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Research Paper

Porous vitalium-base nano-composite for bone replacement: Fabrication, mechanical, and in vitro biological properties

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ABSTRACT

Porous nano-composites were successfully prepared on addition of 58S bioactive glass to Co-base alloy with porosities of 37.2–58.8% by the combination of milling, space-holder and powder metallurgy techniques. The results of X-ray diffraction analysis showed that induced strain during milling of the Co-base alloy powder and also isothermal heat treatment during sintering process led to HCP↔FCC phase transformation which affected mechanical properties of the samples during compression test. Field emission scanning electron microscopy images showed that despite the remaining 58S powder in nanometer size in the composite, there were micro-particles due to sintering at high temperature which led to two different apatite morphologies after immersion in simulated body fluid. Calculated elastic modulus and 0.2% proof strength from stress–strain curves of compression tests were in the range of 2.2–8.3 GPa and 34–198 MPa, respectively. In particular, the mechanical properties of sample with 37.2% were found to be similar to those of human cortical bone. Apatite formation which was identified by scanning electron microscopy (SEM), pH meter and Fourier-transform infrared spectroscopy (FTIR) analysis showed that it could successfully convert bioinert Co-base alloy to bioactive type by adding 58S bioglass nano-particles. SEM images of cell cultured on the porous nano-composite with 37.2% porosity showed that cells properly grew on the surface and inside the micro and macro-pores.

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1. Introduction

The Co-base alloys conforming to the ASTM F75 (vitalium) have been widely used for biomaterials because of their appropriate biocompatibility and high mechanical properties (Ping et al., 2006; Dourandish et al., 2008; Giacchi et al., 2011). But, the mismatch between elastic modulus of this alloy (~240 GPa) and human bone (cancellous: <3 GPa and cortical bone: 3–20 GPa) is a major problem which lead to stress-shielding and micro-movement between the implant and the adjacent bone and finally promotes separation of implant from surrounding tissue after implantation (Jurczyka et al., 2011; Torres et al., 2012).

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In the recent years, particular attention was paid to the synthesis of porous metals, with a unique combination of metals and foam properties, to allow the ingrowth of bone tissue, which improves the mechanical fixation of the implant at the implantation site and also provide a system that enables stresses to be transformed from the implant to the adjacent tissue (Schneider et al., 1989). In addition, because porous metals have lower elastic modulus rather than bulk metals (Gibson and Ashby, 1999), using these materials is a way to overcome stress-shielding and micro-movement between the implant and surrounding bone.

Almost all metallic biomaterials, including Co-base alloys, are classified as bioinert materials because they cannot effectively interact with surrounding bone and form a chemical bond while they are implanted. On the other hand, bioglasses and bioceramics such as 58S bioglass (58% SiO_2, 38% CaO and 4% P_2O_5 in molar percentage), with insufficient mechanical properties in load bearing conditions, can direct bond to soft and hard tissues and thus can be the possible solution to this bioinert alloy. Synthesis of Co-base composite with bioglass might be an approach to take advantages of bioglass and metals.

Ball milling can be considered as a solid state powder metallurgy process in which an important amount of defects such as dislocations, vacancies, stacking faults are created during milling. These defects and diffusion at the atomic level which activated by high-energy ball milling, allows production of various non-equilibrium phases.

Generally, the methods for porous metal fabrication are categorized into four groups of production: liquid, solid, gas and aqueous solutions, which among of them powder metallurgy techniques, are promising because of their considerable advantages. The space-holder technique which classified as a powder metallurgy process was first employed by Zhao and Sun (2001) for producing porous aluminum. In this method, the spacer particles create the space within the structure and therefore allow simple and accurate control of pore fraction, shape and interconnectivity in the structure.

Considering benefits of bioglass nano-particles and porous materials, the aim of this work was to fabricate porous Co-base nano-composite using milling, space-holder and powder metallurgy techniques. The present study focused on the structural, mechanical and in vitro biological characteristics of fabricated samples.

2. Materials and methods

2.1. Raw materials and sample preparation

Co-Cr-Mo (Co–28%Cr–5%Mo; all in weight percent) powder according to ISO standard 58342-4 (E) (ISO-1996) was purchased from Carpenter Co., Sweden. As shown in Fig. 1, this powder has spherical morphology with a mean diameter of about 130 μm. This powder was milled in a PM 4000 Retch planetary ball mill, using a rotational speed of 300 rpm. The 150 mL stainless steel container was charged with 12.7 g of the powder and three different stainless steel balls in size; the whole number of balls were eight: four balls with 10 mm, two balls with 19 mm, and two balls with 21.3 mm in diameter. The milling experiments were done with a ball to powder weight ratio of 12:1 with 3 cc ethanol as process control agent (PCA) under high purity argon gas. The powder after 12 h of milling was employed to fabricate the porous Co-base nanocomposite.

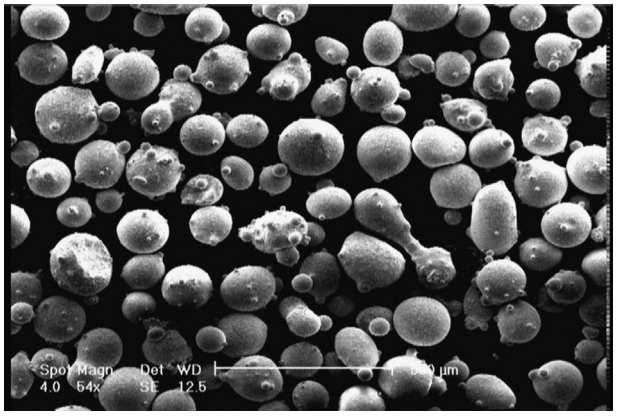

Fig. 1 – SEM micrograph of gas-atomized Co-base alloy powder.

The 58 S bioglass nano-powder (58%SiO_2–38%CaO–4%P_2O_5; all in mol%) was synthesized by sol–gel method (Taghian Dehaghani et al., 2015) and used as a bioactive material. Transmission electron microscopy (TEM, CM120; Philips, Eindhoven, the Netherlands) of the synthesized 58S bioglass nano-powder is shown in Fig. 2. As can be observed, the sizes of these particles are approximately smaller than 100 nm and show quasi-spherical morphology.

Ammonium hydrogen carbonate (NH_4HCO_3) having nearly cubic shapes, distributed in the range of 250–500 μm (Fig. 3), and PVA solution (5 wt% PVA+95 wt% water) were used as space holder and organic binder materials, respectively.

The ball milled powder, 15 wt% of bioglass nanopowder, different amount of NH_4HCO_3 and two weight percent of PVA solution were mixed properly. The blended powders were then cold compacted at pressure of 200 MPa into cylindrical green compacts with 5 mm in diameter and 7.5 mm in height. The green compacts were sintered under high purity argon gas through two steps. The samples were heated at 175 °C for 2 h and 1250 °C for 2 h to burn out the space-holder particles and sintering, respectively.

2.2. X-ray diffraction analysis

The structure and phase composition of as-received, milled powders and sintered samples were characterized by means of Philips X'pert-MPD X-ray diffraction instrument with Cu K_α radiation ($\lambda_{CuK\alpha}$=0.154186 nm at 30 mA and 40 kV) in the range of $30^\circ<2\theta<100^\circ$ (step size: 0.05 and time per step: 1 s).

The relative volume fractions of transformed HCP phase were calculated using the following formula (Balagna et al., 2012).

$$HCP(wt\%) = I(10\bar{1}1)_{HCP}/(I(10\bar{1}1)_{HCP} + 1.5I(200)_{FCC}) \quad (1)$$

where $I(200)_{FCC}$ and $I(10\bar{1}1)_{HCP}$ are the integrated intensities of the (200) and $(10\bar{1}1)$ XRD peaks of the FCC and HCP phase, respectively.

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