Conducting heat treatment of the TiNiTa alloy and studying the mechanical properties depending on the heat treatment

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Abstract. In this work, a TiNiTa wire with a tantalum content of 10% (weight) was obtained, heat treatment of wire samples was carried out at various temperatures in a vacuum furnace, mechanical tests were carried out after heat treatment to determine the optimal heat treatment mode after the final drawing step to remove hardening and obtain the best complex mechanical properties for the use of wire in the manufacture of medical stents.

1. Introduction

Titanium alloys, in particular alloys with a shape memory effect, are widely used for the manufacture of medical implants due to a unique combination of properties: sufficient strength, low specific gravity, corrosion resistance, biocompatibility, low Young’s modulus. The most interesting in the study now are titanium alloys with a shape memory effect, in particular, NiTi is used as a material for stents. Due to the specific combination of properties, they are considered as functional materials for non-standard solutions to important medical problems. Shape memory alloys are used in various fields of technology (power engineering, mechanical engineering, robotics, agriculture, household and aerospace equipment, etc.), medicine, etc. [1-7]. The main representative of such alloys is NiTi, which has optimal biomechanical properties. However, this material has several disadvantages. The first of them is a high content of carcinogenic nickel, whose ions can enter the body, accumulate and cause negative consequences. The second - since titanium nickelide is a chemical compound, even a slight deviation in the composition affects its mechanical properties, in particular, the austenite-martensite transition temperature.

The TiNiTa alloy looks promising, since doping of the initial TiNi with tantalum made it possible to block the release of nickel ions into the body. Like TiNi, it has the necessary properties, such as the shape memory effect, biocompatibility, but is safer in long-term use. It is interesting to study the effect of heat treatment on the mechanical characteristics of this alloy to identify the best conditions.

The object of the study was samples of TiNiTa alloy with 10% tantalum (weight) in the form of a wire with a diameter of 280 μm. Getting the wire consisted of several stages. The composition of the alloy is shown in table 1. At the first stage, an ingot was produced in a vacuum-arc remelting of the starting metals, which was subjected to 6 remelting and homogenizing annealing at a temperature of 600 °C in a vacuum medium for 16 hours. Next, rolling was carried out at a temperature of 20 °C in several passes with intermediate annealing to remove hardening. Cold rolling is necessary to protect the material from oxidation. Then the workpiece is subjected to rotational forging. At the last stage, drawing was carried out to a diameter of 280 μm with intermediate annealing.
The samples were tested on an Instron 3382 universal testing machine for static tension with a loading speed of 1 mm / min. At least one sample was tested on one experimental point.

2. Main
The studied samples with a working part length of 28 mm were subjected to thermal action according to the selected modes (Table 2). To reduce the influence on the test results of heterogeneous surface heterogeneities, the samples were polished after temperature treatment on a specially created polishing machine based on the SNP-0.1-150V machine to remove the oxide layer, remove contaminants, and reduce roughness. The detailed chemical composition of the wire is presented in table 3.

For polishing, felt disks moistened with a polycrystalline diamond slurry with Akasel DiaMaxx lubricant with sizes of diamond chips 1, 3 and 6 μm with a concentration of 20 carats were used. The number of polishing cycles varied for each diamond chip size from 1 to 50. The wire winding speed was 0.3 m / s. The rotation speed of each of the felt disks is 2500 rpm. Every 25 cycles, felt discs were wetted with a suspension of 20 ml.

Before heat treatment, ultrasonic cleaning of the surface from lubrication after drawing was also carried out. A 5% aqueous solution of Detalan-A10M was chosen as a cleaning liquid. The wire was cleaned for 1 hour in an ultrasonic bath. The frequency of ultrasound is 37 kHz, the power of ultrasound is 30 watts.

Next, the wire was polished by pulling through P1000 paper. The length of the processed section of the wire is 2 m, the number of cycles is 25, the speed of drawing the wire through the rotating disks with grinding paper is 0.3 m / s, the speed of rotation of the disks is 2000 rpm.

Next, ultrasonic cleaning of the wire from the remnants of grease and abrasive material of sanding paper for 30 minutes. As a cleaning fluid - 5% aqueous solution of Detalan-A10M. The frequency of ultrasound is 37 kHz, the power of ultrasound is 30 watts.

Finally, the wire was washed in alcohol to remove residual cleaning agent. The heat treatment was carried out in vacuum furnaces to prevent the formation of titanium oxide. The selected heat treatment modes are presented in table 2.

### Table 1. The chemical composition of the wire.

|          | Ti, % (weight) | Ni, % (weight) | Ta, % (weight) |
|----------|----------------|----------------|----------------|
|          | 39.4           | 50.6           | 10             |

The mechanical properties of the samples depending on the heat treatment are shown in table 3.

### Table 2. Heat treatment mode.

| №1 mode | 30 minutes, 600 °C |
| №2 mode | 30 minutes, 700 °C |
| №3 mode | 30 minutes, 800 °C |
| №4 mode | 30 minutes, 900 °C |

### Table 3. TiNiTa mechanical test results.

| Mode   | Relative extension, % | Tensile strength, MPa | Yield strength, MPa | Average diameter, mm |
|--------|-----------------------|-----------------------|---------------------|---------------------|
| №1 mode | 0.944                  | 518                   | 437                 | 0.277               |
| №2 mode | 1.371                  | 678                   | 530                 | 0.279               |
| №3 mode | 0.696                  | 694                   | 617                 | 0.279               |
| №4 mode | 0.051                  | 548                   | 509                 | 0.274               |
3. Results and conclusions.
The heat treatment at 700 °C leads to an increase in strength characteristics. Heat treatment at 600 °C seems to be insufficient. Heat treatment at 800 °C leads to grain growth and a decrease in elastic properties. Heat treatment at 900 °C leads to embrittlement of the material. The maximum improvement in properties is manifested during heat treatment in vacuum at a temperature of 700 °C.

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