transmission electron microscopy

1. Introduction

Hydroxyapatite (HA), with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, is one of the most attractive materials for human hard tissue implants because of its close resemblance to chemical composition (Ca/P ratio) of teeth and bones. This promotes rapid bone growth and bonding between bony tissue and implant surfaces. HA has been clinically applied as a dense, sintered material and as coatings on bioinert metallic implant [1]. As a coating on stems of hip joint prostheses, HA can enhance biological fixation. However, HA coatings with different microstructural characteristics profoundly influence subsequent biological performance, leading to miscellaneous biological responses after implantation [2]. HA coatings have been applied by a wide range of surface deposition techniques including plasma spraying, high-velocity oxy-fuel spraying (HVOF), pulsed laser ablation, ion-beam sputtering, electrophoretic deposition, radio frequency magnetron sputtering, sol–gel and conventional ceramic processes that involve pressing and sintering [3–13]. Among these techniques thermal spray techniques offer the attractive prospect of economy and efficient deposition of HA [1].

HA is known for its good biocompatibility. However, a major drawback is its relatively poor mechanical properties compared to natural bone, making it unsuitable for high-load applications. Its fracture toughness is only about 1 MPa m$^{1/2}$. In order to achieve the mechanical characteristics needed for biomedical applications, blending with a tough phase is essential. Titanium metal, Ti-6Al-4V alloy, yttria-stabilised zirconia (YSZ), Ni_3Al and Al_2O_3 ceramics have been considered good candidates as the reinforcing phases [14–17]. The mechanical properties of such HA-based composites are strongly dependent on the reinforcement fillers and interfacial adhesion.

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