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Biocompatibility of hydroxyapatite coatings deposited by pulse electrodeposition technique on the Nitinol superelastic alloy

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ABSTRACT

The present study deals with pulse electrochemical deposition of HA on NiTi alloy and in vitro evaluation of coatings. At first step, a thermo-chemical surface modification process was applied to control the Ni release of the alloy. The electrochemical deposition of Ca—P coatings was examined at both dilute and concentrated solutions. The morphology and the composition of coatings were studied using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). Plate like and needle like morphologies were formed for dilute and concentrated solution respectively and HA phase was formed by increasing the pulse current density for both electrolyte. The thickness of the samples was measured using cross sectioning technique. Fibroblast cell culture test on the coated samples revealed that the HA coating obtained by dilute solution shows the best biocompatibility. Also, MTT assay showed the highest cell density and cell proliferation after 5 days for the HA coating of dilute solution. The contact angle of samples was measured and the coated samples showed a hydrophilic surface. Soaking the sample in SBF revealed that the crystallization rate of calcium-phosphate compounds is higher on the plate like HA coating as compared to the needle like morphology. The P release of the HA coated samples was measured in a physiological saline solution and the results show that the ions releasing in the plate like coating are less than the needle like coating. It seems that the stability of the plate like coating in biological environments is responsible for the better biocompatibility of the coating.

1. Introduction

Titanium and its alloys are most popular load-bearing implants due to their low cost, high corrosion resistance, excellent biocompatibility and also mechanical properties [1]. Among Ti alloys, NiTi has gained superior attraction because of the similar elastic modulus and mechanical properties with human bone. This behavior makes the alloy an appropriate candidate for orthopedic applications [2]. Furthermore, superelastic and shape memory effects of the alloy can be used in various implants to improve the healing process [3]. However, the high Ni content of the alloy (>50 at.%) is one of the important issues in biomedical applications because the Ni ions cause allergy symptom in human body [4]. It was shown that, chemical and thermal treatments can be used to eliminate the surface Ni content of the alloy and formation of a titanium oxide film on the surface of the alloy [5,6]. Titanium oxide layer is biocompatible which means that there is not any poisoning effect of the layer which can result in cells death. However, this oxide has a poor bioactivity which means the human cells could not be connected to the oxide surface properly [7].

Although calcium-phosphate (Ca—P) based compounds such as hydroxyapatite offer an excellent medium for cells growth, they cannot be used as bulk materials in implants because of the poor mechanical properties. Application of Ca—P based coatings on the surface of metallic implants is a helpful method to achieve the both advantages of metallic implants and Ca—P compounds [8].

There are different methods to deposit calcium phosphate coatings such as plasma spray [9,10], electrophoretic deposition [11,12], sol-gel [13,14], biomimetic method [15], ion beam deposition [16] and electrochemical deposition [17,18]. Electrochemical deposition process is one of the useful methods for the deposition of these compounds in low cost and low temperature [19–21]. It was shown that pulse electrodeposition, can produce a uniform and crystalline HA coating with increased adherence. Also, the produced coatings by the pulse method have a long-term stability and a strong coating-substrate interface which guarantee a success implanting operation [22–24].

Xibo Pei et al. demonstrated that the inoculated primary osteoblasts attach and grow on pure HA better than Ti surface [25]. Noam Eliaz et al. was used mouse osteogenic cell line MBA-15 and showed that these

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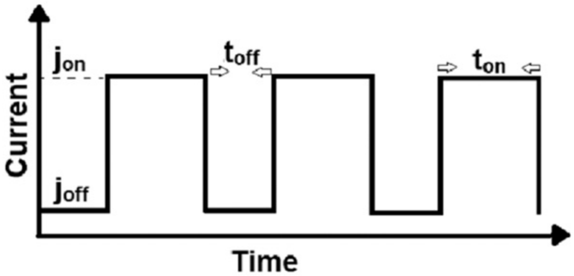

Fig. 1. Schematic representation of the pulse current used in the electrodeposition of Ca—P coatings.

cells attached and extended on the HA coated sample better than pure Ti [26].

Monchau et al. [27] were investigated the human and rat osteoclast cells activity on hydroxyapatite, β-tricalcium phosphate and calcium carbonate. According to the data, Ca and P concentrations in the culture media after 48 h culture test are lowest for the HA coating which can be related to the lowest degradation rate in HA coating. Etminanfar et al. [2] reported that human endothelial cells attachment to Ca—P coatings increases with increasing the Ca/P ratio up to that of the hydroxyapatite ratio (~1.7). According to the literature, most of research works investigated the effect of Ca/P ratio in Ca—P coatings. However, at a same Ca/P ratio the effect of surface morphology on the biological behavior of the coatings is rarely reported.

In this study, the bioactivity of pulse electrodeposited calcium phosphate coatings on shape memory alloy was investigated using soaking samples in SBF solution. Also, cell culture tests were carried out using fibroblast cells which are connective tissue cells and produce extracellular matrix (ECM) and collagen [28].

2. Materials and methods

2.1. Preparation of specimens

In this work, a medical grade NiTi rod with nominal composition of 50.9% Ni was used as substrates. At first, the samples were abraded with P80 to P600 grits and then were etched in a solution composed of 1 vol. HF - 4 vol. NHO_3 - 5 vol. H_2O for 4 min, and finally were soaked in distilled boiling water for 20 min (chemically etched and boiled

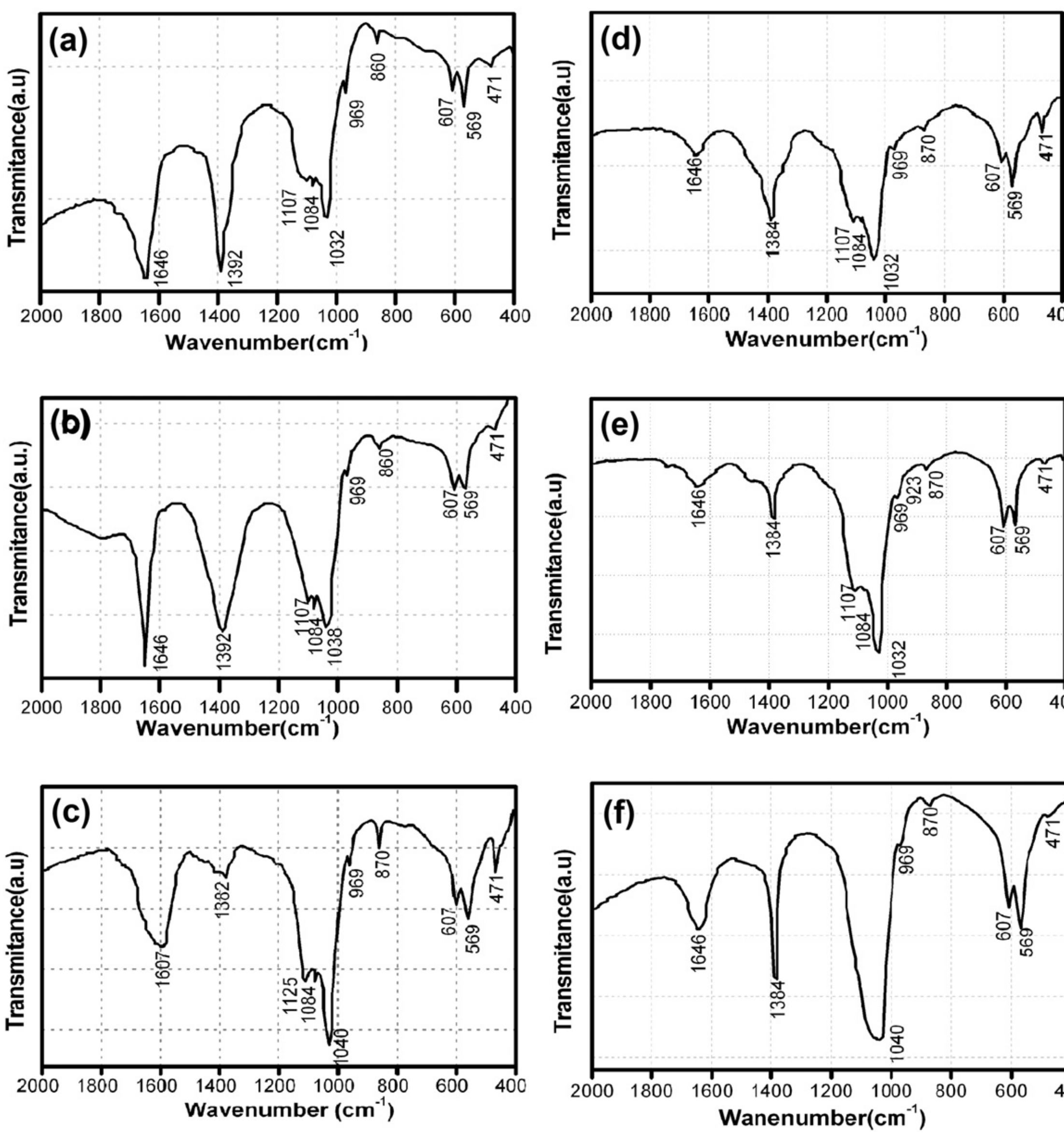

Fig. 2. FTIR spectra of the calcium phosphate coatings electrodeposited at a, b, c) 1.5, 3, 5 mA/cm^2 at (B) electrolyte and d, e, f) 1.5, 5, 15 mA/cm^2 at (A) electrolyte.