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Original article

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STRUCTURE AND PROPERTIES OF SURFACE LAYER OF TiNi ALLOY SUBJECTED TO ION-PLASMA- AND ULTRASONIC TREATMENT

Abstract. The paper presents the results of research on morphology, elemental composition, microhardness, corrosion properties of the surface layer of TiNi alloy subjected to ion-plasma (vacuum-arc method) deposition of TiN coating and ultrasonic treatment (UT) with different number of passes (*n*). The SEM method showed that ultrasonic treatment provides a significant reduction in the amount of the droplet phase on the TiN coating surface. The surface discontinuity of TiN coating at local points was observed with an increase in the number of passes during ultrasonic treatment. The effect of combined processing methods on the microhardness of TiNi sample was studied. It was shown that the synergistic effect can be observed for two hardening methods. The combined strengthening method increased the microhardness of TiNi alloy (1.6 GPa in the as-received state): due to the deposition of a TiN coating – up to 10.9 GPa, due to subsequently ultrasonic treatment – up to 14.5–18.4 GPa depending on the number of passes. For UT + TiN scheme, it was shown that the open circuit potential E_{corr} was about –250 mV which is practically independent of the number of passes and determined by the potential value of TiN coating. For TiN + UT scheme, it was found that with an increase in the number of passes, the value of E_{corr} shifts towards more negative values approaching the open circuit potential value of the TiNi sample in the as-received state (–350 mV). The analysis of Scanning Vibrating Electrode Technique (SVET) results showed high electrochemical compatibility of substrate (TiNi) and coating (TiN) materials in a chloride environment with minimal current density fluctuations for the samples subjected to UT + TiN and TiN + UT (n = 1). The proposed method for TiNi alloy treatment according to TiN + UT scheme (n = 1) promotes an improvement of surface morphology and corrosion resistance.

Keywords: TiNi, TiN coating, vacuum-arc deposition, ultrasonic treatment, microhardness, open circuit potential

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Conflict of interest: the authors declare that there is no conflict of interest.

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Оригинальная статья

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СТРУКТУРА И СВОЙСТВА ПОВЕРХНОСТНОГО СЛОЯ TiNi-СПЛАВА, ПОДВЕРГНУТОГО ИОННО-ПЛАЗМЕННОЙ И УЛЬТРАЗВУКОВОЙ ОБРАБОТКЕ

Аннотация. Проведено исследование морфологии, элементного состава, микротвердости и коррозионных свойств поверхностного слоя сплава TiNi после комбинированной обработки, которая включала ионно-плазменное (вакуумно-дуговым методом) осаждение TiN-покрытия и ультразвуковую обработку (УЗО) с различным количеством проходов (*n*). С использованием сканирующей электронной микроскопии установлено, что ультразвуковая обработка способствует существенному снижению количества капельной фазы на поверхности TiN-покрытия, однако с увеличением числа проходов при УЗО наблюдается нарушение сплошности TiN-покрытия в локальных точках. Исследовано влияние комбинированной обработки образцов TiNi на микротвердость и выявлен синергетический эффект двух упрочняющих технологий, который заключается в увеличении микротвердости сплава TiNi (1,6 ГПа в режиме поставки): за счет осаждения TiN-покрытия – до 10,9 ГПа, за счет последующей УЗО – от 14,5 до 18,4 ГПа в зависимости от количества проходов. Установлено, что для схемы УЗО + TiN величина потенциала коррозии E_{corr} практически не зависит от числа проходов, составляет порядка –250 мВ и определяется величиной потенциала TiN-покрытия. Для схемы TiN + УЗО выявлено, что с увеличением числа проходов величина E_{corr} смещается в сторону более отрицательных значений, приближаясь к значению потенциала коррозии TiNi в состоянии поставки (–350 мВ). С использованием метода сканирующего вибрирующего зонда (SVET) для образцов, подвергнутых обработке УЗО + TiN и TiN + УЗО (n = 1), выявлена высокая электрохимическая совместимость материалов основы (TiNi) и покрытия (TiN) в хлоридной среде с минимальными флуктуациями плотности тока. На основании полученных экспериментальных данных предложен технологический процесс обработки сплава TiNi по схеме TiN + УЗО (n = 1), позволяющий достичь синергетического эффекта упрочнения поверхностного слоя сплава TiNi в сочетании с высокими коррозионными свойствами и улучшенной морфологией поверхности.

Ключевые слова: TiNi, TiN-покрытие, вакуумно-дуговое осаждение, ультразвуковая обработка, микротвердость, потенциал коррозии

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Introduction. Ion-plasma coating deposition methods have been widely used for production of barrier layers and functional coatings, particularly titanium nitride (TiN). Although TiN coatings have been shown to have positive effects on hardness, wear resistance, corrosion resistance, and friction reduction, their biomechanical compatibility sometimes makes them less effective in medicine [1, 2].

Having high fragility, they do not always provide sufficient adhesion strength to substrate, particularly, in dynamic conditions. The coating integrity violation can cause the release of potentially toxic elements of a substrate material (Ni, V, Al, Co, etc.) into a biological environment with their subsequent accumulation in human tissues. For TiN coatings used in medicine, mechanical properties should be considered. These properties must be maximally consistent with a metal substrate reducing anisotropy of product's properties over its thickness.

The attempts have been made to produce coatings with high hardness, fatigue wear and crack resistance [3]. That applies to multilayer and composite coatings that combine hard and soft layers.

A promising method for balancing mechanical properties of a coating and a substrate is high-energy treatment of a product surface for hardening before deposition process [4–7] or high-energy treatment of a coating deposited on a product surface for stress relaxation and softening [8, 9].

A very wide range of effects associated with the influence of ultrasound on materials is known [4]: structural changes, reduction of internal stresses in deformed metals, increase of plastic properties (acoustoplastic effect), etc. Depending on frequency, amplitude, and locality, it is possible to achieve both strengthening, softening and plasticity enhancement of a material over time. This opens up opportunities for ultrasonic treatment (UT) to be used not only as an independent operation for hardening surface of metals and alloys, but also as one of operations of combined processing to prepare surfaces for coating or improve performance properties of previously formed coatings [10–12].

The aim of the present paper was to study the structure and properties of the surface layer of a medical TiNi alloy subjected to ion plasma (deposition of TiN coating) and ultrasonic treatment.

Experimental. The flat wire samples of Ti – 55.16 wt.% Ni alloy (further named TiNi) with cross-section dimensions of 0.3 × 8 mm were used for the study. The combined treatment of the samples was carried out according to two schemes:

1) TiN coating with a thickness of ~ 1 μm followed by ultrasonic treatment (TiN + UT);

2) ultrasonic surface treatment followed by coating (UT + TiN).

The TiN coatings were obtained in the Bulat-6 vacuum arc installation. We used a deposition mode based on the calculation of the substrate temperature during ion plasma treatment [13]:

– Ti-ion bombardment at $U = -800$ V, $I = 100$ A for 20 minutes (this treatment provides a substrate temperature of 700 °C which corresponds to recrystallization of TiNi);

– the deposition of Ti sublayer at $U = -100$ V, $I = 100$ A for 2 minutes (the sublayer provides a higher adhesion of TiN coating to the substrate);

– the deposition of TiN coating at $U = -100$ V, $I = 100$ A, $P_{N_2} = 4 \cdot 10^{-1}$ Pa for 15 minutes.

Ultrasonic treatment was carried out using an experimental installation for ultrasonic hardening and finishing of flat workpieces developed by the authors [14], according to regimes represented in the Table.

Ultrasonic treatment regimens of TiNi samples

Parameters	Designation	Unit	Value
Frequency	f_r	kHz	19.5
Amplitude	A	μm	15
Speed of sample movement relative to the tool	V	mm/s	6.25
Number of tool passes	n	–	1÷6
Static force	F_s	N	40

The surface morphology was studied by scanning electron microscopy (SEM) using a Tescan Mira microscope (Czech Republic). The qualitative and quantitative elemental analysis of the sample surface was identified by X-ray spectral analyzer by Oxford Instruments Analytical Ltd (UK).

The microhardness of the TiNi samples after combined treatment was measured using a PMT-3M device. The Vickers and Knoop indenters were used with 20 g and 2 g loads, respectively.

Two methods were used to evaluate the corrosion properties of TiNi alloy with TiN coatings: the measurement of the open circuit potential E_{corr} and Scanning Vibrating Electrode Technique (SVET). The tests were carried out in 0.9 % NaCl solution (a universal model medium for conducting corrosion tests for medical devices) at room temperature for 15 hours.

Results and discussion. *Morphology and elemental composition.* The presence of a droplet phase is a significant disadvantage of vacuum-arc method [15]: its presence on a coating surface leads to an increase in roughness, in friction coefficient and, hence, a decrease in wear resistance. In addition, as the droplet phase content increases, the number of macrodefects in the coating increases. Heterogeneity of physical properties in the volume and on the coating surface increases due to differences in chemical composition of the droplets and the coating. The droplet phase in the form of predominantly spherical particles of different diameters can be observed in the SEM image of the TiN coating surface (Figure 1).

This type of surface defect was found for the TiNi samples treated according to UT + TiN scheme (Figure 2), i.e. when the final operation was to apply a nitride titanium coating. A large variation in droplet sizes can be observed: from submicron to ~ 20 μm. However, most of them were about 1 to 5 μm in diameter.

Figure 3 shows the surface of the TiNi sample with a TiN coating with a uniform Ti + N composition. However, there were also defects with different elemental compositions: a droplet phase (mainly Ti), as well as porosity with a high content of the substrate element – Ni.

Similarly, the element distribution maps were obtained for the TiNi samples after combined treatment according to different schemes and with different number of passes during ultrasonic treatment. For UT + TiN scheme, the observed picture was almost similar to the case considered in Figure 3. A different picture was for TiN + UT scheme (Figure 4). No droplet defects were found which can be explained by the intense plastic deformation of the surface layers leading to the introduction of such defects into the coating thickness. The observed elongated areas with the high content of nickel detected can be due to cracking of TiN coating. The similar picture was observed on the surface of TiN + UT samples ($n = 3$ and $n = 6$).

The presence of nickel (toxic and carcinogenic metal [16–19]) is the main factor limiting TiNi for a broad use for medical application. In this study, we focus on the quantitative elemental analysis of nickel. Significant differences in the dynamics of change and absolute values of nickel concentration were found for two treatment schemes (Figure 5):

for UT + TiN scheme, a decrease in C_{Ni} value from 8.9 wt.% ($n = 1$) to 2.8 wt.% ($n = 6$) was observed;

for TiN + UT scheme, the value of C_{Ni} was no less than 10.2 wt.% ($n = 1$), and for $n = 3$ was 26.3 wt.%.

This confirms the earlier finding regarding the integrity of TiN coatings being damaged during ultrasonic treatment with a number of passes ≥ 3.

Microhardness. One of the expected results of this study was to achieve a synergistic effect via the application of two different surface engineering methods – ultrasonic strengthening treatment and hard TiN coating ($HV \geq 17$ GPa [20]) deposition.

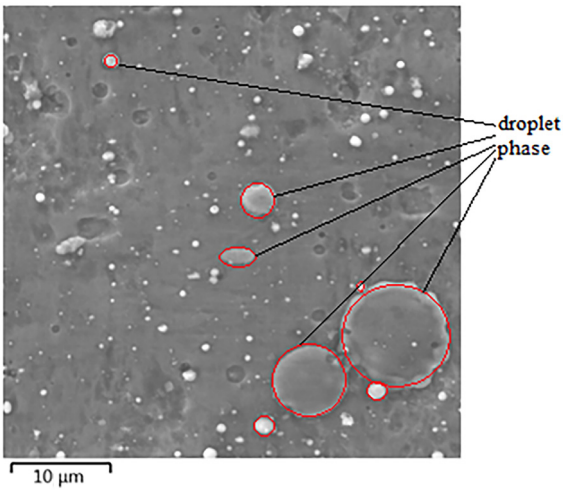
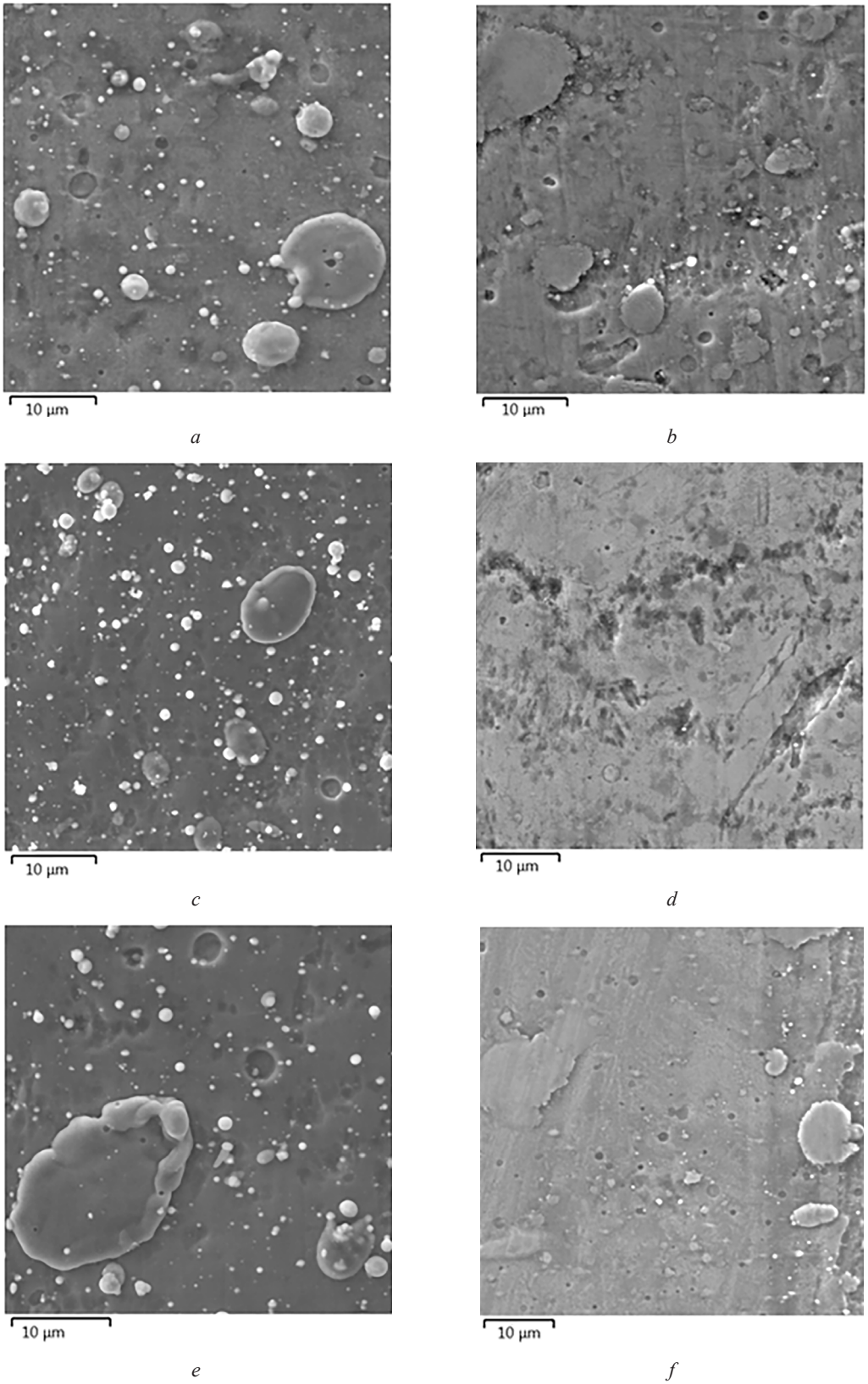


Figure 1. SEM image of TiNi surface sample with TiN coating



a *b* *c* *d* *e* *f*

Figure 2. SEM images of the surface of TiNi samples after combined treatment:
a, *c*, *e* – UT + TiN; *b*, *d*, *f* – TiN + UT; *a*, *b* – *n* = 1, *c*, *d* – *n* = 3; *e*, *f* – *n* = 6

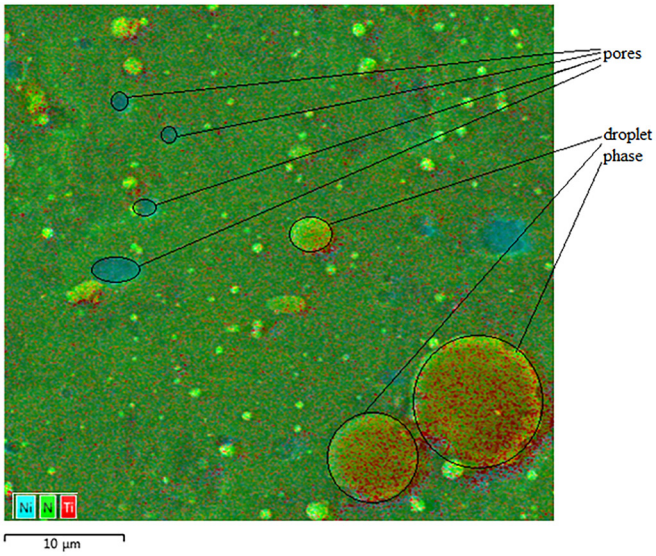


Figure 3. Surface image of TiNi sample coated with TiN in characteristic X-ray in overlay of colour fields of the substrate and coating elements

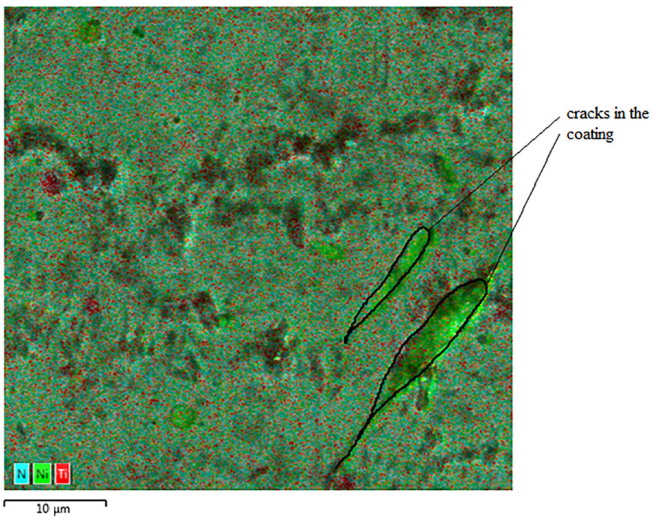


Figure 4. Surface image of TiNi sample subjected to TiN + UT (n = 3) in characteristic X-ray in overlay of colour fields of the substrate and coating elements

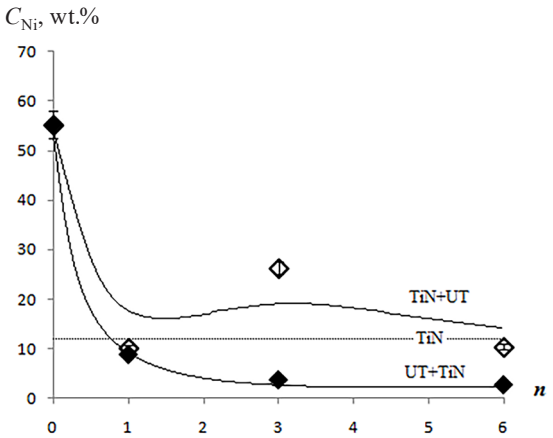


Figure 5. Change in Ni concentration on the surface of TiNi sample after combined treatment depending on the number of UT passes

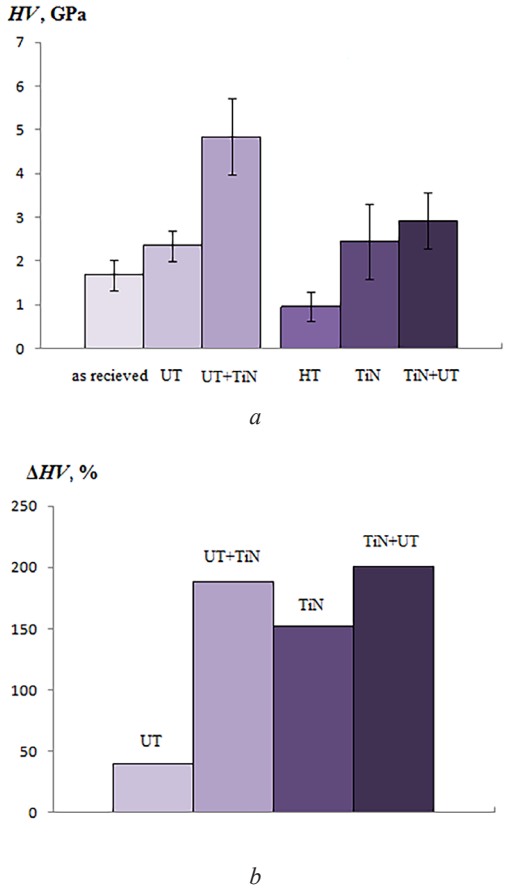


Figure 6. Vickers microhardness of TiNi samples in the as-received state after heat treatment (HT), ultrasonic surface treatment, deposition of TiN coating and their combination (*a*); increase in Vickers microhardness depending on the type of surface treatment (*b*)

In fact, ultrasonic treatment (n = 1) of the TiNi sample in the as-received state led to an increase in its microhardness from ~ 1.7 to ~ 2.4 GPa (Figure 6, *a*), and subsequent deposition of TiN to ~ 4.9 GPa.

The total increase in microhardness ΔHV calculated by formula (1) after treatment according to UT + TiN scheme was ~ 190% (Figure 6, *b*).

$$\Delta HV = (HV_i - HV_0)/HV_0 \cdot 100\ \%, \quad (1)$$

where HV_i – microhardness of TiNi samples after treatment according to different schemes, HV_0 – microhardness of TiNi in the as-received state.

Analyzing the treatment contribution to the rise in microhardness value using TiN + UT scheme reveals that when TiN coating is applied to the sample in its as-received state, a change in the structure of TiNi occurs that is comparable to heat treatment process. Its value was compared with the microhardness of the sample after heat treatment at 700 °C for 20 minutes (Figure 6, *a*). It was ~ 1 GPa, i.e. it was significantly less than for the sample in the as-received state. Due to differences in TiNi microhardness before combined treatment, for TiN + UT scheme (n = 1*)* the microhardness values of ~ 2.9 GPa were obtained, different from UT + TiN scheme, the total increase (~ 200 %) was comparable (see Figure 6, *b*).

The obtained microhardness values were significantly lower than the hardness of traditional hardening TiN coatings due to small thickness of the coating and impossibility of meeting the condition $H \leq 10h$ [21], where H is the indentation depth, μm; h – coating thickness, μm. This indicates that a softer substrate has a major influence on measurement findings, and that can be avoided with much smaller loads.

The measurement was carried out using the Knoop hardness testing with applied load of 2 g when analyzing the influence of number of passes of ultrasonic treatment on the microhardness value of TiNi samples (Figure 7).

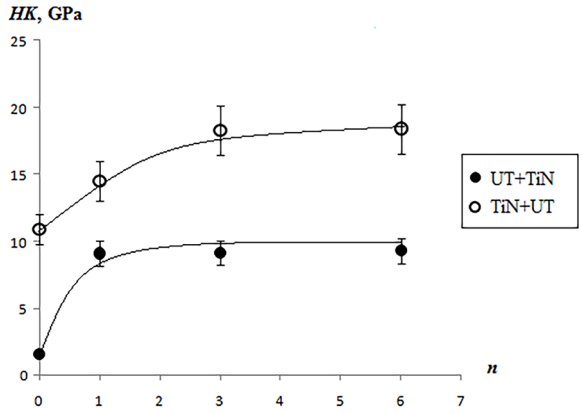


Figure 7. Dependence of the Knoop microhardness of TiNi samples on the number of UT passes after combined treatment

Based on the obtained dependencies, the following conclusions were made:

– for the samples treated according to UT + TiN scheme, the Knoop microhardness was practically the same for different n (9.1÷9.3 GPa) and was close to the microhardness value of TiN (10.9 GPa) which can be explained by relaxation of residual stresses arising during ultrasonic treatment due to high-temperature exposure upon deposition;

– for the samples treated according to TiN + UT scheme, the synergistic effect of two strengthening technologies was observed: the microhardness value of TiNi sample (1.6 GPa) first increases to 10.9 GPa due to deposition of TiN, and subsequent ultrasonic treatment promotes its even greater growth – up to 14.5 GPa at $n = 1$ and 18.3 GPa at $n = 3$. With a further increase in the number of passes, the microhardness value remains at the same level – 18.4 GPa ($n = 6$).

Corrosion properties. Figure 8 shows the measured values of the TiNi samples open circuit potential E_{corr} following different treatments. The value of E_{corr} for TiNi shifted to the area of more positive values after treatment in comparison with in the as-received state, where $E_{corr} = –350$ mV. Following the application of TiN + UT ($n = 1$), the sample shift ($\Delta E_{corr} = 140$ mV) was found.

The study of the dependence of the E_{corr} value on the number of UT passes for UT + TiN scheme (Figure 9) showed that it was practically independent of n (–250 mV). For TiN + UT scheme with $n = 1$, the E_{corr} value was ≈ –220 mV (see Figure 8). The value of E_{corr} shifted towards more negative values as the number of passes increased, approaching the value of the open circuit potential of the TiNi sample in the as-received state.

SVET is used to study electrochemical activity of the material at the micro level. The tests were carried out according to [22]. The current density distribution on the surface of the TiNi samples (Figure 10) shows significant inhomogeneity only for the TiN + UT sample ($n = 3$), which, as mentioned above, results from the destruction of the integrity of TiN coating at local points.

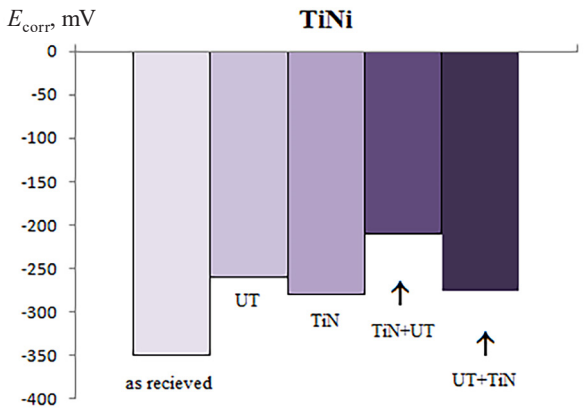


Figure 8. Open circuit potentials of TiNi samples after UT ($n = 1$), TiN coating deposition and their combinations

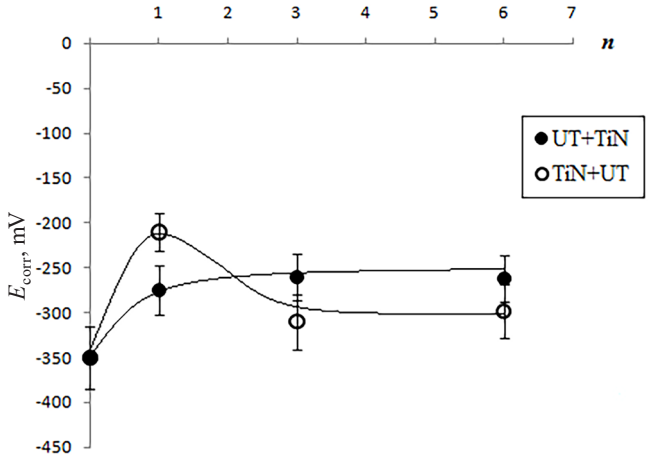
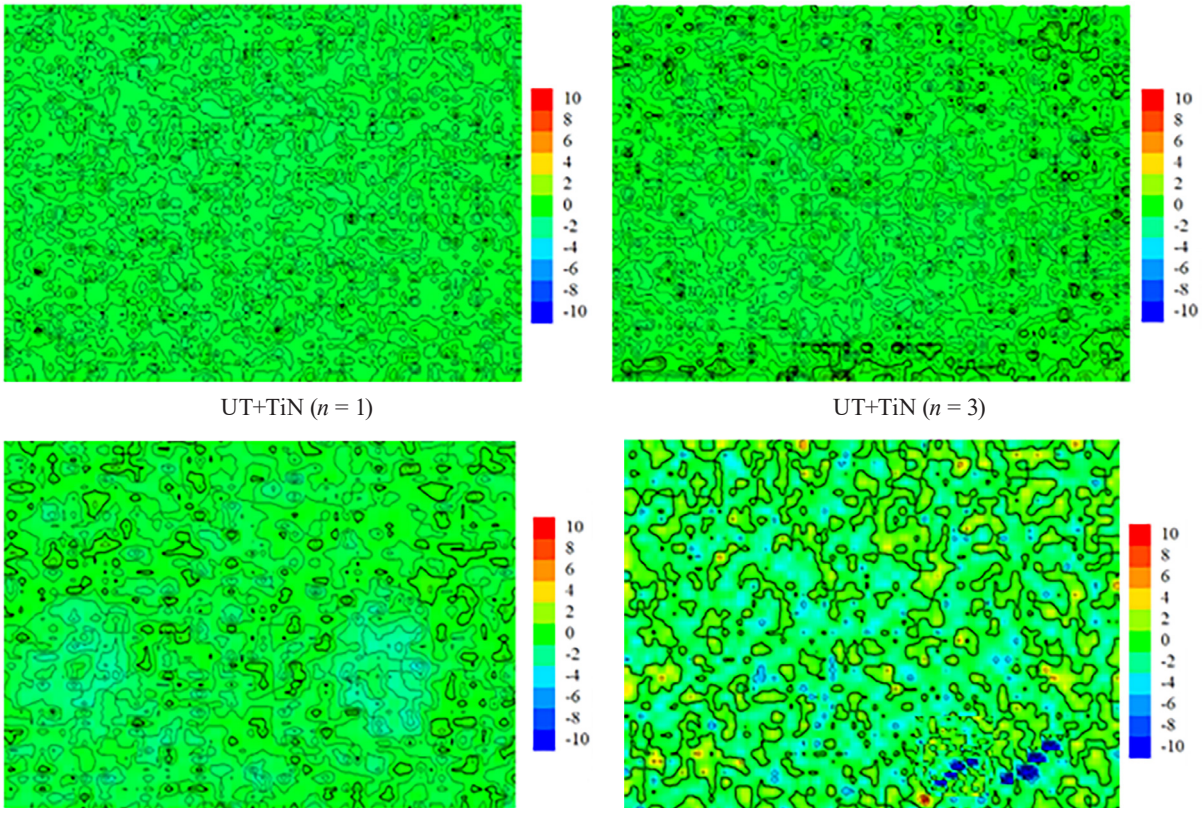


Figure 9. Dependence of the E_{corr} value of TiNi samples on the number of UT passes after combined treatment



UT+TiN (n = 1) UT+TiN (n = 3)

TiN + UT (n = 1) TiN + UT (n = 3)

Figure 10. Current density distribution (μA / mm^2)
on the surface of TiNi samples after exposure to 0.9% NaCl solution

The defective areas had more electronegative potential compared to the rest of the study surface. The samples of UT + TiN and TiN + UT (n = 1) demonstrated high electrochemical compatibility of the substrate and the coating material in a chloride media with minimal fluctuations in current density.

Conclusions. The surface morphology and the distribution of elements in the surface layer of the TiNi samples after combined treatment according to UT + TiN and TiN + UT schemes were studied. The macroparticles of the droplet phase with an average diameter of 1 to 5 μm were found. The subsequent ultrasonic treatment has been shown to significantly reduce the amount of the droplet phase. With an increase in n, cracking is observed on the surface of the TiN coating. The quantitative analysis of nickel content showed that the value of C_{Ni} varied from 8.9 wt.% (n = 1) to 2.8 wt.% (n = 6) for UT + TiN scheme; it was not less than 10.2 wt.% for TiN + UT scheme.

The effect of combined treatment of the TiNi samples on the Knoop microhardness was studied. The synergistic effect of two strengthening methods (TiN + UT) was found. The microhardness of TiNi was increased (1.6 GPa in the as-received state): due to deposition of TiN coating – up to 10.9 GPa, due to subsequent ultrasonic treatment – up to 14.5 GPa (n = 1), 18.3 GPa (n = 3) and 18.4 GPa (n = 6).

The corrosion studies of TiNi after combined treatment showed that for UT + TiN scheme the value of E_{corr} was practically independent on the number of passes n and was about of –250 mV and determined by the potential of TiN-coating. For TiN+UT scheme, it was found that with an increase in the number of passes, the value of E_{corr} shifted towards more negative values, approaching the value of the open circuit potential of the TiNi sample in the as-received state (–350 mV). High electrochemical compatibility of the substrate and the coating material in a chloride media with minimal current density fluctuations was revealed for the samples subjected to UT + TiN and TiN + UT (n = 1).

A method for treatment TiNi according to TiN + UT scheme is proposed including:

– deposition of TiN coating with a thickness of ~ 1 μm using the vacuum-arc deposition method at arc current of 100 A, a bias potential from –800 V (annealing at ~ 700 °C) to –100 V (deposition of Ti-sublayer and TiN coating) and nitrogen pressure ~ 0.4 Pa;

– ultrasonic treatment at frequency ~ 19.5 kHz, amplitude ~ 15 μm, number of tool passes n = 1.

This treatment achieves the synergistic effect of strengthening the TiNi surface layer showing improvement of surface morphology and corrosion resistance and can find application in technological processes for fabrication of biomedical devices from shape memory alloys.

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