Demonstrating a duplex TRIP/TWIP titanium alloy via the introduction of metastable retained β-phase

Karri Sri Naga Sesha a,b, Kenta Yamanaka a, Manami Mori a,c, Yusuke Onuki d, Shigeo Sato e, Damien Fabrègue f,g and Akihiko Chiba a

aInstitute for Materials Research, Tohoku University, Sendai, Japan; bDepartment of Materials Processing, Graduate School of Engineering, Tohoku University, Sendai, Japan; cDepartment of General Engineering, National Institute of Technology, Sendai College, Natori, Japan; dFrontier Research Center for Applied Atomic Sciences, Ibaraki University, Tokai, Japan; eGraduate School of Science and Engineering, Ibaraki University, Hitachi, Japan; fUniversité de Lyon, INSA-Lyon, Villeurbanne, France; gELyT Max, Tohoku University, Sendai, Japan

ABSTRACT
This study demonstrates transformation-induced plasticity (TRIP) in conjunction with twinning-induced plasticity (TWIP) in α + β titanium alloys by introducing metastable retained β-phase. By annealing at 850 °C followed by water quenching, metastable retained β-phase (~25%) was obtained in Ti–6Al–4V alloy. Stress-induced phase transformation in the retained β-phase produced orthorhombic α′′-martensite associated with (021)α′′ twinning, which significantly increased the work-hardening rate and uniform elongation. The findings reveal that the minor retained β-phase is responsible for macroscopic deformation behavior and could aid in novel alloy design that can increase work hardenability with fewer alloying elements than currently available metastable β-titanium alloys.

IMPACT STATEMENT
The minor metastable retained β-phase in an α + β titanium alloy with low alloy content enhanced work hardening because of the combined TRIP/TWIP effect.

1. Introduction

Ti–6Al–4V is a duplex titanium alloy comprising a hexagonal close-packed (hcp) α-matrix and a body-centered cubic (bcc) β-phase. This alloy is a suitable material for aerospace, defense, and biomedical applications owing to its high strength-to-weight ratio, corrosion resistance, and biocompatibility [1,2]. Moreover, titanium alloys exhibit non-equilibrium phases such as α′- and α′′-martensites with hcp and orthorhombic structures, respectively, and the ω-phase, depending on the composition and heat-treatment conditions [3,4]. Among these, the effect of α′-martensite on mechanical properties has been investigated [5–9]. Matsumoto et al. [6] demonstrated that Ti–6Al–4V alloys with a fully α′-microstructure exhibit extraordinary strength, whereas a duplex α + α′ microstructure exhibits a good balance of strength and ductility. Imam et al. [5] asserted that the strain-induced martensitic transformation (SIMT) from the retained β-phase to α′-martensite of Ti–6Al–4V alloys quenched from 900 °C improved the fatigue strength. The formation of α′-martensite is accountable for the increased strength of additively manufactured Ti–6Al–4V alloys [10–12].

α′′-Martensite, on the contrary, is commonly found in metastable β-titanium alloys and has been used to impart shape memory effect and superelasticity [13–17]. Recently, metastable β-titanium alloys with transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) have been developed utilizing a stress-induced β-to-α′ martensitic transforma-
quenching from the between 750 and 900 °C. In comparison, the stability of TRIP/TWIP effects were demonstrated in
ogy with retained austenite in low-alloy TRIP steels, and
According to Welsh et al. [35], the retained high-temperature duplex phase field was investigated.
phase in the Ti–6Al–4V alloy, water quenching from
titanium alloys. To introduce the metastable retained
the as-HT-850.
hereinafter, the specimen is referred to as HT-850.
metastable retained austenite (10–50 vol.% [25,26]) have exhibited significant work hardening [27–32]. This indicates that duplex \( \alpha + \beta \) titanium alloys can exhibit TRIP/TWIP effects. Through precipitation of the \( \alpha \)-phase in a metastable \( \beta \)-matrix, researchers have recently developed duplex \( \alpha + \beta \) titanium alloys and successfully achieved high strength without a loss of ductility [33,34]. However, the primary phase of such alloys was the metastable \( \beta \)-phase, requiring a relatively large amount of alloying elements. Thus, analogous to low-alloy steels with a metastable secondary phase, no studies have been reported on dual-phase TRIP/TWIP titanium alloys with a low alloy content.

In this study, the retained \( \beta \)-phase was utilized in analogy with retained austenite in low-alloy TRIP steels, and TRIP/TWIP effects were demonstrated in \( \alpha + \beta \) duplex titanium alloys. To introduce the metastable retained \( \beta \)-phase in the Ti–6Al–4V alloy, water quenching from the high-temperature duplex phase field was investigated. According to Welsh et al. [35], the retained \( \beta \)-phase can be obtained in Ti–6Al–4V alloys when quenched between 750 and 900 °C. In comparison, the stability of the \( \beta \)-phase decreases with increasing temperature, and quenching from \( \geq 900 \) °C eventually results in the formation of deformation-induced \( \alpha' \)-martensite [5]. Thus, water quenching at 850 °C was selected to obtain a duplex microstructure comprising entirely of the \( \alpha \)- and metastable \( \beta \)-phases and devoid of \( \alpha' \)-martensite.

2. Material and methods

A Ti–6Al–4V alloy rod that was used in our previous study [9] was utilized as a starting material. The as-received specimen was finished with annealing treatment at 704 °C for 2 h, followed by air cooling. Table 1 shows the chemical composition of the as-received rod. The as-received specimen, with a diameter of 12 mm and a length of 60 mm, was encapsulated in an argon-filled quartz tube and heat-treated for 2 h at 850 °C, followed by water quenching. Hereinafter, the specimen is referred to as HT-850.

| Al  | V   | Fe  | C   | N   | O   | H   | Ti  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| 6.01| 3.93| 0.18| 0.015| 0.005| 0.12| 0.0025| Bal.|

Microstructural observations were made using field-emission scanning electron microscopy (FE-SEM; JSM-IT800, JEOL, Japan) operated at an acceleration voltage of 15 kV. Elemental mapping was conducted using field-emission electron probe microanalysis (FE-EPMA) (JXA-8430F, JEOL, Japan) operated at an acceleration voltage of 15 kV. The grain sizes of the \( \alpha \)-phase (intercept method) and \( \beta \)-phase fractions were determined from the SEM-backscattered electron (BSE) images using ImageJ software (NIH, USA). X-ray diffraction (XRD) measurements were conducted using an X’Pert MPD diffractometer (PANalytical, Netherlands). Microstructural observations were conducted on specimens that were ground using emery papers and then mirror-finished with a 0.04-μm colloidal silica solution. The time-of-flight neutron diffraction measurements were conducted using specimens with a diameter of 12 mm and a length of 60 mm at iMATERIA (BL20), J-PARC, Japan [36]. The textures of the specimens were analyzed using the Rietveld texture analysis (RTA) method [37,38]. Scanning transmission electron microscopy (STEM) and energy-dispersive X-ray spectroscopy (EDS) were conducted using a TITAN3 G2 60–300 S/TEM (FEI, USA). Thermodynamic calculations were carried out using Thermo-Calc 2017a software in conjunction with the Thermo Tech Ti-based Alloys database ver. 3.1 (Thermo-Calc, Sweden). The Vickers hardness of the \( \alpha \)-matrix was determined using a micro-Vickers hardness meter (HMV-G31, SHIMADZU, Japan). A force of 98 mN was applied for an indentation time of 10 s; a total of 10 measurements were performed, and the average values were reported. Additionally, uniaxial tensile testing was carried out at room temperature using an Instron 5969 machine with an initial strain rate of \( 1.0 \times 10^{-4} \) s\(^{-1}\). Tensile strains were measured upon loading using a non-contact video extensometer (AVE2, Instron, USA). Electrical discharge machining was used to prepare flat dog bone-shaped specimens with a gauge length of 16 mm, a width of 2 mm, and a thickness of 1 mm. In total, three samples were tested for each condition (as-received and HT-850) to ensure reproducibility.

3. Results and discussion

Figure 1(a1) and 1(a2) show SEM-BSE images of the as-received and HT-850 specimens, respectively. Both specimens had equiaxed \( \alpha + \beta \) microstructures. The HT-850 specimen exhibited slight grain growth with an average grain size of 1.0 and 3.7 μm for the as-received specimen and the HT-850 specimen, respectively. XRD patterns of the investigated specimens, which show the duplex \( \alpha + \beta \)
microstructures, are shown in Figure 1(b1) and 1(b2). No additional phases were identified clearly. The area fractions of the \( \beta \)-phase were determined to be 5.5% and 25% for the as-received and HT-850 specimens, respectively. The \( 100_\alpha \) and \( 002_\alpha \) pole figures in Figure 1(c1) and 1(c2), as determined by RTA, indicate a negligible difference in the crystallographic orientation distribution, indicating the preservation of \( \alpha \text{L}100\alpha \text{L} \) fiber texture along the longitudinal direction (LD) of the rods. STEM-EDS analysis of the HT-850 specimen is depicted in Figure 1(d). A retained \( \beta \)-phase was captured, which can be recognized as a V-rich region. While the annular bright-field (ABF)-STEM and corresponding high-angle annular dark-field (HAADF)-STEM images depict some contrasts in the \( \beta \)-phase, the selected area electron diffraction (SAED) pattern recorded with the beam direction (BD) along the [001]\( \beta \) direction exclusively revealed \( \beta \)-reflections. In contrast, the diffuse streaks that appeared when the BD was parallel to the [011]\( \beta \) direction implied the formation of a small amount of an athermal \( \omega \)-phase, although no \( \omega \)-reflections were identified. Thus, these contrasts correspond primarily to the dislocation substructures introduced during the heat treatment via quenching and/or volume expansion due to \( \alpha \rightarrow \beta \) phase transformation [12].

The SEM-BSE images and corresponding EPMA elemental maps for the as-received and HT-850 specimens are shown in Figure 2. The \( \beta \)-phase was enriched in V and Fe, whereas Al was distributed preferentially in the \( \alpha \)-phase. The heat treatment had no discernible effect on the composition of the \( \alpha \)-phase [Figure 2(c)]. In contrast, the \( \beta \)-phase concentrations were significantly dependent on the heat-treatment temperature. The concentration of V in the \( \beta \)-phase decreased from 15 mass% in the as-received specimen to 8 mass% in HT-850 [Figure 2(c)]. Fe content in the \( \beta \)-phase exhibited a similar trend. Specifically, as the \( \beta \)-phase fraction increased with heat treatment, the concentration of the \( \beta \)-stabilizing elements (V and Fe) in the \( \beta \)-phase decreased. Overall, the elemental distributions matched those found in the equilibrium state for each condition.
Figure 2. SEM-BSE images and corresponding EPMA elemental maps of the (a) as-received and (b) HT-850 specimens. (c) Variations in the alloying elemental concentrations in the $\alpha$-phase and $\beta$-phase of as-received and HT-850 specimens. The equilibrium compositions of each phase for the Ti–6.01Al–3.93V–0.18Fe–0.12O (mass%) system (704 and 850 °C for the as-received and HT-850 specimens, respectively), as calculated using Thermo-Calc, are also shown as black lines for comparison.

Table 2. Mechanical properties of the as-received and HT-850 specimens.

|                  | 0.2%PS (MPa) | UTS (MPa) | Elongation-to-failure (%) | Uniform elongation (%) | Vickers hardness ($\alpha$-matrix) |
|------------------|--------------|-----------|---------------------------|------------------------|----------------------------------|
| As-received      | 902 ± 3      | 980 ± 6   | 13.1 ± 1.3                | 6.1 ± 0.8              | 309 ± 7.3                        |
| HT-850           | 655 ± 14     | 894 ± 6   | 18.0 ± 0.9                | 11.1 ± 0.9             | 300 ± 6.8                        |

Figure 3. (a) Stress-strain curves of the as-received and HT-850 specimens and (b) true stress (solid lines) and work-hardening rate (broken lines) as a function of true strain for the as-received and HT-850 specimens.

The stress–strain curves for the as-received and HT-850 specimens, as determined by tensile testing, are shown in Figure 3(a). Additionally, the tensile properties of the specimens are presented in Table 2. The as-received specimen exhibited no significant work hardening, which is typical for Ti–6Al–4V alloys. Compared to the as-received specimen, the HT-850 specimen demonstrated a decrease in the 0.2% proof stress (0.2%PS); however, the decrease in the ultimate tensile strength (UTS) was less marked. Notably, significant work hardening was observed in the HT-850 specimen upon tensile loading. The evolution of the work-hardening rate
(θ), which can be calculated as follows, is depicted in Figure 3(b).

\[ \theta = \frac{d\sigma}{d\varepsilon} \]  (1)

where \( \sigma \) and \( \varepsilon \) are true stress and true strain, respectively. While the \( \theta \) for the as-received specimen decreased monotonically with increasing \( \varepsilon \), the HT-850 specimen exhibited a consistently higher \( \theta \) across the strain range. Notably, after initially decreasing, the HT-850 specimen demonstrated an extraordinary increase in \( \theta \) with an increase in \( \varepsilon \), reaching a very high \( \theta \) of \( \sim 3 \) GPa. The increased work hardening at the later stages of the tensile loading process prevented early onset of plastic instability, as defined by the Considère criterion.

\[ \frac{d\sigma}{d\varepsilon} \leq \sigma \]  (2)

As illustrated in Figure 3(b), both specimens satisfy Eq. (2). Thus, the uniform elongation of the HT-850 specimen (11.1%) was nearly twice that of its as-received counterpart. Notably, the Vickers microhardness of the \( \alpha \)-matrix was comparable between the investigated specimens (Table 2), indicating that the \( \alpha \)-phase in the investigated specimens possesses no discernible difference in strength or plasticity. Thus, the difference in mechanical performance discussed above is primarily due to the retained \( \beta \)-phase.

To gain a better understanding of the effect of the retained \( \beta \)-phase on work-hardening behavior, SEM-BSE images of the HT-850 specimen in the as-quenched state and after tensile failure were captured in the same observation area (Figure 4). Tensile loading resulted in the introduction of deformation-induced products into the retained \( \beta \)-phase (brighter contrasts).

The ABF-STEM image of the HT-850 specimen following tensile failure is shown in Figure 5(a). In the retained \( \beta \)-phase, a deformation-induced microstructural evolution was observed. The absence of apparent dislocation substructures in the surrounding \( \alpha \)-matrix indicates that plastic deformation took place preferentially in the retained \( \beta \)-phase. The high-magnification image in Figure 5(b) illustrates the formation of plate-like products as a result of deformation. According to the nanobeam diffraction (NBD) patterns obtained from circles c1 and c2 in Figure 5(c), the plate-like microstructure comprised \( \alpha'' \)-martensite. Thus, the retained \( \beta \)-phase in the HT-850 specimen was mechanically metastable and converted to orthorhombic \( \alpha'' \)-martensite via SIM upon tensile loading. Additionally, SAED analysis [Figure 5(d)] revealed that \( \alpha'' \)-martensite was associated with nanoscale (021)-type twinning [39,40].

Here, the phase stability of the retained \( \beta \)-phase is considered using the ‘\( d \)-electron alloy design method’ proposed by Morinaga et al. [41–43]. The mean \( d \)-orbital energy level (\( M_d \)) and mean bond order (\( B_o \)) were taken into account in this method, which is one of the tools used to design low-modulus \( \beta \)-titanium alloys [42,43] and metastable \( \beta \)-titanium alloys [18,21]. The \( M_d \) and \( B_o \) values for the retained \( \beta \)-phase in the HT-850 specimen were determined to be 2.35 and 2.77, respectively, using the chemical composition analyzed by STEM-EDS. The retained \( \beta \)-phase in the HT-850 specimen can be classified into the TRIP/TWIP deformation mode using the modified \( B_o \) vs \( M_d \) map [44]. Thus, the retained \( \beta \)-phase in the HT-850 specimen functions as a metastable phase that undergoes SIM to form \( \alpha'' \)-martensite.

The transformation to \( \alpha'' \)-martensite during tensile loading enables the specimen to accommodate strong local strains that could otherwise result in failure. Additionally, the dynamic Hall-Petch effect is widely accepted.
as a mechanism for explaining the high work hardening and elongation observed in TRIP/TWIP-assisted metastable titanium alloys [18–23]. The newly formed interfaces/twin boundaries decrease the effective grain size and act as barriers to dislocation motion, thereby limiting the mean free path of dislocations. Notably, the TWIP effect in β-titanium alloys has been attained via twinning in the β-phase, such as the 332[110]113[111] and 112[111]111[112] systems [18–23]. In contrast, the HT-850 specimen did not show these mechanical twins in the retained β-phase. Instead, α′′-martensite was associated with nanoscale (021)α′′-type twinning. The formation of such transformation twins could contribute to the work hardening of the HT-850 specimen. Thus, the term ‘TRIP/TWIP effect’ is employed in this paper. Enhancing the work hardenability of titanium alloys is critical for increasing the ductility and durability of critical titanium components that are subjected to fatigue loading. Notably, the results suggest that a combined TRIP/TWIP effect in the retained β-phase, which exists as a minor phase in the duplex microstructure, can control the macroscopic mechanical behavior, because the α-phase in titanium alloys does not show remarkable work hardening. As a result, the high work-hardening rate of HT-850, ~3 GPa, is comparable to or greater than that of single-phase or α-precipitated metastable β-titanium alloys [18,22,33,34,45,46]. Thus, incorporating the TRIP/TWIP concept into the α + β alloys results in the creation of a new class of titanium alloys with increased work hardenability; it also enables the design of TRIP/TWIP titanium alloys with lower alloying contents and consequently lower material costs.

Additionally, the yield stress of the HT-850 specimen was generally greater than that of some β-titanium alloys. The strength of α + β titanium alloys has frequently been described in the following manner [47]:

$$\sigma = \sigma_\alpha(1-f_\beta) + \sigma_\beta f_\beta$$

(3)

where $\sigma_\alpha$ and $\sigma_\beta$ are the strength of the α- and β-phases, respectively, and $f_\beta$ is the volume fraction of the β-phase. Given that the Vickers hardness (Table 2) and the crystallographic orientation distribution [Figure 1(c)] of the α-phase were similar among the investigated specimens, the contribution of the α-phase to the yield stress of the HT-850 specimen was calculated to be ~675 MPa, which exceeds the 0.2%PS, for $f_\beta = ~25\%$, by assuming that the α-phase strength of the HT-850 specimen is identical to that for the as-received specimen. This implies that Eq. (3) does not properly describe the yield stress of the HT-850 specimen. In contrast, the absence of apparent dislocation substructures in the α-matrix following tensile failure [Figure 5(a)] suggests that the α-matrix plays a minor role and that plastic deformation occurs preferentially in the softer β-phase. Thus, the yield stress of the HT-850 specimen primarily corresponds to that of the retained β-phase, and the solution strengthening
caused by the V enrichment could play an important role. Despite this, the onset of yielding in the retained β-phase falls into the lower 0.2%PS than that of conventional Ti–6Al–4V alloys. Further improvements in yield strength will be pursued in future studies by further optimizing the heat treatment, modifying the deformation mechanism [45], and ω-phase precipitation [46].

4. Conclusions

In summary, the HT-850 specimen with a retained metastable β-phase exhibited the combined TRIP and TWIP effects, exhibiting a remarkably high work-hardening rate (∼3 GPa) upon tensile loading. The macroscopic mechanical behavior was governed by the retained β-phase, which formed a SIMed α′′-phase associated with the (021)α′ twinning. This study shed light on the potentiality of the minor β-phase in low-alloy duplex α + β titanium alloys, which has not been explored previously, thereby providing a new avenue for a high work-hardening rate with low cost because of low alloy contents. Thus, the obtained results could contribute to the alloy design for low-cost TRIP/TWIP titanium alloys and to the improvement of plastic deformation in duplex α + β alloys.

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Disclosure statement

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ORCID

Kenta Yamanaka http://orcid.org/0000-0003-1675-4731
Yusuke Onuki http://orcid.org/0000-0001-9763-0970
Shigeo Sato http://orcid.org/0000-0003-3294-3348
Akihiko Chiba http://orcid.org/0000-0001-8227-7975

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