Deformation-force effects of shape memory of TiNi alloys under conditions of incomplete martensitic transformation

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Abstract. The article is devoted to studying the influence of preliminary and plastic processing, applied stress, degree of phase transformation on the deformation effects manifested in conditions of incomplete martensitic transformation. The laws of generation and relaxation of reactive stresses were studied depending on the stiffness of the reaction, the magnitude of the preliminary deformation and the selected interval of heat changes. It is shown that the accumulation and return of deformation, the effect of generation and relaxation of reactive stresses in a narrow temperature range occurs only in structurally inhomogeneous objects, determined with the statistical spread of characteristic temperatures.

1. Introduction

Materials with the effect of shape memory, due to their unique properties, are widely used in many fields of technology [1, 2]. Most often, titanium nickelide and alloys based on it are used as thermosensitive elements. In a series of materials with reversible martensitic transformations, TiNi-based alloys occupy a special place due to the unique combination of mechanical and physical properties and high values of the shape memory effect. However, the presence of a temperature hysteresis of 40-60 K complicates the task of using a material with shape memory effect in devices that maintain a given temperature regime in a narrow range of 5–15 K (relay sensors, instrument containers, thermostats, etc.) [3]. It is not possible to significantly reduce the conversion hysteresis due to a change in the chemical composition of the alloy or in plastic and heat treatment [4, 5]. The only possible way to perform regulatory functions with thermostet elements of titanium nickelide in a narrow temperature range is to realize the effects of shape memory, generation and relaxation of reactive stresses under conditions of incomplete phase transformation. The urgency of the problem is also determined with the fact that the properties of shape memory during heat changes in the interval of incomplete martensitic transformation (IMT) are practically not studied. There are no systematic studies on the influence of the structural state of the material, loading conditions, and the interval of heat changes on the deformation-force effects of shape memory. Without these data, it is impossible to design and create workable mechanisms and devices operating in a narrow temperature range.

2. Objects and research methods

As objects of study, TiNi alloys with a content of 49.5, 50.0, 50.2, 50.5 at % Ni were chosen, in which, depending on the heat and plastic processing, various phase transition chains and, accordingly, various
loop shapes of hysteresis can be observed. The samples were made of wire with a diameter of 2 mm and cantilevered beams of polycrystalline materials subjected to the following types of pretreatment.

1) Quenching from 1073 K in water.
2) Cold rolling with compression strain \( \varepsilon = 15–20\% \) and subsequent annealing at a temperature of 723K for 2 hours.
3) Drawing at a temperature of 823-873K with a reduction ratio of 10–12% and subsequent annealing, \( T_{\text{TOT}} = 723\text{K}, \tau_{\text{TOT}} = 2 \text{ hours} \).

The experimental studies were performed on two specially designed installations, which allow measuring the deformation-force parameters of selected objects in a given temperature range under conditions of constantly applied stress, constrained deformation with finite and infinite stiffness of the opposing system. X-ray diffraction studies of the phase composition of alloys at various temperatures were carried out on a DRON-2.0 X-ray diffractometer using an attachment for heating (cooling) the test sample.

3. Experimental results and discussion

Thermomechanical loops in the incomplete MT interval were formed on the complete hysteresis curve obtained after 10–15 preliminary thermal cycles. At the stage of direct MT at the temperature of \( M_{\text{KN}} \) (the temperature of the end of the incomplete martensitic transformation), cooling was replaced with heating to the temperature of \( A_{\text{KN}} \) (the temperature of the end of the incomplete austenitic transformation) and thermal cycling was performed in this interval \( \Delta T_{\text{H}} = A_{\text{KN}} - M_{\text{KN}} \). In a similar way, small loops were formed from the heating stage. The experiments showed (figure 1) that regardless of the location of the heat transfers interval relative to the characteristic temperatures and its magnitude, the thermal deformation line always closes, forming a hysteresis loop (small loop). That part of the deformation that has accumulated during the cooling process is completely restored during the heating process.

![Figure 1](image-url)  
Figure 1. The accumulation and return of deformation in a Ti-50 at% Ni alloy under a constant voltage \( \sigma = 35 \text{ MPa} \) during heat transfer under conditions of IMT from the stage of cooling (a) and heating (b).

It was established that the nature of the change in deformation during heat changes in a limited temperature range is determined with the structural state of the material, the shape of its full hysteresis figure (figure 2).
In quenched alloys with a homogeneous structure and a wide hysteresis loop (ratio of characteristic temperatures ($A_K > M_H$)), the shape of the thermodeformation curves of the incomplete transformation does not depend on the temperatures of the onset of heat changes (formation conditions) and it is characterized with a wide hysteresis of termination. Accumulation and return of deformation does not occur until the interval of temperature change exceeds the width of the total hysteresis (figure 2a). An increase in the heterogeneity of the structure of the alloys, associated with an increased nickel content ($C_{Ni} = 50.2–50.5$ at%) and plastic processing, leads to a change in the shape of the full hysteresis loop ($A_K < M_H$) and affects the kinetics of accumulation and return of deformation in the temperature range of the IMT. The shape of the small hysteresis loops changes depending on the specified heat exchange interval, and deformation is observed even with a slight change in temperature (figure 2b).

X-ray structural studies showed that in the quenched alloys with a nickel content of 49.5–50.2 at%, only the $B_2 \rightarrow B_{19}'$ martensitic reaction occurs, and in the plastic treated alloys Ti-50.5 at% Ni, along with the $B_2 \rightarrow B_{19}'$ reaction, phase reaction occurs according to the $B_2 \rightarrow B_{19}'$ scheme, characterized by a narrow transformation hysteresis. As a result, it can be noted that the structural heterogeneity of the alloy and the presence of volumes with different intervals of martensitic transformations make it possible to accumulate and return strain at sufficiently small temperature changes, which was also noted in other studies [6].

The study of the influence of the heat change interval on the magnitude of the deformation of the small loop (the value of the reversible deformation that accumulated during cooling and fully recovered during heating $\varepsilon_{cool}^r$) shows the following result. The maximum value of the reversible deformation is achieved if the initial temperatures of the heat change interval $M_K$ or $A_K$ are located in the middle of the $M_H$–$M_K$ intervals or $A_H$–$A_K$, that is with the degree of phase transformation $\eta = 0.4–0.6$ (figure 2).

The degree of direct $\eta_M$ and reverse $\eta_A$ martensitic transformation is determined with the relation

$$\eta_M = \frac{M_H - M_K}{M_H - M_K} \text{ or } \eta_A = \frac{A_K - A_H}{A_K - A_H}$$

(1)
Figure 3. Change in the reversible deformation $\varepsilon_{\text{re}}$ in the Ti-50 at% Ni alloy under a stress of $\sigma = 35$ MPa as a function of the degree of phase transformation $\eta$ during heat changes from cooling stage (a) and heating (b) in a given temperature range $\Delta T_H$.

It was established that the kinetics of changes in reactive stresses is also determined with the structure of the material, the shape of its complete hysteresis loop [7, 8]. The effect of stress generation and relaxation during heat changes in a narrow temperature range not exceeding the width of the total hysteresis is observed only in structurally inhomogeneous alloys (figure 4).

Figure 4. The effects of the generation and relaxation of reactive stresses in the conditions of IMT with infinite stiffness of reaction ($K \rightarrow \infty$, $\varepsilon_{\text{pr}} = 1.2\%$) in alloys: a) Ti-49.5 at% Ni (quenching from 1073 K in water); b) Ti-50.2 at% Ni (rolling $\varepsilon_{\text{ob}} = 15\%$, $T_{\text{OT}} = 723$K, $\tau_{\text{OT}} = 2$ hours).

The influence of the heat change interval, the degree of preliminary deformation, the stiffness of the counteraction on the level of reactive stresses $\sigma_{\text{r}}$ generated when heated to the temperature of the $\Delta T_H$, and the voltage swing $\Delta \sigma$ of the small hysteresis loop are studied. It was found that the stress range $\Delta \sigma$ increases linearly with increasing maximum reactive stresses $\sigma_{\text{r}}$ generated in the alloy upon heating above $AK_\sigma$ temperature (figure 5).
Figure 5. The dependence of the magnitude of the stress range $\Delta \sigma$ of the small loop on the maximum level of generated stresses $\sigma_{r\text{max}}$ in the Ti alloy – 50.2 at% Ni.

Studies have shown that the proportionality coefficient (relative stress range) $\alpha = \Delta \sigma / \sigma_{r\text{max}}$ depends on the cycling interval and the degree of reverse phase transformation (Fig. 6). The level of reactive stress $\sigma_r^{\text{m}}$ is also determined with the maximum stresses $\sigma_{r\text{max}}$ and can be found from the relation.

$$\sigma_r^{\text{m}} = \eta_A \sigma_{r\text{max}}$$  \hspace{1cm} (2)

where $\eta_A$ is the degree of austenitic transformation.

It was established that an increase in the statistical dispersion of characteristic temperatures leads to a transformation of the complete hysteresis loop (the slope of the hysteresis figure increases, the temperature Mn exceeds the temperature of the $A_K$) and to a narrowing of the incomplete conversion hysteresis loop. In structurally heterogeneous materials, a change in deformation or stress occurs virtually at any temperature change.

Figure 6. Change in the relative amplitude of the stress $\Delta \sigma / \sigma_{r\text{max}}$ of the small loop in the Ti-50.2 at% Ni alloy from the cycling interval $\Delta T_{\text{H}}$ at specified values of the degree of inverse MP ($\eta_A$).

4. Conclusion
Studies have shown that the thermomechanical hysteresis of an incomplete transformation depends on the shape of the full hysteresis figure. In alloys with a wide hysteresis loop, accumulation or return of deformation in the temperature range less than the width of the total hysteresis is not observed. In alloys
with a narrow hysteresis of phase transformations, thermomechanical small loops are formed in a small temperature range (5–10 K). It has been established that the structural heterogeneity of the alloy, with the presence of volumes with different intervals of martensitic transformations, makes it possible to manifest the effects of shape memory, generation and relaxation of reactive stresses at sufficiently small temperature changes. The regularities of the change in the shape of small hysteresis loops depending on the location of the heat change interval relative to the characteristic temperatures are revealed. The maximum value of the deformation range or stresses of the small hysteresis loop is achieved during heat changes with an initial degree of phase transformation lying in the range 0.4–0.6. It was found that with increasing stress, the magnitude of the reversible deformation $\varepsilon_{\text{opf}}$ during heat changes in the temperature range not exceeding the width of the total hysteresis increases slightly, and it is determined largely with the initial temperature and thermal cycling interval.

It was found that the magnitude of the thermosilic hysteresis stresses increases linearly with increasing maximum level of reactive stresses generated in the alloy, and the proportionality coefficient (relative stress range) is determined with the heat change interval, the degree of phase transformation, and the alloy structure.

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