Modification of physical-mechanical properties of NiTi alloy by electron beam in surface melting mode

F A D'yachenko¹, S N Meisner¹, E V Yakovlev², A A Atovullaeva³ and L L Meisner¹, ³

¹Institute of Strength Physics and Materials Science SB RAS, Tomsk, Russia
²Institute of High-Current Electronics SB RAS, Tomsk, Russia
³National Research Tomsk State University, Tomsk, Russia

E-mail: frozennonetroll@mail.ru

Abstract. This paper presents a research data on investigation of physical-mechanical properties of the surface of NiTi shape memory alloy after low-energy high-current pulsed electron beam (LEHCPEB) treatment in surface melting mode. Investigation of physical-mechanical and functional properties (Young’s modulus $E_T$, dynamic hardness $H_T$, plasticity characteristic $\delta_H$, and parameter of shape recovery ratio $\eta$) was held by method of instrumented nanoindentation test. Our data suggest that after LEHCPEB treatment physical-mechanical properties of the surface layers of ~500 nm thickness change in depth from the surface without critical influence to the pseudoelastic properties of NiTi alloy.

1. Introduction

Methods of electron beam treatment modify the structure and, accordingly, change the physical-mechanical properties of the surface layers of metallic materials [1]. It was found [2], that the morphology of surface and structure of the surface layers of nickel-titanium alloy (NiTi) were changed by the low-energy high-current pulsed electron beam treatment (LEHCPEB). However, as was shown in study [3], the influence of LEHCPEB with a large number of pulses $n$ leads to a change of physical-mechanical properties of the surface layers of more than ~2 μm thickness.

Currently, it was established [4, 5] that the modified surface layers of NiTi alloy are residual elastic stress concentrators. It is possible to reduce the level of residual stresses in modified layers of the material by decreasing the number of pulses $n$ of electron beam and/or decreasing the energy density $E_{s}$ up to a limiting value. This mode of LEHCPEB treatment is called a surface melting mode.

It can be assumed that the use of LEHCPEB treatment in surface melting mode will allow achieving optimal physical-mechanical characteristics. This, in turn, will not have a critical impact on the important functional (pseudoelastic) properties of the NiTi alloy (shape memory effect and superelasticity, SME-SE).

Here, using the LEHCPEB in surface melting mode, we investigate formed modified surface and its physical-mechanical properties.

2. Experimental
2.1. Material
The material investigated here is 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 %).

Samples were cut to dimensions of 10x10x1 mm by laser, mechanically ground and polished (abrasive grit: P120, P320, P600, P1200), electrolytically polished (acid solution: 3 part CH₃COOH + 1 part HClO₄), and washed in an ultrasonic bath with a distilled water (duration t = 15 min).

Experimental studies of surface of initial NiTi alloy and NiTi alloy after electron beam treatment in surface melting mode were studied on 8 samples (4 – initial NiTi; 4 – after LEHCPEB).

2.2. LEHCPEB-treatment
Electron beam modification in surface melting mode of the surface of NiTi alloy was carried out on the modified automatic setup RITM-SP (Microslov, Russia) [6]. Working characteristics of the setup are: the current of electron beam is up to 25 kA; the accelerating voltage is up to 19 kV; the beam diameter is up to 10 cm; the working gas (argon) pressure is 0.02–0.2 Pa. Parameters of surface melting mode are: the number of pulses of electron beam n = 5, the pulse duration τ = 2–2.5 μs, the beam energy density Eₛ = 1.1 J/cm².

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 optical metallography on the setup Axiovert 200MAT (Zeiss, Germany) using the optical light and dark-field and function of the differential interference contrast (DIC).

Investigation of physical-technical parameters of modified surface layers was examined by method of instrumented nanoindentation test on a Nano Hardness Tester (CSM, Switzerland). As a result of the experiment, according to the standarts ISO 14577 [7], a series P-h diagrams were obtained (dependences of the depth of the indener h on the maxium load P). These diagrams represent information of local deformation under the indenter. Using the Oliver-Pharr method [8] and standart ISO 14577 [7], the Young’s modulus EᵢT, dynamic hardness HᵢT, and static Vickers hardness HV were determined.

The plasticity characteristic δᵢT of surface layer shows the propensity of the material to deform under the action of the load. This parameter δᵢT was estimated [9]:

\[
δᵢT = 1 - 14.3 \left(1 - \nu - 2 \sqrt{\nu} \right) \frac{HV}{EᵢT}
\]  

(1)

where ν – Poisson’s ratio; HV – Vickers hardness; EᵢT – Young’s modulus determined by instrumented nanoindentation.

Pseudoelastic properties of surface layers were estimated using the parameter η. This parameter characterizes the degree of contribution of pseudoelastic recovery of area print of indenter [10]:

\[
η = \frac{h_{max} - h_{plastic}}{h_{max}} \times 100
\]  

(2)

where h_{max} – maximum indentation depth under P_{max}; h_{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 LEHCPEB treatment in surface melting mode (each image from one tested sample of NiTi alloy before and after LEHCPEB). Before electron beam irradiation of initial NiTi sample (figure 1a, b, c, d) the morphology of surface is determined by the rolling texture. In figures 1c, d obtained in the DIC and dark-field regimes, it is clearly seen that inclusions are located on the surface. It was established that the size of the inclusions varies from ~0.5 up to 5 μm.
In studies [11, 12], it was found that inclusions on the surface of the NiTi alloy are the phases of: Ti(C, O) – titanium carbides and oxides; Ti₄Ni₂Oₓ – oxides based on the Ti₂Ni intermetallic phase. The presence of inclusions of these phases on the surface of the NiTi alloy is associated with the stages of its technological production (rolled sheets by vacuum induction melting).

![Figure 1](image_url)

**Figure 1.** Optical light-field (a, b, e), dark-field (d) and DIC (c, f, g, h) images of surface of NiTi alloy: a, b, c, d – initial NiTi (before LEHCPEB treatment); e, f, g, h – NiTi after treatment

After LEHCPEB treatment of the surface of NiTi alloy (figure 1e, f, g, h) a crater-like relief is seen on the surface, as well as a small number of inclusions that were not melted by the electron beam. As a result of LEHCPEB treatment, according to [12], the occurrence of crater-like relief is due to the presence if inclusions on the surface of initial NiTi alloy. These inclusions were not completely melted under the impact of the electron beam in surface melting mode.

3.2. **Physical-mechanical properties**

The physical-mechanical properties of surface layers of NiTi samples before and after LEHCPEB treatment can be addressed from figure 2 showing Young’s modulus $E_{IT}$ (1), dynamic hardness $H_{IT}$ (2), and plasticity characteristic $\delta_{IT}$ (3) as a function of maximum indentation depth $h_{max}$ under the increasing load $P$ from 5 up to 300 mN.

![Figure 2](image_url)

**Figure 2.** Dependences of $E_{IT}$ (1), $H_{IT}$ (2) and $\delta_{IT}$ (3) on the maximum indentation depth $h_{max}$ for: a – initial NiTi (before LEHCPEB treatment); b – after LEHCPEB treatment in surface melting mode

Near the surface the parameters of Young’s modulus $E_{IT}$ and dynamic hardness $H_{IT}$ of NiTi alloy before LEHCPEB treatment take a values: $E_{IT} = 60$ GPa, $H_{IT} = 4$ GPa (figure 2a, curves 1, 2). These
parameters change linearly to the values of: $E_{IT} = 50 \text{ GPa}$, $H_{IT} = 2.5 \text{ GPa}$ at a depth $h$ of more than $\sim 1.25 \text{ μm}$ thickness. High values of physical-mechanical parameters at a depth $h$ up to $\sim 1.25 \text{ μm}$ are associated with mechanical hardening of surface. This is due to the manufacturing of NiTi alloy that was fabricated by VIM and subsequent mechanical grinding of the surface of the NiTi samples before electron beam treatment.

Analysis of the physical-mechanical parameters of the NiTi samples after LEHCPEB treatment showed that modification of surface in melting mode leads to a change of strength characteristics in a depth from the surface. In figure 2b (curves 1, 2), it can be seen that electron beam treatment led to the hardening of the surface to a depth $h$ of $\sim 500 \text{ nm}$. However, at a depth $h$ of more than $\sim 500 \text{ nm}$, the values of the physical-mechanical parameters correspond to the parameters that represent the properties in bulk of initial NiTi sample. It is important to note the LEHCPEB treatment in surface melting mode did not lead to a highly change of parameters to a depth greater than $\sim 500 \text{ nm}$. A further linear horizontal view of the curves of the physical-mechanical parameters $E_{IT}(h_{\text{max}})$, $H_{IT}(h_{\text{max}})$, and $\delta_{H}(h_{\text{max}})$ at a depth more than $\sim 500 \text{ nm}$ (figure 2b, curves 1, 2, 3) means that the mechanical hardening of surface due to the manufacturing of NiTi alloy and subsequent mechanical grinding of the surface of samples was relaxed after melting of surface layers by LEHCPEB treatment.

The change in the values of the plasticity characteristic $\delta_{H}$ before and after LEHCPEB treatment is presented in figure 2a, b (curves 3). It was found that in modified layer of $\sim 500 \text{ nm}$ thickness figure 2b (curve 1), the parameter $\delta_{H}$ varies from 40 to 59% relative to the initial NiTi sample, which has a value of the parameter $\delta_{H} = 48\%$ at given depth. In the NiTi sample before and after treatment at a depth $h$ of more than 1.5 μm, the plasticity characteristic $\delta_{H}$ is equal to 0.6%.

### 3.3. Functional (pseudoelastic) properties

In this study to estimate pseudoelastic properties of the modified surface layers after LEHCPEB treatment, we calculated parameter $\eta$ [10]. This parameter allows estimating the pseudoelastic shape recovery of the print, which formed under the indenter. Figure 3 shows the dependence of the shape recovery ratio of the print on the maximum indentation depth $h_{\text{max}}$ for the sample of the initial NiTi alloy (curve 1) and for the sample of NiTi alloy after electron beam treatment in surface melting mode (curve 2).

![Figure 3](image_url)

**Figure 3.** Dependences of shape recovery ratio $\eta$ on the maximum indentation depth $h_{\text{max}}$ for: 1 – initial NiTi (before LEHCPEB treatment); 2 – after LEHCPEB treatment in surface melting mode

It is seen, that after treatment the parameter of shape recovery ratio $\eta$ varies from 50 to 40% inside a layer of no more than $\sim 500 \text{ nm}$ thicknesses. At a depth from $\sim 500 \text{ nm}$ and more than 2 μm, the parameter
η does not change and retains its value and equal to the value of the parameter η in the volume of the initial NiTi sample before electron beam treatment. This result means that LEHCPEB treatment in surface melting mode did not have a critical influence on the pseudoelastic property of the NiTi shape memory alloy.

4. Conclusion
The main conclusions obtained in the work are as follows:

– the mechanical hardening of surface due to the manufacturing of NiTi alloy and subsequent mechanical grinding of the surface of samples was relaxed after melting of surface layers by LEHCPEB treatment;

– the physical-mechanical properties of the surface layers of ~500 nm thickness change in depth from the surface without critical influence to the pseudoelastic properties of NiTi alloy.

Acknowledgments
Electron beam treatment of the surface of NiTi alloy was supported by grant of the Russian Science Foundation No. 18-19-00198 (26.04.2018); analysis of morphology and physical-mechanical properties of surface of NiTi alloy before and after treatment were performed with the frame of the Fundamental Research Program of the State Academies of Science for 2013–2020, line of research III.23.

References
[1] Poate J M, Foti G, and Jacobson D C 1983 Surface Modification and Alloying: by Laser, Ion, and Electron Beams (New York: Plenum Press)
[2] Zhang K M, Zou J X, Grosdidier T, Gey, N, Yang D Z, Hao S Z and Dong S 2007 J. Alloys Compd. 434 682–685
[3] Meisner S N, Yakovlev E V, Semin V O, Meisner L L, Rotshtein V P, Neiman A A and D’yachenko F 2018 Appl. Surf. Sci. 437 217–226
[4] Meisner L L, Semin V O, Mironov Yu P, Meisner S N and D’yachenko F A 2018 Mater. Today Commun. 17 169–179
[5] Dong C et al. 2003 Surf. Coat. Technol. 163 621–624
[6] Markov A B, Mikov A V, Ozur G E and Padei A G 2011 Instrum. Exp. Tech. 54(6) 862–866
[7] International Standard Organization, ISO 14577-1:2002, Metallic Materials – Instrumented indentation test for hardness and materials parameters – Part 1: Test method
[8] Oliver W C and Pharr G M 2004 J. Mater. Res. 19(1) 3–20
[9] Milman Yu V 2008 J. Phys. D: Appl. Phys. 41 074013
[10] Crone W C, Shaw G A, Stone D S, Johnson A D and Ellis A B 2003 SEM Annual Conference Proceedings 1–6
[11] Coda A, Zilio S, Norwich D and Sczerzenie F 2012 J. Mater. Eng. Perform. 21(12) 2572–77
[12] Meisner L L, Rotshtein V P, Markov A B, Meisner S N, Yakovlev E V and D’yachenko F 2016 Procedia Struct. Integrity 2 1465–72