



Evidence of a defect structure in high fluence nitrogen ion implanted titanium and its effect on corrosion

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ABSTRACT

Commercially pure titanium grade 2 was implanted with nitrogen ions with a fluence of $1 \cdot 10^{18} \text{ cm}^{-2}$ and an accelerating voltage of 90 kV. Surface blisters, a typical sign of oversaturation, were found to be sputtered without re-forming. Massive cracking of the surface layer and the formation of surface pores were observed. Self-ordered pores covered the entire surface, including the area of sputtered blisters. Due to the surface defects, the implanted titanium surface had lower corrosion resistance than non-implanted titanium.

Introduction

Titanium materials are used in many areas of engineering applications in the aerospace, automotive, chemical and biomedical industries, due to their high strength, low specific weight and good stability in many corrosive environments [1–3]. However, a significant disadvantage of these alloys is the increased friction and wear, especially when they are used in moving parts [4]. The formation of nitride compounds is an effective approach aimed at improving their surface properties, e.g. hardness, friction coefficient and resistance to wear and corrosion [5,6]. Ion implantation is an advanced nitriding technique that enables nitrogen doping of titanium and titanium-based structures at low temperatures [7–9]. Implantation with fluence above $5 \cdot 10^{16} \text{ cm}^{-2}$ leads to nitrogen enrichment of ones and tens of atomic percent and the formation of a nitride mixture layer [10,11]. Many studies have confirmed increasing hardness and roughness of implanted titanium alloys with increasing fluence of nitrogen [12,13]. The corrosion behaviour of nitrogen-implanted titanium in an NaCl 3.5% solution in dependence on the applied fluence in the range up to $1 \cdot 10^{16}$ to $8 \cdot 10^{17} \text{ cm}^{-2}$ was investigated by Svaloni et al. [14]. They concluded that the corrosion potential and the passive current density shift to noble values with increasing nitrogen fluence, resulting in an improvement in corrosion resistance. The best corrosion resistance was found for titanium samples implanted with a fluence of $5 \cdot 10^{16} \text{ cm}^{-2}$. The authors attributed the worse corrosion behaviour at higher fluences to increasing roughness.

This paper brings new insights into the formation of surface defects and the porous structure of high-fluence ion-implanted titanium. The effect of the porous structure on the electrochemical behaviour is demonstrated.

Experimental

Commercially pure titanium grade 2 was used as a substrate material. Samples 3 mm in height and 10 mm in diameter were ground with a series of abrasive papers in the range from 80 to 2500 grit. Final polishing to a mirror-like finish was performed with $1 \mu\text{m}$ diamond paste. The polished samples were electrochemically etched and were then cleaned in ultrasound, using deionized water and isopropyl alcohol.

Nitrogen ion implantation was performed at the normal incidence angle of the ion beam, at an accelerating voltage of 90 kV and a fluence of $1 \cdot 10^{18} \text{ cm}^{-2}$. The ion current was measured using a Faraday cup, and the ion current density was below $2 \mu\text{A cm}^{-2}$, in order to keep the temperature of the samples below $100 \text{ }^\circ\text{C}$. The temperature of the sample was measured by a thermocouple located in the holder of the samples.

The depth distribution of the nitrogen was investigated by Glow Discharge Optical Emission Spectroscopy (GD-OES). The microstructure was measured by a Transmission Electron Microscope (TEM) and by X-ray diffraction (XRD). The implanted surface was analysed by a scanning electron microscope (SEM) and an atomic force microscope (AFM). The standard three-electrode setup was used for an investigation of the electrochemical behaviour. Potentiodynamic curve measurements ($-0.05 \text{ V}/E_{\text{ocp}}$ to $1 \text{ V}/\text{SSCE}$, 1 mV/s) were performed after twelve-hour stabilization of the open circuit potential in a physiological saline solution (9 g/L NaCl).

Results and discussion

The typical morphology and exposed grain boundaries of the non-

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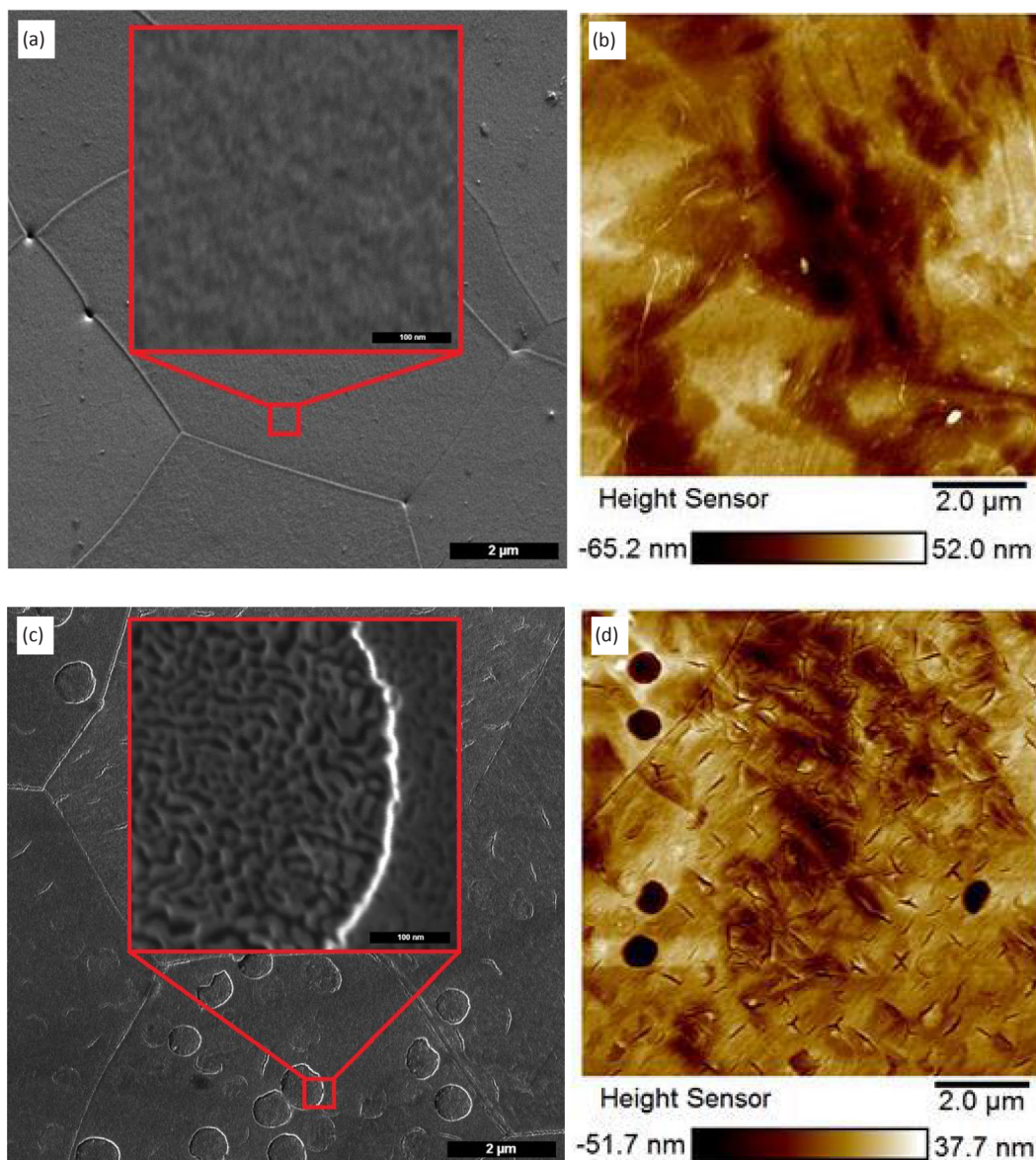


Fig. 1. SEM top view micrographs of the non-implanted titanium (a) and the high fluence ion implanted titanium (c) and AFM images of the surface of the non-implanted titanium (b) and the high fluence ion implanted titanium (d). The insert in Fig. 1c presents the formation of a porous structure.

implanted titanium are visible after electrochemical polishing, see Fig. 1a and b. The SEM micrograph in Fig. 1c shows surface defects of the high fluence ion implanted titanium in the form of cracks and circular craters. The AFM image (Fig. 1d) confirms the distribution and the nature of the observed defects. These results indicate the formation of surface blisters that are sputtered under the applied fluence. No closed blisters were found. Massive sputtering can be seen, which smoothed the surface craters without re-forming them. Surface sputtering of titanium under a comparable fluence is confirmed by Li et al. [12], who did not report the formation of surface defects, and also by Aghajani et al. [15], who implanted titanium with a fluence of $2.1 \cdot 10^{18} \text{ cm}^{-2}$. The insert in Fig. 1c shows the porous structure. The pores with a typical diameter up to 20 nm cover the entire surface, including the area with sputtered blisters. In Fig. 2, the cross-sectional TEM micrograph of implanted titanium documents the formation of a continuous surface layer with a porous structure. The pores are formed from the central region of the modified layer towards the surface. The observed surface morphology and the cross-sectional structure of the modified area prove nitrogen oversaturation and nitrogen effusion.

The XRD measurements were carried out under an incident angle of

0.3° , in order to analyse the phase composition of the modified surface layer more precisely. Fig. 3 shows X-rays diffractograms of the non-implanted and high fluence ion implanted titanium. In the diffractogram of the non-implanted titanium, all peaks correspond to pure titanium of the (hcp) α -Ti phase (ICSD #76265). The diffractogram measured on the implanted titanium shows that the α -Ti reflections have completely disappeared, and only reflections corresponding to the cubic (fcc) TiN (ICSD #1547) and Ti + N mixed interstitially located nitrogen in the α -Ti (ICSD #108614) were detected. The presence of interstitially located nitrogen in implanted titanium was observed by some researchers [11,16]. Phase analysis indicates that the surface layer of the titanium was completely transformed after ion implantation into a titanium nitride layer with a residual amount of interstitially located nitrogen.

Fig. 4 depicts the depth distribution of nitrogen in the implanted titanium. A maximum concentration higher than 60 at% is reached at a depth of approx. 77 nm. This result is in good agreement with the simulated distribution of nitrogen from the SRIM2013 code [17]. The observed high nitrogen concentration is the maximum that the titanium matrix can retain under the applied fluence. The evolution of the

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