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Formation of a defective surface layer on a superelastic Ti–18Zr–15Nb alloy for medical purposes by ultrasonic treatment

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The use of high-intensity ultrasound offers a simple, environmentally friendly and versatile tool for surface modification of metallic materials. This work is devoted to studying the effect of ultrasonic treatment parameters on the formation of a porous layer on the surface of a superelastic Ti–18Zr–15Nb alloy. Scanning electron microscopy analysis revealed that when optimal ultrasonic treatment parameters are selected, a homogeneous region with a porous structure is formed on the alloy surface.

Keywords: ultrasonic treatment, superelastic alloys, porous structure.

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Over the past decades, ultrasonic treatment has loomed large among the methods for modifying the surface of metallic materials. Studies show its effectiveness for titanium-based alloys, aluminum-based alloys, heat-resistant alloys and carbon steels [1]. Ultrasonic action in a liquid medium generates cavitation bubbles, the collapse of which creates localized areas of high pressure and temperature. The mechanical impulse forms a defective porous layer on the metal surface [2].

Ultrasonic treatment is a highly effective alternative to traditional methods of metal surface modification [3]. Its key benefits are high productivity, equipment availability and ease of process deployment.

The formation of a defective porous structure on the surface of metal implants using ultrasonic treatment deals with two key issues. First, high surface roughness promotes increased adhesion and cell proliferation, which accelerates osseointegration and reduces the risk of product rejection [4,5]. Second, in certain conditions, such a surface can exhibit antibacterial properties, mechanically damaging bacterial cells and preventing the formation of biofilms [6,7], which is critically important for the prevention of implant-associated infections. In addition, the defective surface layer created by ultrasound can be used to incorporate antibacterial particles into it.

Titanium alloys are widely used in medicine due to their high strength to corrosion resistance ratio and low modulus of elasticity [8,9]. In particular, the superelastic alloy Ti–18Zr–15Nb is considered one of the most promising materials for bone implants [10,11]. Its benefits include increased biocompatibility, high crystallographic reversible deformation resource ($\sim 6\%$) and increased service life under cyclic loads [12].

This study is aimed at establishing the possibility of forming a surface porous layer of Ti–18Zr–15Nb alloy by ultrasonic exposure in an aqueous environment.

A superelastic alloy Ti–18Zr–15Nb was taken as a surface modification object. Cylindrical specimens 2 mm high and 10 mm in diameter were taken. The surface of the specimens was prepared by successive grinding with abrasive paper (SiC grain size from P320 to P4000) and subsequent polishing using Al_2O_3 suspension (dispersity $1\text{--}3\text{ }\mu\text{m}$).

The experimental setup is shown in Fig. 1. The treatment was carried out in a glass laboratory beaker, which was placed in a water-cooled metal casing to prevent heating and maintain the temperature of the environment at about $20\text{ }^\circ\text{C}$. To ensure reliable fixation of the substrate, a polymer holder was designed and manufactured. It was attached to the bottom of the glass and prevented the specimen from shifting under the impact of ultrasonic treatment.

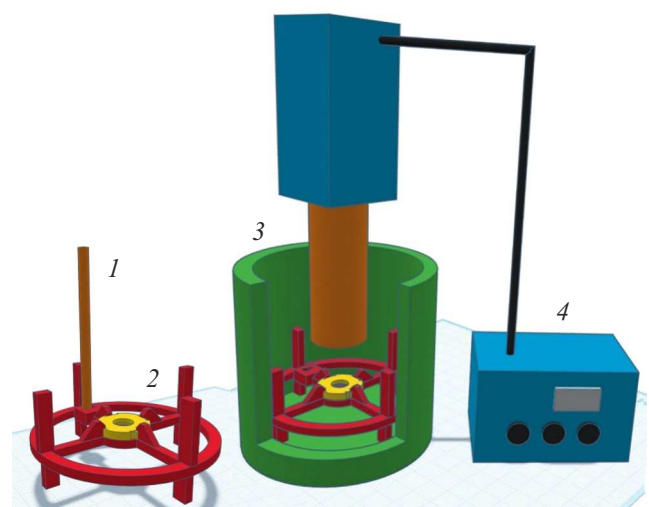


Figure 1. Design of experiments. 1 — specimen holder, 2 — specimen, 3 — continuous cooling container, 4 — ultrasound homogenizer.

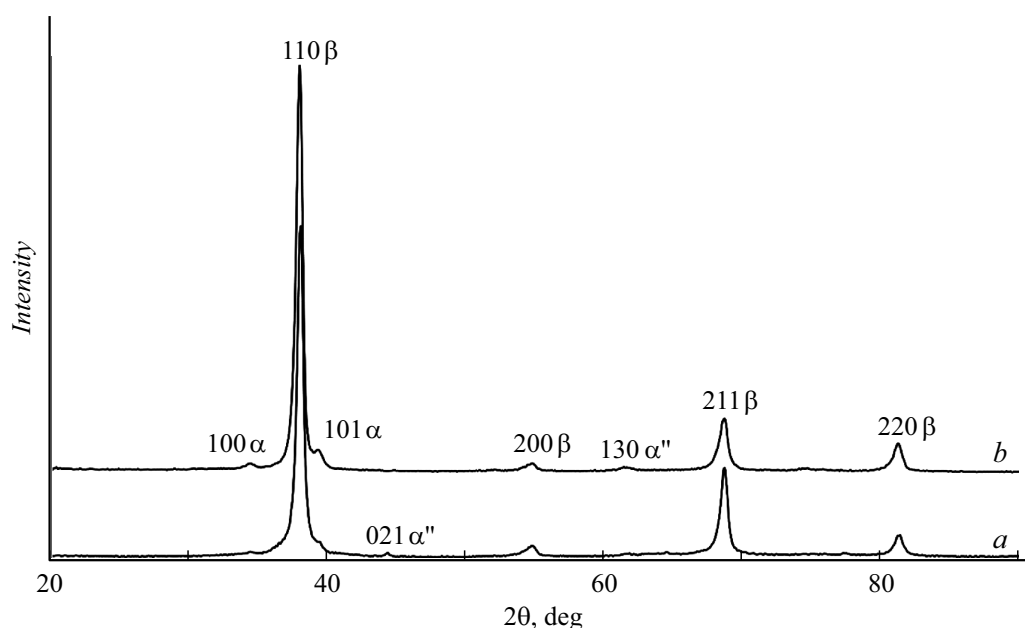


Figure 2. Diffraction patterns of the alloy specimens in the initial state (*a*) and after surface treatment using ultrasonic exposure (*b*).

Distilled water (volume 500 ml) was used as a working medium. In each experiment, the specimen was immersed in water and the distance from its surface to the emitter was varied by moving the sonotrode on a stand. The sonotrode immersion depth was from 3 to 10 cm. A titanium rod with a diameter of 40 mm and a smooth working surface was used as an emitter. The ultrasonic treatment was made for 30 min using Hielscher Ultrasound Technology UIP 1000 hdT generator at a power of 350 W and amplitude 20 % (this corresponds to the sonotrode oscillations to a value of $\sim 5\ \mu\text{m}$). The sonotrode was aligned axially with the specimen at a distance of 2, 5 or 7 cm from its surface. The distance step was chosen with a reserve to cover the possible range of interference effects of ultrasonic waves.

Phase composition was analyzed using diffractometer Rigaku Miniflex 600 ($\text{CuK}\alpha$ -radiation) in the range of $2\theta = 20\text{--}90^\circ$ with a step of 0.1° and 3 s exposure time. The surface morphology was examined using a Scios (FEI) field emission scanning electron microscope in secondary electron mode. The analysis of pore size distribution was carried out using the random intercept method.

Fig. 2 shows the diffraction patterns of the initial (*a*) and ultrasonically treated (*b*) alloy. The main phase in all specimens is the body-centered cubic β phase. Traces of the equilibrium α -phase (due to the alloy production technology) and the martensitic α'' -phase are also observed. The presence of a metastable martensite phase is caused by strain hardening of the alloy surface during mechanical polishing [13]. According to the diffraction patterns shown, ultrasonic treatment does not affect the phase composition of the material, in particular, it does not lead to a rise of the martensite phase.

The findings from the study of the surface morphology of materials modified by ultrasonic exposure are illustrated in Fig. 3. It was found that the nature of the formed porous layer depends on the distance between the sonotrode and the specimen.

Fig. 3, *a* shows the specimen surface treated at a distance of 2 cm from the sonotrode. When analyzing the surface using low magnification, it was found that defects were formed predominantly on the periphery of the specimen, generating a ring-shaped area around the central part, which retained its original state after grinding. The interface between the defective and original areas is clearly visible (Fig. 3, *d*). The defective structure in the treated area is primarily manifested in the form of irregularities on the surface, and is also represented by rare submicron pores.

The specimen surface after modification at a distance of 7 cm is shown in Fig. 3, *c*. As in the case of processing at a distance of 2 cm, defects are formed not over the entire surface, but mainly along the periphery, forming a ring zone. However, the nature of the defective structure is not changed. An increase in the size of pores and irregularities is observed: the pores become of the micron size, and the surface irregularities, albeit similar in type to those observed for the specimen treated at a distance of 2 cm, become more extended and wider.

The surface of the specimen treated at a distance of 5 cm from the sonotrode is shown in Fig. 3, *b*. In this case, unlike the previous modes, almost the entire surface of the specimen (except for the peripheral zones adjacent to the holder) was subjected to uniform modification. The defective structure is represented mainly by clusters of pores in the micron and submicron ranges. In addition, submicron linear defects are observed, lining up into characteristic

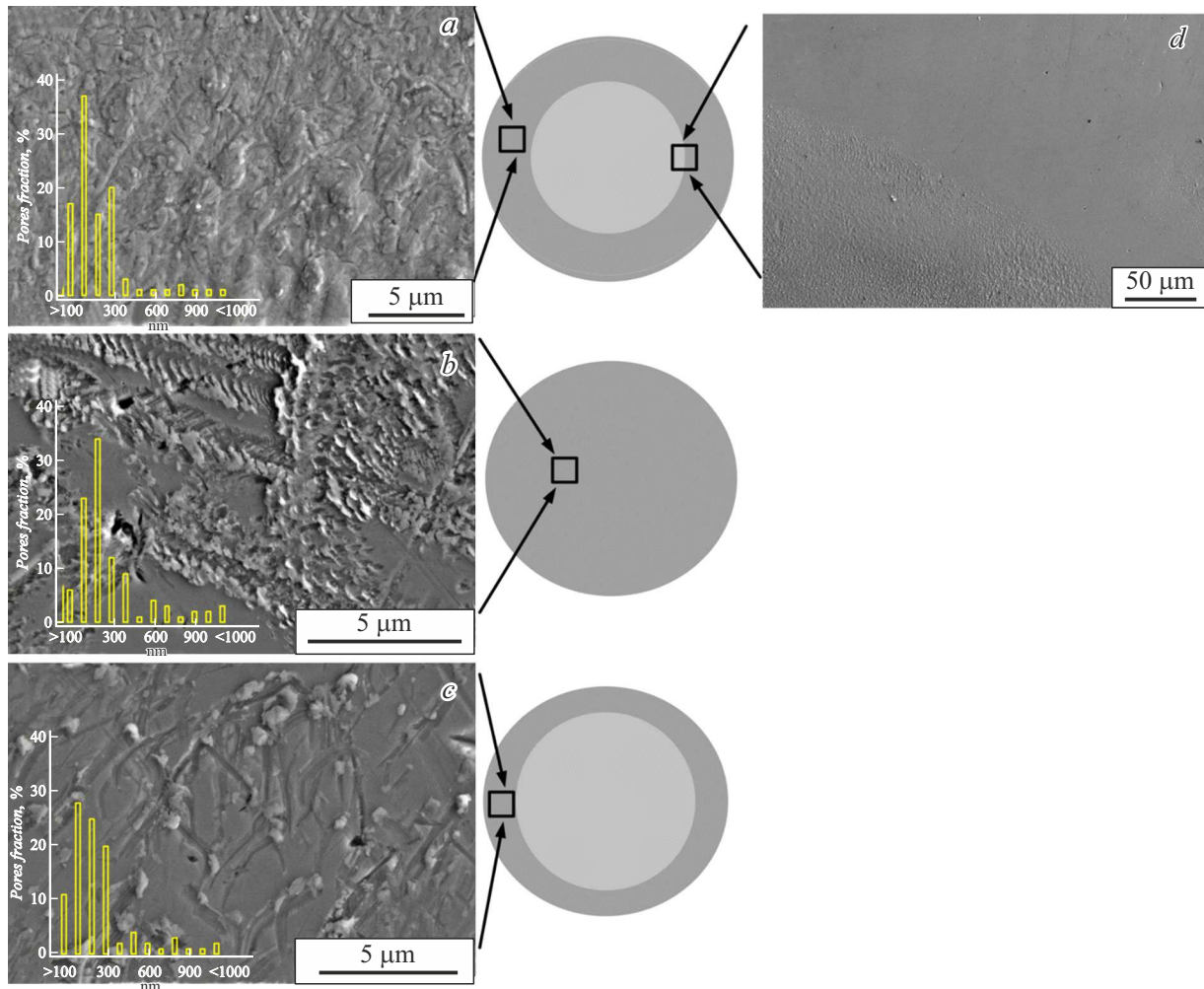


Figure 3. Micro-photos of the specimen surface and histograms of pore size distribution after ultrasonic irradiation at a distance of 2 (a, d), 5 (b) and 7 cm (c).

stripes. Due to this treatment it became possible to achieve the most homogeneous and evenly distributed porous structure over the entire surface. The pore size distribution shows that the largest proportion of pores are in the size range from several tens of nanometers to 500 nm.

The findings demonstrate that varying the distance between the sonotrode and the specimen surface in the range from 2 to 7 cm is an effective way to control the morphology of the formed porous layer. The observed nonlinear dependence of morphology and homogeneity of the porous layer on the emitter–specimen distance indicates the complex nature of the interaction of the ultrasonic field with the specimen surface. Uniform modification of the surface at a distance of 5 cm from the sonotrode, in contrast to the peripheral nature of the processing at 2 and 7 cm, can be associated with the spatial distribution of the acoustic pressure at the specimen surface, which is determined by both the parameters of the radiating system and the geometry of the working chamber.

Thus, within the framework of this study, the possibility of modifying the surface of the superelastic alloy

Ti–18Zr–15Nb by ultrasonic action, leading to the formation of a defective structure, was demonstrated. It was shown that the type and distribution of pores depended on the process parameters. It was found that this treatment had no impact on the phase composition of the material. It was proved that placing the sonotrode at a distance of 5 cm from the specimen surface allowed to form the most uniform defective surface with controlled morphology (pores and linear defects), uniformly distributed over the entire area of the specimen. This treatment is promising for the formation of surfaces capable to raise the biocompatibility of the alloy.

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Conflict of interest

The authors declare that they have no conflict of interest.

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