Effect of Post-Deformation Annealing on Structure and Properties of Nickel-Enriched Ti-Ni Shape Memory Alloy Deformed in Various Initially Deformation-Induced Structure States

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Abstract: The effect of post-deformation annealing (PDA) of nickel-enriched Ti-50.9 at.% Ni shape memory alloy deformed in various initially deformation-induced structure states on the structure and properties was studied. The phase composition, temperature ranges of martensitic transformations and structure were studied using X-ray diffractometry and TEM. Mechanical and functional properties were determined using Vickers hardness tests and thermomechanical method using a bending mode for recovery strain inducing. The PDA at 430 °C (1 h) of the nickel-enriched Ti-Ni SMA with the dynamically recovered, dynamically polygonized or dynamically recrystallized structures after compressing deformation leads to the precipitation of finely dispersed (nanosized) Ti3Ni4 particles. The most significant increase of the completely recoverable strain (from 8.8 to 11.8%) and shape recovery rate (from 88 to 100%) as compared to the reference treatment is observed after PDA of the alloy with the dynamically polygonized structure.

Keywords: shape memory alloys; Ti-Ni; thermomechanical treatment; aging

1. Introduction

Among the advanced functional materials, Ti-Ni-based shape memory alloys (SMA) are commonly characterized as a unique one owing to perform shape memory and superelasticity effects. The application of Ti-Ni SMA expands in various industrial fields, including medical engineering. Hyper-equiaxiomorphic nickel-enriched aging Ti-Ni SMA (containing from 50.6 at.% to 51.0 at.% Ni) are widely applied for the production of medical devices due to superelastic behavior at a human body temperature in combination with high operational characteristics [1–8].

The expansion of the application scope and the complexity of medical devices led to an increase in requirements to mechanical and functional properties of nickel-enriched Ti-Ni SMA. Therefore, the development of new approaches for the improvement of properties of nickel-enriched Ti-Ni SMA, including the optimization of rational production technologies, is of first priority [9–13]. Along with conventional manufacturing, new opportunities for obtaining finished products with a shape memory effect are opened up by additive technologies [4,14–18].

It is well known that properties of Ti-Ni SMA are structure-sensitive and the formation of well-developed dislocation substructure in ultrafine-grained (UFG), or especially, nanocrystalline (NC) size range of structural elements allow considerably improving operational characteristics of Ti-Ni SMA [19]. However, NC structure today is obtained only
in thin experimental samples, while attempts to produce bulk NC semi-finished products are almost unsuccessful [19–26]. Recently, the completely NC structure in the bulk sample was only obtained by the application of the MaxStrain deformation [12,27]. However, for the industrial application of nanocrystalline Ti-Ni-based SMA, the size of sample of about 1 cm³ is insufficient and must be increased. The development of industrial production of Ti-Ni bulk samples is hampered by insufficient technological plasticity in conditions of conventional metal forming processes in the temperature range the most favorable for the formation of the NC structure. Moreover, the deformation behavior of Ti-Ni alloys in the temperature range below 600 °C has been insufficiently studied. Thus, the previous study [28] was aimed at determining the features of the deformation behavior, structure and properties of the Ti-50.9 at.% Ni alloy subjected to uniaxial compression deformation over a wide temperature range, especially below 600 °C. Obtained results allowed defining a temperature border for the transition from low- to high-temperature type of deformation diagram, the deformation modes allowing to obtain the highest shape recovery properties (a total recovery strain \( \varepsilon_{rt} \) of 11%). and the temperature ranges for the development of dynamic softening processes in the Ti-50.9 at.% Ni SMA, as follows: dynamic recovery from 100 to 300 °C, dynamic polygonization from 300 to 600 °C and dynamic recrystallization above 600 °C.

The present article is a development of the reported study [28] focused on the assessment of the effect of post-deformation annealing (PDA) processes on the change of the structural state, resulting in the change of the mechanical and functional properties. The carrying out of PDA after any thermomechanical treatment (TMT) is defined by the necessity for this treatment inducing the required operational ("remembered") shape and temperature range of superelasticity effect (SE) or shape memory effect (SME) to a device before the practical application and by the need to analyze the stability of the structure and properties obtained after TMT. The temperature of PDA was chosen based on the previous experience, which showed that the most intensive aging processes in nickel-enriched Ti-Ni SMA were observed at 430 °C [29–33]. During aging, the depletion of the B2 matrix in nickel with the precipitation of Ti₃Ni₄ phase particles occurs. Aging processes lead to a hardening and a corresponding increase in the yield strength of the alloy. The knowledge about the effect of PDA of Ti-50.9 at.% Ni SMA after compression in a wide temperature range will help to determine the possibilities for precise control of the final structural state and functional properties and optimize the production technology of various medical devices.

2. Materials and Methods

Ti-50.9 at.% Ni alloy obtained by hot rotary forging at the temperature of 800 °C was used as the experimental material. Samples with the diameter of 5 mm and the height of 10 mm were cut by the electro discharge method from the initial rods. TiNi samples before the compression were heated at 700 °C for 30 min in an electrical furnace and quenched in water. This operation was chosen as a reference treatment (RT). Obtained samples were subjected to uniaxial compression deformation to true strain of \( \varepsilon = 0.5 \) in the temperature range from 100 °C to 900 °C and annealed after compression at 430 °C for 1h.

The critical martensitic transformation temperatures of studied alloy in the state after RT were determined by differential scanning calorimetry as follows: Starting and finishing temperatures of forward martensitic transformation \( \text{M}_f = -85 \) °C and \( \text{M}_t = -95 \) °C, starting and finishing temperatures of reverse martensitic transformation \( \text{A}_f = -65 \) °C and \( \text{A}_t = -45 \) °C.

The structural-phase state was studied using a DRON-3.0 X-ray diffractometer in Cu Kα radiation at room temperature in the 2θ ranges from 36° to 47° and a JEM-2100 TEM at an accelerating voltage of 200 kV. Bright field (BF) and dark field (DF) images and the electron diffraction patterns, with the identification of main phases (the high-temperature phase is a long-range-ordered cubic (CsCl-type) B2-austenite, the low-temperature phase is a monoclinic B19’-martensite, and the intermediate phase is a rhombohedral R-phase) were investigated. The samples for the structural analysis were cut using electro discharge machining along the compression direction. The procedure for preparation of thin foils
included the mechanical polishing of precut discs from 0.5–0.3 mm to 0.1 mm and then thinning via electrolytic polishing in an acid solution HCLO₄ + CH₃COOH.

Mechanical (hardness) and functional (maximum completely recoverable strain and shape recovery rate) properties were determined by the Vickers hardness tests at room temperature using LECOM 400-A hardness tester under a load of 1N with indenter exposure of 10 s and a thermomechanical method using a bending mode for strain inducing (the detailed description of the conventional thermomechanical method for evaluation of the shape recovery properties can be found in [34]), respectively. For the average hardness evaluation, no less than ten indentations were performed for each sample in the transverse and longitudinal directions. Samples for the thermomechanical method with dimensions of 0.5 mm × 0.6 mm × 10 mm were cut from the sample after compression using an electro discharge machine. The samples were etched and the width was reduced by 50 µm (to remove a possibly modified layer). The schematic representation of the location of the sample extracted for functional property determination is also presented in Figure 1. Such characteristics of shape memory effect, as total recovery strain $\epsilon_r$, summarize recovery strains due to superelasticity $\epsilon^{SE}_r$ (recovered right after unloading and, in fact, including an elastic component) and shape memory effect $\epsilon^{SM}_r$ (recovered after heating above the As temperature), and shape recovery rate (SRR) calculated as the ratio of the total recovery strain to the induced strain $\epsilon_i$. The recovery strain was induced by bending the sample in the martensitic state in liquid nitrogen at minus 196 °C. Shape recovery, commonly, occurred upon heating to room temperature. The lower critical temperature of the temperature range of shape recovery (TRSR) (As) was always below room temperature.

$$e = 0.5$$

![Figure 1](image_url)

**Figure 1.** Schematic representation of the location of the sample extracted for functional property determination.

### 3. Results and Discussion

#### 3.1. XRD Study

The results of the X-ray diffraction analysis of nickel-enriched Ti-Ni SMA after compression and PDA are presented in Figures 2 and 3.

A single X-ray line profile is visible in a position of [110]$_B$ in the initial state at room temperature after RT (Figure 2). However, the [110]$_B$ line width of 0.38 2θ° (Figure 2) is twice that inherent in the quenched recrystallized B2-austenite [23]. This points to the presence of R-phase formed as a result of a partial B2→R transformation. Indeed, a (330)$_R$ − (330)$_R$ doublet overlaps the [110]$_B$ from both sides and thus must lead to broadening of the general line profile. The (110)$_B$ line width abruptly increases as compared to RT. Then it gradually decreases after compression at 100 °C due to a decrease in the deformation temperature. An abrupt increase in the density of lattice defects (dislocations, sub-boundaries) as a result of processing development of the processes accompanied with a decrease in the dislocation density, that is, dynamic polygonization and dynamic recrystallization (Figure 3) [28].
Figure 2. X-ray line profiles in the vicinity of [110]_{B2} of Ti-50.9 at.% Ni SMA at room temperature after compression and PDA.

Figure 3. Dependence of [110] B2-austenite X-ray line width measured at room temperature in as-deformed state (solid line) and after PDA at 430 °C, 1 h (dotted line) on the deformation temperature.
PDA leads to a slight decrease in the \([110]_{B2}\) B2-austenite line width after compression at 400 °C and lower; however, the X-ray line profile views remain almost unchanged (Figure 2). The general pattern of the effect of PDA on the X-ray line width can be formulated as follows: annealing at a temperature exceeding the deformation temperature leads to a decrease in the lattice defects and a corresponding decrease in the X-ray line width; annealing at the temperature below the deformation temperature does not lead to a decrease in the lattice defects and consequently does not noticeably change the X-ray line width.

3.2. TEM Study

A more detailed electron microscopy study of the obtained structural-phase state was performed after compression at temperatures of 200 °C, 400 °C, and 600 °C, presumably corresponded to the development of dynamic recovery, polygonization, and recrystallization, respectively. The analysis of bright and dark field obtained via TEM is shown in Figures 4–6.

![Figure 4. Microstructure of nickel-enriched Ti-Ni SMA after compression at 200 °C (a–c) and after PDA at 430 °C for 1 h (d–f); transmission electron microscopy. Bright (a,d) and dark (b,e) field images and SAED patterns (c,f). Numbers 1 and 2 in SAED pattern (f) show reflexes in which the DF 1 and DF 2 images were obtained, respectively. Some reflexes characteristic of B2-, R- and Ti\(_3\)Ni\(_4\) phases are indexed in SAED patterns.](image-url)
Microstructure of nickel-enriched Ti-Ni SMA after compression at 400 °C (a–c) and after PDA at 430 °C for 1 h (d–f); transmission electron microscopy. Bright (a,c) and dark (b,e) field images, and SAED patterns (e,f). Numbers 1 and 2 in SAED pattern (c) show reflexes in which the DF 1 and DF 2 images were obtained, respectively. Some reflexes characteristic of B2-, R- and Ti₃Ni₄ phases are indexed in SAED patterns.

Microstructure of nickel-enriched Ti-Ni SMA after compression at 600 °C (a–c) and after PDA at 430 °C for 1 h (d–f); transmission electron microscopy. Bright (a,c) and dark (b,e) field images, and SAED patterns (e,f). Numbers 1 and 2 in SAED pattern (f) show reflexes in which the DF 1 and DF 2 images were obtained, respectively. Some reflexes characteristic of B2-, R- and Ti₃Ni₄ phases are indexed in SAED patterns.
Compression at 200 °C leads to the formation of a well-developed dislocation substructure with a very high dislocation density (Figure 4a,b). Characteristic B2-austenite and R-phase reflexes can be identified in the SAED microdiffraction pattern (Figure 4c). Evidence of the development of dynamic strain-aging processes that is the presence of the Ti₃Ni₄ phase is not found.

PDA at 430 °C for 1 h results in a noticeable change as compared to the structure right after compression (Figure 4d–f). Strong contrast in a form of finely dispersed (nanosized) dotty ripples is observed in the bright field image (Figure 4d). This contrast veils the dislocation substructure and shades the boundaries of deformation bands in the B2-austenite and crystals of the R-phase. Additional, mostly very weak and blurred, reflections appear in the electron diffraction pattern. Attempts to attribute them as reflections of the Ti₃Ni₄ phase were successful in some cases, as it is shown in the corresponding electron diffraction pattern (Figure 4f). The most reliable confirmation of the precipitation of the Ti₃Ni₄ phase after PDA was obtained using a comparative analysis of the dark field images taken from different reflexes of SAED pattern. Figure 4e shows two dark field images (DF 1 and DF 2) obtained from two different strong reflexes 1 and 2 of the diffraction pattern (Figure 4f) taken from the same area shown in Figure 4d. Each of these two reflexes is clearly formed by the superposition of [110]R, (330)R and/or (330)R reflexes, and presumably (112) reflex of the Ti₃Ni₄ phase, as shown in Figure 4f for the reflex 2. In the left dark field image DF 1 taken from the reflex 1, the right area of the frame is bright, while in the right dark field image DF 2 taken from the reflex 2, the left area is bright. In the left dark field image DF 1, the left area is dark, while in the right dark field image DF-2, the right area is dark. The bright areas in both DF 1 and DF 2 can be obviously attributed to B2- and/or R-phases with different orientations, and, presumably, to Ti₃Ni₄ phase. Besides, in each dark area of both dark field images, the dispersion of nanosized bright particles is observed. The appearance of this dispersion indicates the precipitation of particles of the Ti₃Ni₄ phase, which have a corresponding orientation within both reflexes 1 and 2.

After deformation at 400 °C, well-developed dynamically polygonized substructure of B2-austenite, with high dislocation density visible against the background of deformation bands and R-phase crystals, is present (Figure 5a,c). At the same time, analysis of dark field images and SAED patterns revealed a dispersion of nanoscale bright precipitates which represent particles of Ti₃Ni₄ phase. Therefore, dynamic deformation aging develops in an explicit form during compression at 400 °C, in contrast to the compression at 200 °C.

Analysis of the bright- and dark field images and SAED patterns shown in Figure 5b reveals the existence of B2-, R- and Ti₃Ni₄ phases after PDA (process of static aging), wherein particles of Ti₃Ni₄ phase are noticeably grown. It is difficult to estimate the dislocation substructure after this treatment because of the superposition of R-phase images and Ti₃Ni₄ particles precipitated during aging. For this purpose, the results of X-ray structural analysis are more reliable.

Deformation at 600 °C leads to the formation of recrystallized B2-phase grains with a relatively low dislocation density of about 10⁹ cm⁻² (Figure 6a). Strong reflexes of B2- and R-phases as well as weak reflexes of Ti₃Ni₄ phase can be identified in the SAED patterns (Figure 6c). Particles of Ti₃Ni₄ phase are distributed in the matrix or decorate dislocations (Figure 6a,b). PDA after compression at 600 °C leads to the precipitation of Ti₃Ni₄ particles with increased size as compared to the compression at lower temperatures, followed by the same PDA (Figure 6d–f). The mentioned increased particle size is a consequence of much lower dislocation density, which provides a much smaller number of nucleation sites for precipitating particles.

The obtained results of structural analysis reveal that PDA at 430 °C for 1 h of samples with dynamically recovered, dynamically polygonized and dynamically recrystallized structures leads to the precipitation of finely dispersed (nanosized) Ti₃Ni₄ particles, wherein the size of particles noticeably increases after deformation at 600 °C. PDA of samples with the dynamically polygonized structure leads to the additional precipitation of the Ti₃Ni₄ particles against the background of the increase in the size of particles, precipitated before
PDA during compression accompanied by dynamic aging. Thus, the process of static aging develops regardless of the initial structural state of the nickel-enriched Ti-Ni SMA, and after PDA all three main phases are present: B2-, R- and Ti$_3$Ni$_4$ phases.

3.3. Hardness

The strength characteristics of SMAs, such as the ultimate tensile strength, transformation and dislocation yield stresses, and hardness, largely affect the value of the completely recoverable strain, which is a main SMA functional property. The strain, which can be “induced” without involving of dislocation slip and, accordingly, “returned” (during heating or unloading) by only strain recovery mechanisms such as thermoelastic martensitic transformation or reorientation of martensitic crystals, also increases with the increase in the resistance to plastic deformation.

Average hardness values (HV) of the nickel-enriched Ti-Ni SMA after studied regimes of compression and PDA are shown in Figure 7. With the increase in the deformation temperature, the hardness gradually decreases down to RT level, which is reached at 600 °C. This decrease in hardness value is explained by the intensification of dynamic softening processes with an increase in the deformation temperature.

![Figure 7. Hardness test results after studied regimes of compression and PDA.](image)

PDA of nickel-enriched Ti-Ni SMA at 430 °C after deformation in the range of 100–700 °C leads to the increase in the hardness value of 40–60 HV (Figure 7); that is explained by the precipitation hardening. This behavior of aging alloy differs from the equiatomic one, for which occurrence of PDA does not lead to a significant change in the hardness value [35]. Thus, the increase in hardness, observed in the nickel-enriched Ti-Ni SMA, can be attributed to the additional strengthening effect of Ti$_3$Ni$_4$ particles, precipitated during PDA.

3.4. Functional Properties

After all studied regimes of compression and PDA, the completely recoverable strain equals not less than 7% (Figure 8a). Nickel-enriched Ti-Ni SMA usually exhibits better shape recovery behavior as compared to equiatomic ones [35]. This tendency can be explained by the development of martensitic transformations through the intermediate R-phase in case of nickel-enriched Ti-Ni SMA, while in the equiatomic one, the main mechanism of shape recovery is the reorientation of martensitic crystals. Figure 8a demonstrates a certain advantage in $\varepsilon_{rt,1}^{max}$ in the case of dynamically polygonized structure formation in 400 to 600 °C range.
The ratio of the SME and SE contributions to the completely recoverable strain after most of the studied regimes of TMT favors SME. After compression at temperatures below 300 °C, these contributions of SME and SE to the completely recoverable strain become approximately equal [23]. Application of PDA after all studied regimes of compression slightly decreases the contribution of SE. The adjustment of the contributions of SME and SE components to the completely recoverable strain using the control of processing regimes can be applied to the development of devices relying on the SE or SME, respectively.

The use of PDA leads to an increase in the functional characteristics of shape recovery (Figure 8). Thus, the maximum value of total completely recoverable strain is equal to 11.8% after PDA against and 8.8% without PDA (Figure 8a). However, in contrast to the equiatomic alloy [30], this positive effect of PDA is distinctly higher when the temperature of PDA is equal to or lower than the compression temperature. This feature of the nickel-enriched Ti-Ni SMA is explained by the development of static aging processes during PDA. The hardening at 430 °C by the precipitation of Ti$_3$Ni$_4$ particles has a more noticeable effect on the alloy in a relatively weak hardened state after compression at elevated temperatures than in a strongly hardened state in case of low-temperature compression.

PDA provides an increase in the shape recovery rate (SRR) to 100% when the PDA temperature is equal to or lower than the compression temperature (Figure 8b). This fits into the general pattern of the effect of PDA on the functional properties of the alloy. Thus, the highest SRR values were obtained with total induced strain of 10–12% (which is close to a theoretical limit of the transformation lattice strain) as a result of PDA after compression in the range of 400–600 °C, i.e., in the temperature range of dynamic polygonization.

PDA of the nickel-enriched Ti-Ni SMA at 430 °C (1 h) leads to an increase in the functional characteristics of shape recovery because of the dispersion strengthening from...
precipitated particles of the Ti$_3$Ni$_4$ phase. It causes an increase in the dislocation (true) yield stress and an increase in the difference between the dislocation and transformation yield stresses. This enhances realization of the functional characteristics of shape recovery.

Thus, it has been found that the application of post-deformation annealing to nickel-enriched Ti-Ni alloy improves the functional characteristics of shape recovery. PDA is accompanied by the development of precipitation hardening processes due to the precipitation of Ti$_3$Ni$_4$ particles. The most significant increase in properties is observed when the PDA temperature of 430 °C (1 h) is lower than the compression temperature, and the aging process occurs in the dynamically polygonized dislocation substructure.

4. Conclusions

Analysis of the effects of the post-deformation annealing after compressing deformation with a true strain of 0.5 on the structure and properties of nickel-enriched Ti-50.9 at.% Ni SMA allows formulating the following conclusions.

1. When the PDA temperature of 430 °C (1 h) is below the deformation temperature, it does not lead to a decrease in the lattice defects of the initially deformed B2-austenite.
2. PDA of nickel-enriched Ti-Ni SMA at 430 °C (1 h) of samples with the dynamically recovered, dynamically polygonized or dynamically recrystallized structures after compressing deformation leads to the precipitation of finely dispersed (nanosized) Ti$_3$Ni$_4$ particles. All three main phases are presented after PDA: B2-, R- and Ti$_3$Ni$_4$.
3. PDA of the nickel-enriched Ti-Ni SMA at 430 °C (1 h) after deformation in the 100–700 °C range leads to the increase of the hardness by 40–60 HV due to a precipitation hardening as a result of precipitation of nanosized Ti$_3$Ni$_4$ particles.
4. PDA of the nickel-enriched Ti-Ni SMA at 430 °C (1 h) leads to an increase in the functional characteristics of shape recovery. The most significant increase of the completely recoverable strain (from 8.8 to 11.8%) and shape recovery rate (from 88 to 100%) as compared to the reference treatment is observed after PDA of the alloy with the dynamically polygonised structure.

Author Contributions: Conceptualization, V.K. and R.K.; methodology, I.K.; validation, D.G.; formal analysis, V.Y.; investigation, V.K. and R.K.; writing—original draft preparation, V.K. and R.K.; writing—review and editing, I.K. and V.Y.; supervision, I.K. and D.G. All authors have read and agreed to the published version of the manuscript.

Funding: The reported study was funded by RFBR (project number 19-33-60090) in the part of structural and X-ray analysis, study of properties; Federal Academic Leadership Program Priority 2030 in the part of thermomechanical treatment. The TEM characterization was carried out on the equipment of the Center Collective Use “Materials Science and Metallurgy” with financial support by the Ministry of Education and Science, Russia (No. 075-15-2021-696).

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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