Materials Science Forum *Online: 2010-12-30*
ISSN: 1662-9752, Vols. 667-669, pp 1071-1076
doi:10.4028/www.scientific.net/MSF.667-669.1071

Effect of Equal-Channel Angular Pressing on the Microstructure and Superelastic Behavior of Ti-25 at. % Nb Shape Memory Alloy

Jie Song[1a], Xiaoning Zhang[1b], Xiaogang Sun[1c], Chaoying Xie[1d]

[1]School of Materials Science and Engineering, Shanghai Jiao Tong University,
Shanghai 200240, China

[a]jies@sjtu.edu.cn, [b]zn100@ sjtu.edu.cn, [c]sxgsxg@sjtu.edu.cn, [d]cyxie@ sjtu.edu.cn

Keywords: Ti-Nb alloy; ECAP; superelastic behavior

Abstract: Microstructure and mechanical behavior of Ti-25 at. % Nb shape memory alloy processed by equal-channel angular pressing (ECAP) at 673 K have been investigated. The effect of multi-passes ECAP on the martensitic transformation temperature and superelastic recovery strain of Ti-26 at. %Nb alloy have been analyzed. It is found that the Ti-25 at. % Nb alloy exhibits superelasticity with a recovery strain of 1.6 % after one pass ECAP process. As the ECAP pass numbers increases to four passes, the superelasticity increases a little. The yielding stress ($\sigma_{0.2}$) of Ti-25 at. % Nb alloy increases to 430 MPa and the ultimate tensile strength (UTS) is near 678 MPa after one pass ECAP process at 673 K, but the yielding stress and ultimate tensile strength increases little after four pass ECAP. The grain size decreases sharply from 100 μm to no more than 500 nm after four passes ECAP process. Effect of ECAP on the microstructure evolution and Young's modulus has been analyzed.

Introduction

Recently, Ti-Nb based β-type Titanium alloys with shape memory effect and superelasticity have attracted attention as promising functional materials for medical applications[1-7]. Binary Ti-(16-25) at.% Nb alloy exhibits the shape memory effect (SME) and binary Ti-(25.5-27) at.% Nb alloy exhibit superelasticity at room temperature. But, the low critical stress for slip deformation of binary Ti-Nb alloys results in a small recoverable strain[5]. In order to improve the shape memory effect and superelastic recoverable strains, alloying elements were added to increase the mechanical properties, especially the yielding stress, of Ti-Nb alloys. Ti–Nb–Ga, Ti–Nb–Ge[8], Ti–Nb–Zr[9], Ti–Nb–Al [2], Ti-Nb-Ta[6]. Ti-Nb-O[10], Ti-Nb-Ta-Zr[11], Ti-Nb-Sn-Zr[12] alloys with higher yield stress and ultimate tensile strength were developed, exhibiting better shape memory effect or superelasticity.

The thermo-mechanical treatment is another method to improve the mechanical properties of alloys. Severe plastic deformation (SPD) is an important method, including equal channel angular pressing (ECAP), high-pressure torsion (HPT) and multiple forging (MF), etc. The microstructure and properties of shape memory Ti-Ni and Ti-Ni based alloys after ECAP and HPT had been studied by many investigators [13-15], but the study on the microstructure and properties of metastable β-type

Ti-Nb alloys processed by equal-channel angular pressing (ECAP) is little. In the present paper, the ECAP effect on superelastic behavior of binary Ti-25 at. % Nb alloy has been investigated.

Experimental

Ti-25 at. % Nb alloy was prepared from 99.7 % sponge titanium and 99.6 % niobium by magnrtic induction furnace with copper crucible. The ingots were hot forged into bars with a cross dimension of 12×12 mm^2, then, solution treated (ST) at 1143K for 1 hour and quenched into water. Specimens for ECAP were cut from the solution treated bars by spark cutting, with a dimension of 10 mm× 10 mm×140 mm . The ECAP processes were applied at 673 K along Bc route with a ECAP set described in Ref[13].

Specimens of 3 mm×0.8 mm×50 mm for tensile test were cut from these square bar. The surfaces of the specimens were ground to remove the surface oxide layer formed during aging and electric spark cutting. Tensile tests were carried out under a strain rate of 1.0×10^{-4} s^{-1}. Strains were measured using a clip-on extensometer with a gage length of 25 mm. Specimens for microstructure observation were mounted, polished and then etched in a solution of water, nitric acid and hydrofluoric acid with a volume fraction of 80: 15: 5. Microstructures were examined by optical microscopy and TEM (JEOL2100). For TEM observation, thin foil samples were obtained by mechanical grinding to 50 μm thickness and twin jet eletropolishing (5 % perchloric acid, 35 % butyl alcohol and 60 % methyl alcohol; 30V; 243 K). Phase constitution was identified by X-ray diffraction (XRD) with Cu $K\alpha_1$ radiation, obtained from a tube operated at 200 mA, 40 kV. The scan speed is 5 °/s.

Results and discussion

Fig.1 shows the microstructure and the result of X-ray diffraction (XRD) of Ti-25 at. % Nb alloy after solution treated (ST). The solution treated specimen is entirely of single β-phase which is confirmed by X-ray diffraction (XRD).

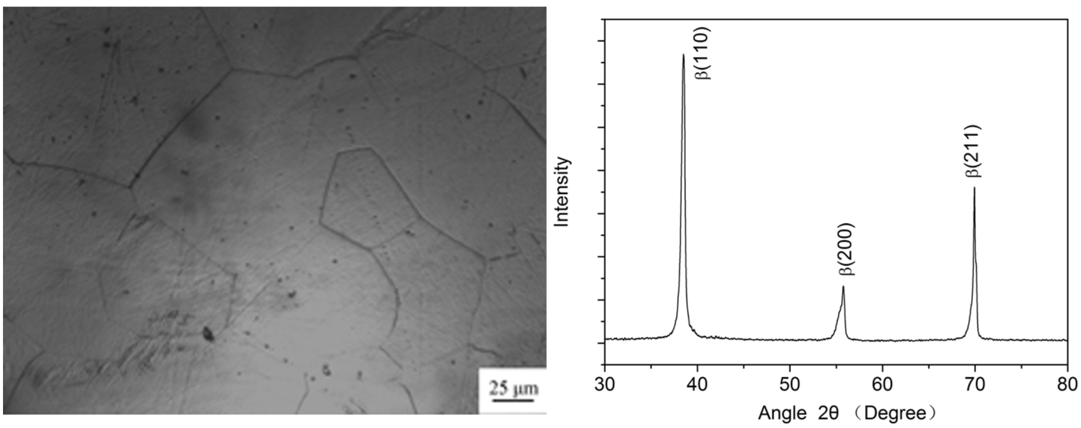

Fig.1 Microstructure and X-ray diffraction result of Ti-25 at.% Nb alloy after solution treatment

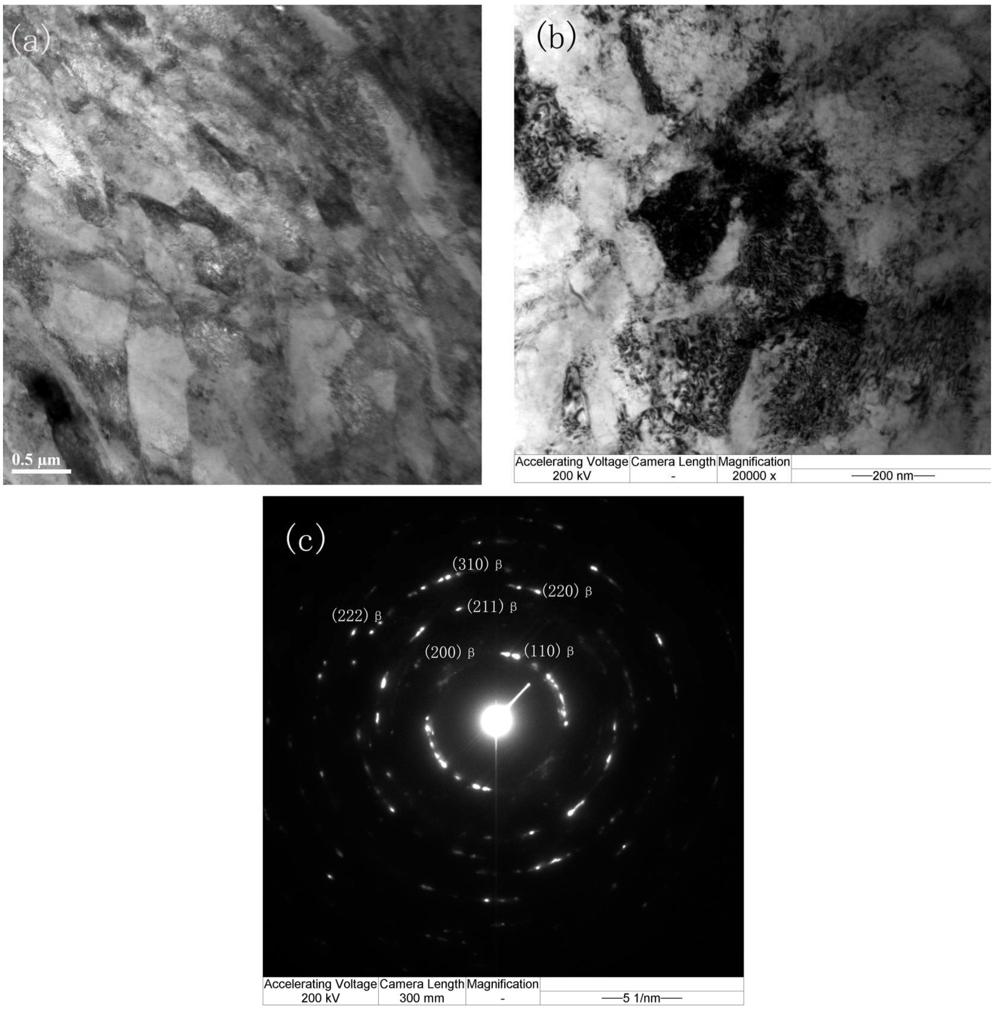

Fig. 2 The TEM microstructure of Ti-25 at.% Nb after ECAP processes, (a) two passes ECAP, (b) four passes ECAP and the corresponding SAD pattern (c).

After one pass ECAP process, the grains were elongated along a direction 45º to the extrusion direction, presenting as the fiber band. After two passes ECAP process, the width of the fiber band becomes narrow only 500 nm. At the same time, some grain have become equaixed grain, as shown in Fig. 2(a). The equaixed grain size is about 500 nm. After four passes ECAP, the fiber band disappeared and the almost entirely grain become equaixed grain. It is evident that a homogenous ultrafine-grained structure with a mean grain size of about 200 nm is formed as shown in Fig. 2(b) and Fig. 2(c).

Fig.3 shows the cyclic stress-strain curves obtained at room temperature for the Ti-25 at.% Nb alloy after solution treatment and ECAP process. As seen in Fig. 3(a), the superelastic recovery strain of solution treated specimen is small. When the specimen is unloaded from tensile strain of 1.5 %, the recovery strain is 1.3 % and the remained stain is 0.2 %. Superelastic recovery is 86 %. As the strain increases during loading-unloading cycle, the recovery strain maintains 1.3 %, while the remained strain increases.

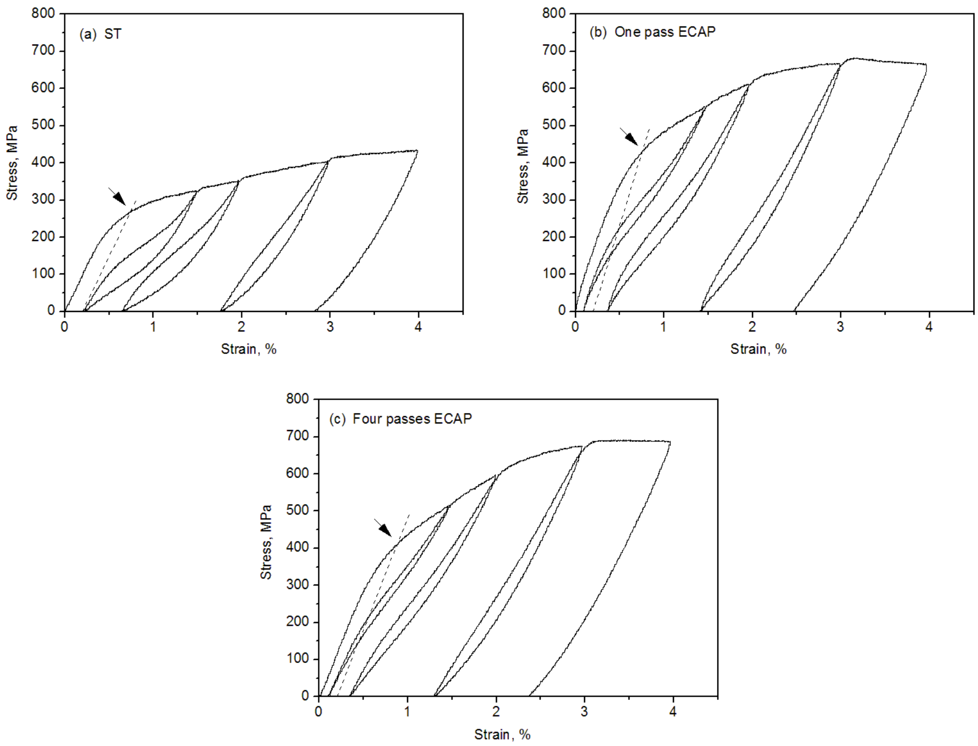

Fig. 3 Cyclic stress-strain curves of Ti-25at. % Nb alloy, (a) ST, (b) one pass ECAP and (c) four passes ECAP.

After one pass ECAP at 673 K, the yielding stress (σ_M) increases obviously, and the recovery strain increases also, as seen in Fig. 3(b). The work hard phenomena become clearer. When the specmen is unloaded from tensile strain of 1.5 %, the recovery strain is 1.4 % and the remaining strain is only 0.1 %. Superelastic recovery is 93 %. When the specimen is unloaded from strain of 2 %, the recovery strain is 1.6 %. With the increase of the pass number of ECAP process, the yielding stress (σ_M) and the ultimate tensile strength (UTS) change little. As shown in Fig. 3(c), after four passes ECAP process at 673 K, the recovery strain inreases to 1.7 %.

When the tensile test is carried out at room temperature, the stress-induced matensitic phae can remain after unloading, Ti-25 at.% Nb alloy exhibts partial superelasticity behavior, as shown in Fig. 3(a). The thermo-mechanical treatment can affect the martensitic transformation start point *Ms*. It is reported that the ECAP process decrease the martensite transformation temperature *Ms* and *As*[16]. So, after one pass ECAP process, the stress-induced matensitic phase cannot remain after unloading. The Ti-25 at. % Nb alloy ECAPed exhibits obviously the superelasic behavior, as seen in Fig. 3(b), which implies that the *Ms* temperature decreases more. It is noticed that the superelasticity of specimen after four passes ECAP is similar to that of the specimen after one pass ECAP process, which implies that the *Ms* temperature change little after further ECAP process[16].

Fig. 4 shows the values of Young's modulus, yielding stress (σ_M) and ultimate tensile strength of specimens solution treated and treated by ECAP. It is clear that the Young's modulus increases to near 70 GPa after one pass ECAP process from 50 GPa of ST specimen. After four passes ECAP prcoses, the Young's modulus decreases again, near 60 GPa. The change of Young's modulus maybe associate with the precipitaed phase (α-phase, ω-phase, etc)[5], which precipitatd during preheating treatment at 673K before ECAP process. Comparing the yeilding stress (σ_M) and ultimate tensile strength (UTS) of specimens after solution treated and ECAP prcoess, it is clear that both the σ_M and UTS increase obviously. The Ti-25 at. % Nb alloy after ECAP process exhibits high yeilding stress, high UTS and improved superelasicity may has potential application as medical materials.

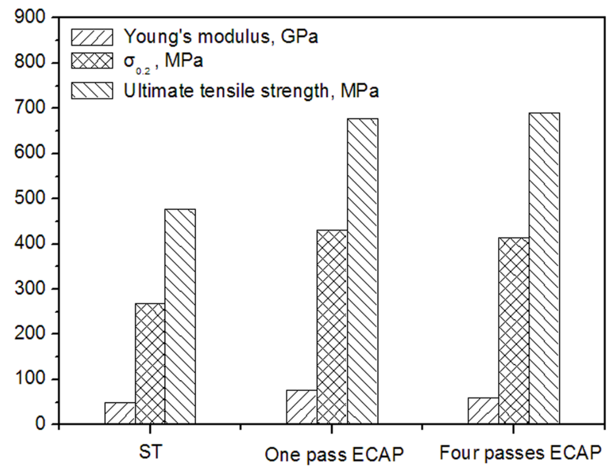

Fig. 4 Young's modulus, yielding stress ($\sigma_{0.2}$) and ultimate tensile strength of ST specimen and treated by ECAP

Summary

The superelasticity of Ti-25 at. % Nb alloy after ECAP process at 673 K has been studied. The summary is as follows:

The grain of Ti-25 at. % Nb alloy can refine to near 200 nm after four passes ECAP process and the almost all of the grains become equaxed ultra-fine grains. The superelasticity is improved after ECAP process. The yielding stress and the ultimate tensile strength increase from 269 MPa, 478 MPa to 430 MPa, 678 MPa, respectively, after one pass ECAP process. As the pass number of ECAP process increases to four, the yielding stress and the ultimate tensile strength increase little.

Acknowledgements

We thank for the financial support from the National Science Fund of China (No.A50671067).

References

[1] L. M. Elias, S. G. Schneider, S. Schneider, H. M. Silva, F. Malvisi: Materials Science and Engineering A, 2006, 432 : 108-112.

[2] H. Hosoda, Y. Kinoshita, Y. Fukui, T. Inamura, K. Wakashima, H.Y. Kim, S. Miyazaki: Materials Science and Engineering: A, 2006, 438-440 : 870-874.

[3] H. Y. Kim, S. Hashimoto, J. I. Kim, T. Inamura, H. Hosoda, S. Miyazaki: Materials Science and Engineering: A, 2006, 417: 120-128.

[4] H. Y. Kim, Y. Ikehara, J. I. Kim, H. Hosoda, S. Miyazaki: Acta Materialia, 2006, 54: 2419-2429.

[5] H.Y. Kim, J.I. Kim, T. Inamura, H. Hosoda, S: Materials Science and Engineering: A, 2006, 438-440: 839-843.

[6] H. Y. Kim, T. Sasaki, K. Okutsu, J. I. Kim, T. Inamura, H. Hosoda: Acta Materialia, 2006, 54: 423-433.

[7] W.Y. Kim, H.S. Kim: Rare Metals, 2006, 25 (6, Supplement 2): 163-167.

[8] T. Inamuraa, Y. Fukuib, H. Hosodaa, K. Wakashimaa, S. Miyazaki: Materials Science and Engineering C 2005, 25 425-432.

[9] J. I. Kim, H. Y. Kim, T. Inamura, H. Hosoda, S. Miyazaki: Materials Science and Engineering: A, 2005, 403: 334-339.

[10] J. I. Kim, H. Y. Kim, H. Hosoda, S. Miyazaki: Mater. Trans., 2005, 46: 852-857.

[11] S. J. Li, R. Yang, M. Niinomi, Y. L. Hao, Y. Y. Cui, Z. X. Guo: Materials Science and Technology, 2005, 21: 678-686.

[12] Y. L. Hao, S. J. Li, S. Y. Sun, C. Y. Zheng, R. Yang: Acta Biomaterialia, 2007, 3 (2): 277-286.

[13] Z. Fan, C. Xie. Materials Science Forum, 2007; Vol. 561-565, 2313-2316.

[14] J. Burow, E. Prokofiev, C. Somsen, J. Frenzel, R. Z. Valiev, G. Eggeler: Materials Science Forum, 2008; Vol. 584-586, 852-857.

[15] I. P. Semenova, R. Z. Valiev, E. B. Yakushina, G. H. Salimgareeva, T. C. Lowe: J. Mater. Sci., 2008, 43: 7354-7359.

[16] Zhiguo Fan, Chaoying Xie: Materials Letters, 2008, 62: 800-803.