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Surface modification of investment cast-316L implants: Microstructure effects

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ABSTRACT

Artificial femur stem of 316L stainless steel was fabricated by investment casting using vacuum induction melting. Different surface treatments: mechanical polishing, thermal oxidation and immersion in alkaline solution were applied. Thicker hydroxyapatite (HAP) layer was formed in the furnace-oxidized samples as compared to the mechanically polished ones. The alkaline treatment enhanced the precipitation of HAP on the samples. It was also observed that the HAP precipitation responded differently to the different phases of the microstructure. The austenite phase was observed to have more homogeneous and smoother layer of HAP. In addition, the growth of HAP was sometimes favored on the austenite phase rather than on ferrite phase.

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1. Introduction

Fabrication of artificial joints and stems from biomaterials is an essential area of large potential and impact on the improvement of human and animal quality of life. For any material to act as a biomaterial, it must satisfy two essential characteristics: biofunctionality and biocompatibility. Biofunctionality is related to a set of properties which allows a device to perform a required function, while biocompatibility refers to the ability of the device to continue performing that function effectively in the body, and as long as necessary [1].

Hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(OH)_2$) is a common biomaterial that has excellent properties such as bioactivity, biocompatibility and ability to induce bone growth [2]. After implantation into the body, the HAP forms strong chemical bonds with bone tissues and enhances new bone growth since its chemical and mineralogical composition and crystallographic structure are similar to those of the apatite of the human bone. However, HAP does not offer sufficient mechanical properties, which impedes its clinical use in long term load-bearing applications [3,4]. However, when it was used in association with a biomaterial such as stainless steel 316L, this problem was overcome [5].

The most widely used stainless steel for medical and dental applications is the austenitic grade AISI 316L [5–7]. It was also reported that the stainless steels do not corrode in-vivo [8]. The breakdown potential of the chromium oxide layer is close to the in-vivo rest potential [9]. If the corrosion process occurred, the release of metallic ions such as Ni^{+2}, Cr^{+3} and Cr^{+6}, would produce serious health risks [10]. In order to reduce corrosion and wear rates of the 316L stainless steel, as well as to increase its fatigue strength, several surface treatments were used such as anodization, passivation, alkaline treatment and others [11]. The implant surfaces treated by these methods can provide a corrosion resistant surface, however it may not satisfy the "in-vivo" requirements [12]. Therefore, biomimetic methods [13–15] have been proposed and developed by many researchers.

Biomimetic process is a physicochemical method in which a substrate is soaked in a solution that simulates the physiologic conditions. The soaking time is long enough to form a desirable layer of calcium phosphate on the substrate [16]. Prior to the immersion in the simulated body fluid, the substrate is usually treated to facilitate the build-up of calcium phosphate (hydroxyapatite) layer [12]. Many research works were done for this purpose on some stainless steels and titanium alloys as reviewed by Bauer et al. [17]. However, neither of these works was devoted to study the influence of the substrate microstructure and its constituting phases on the precipitation of HAP from the simulated body fluid (SBF). The purpose of this study was to apply such pre-biomimetic surface treatments on a real 316L implant, fabricated by investment casting, and to investigate the effect of several surface treatments on the nucleation of hydroxyapatite. In addition, the effect of substrate microstructure on the CaP formation was also studied.

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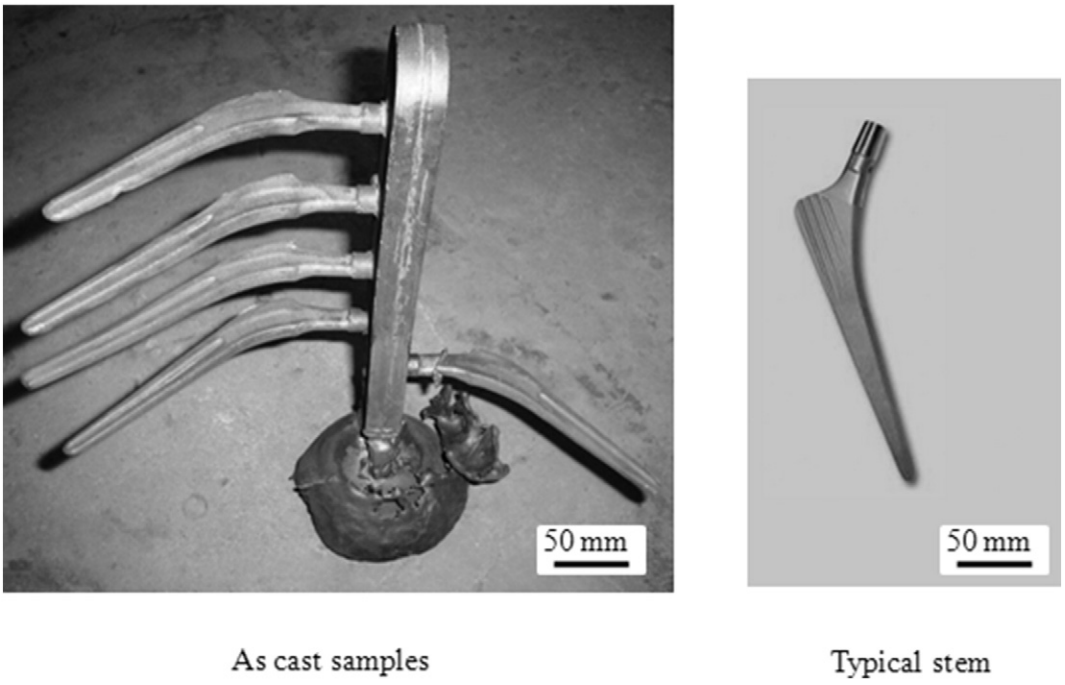

Fig. 1. As cast stems and a typical stem.

2. Experimental

2.1. Casting

Pieces of 316L stainless steel scrap were melted in a vacuum induction skull melting (ISM) furnace. Double vacuum melting process [18] was performed to ensure the quality, purity and properties of the material. In the current experiments, investment casting method was used for fabrication of stems. Investment casting process, which is also known as the lost-wax technique, has the advantage of reproducing complex shapes of tiny sizes at relatively low cost, scalability from single items to large numbers of identical items, and low wastage of raw materials [19,20].

In our experiments, a wax pattern was made typical to the artificial femur stem profile and dimensions using rubber silicon, Fig. 1. A ceramic mold was prepared by investment method [21]. The molten metal was then poured into the mold cavity. When the metal solidified, the ceramic shell was broken off and the cast stems were cut away from the central sprue using a high speed friction saw. A selected stem, free from casting defects, was cut transversally into small samples and finely grinded for surface treatment purposes.

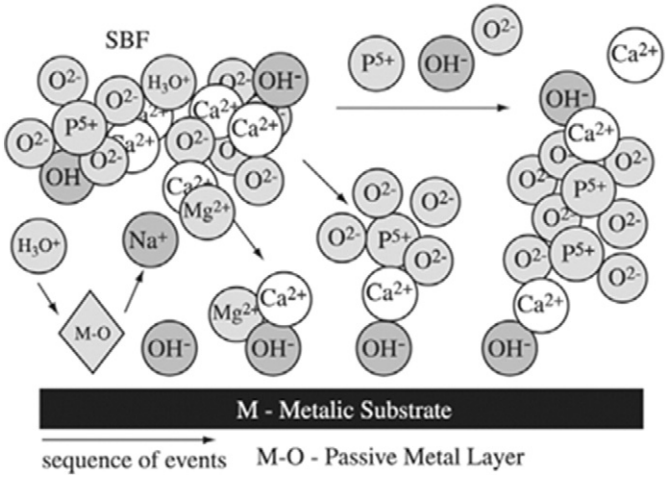

Fig. 2. Schematic representation of alkaline treatment.

2.2. Surface treatments

2.2.1. Mechanical polishing

The as-grinded samples were mechanically polished using alumina paste until a mirror-like surface was obtained.

2.2.2. Thermal oxidation

The austenitic stainless steel (316L) forms a thin chromium oxide layer when exposed to air. This layer plays an important role in increasing corrosion resistance and apatite formation [1,2]. In order to increase this oxide layer thickness, the sample was thermally soaked in a resistance furnace at 648 K (375 °C) for 10 h.

2.2.3. Alkaline treatment (gel layer formation)

The alkaline treatment was performed as follows. Polished samples were immersed in NaOH (0.5 mol/l) alkaline solution for 7 days. A hydrous gel layer formed on the metal surface, which is known to be weakly coherent and low in crystallinity as stated by Oshida [22]. The samples were then removed from the solution, and placed in a furnace at 353 K (80 °C) for about 3 h. The purpose of this treatment was to dry the gel layer and enhance its bonding to the substrate, as well as reverting it highly crystalline [22]. Pereira et al. [23] described the alkaline treatment as a sequence of the following steps; firstly the surface of

Table 1
Chemical composition of Hanks' physiological solution.

Substance	Composition (g/L)
NaCl	8.0
KCl	0.4
$NaHCO_3$	0.35
$NaH_2PO_4 \cdot H_2O$	0.25
$Na_2HPO_4 \cdot 2H_2O$	0.06
$CaCl_2 \cdot 2H_2O$	0.19
$MgCl_2$	0.19
$MgSO_4 \cdot 7H_2O$	0.06
$C_6H_{12}O_6$ (glucose)	1.0

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