INFLUENCE OF TEMPERATURE OF THE SHORT-PERIOD HEAT TREATMENT ON MECHANICAL PROPERTIES OF THE NiTi ALLOY

Jaroslav Čapek[1,*], Jiří Kubásek [1]

[1] Department of Metals and Corrosion Engineering, Institute of Chemical Technology, Prague, Technická 5, 166 28 Prague 6, Czech Republic

[*] corresponding author: Tel.: +420220444055, e-mail: capekj@vscht.cz

Resume

The equiatomic alloy of nickel and titanium, known as nitinol, possesses unique properties such as superelasticity, pseudoplasticity, shape memory, while maintaining good corrosion resistance and sufficient biocompatibility. Therefore it is used for production of various devices including surgery implants. Heat treatment of nickel-rich NiTi alloys can result in precipitation of nickel-rich phases, which strongly influence tensile and fatigue behaviour of the material.

In this work we studied influence of short-period heat treatment on tensile behaviour and fatigue life of the NiTi (50.9 at. % Ni) wire intended for fabrication of surgery stents.

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1. Introduction

The approximately equiatomic alloy of nickel and titanium, known as nitinol, possesses unique properties such as superelasticity, pseudoplasticity, shape memory; moreover it maintains good corrosion resistance and sufficient biocompatibility [1–3]. Therefore, it is used for fabrication of various devices including surgery implants. Nitinol is commonly fabricated by vacuum induction melting (VIM), vacuum arc remelting (VAR), or by their combination [4]. After fabrication, ingots are usually hot and cold worked (forging, rolling, or wire drawing) and these semi-finished products are then treated into the final shape. The fabrication process usually continues with a heat treatment and sometimes with a surface treatment [1, 3].

Heat treatment is very important step to obtain desired properties of the product. Characteristic properties such as transformation temperatures, stress, strength, hardness, ductility and fatigue life could be set very accurately by the right regime of the annealing conditions (temperature, time, presence of stress and environment) [1–3, 5]. It is due to precipitation of nickel rich phases (temperatures up to 600 °C), or their dissolution (higher temperatures), annihilation of dislocation, growth of grains, or recrystallization [6, 7]. Transformation characteristics are mostly influenced by precipitation of nickel rich phases, during which the NiTi matrix is depleted of nickel, which leads to an increase of transformation temperatures. Moreover, the precipitates strengthen the alloy. Therefore, this regime of the heat treatment is often used for superelastic NiTi alloy, which are used for production of medical implants and instruments [1–3, 5, 8, 9].

The final heat treatment is usually performed at temperatures between 300 and 600 °C; in the case of nickel-rich alloys containing up to 51 at. % of nickel, which are used for production of stents. At temperatures up to approximately 500 °C formation of $Ni_{14}Ti_{11}$ and Ni_4Ti_3 [9] is preferred. These precipitates are coherent with the B2 matrix and they act as barrier for dislocation motion. Moreover, precipitation of such nickel-rich phases leads to a decrease of nickel-content in the NiTi matrix, which causes increase of transformation temperatures. Because of very slow recrystallization rate at these temperatures, no significant growth of grains and annihilation of dislocations take place and the material keeps its mechanical strength. It was reported that during annealing at these temperatures lenticular precipitates are formed. Homogeneity of distribution of the precipitates is influenced by the presence of stress. While stress free heat treatment leads to precipitation at grain boundaries and in their vicinity, stress assisted annealing results also in precipitation in grain interior [1–3, 5, 10].

Annealing at higher temperatures leads to the dissolution of the above mentioned precipitates. It causes enrichment of the matrix by nickel and subsequent decrease of transformation temperatures and strength. Moreover, recrystallization becomes significant. Prolongation of the annealing period causes to precipitation of new phases (Ni_3Ti_2 and Ni_3Ti). It results in a repeated increase of transformation temperatures. These precipitates do not have such strengthening effect as the precipitates obtained at lower temperatures. Also longer time of annealing leads to extended recrystallization and grain growth. As a consequence, material has lower strength, but higher ductility and fatigue life [1–3, 5].

A lot of studies have focused on influence of heat treatment conditions on the behaviour of NiTi alloy. Only a few of them; however, were focused on short-time annealing. Therefore, in this study, we studied influence of short-period heat treatment temperature on microstructure and mechanical properties of the Ni-rich NiTi wires.

2. Experimental material and methodology

The nickel-rich NiTi (50.9 at. % of Ni) wire with the diameter of 0.28 mm intended for the fabrication of surgery stents was used as the studied material. The wire was heat treated at different temperatures (450, 510, 530 and 600 °C) for 10 minutes in air. After that the samples were quenched into water at a temperature of 20 °C. The as-received as well as the heat treated wires were subjected to the tensile test using deformation rate $1.667 \cdot 10^{-3}$ s^{-1} on the LabTest5.250SP1-VM universal loading machine and to the bending fatigue test (bending range between 30 and 120 degrees, frequency 3 Hz). Both of these tests were carried out at the temperature of 23 °C and three samples of each set were measured in both tests.

Microstructure of selected samples (as-received and 600 °C) was investigated by a transmission electron microscope (TEM) (Jeol 3010, accelerating voltage of 300 kV, LaB_6) equipped with an energy dispersion spectrometer (EDS).

3. Experimental results

3.1 Microstructure

In Figs. 1-2, there are obvious significant differences between microstructures of the as-received sample and the sample annealed at the temperature of 600 °C. The as-received sample shows typical features of cold working, like large amount of dislocations and elongated grains. On the other hand,the sample annealed at the temperature of 600 °C possesses fully recrystallized microstructure with significantly coarser grains and no precipitates.

3.2 Tensile behaviour

The stress-strain curves of the studied samples are shown in Fig. 3 and the exact values are mentioned in the Table 1. One can see that all curves show characteristic plateau, typical for superelastic materials, and that the tensile behaviour is sensitive to the annealing temperature. The decrease of transformation stress with increasing temperature of the heat treatment is probably caused by partially annihilation of dislocations, precipitation of nickel-rich phases leading to the matrix depletion of nickel in the case of samples annealed at temperatures up to 530 °C. At the temperature of 600 °C; however, recrystallization, dissolution of precipitates and grain coarsening take place. Vanishing of lattice defects and stress in their surroundings, and grain coarsening lead to the decrease of transformation stress.

While transformation stress decreases with increasing annealing temperature, tensile strength increases initially (sample annealed at 450 °C). Higher annealing temperatures have caused that the tensile strengths are lower than the tensile strength of the as-received wire. This could be explained by the superposition of precipitation strengthening and decreasing of transformation stress and softening of the material. Very interesting is the difference between tensile strengths of the samples annealed at temperatures of 510 and 530 °C. While transformation stress of these samples is approximately the same, the tensile strength of the first one is increased by approximately 400 MPa in comparison to the second one. It suggests that coherency of precipitates is changed.

The most significant decrease of the tensile strength was observed in the case of the sample annealed at 600 °C. This decrease is the result of recrystallization and grain coarsening, which is obvious from the shape of the stress-strain curve (high degree of plasticity and high ductility).

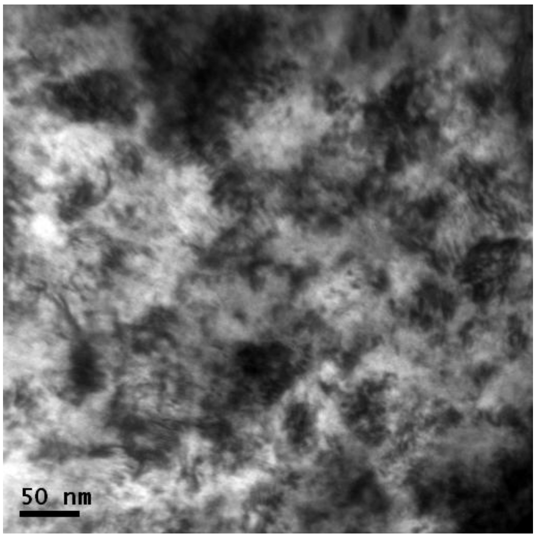

Fig. 1. TEM micrograph of the as-received wire.

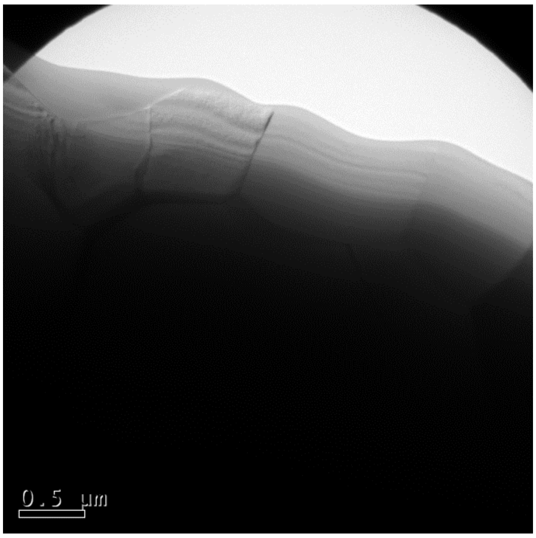

Fig. 2. TEM micrograph of wire annealed at 600 °C.

Table 1

Tensile properties of the studied samples

Sample / property	Transformation stress (MPa)	Tensile strength (MPa)	Ductility (%)
As-received	644	1634	15.3
450 °C	560	1667	14.3
510 °C	452	1514	14.0
530 °C	446	1344	13.1
600 °C	405	991	53.9

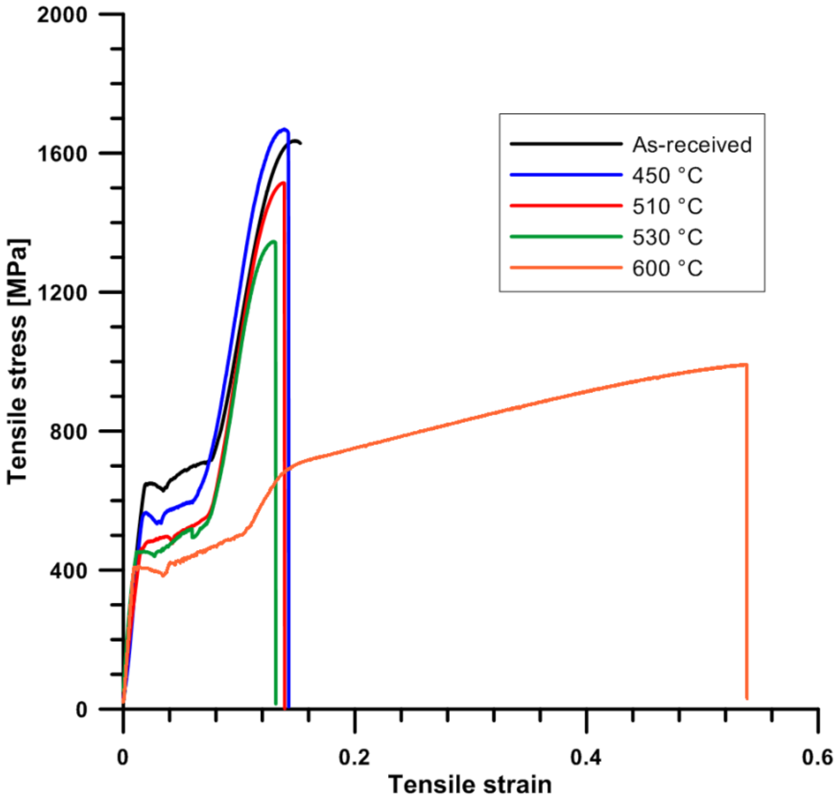

Fig. 3. Stress-strain curves of the studied samples.
(full colour version available online)

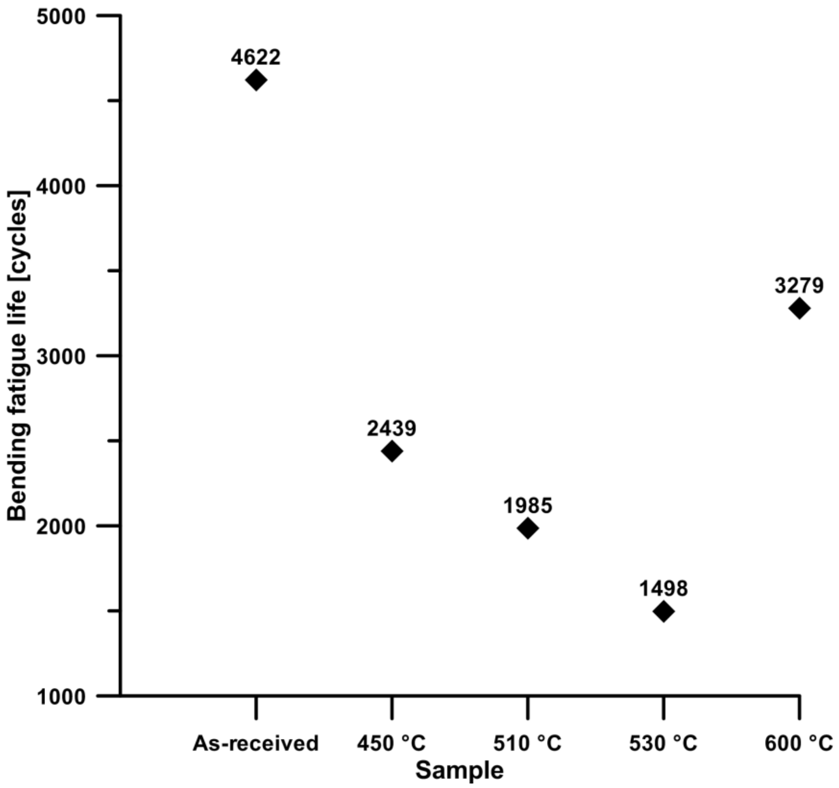

Fig. 4. Fatigue behaviour of the studied samples.
(full colour version available online)

3.3 Fatigue behaviour

The Fig. 4 shows that fatigue life decreases with increasing annealing temperature up to 530 °C. It is probably caused by decreasing of the yield strength of the material. The increase of the fatigue life in the case of sample treated at 600 °C is caused by plasticity of the stress-induced martensite. It is necessary to mention that the obtained values of fatigue life have only an informative character, due to the low amount of tested samples.

4. Conclusions

In this work Ni-rich (50.9 at. % Ni) NiTi wire was heat treated for short-time (10 minutes) at temperature interval 450 – 600 °C in air. Influence of the treatment temperature on tensile behaviour and fatigue life was studied.

An increase of the annealing temperature leads to a decrease of transformation stress. The highest tensile strength was achieved by heat treatment at the temperature of 450 °C. Higher annealing temperatures resulted in a decrease of tensile strengths. It was suggested that this is caused by precipitation of different nickel-rich phases and annihilation of dislocations. The sample annealed at 600 °C; moreover, possessed high ductility. On the other hand, its tensile strength was significantly lower in comparison with the other studied samples. It was explained by TEM observation of this sample. Its microstructure showed features of recrystallization and grain coarsening.

The evolution of microstructure has resulted in changes in fatigue behaviour as well. The highest fatigue life possessed the as-received wire. Heat treatments at temperatures up to 530 °C lead to decrease of fatigue life with increasing treatment temperature. Annealing at 600 °C caused an increase of the fatigue life in comparison with the others annealed samples, but its fatigue life was still lower than fatigue life of the as-received sample.

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