



Modified surface morphology of a novel Ti–24Nb–4Zr–7.9Sn titanium alloy via anodic oxidation for enhanced interfacial biocompatibility and osseointegration



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ABSTRACT

The Ti–24Nb–4Zr–7.9Sn titanium alloy (Ti2448) has shown potential for use in biomedical implants, because this alloy possesses several important mechanical properties, such as a high fracture strength, low elastic modulus, and good corrosion resistance. In this study, we aimed to produce a hierarchical nano-structure on the surface of Ti2448 to endow this alloy with favorable biological properties. The chemical composition of Ti2448 (64.0 wt% Ti, 23.9 wt% Nb, 3.9 wt% Zr, and 8.1 wt% Sn) gives this material electro-chemical properties that lead to the generation of topographical features under standard anodic oxidation. We characterized the surface properties of pure Ti (Ti), nanotube-Ti (NT), Ti2448, and nanotube-Ti2448 (NTi2448) based on surface morphology (scanning electron microscopy and atomic force microscopy), chemical and phase compositions (X-ray diffraction and X-ray photoelectron spectroscopy), and wettability (water contact angle). We evaluated the biocompatibility and osteointegration of implant surfaces by observing the behavior of bone marrow stromal cells (BMSCs) cultured on the surfaces in vitro and conducting histological analysis after in vivo implantation of the modified materials. Our results showed that a hierarchical structure with a nanoscale bone-like layer was achieved along with nanotube formation on the Ti2448 surface. The surface characterization data suggested the superior biocompatibility of the NTi2448 surface in comparison with the Ti, NT, and Ti2448 surfaces. Moreover, the NTi2448 surface showed better biocompatibility for BMSCs in vitro and better osteointegration in vivo. Based on these results, we conclude that anodic oxidation facilitated the formation of a nanoscale bone-like structure and nanotubes on Ti2448. Unlike the modified titanium surfaces developed to date, the NTi2448 surface, which presents both mechanical compatibility and bioactivity, offers excellent biocompatibility and osteointegration, suggesting its potential for use in orthopedic applications.

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1. Introduction

Titanium and titanium-based alloys are well established as suitable implant materials in the fields of osteosynthesis, oral implantology, and in certain joint prosthetics applications [1–4]. The attractiveness of titanium alloys for implant fabrication is based on a combination of favorable characteristics including corrosion resistance, biocompatibility, high strength, low elastic modulus,

high density, and the capacity of titanium to integrate with bone and other tissues [5–7]. Therefore, new generation Ti-based alloy implants are expected to combine appropriate biological properties and ideal biomechanical performance to reduce the risk of aseptic loosening due to poor osteointegration and stiffness mismatch-derived stress shielding [7–9]. Various types of biomaterials and surface modifications have been applied with the goal of improving implant–bone interaction, but to date, none of the proposed systems form a stable interface with the strength required to support functional loading and the bioactivity to promote osseointegration [10,11].

Biologically, surface chemistry and morphology are two of the important properties that determine the biological performance

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of biomaterial implants [12]. Therefore, an important factor in selecting an orthopedic implant material is identifying the correct chemistry and morphology to support or stimulate the desired host response [13]. In comparison to smooth surfaces, surfaces with micrometer-sized or nanometer-sized morphological structures not only have positive effects on cell attachment, proliferation, and differentiation, but also present a larger surface area for integration with newly formed bone [14,15]. Previous research has shown that nanoscale structures in combination with micro-/submicro-scale roughness can improve osteoblast differentiation and local factor production [16]. In addition, other studies in the last 10 years show that other surface properties such as roughness, crystallinity and wettability could be important factors for biological performance of biomaterial implants [17,18]. Many techniques for modifying the implant surface have been proposed to improve osseointegration, such as sol-gel modification, hydroxyapatite coating, surface roughening, etc. [13]. Although these methods have been shown to improve osseointegration, they also result in disadvantages that, to varying degrees, compromise the long-term stability of the implant in vivo [14]. Implant surfaces also have been modified by incorporating biocompatible trace elements that are essential for normal bone metabolism [13], such as Sn, Si, and Zn, and improved biological performance of the modified implant has been reported [19].

Biomechanically, many types of alloys have been developed for use in orthopedic applications over the past five decades [20]. However, implants fabricated from these alloys have a variety of deficiencies, such as elastic modulus mismatch between the implant and surrounding bone that causes resorption and implant loosening and the release of aluminum (Al), vanadium (V) and nickel (Ni) ions, which have toxic effects on the surrounding tissues [21]. In addition, titanium (Ti) allergy can be detected in dental implant patients, even though its estimated prevalence is low (0.6%) [22]. Thus, a novel implant with an ultra-low elastic modulus, high strength, and minimal toxicity is needed. With advances in materials science, second generation β Ti-alloys have been invented for orthopedic applications, and examples such as the biomedical Ti-Nb, Ti-Nb-Zr, and Ti-Ta alloys show significantly improved elastic moduli, strength, and non-toxic composition [23].

The novel Ti-24Nb-4Zr-7.9Sn alloy (Ti2448) was developed recently by the Institute of Metal Research of the Chinese Academy of Science (PCT/CN2004/001352). Ti2448 contains only non-toxic and minimally allergenic elements and also exhibits a high strength (~ 850 MPa) and low elastic modulus (~ 45 GPa) [24–28], which are close to those of human bone. In addition, Bai et al. showed that the Ti2448 alloy exhibits good corrosion resistance due to the formation of a protective passive film [29]. Thus, the chemical and biomechanical properties of this novel alloy are advantageous for medical applications. In the present study, the surface of Ti2448 was modified by anodic oxidation to create a surface presenting a hierarchical nanoscale structure and a biocompatible chemical environment. We employed anodic oxidation because it is a convenient and reliable surface modification technique that can not only create nanoscale tubular structures on previously smooth titanium surfaces but also allows for control of the chemical properties via alteration of parameters such as the voltage, density, and electrolyte chemistry [30,31]. Nanotube structures created by anodic oxidation on the surface of Ti2448 alloy have been reported to stimulate rapid cell proliferation and osteoblast differentiation as well as to accelerate osseointegration in vivo [21,32]. However, in this study, a hierarchical bone-like structure consisting of irregular deep grooves and nanotubes was formed on the Ti2448 surface in order to facilitate transport of nutrients and bone ingrowth. Therefore, we investigated the osseointegration potential of this novel sur-

face through in vitro evaluation of bone marrow cell behavior on the surface and through an in vivo animal study.

2. Materials and methods

2.1. Preparation of surfaces and grouping

Medical-grade pure titanium and Ti2448 were provided by the Institute of Metal Research of the Chinese Academy of Sciences (Shenyang, China). Specimens for cell cultivation were prepared from a plate with dimensions of $\Phi 8$ mm $\times$ 1 mm, and samples for hard issue implantation were $\Phi 5$ mm $\times$ 10 mm. The surfaces were polished using SiC papers with a grain size ranging from 10 to 57 μ m using a grinding and polishing appliance (Saphir 360, ATM, Mammelzen, Germany). Then they were ultrasonically cleaned in acetone, alcohol, and deionized water (each for 10 min) and finally dried in air. The following groups were prepared and characterized: pure Ti (Ti), nanotube-Ti (NT), Ti2448, and nanotube-Ti2448 (NTi2448). The NT and NTi2448 samples were treated by anodic oxidation, which was performed at room temperature in a neutral electrolyte with 1 mol/L $(\text{NH}_4)_2\text{SO}_4$ and 0.15 mol/L NH_4F (pH = 6.7) prepared from analytical grade chemicals and deionized water. A direct current power supply was used to keep the potential at a constant value several times after the potential was swept from the open circuit potential to 20 V with a sweep rate of 0.5 V/s. A two-electrode system with stainless steel as the cathode and sample as the anode was used to fabricate the bone-like layer combined with the nanotube oxide structure under non-stirring conditions on the NTi2448 samples. For the NT samples, round TiO_2 nanotubes were generated by anodic oxidation as described previously [33]. All these processes were performed by the Institute of Metal Research of the Chinese Academy of Sciences.

2.2. Surface characterization

2.2.1. Surface morphology

The topography of all samples was characterized by field emission scanning electron microscopy (FESEM; Inspect F, FEI, Eindhoven, The Netherlands). The three-dimensional (3D) surface topography of samples was assessed using a Nanoscope MultiMode & Explore SPM 9700 atomic force microscope (Shimadzu Corporation, Kyoto, Japan). AFM measurements were conducted in ambient air under the tapping mode with a scan rate of 0.7016 Hz and a scan size of $10 \times 10 \mu\text{m}^2$. The surface roughness, vertical range, and surface area difference (the difference between the selected surface areas in each group) were estimated with the aid of Nanoscope imaging software (SPM-9700, Shimadzu Corporation). Measurements were taken at 10 random areas on the sample surface, and the average values were obtained.

2.2.2. Surface phase composition and chemical composition

The X-ray diffraction (XRD) patterns of the Ti, Ti2448, NT, and NTi2448 samples were determined using a glancing angle X-ray diffractometer (GAXRD; D/max2400, Rigaku, Japan) employing a $\text{CuK}\alpha$ radiation source with an accelerating voltage of 40 kV and a current of 250 mA. The chemical composition of the passive films on NT and NTi2448 samples were analyzed using X-ray photoelectron spectroscopy (XPS, Escalab250) in a vacuum chamber at a base pressure at $\sim 3.5 \times 10^{-8}$ Pa. The chemical distribution along the nanotube was previously analyzed by Zhao et al., and their processing technique was described previously [34].

2.2.3. Surface wettability

The surface wettability of samples in the four groups was characterized through the sessile drop method using a RaméHart 290 automated goniometer/tensiometer (Succasunna, NJ, USA). The

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