



## Surface Activation of Ni-Ti Alloy Using Electrochemical Process for Biomimetic Deposition of Hydroxyapatite Coating

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### ABSTRACT

Electrochemical depositions of calcium phosphide layer on Ni-Ti alloy in concentrated simulated body fluid (SBF×5) were carried out by cathodic electrodeposition. This layer was deposited on Ni-Ti alloy substrate under 10mA/cm<sup>2</sup> current density for 2 hours at room temperature. Then, in order to investigate the bioactivity of the pre-calcified samples, they were put in SBF for 1 and 3 days at room temperature. The microstructure, chemical composition, and bioactivity of the coatings were evaluated using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) techniques. Results showed that the activation of the surface of the Ni-Ti alloy by electrochemical process can significantly enhance the biomimetic deposition during time. On the other hand, by increasing immersion time of pre-calcified samples in SBF from 1 to 3 days, the biomimetic coating uniformly covered the surface of the sample. The ratio of the Ca/P for the pre-calcified sample after immersion in SBF for 3 days was about 1.5 which is very close to the Ca/P ratio of stoichiometric hydroxyapatite.

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## 1. INTRODUCTION

Biomaterials are used in the human body to replace or interact with a living tissue or biological systems. Titanium and its alloys have been extensively used as implant materials in orthopedic and dental surgery due to their excellent corrosion resistance and mechanical properties close to human bone [1-5]. Between Ti alloys, Ni-Ti alloy with a nearly equal atomic ratio of Ni and Ti has been increasingly used in medical and dental applications due to its high corrosion resistance and good biocompatibility [6-8]. Unfortunately, titanium and its alloys are bioinert and easily encapsulated after implanting into the living body by fibrous tissue that isolates them from the surrounding bone [9-11]. Furthermore, the high Ni content in the Ni-Ti alloy is one of the great concerns with regards to its biocompatibility. Because, it has been reported that Ni can be dissolved from Ni-Ti alloy due to the

corrosion. Then, it causes in the clinic toxic and allergic reactions [6]. Therefore, different surface modifications have been developed on titanium and its alloys in order to enhance their biocompatibility and bone-bonding [1, 2, 5, 12-14]. Among these techniques, plasma spraying of hydroxyapatite (HAp) coating is the most acceptable and commercial method, but it has some specific limitations related to its extremely high processing temperature. To overcome drawbacks of this method, several other deposition processes have been proposed [2, 3, 5, 14, 15]. One of them is biomimetic deposition which has some problems. It has been reported that this method will last about 7-14 days with daily refreshments [3]. Another promising alternative method is electrochemical deposition which is an attractive process because of its unique properties such as the easy of processing control, inexpensiveness, a low operating temperature, variability of the coating composition, possibility of protein delivery and suitability for complex implant geometries [1, 4, 5, 14, 16-18]. Recently, calcium

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phosphate layer on titanium and its alloys have become of great interest owing to their excellent combination of mechanical properties and biocompatibility [19]. Electrochemical deposition can deposit bioactive calcium phosphide layer on any titanium surface that contacts the solutions with supersaturated calcium and phosphor at room temperature [14, 20]. Some studies proved that the simulated body fluid (SBF) has almost the same ion concentrations as those of human blood plasma. So, it is a most efficient and usual method to soak the Ni-Ti alloy into SBF to investigate its biological behavior [6]. So far, numerous papers have been published on the apatite formation on Ti alloy, however, the formation of apatite coating on Ni-Ti alloy has not been still reported. Therefore, in this work an attempt was made for formation of calcium phosphide layer on Ni-Ti alloy substrate by electrochemical deposition in a simulated body fluid concentrated by a factor 5 (SBF×5) in order to deposit the precursor of HAp at room temperature. The morphology, phase composition and chemical composition of the coatings were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), energy dispersive spectroscopy (EDS) and Fourier transform infrared spectroscopy (FTIR). Untreated Ni-Ti alloy was used as control.

## 2. EXPERIMENTAL PROCEDURES

A commercial Ni-Ti alloy samples (discs with 15 mm diameter and 5 mm thickness) with equiatomic ratio of Ni and Ti were used as substrate in the experiments. The specimens were polished with SiC abrasive papers (up to 1000 grit) and then were mirror polished with 0.5 μm diamond pastes and finally ultrasonically degreased in acetone. In this work, SBF concentrated by a factor 5 (SBF×5) (its chemical composition is given in Table 1) was used an electrolyte. Cathodic electrodeposition was performed with the potentiostat/galvanostat instrument (EG&G Princeton Applied Research 273A) in the cell which had three electrodes: a Ni-Ti alloy sample as the working electrode, a platinum grid as the counter electrode and a saturated calomel electrode (SCE) as the reference electrode. In order to investigate the effect of current density on the properties of the calcium phosphide layer, two different current densities (5 and 10 mA/cm<sup>2</sup>) were chosen in the electrochemical deposition processes. After deposition, the samples were rinsed in distilled water to remove the residual electrolyte and then dried with the warm air. After that, the samples were soaked in SBF solution for 1 and 3 days at room temperature in order to study their biocompatibility. A simulated body fluid (SBF) with pH=7.4 and ion concentrations (in mM: Na<sup>+</sup> 142.0, K<sup>+</sup> 5.0, Mg<sup>2+</sup> 1.5, Ca<sup>2+</sup> 2.5, Cl<sup>-</sup> 147.8, HCO<sub>3</sub><sup>-</sup> 4.2,

HPO<sub>4</sub><sup>2-</sup> 1.0, SO<sub>4</sub><sup>2-</sup> 0.5) nearly equal to those of human blood plasma was reported [21]. The SBF was made by dissolving reagent grade chemicals of NaCl, NaHCO<sub>3</sub>, KCl, K<sub>2</sub>HPO<sub>4</sub>·3H<sub>2</sub>O, MgCl<sub>2</sub>·6H<sub>2</sub>O, CaCl<sub>2</sub> and MgSO<sub>4</sub> into distilled water and buffering at 7.4 with tris-hydroxymethylaminomethane ((CH<sub>2</sub>OH)<sub>3</sub> CNH<sub>3</sub>) and hydrochloric acid at 37 °C.

Finally, samples removed from the SBF were ultrasonically washed in distilled water and then air-dried. The surface morphology, microstructure and surface chemistry of the samples were evaluated by using scanning electron microscopy (SEM, Philips XL-30) equipped with energy dispersive analysis. Before the SEM analysis, the samples were sputtered with a thin gold layer to make their surfaces conductive. The phase compositions of the specimens were studied by X-ray Diffraction (XRD) analysis. Furthermore, the deposits on the samples were examined by Fourier transform infrared spectroscopy (FTIR). All of the SEM, EDS and XRD tests were carried out on samples before and after immersion in SBF.

## 3. RESULTS AND DISCUSSION

**3. 1. Characterization of Bioactive Films** In order to investigate the effect of the current density on the progress of deposition of calcium phosphide layer on the Ni-Ti substrates in SBF×5 solutions, the electrochemical deposition were carried out in two different current densities (5 and 10mA/cm<sup>2</sup>) for two hours. Figure1 shows the SEM micrographs of the calcium phosphide layer deposited on Ni-Ti alloy substrates at 5 and 10mA/cm<sup>2</sup>. It can be clearly seen that the layer deposited in 5mA/cm<sup>2</sup> (Figure.1 (a,b)) is inhomogeneous, i.e. the calcium phosphide layer can't cover the surface of the substrate completely. While, with increasing the current density from 5mA/cm<sup>2</sup> to 10mA/cm<sup>2</sup>, the surface of the substrate is covered with the layer of calcium phosphide, as shown in Figure.1 (c,d). So, it can be concluded that the current density of the electrochemical process plays an important role in the progress of the formation of the calcium phosphide layer on the Ni-Ti alloy substrate. On the cathode surface due to the high concentration of OH<sup>-</sup> ions in the electrolyte nearby the electrode surface, pH is high enough for HA formation. Because, the following reaction can occur on the surface of cathode:



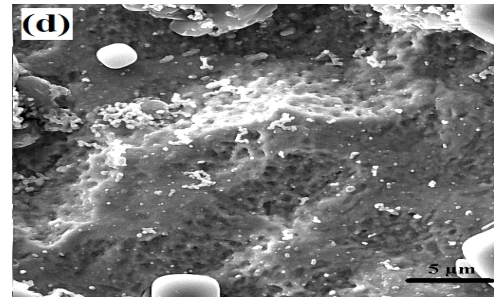
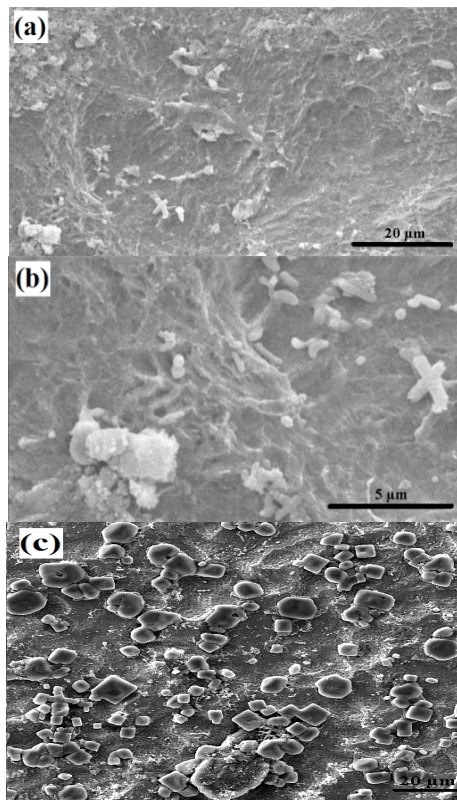
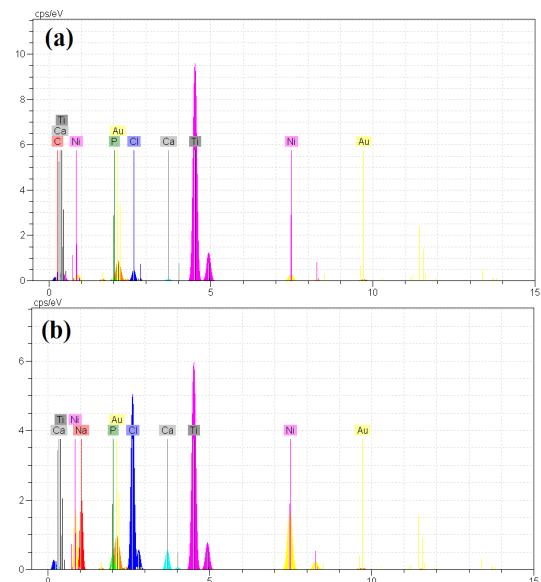
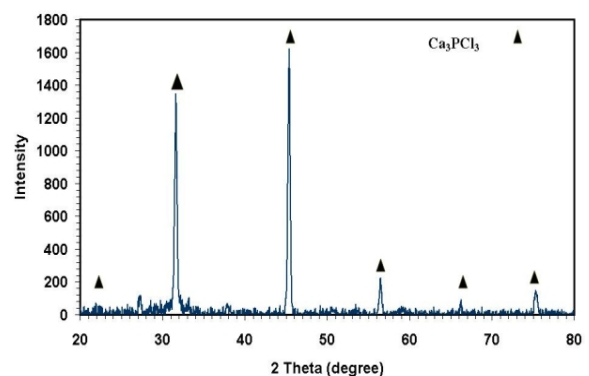
With increasing current density from 5 to 10 mA/cm<sup>2</sup>, rate of OH<sup>-</sup> formation is enhanced, therefore, formation rate of the calcium phosphide layer on the Ni-Ti alloy substrate is raised. As a result, a homogenous film can be made. Figure 2 shows the EDS spectra of the samples electrochemically deposited in different current densities which indicates

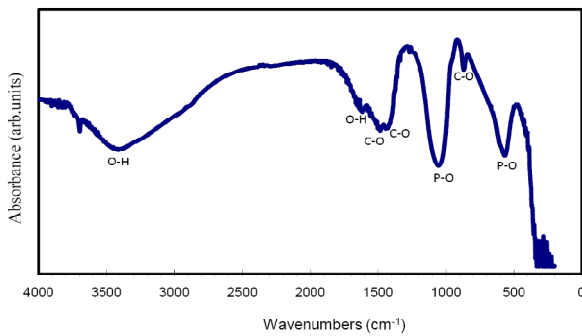
**TABLE 1.** Chemical composition of concentrated simulated body flood (SBF×5)

Matetials	NaCl	CaCl <sub>2</sub> .6H <sub>2</sub> O	MgCl <sub>2</sub> .6H <sub>2</sub> O	NaHCO <sub>3</sub>	Na <sub>2</sub> HPO <sub>4</sub> .2H <sub>2</sub> O
Concentration (g/lit)	42.866	2.738	1.525	1.764	0.318

that the signals from calcium and phosphorous are detected for the electrodeposited sample in higher current density(10mA/cm<sup>2</sup>), while these signals aren't visible for the electrodeposited sample in 5mA/cm<sup>2</sup>. This can be due to the inhomogeneous and very thin layer formation on the surface of this sample, as shown in SEM micrographs in Figure 1 (a,b). XRD analysis result for the electrodeposited sample in 10mA/cm<sup>2</sup> is shown in Figure3 which indicates that the layer is mainly composed of calcium chloride phosphide (Ca<sub>3</sub>PCl<sub>3</sub>). The peaks of calcium chloride phosphide are (110), (200), (210), (211), (220), (300), (310), (320), (400) and (421). These peaks are observed at 22.1, 31.4, 35.3, 32.9, 38.8, 45.1, 47.9, 50.6, 55.9, 65.7 and 76.8 at diffraction angel of degree, respectively.

FTIR spectrometry of the coating under 10mA/cm<sup>2</sup> was measured to determine the inorganic groups, and its result is shown in Figure4. On the spectra, it is evident that all the main peaks in Figure 4 can be assigned to HA and are similar to those of mineral phase of bones [22-24].This is in agreement with the EDS and XRD analysis results which are shown in Figure 2 and 3, respectively.

**Figure 1.** SEM micrographs of samples deposited at (a,b) 5mA/cm<sup>2</sup> and (c,d) 10mA/cm<sup>2</sup> for 2 hours.**Figure 2.** Surface EDS spectra of the samples calcified in different current densities: (a) 5mA/cm<sup>2</sup> and (b) 10mA/cm<sup>2</sup>.**Figure 3.** XRD pattern of the surface of the sample treated in 10mA/cm<sup>2</sup> for 2 hours



**Figure 4.** FTIR spectra of the sample treated in  $10\text{mA}/\text{cm}^2$  for 2 hours.

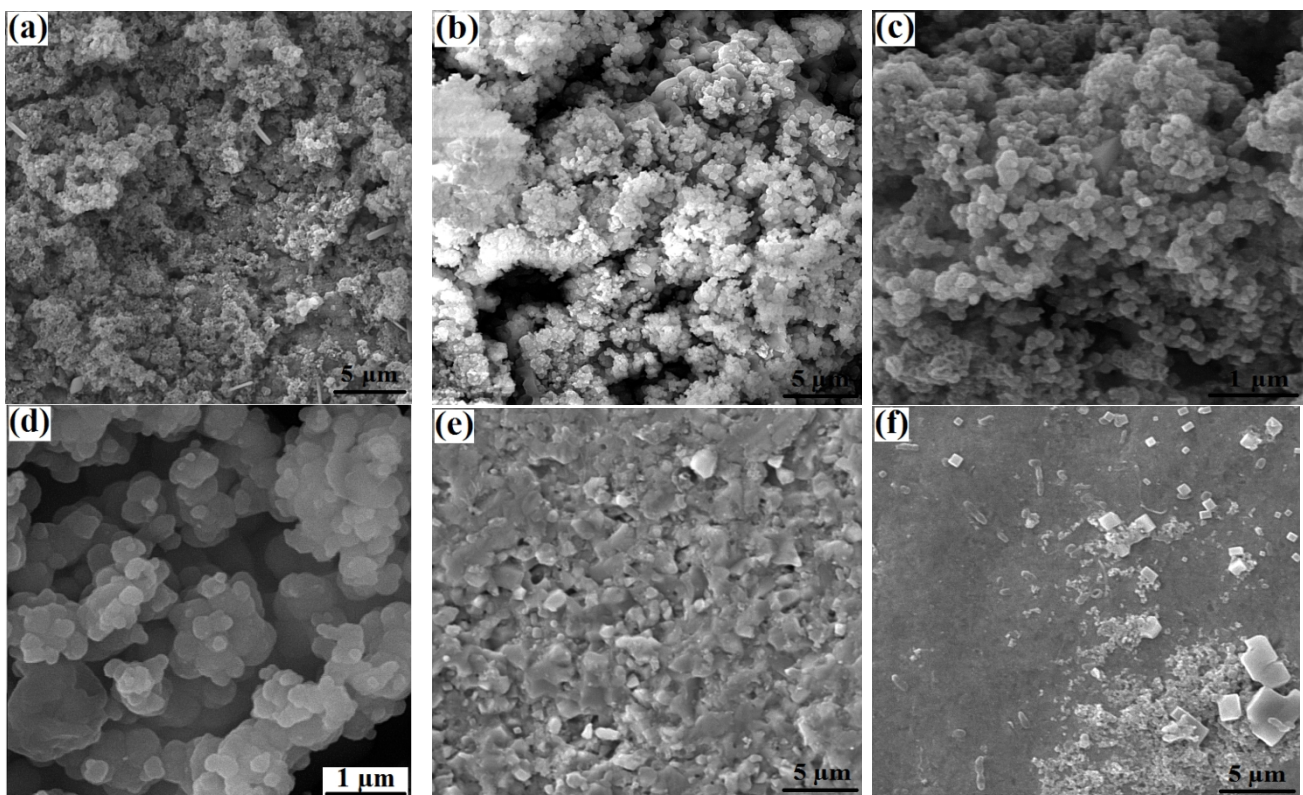
### 3. 2. Characterization of Biomimetic Layer

As discussed in the previous section, current density played an important role in the formation of calcium phosphide layer on the Ni-Ti alloy substrate. Results showed that the electrodeposited sample with  $10\text{mA}/\text{cm}^2$  has relatively more uniform layer than the electrodeposited sample with  $5\text{mA}/\text{cm}^2$ . So, in this section, it is decided to investigate the biomimetic properties of the electrodeposited sample in  $10\text{mA}/\text{cm}^2$ . In order to study the influence of the immersion time in

the SBF solution on the bioactivity of the biomimetic films, two different immersion times (1 and 3 days) were chosen.

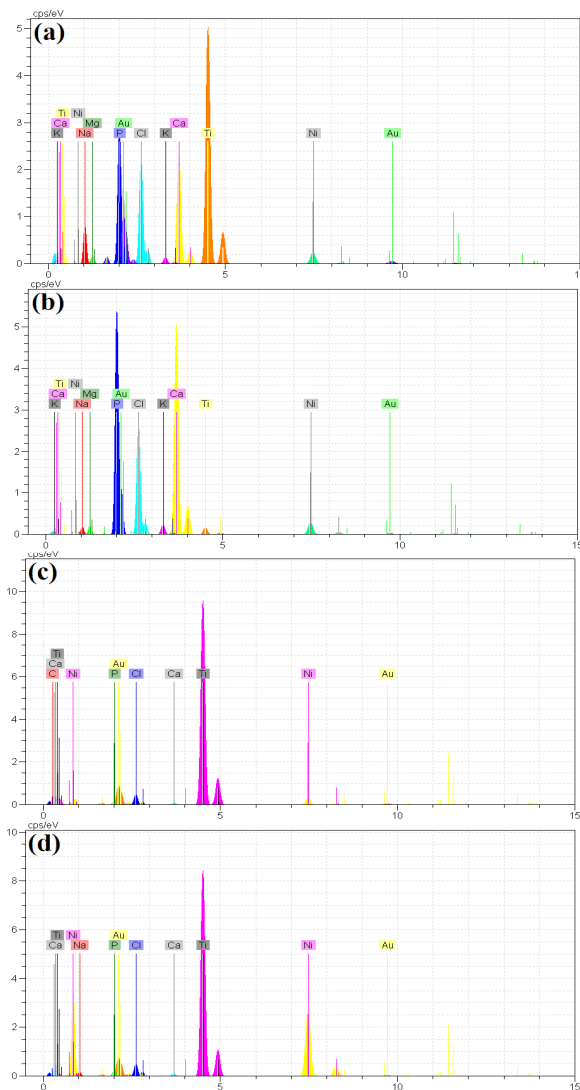
Figure 5 shows the surface SEM micrographs of the pre-electrodeposited specimens after soaking in SBF for various times. After soaking in SBF for a day, numerous fine granules appeared on the surface of the sample, as shown in Figure 5 (a,b). The number of the grains increased obviously with increasing the soaking time (Figure 5 (c) and (d)) and relatively complete coating was formed on the surface after 3 days. In order to investigate the effect of the pre-calcification using electrochemical process on the formation of HA coating, the surface morphology of the untreated Ni-Ti alloy after 3 and 10 days immersion in SBF are shown in Figure 5 (e) and (f), respectively.

As can be seen, without electrochemical deposition, the Ni-Ti sample shows poor HA deposition. After soaking of 10 days in SBF (Figure 5 (f)), no coating was formed and only a few isolated cubic particles were detected on the sample surface. Therefore, cathodic deposition has significant effects on biomimetic coating on Ni-Ti alloy substrate.

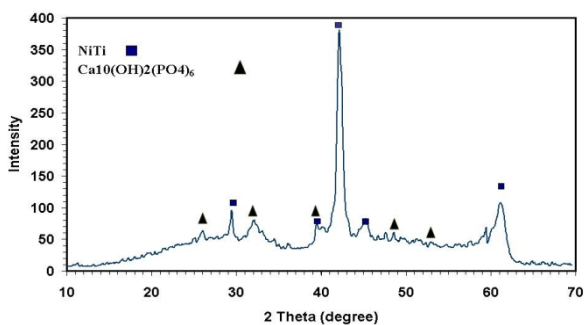


**Figure 5.** SEM micrographs of the biomimetic Ca-P coating: (a,c) on electrochemically treated surface after 1 day immersion; (b,d) on electrochemically treated surface after 3 days immersion; (e) untreated Ni-Ti surface after 3 days immersion and (f) untreated Ni-Ti surface after 10 days immersion.





**Figure 6.** EDS spectra of the biomimetic Ca-P coating: (a) on electrochemically treated surface after 1 day immersion; (b) on electrochemically treated surface after 3 days immersion; (c) untreated Ni-Ti surface after 3 days immersion and (d) untreated Ni-Ti surface after 10 days immersion.



**Figure 7.** XRD pattern of the pre-calcified sample after immersion in SBF for 3 days.

Figure 6 shows the EDS spectra of the pre-electrodeposited Ni-Ti alloys after soaking in SBF for various times. The EDS spectra of Ni-Ti sample, without electrodeposition, after soaking for 3 and 10 days are shown as control samples. The signals from calcium and phosphorous were detected for both samples which were electrochemically activated, but these signals are weak for the immersed sample for 1 day in SBF.

The chemical composition percentage of Ca and P on the specimen surface increased with increasing immersion time and reached about 1.5 after 3 days immersion, which is very close to the Ca/P ratio of stoichiometric HA. The EDS spectra of Ni-Ti alloys, without electrodeposition, after immersion for 3 and 10 days are shown in Figure 6 (c) and (d), respectively. It is evident that the intensity of the Ca and P signals for both samples are insignificant which is due to the very thin and non-uniform coating of the surface of these samples.

XRD pattern of the electrochemically calcified sample after immersion in SBF for 3 days is shown in Figure 7. The main phase present on the surface layer of this sample is HA which is in agreement with the EDS results. It is noted that HA layer has a semi-amorphous state, because, HA layer has been formed at room temperature on the surface of the electrochemically calcified sample. The peaks of HA are (002), (211), (112), (300), (212) and (213) which are observed at 25.9, 31.8, 32.2, 32.9, 39.8 and 49.6 at diffraction angle of degree, respectively.

#### 4. CONCLUSIONS

The deposition of calcium phosphide layer on the surface of Ni-Ti alloy using electrochemical process in concentrated simulated body fluid (SBF×5) was obtained under 10 mA/cm<sup>2</sup> current density at room temperature for two hours. The bioactivity of the alloy surface was significantly improved.

A relatively dense and uniform calcium phosphate layer composed mainly of hydroxyapatite (HA) was deposited on the pre-calcified Ni-Ti alloy substrate in SBF within three days. The ratio of the Ca/P for the pre-calcified sample after immersion in SBF for three days was about 1.5 which is very close to the Ca/P ratio of stoichiometric hydroxyapatite.

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# Surface Activation of NiTi Alloy Using Electrochemical Process for Biomimetic Deposition of Hydroxyapatite Coating

TECHNICAL  
NOTE

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تشکیل لایه فسفات کلسیم بر روی آلیاژ نایتانول از محلول غلیظ شبیه‌سازی شده بدن توسط فرآیند رسوب الکتریکی بصورت کاتدی انجام شد. این لایه بر روی آلیاژ نایتانول با اعمال چگالی جریان برابر با ۱۰ امپر بر سانتی‌متر مربع در دمای اتاق و در مدت زمان ۲ ساعت ایجاد شد. سپس، برای بررسی زیست فعال شدن سطح نمونه‌هایی لایه دار، این نمونه‌ها به مدت زمان ۱ و ۳ روز در دمای اتاق در داخل محلول شبیه‌سازی شده بدن قرار داده شدند. برای مشخصه‌یابی ریزساختار، ترکیب شیمیایی، ریخت‌شناسی و زیست فعالی نمونه‌هایی لایه‌دار به ترتیب از میکروسکوپ الکترونی، طیف‌سنجی تفکیک انرژی، پراش‌سنجی پرتوهای ایکس و طیف‌سنجی پرتوهای فراسرخ استفاده گردید. نتایج حاصل نشان داد که زیست فعال شدن سطح آلیاژ نایتانول توسط فرآیند الکتروشیمیایی بطور قابل ملاحظه در حین رسوب لایه بایومیمتیک افزایش یافت. به عبارت دیگر، با افزایش زمان قرارگیری نمونه‌هایی لایه دار در داخل محلول شبیه‌سازی شده بدن از ۱ روز به ۳ روز، لایه بایومیمتیک بر روی سطح نمونه‌ها بطور یکنواخت ایجاد می‌شود و نسبت کلسیم به فسفر در لایه تشکیل شده از محلول شبیه‌سازی شده بدن در مدت زمان سه روز به سمت نسبت استوکیومتریک لایه شبه آپاتیت میل نموده و به مقدار ۱/۵ نزدیک می‌شود.

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