



Microstructures, mechanical properties and corrosion resistance of the Zr–xTi (Ag) alloys for dental implant application



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HIGHLIGHTS

- The quenched Zr₂₀Ti and Zr₄₀Ti obtain acicular martensite microstructure.
- Zr₂₀Ti and Zr₄₀Ti possess high hardness, strength and strain to fracture.
- Increasing Ti content decreases corrosion current density.
- Adding Ag enhances passivation breakdown potentials of Zr₂₀Ti and Zr₄₀Ti.
- Zr₄₀Ti₆Ag has optimum mechanical properties and pitting corrosion resistance.

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ABSTRACT

The Zr–xTi (Ag) alloys were designed for the application of dental implants. The microstructures of Zr–20Ti and Zr–40Ti alloy were observed using optical microscope and transmission electronic microscope. The hardness and compressive tests were performed to evaluate the mechanical properties of the Zr–xTi alloys. The electrochemical behavior of the Zr–xTi alloys with and without 6% Ag was investigated in the acidified artificial saliva containing 0.1% NaF (pH = 4). For comparison, the electrochemical behavior of cp Ti was examined in the same condition. The results show that the quenched Zr–20Ti and Zr–40Ti alloy exhibit acicular martensite microstructures containing twin substructure. They display good mechanical properties with the hardness of ~330HV, the yield strength of ~1000 MPa and the strain to fracture of ~25% at room temperature. Adding 6% Ag to Zr–20Ti alloy enhances the passivity breakdown potential and the self-corrosion potential, but hardly affects the corrosion current density and the impedance modulus. 6% Ag in Zr–40Ti alloy distinctly increases pitting corrosion resistance, which is attributed the formation of thick, dense and stable passive film under the joint action of titanium and silver. In comparison with cp Ti, Zr–40Ti–6Ag alloy possesses the same good corrosion resistance in the rigorous oral environment as well as the superior mechanical properties.

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1. Introduction

Commercially pure titanium (cp Ti) has been used for dental implant owing to its excellent corrosion resistance, good biocompatibility and low elastic modulus [1]. However, the low strength of cp Ti increases the risk of fatigue fracture because the small diameter screw (≤ 3.5 mm) is used frequently as load-bearing part. It has been reported that at least 5% fracture of dental implants arises from fatigue over the last decades [2]. Increasing the strength

of the materials is an effective approach to prolong the service life of the implants. Ti–6Al–4V alloy has a moderate strength, but the toxicity of V and Al limits the dental application of this alloy. Zirconium is believed to be an ideal alloying element for titanium in biomedical applications. Zr–xTi (or Ti–xZr) alloys exhibit the similar strength and good osseointegration in animal and clinical studies to Ti–6Al–4V alloy [3–7]. In addition to this, Zr–Ti binary alloys present the other advantages, such as low melting point, low magnetic susceptibility, low linear expansion coefficient and less hydrogen absorption compared with cp Ti and Ti alloys [8], which make them become the important candidate materials as permanent prosthesis in dental or orthopedic treatments [9,10].

In the oral environment, the corrosion resistance of the metallic

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materials is crucial in evaluating their roles as dental implants because the released ions arising from the corrosion of metal or alloy stimulate the growth of the macrophage. The rough surface on the corroded metal also promotes the adhesion of various bacteria and the formation of plaque, which are considered as one of the factors inducing periodontitis and gingivitis [11]. The fatigue failure of dental implant has been reported to be closely related to the corrosion of the metal material [12,13]. Though zirconium metal offers superior corrosion resistance in acid or alkali solutions, it is susceptible to localized corrosion in the media containing chloride or fluoride. Increasing titanium content reduces the localized corrosion of the Zr–Ti alloys. Nevertheless, the pitting corrosion resistance of the Zr–Ti alloys is still inferior to cp Ti [14,15].

Recently, it has been reported that silver plays an important role in improving the corrosion resistance of titanium alloy in biological environment [16–19]. The beneficial effect of silver is attributed to its higher standard electrode potential ($E_{Ag}^{\circ} = 0.799V$) than titanium ($E_{Ti}^{\circ} = -0.98V$), which promotes the formation of stable titanium oxide film. In addition to this, the titanium–silver alloys show none or mild cytotoxicity in the agar overlay tests [20]. Till date, most of the investigations have been focused on the corrosion resistance of Ti–Ag binary alloy or Ti–Ag–Cu, Ti–Ag–Fe ternary alloys in neutral artificial saliva [21]. Seldom study deals with the corrosion behavior of the Zr–Ti (Ag) alloys in the acidic medium containing fluoride. It is known that the dental materials are attacked frequently by fluoride ions from toothpastes, mouth rinses, orthodontic gels and dietary supplements. Other factors, such as acidic foods, bacteria and inflammation in the oral cavity, accelerate the destruction of fluoride ions on the passive film of metallic materials.

The aims of this study were to examine the microstructures and mechanical properties of the Zr–xTi alloys. The electrochemical corrosion behaviors of the Zr–xTi alloys with and without silver in fluoride-containing acidified artificial saliva were evaluated. The effects of titanium and silver on the corrosion resistance of the Zr–xTi alloy were clarified. For comparison, the corrosion behavior of cp Ti was also investigated in the same corrosive condition.

2. Materials and methods

High purity titanium (99.97%), zirconium (99.98%) and silver foil (99.9%) were used to prepare Zr–20Ti, Zr–20Ti–6Ag, Zr–40Ti and Zr–40Ti–6Ag alloy. The button ingots were re-melted six times by using arc melting in argon atmosphere. The chemical compositions (wt%) of the four alloys were analyzed by EDS to be as follows: 20.8Ti; 21.3Ti, 5.8Ag; 39.6Ti; 40.7Ti, 6.1Ag; balance Zr. The ingots were hot forged into the rectangular slabs at 800 °C. After removing the oxidized layers, the small specimens for mechanical and electrochemical tests were machined by electric spark cutting, and then encapsulated in vacuum quartz tube for solution heat-treatment at 750 °C for 2 h followed by water cooling. Cp Ti plate was supplied by Zhipu Nonferrous Metals Processing Co. LTD (Baoji, China).

The phase compositions of the Zr–xTi (Ag) alloys were analyzed by means of X-ray diffraction analysis (SmartLab, Japan) using Cu K α radiation with the scan rate of 3°/min. The microstructures of Zr–20Ti and Zr–40Ti alloy were observed by optical microscope (Olympus GX71, China) and transmission electron microscopy (JEM-2100, Japan). TEM foils were prepared by mechanical grinding and two jet electrolytic polishing. Compression tests were performed on electronic universal testing machine (AG-Xplus100, Japan) with the specimens dimension of 5 × 5 × 8 (mm). The strain rate was 2 × 10⁻³ s⁻¹. Vickers microhardness of the Zr–xTi alloy was measured by a hardness tester (401 MVDTM, China) with a load of 100 g and a loading time of 30 s.

Electrochemical tests were performed on a three-electrode

electrochemical working station (Zahner, Germany). A saturated calomel electrode was used as reference electrode, a platinum plate as counter electrode and the specimens as working electrodes (1 cm² exposed area to the solution). The temperature of the electrochemical cell was maintained at 37 °C. According to ISO/TR10271 standard, the artificial saliva consisted of the following components: NaCl (0.4 g/l), KCl (0.4 g/l), NaH₂PO₄·2H₂O (0.78 g/l), CaCl₂·2H₂O (0.795 g/l), Na₂S·2H₂O (0.005 g/l), urea (1.0 g/l) and 1000 ml distilled water. In order to evaluate the corrosion properties of the experimental materials in the rigorous oral environment, 0.1% NaF was added into the artificial saliva solution. The pH value of the solution was adjusted to 4 by citric acid. All chemicals were provided by Guoyao Group Chemical Reagent Co. Ltd (Shenyang).

Before electrochemical tests, the specimens were ground with SiC abrasive paper up to 3000 grit, then ultrasonically cleaned in alcohol for 10 min, and finally immersed in the artificial saliva for 1 h in order to attain stable open circuit potential (E_{oc}) which was identified with potential fluctuation less than 20 mV. Electrochemical impedance spectra (EIS) were measured by applying a sinusoidal potential perturbation of 5 mV with a scan frequency ranging from 10⁻² Hz–10⁵ Hz in logarithmic increment at the open circuit potential. The impedance parameters were obtained by the Thales software. Potentiodynamic polarization curves (PPC) were recorded over the potential range of –2.0 V to +2.0 V at a scanning rate of 1 mV/s. The surface morphologies of the corroded specimens were observed by scanning electron microscope (SSX-550, Japan). For comparison, the PPC and EIS of cp Ti were measured in the same conditions. All tests were repeated three times on individual specimen to ensure the reproducibility of the measurements. Statistical significance was analyzed by means of one-way analysis of variance (ANOVA) with a confidence level of 95% ($p < 0.05$).

3. Results

3.1. Microstructures and mechanical properties

The optical micrographs of Zr–xTi alloys are shown in Fig. 1. Acicular martensite (α') is seen within the primary beta grain boundaries. The microstructures of Zr–20Ti and Zr–40Ti alloy have little difference except for the smaller primary β grain size of Zr–20Ti alloy due to the higher phase transformation temperature (T_{β}). Fig. 2 shows that the single α' phase was analyzed by XRD patterns in Zr–xTi and Zr–xTi–Ag alloys. The diffraction angles of the Zr–40Ti (Ag) alloys are higher than the ones of the Zr–20Ti (Ag) alloys, indicating the larger lattice distortion in the Zr–40Ti (Ag) alloys. The atomic radius of titanium is smaller than zirconium. Increasing titanium content in Zr–Ti alloy decreases the lattice interplanar spacing, which leads to the shifting of the diffraction peaks towards high angle. In addition to this, no retained β phase and TiAg or ZrAg intermetallic compound were found from the XRD patterns. The results are slightly different from the former publications, which reported the small amount of retained β phase in Ti–Zr alloys [22,23] and TiAg intermetallic compound in cast Ti–Ag alloy [24].

The compressive stress-strain curves of Zr–20Ti and Zr–40Ti alloy are shown in Fig. 3. Unlike the compression deformation behaviors of pure Ti or Zr, Zr–20Ti and Zr–40Ti alloy exhibit high work hardening rates after yield point. The yield strength of the two alloys is 1005 MPa and 1008 MPa, the ultimate strength is 1630 MPa and 1884 MPa, and the strains to fracture are 25.2% and 26.8%, respectively. Table 1 shows that the microhardness of Zr–20Ti and Zr–40Ti alloy reaches 329 ± 14 HV and 340 ± 17 HV, respectively. The above results indicate that the Zr-(20–40) Ti alloys exhibit good mechanical properties and workability at room

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