



Atmospheric plasma sprayed silica–hydroxyapatite coatings on magnesium alloy substrates

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Abstract

Silica-doped hydroxyapatite as a bioactive coating presents some advantages compared to the pure one, such as: increased in vivo bioactivity and early bone ingrowth. The aim of this study is to obtain a deposition of silica-doped hydroxyapatite on magnesium alloy plates by atmospheric plasma spraying. The coating material was prepared by a precipitation method with sodium silicate addition as a source of silica, and various methods were used to characterize it. Spraying conditions including powder feed rate and current values were varied. The coating properties were defined by determining the purity, phase composition, morphology and corrosion protection of the HAP–Si deposits on the magnesium plates. © 2015 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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1. Introduction

Hydroxyapatite (HAP), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ is a major bone component. For this reason, it has been mostly used as a biomaterial for over 20 years in medicine and dentistry [1]. Although their mechanical properties are insufficient to use them as load-bearing implants, HAP ceramics can be applied as coatings on metallic implants.

One way to enhance the mechanical strength of hydroxyapatite is by obtaining *substituted apatites*, which resemble the chemical composition and structure of the mineral phase in bones. From this necessity a new generation of silicon substituted hydroxyapatite (HAP–Si) bioactive material has emerged. It has been demonstrated that hydroxyapatite modified with the inclusion of small concentrations of silicon improves

the in vivo bioactivity [2] and benefits the early bone ingrowth and repair through the incorporation of Si [3–5]. This means that the implant is able to chemically join the bone through a strong “bioactive bond”. HAP–Si was found to have increased surface adhesion compared to un-substituted HAP [6,7]. In this way, the osteointegration and the good performance of the implant are ensured [7].

Another important factor in implant design, which has to be taken into consideration, is the implant *substrate material*. Titanium (Ti) and its alloys are the most commonly used metallic materials, but stainless steel, cobalt–chromium alloys, ceramics and polymer composites were also studied [8]. Each has its advantages and limitations, too. For instance, the density, elastic modulus and yield strength of magnesium are closer to the bone tissue than that of the conventional implants. Due to these excellent mechanical properties, as well as for their biocompatibility, magnesium and its alloys have attracted ever-increasing attention for load-bearing biocompatible implant

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applications [9]. Moreover, Mg is also an essential element for bone metabolism that promotes the formation of new bone tissue [10]; being a cofactor for many enzymes, it stabilizes the structures of DNA and RNA [11]. The major drawback of using magnesium in many engineering applications is its low corrosion resistance, which may manifest in pitting corrosion [12], especially in electrolytic, aqueous environments. The corrosion behavior of the magnesium substrates was briefly studied in different media, such as NaCl solution, simulated body fluid and Hanks' balanced salt solution, using unpolarized and polarized [13] techniques.

Several approaches are applied to reduce the corrosion rate or even stop the corrosion of magnesium by using alloying elements and protective coatings; of course, these processes must lead to a non-toxic, biologically compatible material. The most effective approach in order to improve the corrosion resistance of magnesium alloys is probably by covering their surfaces with an additional layer [14]. Coatings may induce biodegradation at a controlled rate, thus offering a limited barrier function.

Coatings on magnesium and magnesium alloys can be manufactured through different methods [15] and all these were thoroughly studied in the past. Calcium phosphates represent a big promise for they can be used as biomedical coatings on magnesium substrates [16]. The literature of the last couple of years enumerates several methods for hydroxyapatite deposition on magnesium/Mg alloys: sol-gel coating [17], electrodeposition [18,19], a cathodic arc PVD technique [20], chemical solution deposition [21–23], dipping and immersion [24,25], cold spray deposition [26], plasma electrolytic oxidation [27,28], and a transonic particle acceleration deposition process [29]. However, the available literature presenting hydroxyapatite coatings deposition with atmospheric plasma spraying on magnesium alloy substrates is incomplete.

Plasma spraying is used to produce almost all commercially available HAP coatings for orthopedic and dental implants [7,30,31]. The major drawback of this process is that when HAP is plasma sprayed, it may transform into other crystalline phases of calcium phosphate, such as α - or β -tricalcium phosphate, tetracalcium phosphate or calcium oxide; the crystallinity of HAP may also be lowered due to the rapid solidification. These alterations in chemistry and crystallinity often interfere with the novel bioactive properties of HAP, as well as with its adhesion to the implant [26]. The amorphous material and other phases are significantly more soluble than hydroxyapatite, therefore these are preferentially dissolved in vitro [32]. The advantage of this method is that plasma spraying is a well-established tool to deposit HAP on metallic implants [7]; also, many studies were performed to investigate the microstructure, phase transformation, influence of post-treatment on the properties of sprayed HAP in vitro and in vivo [33,34].

The aim of this work is to obtain a deposition of silica doped hydroxyapatite bio-compound on magnesium alloy substrate material using a plasma spraying technique. The novelty of the work relies on the application of plasma spraying to achieve HAP-Si coatings on magnesium alloy, which considering the studied literature, was barely applied before.

2. Material and methods

2.1. Material preparation and characterization

Silica doped hydroxyapatite with 1.5 wt% silica content was prepared by precipitation, under controlled conditions, as described in one of our previous article [35]. The following materials were used: calcium nitrate tetrahydrate, diammonium hydrogen phosphate, 25% ammonia solution (Merck, Germany), sodium silicate (Lach:ner, Czech Republic). The reaction time was 20 h. After filtration, the resulting material was dried for 24 h at 105 °C. The heat treatment was performed at 1000 °C.

Various methods were used to characterize the calcined material. The characteristic vibrations of the material were identified by infrared spectroscopy (Bruker Vertex 70 FT-IR); the morphological structure was studied with a Philips XL30 scanning electron microscope; and the particle size distribution was determined with a SALD-7101 powder and particle size analyzer (Shimadzu), with an ultrasonic dispersion time of 10 s.

In order to identify the phase composition and content, X-ray powder diffraction measurements were made using CuK α radiation (typically 40 kV, 40 mA) and a Siemens D5000 diffractometer. The XRD data were obtained over 2θ range of 20–80° at 0.04° step size. The evaluation of the diffraction patterns was obtained by full profile fitting techniques.

2.2. Plasma spraying process

The coated substrates were magnesium plates (52 mm \times 30 mm \times 2.5 mm), which were grit blasted by corundum, prior to spraying, in order to enhance roughness and adhesion of the coating. After that, the specimens were ultrasonically cleaned in water and rinsed with acetone.

The composition of the magnesium plates was determined by an XRF analyzer (NITON, XL3t 900).

The atmospheric plasma spraying was carried out with a commercial plasma spray gun (Metco 9MB). The main operating conditions – including the applied voltage, current, gas flow rates and powder feed rate – are summarized in Table 1. The applied conditions were determined in preliminary tests that are not detailed here. During the main tests, only the feeding rate was varied, by which we could investigate the effect of the specific energy on the crystalline phase of the coating.

2.3. Coating heat treatment

Heat treatment on the sprayed coatings was also employed in order to investigate its effect on possible post-phase transformation. It was carried out in two different ways: i) at 600 °C in air for one and three hours; ii) hydrothermal treatment at 450 °C in water vapor enriched media for 1 h.

2.4. Coating characterization

The coatings were characterized by X-ray diffraction, using CuK α radiation 2θ range 10–80° at 0.01° step size to determine

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