PREPARING BETA-TITANIUM ALLOY FOR NITROGEN ION IMPLANTATION

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Abstract

The paper focuses on preparing beta-titanium alloy Ti39Nb for nitrogen ion implantation, a process in which the surface is bombarded with nitrogen ions. To successfully introduce ions into the titanium surface, it is crucial to have a high-quality surface preparation. In addition to grinding and polishing, removing the mechanically affected surface layer is necessary to eliminate residual stress introduction by pre-treatments. In this case, the affected surface layer was removed using ion sputter-etching. Several analytical methods were chosen to characterize the state of the titanium alloy surface after ion sputter-etching. These methods included X-ray diffraction to verify residual stress removal, confocal microscopy, and atomic force microscopy to examine surface morphology. For experimental comparison to Ti39Nb, alpha titanium grade 2 was utilized.

Keywords: Nitrogen ion implantation, beta-titanium alloy, surface preparation

1. INTRODUCTION

In recent years, there has been a growing interest in beta-titanium alloys as biotolerant materials in the biomedical industry [1]. These alloys are seen as an alternative to the widely used alpha-beta titanium alloy Ti6Al4V. Beta-titanium alloys have a lower modulus of elasticity closer to human bone and do not contain elements toxic to the body, such as vanadium and aluminum [2,3]. Implant titanium materials require hardness, wear resistance, biocompatibility, and corrosion resistance, which nitriding can enhance. Nitrogen ion implantation, a modern approach to nitriding, can improve these properties [4,5]. It involves bombarding the solid surface with accelerated nitrogen ions. The process of kinetic nitriding enables nitrogen saturation of the surface at low temperatures [6]. Ion implantation is especially beneficial for temperature-sensitive materials. It offers a significant improvement over conventional nitriding techniques that require high nitriding temperatures to be achieved. A high-quality surface preparation is necessary to study ion implantation-induced microstructural changes successfully. Grinding and polishing introduce residual stress into the mechanically affected surface. In [7], the stressed surface area of commercially pure titanium increased hardness, and by removing the mechanically affected area using several etching methods (electrochemical, chemical, and ion sputter-etching), it was determined that residual stress and hardness were decreased. The residual stress can affect the nitriding process and the correct understanding of the interaction of the incident ions with atoms of the implanted surface.

2. EXPERIMENTAL PART

The selected beta-titanium alloy for the experiment was Ti39Nb. Ti39Nb beta titanium alloy has been used in this study. The commercially pure titanium grade II (alpha titanium structure) was chosen for comparison with the beta titanium alloy. The samples of both materials were cylindrical, with a diameter of 14 mm and a height of 8 mm; typical sample is shown in **Figure 1**. These samples were mirror polished on an Automet250 polisher using a ChemoMet pad and colloidal silica named MasterMet2. Subsequently, after polishing, the samples were ion sputter-etched to remove the surface layer to reduce the residual stress introduced by the previous pretreatments. **Figure 1** shows the apparatus used for ion sputter-etching, with an inside view of the vacuum chamber. A Kaufman ion source with an MPS-3000FC IONTECH, INC control module was used. Ion sputter-etching was performed by bombarding the surface with argon ions accelerated to an energy of 700 eV. The ion sputter-etching rate of 1,1-1,2 Å/s was measured using a quartz crystal coated with titanium layer. A controller from MAXTEK, INC processed the output signal. The flow rate of argon gas was 7,2 ml/min. The thicknesses of the layers removed from both materials (Ti39Nb, Ti grade II) were approximately 7 μm.

Figure 1 Ion sputter-etching apparatus (left) and view of the inside of the vacuum chamber (right); 1 – titanium-coated quartz crystal for ion sputter-etching rate measurement, 2 – samples placement, 3 – Kaufman ion source, and 4 – sample of Ti39Nb after ion sputter-etching.

The samples (before and after the process of ion sputter-etching) were analyzed using X-ray diffraction, atomic force microscopy (AFM), and confocal microscopy. X-ray diffraction (XRD) was used to verify the removal of the mechanically influenced layer from previous treatments and reduce residual stress. The XRD measurement was conducted using an X'Pert PRO diffractometer with a Co anode, parallel beam geometry, a parallel beam mirror in the incident beam, and a parallel beam collimator in the difracted beam (with an acceptance of 0,09°). The incident angle was set at 3°. Subsequently, the samples were analyzed using confocal and atomic force microscopy (AFM); these methods determined the materials‘ surface morphology and microstructure. A LEXT-OLS 5000 confocal microscope was used to analyze the microstructure of the materials. The measured area of the microstructures was 1280x1280 μm. For atomic force microscopy, a NanoWizard3 microscope was utilized. AFM measurement was performed in contact mode with the CONTV-A probe, which has an Al layer to enhance the laser response. The goal was to assess the morphology and roughness of the analyzed surfaces. To determine surface roughness (parameter Ra), an area (100x100 μm) was measured in 10 locations on each sample, and the images were evaluated in JPK Data Processing.

3. RESULTS

The left side of **Figure 2** displays the microstructure of Ti grade II, while the microstructure of Ti39Nb is shown on the right. The difference between Ti grade II and Ti39Nb is in their grain size. Ti grade II has a fine-grained microstructure (grain size up to 50 μm), while Ti39Nb significantly increases the grain size (grain size in the range of hundreds of microns) due to its niobium content. The phases of individual materials were determined using X-ray diffraction. **Figure 3** displays the diffractogram for two materials - titanium grade II and Ti39Nb. The analysis of titanium grade II revealed the presence of hexagonal close-packed (HCP) crystal lattice of titanium. The Ti39Nb alloy has been found to contain a body-centered cubic (BCC) lattice. X-ray diffraction confirmed the reduction of residual stress in the surface layer by removing the mechanically affected area from pretreatment. The residual stress in the surface layer can be evaluated using the measured microstrain by X-ray diffraction. The microstrain introduced into the surface layer before ion sputter-etching was 0,04 % for Ti39Nb and 0,10 % for Ti grade II. After ion sputter-etching, the microstrain dropped to 0,001 % for Ti39Nb and 0,02 % for Ti grade II. A significant decrease in microstrain of up to forty times was recorded on the Ti39Nb sample; for Ti grade II, it was five times. The decrease in microstrains indicates that ion sputter-etching effectively reduces residual stress. The difference in microstrain reduction between Ti39Nb and Ti grade II could be explained by the different ion sputter-etching rates, which depend on individual grain size and crystallographic orientation.

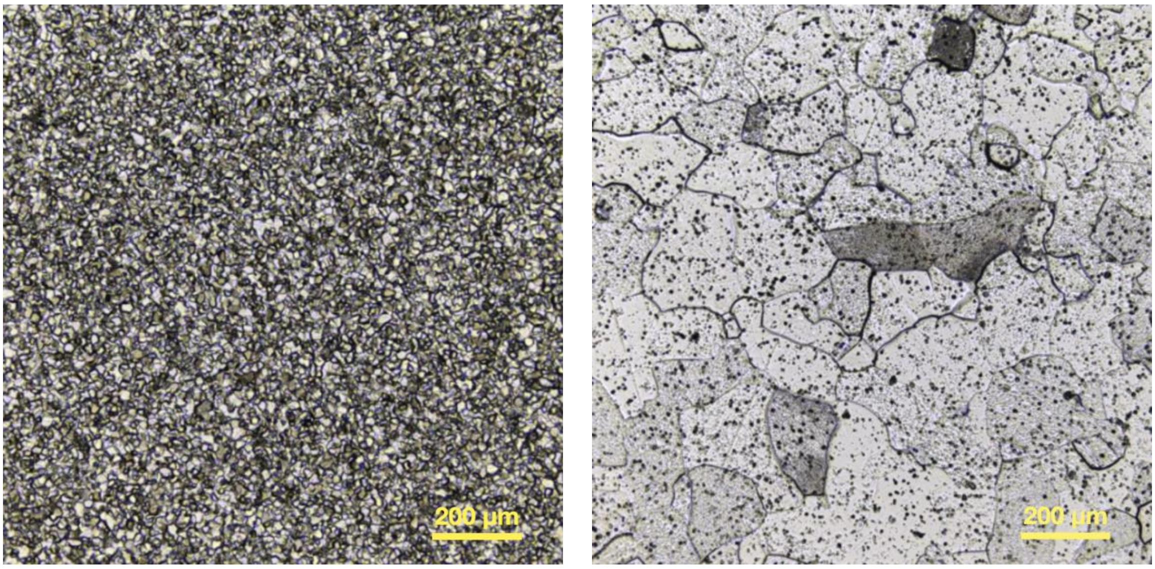

Figure 2 Microstructures of Ti grade II (left) and Ti39Nb (right).

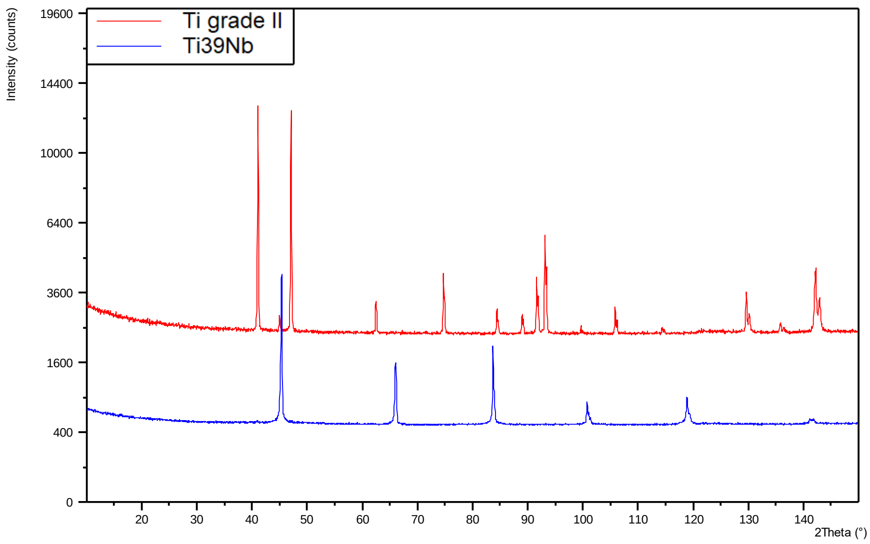

Figure 3 Diffractogram of titanium grade II and Ti39Nb.

The effect of ion sputter-etching on the morphology and surface roughness was analyzed using atomic force microscopy. **Figure 4** shows the surface morphology of polished and ion sputter-etched Ti39Nb and ion sputter-etched alpha titanium grade II. A very smooth surface, can be observed after polishing (**Figure 4 left**). There are visible light spots that idicate a significant height difference. These objects probably represent surface impurities, polishing residues or dust particles. The ion sputter-etched surface of Ti39Nb displays significant uneven etching of individual grains and height differences between them, as shown in **Figure 4** (middle), where a grain boundary separates two differently colored grains. **Figure 4** (middle) documents pitting etching, which may have been caused by material inhomogeneity. For Ti grade II, the etched fine-grained structure is shown. In **Figure 5**, the impact of ion sputter-etching on surface roughness is demonstrated. The surface roughness of polished state of Ti39Nb is 6 nm, compared to 200 nm for ion sputter-etched Ti39Nb and 482 nm for ion sputter-etched Ti grade II. Surface roughening in polycrystalline materials is caused by the different rates of sputtering of individual grains depending on their crystallographic orientation, which causes height differences between them.

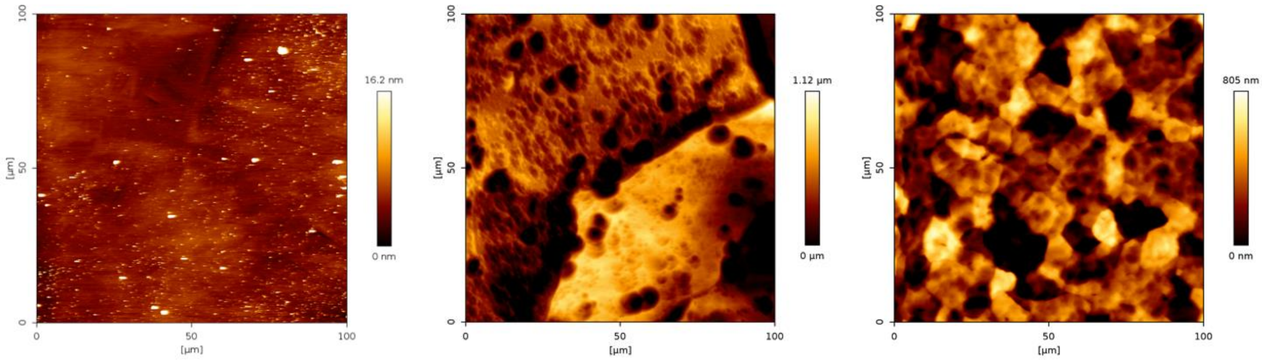

Figure 4 AFM images: polished state of Ti39Nb (left), ion sputter etched Ti39Nb (middle), and ion sputter-etched Ti grade II (right).

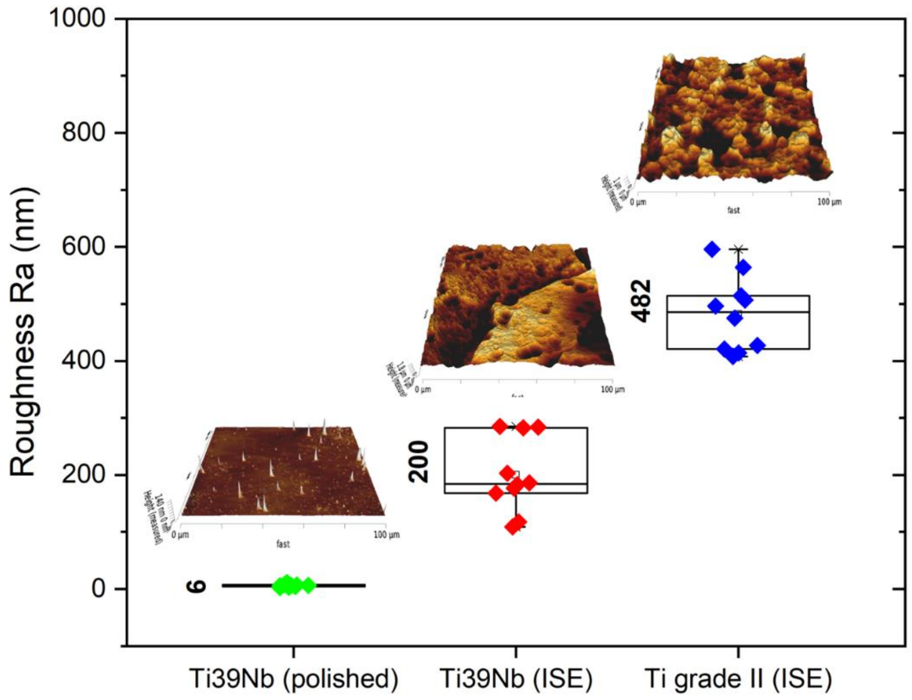

Figure 5 Roughnesses of polished and ion sputter-etched (ISE) states with 3D images from AFM.

4. CONCLUSION

Removing the 7 μm thick surface layer reduced microstrains in the surface area. For Ti39Nb alloy, it decreased from 0,04 % to 0,001 %, and for Ti grade II, it decreased from 0,10 % to 0,02 %. This reduction confirmed the positive effect of ion sputter-etching on removing residual stress introduced into the surface from previous mechanical pretreatments. Ion sputter-eching significantly increases surface roughness. Polished states vary in units of nanometers (Ti39Nb has 6 nm), while ion sputter-etched Ti39NB has a roughness of 200 nm, and ion sputter-etched Ti grade II has a roughness of 482 nm.

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