The influence of the energy density of the electron beam on the morphology of surface and mechanical properties of the surface layer of NiTi alloy

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Abstract. This work comprises a study of the influence of the energy density \( E_s \) of the low-energy high-current electron beam (LEHCEB) exposure on the morphology of surface and mechanical properties of the surface layer of nickel titanium alloy (NiTi alloy). It was found that the formation of crater-like relief on the surface of NiTi alloy after electron beam treatment is determined by the structure and distribution of the particles of second and impurity phases. The mechanical properties in a modified surface layer of no more than \( \sim 500 \) nm thickness change at different values of the energy density \( E_s \) of electron beam.

1. Introduction

The technologies of processing by the pulsed low-energy high-current electron beams (LEHCEBs) allow for smoothing the surface, homogenizing the surface layers, modifying the structure, and changing the mechanical properties of the surface layers of metallic materials [1]. In particular, electron beam processing is one of the techniques for modifying the surface of a nickel titanium alloy (NiTi alloy), which has the effect of shape memory and superelasticity (SME-SE). Today, the studies conducted on the NiTi alloys after LEHCEB-treatments are mainly presented as an analysis of the structure and strength properties of the modified layers. Whereas, the results of the complex investigations of the influence of the modes of LEHCEB-treatments both on the morphology of surface and mechanical properties are insufficient.

Here, using the LEHCEB-treatment, we investigate its influence on the morphology of the surface and mechanical properties of the surface layer of NiTi alloy.

2. Experimental

2.1. Material

The material researched here is a commercial NiTi alloy (MATEK-SMA, Russia) produced as rolled sheets by vacuum induction melting (VIM). The chemical composition of the alloy was: Ti (balance); 55.08 Ni; 0.051 C; 0.03 O; 0.002 N (wt %). Initial NiTi samples were cut down to dimensions of 15×10×1 mm by the method of laser cutting. The stages of preliminary surface treatments of NiTi samples were described in [2].
2.2. *LEHCEB*-treatment

The electron beam treatment in the surface melting mode of the surface of NiTi alloy was carried out on the modified automatic setup RITM-SP (Microsplav, Russia). Parameters of LEHCEB-treatment: the number of pulses of electron beam \( n = 5 \), the pulse duration \( \tau = 2\text{–}2.5 \mu s \), the beam energy density \( E_s = 1.1 \text{ J/cm}^2 \), \( E_s = 1.7 \text{ J/cm}^2 \), \( E_s = 2.7 \text{ J/cm}^2 \), \( E_s = 3.7 \text{ J/cm}^2 \). The parameters of the electron beam setup were described in [3].

2.3. Methods of investigation

The study of the morphology of surface of NiTi samples before and after electron beam treatment was carried out by the method of the optical metallography on the setup Axiovert 200MAT (Zeiss, Germany) using the function of the differential interference contrast (DIC).

The investigation of the mechanical properties of modified surface layers of NiTi samples were examined by the method of instrumented indentation on a Nano Hardness Tester (CSM, Switzerland). As a result of the experiment, were obtained series \( P-h \) diagrams (loading/unloading) in the form of dependence of the depth of the indenter \( h \) on the maximum load \( P \). Values of the hardness parameter \( H \) were determined by the Oliver-Pharr method [4] and standard ISO 14577 [5].

Pseudoelastic properties of surface layers were estimated using the parameter \( \eta \). This parameter characterizes the degree of contribution of pseudoelastic recovery of area print of indenter [6]:

\[
\eta = \frac{h_{\text{max}} - h_{\text{plastic}}}{h_{\text{max}}} \times 100\%
\]  

where \( h_{\text{max}} \) – maximum indentation depth under \( P_{\text{max}} \); \( h_{\text{plastic}} \) – residual depth after unloading.

3. Experimental results and discussion

3.1. Morphology of surface

Figure 1 shows optical metallographic images of the surface of NiTi before and after LEHCEB-treatment in the surface melting mode under different values of beam energy density \( E_s \). A detailed description of the surface morphology and topographical parameters of samples of NiTi alloy in the initial state was presented in [2, 7].

![Figure 1](image-url)
As can be seen from figure 1a, on the electro-mechanically treated surface of the NiTi sample before LEHCEB treatment were found a large number of inclusions. The result shows that the stages of preliminary electro-mechanical preparation (mechanical grinding and polishing, electrolytically treatment) do not allow to completely get rid of inclusions located on the surface. Based on this result, we conclude the feasibility of performance of LEHCEB-treatment as a tool for smoothing the surface and homogenizing the surface layers of NiTi alloy.

Figure 1 (b-e) demonstrates morphology of the surface of samples of NiTi alloy after LEHCEB-treatment at constant number of pulses of electron beam \( n = 5 \) and different values of beam energy density \( E_s \). A visual assessment shows that the result of the LEHCEB-treatment at the mode \( E_s = 1.1 \text{ J/cm}^2 \) the surface of the NiTi sample was smoothed (figure 1b). This is due to the fact that under the action of LEHCEB occur melting and particles evaporation, and homogenization of the phase and chemical composition on the surface of the NiTi alloy.

After LEHCEB-treatments of NiTi samples at modes with electron beam energy density \( E_s = 1.7, 2.7, \text{ and } 3.7 \text{ J/cm}^2 \) a crater-like relief is seen on the surface. The images in figure 1 (c-e) illustrated the morphological features of the surface topography. This suggests that the formation of a crater-like relief on the surface of NiTi samples as a result of LEHCEB-treatments is due to the presence of inclusions on the surface of initial NiTi alloy, that were melted under the influence of an electron beam. An increase the energy density of the electron beam \( E_s \) leads to decrease in the intensity of crater formation, as well as their size.

3.2. Physical-mechanical properties

Figure 2 shows dependences of the hardness parameter \( H \) on the maximum indentation depth \( h_{max} \) at different values of the maximum load \( P_{max} \) on the indenter. For ease of analysis, at these graphs are identified the areas of surface layers at depths of 500 and 1500 nm. The analysis of the mechanical properties of initial NiTi-substrate before LEHCEB-treatment was presented in [2].

The hardness values \( H \) in NiTi samples after LEHCEB-treatment have higher values than in the initial sample. So, in surface layers (of ~ 500 nm thickness) parameter \( H \) is: ~ 7000 MPa at the electron beam energy density \( E_s = 1.1 \text{ J/cm}^2 \), ~ 5500 MPa at \( E_s = 1.7 \text{ J/cm}^2 \), ~ 4000 MPa at \( E_s = 2.7 \text{ J/cm}^2 \), and ~ 6000 MPa at \( E_s = 3.7 \text{ J/cm}^2 \). At a depth of more than ~ 2 μm from the surface, the hardness parameter for these samples takes values close to the values of the initial NiTi substrate.

![Figure 2](image-url)

**Figure 2.** The dependences of hardness \( H \) on the maximum indentation depth \( h_{max} \): after preliminary electro-mechanical preparation – curve 1 (a), (b); after LEHCEB-treatment at mode: \( E_s = 1.1 \text{ J/cm}^2 \) (curve 2, a); \( E_s = 1.7 \text{ J/cm}^2 \) (curve 3, a); \( E_s = 2.7 \text{ J/cm}^2 \) (curve 2, b); \( E_s = 3.7 \text{ J/cm}^2 \) (curve 3, b).

From the analysis of the experimental data obtained, it follows that the LEHCEB-treatment mode at \( E_s = 1.1 \text{ J/cm}^2 \), which, as shown (figure 1b), leads to a smooth surface, produces a hardened surface layer of ~ 500 nm thickness (figure 2a, curve 2). The increase of hardness of the surface layer of ~ 500
nm thickness probably due to the occurrence of a columnar structure, formed after LEHCEB-treatment. Conversely, for the sample after LEHCEB-treatment at $E_r = 2.7$ J/cm², despite the detected local inhomogeneities after remelting the surface layer (figure 1d), the values of $H \approx 4500$ MPa (figure 2b, curve 2). This result shows that LEHCEB-treatment at $E_r = 2.7$ J/cm² does not lead to an increase of hardness of the surface layer.

From the analysis of the obtained dependences (figure 2), it follows that an increased energy density of the electron beam $E_s$ does not lead to a regular change in the hardness parameter $H$ in the modified layer of ~ 500 nm thickness.

3.3. Functional (pseudoelastic) properties

In this study to estimate the pseudoelastic properties of the modified surface layers after LEHCEB-treatment, we calculated parameter $\eta$. This parameter allows estimating the pseudoelastic shape recovery of the print, which formed under the indenter [6]. Figure 3 shows the dependence of the shape recovery ratio $\eta$ of the print on the maximum indentation depth $h_{max}$.

In a sample of a NiTi alloy before LEHCEB-treatment (figure 3a, b, curve 1), the parameter of shape recovery ratio $\eta$ in a surface layer of no more than ~ 500 nm thickness is equal to ~ 55%, and in a surface layer with a thickness of ~ 500 to ~ 1500 nm the parameter $\eta$ decreases to 50%. At a depth of more than ~ 2 μm, the value of the parameter $\eta$ is equal to 40%.

At different modes of LEHCEB-treatment, the parameter of shape recovery ratio $\eta$ changes relative to the values in the initial NiTi sample as follows at figure 3a, b, (curves 2, 3): in a surface layer of ~ 500 nm thickness: decreases by 10%, at $E_r = 1.1$ J/cm², increases by 10% at $E_r = 1.7$ J/cm², decreases by 15% at $E_r = 2.7$ J/cm², decreases by 5% at $E_r = 3.7$ J/cm². In a surface layer of ~ 1500 nm thickness, regardless of the energy density in the electron beam, $\eta$ decreases by 15%.

So, it can be concluded that, regardless of the mode of LEHCEB-treatment, there is a general tendency to decrease the parameter of shape recovery ratio $\eta$ by about 15% in near-surface layers at a depth of ~ 2 μm. When the maximum indentation depth $h_{max}$ increases (more than ~ 2 μm), the values of the parameter $\eta$ in the samples after LEHCEB-treatment close to the values of this parameter in the initial NiTi sample.

4. Conclusion

As a result of LEHCEB-treatments of the NiTi alloy at different energy densities of the electron beam $E_r$ and a constant number of pulses $n = 5$, it was found that:

![Figure 3](image-url)
1) regularities of crater-like relief formation on the surface of NiTi alloy are determined by the structure of rolled samples and the specificity of distribution particles of the second and/or impurity phases;

2) an increase energy density of the electron beam $E_s$ does not lead to a regular change in the hardness parameter $H$ in the modified layer of $\sim 500$ nm thickness. At a depth of more than $\sim 2 \mu m$ from the surface, the hardness parameter for these samples takes values close to the values of the initial NiTi substrate;

3) in a modified surface layer of $\sim 500$ nm thickness, the functional (pseudoelastic) properties, are lower than the functional properties of the initial NiTi substrate by no more than 15%.

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