Influence of Mo or Ta Thin Coatings on Nickel Titanium Inelastic Behavior[1]

A.A. Neyman, L.L. Meysner, A.I. Lotkov, and K.P. Redlich

Institute of Strength Physics and Material Science SB RAS, 2/1, Akademichesky ave., Tomsk, 634021, Russia
Phone: +8(3822) 28-68-54, E-mail: nasa@ispms.tsc.ru

Abstract – Using of materials with shape memory effect on the base of titanium-nickel alloys as implants finds wide application in medicine. Protection of a surface of a material or a product against influence of environment also was a problem of the given research.

In this paper the influence of a titanium-nickel surface modification on its physic mechanical properties was investigated. The modification was consisted in depositing of thin films by thickness of 200 and 500 nm from elements Mo and Ta. Such physic mechanical parameters, as superelasticity and reactive strain for initial polished samples and for samples with a covering were determined. Influence of a thin-film covering, on properties of an initial material was established.

1. Introduction

There are number of reasons defining a choice as surface modification the magnetron sputtering method. First, this method allows spending thin superficial layers modification of metals and alloys with the control and their chemical compound exact reproduction, crystal structure, physical and mechanical properties, improving physic mechanical superficial layers properties that leads to materials biological compatibility increase.

The purpose of the work is researching of magnetron sputtering method influence on superelasticity effect, TiNi samples with various superficial layers conditions revealed.

2. Materials and experimental technique

Researches carried out on $Ti_{49.5}Ni_{50.5}$ alloy specimens with temperatures of martensate transformation B2 ↔ B19: M_s = 283 K, M_f = 261 K, A_s = 299 K, and A_f = 322 K (M_s, M_f, A_s, and A_f are the temperatures of start and finish of direct and return MT, accordingly).

For researching by Auger-electronic spectroscopy method plate form samples and also by torsion method for carrying out the experiments needle form square profile samples with a working part 1×1×16 mm were made made [1]. All samples have been cut out by electro erosion method from a single ingot. Surfaces of all samples prepared by mechanical grinding and subsequent electrolytic polishing.

Three samples groups have been prepared: (1) – with initial (electrolytic polished) surface (Ini.), (2) – with 200 and 500 nm Mo coatings (Mo 200, Mo 500) and (3) – with 200 and 500 nm Ta coatings (Ta 200, Ta 500) created by magnetron sputtering method.

Deposition on samples was made on magnetron sputtering plant Leibold Z-80, Germany (EDI "RETC" TSC SD RAS). The deposition was spent at following conditions: chamber vacuum $P = 10^{-4}$ Pa, samples temperature at deposition T = 473 K, Ar pressure chamber P_{Ar} = 0.3 Pa, cathode current I = 2.5 A, U = 270 V, samples voltage U = 0 V, distance from Ta, Mo target to the sample 70 mm, thickness layer growth rate v = 10 micron/h. For creation steady coatings thickness the needle form samples rotated during deposition.

The chemical compound analysis was spent on Auger-electronic spectroscope "Schkhuna-2" (Russia) (SRINP).

Mechanical tests were spent on the plant by return torsion oscillation method under two loading – unloading schemes:

Scheme 1: For reception of loading – unloading vs. deformation diagrams the sample heated or cooled up to the certain temperature which was supported constant during a cycle of test. Then the sample with constant speed twisted up to deformation value equal 6%. Then the specimen smoothly unloaded. Experiment repeated at four temperatures chosen according to temperatures of MT in the given alloy so that to receive samples with a various initial structural condition: in single-phase with martensate structure B19′ (T_1 = 273 K), in the field of temperatures of return MT ($T_{2,3}$ = 296 and 306 K) and in single-phase with stable in relation to MT structure B2 (T_4 = 323 K). Between individual cycles of tests, the sample warmed up to temperature 373 K.

Scheme 2: For an estimation of jet pressure size sample cooled up to temperature 273 K at which the material completely contained in martensate condition. Then the sample twisted up to deformation value equal 6% and started to evenly heat up to temperature 720 K [2, 3].

3. Results and discussion

The analysis of Auger-electronic spectroscopy data on distribution of elements in samples superficial area

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initial and with the coatings (Fig. 1) allows conditionally allocating some layers on samples depth. In the initial sample is a top layer, extent 30 nm, with big carbon and oxygen concentration presence, and internal – with equiatomic Ti and Ni structure. In samples with a Mo and Ta coatings it is possible to allocate 3 layers: directly film (I), a transition layer (II) which contains a films material as well as Ti and Ni oxides and carbides and the last one – the layer of substrate material from titanium nickel (III).

As film deposition speed – magnitude known and practically constant it is easy to calculate a layer thickness from time.

Figure 1 illustrates the coating layers (I) correspond to the set thickness in 200 and 500 nm. It is necessary to note that the layer II can have various extent and a various chemical ratio.

From Fig. 2, *a* it is visible that the qualitative loading-unloading diagrams image and their changing in depending on tests temperatures (T_{1-4}) for all groups of samples is similar. Diagrams of the modified samples are characterized by presence extensive martensate yield plateau. The martensate shearing size τ_m increases with growth of tests temperature that is well visible from Fig. 2, *b*.

The temperature vs. deformation-power superelasticity parameters dependences received from loading-unloading diagrams of initial and modified samples $Ti_{49.5}Ni_{50.5}$ alloy are presented in Fig. 3.

Apparently, deformation contributions (Figs. 3, *b*–*e*) depend on superficial modification in a lesser degree, than power parameters sizes (Fig. 3, *a*). For all samples in an interval of temperatures 273–323 K a martensate shearing tension τ_m is characterized by linear temperature dependences. In case of modification superficial layers appreciable decrease, approximately in 2 times, sizes of a martensate shearing tension τ_m is observed. They are easily deformed at rather low sizes of the applied pressure (100–350 MPa). At the same time the reversible deformations size remains comparable with its value in the initial sample.

It is established (Fig. 3, *b*) that change deformation γ_m size (corresponding τ_m) correlates with τ_m behavior depending on a coatings kind on surface. Temperature dependence γ_m for samples with Ta 500 nm coatings is located above the initial sample. The presence Ta 200 nm, Mo 500 and 200 nm coatings on samples practically has not affected this parameter.

In Figures 3, *c* and *d* temperature dependences of deformation γ_{res} and γ_{ur} sizes are resulted. It is visible that initial samples return the set deformation after unloading (Fig. 3, *e*, curve 1) is better. Accumulation of residual deformation (after heating samples) in all samples begins with T_3 = 306 K. With test temperature rise in samples with any condition of a surface intensive accumulation γ_{res} begins, however samples with an initial surface accumulate it in a greater degree (Fig. 3, *c*, curve 1).

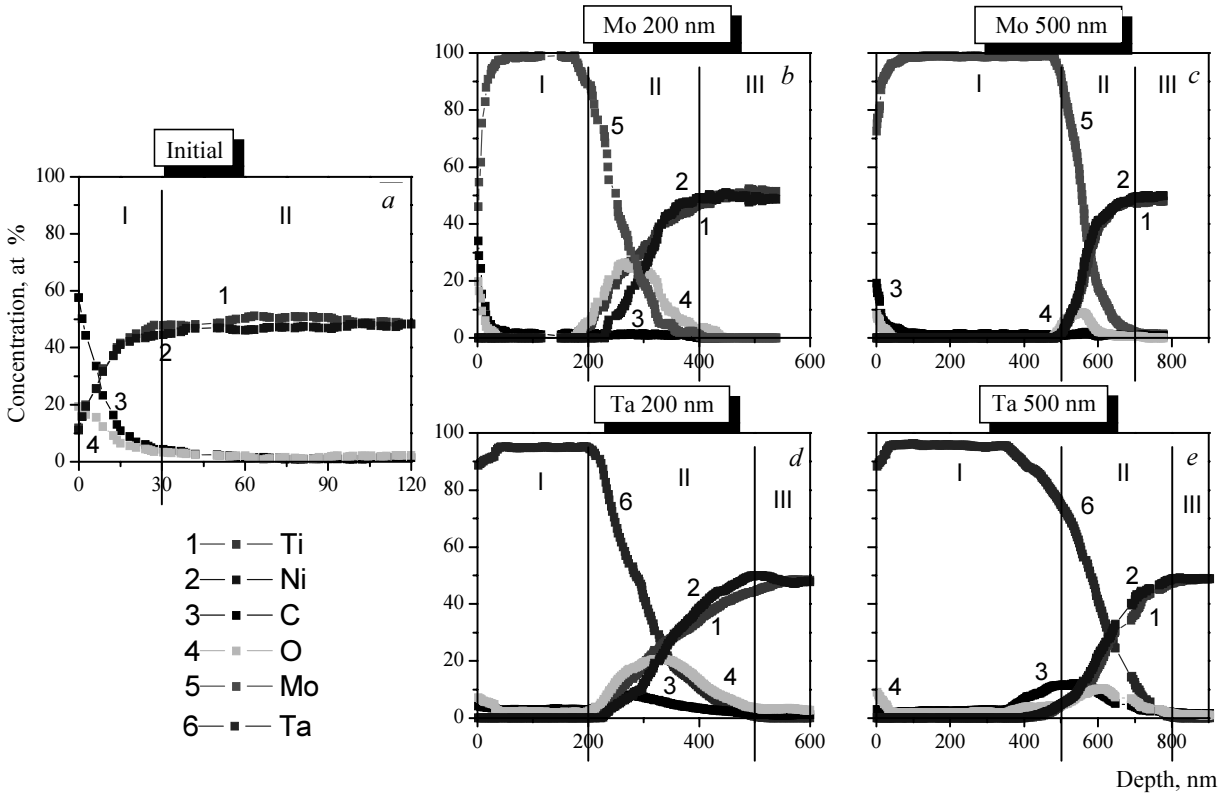

Fig. 1. Elemental composition distribution in superficial layers of alloy $Ti_{49.5}Ni_{50.5}$ with: *a* – electrolytic polished (initial) surface; *b*, *c* – after 200 and 500 nm Mo layer deposition; *d*, *e* – after 200 and 500 nm Ta layer deposition

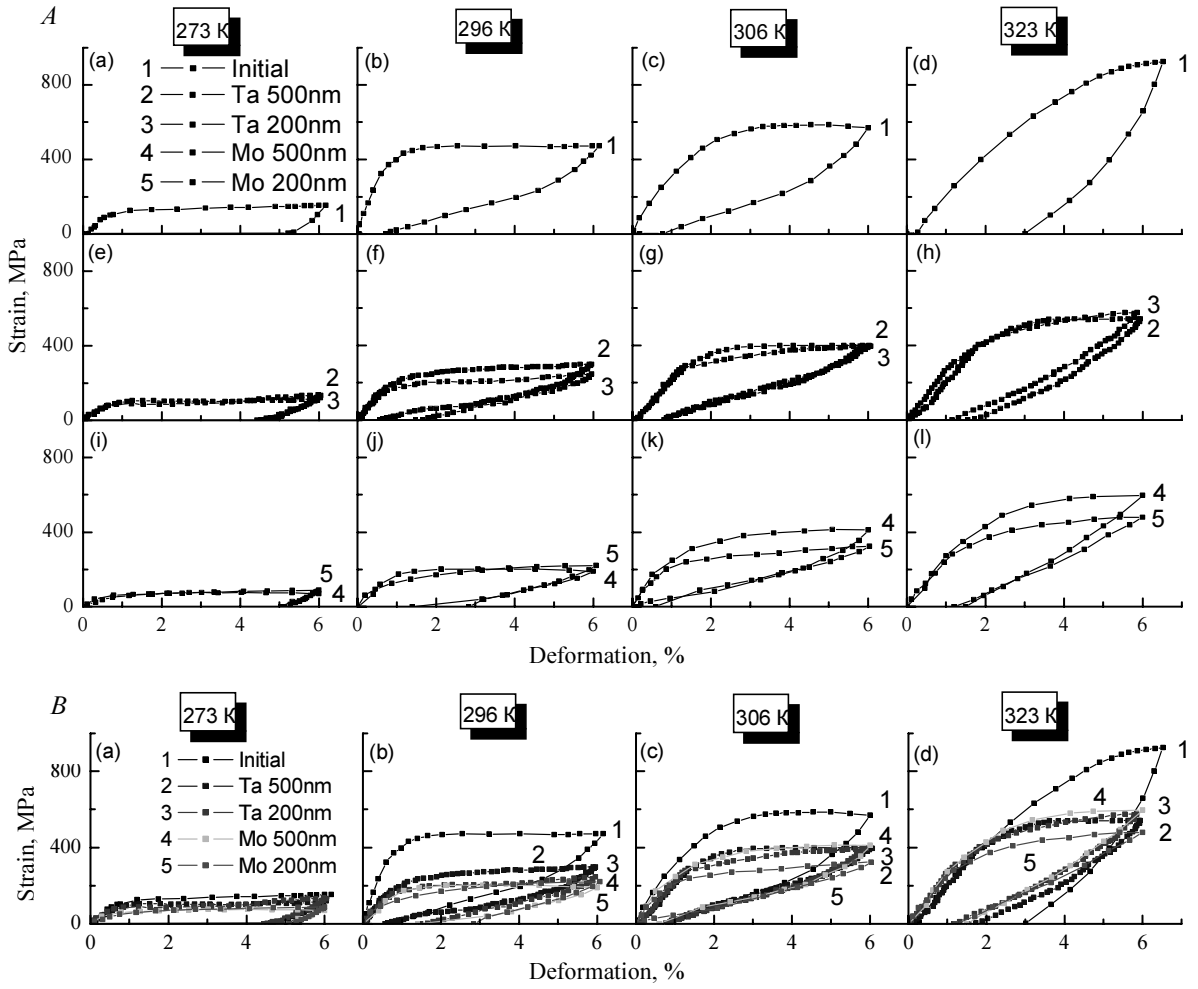

Fig. 2. Loading-unloading diagrams of samples from $Ti_{49.5}Ni_{50.5}$ alloy with a surface: 1 – initial; modified magnetron deposition Ta with thickness of 500 and 200 nm (2–3); Mo with thickness of 500 and 200 nm (4–5)

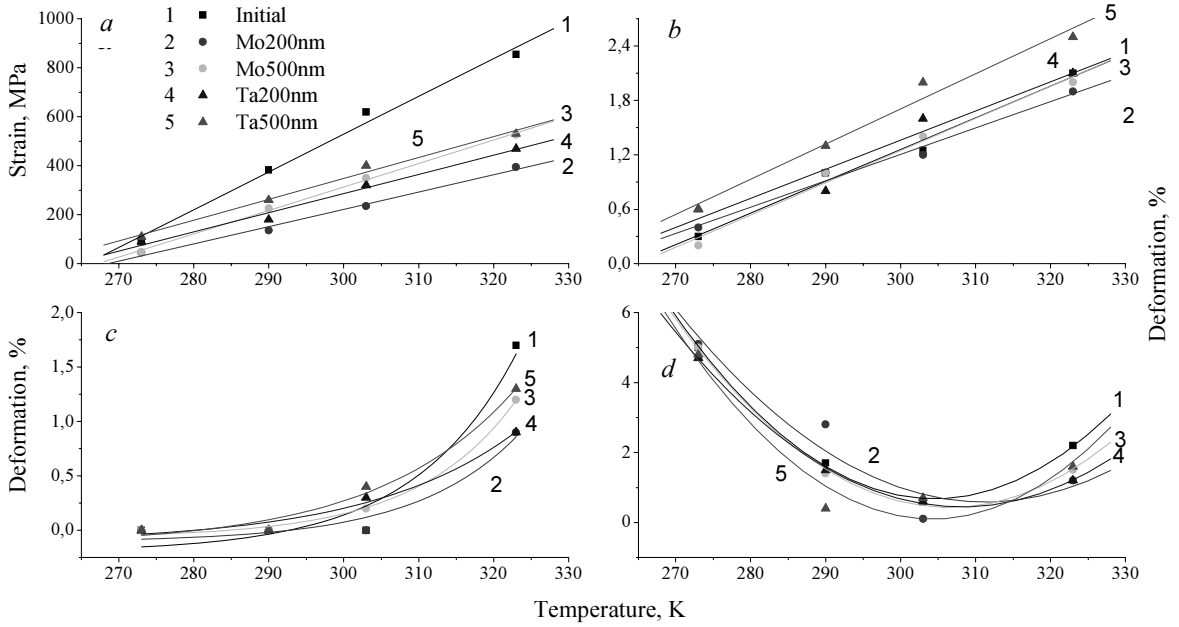

Fig. 3. Temperature dependences: tension of the beginning of a martensate yield plateau τ_m (*a*), deformation of the martensate yield plateau formation beginning γ_m (*b*), residual deformation γ_{res} (*c*), and underretern deformation γ_{ur} (*d*) in initial $Ti_{49.5}Ni_{50.5}$ samples and samples with Mo and Ta coatings

Researches of alloy $Ti_{49.5}Ni_{50.5}$ samples on reactive strain (scheme 2) with the initial and modified surfaces have shown (Table 1) that in an interval of temperatures 273÷720 K samples with the polished surface generate reactive strain magnitude ≈ 500 MPa.

Table 1. Reactive strain generated by alloy $Ti_{49.5}Ni_{50.5}$ samples with different surface condition

Surface condition	σ_p, MPa
Initial	520
Ta200	430
Ta500	400
Mo200	440
Mo500	450

It is revealed that surface modification by deposition thin-film coverings from Mo and Ta reduces magnitude of reactive strain on 15–25%.

4. Conclusion

Researches of $Ti_{49.5}Ni_{50.5}$ based alloy material with various different surface modification with use of a torsion method on superelasticity effect allows to draw following summaries:

1. Samples surface modification by a magnetron deposition method qualitatively has not affected on inelastic behavior of a material with shape memory effect.

2. In case of superficial layers modification appreciable sizes decrease, approximately in 2 times, of a martensate shearing tension τ_m is observed, thus the kind of an deposited element does not influence this size.

3. Surface modification allows raising deformation parameters of superelasticity effect in the given alloy.

4. Surface modification by deposition a thin-film Mo and Ta coverings reduces ability of a material to generate develop reactive strain on 15–25%.

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References

[1] K. Oura, V.G. Lifshits, A.A. Saranin, A.V. Zotov, and M. Katayama, *Surface science. An introduction*, Moscow, Nauka, 2006, 500 pp.

[2] V.G. Pushin, *Nickel titanium alloys with shape memory*, Yekaterinburg, IMP UD RAS, 2006. 438 pp.

[3] V.A. Lichacev, S.L. Kyzmin, and Z.P. Kamentceva, *Shape memory effect*, Leningrad, 1987, 216 pp.