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Improvements in the corrosion resistance and biocompatibility of biomedical Ti–6Al–7Nb alloy using an electrochemical anodization treatment

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ABSTRACT

The biocompatibility of an implant material is determined by its surface characteristics. This study investigated the application of an electrochemical anodization surface treatment to improve both the corrosion resistance and biocompatibility of Ti–6Al–7Nb alloy for implant applications. The electrochemical anodization treatment produced an Al-free oxide layer with nanoscale porosity on the Ti–6Al–7Nb alloy surface. The surface topography and microstructure of Ti–6Al–7Nb alloy were analyzed. The corrosion resistance was investigated using potentiodynamic polarization curve measurements in simulated blood plasma (SBP). The adhesion and proliferation of human bone marrow mesenchymal stem cells to test specimens were evaluated using various biological analysis techniques. The results showed that the presence of a nanoporous oxide layer on the anodized Ti–6Al–7Nb alloy increased the corrosion resistance (i.e., increased the corrosion potential and decreased both the corrosion rate and the passive current) in SBP compared with the untreated Ti–6Al–7Nb alloy. Changes in the nanotopography also improved the cell adhesion and proliferation on the anodized Ti–6Al–7Nb alloy. We conclude that a fast and simple electrochemical anodization surface treatment improves the corrosion resistance and biocompatibility of Ti–6Al–7Nb alloy for biomedical implant applications.

1. Introduction

Orthopedic implant designs often use alpha/beta (α/β) phase Ti–6Al–4V alloy, due to its favorable mechanical properties and acceptable biocompatibility. However, potential biological side effects of vanadium have been reported [1–5]. The presence of vanadium ions may increase the expression of pro-inflammatory factor and osteolytic mediator, leading to implant failure [1]. The presence of vanadium pentoxide on the alloy surface can be cytotoxic [2]. Furthermore, vanadium ions may also decrease fertility; embryo lethality, fetotoxicity, and teratogenicity can occur in animals after contact with vanadium [3–5].

The vanadium component, a β-phase stabilizer, can be replaced by another β-phase stabilizer element, niobium, to produce the new generation of Ti alloy (Ti–6Al–7Nb) for biomedical implant applications. As with the Ti–6Al–4V alloy, the Ti–6Al–7Nb alloy provides good mechanical properties but with fewer potential biological side effects [6]. However, notably limited information on the corrosion resistance and biological responses of the Ti–6Al–7Nb alloy is available.

The creation of porous structural features on the Ti-based implant material's surface is important when biocompatibility is a concern. Thus, there has been much effort to develop methods to create and modify porous Ti-based implant surfaces to provide the ideal biological response [7–11]. However, these methods used in the literature are sometimes time-consuming and/or too complex for potentially clinical applications. In contrast, researchers have used simple electrochemical methods to create ordered TiO_2 nanotubes on pure Ti surfaces under a fluoride-containing solution [12,13]. Our previous studies have used a fast electrochemical method to produce a nanonetworked TiO_2 layer on a commercially pure Ti surface for the improvement of blood coagulation and cell growth [14,15]. Nevertheless, complete information on the corrosion resistance and biocompatibility of porous and nanoscale surfaces for the new generation of biomedical Ti alloys, such as Ti–6Al–7Nb, with higher mechanical properties is not yet available.

We hypothesized that a nanoscale oxide layer on the Ti–6Al–7Nb alloy surface would improve the corrosion resistance and biocompatibility of the alloy. In this study, we used a fast and simple electrochemical anodization treatment to produce a nanoporous oxide layer on the Ti–6Al–7Nb alloy surface for biomedical implant applications. Additionally, we studied the corrosion resistance, cell adhesion, and

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cell proliferation of the anodized Ti–6Al–7Nb alloy surface with the nanoporous oxide layer.

2. Materials and methods

2.1. Material preparations

Commercial biomedical Ti–6Al–7Nb disks (diameter: 15 mm and thickness: 1 mm) were used as test substrates. The substrate surfaces were sequentially polished with SiC papers from #240 to #1200. An anodic current (0.2 A) was applied to polished Ti–6Al–7Nb specimens by a potentiostat for 12 min in alkaline solution (5 M NaOH) at room temperature (approximately 25 °C). Anodized Ti–6Al–7Nb specimens were designated as Ti6Al7Nb-A. Polished Ti–6Al–7Nb specimens without anodization treatment were designated as Ti6Al7Nb-P.

The surface topography and microstructure of the Ti–6Al–7Nb alloys were analyzed using field emission-scanning electron microscopy (FE-SEM) and X-ray photoelectron spectroscopy (XPS), respectively. The transmission electron microscope (TEM) specimen of Ti6Al7Nb-A was prepared for cross-sectional analysis with the focused ion beam (FIB) milling process. The adherence of the anodized layer on Ti6Al7Nb-A specimen was evaluated using American Society for Testing and Materials (ASTM) D3359: Standard Test Methods for Measuring Adhesion by Tape Test.

2.2. Corrosion resistance

A potentiostat was used to perform the corrosion tests in terms of the potentiodynamic polarization curve measurements with Ti–6Al–7Nb specimens as the working electrodes. A saturated calomel electrode (SCE) and platinum sheet were used as the reference electrode and counter electrode, respectively. The corrosion test electrolyte was neutral (pH 7.4) simulated blood plasma (SBP) maintained at 37 °C. The SBP consisted of NaCl (5.403 g/L), Na_2CO_3 (2.046 g/L), Na_2SO_4 (0.072 g/L), $NaHCO_3$ (0.740 g/L), KCl (0.225 g/L), $CaCl_2$ (0.293 g/L), $MgCl_2 \cdot 6H_2O$ (0.311 g/L), $K_2HPO_4 \cdot 3H_2O$ (0.230 g/L), and $C_8H_{18}N_2O_4S$ (11.928 g/L) [16]. The potentiodynamic polarization curves of Ti–6Al–7Nb specimens were measured from −1.5 V (vs SCE) in the anodic direction with a scan rate of 1 mV/s. The test electrolyte was deaerated with nitrogen gas for 1 h before the corrosion test started. The corrosion parameters, including the corrosion potential (E_{corr}), corrosion rate (I_{corr}), and passive current (I_{pass}), obtained from the potentiodynamic polarization curves were used to evaluate the corrosion resistance of the test Ti–6Al–7Nb specimens. At least 5 samples were used for each test condition.

2.3. Cell responses

The cell responses investigated in this study included cell adhesion and cell proliferation. Human bone marrow mesenchymal stem cells (hMSCs) with a multi-lineage potential were used. The cell culture medium was RPMI-1640 supplemented with 5% fetal bovine serum and 10% horse serum.

For the cell adhesion assay, the hMSCs were cultured on test Ti–6Al–7Nb specimens for 1 and 48 h, then fixed with 1% osmium tetroxide, and sequentially dehydrated in a series of ethanol and distilled water baths containing 30–100% volumes of ethanol. The cell adhesion morphology was observed using FE-SEM after coating a thin platinum film onto the Ti–6Al–7Nb specimens. Additionally, an immunofluorescent staining technique was used for the cell adhesion assay. The hMSCs were cultured onto test specimens (5×10^4 cells/cm^2) in an incubator with 5% CO_2 content at 37 °C. After 6 h of incubation, the cells were fixed in 10% formalin, and then permeabilized with 0.2% Triton X-100 in phosphate buffered saline (PBS), washed with PBS, and incubated with diamidino-2-phenylindole (DAPI) for nuclei staining and with rhodamine phalloidin for actin filament staining. Images of the

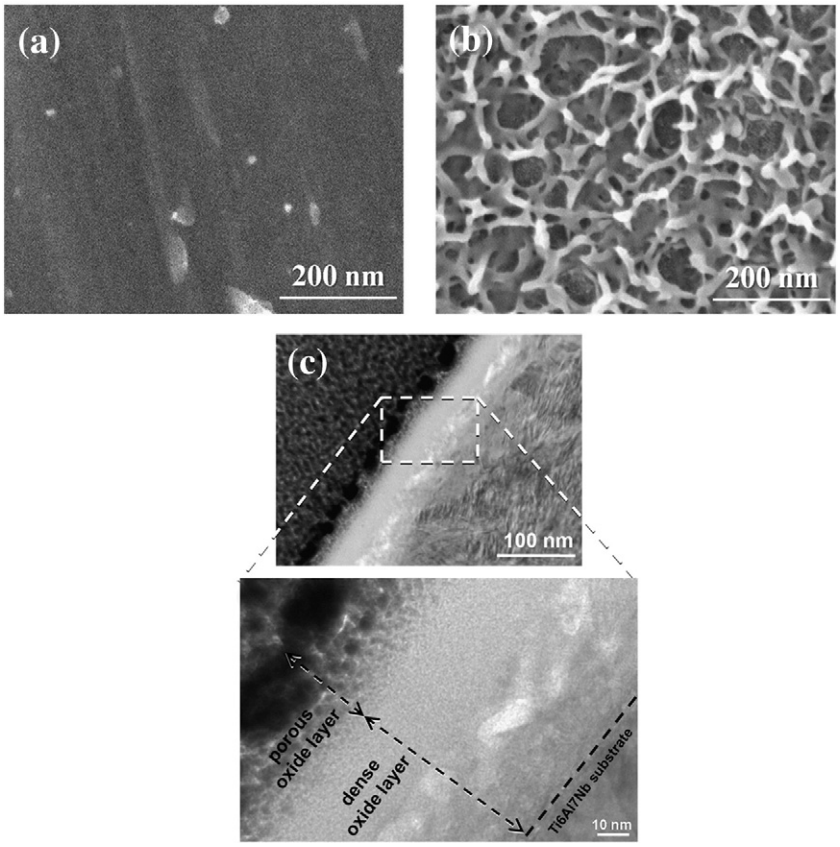

Fig. 1. FE-SEM micrographs of the (a) Ti6Al7Nb-P and (b) Ti6Al7Nb-A specimens; (c) cross-sectional TEM micrograph of the Ti6Al7Nb-A specimen.