Mechanical properties and fractographic analysis of the Ti$_{49.14}$Ni$_{50.86}$ alloy in a coarse-grained state during multiple martensitic transformations

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Abstract. The mechanical behavior of the coarse-grained Ti$_{49.14}$Ni$_{50.86}$ alloy after multiple martensitic transformations B2-B19' was investigated. A fractographic analysis of the samples after mechanical tests was carried out. The fracture pattern of the Ti$_{49.14}$Ni$_{50.86}$ alloy in the coarse-grained state has a viscous and heterogeneous character with microdepths on the fracture surface, the average size of which decreases as the number of thermal cycles increases.

1. Introduction
TiNi alloys with the shape-memory effect (SME) possess high functional characteristics, enhanced strength, ductility, corrosion resistance, biocompatibility, etc. [1-2]. They have a unique set of properties - high values of recoverable deformation and developed stresses, high corrosion resistance, due to which they are widely used in many industries and medicine [3-4].

At the same time, for many applications, especially in critical or small-sized products, the level of mechanical and functional properties possessed by TiNi alloys in the usual coarse-grained state is insufficient. Since physicomechanical properties are structurally sensitive, methods of deformation-heat treatment are traditionally used to increase them, allowing to obtain various types of structures.

For these alloys, various metallurgical and metallurgical aspects are developed, new alloys and technologies for their processing are created, a systematic study of the structural and phase transformations in them, various properties, including the thermomechanical memory effects, unique in their temperature, deformation and power characteristics, is carried out.

In the development of products made of SMA (shape memory alloys), the following functional characteristics are most important: temperature interval of shape recovery, maximum reversible deformation, maximum fully reversible deformation, degree of shape recovery, similar characteristics of the reversible shape memory effect and superelasticity [1-10].

The heat treatment of this alloy requires high accuracy, as it greatly affects its properties. Heat treatment is the main means for finely controlling the temperature of the phase transformation. The duration and temperature of the heat treatment affect the deposition of various phases Ni-rich and thereby determine the amount of nickel deposited on the nickel-titanium crystal lattice; depleting the nickel matrix, heat treatment leads to an increase in the temperature of the phase transformation. The combination of heat and cold treatment is the main means of regulating the properties of this alloy. An effective method of hardening of structural elements and increasing strength and functional
characteristics is heat treatment and thermal cycling. Thermocyclic treatment (TCT) is accompanied by multiple phase transformations during heating and cooling with optimal speeds.

Shape memory alloys have a unique ability to restore shape upon heating, and it is known that multiple heat exchanges through the temperature range of martensitic transformations lead to a change in their functional properties [1,2].

During thermal cycling in a free state (without stress), a decrease in the temperature of phase transitions and a change in their sequence are observed [1]. If external stress acted upon the sample during thermal cycling, along with a change in the characteristics of martensitic transformations, the parameters of the shape memory effects change, and one-sided irreversible deformation accumulates.

Thermal cycling can have a beneficial effect on the formation of the structure and properties of metallic materials and alloys. In this regard, thermal cycling is included as one of the operations in the production technology of materials. It is important to determine the effect of multiple martensitic transformations on the mechanical behavior and nature of fracture of TiNi alloy in coarse-grained state.

2. Material and methods

In this work, as the material for investigation we used a TiNi intermetallic – Ti$_{49.14}$Ni$_{50.86}$ (manufactured by MATEX, Russia), having a B2 crystalline lattice in the initial state after quenching from a temperature of 800 °C in water. Thermal cycling of the samples was performed via a successive immersion of the samples into liquid nitrogen (-196 °C) followed by heating to a temperature of 150±5 °C, the temperatures which are known to be lower and higher than the temperature of the direct martensitic transformation, $M_s$ and that of the reverse martensitic transformation, $A_s$. The number of “heating – cooling” thermal cycles was from 0 to 250. The exposure time was t=8 min to ensure the complete heating/cooling of the samples. Tensile mechanical tests of small-sized flat samples with a gauge portion of 1*0.25*4 mm were conducted at room temperature with a strain rate of 1*10$^{-3}$ s$^{-1}$ on a Shimadzu AG-50kNXD machine. The structure of the material was studied with a JEM JSM 6390 scanning electron microscope (SEM).

3. Results and discussions

Mechanical tests of the Ti$_{49.14}$Ni$_{50.86}$ alloy were performed. Table 1 shows the results of the study. Based on the data, a consistent increase in the values of $\sigma_{UTS}$ and $\sigma_{YS}$ (yield strength) to n = 100 thermal cycles, starting from n = 150. There is a fluctuation in the parameters $\sigma_{UTS}$, $\sigma_{YS}$ within the measurement error, which it indicates stabilization of the values. The phase yield stress $\sigma_m$ also reaches its maximum value at n = 100 cycles; no further increase in characteristics is observed due to the effect of saturation of the material with defects during thermal cycling. Relative elongation has a maximum value in the initial coarse-grained state; as a result of thermal cycling with a different number of cycles, the value of elongation decreases.

| States | $\sigma_{UTS}$, MPa | $\sigma_{YS}$, MPa | $\sigma_m$, MPa | $\delta$, % |
|--------|---------------------|-------------------|----------------|-----------|
| CG     | 940±15              | 495±10            | 320±5          | 70±3      |
| CG + TC n=50 | 1050±10 | 615±10            | 515±5          | 45±2      |
| CG + TC n=100 | 1065±10 | 700±10            | 550±7          | 35±2      |
| CG + TC n=150 | 1040±15 | 700±10            | 405±8          | 35±3      |
| CG + TC n=200 | 1080±12 | 675±7            | 515±5          | 50±4      |
| CG + TC n=250 | 1075±10 | 685±10            | 505±5          | 55±5      |

Consider fractography of a Ti$_{49.14}$Ni$_{50.86}$ alloy in a coarse-grained state after quenching. According to the SEM analysis (figure 1), the fracture is viscous and heterogeneous. The surface of the fracture
contains numerous cup-like microdimples (pits). The size and shape of the pits are heterogeneous. Discontinuities are observed along the boundaries of the structural element.

![Figure 1](image1.png)

**Figure 1.** Fractography of Ti<sub>49.14</sub>Ni<sub>50.86</sub> alloy in a coarse-grained state. Figure (a) shows a general view. In figure (b) the fracture surface with microdimples.

After thermal cycling with the number of cycles equal to 50, analyzing the images obtained using scanning electron microscopy (figure 2), shear fracture zones were observed at the fracture of the sample. Tearing-off pits are noticeable throughout the fracture area. The microrelief of this fracture consists of microdimples on the fracture surface, which are exposed micro-hollow surfaces. The average pit size is \( \sim 10 \pm 2 \, \mu\text{m} \).

![Figure 2](image2.png)

**Figure 2.** Fractography of Ti<sub>49.14</sub>Ni<sub>50.86</sub> alloy in a coarse-grained state after 50 thermal cycles. Figure (a) shows a general view. In figure (b) the fracture surface with microdimples.

With an increase in the number of thermal cycles to \( n = 100 \), it can be seen that the nature of the fracture is inhomogeneous (figure 3). The kink is characterized by the presence of zones that differ in macrorelief. In one of them, the formation of discontinuities along the boundaries of the structural element are notice. The border between the zones at the macro level is highlighted by a color change during the transition from one zone to another.
Figure 3. Fractography of Ti_{49.14}Ni_{50.86} alloy in a coarse-grained state after 100 thermal cycles. Figure (a) shows a general view. In figures (b) and (c) the fracture surface with microdimples.

In a coarse-grained alloy, which underwent thermal cycling with the number of cycles n = 250, the microrelief of the fracture of the sample has a dimple character, inside of which inclusions are visible (figure 4). The average pit size is approximately 5 ± 2 μm.

Figure 4. Fractography of Ti_{49.14}Ni_{50.86} alloy in a coarse-grained state after 250 thermal cycles. Figure (a) shows a section of an inhomogeneous fracture. In figure (b) the fracture surface with microdimples.
4. Conclusions
As a result of these studies, we can conclude that with multiple martensitic transformations, the values of tensile strength, yield strength, and phase yield strength increase to \( n = 100 \) thermal cycles, then, starting from \( n = 150 \) cycles, the values of mechanical characteristics are stabilized. Relative elongation has a maximum value in the initial coarse-grained state; during thermal cycling and an increase in the number of cycles, the value of elongation decreases.

The fracture pattern of the Ti\(_{49.14}\)Ni\(_{50.86}\) alloy in the coarse-grained state has a viscous and heterogeneous character with microdimples on the fracture surface, the average size of which decreases with an increase in the number of thermal cycles.

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