INFLUENCE OF QUASI-CONTINUOUS ECAP WITH VARIOUS CHANNEL INTERSECTION ANGLES ON THE STRUCTURE FORMATION, MECHANICAL AND FUNCTIONAL PROPERTIES OF Ti-Ni SHAPE MEMORY ALLOYS

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Abstract

Influence of quasi-continuous ECAP with channel intersection angles of 110 and 120° on the structure formation and properties of Ti-50.1 at% Ni shape memory alloy was studied and compared. The structure was studied using X-ray diffraction analyses and transmission electron microscopy. The mechanical properties were determined by the uniaxial tensile and hardness tests. The maximum completely recoverable strain and temperatures of reverse martensitic transformation were estimated by a thermomechanical method using a bending mode for strain inducing. After ECAP with channel intersection angle of 120° for 3 passes at 400 °C a mixed ultrafine-grained structure with high density of free dislocations and incompletely equiaxed structural elements size of about 130-150 nm is formed. In comparison, after ECAP with channel intersection angle of 110° an ultrafine-grained structure with deformation bands elongated in the direction perpendicular to the sample axis, consisting of the less uniform equiaxed and ellipsoid structural elements with the size of about 50-150 nm is obtained. After both studied ECAP regimes samples have relatively high values of hardness (220 HV) and strength characteristics (σ_y = 850/1000 MPa, σ_B = 1040/1020 MPa, 120/110°). Maximum completely recoverable strain of 7.1 % is obtained after ECAP with channel intersection angle of 110°, and post-deformation annealing at 400 °C, 1 h.

Keywords: Shape-memory alloys, titanium nickelide, ultrafine-grained structure, equal channel angular pressing, functional properties

1. INTRODUCTION

Ti-Ni-based shape memory and alloys (SMA) are widely used in different fields of engineering and medicine as a functional material for production of various shape-memory devices [1-6]. It is well-known that formation of ultrafine-grained (UFG) structure in Ti-Ni SMA allows considerably improving mechanical and functional properties. Severe plastic deformation (SPD) is one of the most attractive methods of thermomechanical treatment used for structure refinement [7-11]. In their turn, the most promising SPD method for production of bulk UFG billets from Ti-Ni SMA is equal channel angular pressing (ECAP) [12-16]. The best combination of properties in Ti-Ni SMA can be achieved after formation of completely nanocrystalline structure (60-80 nm) [14]. Traditional mode of the ECAP (with additional heating in the pauses between the passes) allow obtaining only structure with the average grain/subgrain size of elements about 150-250 nm [8]. According to [17], for the further grain refinement ECAP in quasi-continuous mode can be used. It allows obtaining mixed nanosubgrained and nonocrystaline structure (average size 103 ± 5 nm) and increase the completely recoverable strain (one of the primary functional property of SMA) to 9.5 % due to the peculiarities of mixed nanograined/nanosubgrained structure with a high dislocation density which prevents plastic deformation by dislocation slip during deformation inducing shape memory effect [14]. For the additional investigation of the quasi-continuous ECAP process it is necessary to analyze the influence of change of basic ECAP parameters on the evolution of structure and properties. In this context, for the further study of ECAP in quasi-continuous mode, the influence of the channel intersection angle decrease from 120 to 110° on the features of structure formation, mechanical and functional properties is performed in the present work.

2. EXPERIMENTAL PROCEDURE

In the present work, Ti-50.1 at% Ni alloy, supplied by “Industrial Center MATEK-SMA Ltd.”, was studied. The billets for ECAP, rods 20 mm in diameter, were produced by screw rolling at 850-950 °C with reduction of 7-20 % per pass and interpass heating (hot-rolled state). ECAP was carried out after annealing at 750 °C, 0.5 h (reference treatment – RT) in the quasi-continuous mode at the temperature of 400 °C for 3 passes for both channel intersection angels 110 and 120°. A post-deformation annealing (PDA) at the deformation temperature for 1 h was performed after ECAP with channel intersection angel of 110° for the study of stability of structure and properties. The structure was studied at room temperature using “Ultima IV Rigaku” X-ray diffractometer and “JEM-2100” transmission electron microscope. The mechanical properties were determined at room temperature by the uniaxial tensile tests using universal tensile machine “INSTRON 3382”. The Vickers hardness measurements were carried out at room temperature using a “LECOM 400-A” tester under a load of 1 N. The maximum completely recoverable strain and starting and finishing temperatures of reverse martensitic transformation A_s and A_f were estimated by a thermomechanical method using a bending mode for strain inducing.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction analyses

The phase composition of the samples after RT and two regimes of ECAP is estimated using X-ray diffractograms shown in **Figure 1**.

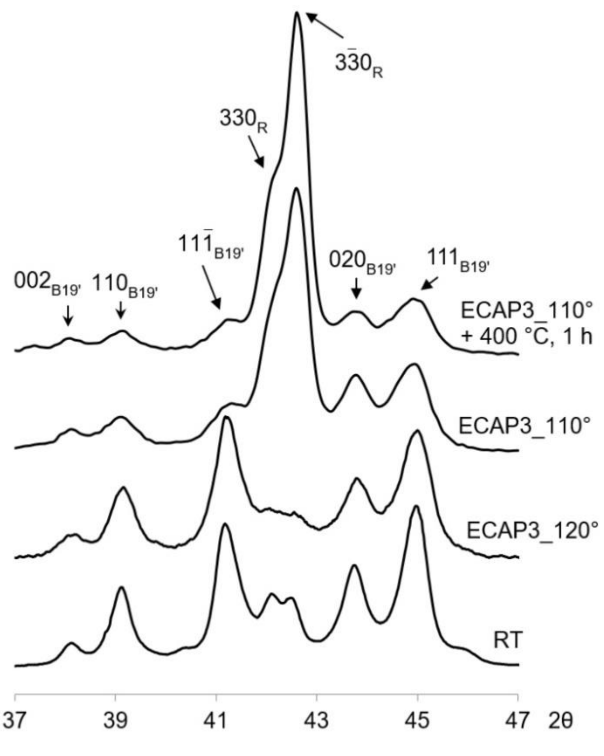

Figure 1 X-ray diffraction patterns of the ECAP under different regimes

After RT mixed phase composition can be seen: the main phase is B19'-martensite, and not more than 20 % of the rhombohedral R-phase and B2 austenite are also present. After ECAP with channel intersection angle of a 120° close phase composition is defined, but with the certain broadening of the $002_{B19'}$ peak width due to an increase in deformation hardening. The lowering of the channel intersection angle to 110° leads to additional broadening of the $002_{B19'}$ peak, and to a decrease in the amount of martensite due to the increase of B2→R transformation temperature and formation of the R-phase. The post-deformation annealing (PDA) does not lead to the significant difference in comparison with the state after ECAP, except the slightly more noticeable separation of R-phase peaks, and some increase in the R-phase amount.

3.2. Transmission electron microscopy

The results of TEM study after ECAP with channel intersection angle of 120° correlates well with X-ray diffraction analyses. A mixed structure with high density of free dislocations and incompletely equiaxed grains and subgrains of submicron size is formed (**Figure 2**).

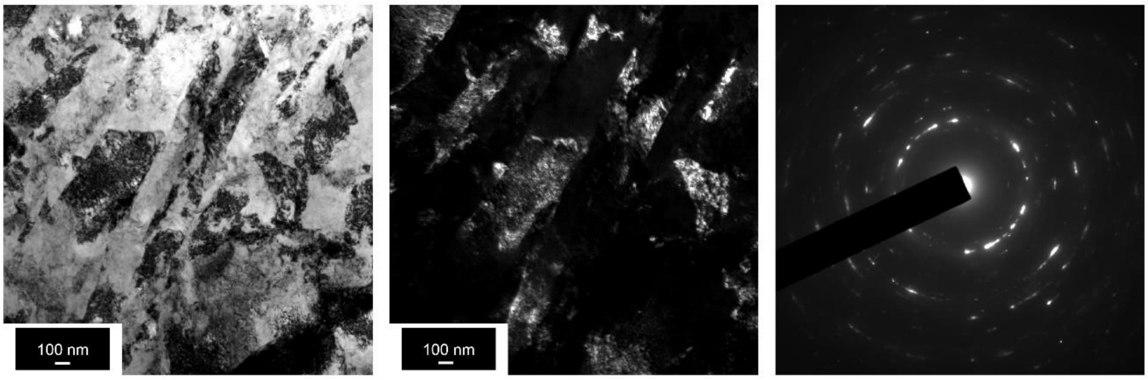

Figure 2 Microstructure of the Ti-Ni alloy subjected to quasi-continuous ECAP with channel intersection angle of 120° for three passes at 400 °C. Transmission electron microscopy: bright field image, dark field image, electron diffraction patterns

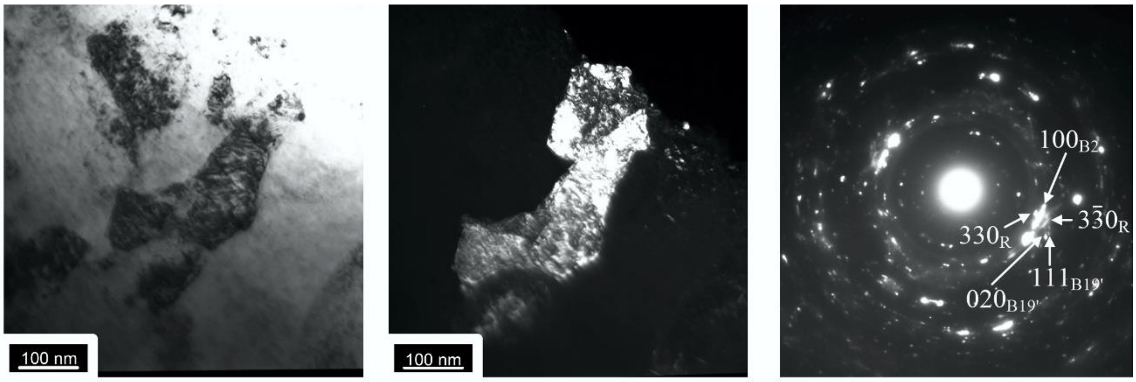

Figure 3 Microstructure of the Ti-Ni alloy subjected to quasi-continuous ECAP with channel intersection angle of 110° for three passes at 400 °C. Transmission electron microscopy: bright field image, dark field image, electron diffraction patterns

In the electron diffraction patterns both discrete and continuous arc reflections are observed. The liner size of structural elements is about 130-150 nm, grains/subgrains with a size of less than 100 nm i.e. are also presented. Analyses of microstructure after ECAP with channel intersection angle of 110° shows that a complex ultrafine-grained structure is formed (**Figure 3**). Patterns of three main phases, i.e., B19'-martensite, R-martensite, and B2-austenite may be observed in **Figure 3**. In comparison with the structure after ECAP with angle of 120° the structural elements are less uniform. In the bright-field images, deformation bands elongated in the direction perpendicular to the sample axis are observed. They consist of equiaxed and ellipsoid structural elements, with long axis also oriented in the direction perpendicular to the sample axis. In the strong reflexes of the first diffraction ring both groups of closely oriented structural elements (subgrains) adjacent to each other as well as individual bright elements (grains with high-angle misorientation) are defined. The annularity of the electron diffraction pattern increases due to an increase in the azimuthal broadening of phase reflections, which indicates an increase in the local crystal lattice distortion because of slight increase in its defectiveness. The sizes of the elements are in the range from 50 to 150 nm. Density of free dislocations is visually higher.

3.3. Comparison of mechanical and functional properties

Mechanical and functional properties of Ti-Ni samples after RT and ECAP with both studied channel intersection angles are performed in **Table 1**.

Table 1 Mechanical and functional properties after different regimes of ECAP

Treatment	σ_{cr} (MPa)	σ_y (MPa)	$\Delta\sigma$ (MPa)	σ_B (MPa)	δ (%)	HV (ea)	$\varepsilon_{r,1}^{max}$ (%)	A_S (°C)	A_f (°C)
Reference	100	430	330	700	28	130	2.0	42	64
ECAP3_120°	140	850	710	1040	17	220	6.3	57	67
ECAP3_110°	150	1000	850	1020	52	220	6.8	47	62
ECAP3_110° + 400 °C, 1 h	120	1000	880	1050	49	220	7.1	46	65

Measurements of the Vickers hardness show high hardness value after both ECAP regimes in comparison with reference treatment (RT – 130 HV, both ECAP regimes – 220 HV). The same hardness value after both ECAP regimes indicates that an increase in the accumulated strain does not lead to an additional hardening. The analyses of mechanical behavior during tensile tests at room temperature shows that after both ECAP regimes the strength characteristics (σ_y = 850/1000 MPa, σ_B = 1040/1020 MPa, 120/110°) are much higher than after RT (σ_y = 430 MPa, σ_B = 700 MPa). The maximum ultimate tensile strength is observed after PDA of samples after ECAP with channel intersection angle of 110° (σ_B = 1050 MPa). High elongation to failure after ECAP with channel intersection angle of 110° should also be noted (δ = 52 %). It may be explained by effect of anomalously high plasticity of Ti-Ni alloys during deformation in the region of the existence of the R- phase near the R→ B19' martensitic transformation (**Figure 1**) [18-19]. Maximum completely recoverable strain of 7.1 % was obtained after ECAP with channel intersection angle of 110° and PDA at 400 °C, 1 h (**Table 1**). It correlates with the maximum difference between the values of the transformation yield stress (σ_y) and critical stress of martensite reorientation (σ_{cr}) $\Delta\sigma$ = 880 MPa. Increase of transformation yield stress value (σ_y) after ECAP with channel intersection angle of 110° may be defined by the increase in deformation hardening and corresponding increase in the value of crystal lattice defectiveness. The comparison of shape recovery temperatures after two studies ECAP regimes and RT shows slight increase of starting temperature of reverse martensitic transformation A_S, especially after ECAP with channel intersection angle of 120°. Finishing temperature of reverse martensitic transformation A_f is practically the same after RF and ECAP. The error limits of the reported values are as follows: ±15 MPa for σ, ± 9 for HV, ± 1.4 % for δ, ± 0.3 % for $\varepsilon_{r,1}^{max}$, ± 3.0 °C for A_S and A_f.

4. CONCLUSION

After ECAP with channel intersection angle of 120° for 3 passes at 400 °C a mixed ultrafine-grained structure with high density of free dislocations and incompletely equiaxed structural elements size of about

130-150 nm in Ti-50.1 at% Ni shape memory alloy is formed. In comparison, after ECAP with channel intersection angle of 110° an ultrafine-grained structure with deformation bands elongated in the direction perpendicular to the sample axis, consisting of the less uniform equiaxed and ellipsoid structural elements with the size of about 50-150 nm is obtained. The closed phase composition after both studied regimes of ECAP at room temperature is presented: B19'-martensite, R-phase and B2 austenite, but with different ratio between the phases. The lowering of the channel intersection angle to 110° leads to a decrease in the amount of martensite and formation of the R-phase due to the increase of B2→R transformation temperature. After both studied ECAP regimes samples have relatively high values of hardness (220 HV), strength characteristics ($\sigma_{0,2}$ = 850/1000 MPa, σ_B = 1040/1020 MPa, 120/110°) and functional properties ($\varepsilon_{r,1}^{max}$ = 6.3/6.8 %, 120/110°). Maximum completely recoverable strain of 7.1 % is obtained after ECAP with channel intersection angle of 110°, plus post-deformation annealing 400 °C, 1 h. It correlates with the maximum value of transformation yield stress (σ_y = 1000 MPa) and corresponding increase of difference between σ_y and critical stress of martensite reorientation σ_{cr}.

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