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TABLE OF CONTENTS

TECHNIQUE

EDUCATION

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MANAGEMENT

Nanoindentation Study of Ultrafine-Grained Titanium-Based Materials

D.R. Barjaktarević [a], M.P. Rakin [a], B.I. Međo [a], V.R. Đokić [a]

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Abstract

The commercially pure titanium (cpTi) and especially titanium (Ti) alloys are materials increasingly used in orthopaedic and dental implants. In order to enhance the implant material properties, Ti-based materials may be modified by severe plastic deformation (SPD) methods. One of the most attractive SPD methods is high-pressure torsion (HPT), as a method for obtaining submicron-sized grains, with the aim to improve, among others, mechanical properties of metallic materials. In the present study, ultrafine-grained titanium (UFG cpTi) and ultrafine-grained Ti-13Nb-13Zr (UFG TNZ) alloy samples were obtained by high pressure torsion (HPT) under a pressure of 4.1 GPa with a rotational speed of 0.2 rpm up to 5 rotations at room temperature. The homogeneity of the material was determined by using Vickers microhardness tester and analysing the obtained microhardness profile along the samples diameters. The results show that materials are reasonably homogeneous after HPT processing. The aim of this study is to determine the mechanical behaviour of the commercially pure titanium and titanium alloy before and after HPT processing using the nanoindentation technique. Obtained results show that ultrafine-grained materials have lower modulus of elasticity than coarse-grained (CG) materials, which means that the values are closer to those of bones, making the discontinuity of mechanical properties at the bone-implant interface less pronounced. On the other hand, UFG materials have higher values of nanohardness than CG materials.

Keywords: biomaterials, high pressure torsion, modulus of elasticity, nanoindentation, titanium alloys

1. INTRODUCTION TO BIOMATERIALS

The commercially pure titanium (cpTi) and especially titanium (Ti) alloys are materials increasingly used in orthopaedic and dental implants [1]. The first generation of titanium alloys has had good clinical results, but the modulus of elasticity of this alloy (110 GPa) is much higher than that of human bone (10-30GPa) [2]. The newly developed $\alpha+\beta$ - type and β - type titanium alloys have better characteristics such as higher corrosion resistance and lower modulus of elasticity, which is closer to that of a bone [2-4]. One of these alloys is Ti-13Nb-13Zr. In order to enhance the implant material properties, commercially pure titanium and titanium alloy may be modified by different techniques, among others by severe plastic deformation (SPD) methods [5]. One of the most attractive SPD methods is high-pressure torsion (HPT) [6, 7], which is a method for obtaining submicron-sized grains, with the aim to improve, among others, mechanical properties of metallic materials, Fig. 1. The possible inhomogeneity of

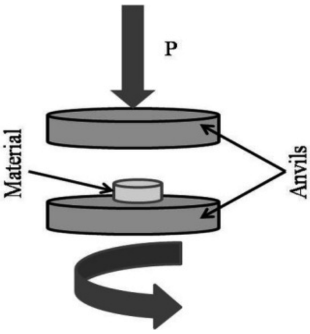

Fig. 1. Schematic of the HPT process

metallic materials after HPT process is a very important issue that should be checked. In this study, inhomogeneity of materials was determined by using Vickers microhardness tester and analysing the obtained microhardness profiles along the samples diameters.

Behaviour of the metallic biomaterials is also regulated by surface properties, which is a crucial factor in interactions of the implant material with the surrounding tissue. Because of that, in this study nanoindentation technique was used in order to determine the modulus of elasticity and nanohardness of the commercially pure titanium after HPT processing and titanium alloy before and after HPT processing.

2. MATERIALS AND METHODS

The commercially pure titanium (cpTi) and Ti-13Nb-13Zr alloy (TNZ) in the initial state were disk-shaped samples with a diameter of 28 mm and thickness about 2.2 mm. The samples were subjected to HPT process in order to obtain ultrafine-grained cpTi (UFG cpTi) and ultrafine-grained Ti-13Nb-13Zr (UFG TNZ) alloy. HPT was performed at room temperature with a device, which has two anvils with circular flat-bottom depressions at the centre. Each disc was placed between the anvils, a pressure of 4.1 GPa was applied and the disc was torsionally strained through rotation of the lower anvil. Strains were imposed on the discs by processing at a constant speed of 0.2 rpm through 5 revolutions. The obtained samples of UFG cpTi and UFG TNZ alloy were disc-shaped with a diameter of 34 mm and thickness approximately 0.7 mm.

2.1. Microhardness measurements

The Vickers microhardness (HV) was measured on the surfaces of the discs samples using TIME HVS-1000 microhardness tester machine under a load of 4.903 N and a dwell time of 5 s with a distance of 1 mm between each indentation. The measurements were performed along the diameters on each sample. The value of HV for each indentation was estimated from the average of four hardness measurements. Before measurements, samples were wet-ground with 150 μm to 4000 μm grit silicon carbide paper and polished.

2.2. Mechanical characterization

The nanoindentation test was performed on the surfaces of the disc samples using Nanoindenter G200, Agilent Technologies. The test was controlled using total displacement of 2000 nm. Ten measurements were made on each sample and the mean values were calculated. All tests were applied to assess the surface mechanical properties of materials after HPT processing.

3. RESULTS

The HV of UFG Ti-13Nb-13Zr alloy and UFG cpTi as a function of the diameter of the disk - shaped samples before and after HPT processing are presented in Fig. 2.

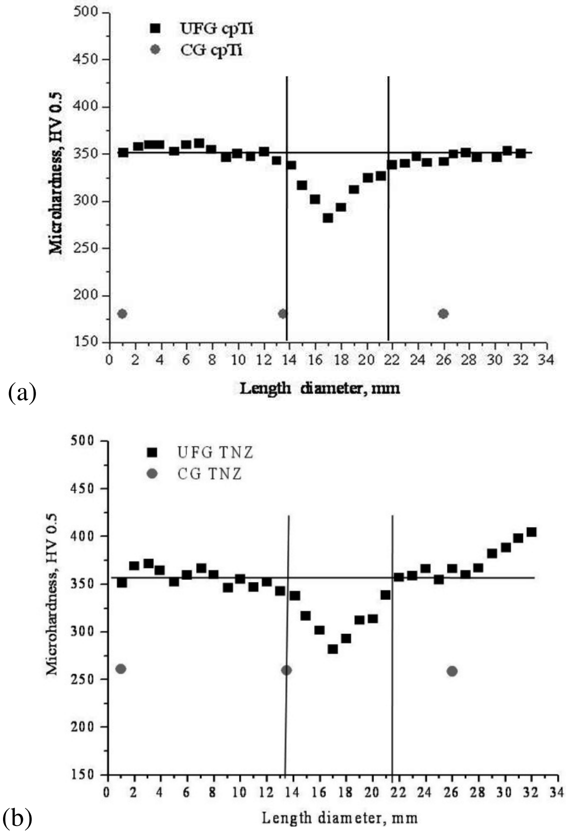

Fig. 2 Vickers microhardness of UFG cpTi (a) and UFG Ti-13Nb-13Zr alloy (b) as a function of the sample diameter

As can be seen in Fig. 2a, the microhardness value of cpTi significantly increases after HPT processing, from 180 HV in the initial state to an average of 350 HV. Also, the microhardness of the Ti-13Nb-13Zr alloy was significantly enhanced by HPT processing such that the average hardness increased from 259.73 HV for the coarse-grained (CG) sample to 350.34 HV, Fig. 2b. Two important conclusions can be drawn from Fig.2. First, the microhardness of cpTi and Ti-13Nb-13Zr samples increases significantly through HPT processing by comparison with the CG one. Second, the HPT-processed cpTi and Ti-13Nb-13Zr samples processed through 5 turns have lower microhardness values in the centre (between two horizontal line in Fig. 2), in comparison with the other parts of the samples. For the UFG Ti-13Nb-13Zr alloy microhardness values are changed from 301.7 HV in the centre to 342.9 HV to the edges, while for the narrow region around the one edge, microhardness value is about 400 HV (this part of sample is cut and is not used in experiments). For the UFG cpTi microhardness values are changed from 300 HV to 350 HV.

Increase of the value of microhardness from the centre toward the edges of the sample is related to the fact that the value of torque is zero in the centre increasing in the radial direction and reaches its maximum at the edges of the samples during the process of HPT. Also, due to the variation in shear strain, γ, across each disc during HPT processing, value of γ at different positions of the disc is estimated by [8]:

$$\gamma = \frac{2\pi NR}{h} \qquad (1)$$

where N is the number of revolutions, R is the radial distance from the centre of the disc and h is the disc thickness.

For the specimen processed through 5 turns, the obtained microhardness values, outside the narrow region near the disc centre (between two vertical lines) and outside the narrow region around the one edge of disc (from 31 mm to 34 mm in diameter length), are narrowly distributed from point to point variation (along the horizontal line) of ± 16 HV, see Fig. 2b. So, it could be said that the reasonably uniform microstructure is achieved after 5 turns of HPT processing, with the exception of a narrow area around the centre of the sample with diameter of about 4 mm and a narrow area around one edge of the disc from 31 mm to 34 mm in diameter length (these parts of the sample are not used in experiments).

The microstructure characterisation of ultra-fine grained titanium and titanium alloys is very difficult to do using usual microscopes, like optical microscope or scanning electron microscope. The microstructure characterisation of ultra-fine grained metallic materials is usually done on a transmission electron microscope, and the available literature shows the homogeneous microstructure after the HPT process [9, 10].

The microhardness value of Ti-13Nb-13Zr, in the initial state, has no large deviations, which indicates a homogenous microstructure. In point of fact, decreasing of the grain size with HPT processing in UFG Ti-13Nb-13Zr alloy leads to a significant increase in the microhardness, while the microstructure remains homogeneous (except in the narrow zone around the centre of the sample and the narrow zone around the one edge of the sample). The results are in agreement with I. Dimić et al. [10], who showed that the average microhardness value of Ti-13Nb-13Zr alloy increases from 230 HV in the initial state to 300 HV, 355 HV and 360 HV after HPT deformation up to 7.8 GPa and ¼, 1 and 5 rotations, respectively. They concluded that microhardness increases with the increase of the number of rotations in the case of this Ti-based alloy. The results in this study confirm the potential of using HPT processing to achieve reasonably homogeneous microstructures.

After the analysis of materials homogeneity, examination of mechanical surface properties was done using nanoindentation (as mentioned previously, the region in the centre of UGF processed samples is excluded from the tests). The displacement during nanoindentation was 2000 nm for each sample, and maximum mean values of load on the sample were approximately 200 mN, 250 mN and 375mN for CG TNZ, UFG TNZ and UFG cpTi, respectively. Fig. 3 represents loading-displacement curves obtained during the nanoindentation tests. Each curve consists of the loading part, the dwell period at the maximum load of the indentation and the unloading part. The diagram shows the change in depth with the dwell period at the maximum load.

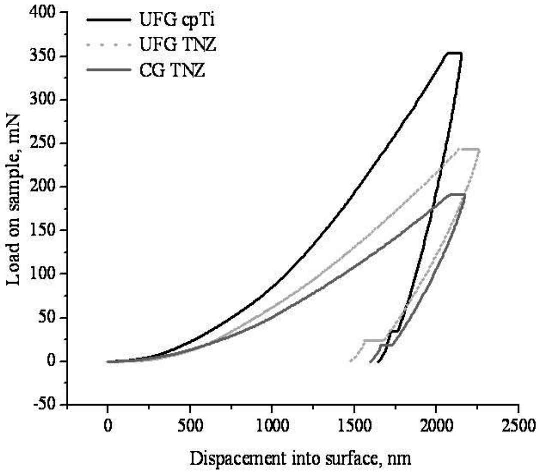

Fig. 3. Loading-displacement curves for examined materials

The difference between the loading part and the unloading part on the diagram indicates the presence of the permanent (plastic) deformation. The mean values of surface mechanical characteristics, nanohardness and modulus of elasticity, obtained from nanoindentation test, are presented in Table 1 and Fig. 4 and 5.

Table 1. Mean values of mechanical properties

Materials	UFG cpTi	CG TNZ	UFG TNZ
Modulus of elasticity, GPa	104.56	61.301	59.987
Nanohardness, GPa	3.54	2.393	2.691
Nanohardness, HV	361	244	274

The results show that the modulus of elasticity is lower after HPT process, Fig. 4. UFG TNZ alloy has the lowest modulus of elasticity, which indicates that it is the most acceptable material for the metallic implant. The lower value of modulus of elasticity and closer to that of a bone is one of the crucial factors in accepting the implant material from the surrounding tissue, and reduces the possibility of slow disappearance of bone in contact with the implant - “shielding effect” [11]. The commercially pure titanium typically has a modulus of elasticity of 120 GPa after the nanoindentation test [12]. This indicates that UFG cpTi has lower modulus of elasticity than the pure titanium.

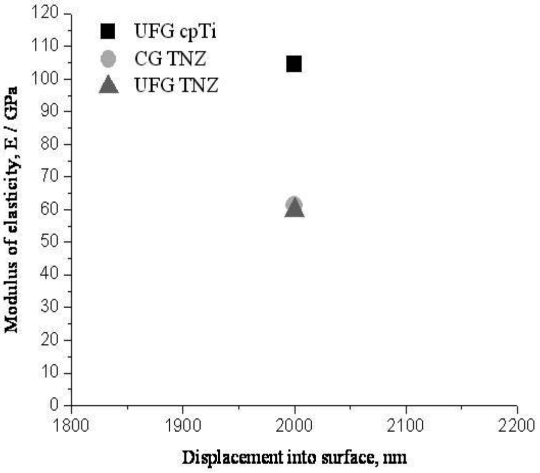

Fig. 4. Modulus of elasticity obtained using nanoindentation for examined materials

On the other hand, the nanohardness values of ultrafine-grained materials are higher than those obtained for coarse-grained materials, Fig. 5.

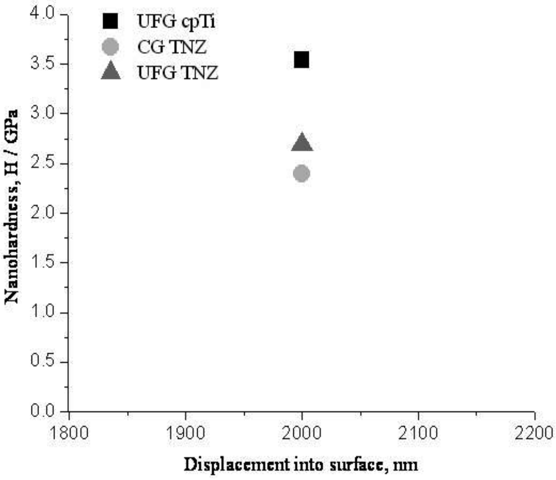

Fig. 5. Nanohardness values obtained using nanoindentation for examined materials

4. CONCLUSIONS

The results show that high pressure torsion is an acceptable process for metallic biomaterials. The materials after HPT process show adequate homogeneous microstructure. The values of microhardness are higher after HPT process. The nanoindentation tests show that UFG materials have lower modulus of elasticity, which make them more acceptable as metallic biomaterials than CG materials. Future examination will include the formation of a nanotubular oxide layer on the surface of the above mentioned materials and the examination of surface mechanical properties using the nanoindentation test.

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