Study of mechanical characteristics by nanoindentation of an ion-implanted Ti-Ni shape memory alloy

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Abstract. This article examines the effect of ion implantation on the change in the mechanical characteristics of the Ti-Ni shape memory alloy. A comparison of the characteristics was carried out in the coarse-grained and nanostructured state on the initial alloy samples and on the samples after ion implantation. The implantation mode was chosen as the most optimal on the basis of previous studies. Irradiation mode parameters: dose - 2.3·10¹⁷ ion/cm², energy - 40 keV, ions - nickel, temperature - did not exceed 200 °C. The characteristics were studied by the nanoindentation method on a NanoHardnessTester, (CSM Instr.) At a load of 2 mN using a Berkovich-type diamond indenter. According to the data of nanoindentation by the Oliver-Pharr method, the value of hardness and Young's modulus of elasticity were determined. Research results show that ion implantation leads to an increase in hardness.

1. Introduction

For many years, interest in studying the properties of various metals and alloys subjected to ion implantation (II) has not disappeared. This is primarily due to the fact that the mechanism of action from AI is insufficiently studied and not fully disclosed, general patterns of changes in certain properties for various metal systems have not been modeled. The effects of AI exposure are different and ambiguous [1, 2].

Thus, as a result of the penetration of implanted ions into the near-surface layers of metallic materials, various structural changes are observed, the occurrence of which explains an increase in strength, hardness, wear resistance, a decrease in fatigue characteristics, a general improvement in surface quality, an increase in corrosion resistance and many other effects [3, 4, 5, 6, 7, 8].

Recently, of particular interest has been the production of materials with a specific structure and specified characteristics. The management of properties should be predictable and occur by varying the conditions for obtaining the material.

In this regard, the study of the influence of ionizing radiation on alloys with the shape memory effect both in the normal (coarse-grained state) and in the nanostate (fine-grained state) may be of broad scientific and practical interest.
2. Nanoindentation
This article presents the results of a study of the effect of ion implantation (II) on the change in the mechanical properties of the Ti-Ni shape memory alloy in the coarse-grained (CG) and nanostructured (NS) state. The tests were carried out by the nanoindentation method (ISO 14577).

For research, samples were prepared from a Ti-Ni alloy in a coarse-grained and nanostructured state in the form of strips 10x20 mm.

To obtain a Ti-Ni alloy in a coarse-grained state, the samples were quenched in water from a temperature of 700 °C.

Samples in the nanostructured state were obtained by electroplastic deformation (e=1.8) followed by annealing at 450 °C. A detailed description of the method for obtaining samples is given in [9].

The surface of the Ti-Ni alloy samples was implanted with Ni+ ions with a dose of $D = 2.3 \times 10^{17}$ ion/cm² with an energy of 40 keV on an ion implantation device.

Investigations of the mechanical characteristics of Ti-Ni samples in the coarse-grained and nanostate before and after ion implantation were carried out using the modern nanoindentation method and the traditional method for measuring the microhardness of thin coatings, which differ in the magnitude of the load and the indentation depth of the indenter.

Nanoindentation was carried out on a NanoHardnessTester (CSM Instr.) At a load of 2 mN using a Berkovich-type diamond indenter.

According to the nanoindentation data by the Oliver-Pharr method, the value of hardness and Young’s modulus of elasticity are measured. The results of calculating the hardness and elastic modulus by the Oliver-Pharr method (ISO 14577) were averaged over 9 measurements by the methods of analysis of variance. According to the recommendations of the ISO 14577 standard for correct measurement of the coating hardness, the indentation depth of the indenter did not exceed 1/10 of its thickness, i.e. for an irradiated layer with a thickness of 1 μm, the penetration depth should be less than 100 nm, for 2 μm - less than 200 nm. The loads on the indenter were chosen so that the maximum indentation depth was no more than 200 nm, thus the maximum load was 2 mN.

3. Data analysis
Data analysis was performed according to the experimental load-penetration depth curves shown in figures 1 and 2.

![Figure 1. Curves of nanoindentation of the Ti-Ni alloy in the coarse-grained state before (1) and after (2) implantation with nickel ions.](image-url)
Figure 2. Curves of nanoindentation of the Ti-Ni alloy in the nanostructured state before (1) and after (2) implantation with nickel ions.

4. Results

The results obtained are presented in Table 1. The average value of the elastic modulus $E$ of the alloy in the coarse-grained state after implantation with nickel ions decreases in comparison with the initial state (55 and 62 GPa, respectively). The reason for the decrease in the elastic modulus during implantation is poorly studied; data are practically absent in the literature and require additional research.

Table 1. Results of nanoindentation at a load of 2 mN.

| Condition       | HV     | E, GPa | R, % | $h_m$, nm | $h_f$, nm |
|-----------------|--------|--------|------|------------|-----------|
| CG (before II)  | 278±22 | 62±4   | 46   | 184        | 127       |
| CG (after II)   | 362±19 | 55±4   | 32   | 169        | 92        |
| NS (before II)  | 400±77 | 52±1   | 43   | 165        | 94        |
| NS (after II)   | 422±48 | 53±4   | 45   | 159        | 88        |

Where: $h_m$ in nm is the maximum penetration depth of the indenter. $h_f$, in nm - residual depth of indenter penetration.

In the alloy implanted with nickel ions in the nanostructured state, there are no noticeable changes in the elastic modulus $E$, in comparison with the initial one (53 and 52 GPa, respectively), since upon nanostructuring of the Ti-Ni alloy, the elastic modulus $E$ is already significantly reduced (~ 10% compared to the coarse-grained state), which is apparently associated with the inelastic behavior of strongly nonequilibrium grain boundaries in the nanostructured state.

The hardness values of the alloy, measured by the nanoindentation method, in both states increase after implantation with nickel ions (Table 1). So, in the coarse-grained state before and after implantation, the hardness $HV$ is 278 and 362, respectively. In the nanostructured state, the hardness values $HV$ before and after implantation with nickel ions are 400 and 422, respectively. An increase in the hardness $HV$ in both cases is apparently associated with the formation of a large number of specific radiation defects during ion implantation, at a depth much greater than the penetration of the implanted ions themselves [1, 2].
Note that the effect of hardening as a result of implantation is noticeably higher in the coarse-grained state ($\Delta HV = 83$) than in the nanostructured state ($\Delta HV = 22$), which indicates the dislocation hardening mechanism, the contribution of which decreases and even disappears with a decrease in the grain size to nanometer range.

The recovery factor $R$ for the coarse-grained Ti-Ni alloy after implantation decreases and has a value of 32% (46% before implantation).

For a Ti-Ni alloy in a nanostructured state after ion implantation, the recovery coefficient $R$ slightly increases from 43 to 45%, respectively, which corresponds to an increase in the area under the nanoindentation curve.

5. Conclusion

According to nanoindentation data, ion implantation of the Ti-Ni alloy in the coarse-grained and nanostructural states leads to an increase in hardness by ~ 20% and ~ 5-7%, respectively.

The modulus of elasticity $E$ and the coefficient of recovery $R$ as a result of ion implantation significantly (20-30%) decrease in coarse-grained samples and practically do not change (within the measurement error) in nanostructured samples.

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