Impact of thermocycling type on nature of changing reversible deformations in titanium nickelide

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Abstract. For cyclic functioning of mechanisms for various functional purposes based on materials with a shape memory effect (SME), it is necessary to possess skills of forming the characteristics of a cyclic shape memory, i.e. the shape memory effect implemented upon heating and plasticity of direct transformation observed upon cooling of the material under load. For this purpose, a systematic experimental research of the impact of a type (mode) of thermocycling of samples of titanium nickelide under torsion on the nature of changing reversible deformations in the process of thermocycling was performed. To assess quantitatively the effectiveness of a thermocycle, the value of a thermocycle completeness coefficient was introduced which is determined as the ratio of the value of the deformation restored for a cycle to the value of the deformation accumulated for a thermocycle. Three modes of loading were studied.

1. The material was loaded in a martensitic state to a certain level, then it was heated through the interval of a reverse martensitic transformation, in the austenitic state it was unloaded and cooled to its original state.
2. The material was loaded in a martensitic state, then heated through the interval of a reverse martensitic transformation to an austenitic state and cooled to its original state.
3. The material loaded in a martensitic state was unloaded. Then it was heated through the interval of a reverse martensitic deformation to an austenitic state in a free state, where it was loaded and the loaded material was cooled to a martensitic state. For all thermomechanical modes of loading upon heating, the value of the deformation restored and accumulated upon cooling was defined experimentally.

It has been experimentally found that the highest value of the restored deformation is implemented in the third loading mode, and the lowest one – in the first one. The intermediate deformation is implemented in the second mode.

The highest value of the deformation determined by the effect of the direct transformation plasticity takes place in the third mode.

1. Introduction
Alloys with a shape memory effect (SME) are capable to demonstrate the reversible shape changing during thermocycling through the intervals of martensitic transformations under a mechanical stress [1]. Herewith, at the stage of heating the SME itself occurs as a rule, and it is implemented in the direction opposite to the acting loading, and at the cooling stage the accumulation of the deformation in the direction of the acting force, or a direct transformation plasticity (DTP), is observed. In the work, the
systematic study of the impact of loading mode type, the values of acting stresses – \( \tau \) and the number of thermocycles – \( N \) on the values of deformations restored when heated, through the interval of reverse martensitic transformation, and accumulated in the process of the direct transformation plasticity has been made.

In the work [2], the example of the TN-1 alloy has shown that, if the thermocycling is made according to the second mode by the way of thermocycling under loading through the intervals of martensitic transitions, then the value of the SME deformation implemented upon subsequent heating will be essentially higher that that after an isothermic loading and subsequent unloading at temperature \(-T_D\). Herewith, the value of the ratio \( \frac{\gamma_p}{\gamma_T} \) depending on the value \(-T_D\) and the level of acting loadings varies from 1.4 to 27.0. All above stated testify that the most effective way of forming the reverse deformation, and, particularly, of the shape memory effect includes thermocyclic variants of impacting on the material under loading.

The purpose of the present work is to perform a systematic experimental study of the impact of a certain type of thermocycling titanium nickelide samples on the nature of changes in reversible deformations, as well as to make an analytical model to quantify this impact.

2. Materials and methods of the experiment

The TN-1 (53.5-56.5 Ni, 43.2-46.5 Ti% by weight) alloy with the characteristic temperatures of martensitic transitions \( M_s = 326 \, K \), \( M_F = 298 \, K \), \( A_S = 365 \, K \), \( A_F = 395 \, K \) was studied. Smooth cylindrical samples, with the length and the diameter of the working part 33 and 4 mm correspondingly, were exposed to low temperature annealing in 823 K for 1 hour in the state before the study. Next, the experiments in three modes detailed in [3, 4] were performed. In all modes, the thermocycling was performed within the interval of temperatures \( T_{\text{min}} - T_{\text{max}} \). Minimal temperature matched the martensitic state at \( T_{\text{min}} = 291 \, K \), and maximal temperature matched the austenitic state at \( T_{\text{max}} = 520 \, K \). In all experiments two complete thermocycles within the interval of temperatures \( T_{\text{min}} \leq T \leq T_{\text{max}} \) were performed according to the following three modes of thermocyclic impact:

**The first mode:** heating from the martensitic state to the austenitic one under the constant stress \( \tau_s = \tau \); unloading in the austenitic state to the stress \( \tau_0 = 0 \); cooling from the austenitic state to the martensitic one in the unloaded state; loading in the martensitic state to the stress \( -\tau_s \), where \( \gamma_p \) – the deformation of the shape memory, figure 1.

![Figure 1. First mode circuit.](image)

**The second mode:** Heating from the martensitic state to the austenitic one and cooling from the austenitic state to the martensitic state under constant stresses \( \tau_s = \tau_0 = \tau \), where \( \tau_s \) and \( \tau_0 \) – stresses at the stages of cooling and heating correspondingly, where \( \gamma_{tp} \) – the deformation of transformation plasticity, figure 2.
The third mode: Unloading in the martensitic state from stress $\tau = \tau_0$ to $\tau = 0$; heating without loading to the austenitic state; loading in the austenitic state to the value of stresses $\tau_0 = \tau$; the next cooling to the martensitic state under stress, figure 3.

Experiments were made according to the techniques detailed in [5]. Thermocycling was made with the average speed of changing temperature $|\dot{T}| \approx 0.08$ degree/sec. Herewith, the measuring of the value of the temperature $T$ was made with the help of torque chromel-copel thermocouple with accuracy to 2°C. At the heating and cooling stages, the steering angle of cross section of the sample $\varphi$ was measured. Herewith, the accuracy of measuring the angle $\varphi$ was 0.009 rad, and that of the torque $M$–0.01H·m. Values of the integral characteristics $M$ and $\varphi$ allowed calculating the estimated values of shear stresses and deformations referred to the outer fiber of the working part of the sample to the approximation of an ideally-plastic hinge. Herewith, the following working hypotheses were used in the basis of estimating:

1. Hypothesis of flat sections is performed: the transverse section of a beam, flat and perpendicular to the longitudinal axis, remains flat and perpendicular to the longitudinal axis after being deformed.
2. Radius drawn from the center of the beam’s cross section remains a straight line (does not bend) after the deformation.
3. The distance between the cross sections does not change after the deformation. The beam’s axis does not bend, the diameters of the cross sections do not change.

These suggestions allow finding estimated values of shear stresses and deformations:
Figure 1–3 demonstrates the functional modes of thermomechanical impact on the samples.

3. Results of the experiments

Figures 1–3 demonstrates the functional modes of thermomechanical impact on the samples.

In figure 1, the deformation $\gamma_p$ is restored when heated, the deformation $\Delta \gamma_A$ is back additionally in the austenitic state under the next isothermal unloading. The deformation does not change with the following cooling from the austenitic state to the martensitic one, the isothermal additional loading of the deformation in the martensitic state gives accumulation of the deformation $-\Delta \gamma_M$.

In figure 2, the heating stage corresponds the deformation restoration by the value $\gamma_p$, and the cooling stage – the accumulation by the value $\gamma_p$.

In figure 3, the deformation $\Delta \gamma_M$ is restored in the martensitic state under isothermal unloading, during the following heating in the free state the deformation is restored by the value $\gamma_p$, the following isothermal additional loading in the austenitic state gives accumulation of the deformation by the value $\Delta \gamma_A$, and during the cooling in the unloaded state the deformation $\gamma_{tp}$ is accumulated. To quantify the experimental results, a coefficient of thermocycle completeness was introduced – as the ratio:

$$k_c = \frac{\gamma_{tp}}{\gamma_{sf}}; \quad (1)$$

where $\gamma_{sf}$ and $\gamma_h$ – the accumulated deformation and the deformation restored for a thermocycle.

In the first mode: at $\tau = \tau_t$; $\tau_0 = 0$; $\gamma_h = \gamma_p + \Delta \gamma_A$; $\gamma_{sf} = \gamma_0 + \Delta \gamma_M = \Delta \gamma_M$; $\gamma_0$ – the deformation implemented during the cooling at $\tau_0 = 0$, which is equal to zero in the described experiments; $\Delta \gamma_A$ – the deformation removed under the isothermal unloading in the austenitic state; $\Delta \gamma_M$ – the deformation accumulated under the isothermal loading in the martensitic state. Then:

$$k_c = \frac{\gamma_h}{\gamma_{sf}} = \frac{\gamma_p + \Delta \gamma_A}{\gamma_0 + \Delta \gamma_M} = \frac{\gamma_p + \Delta \gamma_A}{\Delta \gamma_M}; \quad (2)$$

In the second mode: at $\tau = \tau_t$; $\gamma_h = \gamma_p$; $\gamma_{sf} = \gamma_{td}$; then

$$k_c = \frac{\gamma_h}{\gamma_{sf}} = \frac{\gamma_p}{\gamma_{tp}}; \quad (3)$$

In the third mode: at $\tau = \tau_t$; $\gamma_h = \Delta \gamma_A$; $\gamma_p = \Delta \gamma_A$; $\gamma_0 = \Delta \gamma_M$ – the deformation returned into the martensitic state under the isothermal unloading; $\Delta \gamma_A$ – the deformation accumulated in the austenitic state under the isothermal additional loading.

$$k_c = \frac{\gamma_{sf}}{\gamma_h} = \frac{\Delta \gamma_M + \gamma_{tp}}{\Delta \gamma_A + \gamma_{tp}}; \quad (4)$$

Experimental dependencies of the deformations conditioned by the SME, DTP, as well as of the values $k_c$ of the number of thermocycles for the second loading mode are presented in figure 4–6, which show that $\gamma_p$ increases monotonously with the number of thermocycles, reaching its saturation at $N = 4$, figure 4.

The deformation conditioned by the DTP decreases monotonously during the thermocycling process, coming to saturation at $N = 4$, figure 5. In figure 6, there is a dependence of completeness coefficient on the number of thermocycles for the second mode, which shows that $k_c$ increases monotonously with the number of cycles. Herewith, we can observe that the higher is the level of acting stresses, the lower is the completeness coefficient.
Figure 4. Dependences of memory deformation on the number of thermocycles in the first – (1, 2), curve 1 at $\tau = 300$ MPa, curve 2 at $\tau = 400$ MPa; in the second (3, 4, 5, 6) (curves 3 at $\tau = 100$ MPa; curve 4 at $\tau = 200$ MPa; curve 5 at $\tau = 300$ MPa; curve 6 at $\tau = 400$ MPa) and in the third mode (7, 8) (curve 7 at $\tau = 300$ MPa; curve 8 at $\tau = 400$ MPa).

Figure 5. Dependences of direct transformation plasticity on the number of thermocycles in the second (3, 4, 5, 6) (curves 3 at $\tau = 100$ MPa; curve 4 at $\tau = 200$ MPa; curve 5 at $\tau = 300$ MPa; curve 6 at $\tau = 400$ MPa) and the third thermocycle (7, 8) (curves 7 at $\tau = 300$ MPa; curve 8 at $\tau = 400$ MPa).

Figure 6. Dependences of completeness coefficient on the number of thermocycles in the first – (1, 2) in the second (3, 4, 5, 6) (curves 3 at $\tau = 100$ MPa; curve 4 at $\tau = 200$ MPa; curve 5 at $\tau = 300$ MPa; curve 6 at $\tau = 400$ MPa) and in the third mode (7, 8) (curves 7 at $\tau = 300$ MPa; curve 8 at $\tau = 400$ MPa).
Figures 4–6 presents the impact of the number of thermocycles on the values $\gamma_p$, $\gamma_{tp}$, and $k_c$ for the levels of acting stresses 300 and 400 MPa for the first and third mode, as well as for the levels of acting stresses 100, 200, 300 and 400 MPa for the second mode.

Herewith, the following is justified: the highest values – $\gamma_p$ are implemented in the third mode – heating in the free state, cooling under stress – and reach 15–16%, the intermediate value is implemented in the second mode – thermocycling under stress – and reach 8–9%, and the lowest value in the first mode – heating under stress, cooling in the free state – reach 6%. Herewith, it is important to note that in all cases the value $\gamma_p$ for 300 MPa was higher than at 400 MPa, which is connected with the fact that, under stresses 400 MPa, reversible components of the deformation are suppressed due to irreversible components of deformations because of the effect of thermocyclic creep implemented in the materials [6, 7], so suppression of the SME occurs in the transition from stresses 300 MPa to stresses 400 MPa. Herewith, the following regularities are present: $\frac{d\gamma_p}{dN} \geq 0$; $\frac{d^2\gamma_p}{dN^2} \leq 0$, figure 4.

Figure 5 presents the dependences of deformations conditioned by the effect of a direct transformation plasticity (DTP) on the number of thermocycles for the first and third thermocycling mode. Here, it is important to note that in the second thermocycle the DTP effect itself is absent, as the circuit in figure 2 shows. Figure 5 shows that the DTP deformation is higher under stress 300 MPa than 400 MPa. It is characteristic that the DTP deformation for the third mode is about two times higher than for the second mode. The value of the DTP deformation decreases monotonously with the number of cycles.

Figure 6 presents the dependences of the completeness coefficient on the number of thermocycles. For all modes the monotonous growth of the deformation with the number of cycles is characteristic. Herewith, $\frac{d\gamma_p}{dN} > 0$; $\frac{d^2\gamma_p}{dN^2} < 0$ was characteristic. Herewith, the following was justified: the most complete cycle was observed in the third mode, curves 5–6, and the least complete cycle – in the first mode, curves 1–2. Intermediate values of the completeness coefficient occur in the second mode, curves 3–4.

![Figure 7](image)

**Figure 7.** Dependences $\gamma_p$ curve (1) and $\gamma_{tp}$ curve (2) on the number of a testing mode. $\tau = 300$ MPa, at N=2.

Figure 7 presents the dependences of the deformations conditioned by the SME and DTP on the number of a loading mode which show that the values of the SME deformation are minimal in the first mode, intermediate in the second mode, and maximal in the third loading mode. Deformation conditioned by the DTP is absent in the first mode, and in the third mode it is two times higher than in the second one.
Figure 8. Dependences of completeness coefficient on the mode number, for \( N=2, 4, 6, 8 \) and 10.

Figure 8 presents the dependences of the completeness coefficient on the mode number for different thermocycles. The course of curves show that at the initial thermocycling stage at \( N=2 \) the completeness coefficient is minimal in the first, intermediate in the second, and maximal in the third thermocycling mode. It is associated mostly with the fact that in the first thermocycles the SME has not been yet formed, and this is especially noticeable in the first mode where the SME is being formed mainly due to the isothermal additional loading in the martensitic state. As thermocycles repeat, the SME continues to form and \( k_c \) increases with the number of cycles, this matching the ascending family of dependences of thermocycle completeness on the mode number, figure 8. Failure of the value \( k_c \) for the second mode is conditioned by the fact that the thermocyclic creep [6] implemented in the process of thermal changes more suppresses the SME in those thermocycles, where there is a stress both at the heating stage and at the cooling stage. This determines the course of extreme curves in figure 8, at \( N=2, 6, 8, 10 \).

4. Conclusions
Summarizing the above stated, we may conclude the following: the impact of thermocycling type on the nature of changes of reversible deformations in titanium nickelide conditioned by the SME and DTP, as well as on the completeness coefficient of the thermocycle – \( k_c \) was studied experimentally in the thermocycling process.

It has been found that, regardless of the mode of deformation thermocyclic impact, the conditioned SME as well as the thermocycle completeness coefficient increase monotonously during the thermocycling, the deformations conditioned by the DTP decreasing monotonously.

It has been shown that the deforming effects of the SME and DTP are maximal in the third thermocycling mode, while the minimal deformation associated with the SME is implemented in the first thermocycle.

From the above stated it follows that, for functional elements of constructions working under minor, close to zero, stresses the use of the third thermocycling mode is the most effective.

For forceful functional elements under major acting stresses, the first thermocycling mode is the most favorable, as in this case the high completeness of the cycle will be observed with the sufficiently high values of SME deformations.

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