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Structure and properties of Ti–Ni-based alloys after equal-channel angular pressing and high-pressure torsion

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Abstract

Structure formation and functional properties of Ti–48.5, 50.0, 50.6 and 50.7 at.% Ni and Ti–47 at.% Ni–3 at.% Fe shape memory alloys under conditions of high-pressure torsion (HPT) and equal-channel angular pressing (ECAP) in dependence on deformation temperature and post-deformation annealing were studied using electron microscopy and mechanical testing methods. The upper limiting deformation temperature for nanocrystalline structure formation under continuous severe deformation in HPT were determined for aging (somewhat higher than 400 °C) and non-aging (about 300–350 °C) alloys. As a result of ECAP of Ti–Ni and Ti–Ni–Fe alloys at 350–500 °C in six to eight passes, a submicrocrystalline structure with the grain size of 0.1–0.2 μm (at 350 °C), 0.2–0.4 μm (at 450 °C) and 0.3–0.5 μm (at 500 °C) was obtained. The highest functional properties of Ti–50.6% Ni alloy which exceed the best results provided by traditional thermomechanical treatment, correspond to the finest submicrocrystalline structure formation after ECAP at 350 °C. The low-temperature annealing after ECAP does not deteriorate the functional properties as it does not increase the austenite grain size. For obtaining actual nanocrystalline structure in bulk samples under ECAP conditions, the ECAP temperature should be below 350 °C.

Keywords: Ti–Ni shape memory alloys; Nanocrystalline structure; Severe plastic deformation; Submicrocrystalline structure; Functional properties

1. Introduction

Till present, the most effective method for the regulation and improvement of the functional properties of Ti–Ni shape memory alloys (SMAs) was a thermomechanical treatment (TMT) creating a well-developed dislocation substructure [1]. Application of new TMT schemes involving a severe plastic deformation (SPD) can produce ultra-fine grained, i.e. nano- or submicrocrystalline structure and thus opens extra possibilities for regulation of the structure-sensitive functional properties of SMAs [2–4].

The most popular methods of SPD are high-pressure torsion (HPT) and equal-channel angular pressing (ECAP) [2–6]. The first one is used for research purposes while the second one is interesting for practice as it creates an ultra-fine grained structure in bulk samples. There is a difference between ECAP and HPT procedures. HPT is a continuous process while ECAP is a multipass process which includes interpass annealings at the deformation temperature which leads to grain coarsening [7].

In the present paper, the thermomechanical conditions of SPD in HPT and post-deformation annealing under which the nanocrystalline grained structure forms in Ti–Ni-based SMAs and their functional properties SPD in ECAP compared to the traditional TMT are described.

2. Experimental

Ti–48.5, 50.0, 50.2, 50.6 and 50.7 at.% Ni and Ti–47 at.% Ni–3 at.% Fe SMAs were subjected to HPT at RT and at elevated temperatures (200–400 °C), and ECAP in the 500–350 °C

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Table 1
Grain-size in dependence of HPT temperature for the alloys studied ($N = 10$, $P = 4$ GPa)

HPT temperature (°C)	Grain size (nm)		
	Ti–48.5 at.% Ni, $M_s = 68$ °C	Ti–50.7 at.% Ni, $M_s = -20$ °C	Ti–47 at.% Ni–3 at.% Fe, $M_s = -160$ °C
200	–	5–30	5–30
250	–	5–40	5–40
270	5–30	–	–
300	–	8–50	–
350	30–150	10–60	10–80
400	–	20–80	15–90
450	–	–	30–200

temperature range. To determine the conditions of nano- or submicrocrystalline structure formation, the samples 0.2 mm in diameter × 3 mm long were deformed in the HPT scheme. The number of revolutions N varied from 1 to 15 (true strain from $e = 3.8$ to 6.6). The pressure value was 4 GPa. In the case of ECAP, samples 16 mm in diameter × 70 mm long were used and the number of passes varied from $N = 4$ to 8 ($e = 3.2$–6.5). After HPT and ECAP, the samples were annealed in the temperature range from 200 to 500 °C. Structure and substructure evolution was studied using transmission electron microscope JEM-100C and JEOL 2100F. Tensile tests were performed in a temperature range from −18 to 200 °C. The deformation for recovery stress generation was induced by tension, and the maximum recovery stress was measured upon heating. To determine the maximum completely recoverable strain, the samples were bent around the mandrels of various diameters and then heated above A_f temperature.

3. Results and discussion

The nanocrystalline structure can be obtained as a result of deformation in HPT and also by crystallization of amorphous structure. HPT at elevated temperatures was used to reveal the highest deformation temperature for nano-sized grain formation. Increase of HPT temperature in the austenitic temperature range suppresses amorphization due to decreasing the resistance to lattice plastic deformation, shifting of the deformation temperature to higher temperatures relatively to the M_s and involving lattice diffusion processes [6]. The upper deformation temperature limit for the partial amorphization in the alloy Ti–48.5 at.% Ni having the highest M_s temperature lies about 300 °C. It lowers as the M_s temperature of an alloy lowers. On the other hand, increasing of the deformation temperature promotes nanocrystalline structure formation, however, up to a certain temperature above which not nanocrystalline but submicrocrystalline structure forms. This limiting temperature is about 350 °C for non-aging Ti–48.5 at.% Ni alloy, and it is somewhat above 400 °C for aging Ti–50.7 at.% Ni alloy (Table 1). In the last case, the blocking effect of precipitated particles retards grain growth. For the Ti–Ni–Fe alloy, the temperature for the nanocrystalline structure formation is also somewhat about 400 °C (see Table 1). However, the above mentioned conclusions are related to the continuous HPT deformation. On the contrary, the ECAP is discontinuous processing which includes interpass annealings at a deformation temperature. Therefore, the ECAP in the deformation temperature range of 350–500 °C results in not nanocrystalline (grain size <100 nm) but more coarse-grained submicrocrystalline (grain/subgrain size of 0.1–1 μm) structure.

In Table 2, the structure and functional properties of the SMAs as result of ECAP are presented. Equal-channel angular pressing in the range 500–400 °C of Ti–50.2 at.% Ni alloy ($N = 1$, 4, 8 and 12) leads to formation of a submicrocrystalline structure (grain size of 0.2–0.4 μm) and determines high recovery strain: maximum completely recoverable strain ε_r^{max}, grows from 6% after reference quenching to 9% after ECAP in 4 passes and to 7.5–8% after 8 and 12 passes. The highest value of maximum recovery stress σ_r^{max} is reached after ECAP in 8–12 passes (see Table 2). As it is seen, the combination of ε_r^{max} and σ_r^{max} after such ECAP is somewhat better than after "the best" traditional low-temperature TMT in rolling plus post-deformation annealing (PDA, see Table 2) which creates a polygonized substructure.

In the case of Ti–47 at.% Ni–3 at.% Fe alloy, the submicrocrystalline structure formed as a result of ECAP also improves the functional properties: ε_r^{max} grows to 9% and σ_r^{max} to 695 MPa against to 8% and 420 MPa after traditional high-temperature TMT (HTMT, see Table 2), the latter creates dynamically polygonized substructure.

The Ti–50.6 at.% Ni possessed a better deformability that allowed lowering the ECAP temperature down to 350 °C. Application of ECAP at 350 °C to Ti–50.6 at.% Ni alloy was most successful. In Fig. 1 and Table 2, the results of ECAP at 450 and 350 °C are compared. The highest functional properties of Ti–50.6 at.% Ni alloy which exceed the best results provided by traditional thermomechanical LTMT + PDA, correspond to the finest submicrocrystalline structure formation after ECAP at 350 °C (grain size of 0.1–0.2 μm) (see Fig. 1, regime 2, Table 2). The low-temperature annealing (350 °C) after ECAP does not deteriorate the functional properties as it does not increase the austenite grain size (see Fig. 1, regime 3), while the annealing at 450 °C leads to grain growth to 0.2–0.4 μm and corresponding σ_r^{max} lowering (see Fig. 1, regime 4).

An ultimately high combination of functional properties of given alloy can be obtained when a nanocrystalline structure forms as a result of TMT including SPD [5]. It is shown in Table 2 for Ti–50.0 at.% Ni severely deformed in rolling (true strain $e = 1.9$) and then annealed at 400 °C. However, to obtain