Effects of second phases on mechanical properties and martensitic transformations of ECAPed TiNi and Ti−Mo based shape memory alloys

SONG Jie[1], WANG Li-ming[2], ZHANG Xiao-ning[1], SUN Xiao-gang[1],
JIANG Hong[1], FAN Zhi-guo[1], XIE Chao-ying[1], WU M H[3]

1. State Key Laboratory of Metal Matrix Composites, School of Materials Science and Engineering,
Shanghai Jiao Tong University, Shanghai 200240, China;
2. Jiangsu Fasten Company Limited, Jiangying 214400, China;
3. Advanced Materials Technology, Edwards Life Sciences LLC, CA, USA

Abstract: TiNi and Ti-based shape memory alloys were processed by equal channel angular pressing (ECAP) at 673−773 K along B_c route to obtain ultrafine grains for increasing the strength of parent phase and improving the functional properties. The effects of both thermodynamically stable and metastable second phases on the mechanical properties and martensitic transformations of these alloys were investigated. It is found that thermodynamically stable Ti_2Ni phase has no effect on martensitic transformation and superelasticity of Ti-rich TiNi alloy, thermodynamically stable α phase is harmful for ductility of Ti−Mo−Nb−V−Al alloy, but metastable Ti_3Ni_4 phase is effective for R phase transformation, martensitic transformation and superelasticity of Ni-rich TiNi alloy. The mechanisms of the second phases on the martensitic transformations and mechanical properties were discussed.
Key words: TiNi alloy; Ti−Mo based shape memory alloy; equal channel angular pressing (ECAP); second phase; mechanical property

1 Introduction

TiNi alloy with superior shape memory effect (SME) and superelastic properties has been used in diverse areas of applications. In order to extend its application scopes and improve its properties, equal channel angular pressing (ECAP) technique has been applied successfully to TiNi shape memory alloys, and the microstructure evolution, phase transformations and properties of TiNi alloy after ECAP have been investigated [1−6]. The ultra-fined grains, even nanostructured microstructure in TiNi alloys can be obtained after ECAP [1,2,5]. The martensitic transformation point (M_s) of TiNi alloys would decrease after process of ECAP [3−5], and mechanical properties, such as superelasticity, of TiNi alloy were improved [6]. As well known, martensitic transformation temperatures and functional properties of TiNi and Ti-Mo based shape memory alloys (SMA) are sensitive to alloy components. There are stable and metastable second phases for Ti-rich TiNi, Ni-rich TiNi

or Ti−Mo based SMAs precipitated or redissolved during medium temperature thermomechanical process, which have strong effect on the matrix component contents, phase transformations and functional properties. The precipitation and redissolution behavior of these second phases during ECAP process and their effects, especially the second phase for Ti-rich TiNi alloy, are not quite clear yet. In this work, the effects of second phases on the R phase transformation and martensitic transformation and functional properties of both Ti-rich and Ni-rich TiNi alloys have been investigated.

It should be pointed out that high content of toxic Ni atoms in TiNi alloy brings constrain for its broadening medical application. Recently, Ni-free β-type titanium alloys with shape memory effect and superelasticity have attracted attention as promising functional materials for medical applications [7,8]. The superelasticity and shape memory effect of NiTi alloys are usually superior over that of the Ni-free β-type titanium alloys. Thermomechanical treatment is an effective way to improve the mechanical properties, shape memory effect

and superelasticity of these alloys and the second phases have strong effect on the phase transformation behaviour and functional mechanical properties [7−9]. In order to explore a new way to enhance the functional mechanical properties of Ti−Mo based shape memory alloys, Ti−Mo based metastable β titanium alloy (Ti−9.8Mo−3.9Nb− 2V−3.1Al, mass fraction, %) has been processed by ECAP and the effects of both thermodynamically stable and metastable second phases during severe plastic deformation—ECAP process have been investigated in the present study.

2 Experimental

The initial hot forged Ti−50.9%Ni (mole fraction), Ti−50.7%Ni and Ti−50%Ni alloy rods, received from Jiangsu Fasten Company Limited, China, were annealed at 1023 K for 30 min (solution treatment), and cooled into water. Some solution treated samples were aged at 773 K, and then cooled into water. Ti−9.8Mo−3.9Nb− 2V−3.1Al (mass fraction, %) metastable β titanium alloy was received as hot rolled bars from Memry Corporation, USA. These as-received bars were solution treated at 1143 K for 1 h followed by quenching. The billets for ECAP treatment with a dimension of 10 mm×10 mm×120 mm were cut from the solution treated alloy bars. The ECAP processes were applied at 673−773 K along B_c route (the billets were rotated along clockwise direction, 90° between adjacent passes).

Specimens of 3 mm×0.8 mm×50 mm for tensile test were cut from these square bars. 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 gauge length of 25 mm. Samples for DSC test and microstructure observation were cut from the longitudinal plane of billets parallel to the extruding direction. DSC tests were carried out at heating and cooling rates of 10 °C/min to analyze the phase transformations behaviour. Specimens for microstructure observation were mounted, polished and then etched in a solution of water, nitric acid and hydrofluoric acid with a volume ratio of 80:15:5. Microstructures were examined by LeICA−MEF4M optical microscope and JEOL− JEM−2010 electron microscope (TEM) with an accelerating voltage of 200 kV. For TEM observation, thin foil samples were obtained by mechanical grinding to 50 μm thickness and twin jet electro polishing (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$_{\alpha1}$ radiation, obtained from a tube operated at 200 mA, 40 kV. The scan speed was 5 (°)/s.

3 Results and discussion

3.1 Re-dissolution of metastable Ti$_3$Ni$_4$ precipitate during ECAP process

According to Ti−Ni binary diagram, there is two-phase TiNi$_3$ + TiNi equilibrium for Ni-rich TiNi alloy. However, there is only metastable second phase Ti$_3$Ni$_4$ precipitated during aging at a temperature lower than 873 K for less than 2 h for near equal atomic Ni-rich TiNi alloys solution treated [10]. The microstructure of Ti−50.7%Ni alloy solution treated and aged at 773 K for 20 min is shown in Fig. 1. The Ti$_3$Ni$_4$ precipitates are observed in the aged specimen and the SEAD pattern obtained with a zone axis of [111]$_{B2}$ is shown in Fig. 1(b), where the typical 1/7(123)$_{B2}$ spots for Ti$_3$Ni$_4$ are marked with white arrows.
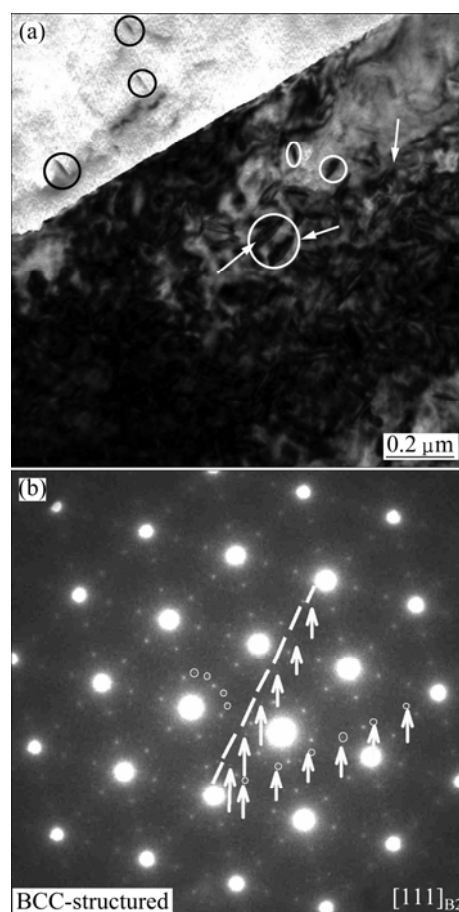
Fig. 1 Microstructures of Ti−50.7% Ni alloys solution-treated followed by aging at 773 K for 60 min: (a) Bright field image; (b) SAED pattern obtained from cycle area in Fig. 1(a) with zone axis of [111]$_{B2}$
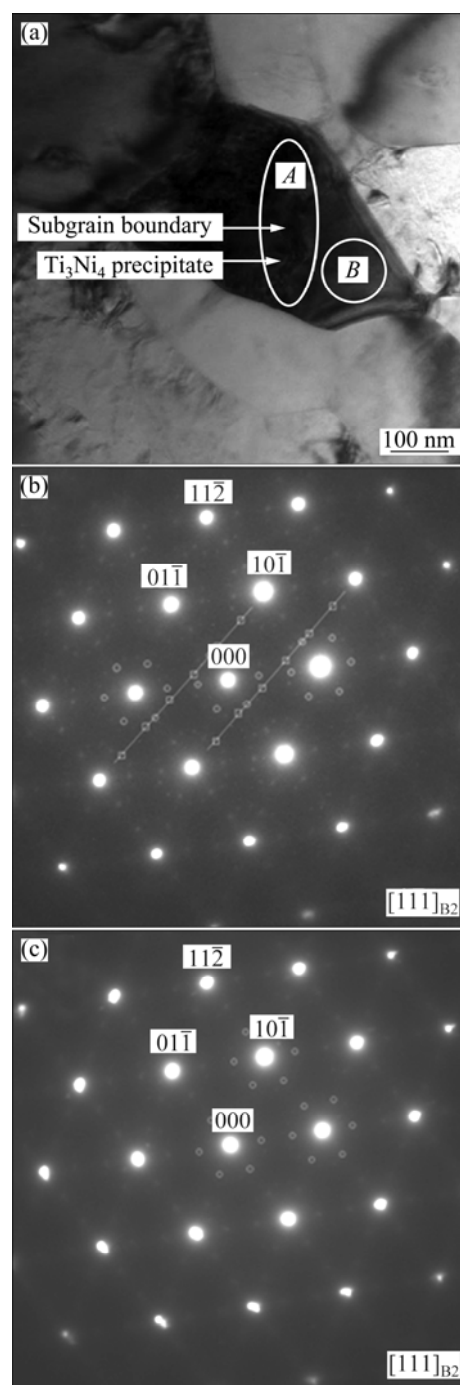
There are few Ti$_3$Ni$_4$ precipitates observed in Ni-rich Ti−50.7%Ni alloy after one pass of ECAP, as shown in Fig. 2, and a few Ti$_3$Ni$_4$ precipitates can be observed only in the regions of subgrain boundaries of Ti−50.9%Ni alloy after eight passes of ECAP treatment.
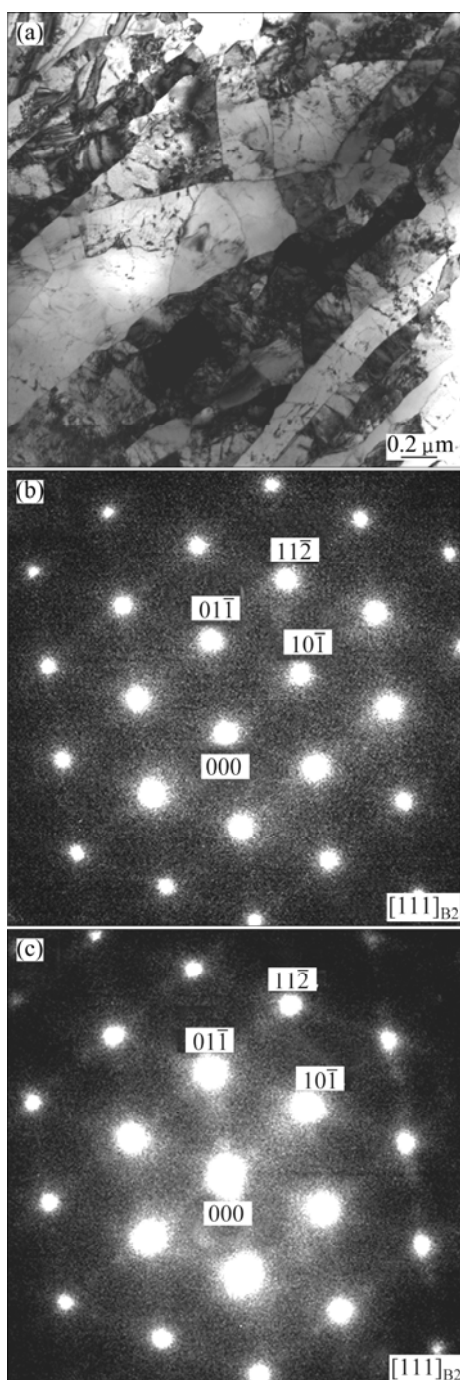
Fig. 2 Morphology of Ti−50.7% Ni alloy after one ECAP pass: (a) Bright field image; (b, c) SAED patterns taken from Fig. 2(a)

Fig. 3 Morphology of Ti−50.9%Ni alloy after the eighth ECAP pass at 773 K by B_C path: (a) Bright field image; (b) SAED pattern from region A in Fig. 3(a) with zone of $[111]_{B2}$; (c) SAED pattern from region B in Fig. 3(a) with zone of $[111]_{B2}$ (Ref. [4])

The SAED pattern is inconspicuous, as shown in Fig. 3, which reveals that a re-dissolving of Ti_3Ni_4 precipitates occurred during medium temperature ECAP treatment.

It is seen from Fig. 3(a) that there is a complex structure containing dislocations and grain boundaries decorated with Ti_3Ni_4 precipitates after the ECAP treatments [4]. Ti_3Ni_4 precipitate re-dissolving has obvious effect on the subsequent martensite transformations. The re-dissolving of Ti_3Ni_4 increases the

Ni content in the matrix, which can lead to the decrease of the temperature of the martensite transformation after the ECAP treatment [3−5].

It has been reported that precipitated second phase can re-dissolve after severe plastic deformation [11−14]. XU et al [15,16] studied Al−4.11%Cu alloy and discovered that thermodynamically metastable θ' phase

precipitated before ECAP almost re-dissolved after ECAP treatment, and the mechanisms of the strain-induced dissolution of precipitates were suggested. The microstructural changes of an aged Al−1.7%Cu alloy associated with severe plastic deformation have been studied, which showed that θ' precipitates were almost completely dissolved after eight passes of ECAP. In the present study, the re-dissolution of Ti_3Ni_4 phase would attribute to lots of dislocation caused by severe plastic deformation (ECAP) providing more positions for the Ni atom dissolution and the activity of Ni atom at medium temperature (773 K) [3,4].

3.2 Phase transformations of ECAPed Ni-rich TiNi alloys

There are three phase transformations related to TiNi shape memory alloys upon cooling or heating in near equal atomic TiNi alloys: the B2⟶B19′, B2⟶R and R⟶B19′ transformations [17]. The DSC curves of Ni-rich Ti−50.7%Ni alloy solution treated and aged at 773 K are shown in Fig. 4. It is seen from Fig. 4(a) that there is only B2⟶B19′ transformation occurring on the cooling curve of solution treated sample. After aging at 773 K for 20 min, the alloy underwent two-step transformations B2⟶R⟶B19′ upon cooling, as seen in Fig. 4(b), and the martensitic transformation temperature (M_p) was slightly decreased compared with the solution treated sample. The R phase is associated with the precipitation of Ti_3Ni_4 phase, which is interpreted in Ref. [17].
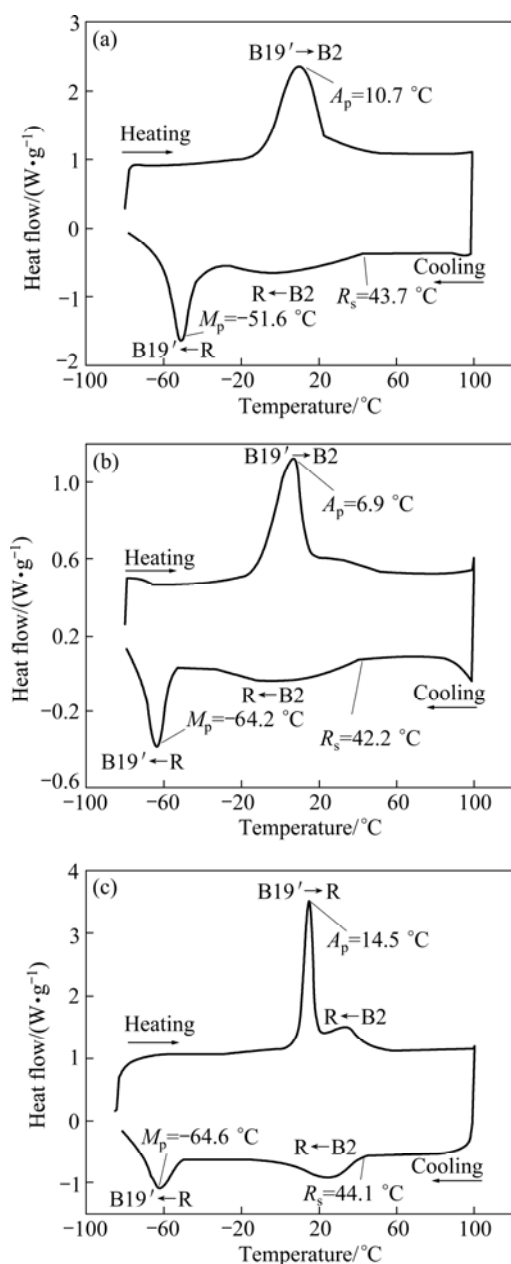
DSC curves of Ti−50.7%Ni alloy processed by ECAP at 773 K are shown in Fig. 5. After one pass of ECAP, the martensitic transformation temperature (M_p) drops a lot, as shown in Fig. 5(a), and the B2⟶R transformation is observed upon cooling, but occurs within large temperature range, in contrast to the one solution treated and aged at 773 K (Fig. 4(b)). The M_p is gradually reduced with the increase of ECAP passes. It is noticed that after eight passes of ECAP, the reverse phase transformations undergo two-step B19′⟶R⟶B2 transformations on heating.

When specimens after one pass of ECAP process were aged at 773 K for 15 min, the sharp and clear B2⟶R transformation curves can be seen in a narrow temperature range, as shown in Fig. 4(c), which is almost the same as that of solution treated and aged specimen (Fig. 4(b)). Furthermore, the martensitic transformation temperature (M_p), R-phase transformation temperature (R_p), austenitic transformation temperature (A_p) of aged TiNi specimens were not changed along with the ECAP treatment [3]. These results reveal that the sharp and clear B2⟶R transformations are attributed to the fine and dense lens-like shape Ti_3Ni_4 phase precipitates formed during aging treatment.
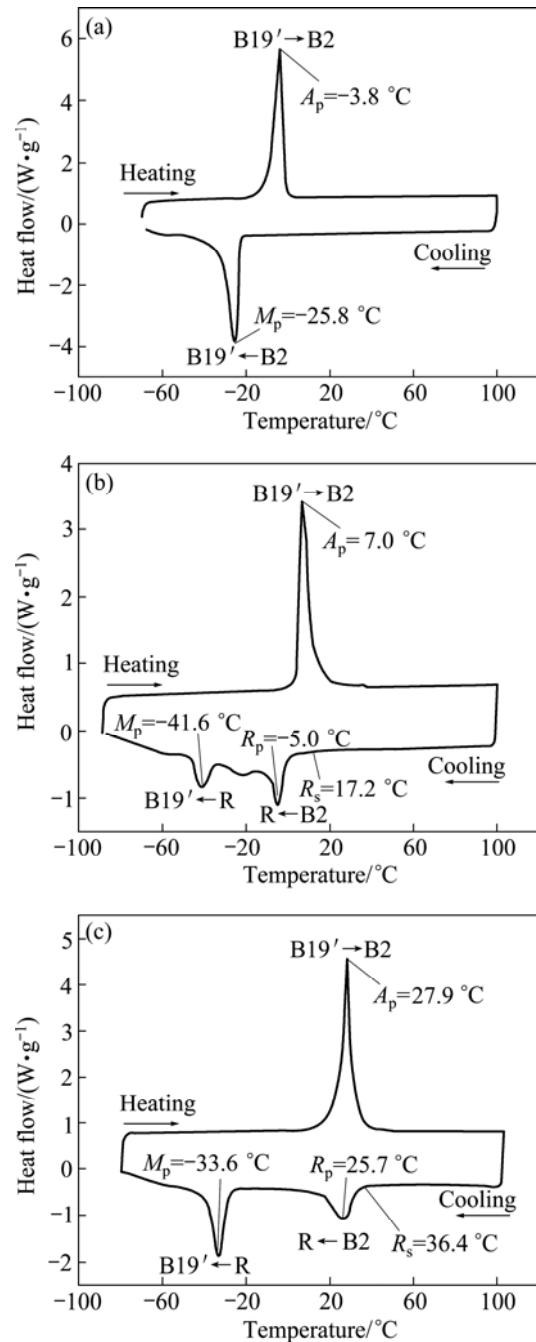
Fig. 4 DSC curves of Ti−50.7%Ni alloy: (a) Solution-treated; (b) Aged at 773 K for 20 min; (c) One ECAP pass +(773 K, 15 min) (Ref. [3])

The broad and smooth B2⟶R phase transformation is obviously not induced by Ti_3Ni_4 precipitation in the ECAPed TiNi specimens, because there is a re-dissolution of Ti_3Ni_4 and few Ti_3Ni_4 precipitate (as shown in Fig. 2). It is reasonably suggested that the broad and smooth B2⟶R phase transformation originates from the refined grains and energy accumulation after ECAP treatments. Because the initial grains are refined dramatically after ECAP treatment, the fraction of grain boundary is largely

Fig. 5 DSC curves of Ti−50.7%Ni alloy processed by ECAP at 773 K: (a) One ECAP pass; (b) 4 ECAP passes; (c) 8 ECAP passes (Ref. [3])

increased and lots of defects such as dislocations are introduced into the grain interior. Extrusion force makes some dislocations slip and pile up around grain boundary. These excessive grain boundaries and dislocations in the grain interior and exterior probably stimulate the B2→R transformation, as well as suppress the R→B19′ transformation and lead to the drop of M_p. Another reason may be that ECAE process usually leads to nonuniform microstructure, which creates local stress fields for inducing the B2→R transformation, but they act as a resistance to R→B19′ transformation. These remaining Ti_3Ni_4 precipitates, as seen in Fig. 3, would

serve as the nucleation sites of the R phase transformation and the presence of Ti_3Ni_4 in the B2 matrix favours the B2→R transformation.

ECAP has advantage in achieving the bulk materials with uniform ultra-fine grains. In this study, the coarse grains of Ti−50.9%Ni alloy decreased from ~70 μm to 300−400 nm after 8 passes of ECAP, and the volume fraction of the grain boundaries increased relatively. All could be attributed to the continuous dynamic recovery/recrystallization during ECAP treatments. The refined submicron grains and microstructures also have a notable effect on the thermo-mechanical properties of the present Ti–50.9%Ni alloy [18]. These geometrical constraints including the submicron grains and the high density grain boundaries would lead to the transformation barrier and change the martensite morphology in the TiNi alloys with ultra-fine grains. They also could bring about the change of the thermal stability and martensite transformation path [19]. Thus, the reduction of martensitic phase transformation temperatures happened. When the martensite transformation was depressed, the sequence B2→R→B19′ in nanostructure TiNi alloys was not observed, only a single B2→R transition could be observed in the nanocrystalline alloy [1,3].

3.3 Effect of stable $TiNi_2$ phase

The XRD pattern of Ti−50%Ni alloy is shown in Fig. 6. There are some Ti_2Ni diffraction peaks and basically B19′ diffraction peaks, which imply that the microstructure of Ti−50%Ni alloy is dual phase (B19′+Ti_2Ni). Ti_2Ni is a stable phase in thermodynamics.
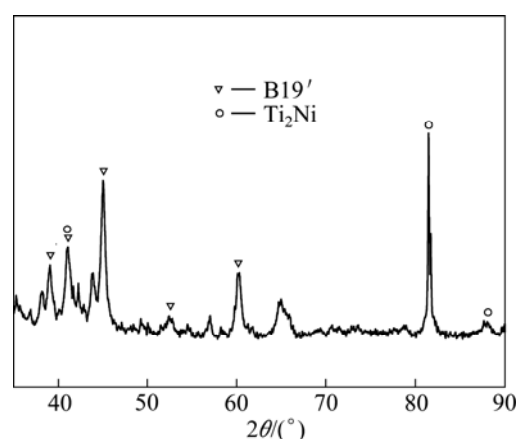
Fig. 6 XRD pattern of solution treated Ti−50%Ni alloy

The microstructures of Ti−50%Ni alloy solution treated and after four ECAP passes are shown in Fig. 7. It is seen that severe plastic deformation occurred after four passes of ECAP process. The DSC curves of solution treated, aged and ECAPed Ti−50%Ni alloy are shown in Fig. 8. There is only B2→B19′ transformation

and no B2→R transformation during cooling. There is no obvious change of martensitic transformation temperature (M_p) after aging or one pass or even four passes of ECAP treatment, which is different from those of Ti−50.7%Ni alloy (Figs. 4 and 5). These results reveal that stable Ti_2Ni phase does not re-dissolve as the metastable phase Ti_3Ni_4 during medium temperature ECAP process or aging treatment. Aging and ECAP treatment do not affect the phase transformation behaviour of Ti−50%Ni alloy, since there is little change in Ni content of matrix.

3.4 Cycle tensile curves of TiNi alloys

Cycle tensile curves of Ti−50.9%Ni alloy after ECAP process are shown in Fig. 9. The remaining strain decreases along with the ECAP process. A completely recoverable strain of 6% is obtained after 8 passes of ECAP process (Fig. 9(e)). However, there are little changes in the tensile curves of Ti−50%Ni alloy after ECAP process, except for a decrease of yield stress, as shown in Fig. 10, which reveals the critic stress for re-orientation of martensite variants decreases along with the ECAP treatment. The shape recovery property was not measured.
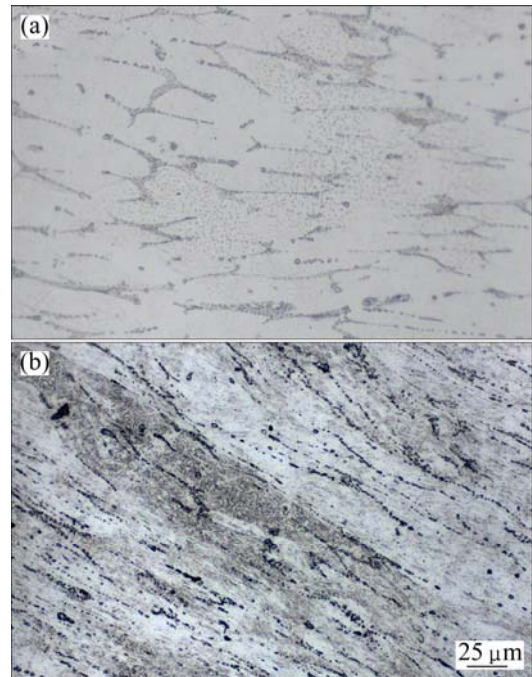
Fig. 7 TEM images showing microstructures of Ti−50%Ni alloy: (a) Solution treated; (b) 4 passes ECAPed
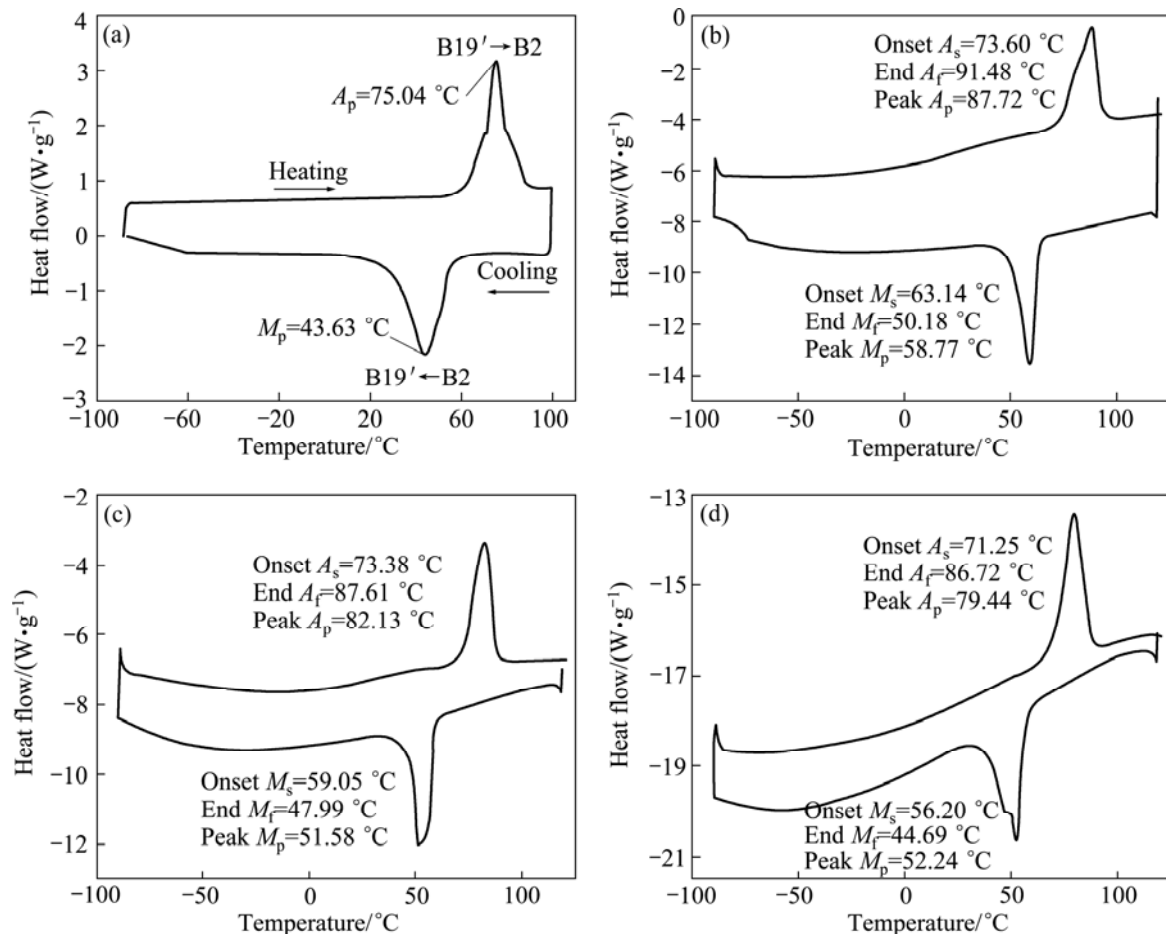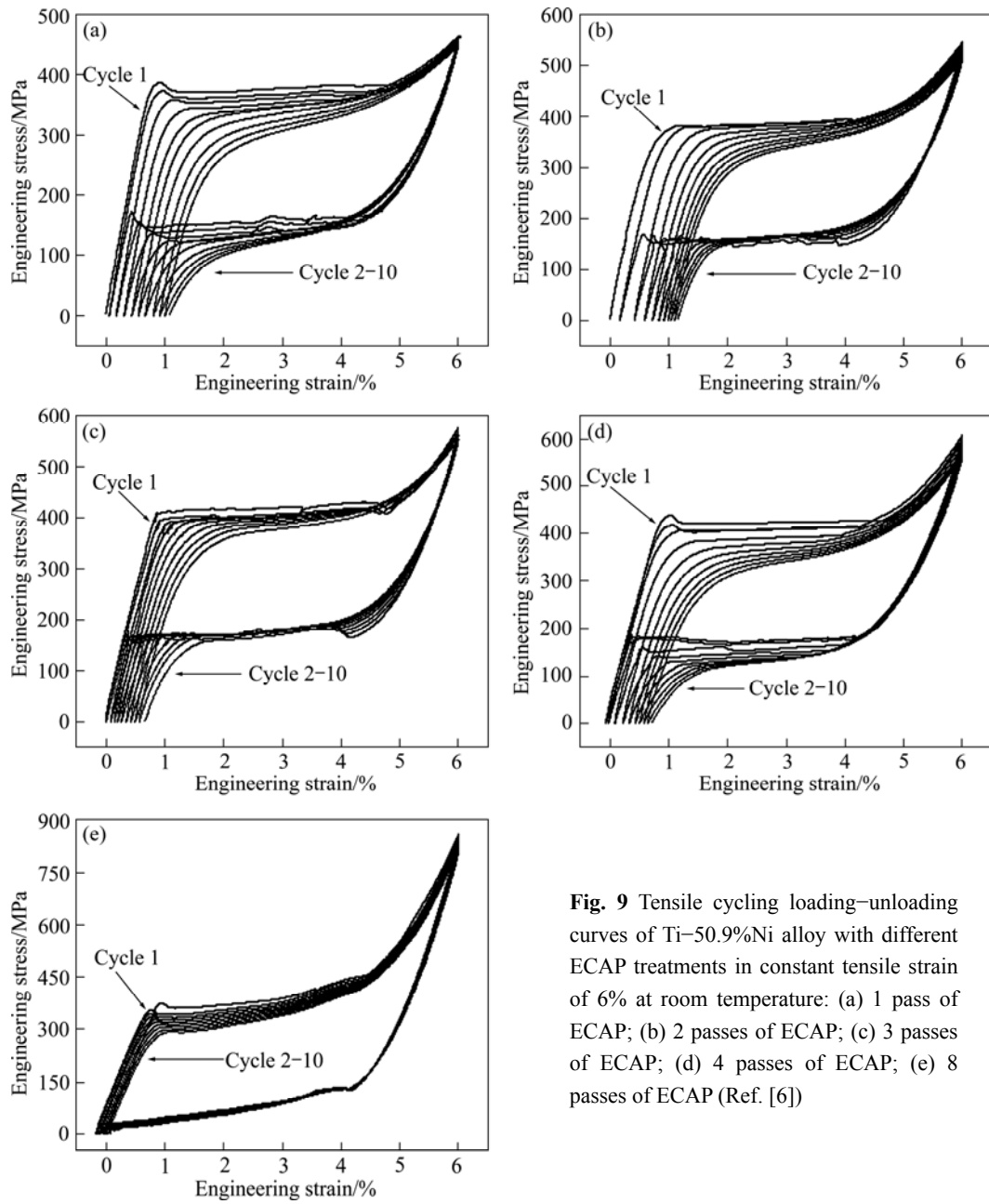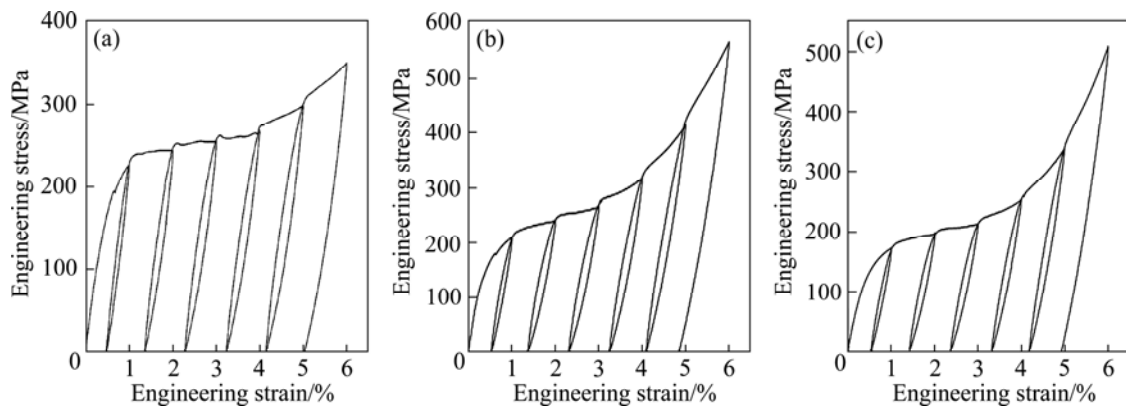
Fig. 8 DSC curves of Ti−50%Ni alloy: (a) Solution treated; (b) Solution treated+773 K aged for 30 min; (c) 1 pass ECAPed; (d) 4 passes ECAPed

Fig. 9 Tensile cycling loading−unloading curves of Ti−50.9%Ni alloy with different ECAP treatments in constant tensile strain of 6% at room temperature: (a) 1 pass of ECAP; (b) 2 passes of ECAP; (c) 3 passes of ECAP; (d) 4 passes of ECAP; (e) 8 passes of ECAP (Ref. [6])

Fig. 10 Mechanical behavior of solution treated and ECAP Ti-rich Ti−50% Ni alloy: (a) Solution treated; (b) 1 pass ECAPed; (c) 4 passes ECAPed

3.5 Effect of stable *α*-phase on Ti−Mo based shape memory alloy

Figure 11 shows the optical micrographs of solution treated and aged Ti−9.8Mo−3.9Nb−2V−3.1Al alloy. The solution-treated specimen is single *β* phase. As the aging temperature increases to 673 K, very fine precipitates appear in grains, as seen in Fig. 11(d). As the aging temperature increases to 773 K, lots of fine *α*-phase distribute inside the grains homogeneously [13], as shown in Fig. 11(f). After aging at 673 K for 30 min, some fine isothermal ω_{iso} phases with ellipsoidal-like precipitate shown in Fig. 12.

The strength of Ti−9.8Mo−3.9Nb−2V−3.1Al alloy increases sharply after ECAP process as shown in Fig. 13. However, the plasticity decreases dramatically, which can be attributed to the precipitation of *α*-phase and ω_{iso} phase, which may be enhanced during ECAP treatment.
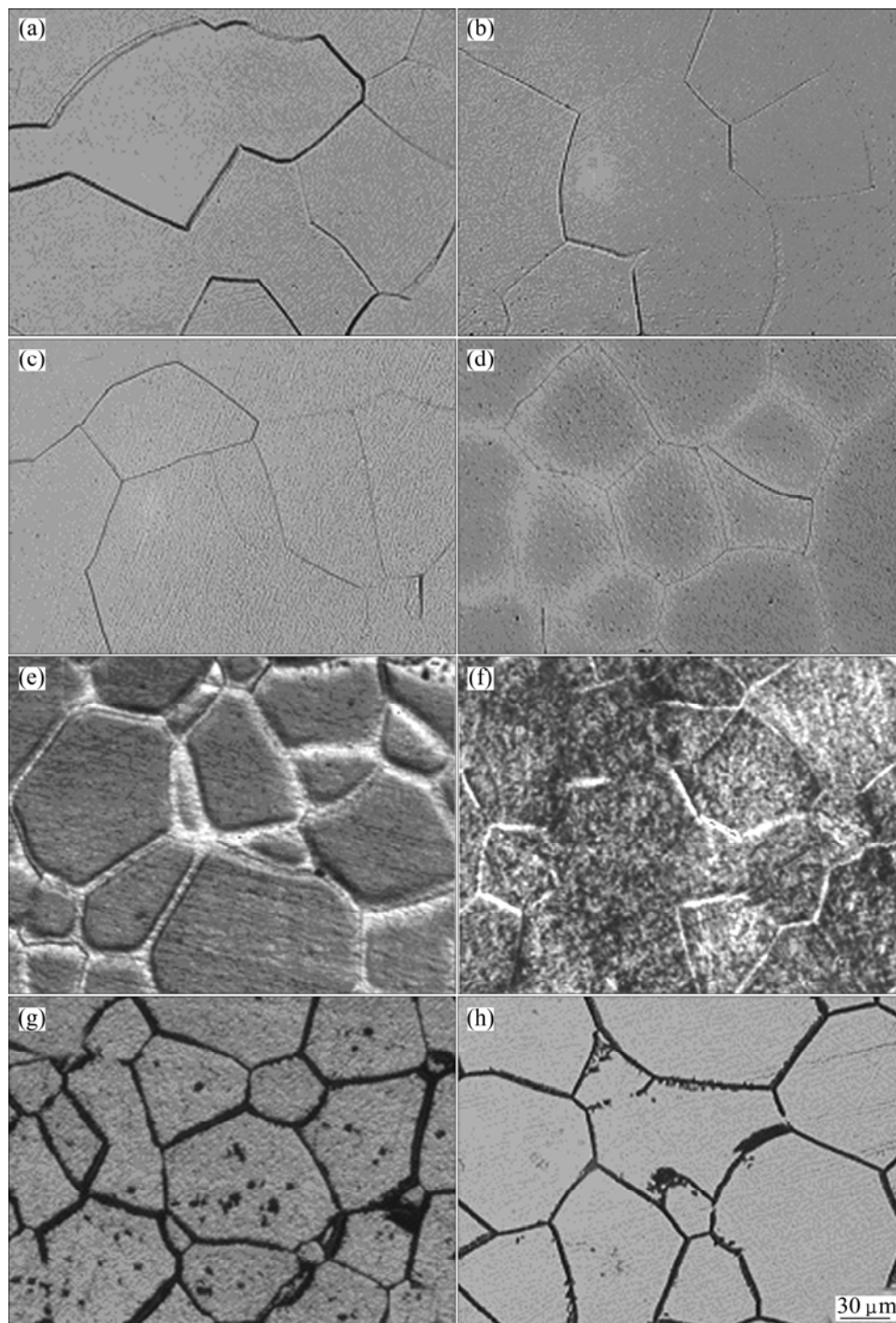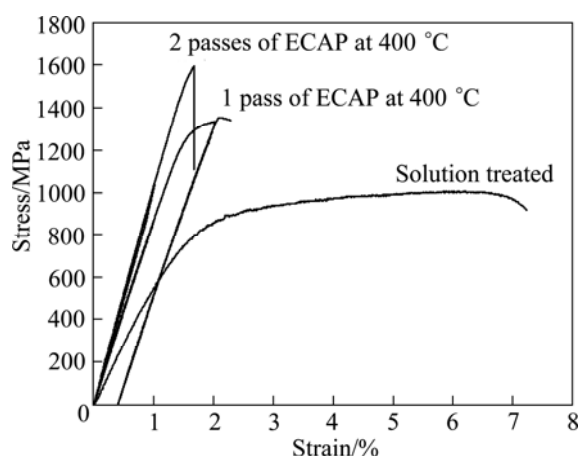

Fig. 11 Optical micrographs of solution treated Ti−9.8Mo−3.9Nb−2V−3.1Al metastable *β* titanium alloy (a) and aged at 573 K (b), 623 K (c), 673 K (d), 723 K (e), 773 K (f), 873 K (g) and 973 K (h)
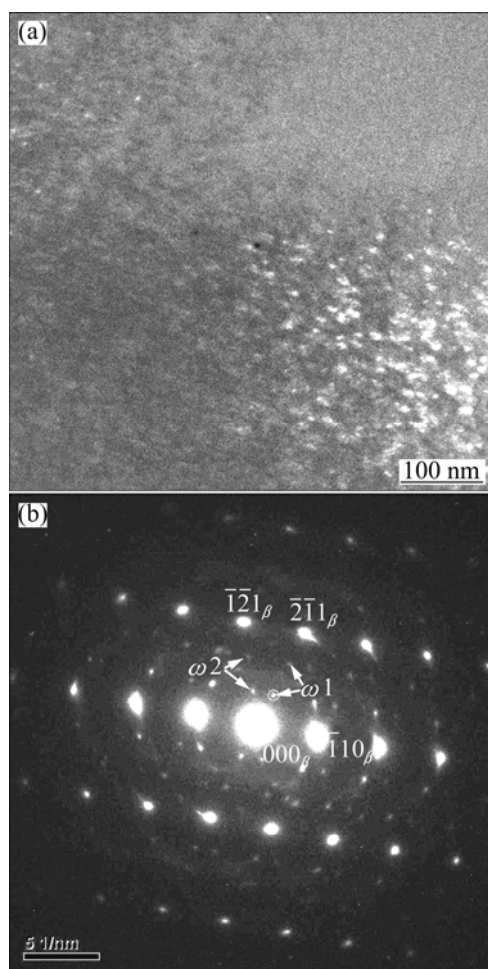
Fig. 12 Dark field image (a) of ω_{iso} phase in Ti−9.8Mo−3.9Nb−2V−3.1Al alloy aged at 673 K for 30 min and SAED pattern (b)

Fig. 13 Stress—strain curves of Ti−9.8Mo−3.9Nb−2V−3.1Al alloy solution treated and ECAP processed at 673 K

After two passes of ECAP process, the plasticity is lost completely. The effects of α-phase and ω_{iso} phase on the ECAP workability and mechanic property are still under investigation.

4 Conclusions

1) For Ni-rich TiNi alloy, there is re-dissolving of metastable Ti_3Ni_4 precipitates during ECAP process. After ECAP process, the B2→R transformation occurs in a broad temperature range. With increasing the passes, the martensitic transformation temperature decreases gradually.

2) For Ti−50%Ni alloy, equilibrium phase Ti_2Ni does not re-dissolve during ECAP process and there is no B2→R transformation on the DSC curves. The cycle tensile curves change little after four passes of ECAP process.

3) For Ti−9.8Mo−3.9Nb−2V−3.1Al alloy, the plasticity decreases dramatically after one pass of ECAP at 673 K, because of dislocation accumulation and the ω_{iso} phase and α-phase. After two passes of ECAP processes, the plasticity is lost completely.

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第二相对等径弯角挤压处理 TiNi 合金和 Ti−Mo 基合金力学性能及马氏体转变的影响

宋 杰[1]，王立明[2]，张效宁[1]，孙小刚[1]，江 鸿[1]，
范志国[1]，谢超英[1]，吴明雄[3]

1. 上海交通大学 材料科学与工程学院，金属基复合材料国家重点实验室，上海 200240；
2. 江苏法尔胜有限公司，江阴 214400；
3. 爱德华兹生命科学公司，欧文 加利福尼亚州 92614，美国

摘 要：借助 ECAP 技术对 TiNi 合金和 Ti−Mo 基记忆合金在 673~773 K 进行挤压处理，挤压路径为 B_c，以获得超细晶组织，从而增强母相的强度，改进材料的性能。研究热力学稳定相及亚稳相对这两种合金的力学性能和马氏体转变的影响。结果表明，对于富钛 TiNi 合金，热力学稳定相 Ti_2Ni 对马氏体转变及超弹性没有影响，而稳定相 α 相则可造成 Ti−Mo−Nb−V−Al 塑性降低。亚稳 Ti_3Ni_4 相对富镍 TiNi 合金的 R 相变、马氏体转变及超弹性有很大影响。并对第二相对 TiNi 合金和 Ti−Mo 基合金力学性能、马氏体转变的影响进行分析。

关键词：镍钛合金；钛钼基形状记忆合金；等径弯角挤压；第二相；力学性能

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