



Surface modification of stainless steel implants using nanostructured forsterite (Mg_2SiO_4) coating for biomaterial applications

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ARTICLE INFO

Article history:

Received 12 June 2014

Revised 16 April 2015

Accepted in revised form 6 June 2015

Available online 9 June 2015

Keywords:

Stainless steel

Implant

Coating

Bioceramic

Forsterite

Biomedical applications

ABSTRACT

The main aim of this research was the preparation of the forsterite (Mg_2SiO_4) coating on the surface of 316L stainless steel (316L SS) substrate. For this purpose, the nanostructured forsterite was coated on the 316L SS substrate using the sol–gel dip coating technique. Structural characterization techniques including X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDX) were utilized to investigate the phase structure, morphology and elemental composition of the uncoated and coated samples. Corrosion properties of samples were studied using the electrochemical measurements in simulated body fluid (SBF). The in vitro bioactivity evaluation of the forsterite coated samples was conducted by soaking the samples in the SBF at the temperature of 37 °C. The results showed that, a crack-free and homogeneous forsterite coating with the crystallite size of around 40 nm was successfully achieved on the surface of 316L SS substrate. The corrosion current density of the forsterite coated samples was lesser than that of the uncoated ones indicating the improvement of corrosion resistance of the metallic substrate using the forsterite coating. Deposition of Ca and P-contained products on the surface of coated samples during the incubation in the SBF solution confirmed the bioactivity behavior of the forsterite coated samples. Consequently, the 316L SS substrate coated with nanostructured forsterite may be an appropriate selection for dental and orthopedic implant applications.

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1. Introduction

Metallic biomaterials such as titanium and its alloys, cobalt-based alloys and stainless steels have been widely used for clinical applications due to their noticeable strength, biocompatibility, durability, and corrosion resistance in physiological environments [1,2]. The high mechanical strength and fracture toughness of the mentioned bio-metals are their most vital advantages compared to bioactive ceramics, which are inherently brittle [3]. Nowadays, stainless steel is employed as bone fixation devices due to the combination of mechanical properties, corrosion resistance and lower cost compared to other conventional metallic implant materials [4]. The biocompatibility of stainless steel implants has been approved by clinical trials [1]. Moreover, the handling of stainless steel implants are easier compared to that of the titanium alloys for required plastic deformations during the surgery [4]. However, stainless steel implants will be corroded in the presence of aggressive ions in bio-fluids. The corrosion process releases the ions which may adversely affect the biocompatibility of the implant [4]. Furthermore, the variation

of relative position of the implant is extremely detrimental and consequently immobilization of a patient may be necessary before the implant fixation. Therefore, during implantation, a close contact between metal prostheses and the host tissue is required for a subsequent ingrowth of the bone tissue into the implant surface [3]. Current clinical researches have reported that about 100 days recovery time can be reduced to only 20 days through the use of bioactive ceramics on the surface of implant due to the quick bond formation between the implant and bone tissue [3].

Among regular techniques for enhancement of corrosion resistance and biocompatibility of metal implants, surface modification has been frequently utilized [5]. Accordingly, selection of a proper coating material for stainless steel implants may highly affect the biological behavior [3]. A designed system including the metal as the substrate and a bioactive ceramics as the coating material combines the mechanical properties of the metallic substrate and biological properties of the bioceramic coating [6].

Among the bioactive ceramics, Mg and Si-contained ceramics have attracted much attention as bone replacement materials [7]. Si is an essential element in skeletal development and Mg is directly associated with mineralization of calcined tissues and ultimately influences the mineral metabolism [8]. Forsterite with the chemical composition of Mg_2SiO_4 is a member of olivine family of crystals in the magnesia-

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silica binary system which is considered as a new bioactive ceramics with appropriate bioactivity and biocompatibility properties [7]. Forsterite as a biocompatible material have in vitro bioactivity, better mechanical properties compared to the hydroxyapatite, and can be applicable as a new biomaterial for hard tissue repairs [7,9]. Moreover, fracture toughness of forsterite is close to that of the natural bone [10].

Hydroxyapatite (HA) is an important achievement of bioceramics as a bone tissue regenerating material. HA is osteoconductive; a property that encourages bone in-growth. However, weak mechanical properties including low toughness have confined its clinical applications. Bulk HA has lesser fracture toughness compared to that of the cortical bone, and more elastic modulus than cortical bone [11,12]. Bioactive glasses (BaG) are also another category of bioactive materials which have been employed for production of tissue engineering scaffolds. BaG indicates osteopductive properties, bonding ability to both of the soft and hard tissues and formation of a bioactive deposited layer (HCA) when exposed to physiological environment [13,14]. In contrast, the poor mechanical properties of these materials have restricted their clinical applications. The high reactivity of BaG is the main benefit for its application in bone repairs, because the deposited reaction products from the body fluids result in the formation of the apatite phase, similar to the inorganic constituent of bone in hard tissues [15,16]. Forsterite could be an attractive material owing to its superior mechanical properties and biodegradability than HA and BaG. In comparison with HA and BaG, forsterite indicates significant fracture toughness and hardness. Research indicates that forsterite has better mechanical properties compared to calcium phosphate ceramics like HA. The fracture toughness of forsterite ceramics is $2.4 \text{ MPa m}^{1/2}$ more than the lower limits reported for bone implants. Enhanced fracture toughness of forsterite coating compared to that of the HA coating will avoid the formation of cracks, pores, and coating delamination [10,17]. The biodegradability of BaG may be accounted as its main disadvantage when it is utilized as a coating material on the permanent implants such as 316L SS. Through the coating degradation, crack and pore is formed on the coating's surface resulting in the direct exposure of substrate to the corrosive media. It will adversely affect the degradation protection properties of coating. Ni et al. [7] indicated that forsterite is a bioceramics with high biocompatibility and might be apt for hard tissue regeneration. In addition, making the nanostructured forsterite suggests that these can be extensively enhanced concerning their fracture toughness ($KIC = 3.61 \text{ MPa m}^{1/2}$) and hardness (940 Hv) compared to the hydroxyapatite ceramics ($KIC = 0.75\text{--}1.2 \text{ MPa m}^{1/2}$ and hardness = 700 Hv). According to aforementioned advantages, we selected the forsterite as a coating material on the 316L SS substrate.

Among coating methods, sol–gel is simple, industrial and cost-effective technique which applies a variety of coatings materials on different substrates, involving the immersion of the substrate into a liquid medium [18].

The nanostructured materials with the high volume fraction of grain boundaries are reported to present improved biocompatibility and increased osteoblast adhesion and proliferation over the normal materials [19]. Regarding the forsterite, the in vitro studies have showed a significant osteoblast adhesion and forsterite nanopowder unlike micron-sized forsterite has indicated the in vitro bioactivity [9].

In this research, we used biomedical stainless steel type 316L SS as the substrate for studying the nanostructured forsterite coating using the sol–gel technique. The stainless steel type 316L SS has been extensively utilized for fabrication of prosthesis devices such as plate, screw, etc., in orthopedic surgery [4]. In this work, we investigate the characteristics of deposited coatings on the surfaces.

2. Materials and methods

2.1. Sample preparation

Rectangular substrates with the dimensions of $10 \text{ mm} \times 10 \text{ mm} \times 2 \text{ mm}$ were machined and prepared from the 316L SS. The prepared

samples were ground with SiC papers progressively to 1200 grit, afterward were ultrasonically cleaned in acetone for about 0.5 h and dried.

2.2. Coating process

The coating solution was prepared similar to our previous procedure on the production of forsterite nanopowders using sol–gel technique [17]. Briefly, the aqueous solution of magnesium nitrate was dissolved in 50 mL distilled water on a magnetic stirrer. Then, colloidal silicon oxide was added to the solution with the ratio of Mg:Si = 2:1. The sucrose was separately dissolved in 100 ml distilled water and was added to the previous solution. After homogenizing for 2 h, a solution containing polyvinyl alcohol in 200 ml distilled water with the ratio of magnesium ion to polyvinyl alcohol of 0.8:1 was added to the solution. The pH of solution was adjusted to be around 1 using nitric acid. The homogenizing operation of solution was conducted at ambient temperature for 2 h at 80 °C. To prepare the forsterite coating on the 316L SS substrate, the prepared solution was first stirred for 15, 30, and 45 min at 200 °C. Then, the samples were vertically immersed for 1, and 2 times in the prepared solution for 30 and 60 s and extracted gently with the constant rate of 27 mm/min. Aging and drying processes were performed on the coated substrates for 24 h at 25 °C and 48 h at 70 °C, respectively. The samples were calcinated for 2 h at 800 °C with the heating rate of 1 °C/min and were cooled inside the furnace. Among the abovementioned parameters, one set of parameters which could create and develop the best quality of the coating in regard to the coating thickness, delaminating and pore formation was selected as follows: stirring time: 45 min, immersing time: 30 s for 1 time, extracting rate: 27 mm/min, heat treatment time and temperature: 24 h at 25 °C, 48 h at 70 °C, and 2 h at 800 °C with a heating rate of 1 °C/min.

2.3. Coating characterization

A low angle X-ray diffraction analysis technique (XRD: Xpert Pro MPD) was conducted to reveal the phase composition of the synthesized forsterite coating. The operating conditions were selected 40 kV and 30 mA using Cu- K_{α} radiation at 2θ range of 10–90°, employing the step size of $0.02^{\circ}/\text{s}$. The broadening of the peaks in XRD patterns was utilized to determine the crystallite size according to Scherrer formula (Eq. (1)) [20]:

$$X_s = 0.9\lambda / \beta \cos \theta \quad (1)$$

where X_s is crystallite size (nm), λ is wavelength of X-ray beam ($\lambda = 1.5418 \text{ \AA}$ for Cu- K_{α} radiation), β is full width at half maximum of the diffraction peaks (radian), and θ is diffraction angle (degree).

The morphology and elemental composition of prepared samples were investigated using a scanning electron microscopy (SEM: Philips XL 30: Eindhoven) equipped with energy dispersive spectroscopy (EDS).

2.4. Electrochemical corrosion measurements

A standard platinum wire was used as the counter electrode and a KCl saturated Ag/AgCl electrode was used as the reference electrode. In order to perform the potentiodynamic polarization experiments, uncoated and forsterite coated 316L SS samples as working electrodes were subjected in 600 ml simulated body fluid (SBF) at $37 \pm 1^{\circ}\text{C}$. The effective surface area of samples was adjusted to be 1 cm^2 . The open-circuit potential (E_{ocp}) of the samples was continuously monitored for 1 h until the potential was reached to a stable value. The electrochemical polarization corrosion examinations were carried out using a potentiostat/galvanostat with the model of AMETEK, PARSTAT 2273. The potential were scanned from -250 mV to $+800 \text{ mV}$ versus open-circuit potential with a scan rate of 3 mV/s . The corrosion

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