Effect of Pre-cold Rolling-Induced Twins and Subsequent Precipitated ω -Phase on Mechanical Properties in a β -Type Ti–Mo Alloy

Li Xiang¹ · Xiao-Hua Min¹ · Xin Ji² · Satoshi Emura² · Cong-Qian Cheng¹ · Koichi Tsuchiya²

Received: 17 May 2017 / Revised: 19 June 2017 / Published online: 15 December 2017
© The Chinese Society for Metals and Springer-Verlag GmbH Germany, part of Springer Nature 2017

Abstract

This paper reported an effectiveness of pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins combined with subsequent isothermal ω -phase formation for enhancement of uniform elongation in a β -type Ti–15Mo alloy with high yield strength level. Mechanical $\{332\} \langle 113 \rangle$ twins were induced by cold rolling with an thickness reduction of 5%, which had little effect on ω -phase precipitation after aging at 573 K for 3.6 ks. Twinning after the cold rolling was further activated during tensile deformation, even with the presence of isothermal ω -phase. This combination of twins and ω -phase enhanced uniform elongation from 0 to 9% at yield strength level of 890 MPa. The high yield strength was mainly dominated by dislocation slip due to the isothermal ω -phase formation, and early onset of plastic instability after yielding was hindered due to the pre-cold rolling-induced twins. Dynamic microstructural refinement was induced by further twinning activation during deformation, which resulted in high work hardening rate corresponding enhancement of uniform elongation.

Keywords β -Type titanium alloy · Pre-cold rolling · Twinning deformation · Isothermal ω -phase · Strength-ductility

1 Introduction

β -Type (body-centered-cubic) titanium alloys have found use in a wide range of aerospace, biomedical application and other industrial fields owing to their high specific strength and good corrosion resistance, ever since the first introduction of Ti–13V–11Cr–3Al (this and all other compositions are expressed in mass%) in the early 1950s [1–3]. One point of superiority for β -type titanium alloys is that they lend themselves to strengthen through control of their α -phase and ω -phase; however, their relatively low ductility is still a problem that affects their applications from an engineering aspect [4]. The other is that they have diversity of deformation mode, such as deformation-induced α'' martensitic transformation, $\{332\} \langle 113 \rangle$ twinning and dislocation slip; however, the mechanical

properties depend strongly on their deformation modes, which result in a relatively bad balance between yield strength and uniform elongation [4, 5].

A simultaneous improvement in both strength and ductility by means of second-phase precipitate or a single deformation mode appears to be a difficult proposition for β -type titanium alloys, but it is desirable for further widening the range of application, especially under complex loading conditions. Judging from a review of the literatures, it appears that such alloys have not received much attention in this regard, at least in comparison to face-centered-cubic Fe–Mn base steels [6–11]. Recently, several studies have shown the possibility of obtaining an improvement in both strength and ductility through a combination of deformation modes. Marteluer et al. [12] have reported that a complex combination of deformation-induced α'' - and ω -phase transformation, $\{332\} \langle 113 \rangle$ and $\{112\} \langle 111 \rangle$ twinning, leads to high tensile strength and large uniform elongation in β -type Ti–12Mo alloy. In previous work, we have reported that a combination of $\{332\} \langle 113 \rangle$ twinning and dislocation slip, caused by the solidification segregation of alloying elements [13], or by artificially inducing a heterogeneous distribution of alloying elements in β -type Ti–Mo base alloys [14, 15],

Available online at <http://link.springer.com/journal/40195>

✉ Xiao-Hua Min
minxiaohua@dlut.edu.cn

¹ School of Materials Science and Engineering, Dalian University of Technology, Dalian 116024, China

² Research Center for Structural Materials, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba 305-004, Japan

provides an effective approach to achieve large uniform elongation while keeping high yield strength.

Based on two typical features of β -type titanium alloys, namely $\{332\} \langle 113 \rangle$ twins corresponding to twinning-induced plasticity effect in Ti–Mo [16, 17], Ti–Nb–Ta–Zr [18], Ti–Nb [19], Ti–Cr–Sn [20] alloys, and isothermal ω -phase as a brittle phase [21, 22], we have also reported that an approach for improving the balance of strength and ductility through a combination of pre-tensile strain-induced $\{332\} \langle 1113 \rangle$ twins with subsequent precipitated ω -phase. The preexisting twins hinder the early onset of plastic instability (necking) after yielding, which is often caused by the presence of isothermal ω -phase, and the enhanced uniform elongation is attributable to further twinning activation during tensile deformation. In addition, the yield strength and uniform elongation characteristics depend strongly on degree of pre-tensile strain and aging condition. Since the $\{332\} \langle 113 \rangle$ twin was easily induced by cold rolling in β -type titanium alloys [23], a combination of pre-cold rolling-induced twins with isothermal ω -phase for improving their mechanical properties is considered to be more practical from an engineering aspect.

Previous research efforts have paid more attention to the effect of pre-cold rolling on the mechanical properties in TRIP/TWIP (transformation-induced plasticity/twinning-induced plasticity) steels [24–26] and magnesium alloys [27, 28]. Liu et al. [29] have reported that the cold rolling-induced ω -phase transformation is effective for changeable Young's modulus of β -type Ti–Cr alloys. However, the effect of pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins and subsequent precipitated ω -phase on mechanical properties is still unclear in β -type titanium alloys. Thus, the aim of this study is to examine the effect of cold rolling on $\{332\} \langle 113 \rangle$ twin formation, ω -phase precipitation and mechanical properties in a β -type Ti–15Mo alloy, and to further explore an effectiveness of pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins and subsequent precipitated ω -phase for improving the strength–ductility relationship.

2 Experimental Procedures

2.1 Materials Preparation

An approximately 1 kg ingot of Ti–15Mo alloy was prepared by cold crucible levitation melting under high-purity argon gas atmosphere. After solidification, the ingot was homogenized at 1273 K for 3.6 ks, hot-forged at 1273 K into a slab of dimensions 90 mm (l) $\times$ 40 mm (w) $\times$ 40 mm (t), and then hot-rolled at 1173 K into a plate of dimensions 290 mm (l) $\times$ 50 mm (w) $\times$ 10 mm (t) followed by air cooling. The plate was cut into 40 mm

(l) $\times$ 50 mm (w) $\times$ 10 mm (t) pieces, which were solution-treated at 1173 K for 3.6 ks followed by water quenching for achieving a single β -phase. The principal axes of the rolled plate are defined as RD (rolling direction), ND (normal direct to the rolling plane), and TD (transverse direction, which are orthogonal to both RD and ND). The solution-treated piece was subjected to pre-cold rolling with a thickness reduction of 5% along the RD for inducing $\{332\} \langle 113 \rangle$ twins. Subsequent aging at a temperature of 573 K for 3.6 ks was carried out followed by water quenching for precipitating ω -phase. The chemical composition of solution-treated sample was 15.3Mo–0.105O–0.012C–0.004N with a balance of Ti. Alloying element of Mo was detected by an inductively coupled plasma optical emission spectrometry. Oxygen, nitrogen, and carbon were detected by helium carrier fusion-infrared absorption method, helium carrier fusion-thermal conductivity method, and infrared absorption method after combustion in high-frequency induction furnace, respectively.

2.2 Mechanical Properties

Four types of samples were used as shown in a schematic figure (Fig. 1): solution-treated (ST), solution-treated and aged (STA) samples as a comparison [4], solution-treated and cold-rolled (STCR), solution-treated and cold-rolled and then aged (STCRA) samples. Tensile specimens with gage dimensions of 18 mm (l) $\times$ 4 mm (w) $\times$ 2 mm (t) were cut out from the four different samples by electric discharge machining along the RD and TD. Tensile testing was performed at room temperature in a tensile test machine Instron-5581 at a strain rate of $2.78 \times 10^{-4} \text{ s}^{-1}$. In addition, a tensometer was used for interrupted tensile testing to control tensile strain. Tensile force was applied parallel to the RD. Vickers hardness was measured with a load of 9.8 N at ten positions on the transverse plane of each sample, with their average taken as the value for that sample.

2.3 Microstructural Characterization

Aforementioned four types of samples before the tensile deformation were sanded with 320 grit sandpaper and then were polished with 9- and 3- μm diamond cloth, respectively, followed by colloidal silica and hydrogen peroxide (5:1 in volume) to obtain a smooth surface. They were etched for 60 s in a solution of distilled water, nitric acid, and hydrofluoric acid (100:3:2 by volume) for observations by optical microscopy (OM). Phase identification was made by X-ray diffraction (XRD) with an EMPYREN diffractometer using CuK_α radiation and operated at 40 kV and 30 mA with scan angle of 30° – 90° , step size of 0.02° , and scan speed $2^\circ/\text{min}$. Figure 2 shows the deformation

Fig. 1 Schematic diagram of **a** ST, **b** STA, **c** STCR, **d** STCRA samples. Black dots represent the ω -phase after aging, and black lines indicate the twins induced by pre-cold rolling

Fig. 2 Deformation microstructure of a half-cut 4% tensile-strained ST sample. Optical micrographs are taken at four different positions (black rectangles) along tensile direction from center to edge for analyzing the area fraction of twins. The horizontal direction is parallel to the tensile direction

microstructure of a half-cut 4% tensile-strained ST sample. For STCR sample and 4% plastic tensile-strained STCRA sample, optical micrographs were taken at four different positions along tensile direction from center to edge of gauge section, respectively. These optical micrographs were analyzed by using the analysis software ImageJ, and area fraction of twins was obtainable based on the image analysis. Deformation microstructures were observed with a field-emission scanning electron microscope (JSM-7001F, JEOL) equipped with an orientation imaging system for conducting the electron backscattered diffraction

(EBSD) analysis at a step size of 1.0 μm and EDAX-OIM software. Transmission electron microscopy (TEM) was performed with a JEOL-2100 system operating at 200 kV. Thin foils were prepared with a twin-jet electropolisher in a solution of methanol, 1-butanol, and perchloric acid (10:6:1 in volume) at 228 K under a voltage of 20 V. All analyses and observations were conducted on the rolling plane.

3 Results

Figure 3 shows the microstructures of ST, STA, STCR, and STCRA samples before tensile deformation. Both ST and STA samples were comprised by β -grains, and no significant difference was observed between them as reported in previous work [4]. Optical micrographs of STCR and STCRA samples exhibited the presence of $\{332\} \langle 113 \rangle$ twins inside β -grains without obvious difference. XRD profiles confirmed the existence of β -phase for each sample as shown in Fig. 4. Peaks of ω -phase was not detected in ST and STCR samples, but appeared in STA and STCRA samples, although they were very weak. TEM dark-field image (Fig. 5a) and selected area electron

Fig. 3 Optical micrographs for **a** ST, **b** STA, **c** STCR, **d** STCRA samples. The rolling plane is observed, and the horizontal direction is parallel to the RD

Fig. 4 XRD profiles for **a** ST, **b** STA, **c** STCR, **d** STCRA samples. The rolling plane is analyzed

diffraction pattern (Fig. 5b) confirmed the formation of isothermal ω -phase in the STA sample, and the $\{332\} \langle 113 \rangle$ twins can be clearly seen in the EBSD inverse pole figure (Fig. 5c) and corresponding boundaries map (Fig. 5d) of STCRA sample. Note that the coincidence site lattice (CSL) of a $\{332\} \langle 113 \rangle$ twin is identified as

a $\Sigma 11$ boundary, which corresponds to a misorientation of 50.5° around the $\langle 110 \rangle$ direction [30]. Thus, the presence of $\{332\} \langle 113 \rangle$ twins was determined by the red lines in the twin boundary map. Isothermal ω -phase formation is accompanied by a decrease in lattice parameter of β -phase as a result of enrichment of alloying elements in the matrix [31]. In addition, the presence of isothermal ω -phase can cause an increase in the hardness [31]. Table 1 lists the changes in β -phase lattice parameter and Vickers hardness of ST, STA, STCR, and STCRA samples. The β -phase lattice parameter was almost the same between ST and STCR samples, while it decreased after aging at 573 K for 3.6 ks. On the other hand, the Vickers hardness of ST and STCR samples exhibited a similar increase in tendency after aging. Based on these results, it implied that the preexisting twins induced by cold rolling with a thickness reduction of 5% had little effect on isothermal ω -phase formation after aging at 573 K for 3.6 ks.

Figure 6a shows the nominal stress–strain curves of ST, STA, STCR, and STCRA samples, and their tensile properties such as yield strength (YS), tensile strength (TS), uniform elongation (uEL), and total elongation (tEL) are also summarized in Table 1. The yield strength corresponds to the proof stress of 0.2% plastic deformation, and the uniform elongation is defined as the plastic strain of tensile strength. ST and STCR samples exhibited low yield strength (439 and 607 MPa, respectively) and large

Fig. 5 **a** TEM dark-field image and **b** selected area electron diffraction pattern for STA sample. Bright area in **a** is isothermal ω -phase and dark area is β -matrix. Zone axis is parallel to $[011]_{\beta}$. **c** EBSD inverse pole figure and **d** corresponding boundaries map for STCRA sample. Red and gray lines in **d** are $\{332\} \langle 113 \rangle$ twin and grain boundaries, respectively

Table 1 Lattice parameter of β -phase (a_{β}), Vickers hardness and tensile properties for yield strength (YS), tensile strength (TS), uniform elongation (uEL), total elongation (tEL) of Ti–15Mo alloy for ST, STA, STCR, and STCRA samples

	a_{β} (nm)	Vickers hardness (HV)	YS (MPa)	TS (MPa)	uEL (%)	tEL (%)
ST	0.3258 ± 0.0001	220 ± 7	439	714	23	49
STA	0.3255 ± 0.0001	324 ± 15	985	985	0	17
STCR	0.3258 ± 0.0001	233 ± 5	607	759	20	33
STCRA	0.3254 ± 0.0001	331 ± 8	890	1000	9	18

uniform elongation (27 and 20%, respectively), while STA sample exhibited high yield strength (985 MPa) with negligible uniform elongation (0%), despite a large total elongation (17%). On the other hand, STCRA sample provided a good balance between yield strength (890 MPa) and uniform elongation (9%), and its tensile strength (1000 MPa) was slightly higher than that (985 MPa) of STA sample. Figure 6b shows their curves of true stress–

strain and work hardening rate. The necking phenomenon is known to explain plastic instability [32, 33], indicating that it is initiated when work hardening rate is equal to true stress. Therefore, uniform elongation is improved if work hardening rate can be increased. Note that STA sample exhibited high yield strength but fast necking after yielding (Fig. 6a). For a comparison, the true stress–strain and corresponding work hardening rate of STA sample were

Fig. 6 **a** Nominal stress–strain curves, **b** true stress (σ)–true strain (ε) curves and work hardening rate ($d\sigma/d\varepsilon$) curves, **c** correlation between work hardening rate and true stress of the ST, STA, STCR, and STCRA samples

calculated by assuming a 5% uniform elongation. As can be seen, the initial work hardening rate of each sample was extremely high but decreased rapidly. STCRA sample exhibited larger work hardening rate compared to STA sample, which enhanced the uniform elongation while keeping high strength. Figure 6c shows the correlation between work hardening rate and true stress. The necking that occurred in both ST and STCR samples exhibited approximately the same work hardening rate around a true stress of 900 MPa. STCRA sample had considerable work hardening behavior during plastic deformation, in which the true stress exceeded 1070 MPa.

Figure 7 shows optical micrographs of fractured ST, STA, STCR and STCRA samples. The deformation microstructures comprised by β -grains, which were fragmented by the plate-like $\{332\} \langle 113 \rangle$ twins, were observed in fractured ST, STCR, and STCRA samples. It was difficult to distinguish the difference among three samples due to the twin and matrix regions being heavily deformed. In addition, the twins in fractured STCR and STCRA samples became dense, while they were not detected very well compared to those samples before deformation (Fig. 3c, d). In fractured STA sample, the line-like slip traces were observed within β -grains instead of $\{332\} \langle 113 \rangle$ twins, indicating that its deformation mode

Fig. 7 Optical micrographs of the fractured **a** ST, **b** STA, **c** STCR, **d** STCRA samples

was dominated only by the dislocation slip. Microstructures of STCR and 4% tensile-strained STCRA samples were observed by EBSD as shown in the inverse pole figure maps (Fig. 8a, c). Corresponding twin boundary maps (Fig. 8b, d) indicated that the plate-like features were identified as $\{332\} \langle 113 \rangle$ twins. The twins in 4% tensile-strained STCRA sample became denser than that of STCR sample, even though considering some degree of grain orientation dependence on the twin formation.

4 Discussion

In this study, we examined the effectiveness of a combination of pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins and subsequent precipitated ω -phase for enhancing uniform elongation in a β -type Ti-15Mo alloy with high yield strength. Figure 9 shows the uniform elongation as a function of yield strength for STCR and STCRA samples

Fig. 9 Uniform elongation as a function of yield strength for STCR (solid circle) and STCRA (solid triangle) samples along with those of ST and STA samples and the previous data [5] (open circles)

Fig. 8 EBSD maps of microstructures for **a, b** the STCR sample, **c, d** the 4% tensile-strained STCRA sample. **a, c** inverse pole figures for the RD, **b, d** boundaries delineated by red lines represent the $\{332\} \langle 113 \rangle$ twins. The rolling plane is observed, and the horizontal direction is parallel to the RD

(solid symbols), along with those of ST and STA samples, and the results from a combination of pre-tensile strain-induced twins and isothermal ω -phase as reported by our previous work (open circles) [5]. Thus, the preexisting twins induced by cold rolling as well as by tensile strain would act to enhance uniform elongation at high yield strength level. Here, two possible factors are considered to discuss the uniform elongation and yield strength characteristics below: One is pre-cold rolling-induced twins; the other is further twinning activation during deformation.

4.1 Pre-cold Rolling-Induced Twins

We first discuss the difference in tensile properties between ST and STA samples before moving on to discuss pre-cold rolling-induced twins. The difference is mainly due to a change in deformation mode from $\{332\} \langle 113 \rangle$ twinning to dislocation slip. At low β -phase stability, $\{332\} \langle 113 \rangle$ twinning leads to low yield strength and large uniform elongation; however, at high β -phase stability, dislocation slip leads to high yield strength and negligible uniform elongation [34]. Note that a Mo-poor isothermal ω -phase forms upon aging, which results in an enrichment of Mo in β -matrix, and as mentioned earlier, a decrease in β -phase lattice parameter. According to the relationship between the lattice parameter of β -phase and the Mo content in the Ti–Mo binary alloy [35], the obtained lattice parameters of β -phase (Table 1) in STA and STCRA samples implied that both samples had approximately the same Mo content in β -matrix, i.e., the same β -phase stability. Thus, STCRA sample as well as STA sample should be deformed by the dislocation slip.

Fine isothermal ω -phase for STA sample are easily cut by dislocation slip [36, 37], resulting in an extremely inhomogeneous slip distribution and a drop in flow stress. Thus, STA samples exhibited necking soon after yielding, even though a total elongation was 17%. However, the necking is obtainable to delay if work hardening rate can be increased as shown in Fig. 6. Thus, we suggest that uniform nucleation and multiplication of dislocations, together with strong obstacles to dislocation pile-up, are needed to attain a high degree of work hardening. Twin plates induced by cold rolling in STCRA sample were strong obstacles to dislocation movement. Although the fine isothermal ω -phase was also cut by dislocations similarly to STA sample, the pre-cold rolling-induced twins reduced the degree of inhomogeneous slip distribution or local stress concentration because of an increase in the area of boundaries. Thus, the high yield strength was mainly dominated by dislocation slip due to isothermal ω -phase formation, and an early onset of plastic instability after yielding was hindered due to preexisting twins in STCRA sample.

4.2 Further Twinning Activation During Deformation

From EBSD analysis (Fig. 8), we found that additional twins were formed in STCRA sample during tensile deformation. Twin density, which we obtained by dividing the length of twin boundaries by scanned area, was used as an indicator of twin quantity. As shown in Fig. 10, twin density of STCR and 4% tensile-strained STCRA samples (solid symbols) were plotted with those of results from a combination of pre-tensile strain and subsequent aging, reported by our previous work (open circles) [5]. Note that the 5.9% plastic strain was roughly calculated in terms of a thickness reduction of 5% by cold rolling in STCR sample. Thus, a total accumulated plastic strain is about 9.9% in the 4% tensile-strained STCRA sample. On the other hand, area fraction of twins in STCR and 4% tensile-strained STCRA samples were also obtained from OM observations. Optical micrographs were selected at four different positions along center line of the half-cut sample as shown in Fig. 2 for evaluating area fraction of twins. Figure 11 shows the typical optical micrographs and corresponding analyzed images. In the analyzed images, both grain boundaries and twins were selected as black lines, while β -matrix was shown in white. The area fraction of twin was obtained by subtracting the value of grain boundaries (1.5%), and they were 38.4 and 41.8% in STCR and 4% tensile-strained STCRA samples, respectively.

Therefore, the mechanical properties in STCRA sample were attributable to not only static microstructural refinement effect due to preexisting twins, but also dynamic microstructural refinement effect caused by further twinning activation during deformation. Influence of deformation twinning on work hardening has been widely discussed

Fig. 10 Plots of $\{332\} \langle 113 \rangle$ twin density versus plastic strain in STCR sample (solid circles) and 4% tensile-strained STCRA sample (solid triangle) along with those of previous data [5] (open circles)

Fig. 11 Deformation microstructures of **a, b** the STCR sample, **c, d** the 4% tensile-strained STCRA sample. **a, c** original optical micrographs, **b, d** corresponding analyzed images by software. The black lines represent the grain boundaries and twin boundaries, and the white area is β -matrix. The rolling plane is observed, and the horizontal direction is parallel to the RD

in references to dynamic Hall–Petch mechanism in TWIP steels [38, 39]. Twins cuts across the grains and thus form boundaries that act as barriers to dislocation motion, causing a decrease in the dislocation mean free path. In recent previous work [40], we found a linear relationship between the inverse square root of dislocation mean free path and true stress at an early stage of deformation in a TWIP β titanium alloy, and concluded that work hardening was due to dynamic microstructural refinement induced by $\{332\} \langle 113 \rangle$ twinning. Here, the enhanced uniform elongation apparent with STCRA sample was mainly attributable to further twinning deformation. However, the twinning activation could not be explained by β -phase stability because the stability of STCRA sample was similar to that of STA sample.

A possible explanation of the twinning activation in STCRA sample is related to $\{332\} \langle 113 \rangle$ twinning mechanism. Crocker [41] proposed that a $\{332\} \langle 113 \rangle$ twin is formed by a shear-shuffling mode. Recently, Tobe et al. [42] proposed that lattice modulation plays a key role in facilitating the formation of $\{332\} \langle 113 \rangle$ twin in terms of magnitude of shear and complexity of shuffle. On the other hand, Rusakov et al. [16] and Kawabata et al. [43] have reported a dislocation mode for $\{332\} \langle 113 \rangle$ twinning. A $\{332\} \langle 113 \rangle$ twin is formed as a result of successive slip of zonal dislocations with a small Burgers vector of

$a/22 \langle 113 \rangle$ in pairs of neighboring $\{332\}$ planes. Nevertheless, based on special features of $\{332\} \langle 113 \rangle$ twins, twin boundaries are thought to act not only as dislocation sources but also as places of preferred twin nucleation [16]. In addition, formation of $\{332\} \langle 113 \rangle$ twins becomes possible through intersections of twin–twin and twin–grain boundary to accommodate local stress concentrations and geometric constraint between neighboring grains [30]. Figure 12 shows the optical micrographs observed from the same area of STCRA sample before and after plastic deformation, respectively. Additional twins appeared in this sample with further tensile deformation as shown in Fig. 12b indicated by the white arrow.

In previous work [5], we reported that enhancement of uniform elongation depends strongly on degree of pre-tensile strain and aging conditions in a β -type Ti–15Mo alloy with high yield strength. Due to twinning polarity [44, 45], pre-induced twinning systems by cold rolling is different from those by tensile strain, which affects the further twinning activation during tensile deformation. Optimizing combination of pre-cold rolling and aging conditions is also needed to control the preexisting twins and subsequent twinning activation during deformation. In addition, due to preexisting twins induced by cold rolling, anisotropic effect on mechanical properties should be considered but small because of a small thickness reduction

Fig. 12 Optical micrographs of **a** STCRA, **b** 4% tensile-strained STCRA samples. The rolling plane is observed, and the horizontal direction is parallel to the tensile direction

used in this study, which benefits from an engineering aspect.

5 Conclusions

Through pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins, we examined the effect of preexisting twins on isothermal ω -phase formation and mechanical properties in a β -type Ti–15Mo alloy for improving its strength–ductility relationship. The main results can be drawn as follows:

1. Mechanical $\{332\} \langle 113 \rangle$ twins were induced by cold rolling with a thickness reduction of 5%, while they had little effect on ω -phase precipitation after aging at 573 K for 3.6 ks. Compared to preexisting twins, the twins became denser during further tensile deformation, even with the presence of isothermal ω -phase.
2. A combination of pre-cold rolling-induced $\{332\} \langle 113 \rangle$ twins and subsequent precipitated isothermal ω -phase was confirmed to be effective for enhancing uniform elongation from 0 to 9% in this Mo-bearing alloy at high yield strength level of 890 MPa.
3. The high yield strength was mainly dominated by the precipitation strengthening of isothermal ω -phase, and the enhanced uniform elongation was attributable to both static microstructural refinement induced by preexisting twins and dynamic microstructural refinement due to twinning activation during tensile deformation.

Acknowledgements This work was financially supported by the National Natural Science Foundation of China (Grant No. 51471040).

The authors would like to thank Prof. J. Zhao and Mr. S.H. Xue of Dalian University of Technology for their assistance.

References

- [1] R.R. Boyer, R.D. Briggs, *J. Mater. Eng. Perform.* **14**, 681 (2005)
- [2] R.R. Boyer, *Adv. Perform. Mater.* **2**, 349 (1995)
- [3] K. Wang, *Mater. Sci. Eng. A* **213**, 134 (1996)
- [4] X.H. Min, K. Tsuzaki, S. Emura, K. Tsuchiya, *Philos. Mag. Lett.* **92**, 726 (2012)
- [5] X.H. Min, S. Emura, L. Zhang, K. Tsuzaki, K. Tsuchiya, *Mater. Sci. Eng. A* **646**, 279 (2015)
- [6] H.N. Han, C.S. Oh, G. Kim, O. Kwon, *Mater. Sci. Eng. A* **499**, 462 (2009)
- [7] D.R. Steinmetz, T. Japel, B. Wietbrock, P. Eisenlohr, I.G. Urrutia, A.S. Akbari, T. Hickel, F. Roters, D. Raabe, *Acta Mater.* **61**(494), 494 (2013)
- [8] K. Jeong, J.E. Jin, Y.S. Jung, S. Kang, Y.K. Lee, *Acta Mater.* **61**, 3399 (2013)
- [9] B. Mahato, S.K. Shee, T. Sahu, S.G. Chowdhury, P. Sahu, D.A. Porter, L.P. Karjalainen, *Acta Mater.* **86**, 69 (2015)
- [10] H.K. Zhang, Z.J. Zhang, Z.F. Zhang, *Mater. Sci. Eng. A* **622**, 184 (2015)
- [11] B.B. He, H.W. Luo, M.X. Huang, *Int. J. Plast.* **78**, 173 (2016)
- [12] M. Marteleur, F. Sun, T. Gloriant, P. Vermaut, P.J. Jacques, F. Prima, *Scr. Mater.* **66**, 749 (2012)
- [13] X.H. Min, K. Tsuzaki, S. Emura, K. Tsuchiya, *Mater. Sci. Eng. A* **554**, 53 (2012)
- [14] G.U. Ivan, C.L. Li, S. Emura, X.H. Min, K. Tsuchiya, *Sci. Techno. Adv. Mater.* **17**, 220 (2016)
- [15] X.H. Min, S. Emura, F.Q. Meng, G.B. Mi, K. Tsuchiya, *Scr. Mater.* **102**, 79 (2015)
- [16] G.M. Rusakov, A.V. Litvinov, V.S. Litvinov, *Met. Sci. Heat Treat.* **48**, 244 (2006)
- [17] J.Y. Zhang, J.S. Li, Z. Chen, Q.K. Meng, F. Sun, B.L. Shen, *J. Alloys Compd.* **699**, 775 (2017)
- [18] M.J. Lai, C.C. Tasan, D. Raabe, *Acta Mater.* **111**, 173 (2016)
- [19] H.Y. Zhan, G. Wang, D. Kent, M. Dargusch, *Acta Mater.* **105**, 104 (2016)
- [20] C. Brozek, F. Sun, P. Vermaut, Y. Millet, A. Lenain, D. Embury, P.J. Jacques, F. Prima, *Scr. Mater.* **114**, 60 (2016)

- [21] S. Banerjee, R. Tewari, G.K. Dey, *Int. J. Mater. Res.* **97**, 963 (2006)
- [22] S. Dubinskiy, A. Korotitskiy, S. Prokoshkin, V. Brailovski, *Mater. Lett.* **168**, 155 (2016)
- [23] Y. Wang, J. Zhao, S.J. Dai, F. Chen, X.Q. Yu, Y.F. Zhang, *J. Mech. Behav. Biomed. Mater.* **27**, 33 (2013)
- [24] Y.F. Shen, C.H. Qiu, L. Wang, X. Sun, X.M. Zhao, L. Zuo, *Mater. Sci. Eng. A* **561**, 329 (2013)
- [25] N.K. Tewary, S.K. Ghosh, S. Bera, D. Chakrabarti, S. Chatterjee, *Mater. Sci. Eng. A* **615**, 405 (2014)
- [26] P. Kusakin, A. Belyakov, C. Haase, R. Kaibyshev, D.A. Molodov, *Mater. Sci. Eng. A* **617**, 52 (2014)
- [27] R.G. Li, R.L. Xin, A. Chapuis, Q. Liu, G.Y. Fu, L. Zong, Y.M. Yu, B.T. Guo, S.G. Guo, *Mater. Charact.* **112**, 81 (2016)
- [28] D. Wu, W.N. Tang, R.S. Chen, E.H. Han, *Trans. Nonferrous Met. Soc. China* **23**, 301 (2013)
- [29] H.H. Liu, M. Niinomi, M. Nakai, K. Cho, *Acta Mater.* **106**, 162 (2016)
- [30] X.H. Min, K. Tsuzaki, S. Emura, T. Sawaguchi, S. Li, K. Tsuchiya, *Mater. Sci. Eng. A* **579**, 164 (2013)
- [31] B.S. Hickman, *J. Mater. Sci.* **4**, 554 (1969)
- [32] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino, *Scr. Mater.* **47**, 893 (2002)
- [33] N. Takata, Y. Ohtake, K. Kita, K. Kitagawa, N. Tsuji, *Scr. Mater.* **60**, 590 (2009)
- [34] X.H. Min, S. Emura, N. Sekido, T. Nishimura, K. Tsuchiya, K. Tsuzaki, *Mater. Sci. Eng. A* **527**, 2693 (2010)
- [35] J.L. Murray (ed.), *Phase Diagram of Binary Titanium Alloys* (ASTM International, Ohio, 1987)
- [36] S. Hanada, M. Ozeki, O. Izumi, *Metall. Mater. Trans. A* **16**, 789 (1985)
- [37] S. Banerjee, U.M. Naik, *Acta Mater.* **44**, 3667 (1996)
- [38] R. Ueji, N. Tsuchida, D. Terada, N. Tsuji, Y. Tanaka, A. Takemura, K. Kunishige, *Scr. Mater.* **59**, 963 (2008)
- [39] H. Idrissi, K. Renard, D. Schryvers, P.J. Jacques, *Scr. Mater.* **63**, 961 (2010)
- [40] X.H. Min, X.J. Chen, S. Emura, K. Tsuchiya, *Scr. Mater.* **69**, 393 (2013)
- [41] A.G. Crocker, *Acta Metall.* **10**, 113 (1962)
- [42] H. Tobe, H.Y. Kim, T. Inamura, H. Hosoda, S. Miyazaki, *Acta Mater.* **64**, 345 (2014)
- [43] T. Kawabata, S. Kawasaki, O. Izumi, *Acta Mater.* **46**, 2705 (1998)
- [44] S. Godet, L. Jiang, A.A. Luo, J.J. Jonas, *Scr. Mater.* **55**, 1055 (2006)
- [45] E. Bertrand, P. Castany, I. Peron, T. Gloriant, *Scr. Mater.* **64**, 1110 (2011)