



# Synthesis and characterization of Ca–Sr–P coating on pure magnesium for biomedical application

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## Abstract

There are growing evidences that Sr-containing calcium phosphate biomaterials can promote better osteo-precursor cell attachment and proliferation than pure calcium phosphate biomaterials. In this study, attempts were made to fabricate two kinds of Sr-substituted calcium phosphate (Ca–Sr–P) coatings on pure magnesium in electrolyte solutions with differing amounts of Sr(NO<sub>3</sub>)<sub>2</sub> for biomedical application. The surface microstructure, composition and chemistry of the coatings were characterized by Scanning Electron Microscope (SEM), Energy-dispersive X-ray Spectroscopy (EDS), and X-ray Diffractometer (XRD), respectively. In addition, electrochemical and immersion tests were performed to evaluate the corrosion resistance of the Ca–Sr–P coated magnesium in phosphate buffered saline solution (PBS). © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

**Keywords:** Strontium; Chemical deposition; Degradation; Bioactive coating; Magnesium

## 1. Introduction

In recent years, magnesium and its alloys have been widely studied for application in orthopedic implants due to their excellent properties, such as low toxicity, well biodegradation, attractive biocompatibility and excellent mechanical properties [1–3]. However, previous studies indicated that the fast degradation rate of magnesium implants in the physiological environment limits their clinical applications [4,5]. Avoiding this drawback, the degradation rate should be controlled to prevent early failure of implants from losing their mechanical integrities before the tissue healed [6]. Surface modification is a promising route to slow down the degradation rate in magnesium. Various surface modification technologies have been developed for magnesium to obtain protective coating, such as chemical deposition [7], biomimetic deposition [8], organic coating [9], sol–gel method [10], cathodic deposition [11,12], micro-arc oxidation [13], and pulsed laser deposition [14]. Calcium phosphate is used as a bone

substitute in dentistry and orthopedics because of its excellent biocompatibility, non-toxicity, bioactivity and osseointegration [15]. Recently, calcium phosphate has also been applied as a coating for the purpose of modifying the surface of the magnesium and its alloys to promote the direct attachment to the surrounding hard tissue and to reduce the degradation rate [16]. For example, Song et al. [17] successfully applied calcium phosphate on biodegradable Mg–Zn alloy to improve its biocompatibility and promote osteointegration. It was also reported that an ion-beam assisted deposition HA coating on an AZ31 magnesium alloy can significantly decrease its degradation rate in a NaCl aqueous solution [18].

Strontium, as a natural bone-seeking trace element, can enhance osteoblast differentiation and inhibit osteoclast differentiation, and used in therapy that promotes bone mass and strength [19]. In vitro and in vivo studies have proven that strontium can increase bone formation, reduce bone resorption and improve bone mechanical strength in humanity [20,21]. Considering the beneficial effects of strontium in bone, strontium has been incorporated into apatite phases for biomedical implantation via various methods, due to the similarities shared with calcium in physical and chemical nature [22–24]. According to Gorustovich et al. [25], Sr-containing calcium phosphate

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ceramics can promote better osteoprecursor cell attachment and proliferation. Qiu et al. [26] also reported that Sr-substituted calcium phosphate could stimulate the proliferation and differentiation of osteoblastic cells in vitro. Furthermore, Strontium has also been introduced into the coatings on traditional metal for the application in biomaterials [27–29]. For example, Chung et al. [30] replaced calcium with differing percentages of strontium in order to fabricate Sr-substituted Hydroxycarbonate coatings to enhance the osseointegration of titanium implants by micro-arc oxidation. The work demonstrated that the Sr-substituted coatings could significantly inhibit the osteoclast differentiation when the content of strontium in Hydroxycarbonate coatings exceeds 38.9 wt%.

To date, notwithstanding many reports come out around Sr-containing calcium phosphate ceramics and cements for bone tissue repair in the last decade [25,31], few research work about the Ca–Sr–P coating on magnesium surface by chemical deposition has been carried out. Chemical deposition, compared with other methods, is an effective way to fabricate Ca–Sr–P coatings on magnesium. Therefore, the aim of this work is to develop Sr-containing calcium phosphate coatings on pure magnesium by chemical deposition with the purpose of slowing down the degradation rate of pure magnesium. Additionally, the investigation of how strontium content affects the surface microstructure, chemistry and magnesium degradation of resultant modified magnesium were also investigated.

## 2. Experiment

### 2.1. Preparation of samples

As-cast pure magnesium bar with a purity of 99.99% (0.0014Fe, 0.0001Ni, 0.0002Cu, 0.0016Si, 0.0004Al, 0.0004Mn and balance Mg, wt%) was cut into plates of 10 mm × 10 mm × 2 mm. These samples were ground with grit SiC papers to 1200 grit, then cleaned in acetone for 10 min, rinsed in distilled water and finally dried at room temperature. Then, the samples were immersed into two kinds of electrolyte solutions at 70 °C for 24 h. After the coating process, the samples were washed with deionized water to remove the residue of the electrolyte solutions. The chemical composition of these electrolyte solutions is listed in Table 1, in which they are denoted as CD-1 and CD-2, in accordance with the content of strontium in the electrolyte solution.

Table 1  
The chemical composition of the electrolyte solutions (g/L).

Composition	Ca(NO <sub>3</sub> ) <sub>2</sub> · 4H <sub>2</sub> O	Sr(NO <sub>3</sub> ) <sub>2</sub>	NaH <sub>2</sub> PO <sub>4</sub> · 2H <sub>2</sub> O
CD-1	36	4	16
CD-2	28	12	16

### 2.2. Measurement of Sr-containing calcium phosphate coating

The morphologies of Sr-containing calcium phosphate coatings were characterized by a scanning electron microscope (SEM, Inspect F50) equipped with energy-dispersive X-ray spectroscopy (EDS). An X-ray diffractometer (XRD, D/MAX-2500PC) was used to identify the phase analysis on coatings. The XRD pattern was made with MDI Jade 5.0 software.

### 2.3. Electrochemical test

An electrochemical test was also conducted to evaluate the protective capacity of the coating on the magnesium substrate in the phosphate buffered saline (PBS) solution at 37 °C. A three-electrode cell was used for the electrochemical measurements. The counter electrode was made of platinum and a saturated calomel electrode (SCE) was used as the reference electrode. A sample with an exposed area of 1 cm<sup>2</sup> was taken as the working electrode. Before the test, all samples were immersed in the PBS solution for 30 min to obtain an electrochemical steady state. The polarization scan was started from an anodic region from 250 mV below open circuit potential at a constant voltage scan rate of 0.5 mV/s.

### 2.4. Immersion test

The further evolution of the corrosion resistance of the Ca–Sr–P coated magnesium was carried out through an immersion test in PBS solution. In order to examine the degradation behavior of the samples, the in vitro immersion test was performed in PBS solution. Each sample was soaked with the PBS solution kept in an airtight container at 37 °C in a carbon dioxide incubator. The ratio of sample area to solution volume was 1.25 cm<sup>2</sup>/mL. The immersion solutions were refreshed every day to simulate the real in vivo condition. The morphologies of coatings after 14 days of immersion were observed on SEM.

## 3. Results

### 3.1. Microstructure and phase identification

The morphologies and element content of coatings are shown in Fig. 1(a–d.) Obviously, the two kinds of coating densely cover on the magnesium substrate without any defects after exposure to a Sr(NO<sub>3</sub>)<sub>2</sub>-containing electrolyte solution. It is clear to see that when the 4 g/L Ca(NO<sub>3</sub>)<sub>2</sub> was replaced by Sr(NO<sub>3</sub>)<sub>2</sub>, only well-crystalline Sr-substituted calcium phosphate is found in the CD-1 coating as shown in Fig. 1(a–b.) But when the concentration of Sr(NO<sub>3</sub>)<sub>2</sub> increased the situation is reversed as shown in Fig. 1c, a distinct change is observed in the structure of the CD-2 coating; at higher magnification (Fig. 1d) it shows two phases coexist on the CD-2 coating: the one presents well-crystalline structure (marked I), and the other presents poor-crystalline structure (marked II), indicating that strontium has a strong influence on the microstructure of the coatings.

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