Effect of Deformation-Induced ω Phase on the Mechanical Properties of Metastable β -Type Ti-V Alloys

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A series of metastable β -type binary Ti-(18–22)V alloys were prepared to investigate the effect of deformation-induced products (deformation-induced ω phase transformation and mechanical twinning) on the mechanical properties of metastable β -type titanium alloys. The microstructures, Young's moduli, and tensile properties of the alloys were systemically examined.

Ti-(18–20)V alloys subjected to solution treatment comprise a β phase and a small amount of athermal ω phase, while Ti-22V alloy subjected to solution treatment consists of a single β phase. Ti-(18–20)V alloys subjected to solution treatment exhibit relatively low Young's moduli and low tensile strengths as compared to cold-rolled specimens. Both deformation-induced ω phase transformation and $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning occur in all of the alloys during cold rolling. The occurrences of $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning and deformation-induced ω phase transformation are dependent on the β stability of the alloys. After cold rolling, all of the alloys comprise a β phase and an ω phase. The Young's moduli of Ti-(18–22)V alloys increase because of the formation of a deformation-induced ω phase during cold rolling. The significant increase in tensile strength is attributed to the combined effect of the deformation-induced ω phase transformation and work-hardening during cold rolling. [doi:10.2320/matertrans.M2012116]

(Received March 26, 2012; Accepted May 16, 2012; Published July 11, 2012)

Keywords: beta-type alloys, deformation-induced omega phase transformation, mechanical twinning, Young's modulus, tensile properties

1. Introduction

Non-equilibrium phases, such as α' (hcp-structured martensite), α'' (orthorhombic-structured martensite), or ω (simple hexagonal structure phase), can appear in certain metastable β -type titanium alloys during deformation.^{1,2} It would appear that the deformation-induced products are related to the amount of β stabilizing element that is present, i.e., the stability of the β phase. As the amount of β stabilizer increases, the stability of the β phase increases so that such products change from deformation-induced martensites (α' and α'') to a deformation-induced ω phase and mechanical twin.³ Over the past few decades, deformation-induced martensites and their effect on the mechanical properties of titanium alloys have been extensively investigated.^{4–10} Zhao *et al.* reported that deformation-induced α' transformation occurs in Ti-30Zr-4Cr alloy and result in decreasing of Young's modulus.⁵ Kim *et al.* reported that stress-induced α'' transformation can take place in the metastable β region in Ti-Nb-Si alloys, and result in pseudoelasticity.⁶ Matsumoto *et al.* released that stress-induced α'' phase is formed by cold rolling in Ti-35Nb-4Sn alloy, and the Young's modulus of the alloy decreases after cold rolling, while the tensile strength increases.⁸ Additionally, there are some reports that have reported on the phenomenon of deformation-induced ω phase transformation in metastable β -type alloys. Kuan *et al.*¹¹ observed that the ω phase was stress-induced during tensile deformation of quenched Ti-16 and Ti-20 mass% V single crystals. In addition, deformation-induced ω phase transformation was also found in metastable β -type Ti-Cr, Ti-Fe and Ti-Mo alloys.^{12,13} Nevertheless, there is very little literature available that has focused on the effect of deformation-induced ω phase transformation on the

mechanical properties, such as Young's modulus and tensile properties, for example.

As mentioned above, the type of deformation-induced products are related to the stability of the β phase.³ There has a possibility that both of deformation-induced martensitic transformation and deformation-induced ω phase transformation occur simultaneously because they are competitive in a certain range of chemical composition.⁵ In this case, it is difficult to clarify the effects of deformation-induced martensitic transformation and deformation-induced ω phase transformation on the mechanical properties separately. It is necessary to eliminate the effect of the deformation-induced martensite on the mechanical properties to clarify the effect of the deformation-induced ω phase on the mechanical properties. Therefore, a relatively large amount of β stabilizer should be added to obtain a relatively high stability in the β phase to avoid deformation-induced martensitic transformation. Vanadium (V) is an effective β stabilizer for designing β -type alloys, and Ti-V alloys have a large composition range capable of producing deformation-induced ω phase transformation.^{11,13} According to the previous reports, a composition range of 18 to 22 mass% V is expected to produce deformation-induced ω phase transformation for the binary Ti-V alloys.^{11,13} Thus, in the present work, the effect of deformation-induced ω phase on Young's modulus and tensile properties of β -type titanium alloys were systematically investigated using the binary Ti-V alloys.

2. Materials and Methods

2.1 Material preparation

A series of binary Ti- x V ($x = 18, 20$ and 22 mass%) alloys were prepared by the following procedure. Appropriate amounts of high-purity sponge Ti (99.7%) and lumps of V (99.7%) were mixed together. The mixtures were then arc melted with a non-consumable tungsten electrode under a

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high-purity argon (Ar) atmosphere. The ingots were inverted and remelted at least six times to ensure compositional homogeneity. The arc-melted ingots of the Ti-*x*V alloys were homogenized at 1373 K for 21.6 ks in an Ar atmosphere, after which they were subjected to water quenching. The alloys were then hot rolled into plates with a reduction ratio of 70% at 1273 K in the same atmosphere and subjected to air cooling. Then, the hot-rolled specimens were subjected to solution treatment at 1123 K for 3.6 ks under vacuum conditions, followed by water quenching. Finally, to introduce deformation-induced products into the Ti-(18–22)V alloys, the solution-treated samples were cold rolled with a reduction ratio of 10% at room temperature. The solution-treated and cold-rolled specimens are labeled ST and CR, respectively. More particularly, from here on they are referred to as Ti-18V-ST, Ti-20V-ST and Ti-22V-ST, and Ti-18V-CR, Ti-20V-CR and Ti-22V-CR, respectively.

2.2 Microstructural analysis

The phase constitutions were identified by X-ray diffraction (XRD) analysis using a Bruker D8 Discover two-dimensional X-ray diffractometer with Cu-K α radiation at an accelerating voltage of 40 kV and a current of 40 mA. The microstructures were examined by optical microscopy (OM; Olympus BX51), electron backscatter diffraction (EBSD; Quanta 200 3D SEM-TSL), and transmission electron microscopy (TEM; JEOL JEM-2000EX). For OM and EBSD observations, the specimens were mechanically polished using SiC waterproof papers of up to #2400 grit and a colloidal SiO₂ suspension. The mirror-polished specimens were then etched with mixed aqueous solutions of hydrofluoric acid and nitric acid. The specimens for TEM were prepared by mechanical polishing and ion milling. Specifically, the samples were first mechanically polished to a thickness of approximately 50 μ m, after which they were dimpled with a phosphor bronze ring to a thickness of around 15 μ m. Finally, the specimens were ion milled to a thin foil. TEM observations were conducted at an accelerating voltage of 200 kV.

2.3 Evaluation of mechanical properties

The mechanical properties of the prepared alloys were evaluated by performing Young's modulus measurements and tensile tests.

Specimens, of size 40 mm $\times$ 10 mm $\times$ 1.5 mm, were cut from ST and CR plates for Young's modulus measurements, with their longitudinal direction parallel to the rolling direction. These specimens were polished using SiC waterproof papers of up to #2400 grit. The Young's moduli of the alloys were measured at room temperature in air using a free resonance method (Nippon Techno-Plus Co., Ltd. JE-RT3).

For the tensile tests, specimens with a thickness of 1.5 mm, width of 3 mm, and gage length of 13 mm were cut from the ST and CR plates. The longitudinal directions of these specimens were parallel to the rolling direction. The tensile test specimens were polished using SiC waterproof papers of up to #2400 grit. The tensile tests were performed at a crosshead speed of 8.33×10^{-6} m s⁻¹, at room temperature in air, using an Instron-type machine (Shimadzu AGS-20kNG). The Young's modulus measurements and tensile tests were performed in triplicate to minimize the experimental errors.

Fig. 1 XRD profiles of Ti-(18–22)V alloys subjected to solution treatment (ST) and cold rolling (CR).

3. Results and Discussion

3.1 Microstructures

Figure 1 shows the XRD profiles of the Ti-(18–22)V alloys subjected to solution treatment and cold rolling. In Ti-18V-ST, small peaks corresponding to the ω phase are detected in combination with the β phase. Only peaks corresponding to the β phase are detected in Ti-20V-ST and Ti-22V-ST, and no peaks corresponding to the ω phase are detected for these specimens. In contrast, after cold rolling, sharp peaks corresponding to the ω phase are detected in Ti-18V-CR. In addition, a weak peak of the ω phase is also found in Ti-20V-CR, while only a single β phase is detected in Ti-22V-CR.

Typical optical micrographs for Ti-(18–22)V alloys subjected to solution treatment and cold rolling are shown in Fig. 2. After solution treatment, all of the alloys consist of equiaxial grains, with an average grain diameter of around 300 μ m and without any precipitates (Figs. 2(a)–2(c)). In contrast, after cold rolling, apparently different optical microstructures are observed compared with those after solution treatment. Many straight bands are observed inside the equiaxial grains of the cold-rolled specimens, as shown in Figs. 2(a')–2(c'). The straight bands in Ti-18V alloy are very fine, while the number of straight bands is large. With an increase in V content, the width of the straight bands clearly increases and the number of straight bands decreases.

Figure 3 shows TEM images that indicate straight bands in Ti-18V-CR. Figures 3(a) and 3(b) show the respective bright field image and dark field image of the straight band observed in Ti-18V-CR. Analysis of the electron diffraction patterns obtained from the straight band boundary region indicates that the straight band observed in Ti-18V-CR is the result of $\{332\}_{\beta}\{113\}_{\beta}$ mechanical twinning (Figs. 3(c) and 3(d)). In addition, Fig. 3(c) clearly shows that the two variants of the ω phase in the twinning are different in density from each other. Specifically, the intensity of one ω variant is much stronger than that of the other. These results are consistent with previous work reported by Hanada *et al.*¹³⁾

Fig. 2 Optical micrographs of Ti-(18-22)V alloys subjected to solution treatment (ST) and cold rolling (CR): (a) Ti-18V-ST, (b) Ti-20V-ST, (c) Ti-22V-ST, (a') Ti-18V-CR, (b') Ti-20V-CR and (c') Ti-22V-CR.

Fig. 3 TEM observations of a mechanical twin in Ti-18V subjected to cold rolling (CR): (a) Bright field (BF) image of Ti-18V after CR; (b) dark field (DF) image of (a) taken by using the spot labeled by the solid circle in (c); (c) Electron diffraction pattern taken from the twin boundary that is labeled by the dashed circle in (a); (d) Key diagram of (c). DIFF patterns were taken with the beam direction parallel to $[110]_{\beta}$.

In Ti-20V-CR and Ti-22V-CR, mechanical twinning is not observed by TEM because the widths of the straight bands are too large. Therefore, EBSD analysis was performed to identify the wide straight bands. Figure 4 shows the EBSD

Fig. 4 EBSD maps of (a) and (b) Ti-20V alloy, and (c) and (d) Ti-22V alloy subjected to cold rolling (CR): (a) and (c): β IPF figures; (b) and (d): boundaries with a misorientation angle of 50.5° along the $\langle 110 \rangle_{\beta}$ direction corresponding to $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ twinning. $\Delta\theta = 5^\circ$: tolerance angle is 5° . IPF: inverse pole figure mapping.

results for Ti-20V-CR and Ti-22V-CR. Many straight bands, as was observed by the optical micrographs, are evident in the orientation image maps of Ti-20V-CR and Ti-22V-CR, as shown in Figs. 4(a) and 4(c). The position of the straight band boundaries in each of these orientation image maps match well with the $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ twin boundaries^{14,15} that are delineated by the red lines in Figs. 4(b) and 4(d). Thus, these straight bands in Ti-20V-CR and Ti-22V-CR are defined as $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning.

To summarize, $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning occurs in all Ti-(18-22)V alloys during cold rolling. In addition, with an increase in V content, the width of the twinning bands clearly increases and the number of twinning bands decreases. This indicates that the occurrence of $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning is dependent on the stability of the β phase. As the V content increases, the stability of the β phase increases and $\{332\}_{\beta}\langle 113 \rangle_{\beta}$ mechanical twinning is difficult to occur.

Figure 5 shows selected area electron diffraction patterns and dark field images of the Ti-(18-22)V alloys subjected to solution treatment and cold rolling. The electron diffraction patterns for the $[110]_{\beta}$ zone of Ti-18V-ST clearly show extra spots that correspond to the ω phase and spots that are derived from the β phase, thus suggesting that a certain amount of the athermal ω phase is formed in Ti-18V-ST during water quenching (Fig. 5(a)). The orientation relationships between the athermal ω phase and β matrix are $\{111\}_{\beta} // \{0001\}_{\omega}$ and $\langle 110 \rangle_{\beta} // \langle 11\bar{2}0 \rangle_{\omega}$. The intensities of the ω reflections in Ti-20V-ST are much weaker than those in Ti-18V-ST (Fig. 5(b)), indicating that the amount of athermal ω phase is decreased in comparison with that in Ti-18V-ST. As the V content increases further, the ω reflections in Ti-22V-ST disappear. The dark field images obtained from the ω reflections in Figs. 5(a) and 5(b) show the

Fig. 5 Selected area electron diffraction patterns and dark field images of Ti-(18–22)V alloys subjected to solution treatment (ST) and cold rolling (CR): (a) and (a'): Ti-18V-ST; (b) and (b'): Ti-20V-ST; (c) and (c'): Ti-22V-ST; (d) and (d'): Ti-18V-CR; (e) and (e'): Ti-20V-CR; and (f) and (f'): Ti-22V-CR. DIFF patterns were taken from the matrix with the beam direction parallel to $[110]_{\beta}$, and dark field images were taken from the diffraction patterns corresponding to the ω phase.

presence of a dispersion of fine ω particles in the β matrix of Ti-18V-ST and Ti-20V-ST, as shown in Figs. 5(a') and 5(b'), respectively. The size and number of ω particles decrease with an increase in V content such that no ω particles are found in the β matrix of Ti-22V-ST (Fig. 5(c')). These results indicate that the formation of the athermal ω phase during water quenching is suppressed by increasing the amount of β stabilizer, i.e., V.

In contrast, after cold rolling, the reflections of the ω phase in the matrix of Ti-18V-CR and Ti-20V-CR specimens (Figs. 5(d) and 5(e), respectively) are much sharper than those in Ti-18V-ST and Ti-20V-ST (Figs. 5(a) and 5(b)), respectively. The size and number of ω particles in the β matrix of Ti-18V-CR (Fig. 5(d')) and Ti-20V-CR (Fig. 5(e')) are greater than those in the β matrix of Ti-18V-ST and Ti-20V-ST (Figs. 5(a') and 5(b'), respectively), indicating that the amount of ω phase increases as a result of cold rolling. In addition, ω phases are also found in the matrix of Ti-22V-CR, as shown in Figs. 5(f) and 5(f'), suggesting that a considerable amount of ω phase is formed in Ti-22V-CR. An increase in the intensity of the ω phase in metastable β -type Ti alloys after deformation has been

observed by other researchers.^{1,2,16,17)} These findings confirm that deformation-induced ω phase transformation occurs in all Ti-(18–22)V alloys during cold rolling. The orientation relationships between the deformation-induced ω phase and β matrix are $\{\bar{1}11\}_{\beta} // \{0001\}_{\omega}$ and $\langle 110 \rangle_{\beta} // \langle 11\bar{2}0 \rangle_{\omega}$, which are the same as the orientation relationships between the athermal ω phase and β matrix. It is found that the size and number of ω particles decrease with an increase in V content, suggesting that deformation-induced ω phase transformation becomes difficult.

In summary, with an increase in V content, the total amount of the ω phase (athermal ω phase + deformation-induced ω phase) in the Ti-(18–22)V alloys decreases. The phase constitutions of Ti-(18–22)V alloys subjected to solution treatment and cold rolling are shown in Table 1.

3.2 Mechanical properties

3.2.1 Young's modulus

Figure 6 shows the Young's moduli of the Ti-(18–22)V alloys subjected to solution treatment and cold rolling. The Young's moduli of the alloys subjected to solution treatment first significantly decrease from 94 GPa in Ti-18V-ST to

Table 1 Summary of phase constitutions of Ti-(18–22)V alloys subjected to solution treatment and cold rolling.

Alloy	Subjected to solution treatment	Subjected to solution treatment and cold rolling
Ti-18V	β + athermal ω	β + athermal ω + deformation-induced ω
Ti-20V	β + athermal ω	β + athermal ω + deformation-induced ω
Ti-22V	β	β + deformation-induced ω

Fig. 6 Young's moduli of Ti-(18–22)V alloys subjected to solution treatment (ST) and cold rolling (CR).

76 GPa in Ti-20V-ST, after which they slightly decrease to 75 GPa in Ti-22V-ST. The microstructural analysis of Ti-(18–22)V alloys reveal that a small amount of the athermal ω phase exists in Ti-18V-ST and Ti-20V-ST. It is well known that the ω phase has a significant effect on the mechanical properties of Ti alloys¹⁸⁾ and that it is likely to increase the Young's modulus. In addition, the solid solution strengthening with V also has an effect on the Young's modulus.¹⁹⁾ Thus, the change in Young's modulus is a combined effect of the athermal ω phase and the solid solution strengthening. However, the athermal ω phase in the β matrix is the dominant factor in changing the Young's modulus. According to the microstructural analysis, as the V content increases, i.e., as we go through the range of Ti-18V to Ti-22V, the amount of the athermal ω phase first significantly decreases and then decreases only slightly. Therefore, the Young's moduli of Ti-(18–22)V alloys first decrease significantly and then decrease slightly.

The Young's moduli of the alloys subjected to cold rolling are increased compared to those subjected to solution treatment, and the biggest change in the Young's modulus is obtained in Ti-20V alloy. Generally, the change in Young's modulus of a Ti alloy is not sensitive to deformation.¹⁸⁾ However, the Young's modulus of Ti alloy is sensitive to the secondary phase or precipitates within the β matrix.¹⁸⁾ The results of microstructural analysis indicate that deformation-induced ω phase transformation occurs in all the alloys. Therefore, the increase in the Young's moduli of the alloys by cold rolling is attributed to the deformation-induced ω phase transformation that occurred during cold rolling. In addition, with an increase in V content, deformation-induced ω phase transformation becomes increasingly difficult. With

Fig. 7 Tensile properties of Ti-(18–22)V alloys subjected to solution treatment (ST) and cold rolling (CR).

an increase in the V content, the total amount of the ω phase (athermal ω phase + deformation-induced ω phase) in the Ti-(18–22)V alloys subjected to cold rolling decreases. Thus, the Young's moduli of Ti-(18–22)V alloys subjected to cold rolling decrease with an increase in the V content.

3.2.2 Tensile properties

Figure 7 shows the tensile properties of the Ti-(18–22)V alloys subjected to solution treatment and cold rolling. Ti-18V-ST shows low 0.2% proof stress and considerable work-hardening, resulting in excellent ductility. With an increase in V content, the 0.2% proof stress increases, while the ductility decreases. According to the results of the microstructural analysis, both $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning and deformation-induced ω phase transformation occur in all of the alloys. There is a small amount of athermal ω phase that exists in Ti-18V-ST and Ti-20V-ST. In general, deformation proceeds via $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning and deformation-induced ω phase transformation results in low 0.2% proof stress and good ductility. By increasing the β stability of the alloys, $\{332\}_{\beta}$ mechanical twinning and deformation-induced ω phase transformation become difficult. Thus, the triggering stress for the $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning and deformation-induced ω phase transformation increases with an increase in the V content. Additionally, the athermal ω phase seems to increase the 0.2% proof stress and decrease the ductility. Nevertheless, the effects of $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning and deformation-induced ω phase transformation are dominant. Therefore, the 0.2% proof stress of the alloys subjected to solution treatment increases with an increase in the V content, whereas the ductility of the alloys subjected to solution treatment decreases.

After cold rolling, the tensile strengths of all the alloys increase significantly, but the elongation decreases. More specifically, Ti-18V-CR shows the highest tensile strength and least elongation. The tensile strengths of Ti-20V-CR and Ti-22V-CR are lower than those of Ti-18V-CR, whereas elongations of Ti-20V-CR and Ti-22V-CR are greater than those of Ti-18V-CR. As mentioned previously, both deformation-induced ω phase transformation and $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning occur during cold rolling. The size and number of ω particles in the β matrix of Ti-18V-CR

are greater than those of Ti–20V–CR and Ti–22V–CR (Figs. 3(d')–3(f')). It is well known that the ω phase is a brittle phase in comparison to the β phase. Therefore, the numerous, coarse ω particles in Ti–18V–CR seem to effectively restrict the slip of dislocations that largely limit the capability of plastic deformation.²⁰⁾ Moreover, the extensive $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning that occurs in Ti–18V–CR during cold rolling produces considerable work-hardening, which is also expected to increase the tensile strength and worsen the ductility. Therefore, the strength of Ti–18V alloy is significantly increased by cold rolling, while the elongation drastically decreases. With an increase in V content, the amount of the ω phase and work-hardening decrease in the Ti–(18–22)V alloys subjected to cold rolling. Therefore, with an increase in the V content, the rate of increase of tensile strengths of Ti–(18–22)V alloys subjected to cold rolling decreases, whereas elongations are improved. These results indicate that the deformation-induced ω phase shows similar effects on the mechanical properties of the alloys as the isothermal ω phase and athermal ω phase. A coarse ω phase in the β matrix increases the tensile strength, but worsens the ductility.

4. Conclusions

Microstructures, Young's moduli, and tensile properties were systematically investigated for binary Ti–(18–22)V alloys subjected to solution treatment and cold rolling. The following conclusions were drawn on the basis of the results obtained:

(1) Ti–18V and Ti–20V alloys subjected to solution treatment comprise a β phase and a certain amount of athermal ω phase. The amount of ω athermal phase decreases with increasing content of the β stabilizer, V. Ti–22V alloy subjected to solution treatment consists of a single β phase.

(2) Deformation-induced ω phase transformation and $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning occur in all of the alloys during cold rolling. Thus, after cold rolling, all of the alloys comprise a β phase and an ω phase. The size and number of ω particles in the β matrix of Ti–(18–22)V alloys subjected to cold rolling decrease with an increase in V content. The occurrences of $\{332\}_{\beta}(113)_{\beta}$ mechanical twinning and deformation-induced ω -phase transformation are dependent on the β stability of the alloys.

(3) The Young's moduli of Ti–(18–22)V alloys clearly increase as a result of the formation of a deformation-induced ω phase during cold rolling. In addition, the tensile strengths significantly increase by cold rolling, whereas the ductility of the alloys decrease. The increase in tensile strength is attributed to a combined effect of the deformation-induced ω phase transformation and work-hardening during cold roll-

ing. Numerous and coarse ω particles in Ti–18V–CR significantly increase the tensile strength, but drastically decrease the ductility.

Acknowledgements

This study was supported in part by the Industrial Technology Research Grant Program in 2009 from the New Energy and Industrial Technology Development Organization (NEDO) of Japan, the Global COE Materials Integration Program, Tohoku University, Sendai, Japan, the Japan Society for the Promotion of Science (JSPS), Tokyo, Japan, and the inter-university cooperative research program "Highly-functional Interface Science: Innovation of Biomaterials with Highly-functional Interface to Host and Parasite, Tohoku University and Kyushu University", Ministry of Education, Culture, Sports, Science and Technology (MEXT), Tokyo, Japan.

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