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Influence of annealing on mechanical properties of TiNi (55.8 mass % of Ni) wire made for medical purposes

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Abstract. Mechanical properties of titanium nickelide processed with various heat treatments are investigated. Fractographic, microstructural and X-ray diffraction analysis' were carried out as well as strength and plasticity measurements. It is shown that titanium nickelide (55.8 mass % of Ni) wires treated by two annealing stages, which consist of heating up to 300^{0}C on the first stage and 450^{0}C on the second, with 4 hours and 15 minutes long exposures on each stage respectively, conducted in an air environment, possess the best strength while saving good elasticity figures.

1. Introduction

Alloys possessing shape memory effect are materials, which are capable of shape and mechanical properties alteration in return for temperature change [1]. Nowadays, among all the variety of materials carrying shape memory effect, titanium nickelide (TiNi) appears to be the most widely used material of such kind. It earned such popularity due to its spectacular properties, such as shape memory effect and superelasticity. Within a small range of temperatures, it can exist with low-temperature body-centered cubic lattice (martensite) as well as a high-temperature face-centered cubic lattice (austenite). Transition temperature approximately varies from -50 to 166°C depending on the chemical composition of the material. Due to its unique properties, TiNi is widely used in the manufacturing of bio-medical equipment such as orthopedic arched wires, bone plates etc [2].

Moreover, titanium nickelide is used as a material for the production of medical articles by the type of stent. Stents are mostly used for distension of narrowed cavities in the human body. In the cooled state, stents are plastically deformed before being placed into the catheter to be inserted in the living organism in the future. Inside the organism, stents regain their original form [2-5].

It is essential to use annealing for structure stabilization and shape fixation of medical products made of TiNi [6-13].

This article describes research methods of 280mkm thick wires made of titanium nickelide with 55.8 mass % of Ni and 44.1 mass % of Ti processed by diverse heat treatments in the temperature range of 300 - 500 ^{o}C.

2. Methodology

Researched samples are presented in a form of 280mkm thick wires made of titanium nickelide consisting of 55.8 mass % of Ni and 44.1 mass % of Ti. Detailed information on chemical composition can be obtained from the table 1.

Table 1. Chemical composition of titanium nickelide wires.

Ni(mass %)	Ti(mass %)	C(mass %)	O(mass %)	Other el.(mass %)
55,8	44,1	0,03	0,037	≤0,001*

*Overall quantity of corresponding elements ≤0,027 %

To obtain TiNi wires with optimal mechanical properties, annealing in vacuum heater or high-temperature chamber furnace (ПВК-1,4-А) was used. Rod silicon carbide (SiC) heaters located across the walls of furnace's working chamber were used for heating samples up. Primarily, the furnace's (figure 1) inner empty space was heating up to the temperatures chosen for annealing, only after that samples placed in ceramic cylinders could be placed inside the furnace.

Figure 1. Figure 1High-temperature chamber electric furnace ПВК-1,4-А.

Electromechanical machine INSTRON 3382 was used to carry out static tests of finished samples. The test speed was 1 mm per min. The accuracy of the traverse speed was ± 0,2% of the value of the set speed. The accuracy of load measurement was ± 0.5% of the measured value and up to 1/100 of the maximum value of the load cell. With the help of static tensile tests, the following mechanical properties were determined: yield stress ($\sigma_{0.2}$), tensile strength (σ_t) and elongation (δ). The phase composition was determined using a X-ray diffractometer "Ultima IV" (Cu Kα-radiation) manufactured by "Rigaku", which includes: 1.graphite monochromator intended for use of various polycrystalline inorganic and organic substances, metals and alloys, films and coatings, composites; 2.vertical goniometer; 3.high-speed semiconductor detector "D / teX" with CuKα radiation, which is used to conduct researches using the Bragg-Brentano method, as well as method of oblique shooting with a fixed angle of rotation of the X-ray tube. The phase composition was determined by the PDXL software using the ICDD database.

After static tests, fractographic studies on samples were carried out with help of a scanning electron microscope (SEM) by the name of TESCAN VEGA II SBU equipped with an attachment for the energy-dispersive analysis INCA.

To investigate the microstructure of titanium nickelide wires, its surface was etched for 2-3 minutes with a mixture of the following composition: 50ml C2H5OH + 50ml HCl + 10g CuSO4. After that, each sample was washed with distilled water several times. On the last step of sample preparation, each of them underwent air drying. The studies were carried out on an optical metallographic microscope Olympus GX51. To study the structure each sample was poured by resin to be further pressed-in on the machine for hot pressing Struers CitoPress-20. The final step of preparation, sampled polishing, carried out in the automatic grinding polishing system Struers Tegramin-25.

3. Results and discussion

To obtain stable structure and properties, each sample wire had undergone annealing before the research was carried out. Wires treated by two annealing stages, which consist of heating up to 300^0C on the first stage and 450^0C on the second, with 4 hours and 15 minutes long exposures on each stage respectively, conducted in an air environment, proved to have the best strength while saving good elasticity figures (table 2).

Table 2. Annealed titanium nickelide resulting mechanical properties obtained by conducting the static stretching tests

Sample	δ(%)	$\sigma_{0,2}$(MPa)	σ_t(MPa)
Original titanium nickelide	9,8	640	1301
Titanium nickelide, annealing 390ºC, 30 minutes, vacuum annealing	7,8	347	1830
Titanium nickelide, annealing 450ºC, 30 minutes, annealing in air environment	6,3	255	1766
Titanium nickelide, annealing 450ºC, 30 minutes, vacuum annealing	4,2	269	1640
Titanium nickelide, annealing 500ºC, 15 minutes, annealing in air environment	13,1	349	1585
Titanium nickelide, annealing 350ºC, 4hours, then 450ºC, 15 minutes (air)	8,8	278	1904
Titanium nickelide, annealing 300ºC, 4hours, then 450ºC, 15 minutes (air)	12	340	1777

In order to research the surface of wires treated with drawing and heating, X-ray phase and microstructural analysis were carried out. As it is shown on figure, the bulk of the alloy consists of the B2 phase with a slight presence of Ti2Ni intermetallides, which size ranges from 0.1μm to 3μm and does not change with an increase in the annealing temperature. This inclusion is formed during the crystallization of the alloy and is much harder than the matrix [14].

As the result of fractographic studies of samples, figures 3 and 4 were obtained. It is immediately apparent that samples destruction occurred with the formation of the neck, the fracture surface is oriented almost perpendicular to the extension axis and consists of a set of heterogeneous self-similar viscous fracture pores ("cups"). Upon stretching the trunk fracture in wire samples arises on the surface and then propagates to the inner layers of the samples. The surface pattern of fracture of the near-surface layers differs from that for the inner layers, being visually more complex and having directed character of the relief, in addition, the number of "cups" on the near-surface layers exceeds those on inner-surface layers, "cups" located on the near-surface layers also have much smaller dimensions. The spread of the cup sizes for the fracture of the inner layers is approximately 2 times greater than for the fracture of the near-surface layers. kinks differences of the inner and near-surface layers of the samples indicate that the processes of structural self-organization at the stage of propagation of the main transverse crack was proceeding in them differently.

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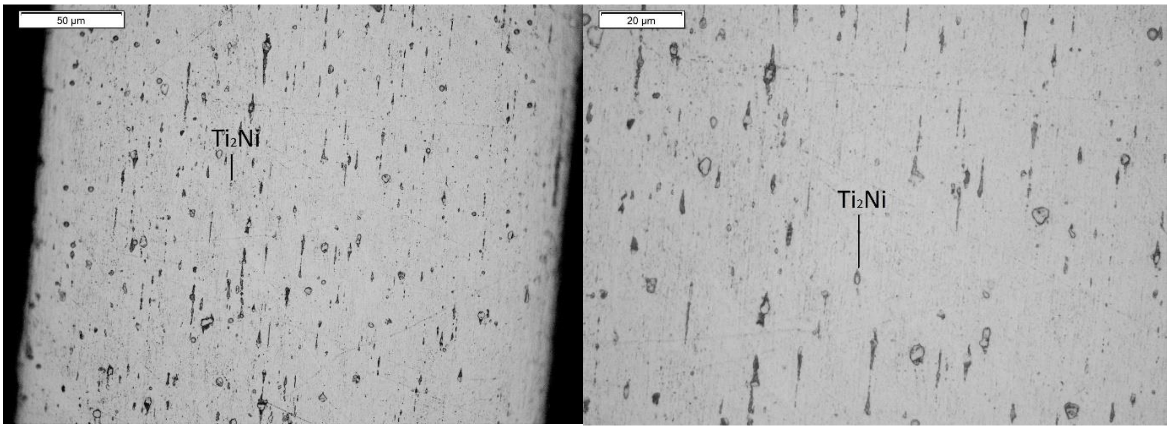
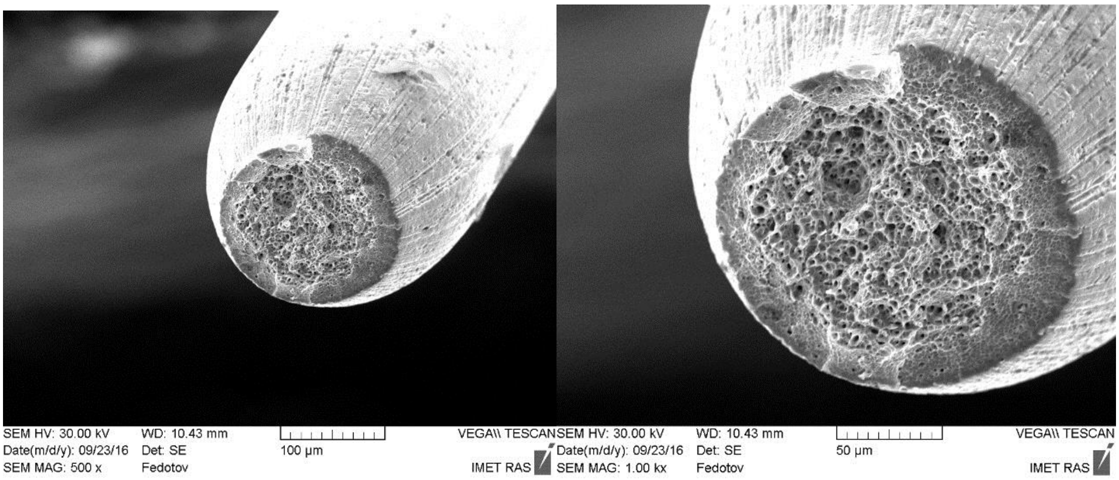
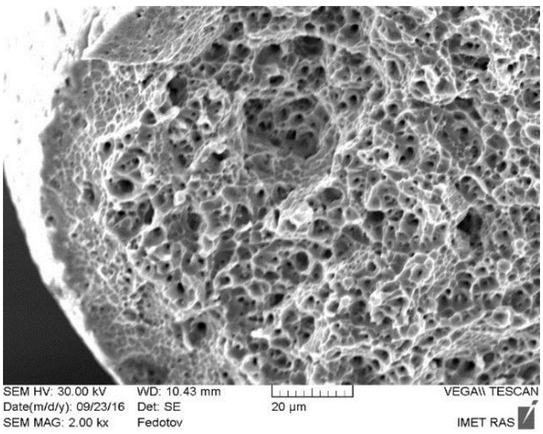


a) b)

Figure 2. Wires microstructure (a – 500x optical magnification, b – 1000x optical magnification).



a) b)



c)

Figure 3. Views on the wire after the destruction (a, b –general view; c - characteristic view on fracture of near-surface layers).

4. Conclusion

The structure of titanium nickelide samples is heterogeneous. Alloy itself consists of the B2 phase and Ti2Ni intermetallides inclusions.

Proper heat treatment mode, which provides titanium nickelide wires with satisfactory mechanical properties, has been obtained. This heat treatment mode consists of two annealing stages, heating up to 300^0C on the first stage and 450^0C on the second, with 4 hours and 15 minutes long exposures on each stage respectively. The whole heating process should be conducted in an air environment.

Acknowledgments

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