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The Effect of High-Temperature and High-Stress Martensite Aging on Martensitic Transformation and Microstructure of Ti–51.5 at % Ni Single Crystals

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Abstract. In this work, the thermomechanical stability of heterophase [001]-oriented Ti–51.5 at % Ni single crystals was investigated. Experiments include a high-temperature and high-stress martensite ageing and also loading/unloading cycling at superelasticity (SE) during forward and reverse B2-B19′ martensitic transformations (MT). It was found that Ti–51.5 at % Ni single crystals containing large Ti_3Ni_4 particles ($d \sim 600$ nm after ageing at 850 K for 1 h) is not resistant to stress-assisted martensite ageing and cycling. Stress-assisted martensite ageing at 423 K, 1.7–2 GPa, for 2–4 h leads to the deformation of large Ti_3Ni_4 particles, the appearance of dislocations at the particle-matrix boundaries, an increase in MT temperatures (by 10 K), and a strong diffusion of the MT (the intervals of forward and reverse MT Δ_1^σ and Δ_2^σ increase in 2 times). During loading/unloading cycling at 423 K and 1.0–2.0 GPa single crystals with large particles were destroyed after 20–25 cycles. It is possible to increase the thermomechanical stability due to the additional precipitation of nanosized Ti_3Ni_4 particles ($d < 30$ nm by ageing at 673 K, 1 h), which appear in the B2 matrix between large particles. In such crystals with a bimodal structure no changes in the microstructure and functional properties were observed after stress-assisted martensite ageing and loading/unloading cycling.

INTRODUCTION

It was shown [1–3], that a combination of a high nickel content ($C_{Ni} > 51.0$ at %), a high-strength orientation [001], and a crystal microstructure including dispersed Ti_3Ni_4 particles is necessary to obtain a high-strength TiNi alloys with high-temperature SE. Precipitation of large Ti_3Ni_4 particles (up to 600 nm) during ageing at 823 K for 1 h provides high strength properties of the B2-phase up to 2 GPa and a wide SE temperature range (from 200 to 450 K) with reversible strain up to 2.3% in [001]-oriented Ti–51.5 at % Ni single crystals [3]. In addition, in the same work [3], a two-stage ageing of 823 K, 1 h + 673 K, 1 h was carried out, that leads to the formation of a bimodal microstructure, where small Ti_3Ni_4 particles with $d < 30$ nm are contained between large particles. Such heat treatment makes it possible to increase the strength properties of the B2-phase, to enlarge the MT temperature, and, accordingly, shift the SE temperature range towards high temperatures.

For the practical application of these TiNi alloys under extreme conditions, it is necessary to find out which microstructure (only large particles or bimodal) is more suitable for use at high temperatures and stresses (above 100°C and 1 GPa). The single crystals with only large particles demonstrate a high-temperature SE with large stress hysteresis, which is 2 times wider than one in single crystals with a bimodal structure. Therefore, it is assumed that a bimodal structure is more resistant to high-temperature tests. To clarify this, it is planned to investigate the effect of high-temperature and high-stress martensite ageing and loading/unloading cycling on the microstructure and stress-induced MT in [001]-oriented Ti–51.5 at % Ni single crystals containing only large Ti_3Ni_4 and bimodal particles distribution (large + small).

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MATERIALS AND METHODS

Single crystals of Ti–51.5 at % Ni alloy were grown by the Bridgman method. Samples for compression deformation had a parallelepiped shape with sizes of $3 \times 3 \times 6$ mm^3. The as-grown crystals were preliminarily annealed at 1253 K for 1 h, followed by water quenching. First all annealed samples were aged at 850 K, 1 h (one-step ageing). Then aged samples were divided into two parts: one part was investigated after one-step ageing, the second part was exposed by subsequent ageing at 673 K, 1 h and mark as two-step aged crystals. The water quenching at each step of ageing was carried out.

The martensite ageing and loading/unloading cycling were conducted at 423 K, when a high-temperature SE is observed [3]. First, the stress-assisted martensite ageing was carried out under conditions when the martensite volume fraction is closed to 100% at a stress level of 1.7–2.0 GPa. Secondly, the loading/unloading cycling was performed during forward and reverse B2-B19′ MT at 1.0–2.0 GPa with a strain rate of 10^{-3} s^{-1}. Mechanical tests were executed on an Instron 5969 testing machine, IMRS-1 dilatometer and UTS 111.2-50-22 high-temperature universal testing machine. The untested aged samples were exposed to the stress-free thermal cycling (10 cycles) in the temperature range of 77–373 K in order to avoid the influence of degradation on the experimental results. Then, control $\varepsilon(T)$ curves were obtained for verifying the absence of degradation (change M_s^σ and thermal hysteresis ΔT_σ). After this all thermomechanical tests and microscopic research were conducted:

– the investigation of B2-B19′ MT during cooling/heating cycles under stress level of 400 MPa was conducted;

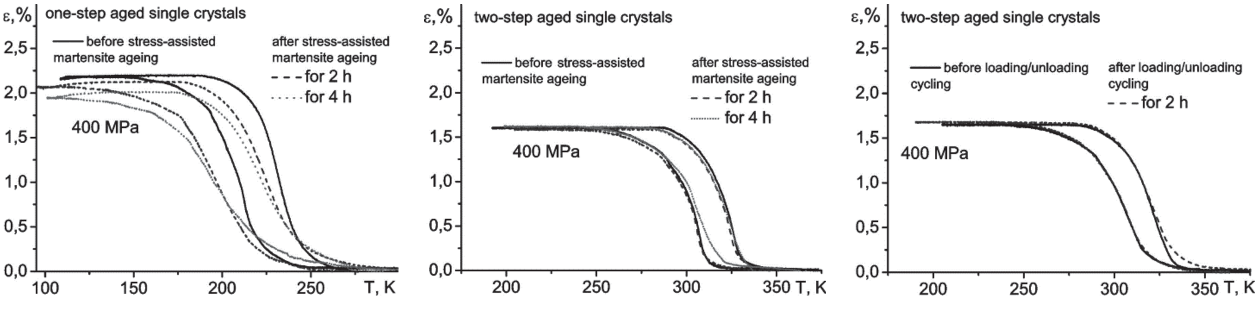
– an electron microscope study of the single crystal microstructure was carried out after martensite ageing and loading/unloading cycling using a Philips CM-12 transmission microscope in the Scientific Center of Tomsk State University and HT-7700, Hitachi in the Krasnoyarsk Regional Center of Research Equipment of Federal Research Center “Krasnoyarsk Science Center SB RAS”.

During the analysis of experimental data, the measurement errors of the strain ±0.3%, temperature ±2 K, and stresses ±2 MPa were taken into account.

RESULTS AND DISCUSSION

The microstructure of the studied single crystals is presented in detail in Ref. [3]. After one-step ageing, Ti–51.5 at % Ni single crystals contain the large Ti_3Ni_4 particles with a diameter of $d \sim 600$ nm, an interparticle distance of 0.05–0.6 μm and a volume fraction of about 20%. Small nanosized particles with $d < 30$ nm [3] appeared between the large particles in a subsequent low-temperature ageing.

Figure 1 shows the $\varepsilon(T)$ curves during shape memory effect (SME) for one-step and two-step aged single crystals before and after martensite ageing and loading/unloading cycling. It was experimentally established that one-step aged single crystals are not resistant to stress-induced martensite ageing (Fig. 1a). An increase in the MT temperature M_s^σ by 10 K and a strong diffusion of the MT (the intervals of forward and reverse MT Δ_1^σ and Δ_2^σ increase in 2 times) are observed after martensite ageing. The loading/unloading cycling led to the destruction of one-step aged single crystals after 20–25 cycles.



(a) (b) (c)

FIGURE 1. The $\varepsilon(T)$ curves for [001]-oriented Ti–51.5at%Ni single crystals: one-step aged single crystals before and after stress-assisted martensite ageing at 423 K, 2 h, 2.0 GPa (a); two-step aged single crystals before and after stress-assisted martensite ageing at 423 K, 2 h, 1.8 GPa (b) and after loading/unloading cycling at 423 K, 2 h (c).

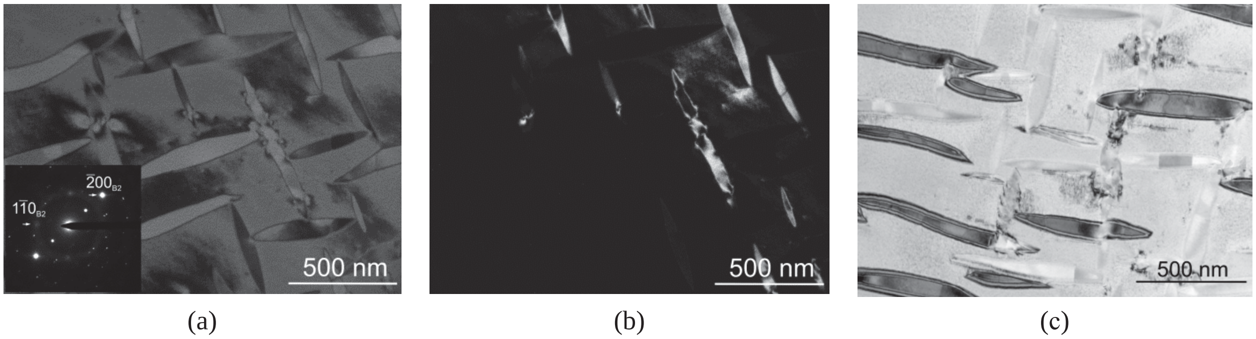


FIGURE 2. Microstructure of one-step aged Ti–51.5 at % Ni single crystals after stress-assisted martensite ageing at 423 K, 4 h, 2.0 GPa: a bright-field image and selected area diffraction pattern, the zone axis is near $[001]_{B2}$ (a, c); a dark-field image in the reflection $[\bar{2}00]_{B2}$, demonstrates large Ti_3Ni_4 particles (b).

The reason for this is the microstructure degradation of one-step aged single crystals (Fig. 2). During high-temperature and high-stress martensite ageing the relaxation of stresses near large Ti_3Ni_4 particles occurred, the dislocations form near the particles, and the steps and kinks at the particle-matrix boundaries appear (Fig. 2). After unloading the internal stress fields arise from deformed particles and contribute to the nucleation of non-oriented martensite, inducing an increase in the MT temperatures and diffusion of the MT upon cooling/heating under stress.

It is important to note that only two of the four particle variants are most susceptible to deformation. Only two variants are visible in the bright field images with the $[001]_{B2}$ zone axis (Fig. 2). Large Ti_3Ni_4 particles have $\{111\}_{B2}$ habit planes [4]. Therefore, all particles are equivalently located with respect to the loading axis of $[001]_{B2}$, however, it is nonequivalent with respect to twins in B19′-martensite. So it can be the reason for the prevalent deformation of only two particle variants. The most common twinning type is II ⟨011⟩ twinning, namely $(0.720531\,\bar{1})\,[011]$ or $(\overline{0.7205}311)\,[01\,\bar{1}]$ [5]. This means that during stress-induced MT two types of particles with habit planes parallel to twin planes (these are $(11\bar{1})$ or $(\bar{1}11)$) can undergo less impact than particles with habit planes locating at an angle of 73.3° to twin planes. The described above structural degradation during martensite ageing in one-step aged single crystals are obviously the cause of the wide stress hysteresis that is observed during high-temperature SE at 423 K and 1.5–2.0 GPa in Ref. [3].

Two-step aged single crystals with a bimodal particle distribution are more resistant to thermomechanical ageing (Figs. 1b, 1c). Two-step aged single crystals endure loading/unloading cycling at 423 K for 2 hours (40 cycles), unlike one-step aged single crystals. After cycling as well as after stress-assisted martensite aging, the temperature M_s^{σ} and thermal hysteresis ΔT_{σ} do not change. The $\varepsilon(T)$ curves before and after ageing almost coincide. No microstructure changes were detected by an electron microscope, and no dislocations were observed around large Ti_3Ni_4 particles (Fig. 3).

The reason for such good stability is the presence of nanosized Ti_3Ni_4 particles, which appeared after ageing at 673 K. First, small particles lead to a change in the twinning type.

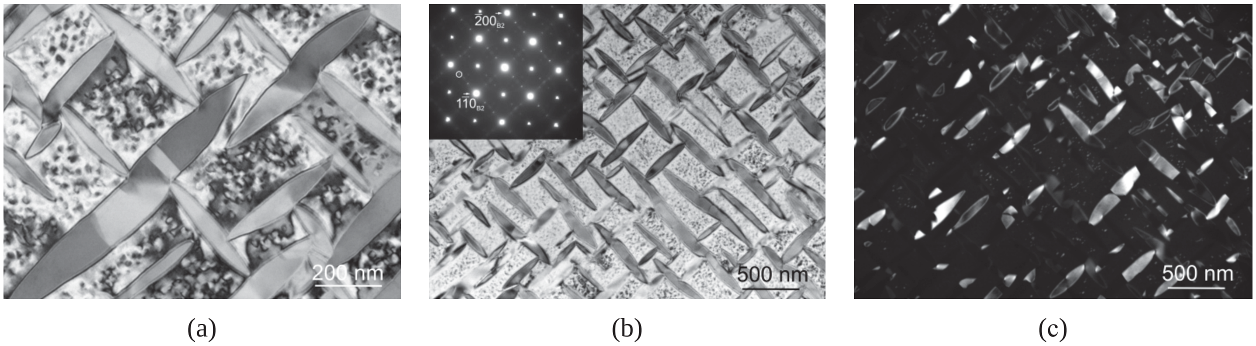


FIGURE 3. Microstructure of two-step aged Ti–51.5 at % Ni single crystals after stress-assisted martensite ageing at 423 K, 4 h, 1.8 GPa: bright-field images and selected area diffraction pattern, zone axis $[001]_{B2}$ (a, b); a dark-field image in a circled reflection, showing large Ti_3Ni_4 particles (c).

Based on by the magnitude of interparticle distances of 0.2–0.7 μm it should be noted that in Ti–51.5 at % Ni single crystals with large particles the both II type of twinning and [001](100) compound twinning can be observed under load [6–9]. The precipitation of small particles leads to the change of twinning type to only compound twinning and a significant, up to 10-fold increase in the density of these twins [9]. Due to this, the habit plane of the twinned B19′-martensite has greater compatibility with a large particle, in contrast to the case of wider II type twins. Second, the particles significantly strengthen the matrix and prevent a deformation of the particles during stress-assisted ageing owing to particles appeared on the particle-matrix boundary. As a result, a structure with a bimodal particle distribution is the most resistant to testing under conditions of high stresses and temperatures. Therefore, in these crystals, the stress hysteresis at high temperature SE at 423 K and 1.2–1.6 GPa is 2 times less than in single crystals with only large particles [3].

SUMMARY

It was experimentally established that [001]-oriented Ti–51.5 at % Ni single crystals containing large Ti_3Ni_4 particles ($d \sim 600$ nm after ageing at 850 K, 1 h) are not resistant to loading/unloading cycling and stress-assisted martensite ageing at high stress levels (up to 2 GPa) and temperature (423 K) for 2–4 hours. Stress-assisted martensite ageing at 423 K, 1.7–2 GPa, for 2–4 hours leads to the deformation of large Ti_3Ni_4 particles, the appearance of dislocations at the particle-matrix boundaries, which contributes to the formation of B19′-martensite, an increase in the MT temperatures (by 10 K) and a strong diffusion of the MT (intervals of the forward Δ_1^σ and reverse MT Δ_2^σ increased in 2 times). Single crystals with large particles were destroyed after 20–25 loading/unloading cycles at 423 K and 1.4–2.0 GPa. It is possible to increase the thermomechanical stability of single crystals due to an additional ageing at 673 K for 1 h, which leads to the precipitation of Ti_3Ni_4 nanoscale particles ($d < 30$ nm) between large particles. No changes in the microstructure and functional properties are observed in Ti–51.5 at % Ni single crystals with a bimodal particle distribution, after similar loading/unloading cycling and stress-assisted martensite ageing.

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