

A. Sibirev et al.: Relationship between the variation in transformation temperatures, resistivity and dislocation density

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Relationship between the variation in transformation temperatures, resistivity and dislocation density during thermal cycling of Ni₅₀Ti₅₀ shape memory alloy

The aim of the present work is to find the relationship between a decrease in transformation temperature and an increase in dislocation density during thermal cycling of NiTi alloy. The resistivity is used as a measure of the dislocation density variation and it allows one to find a linear dependence of the increment in the transformation temperatures on the dislocation density variation starting with the fifth thermal cycle. It is found that the thermal cycling of NiTi alloy within a temperature range of 473 to 273 K is accompanied by less dislocation density variation than in a range of 413 to 273 K. The dependence of transformation temperatures on increase in dislocation density in the first five cycles is not linear, hence it is concluded that the dislocation density variation is not the unique reason for a decrease in transformation temperature during the thermal cycling of equiatomic NiTi alloy.

Keywords: Shape memory alloys; Martensitic transformations; Thermal cycling; Dislocation density; Resistivity.

1. Introduction

Shape memory alloys (SMAs) are often used as working elements of actuators due to their unique capacity for strain recovery and stress generation, Refs. [1, 2]. In actuators, the

SMA elements are subjected to thermal cycling through the temperature range of the martensitic transformations. Thus, the stability of SMA properties on repeated cooling and heating is an important characteristic for applications Refs. [3–6]. The thermal stability of SMA properties depends on the chemical composition Refs. [7–9], yield limit Refs. [6, 10] the value of applied stress Refs. [2, 11, 12] and grain size, Refs. [10, 13].

Thermal cycling of equiatomic NiTi alloys in the stress-free state results in a variation in the martensitic transformation temperatures and a change in the transformation sequence Refs. [14–18]. It is believed that these phenomena are caused by an increase in defect density that occurs on cooling during the forward martensitic transformation due to the transformation from the austenite cubic B2 phase to the martensite monoclinic B19' phase which is accompanied by a maximum 11 % transformation strain that creates high internal stresses, Ref. [19]. To accommodate this stress, dislocation nucleation and slip occur and it increases the dislocation density, Refs. [14, 16, 20]. An increase in the dislocation density leads to an increase in the elastic energy that decreases the transformation temperatures, Ref. [21]. The dislocation density may increase during the mechanical cycling where the pseudoelasticity occurs and it influences the parameters of the transformation, Ref. [22].

Equiatomic TiNi alloy is in martensite phase at room temperature and B19' martensite has many X-ray reflec-

tions in a narrow angle interval, which leads to peak overlapping. This makes estimation of peak broadening very challenging, Ref. [23]. The observation of the dislocations in the martensite phase by transmission electron microscopy is prevented by the complex martensite contrast, Ref. [24]. This is why Pelton et al. [16] estimated dislocation density variation during thermal cycling for a Ti-50.5 Ni alloy, which was in the austenite phase at room temperature. It was shown that the defect density increased from 10^{12} to 10^{13} m^{-2} during the first thermal cycle and it varied to $5 \cdot 10^{14} \text{ m}^{-2}$ during one hundred thermal cycles.

To estimate the dislocation density variation during thermal cycling of the equiatomic NiTi alloy, the measurement of the resistivity variation may be used. According to Matthiessen's rule, the resistivity of the alloy is equal to the sum of the resistivity induced by different sources, including phonons, dislocations, point defects, interfaces and so on, Ref. [25]. There are no data regarding the variation in the concentration of point defects and interfaces during thermal cycling, hence the variation in the resistivity that is observed on thermal cycling may be attributed to an increase in dislocation density, Refs. [14, 16]. The resistivity induced by dislocations may be estimated as $\rho_d = \rho^* \cdot \Delta D$, where ΔD is the dislocation density variation and ρ^* is a constant, Ref. [26]. Therefore, the variation in resistivity that is measured at a fixed temperature in each cycle may be considered as a measure of the dislocation density and it may be used to find the relationship between a decrease in transformation temperatures and an increase in dislocation density during thermal cycling of equiatomic NiTi alloy, which is the aim of the present work.

2. Experimental procedure

The $\text{Ni}_{50}\text{Ti}_{50}$ alloy wire samples with a diameter of 0.5 mm and a length of 140 mm produced by MATEK-SMA Ltd. (Moscow, Russia), wires were subjected to homogenising water quenching from 973 K (15 min) and to remove residual quenching stresses wires were annealed at a temperature of 773 K for 1 h, an annealing of the equiatomic TiNi does not lead to the precipitation of secondary phases particles, thus it does not result in property variation. After annealing, the alloy underwent the martensitic transformation

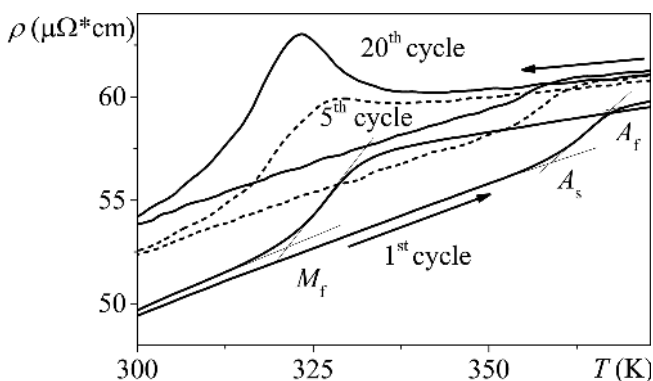


Fig. 1. Dependences of resistivity on temperature obtained in 1st, 5th and 20th cycle during thermal cycling within temperature ranges of 473 to 273 K.

from the cubic B2 phase to the monoclinic B19' phase on cooling and the B19' → B2 reverse transformation on heating.

The samples were subjected to 20 thermal cycles in a temperature range of 413 to 273 K with cooling/heating rate of 7 K min^{-1} in the Shimadzu thermal chamber TCLN-220P. The thermal chamber allowed precise control of cooling/heating rate. During thermal cycling, the resistivity of the samples was measured on cooling and heating by the four-point method and sample's temperature was measured by a thermocouple.

Wang et al. [18] have shown that a thermal cycling in different temperature ranges of 423 to 123 K and 523 – 123 K was accompanied by different transformation temperature variations due to heating to a higher upper temperature leading to dislocation annihilation. In other words, it was found that thermal cycling in different temperature ranges resulted in different dislocation density variations. Taking into account the results that were found by Wang et al. [18], in the present study to investigate influence of maximum thermal cycle temperature on increase of dislocation density and variation in alloy properties on thermal cycling, the annealed samples were additionally subjected to thermal cycling in a temperature range of 473 to 273 K and the procedure described above was repeated.

3. Results

Figure 1 shows dependences of resistivity on temperature obtained in 1st, 5th and 20th thermal cycle in temperature range of 473 to 273 K. It is seen that on resistivity curves there were anomalies related to martensitic transformations. With increase of thermal cycle number these anomalies shifted to lower temperature and the $\rho(T)$ curve rose to higher resistivity values, most of the changes occurred in the first five thermal cycles. In the first thermal cycle the alloy underwent B2 ↔ B19' transformations, but after the 5th thermal cycle the transformation sequence on cooling changed from B2 → B19' to B2 → R → B19'. Using the $\rho(T)$ curves that were obtained during thermal cycling, the transformation temperatures were measured as an intersection of the tangent lines, as shown in Ref. [14]. However it is certain that R-phase formed on cooling only in part of the alloy volume, and the other part underwent B2 → B19' transformation. Using the resistivity measurement method one cannot divide the contribution to overall resistivity of two different transformations which occurred simultaneously in the sample volume, thus the start temperature of B2 → R and B2 → B19' transformations were excluded from further analysis due to inevitably large error in their determination. The variation in transformation temperatures is caused by an increase in defect density, Refs. [14, 21]. To estimate defect density, increase in thermal cycling resistivity was measured at 393 K in each cycle when samples were in the fully austenite state, Ref. [27].

The dependences of M_f , A_s and A_f temperatures on the thermal cycle number are presented in Fig. 2. It is seen that despite the upper thermal cycling temperature, transformation temperatures decrease (Fig. 2a and b) and the resistivity increases (Fig. 2c) during thermal cycling. The comparison of the data obtained during thermal cycling in different temperature ranges shows that the variations in transformation temperatures and resistivity during thermal cycling within a

temperature range of 473 to 273 K are less than in the range of 413 to 273 K and it is in a good agreement with results found in Ref. [18].

The data presented in Fig. 2 were used to obtain the dependences of the transformation temperatures on the resistivity given in Fig. 3. It is seen that the dependences of the transformation temperatures on resistivity increase are not

linear, transformation temperatures decrease rate per resistivity increment decreases with increase in resistivity.

To find the dependence of the transformation temperatures on the dislocation density, the ratio between the resistivity and dislocation density should be found. The variation in resistivity induced by dislocations may be found using:

$$\rho_d = \rho_N - \rho_0 \tag{1}$$

where ρ_N is the resistivity measured in the N^{th} thermal cycle and ρ_0 is the resistivity, measured in annealed sample before cycling Ref. [25]. At the same time, $\rho_d = \rho^* \cdot \Delta D$ Ref. [26] hence, to estimate the defect density it is necessary to find the ρ^* coefficient as the ratio of the ρ_d to the variation in the dislocation density D . Thus, the ρ^* may be determined as:

$$\rho^* = \frac{\rho_1 - \rho_0}{D_1 - D_0} \tag{2}$$

where ρ_0 and D_0 are the resistivity and the dislocation density measured in the annealed sample and ρ_1 and D_1 are the resistivity and the dislocation density measured after the first thermal cycle, respectively. To show how this analysis may be used for the estimation of the linear coefficients between the transformation temperatures and dislocation density the data of Pelton et al. [16] were used. According to Ref. [16], the dislocation density of the annealed NiTi alloy is equal to $D_0 = 10^8 \text{ cm}^{-2}$, and it increases up to

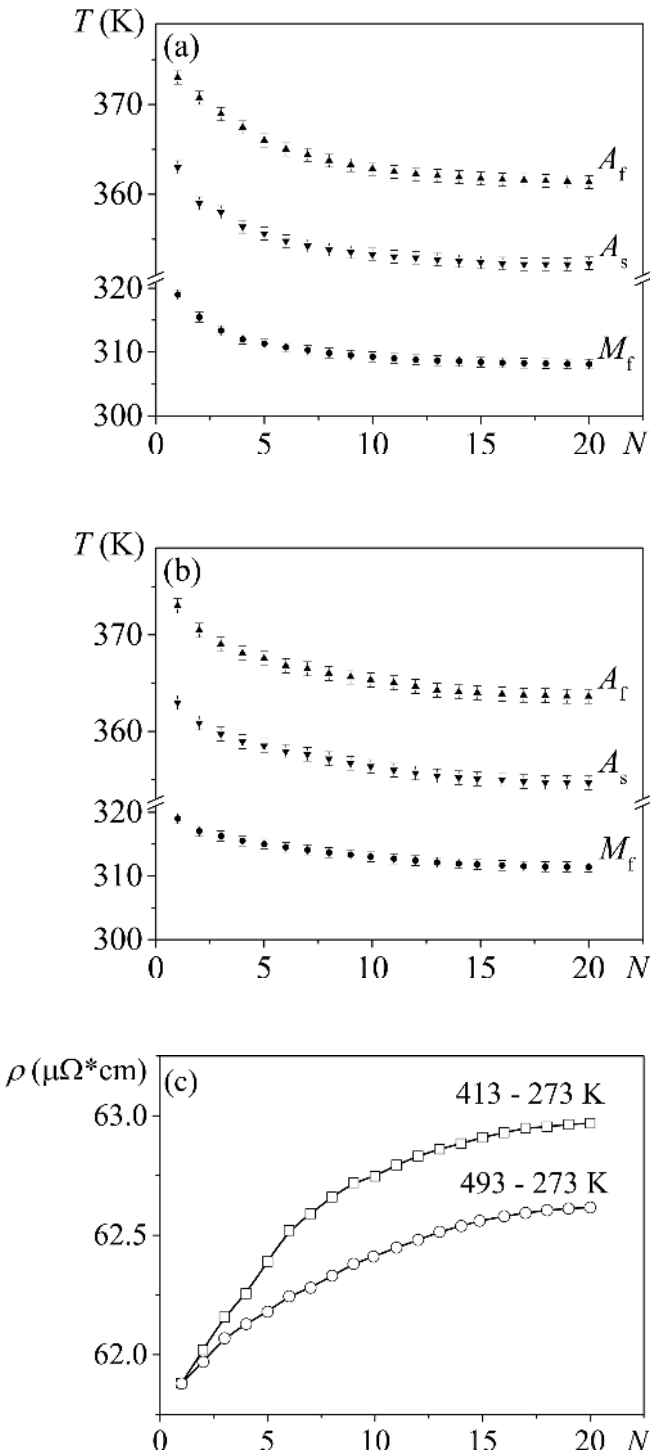


Fig. 2. Variation in (a, b) transformation temperatures, and (c) resistivity during thermal cycling within temperature ranges of (a) 413 to 273 K and (b) 473 to 273 K.

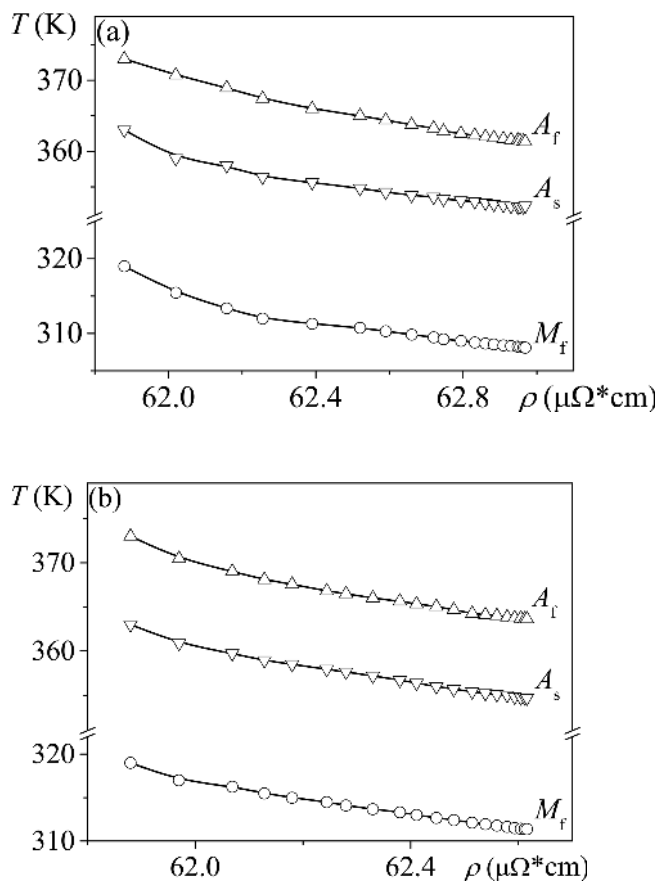


Fig. 3. Dependences of the transformation temperatures on the resistivity that was observed during thermal cycling within temperature ranges of (a) 413 to 273 K and (b) 473 to 273 K.

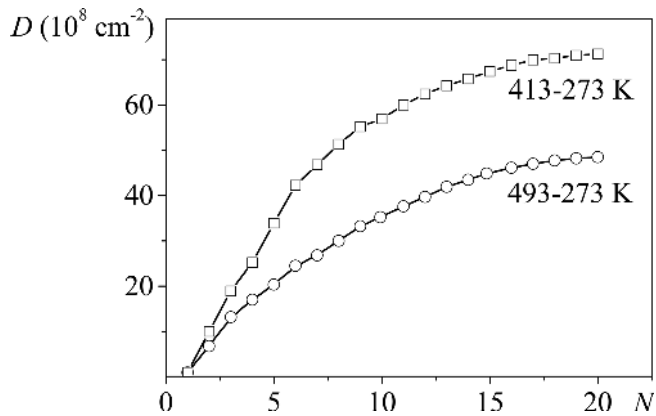


Fig. 4. Dependences of the dislocation density estimated using Eq. (3) on the thermal cycle number.

$D_1 = 10^9 \text{ cm}^{-2}$ during the first cycle. Hence, the $D_1 - D_0$ value is equal to $9 \cdot 10^8 \text{ cm}^{-2}$. Figure 2c shows that $\rho_1 - \rho_0$ is equal to $0.14 \mu\Omega \cdot \text{cm}$. Thus, the value of ρ^* is equal to $1.55 \cdot 10^{-6} \mu\Omega \cdot \text{cm}^3$. In this case, the dislocation density in each thermal cycle may be estimated as:

$$D = D_0 + \Delta D = D_0 + \frac{\rho_d}{\rho^*} = D_0 + \frac{\rho_N - \rho_0}{1.55 \cdot 10^{-6}} \quad (3)$$

The data presented in Fig. 2c were used to estimate the dislocation density in each cycle and Fig. 4 shows the variation in defect density during thermal cycling within the temperature ranges of 413 to 273 K and 473 to 273 K. It is seen that an increase in defect density during thermal cycling in the temperature range of 473 to 273 K is less than in the range of 413 to 273 K and it confirms the assumption given by Wang et al. [18] that an increase in the upper thermal cycle temperature leads to a decrease in dislocation density variation due to a recovery process.

Using the data presented in Fig. 3 and the dislocation density variation given in Fig. 4, the dependences of the increment in transformation temperatures (ΔA_s , ΔA_f , ΔM_f) on the dislocation density were obtained and are shown in Fig. 5. The T_R temperature was excluded from the analysis due to a low accuracy of this temperature determination, especially in the first thermal cycles when the peak on the $\rho(T)$ curve was hardly visible. Figure 5 shows that if the thermal cycling is carried out within a temperature range of 413 to 273 K (Fig. 5a), then the linear segment may be found on the $\Delta M_f(D)$, $\Delta A_s(D)$ and $\Delta A_f(D)$ curves when the dislocation density is larger than $34 \cdot 10^8 \text{ cm}^{-2}$ ($N = 5$). The linear coefficient A^* is equal to $-0.09 \pm 0.002 \cdot 10^{-8} \text{ K} \cdot \text{cm}^2$ for the $\Delta M_f(D)$ and $\Delta A_s(D)$ curves and A^* is equal to $-0.125 \pm 0.002 \cdot 10^{-8} \text{ K} \cdot \text{cm}^2$ for the $\Delta A_f(D)$ curve. If the thermal cycling is carried out within a temperature range of 473 to 273 K (Fig. 5b), then the linear segment may be observed on the $\Delta M_f(D)$, $\Delta A_s(D)$ and $\Delta A_f(D)$ curves when the dislocation density is larger than $20 \cdot 10^8 \text{ cm}^{-2}$ ($N = 5$). The linear coefficient A^* is equal to $-0.130 \pm 0.002 \cdot 10^{-8} \text{ K} \cdot \text{cm}^2$ for the $\Delta M_f(D)$ curve, $-0.140 \pm 0.002 \cdot 10^{-8} \text{ K} \cdot \text{cm}^2$ for the $\Delta A_s(D)$ curve and $-0.143 \pm 0.002 \cdot 10^{-8} \text{ K} \cdot \text{cm}^2$ for the $\Delta A_f(D)$ curve. Thus, the results of the study show that the dependence of the transformation temperatures on the dislocation density is non-linear during the first

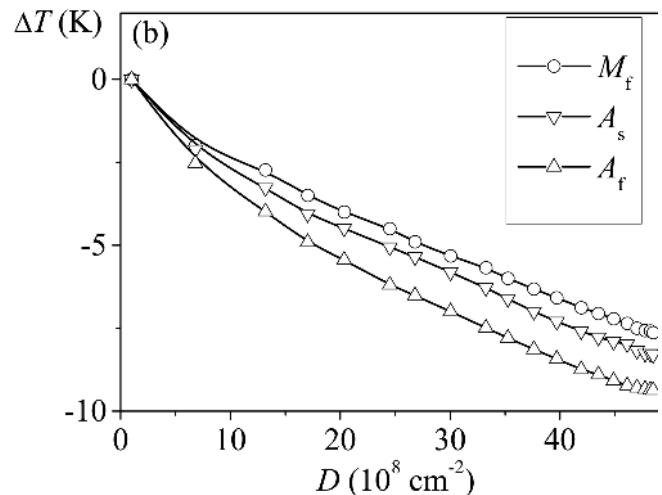
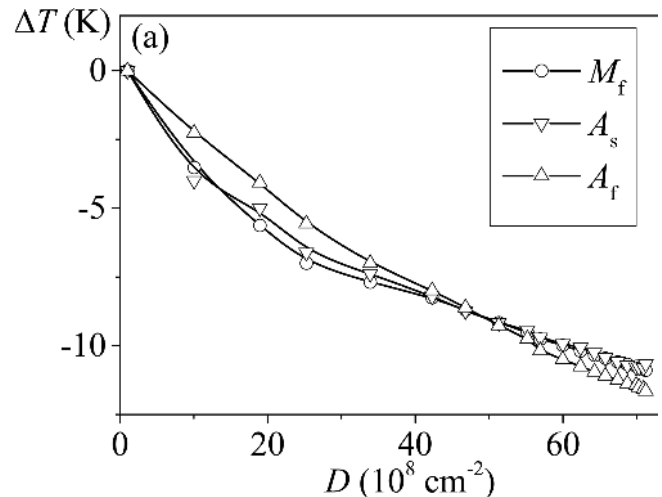


Fig. 5. Dependences of the transformation temperatures increment on the dislocation density estimated according to Eq. (3) obtained during thermal cycling within temperature ranges of (a) 413 to 273 K and (b) 473 to 273 K.

five cycles and it becomes linear on further thermal cycling. The data presented in Fig. 4 show that the linear coefficients are different for various transformation temperatures. The smallest coefficient is found for the $\Delta M_f(D)$ curve and the largest for the $\Delta A_f(D)$ curve. Moreover, it is found that the linear coefficients measured for the transformation temperatures during thermal cycling in a temperature range of 473 to 273 K are larger than in the range of 413 to 273 K. Hence an increase in the dislocation density decreases the transformation temperatures in different ways depending on the upper thermal cycle temperature.

4. Discussion

The relationship between the dislocation density and the martensitic transformation temperatures may be found using the thermodynamic equilibrium equation. In the first cycle, the forward martensitic transformation on cooling occurs when $\Delta G_{\text{ch}} = E_d + E_{\text{cl}}^{A \rightarrow M}$, where ΔG_{ch} is the difference in chemical free Gibbs energy of the austenite and

martensite phases, E_d is the dissipative energy that determines the hysteresis of the transformation and $E_{el}^{A \rightarrow M}$ is the elastic energy associated with the appearance of the martensite phase that determines the temperature range of the martensitic transformation (the sum $E_d + E_{el}^{A \rightarrow M}$ is called non-chemical Gibbs energy), Refs. [25, 27]. According to McCormick and Liu [21], an increase in dislocation density during the thermal cycle leads to an additional contribution to the elastic energy, which should be included to the thermodynamic equilibrium equation. Thus, in the second cycle the forward martensitic transformation on cooling takes place when $\Delta G_{ch} = E_d + E_{el}^{A \rightarrow M} + E_{el}^d$, where E_{el}^d is the elastic energy of dislocations, Refs. [21, 28, 29]. The comparison of the thermodynamic equilibrium equation formulated for the first and the second cycles shows that if an increase in dislocation density gives the contribution to the thermodynamic balance, then the ΔG_{ch} that is measured in the second cycle should be larger than the value of ΔG_{ch} measured in the first cycle. The larger value of ΔG_{ch} may be achieved by additional undercooling of the sample, thus the martensitic transformation occurs in the second cycle at lower temperatures than in the first cycle. McCormick and Liu [21] shown that a variation in transformation temperatures induced by an increase in elastic energy of the dislocations is equal to:

$$\Delta T = - \frac{|E_{el}^d|}{|\Delta S|} \quad (4)$$

where ΔS is the transformation entropy change and it is characteristic of the transformation. Therefore, a decrease in transformation temperatures is proportional to an increase in the elastic energy of dislocations.

According to Ref. [30], the energy of an individual dislocation may be found as the sum of the elastic strain energy and the energy of the dislocation core. In Ref. [30], it is shown that the dislocation core energy did not exceed the 10% of the total energy, hence this term might be ignored. Thus, the elastic energy of the dislocation may be estimated as, Ref. [30]:

$$E_{el}^d = \frac{G \cdot b^2 \cdot L}{4\pi \cdot K} \cdot \ln \frac{R}{r_0} \quad (5)$$

where G is the shear modulus, L is the dislocation length, b is the Burgers vector, K is a constant that is equal to 1 for edge dislocations and $(1-\nu)$ for screw dislocations (ν is the Poisson's ratio), r_0 is the dislocation core radius and R is the cut-off radius. To estimate the elastic energy induced by dislocations in metals and alloys, the following qualitative expression is used:

$$E_{el}^d \approx \frac{G \cdot b^2 \cdot L}{2} \quad (6)$$

If the interaction between the dislocations is not taken into account, then the total energy induced by all dislocations may be estimated as, Ref. [31]:

$$E_{el}^d \approx \frac{G \cdot b^2 \cdot \Sigma L}{2} \quad (7)$$

where ΣL is the total length of the dislocations that may be found as $\Sigma L = D \cdot V$, where D is the dislocation density

and V is the volume. Thus, the expression for the elastic energy induced by dislocations may be rewritten as:

$$E_{el}^d \approx \frac{G \cdot b^2 \cdot D \cdot V}{2} \quad (8)$$

As the G , b , V are the constants, the elastic energy may be estimated as:

$$E_{el}^d \approx A \cdot D \quad (9)$$

where $A = \frac{G \cdot b^2 \cdot V}{2}$. Using Eq. (4), one may estimate the variation in the transformation temperatures in the following manner

$$\Delta T \approx - \frac{A \cdot D}{\Delta S}$$

and as the ΔS is the constant, then this expression may be rewritten as:

$$\Delta T \approx -A^* \cdot D, \quad (10)$$

where $A^* = \frac{G \cdot b^2 \cdot V}{2 \cdot \Delta S}$. This result is in a good agreement with the experimental results found in the present study and shown in Fig. 4.

According to the thermodynamics approach that is described in Refs. [21, 28, 29] and given above, an increase in dislocation density should lead to a decrease in all transformation temperatures in the same manner, however, this contrasts with the data presented in Fig. 5. The fact that the linear coefficients A^* are different for various temperatures may be caused by the shear modulus and Burgers vector of dislocations changing during the martensitic transformation Ref. [23]. Also, the difference in the A^* coefficient that was estimated for the M_f and A_s temperatures may be caused by the influence of the thermal cycling on the elastic modulus in the martensitic state Ref. [32]. Thus, to apply the analysis of the thermodynamic equilibrium equation given for the different transformation temperatures, it is necessary to take into account that the elastic energy of dislocations is different for the martensitic and austenitic states, and this value may depend on the volume fraction of the martensite phase.

A decrease in transformation temperatures should depend linearly on the dislocation density and it is observed experimentally starting from the fifth cycle. During the first cycles, a decrease in transformation temperatures depends on the dislocation density in a non-linear way and this may be due to another reason (or reasons) that influences the transformation temperatures aside from the dislocation density. It may be speculated that the dislocation structure (distribution) may additionally influence the transformation temperatures. For instance, in Refs. [15–17] it is shown that thermal cycling of an equiatomic TiNi alloy resulted in heterogeneous dislocation distribution that led to the observation of the multistage martensitic transformation. Probably, the dislocation structure may be responsible not only for the multistage martensitic transformation but for the transformation temperature shift. Therefore, the results of the study show that the dislocation density variation is not the unique reason for a decrease in transformation temperatures variation during thermal cycling of the equiatomic NiTi alloy.

5. Conclusions

1. A method for the estimation of the dislocation density variation during the thermal cycling of an NiTi alloy using the variation in the resistivity measured was proposed.

2. The thermal cycling within a temperature range of 473 to 273 K was accompanied by lower dislocation density variation than during a range of 413 to 273 K. However, the decrease in transformation temperatures per dislocation density unit (the value of A^* coefficient) was larger during thermal cycling in a range of 473 to 273 K than within a range of 413 to 273 K.

3. The linear dependences of the increment in the transformation temperatures on the dislocation density were observed starting with the fifth thermal cycle. In the first cycles, the transformation temperatures depend on the dislocation density in a non-linear way.

4. The linear coefficients A^* that were measured using the $\Delta M_f(D)$, $\Delta A_s(D)$ and $\Delta A_f(D)$ curves were different. It was assumed that this might be due to the shear modulus and Burger's vector of dislocation and as a result, the elastic energy of dislocation change during the martensitic transformations.

5. The dislocation density variation was not the unique reason for a decrease in transformation temperature variation during thermal cycling of the equiatomic NiTi alloy.

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