



# *Article* **Microstructure Control for Enhancing the Combination of Strength and Elongation in Ti-6Al-4V through Heat Treatment**

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**Abstract:** For the application of Ti-6Al-4V alloys in urban air mobility, safety is very important, so achieving excellent strength and toughness is essential to prevent fractures. Regarding toughness, which is a combination of strength and ductility, it is necessary to derive the optimal heat treatment conditions for this combination of Ti-6Al-4V alloy and further understand its microstructure and fracture characteristics. For this purpose, this study investigated the microstructure in terms of grain size, plate thickness, and element distribution, as well as mechanical properties, including phase hardness and tensile properties, of Ti-6Al-4V alloy subjected to solution treatment and aging (STA) heat treatment under various aging conditions. As a result, this study suggests that solution treatment followed by aging at  $630 °C$  for 480 min can achieve approximately 26% higher toughness than the just-solution treatment process. This is because there is little difference in hardness between the equiaxed  $\alpha$  and basketweave structures, and β plates, which contain an excessive V between  $\alpha$  plates, function like fibers and delay fracture.

**Keywords:** Ti-6Al-4V alloy; heat treatment; microstructure; strength; toughness



In recent years, the urban air mobility (UAM) market has rapidly emerged as a nextgeneration transportation solution that solves the urgent issue of urban traffic congestion and provides efficient transportation  $[1,2]$  $[1,2]$ . One of the most critical problems to be solved in the development of UAM is safety [\[2,](#page-14-1)[3\]](#page-14-2). This requires not only having systems to avoid collisions with other aircraft and obstacles during flight [\[3\]](#page-14-2), but also ensuring that the materials composing the aircraft possess high strength while maintaining high ductility [\[4,](#page-14-3)[5\]](#page-14-4). Specifically, toughness represents the total amount of energy that a material can absorb during the deformation process, and is a mechanical property that is an appropriate balance between strength, which is the material's ability to withstand deformation, and ductility, which is the ability to maintain a load without breaking [\[6\]](#page-14-5). In other words, applying high-strength and high-toughness materials to UAM can prevent sudden structural failures from external impacts or excessive loads, thus enhancing durability and safety. However, because strength and ductility are generally in a trade-off relationship in most metals [\[6](#page-14-5)[,7\]](#page-14-6), it is still difficult to obtain a high-toughness alloy, which has both excellent strength and ductility [\[8\]](#page-14-7).

The Ti-6Al-4V (Ti64) alloy is one of the representative lightweight alloys used in aircraft and boasts excellent properties such as strength, corrosion resistance, and hightemperature properties [\[9](#page-15-0)[–12\]](#page-15-1). To improve strength, solution treatment and aging (STA) heat treatment is commonly used, which involves two cycles of heat treatment consisting of solution treatment (ST) and aging (A) [\[12](#page-15-1)[,13\]](#page-15-2). When performing the STA heat treatment



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below the  $\beta$ -transus temperature, a microstructure consisting of equiaxed  $\alpha$  and transformed β, known as a duplex microstructure, is observed [\[14–](#page-15-3)[17\]](#page-15-4). Numerous studies have been conducted regarding the microstructural property changes under various ST and A conditions. S. Huang et al. [\[18\]](#page-15-5) found that higher ST temperatures lead to greater concentration variations in V and Al in transformed β compared to equiaxed  $\alpha$ . Y.C. Lin et al. [\[19\]](#page-15-6) found that coarsened secondary α and equiaxed α combined to form new curved lamellar α, while N. Kherrouba et al. [\[20\]](#page-15-7) suggested a relationship between the precipitation of secondary  $\alpha$  and partial V diffusion in transformed  $\beta$ . Regarding the mechanical properties under various STA conditions, T. Morita et al. [\[21\]](#page-15-8), S. Tanka et al. [\[22\]](#page-15-9), and A. Ajiz et al. [\[23\]](#page-15-10) observed microstructures and mechanical properties under different aging conditions after quenching at 930 °C for 60 s, and found that aging within the range of 530–580 °C has the highest tensile strength. S.T. Oh et al. [\[24\]](#page-15-11) noted that a higher amount of retained β after ST results in a pronounced TRIP effect during aging, with the best combination of tensile properties observed at 550 °C for 300 s. Y. Vahidshad et al. [\[25\]](#page-15-12) reported that ST at 950 ◦C followed by aging at 500 ◦C was the condition with the highest tensile strength with  $Ti<sub>3</sub>Al. G. Perumal et al. [26] investigated the mechanical properties and wear char Ti<sub>3</sub>Al. G. Perumal et al. [26] investigated the mechanical properties and wear char Ti<sub>3</sub>Al. G. Perumal et al. [26] investigated the mechanical properties and wear char$ acteristics under various STA conditions, revealing that a fine lamellar structure exhibits high hardness, strength, and low wear rate. However, these studies [\[21–](#page-15-8)[26\]](#page-15-13) did not analyze from the perspective of changes in toughness, which is the combination of strength and elongation, according to STA conditions. Recently, with regard to Ti64 STA heat treatment and toughness, Q. Zhu et al. [\[27\]](#page-15-14) and R.N. Elshaer et al. [\[28\]](#page-15-15) reported that air cooling in ST or water cooling and aging in ST showed higher toughness. However, these studies [\[27,](#page-15-14)[28\]](#page-15-15) lack sufficient explanation regarding the microstructural features for enhancing toughness and their correlation with fractographies under different STA conditions.

As in the aforementioned studies, there still remains a lack of research on STA heattreated Ti64 to achieve a high combination of strength and elongation, especially regarding the correlation between microstructural characteristics and fractographies. Moreover, the hardness of each phase at the micro-scale changes due to phase transformation, grain size, and redistribution of elements within the phase during STA, which ultimately affects the macro-scale tensile properties due to the interaction of each phase under tensile loading [\[29\]](#page-15-16). Therefore, in this study, microstructural characteristics such as size and thickness of the phase and V distribution were precisely controlled by changing the aging temperature and time during STA heat treatment, and the phase hardness, strength, elongation and modulus of toughness were compared for each heat treatment condition. Consequently, we propose that aging at 630  $\degree$ C for 480 min is the optimal heat treatment condition for a high modulus of toughness with proper strength.

# **2. Materials and Experimental Procedures**

# *2.1. Material and Heat Treatment Conditions*

In this study, a rod-shaped Ti-6Al-4V alloy ( $\varnothing$ 70 mm  $\times$  2000 mm) fabricated via a vacuum arc remelting (VAR) process by KPC Metal Co., Ltd. (Gyeongsan, Republic of Korea), served as a starting material. The chemical composition of the alloy is obtained using inductively coupled plasma optical emission spectroscopy (ICP-OES, Optima 7300DV, PerkinElmer, Hopkinton, MA, USA) analysis, which is presented in Table [1.](#page-1-0) Additionally, the as-received sample is a fully equiaxed microstructure, which consists with equiaxed  $\alpha$ and  $β$  in Figure [1.](#page-2-0)

<span id="page-1-0"></span>**Table 1.** Chemical composition (wt.%) of Ti-6Al-4V used in this study.

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Balance	6.56	4.15	0.19	19 ∪.⊥	0.021	0.027	0.003

<span id="page-2-0"></span>

**Figure 1.** Optical microstructure (OM) of initial state of Ti64 used in this study. **Figure 1.** Optical microstructure (OM) of initial state of Ti64 used in this study.

Samples for heat treatment were machined to dimensions of  $10 \times 10 \times 55 \; (\text{mm}^3)$ . The samples were solution-treated at 950 °C for 1 h followed by water quenching (WQ) and aging at temperatures ranging from 480 to 630  $\degree$ C for durations ranging from 1 min to 480 min, then followed by air cooling (AC), as illustrated in Figure [2.](#page-2-1) All the heat treatments were performed in a quartz furnace under vacuum ( $6 \times 10^{-5}$  torr).

<span id="page-2-1"></span>

Figure 2. Conditions of solution treatment and aging (STA) heat treatment procedures.

#### $\Omega$  Characterization **Figure 2.** Conditions of solution treatment and aging (STA) heat treatment procedures. *2.2. Characterization*

In Emmanumanum<br>The hardness of all STA samples before and after aging was measured using a microaging condition, which is, in detail, described in Section 3.1, we chose 4 conditions to an-*2.2. Characterization*  Vickers hardness tester (HM–210B, Mitutoyo, Japan) with a load of 1 kgf on a machined  $10 \times 10 \times 5 \text{ (mm}^3)$  area (Figure 3a). Then, in the hardness profile according to the various aging condition, which is, in detail, described in section 3.1, we chose 4 conditions to<br>analyze further microstructural and mechanical properties; these conditions are solution treatment (ST) and 3 aging conditions at  $480^{\circ}$ C for 1 min (A\_480-1), 530  $^{\circ}$ C for 120 min  $(A_530-120)$  and  $630 °C$  for  $480$  min  $(A_630-480)$ . aging condition, which is, in detail, described in Section [3.1,](#page-3-1) we chose 4 conditions to

ment, the incrostructures of 4 conditions (51, A\_400-1, A\_500-120, A\_000-400) were<br>analyzed using X-ray diffraction (XRD, X'pert-Pro MPD, PANalytical, Marvern, UK), optical microscopy (OM, HRM-300, Huvitz, Houston, TX, USA) and field-emission scanning electron microscopy (FE-SEM, NNS-450, FEI, Hilsboro, OR, USA). The XRD was analyzed Then, the microstructures of 4 conditions  $(ST, A_480-1, A_530-120, A_630-480)$  were

with the Cu K $\alpha$  radiation source in the 20 range of 30–90 $\degree$  at a scan speed of 0.02 $\degree$  min<sup>-1</sup>. Then, the same samples were mechanically grinded and polished using a 1  $\mu$ m diamond Then, the same samples were mechanically grinded and poilshed using a 1 functionidal<br>suspension, followed by an oxide suspension (OP-S solution), and etched using Kroll's reagent (5 mL HF and 10 mL HNO<sub>3</sub> in 85 mL distilled H<sub>2</sub>O) for OM and SEM analysis. Grain size and plate thickness were also measured at 7 points in OM images, and at 9 plates in SEM microstructures, respectively. Additionally, the distribution of Al and V in the STA heat-treated samples was analyzed using electron probe micro-analysis (EPMA, JXA-8500F,<br>UCM Televe Japon) for line scenning across the durbay phases JEOL, Tokyo, Japan) for line scanning across the duplex phases.

(a) Sample preparation area in heat-treated Ti64

<span id="page-3-0"></span>



microstructural and mechanical property analysis and (**b**) presents the sample dimensions for the tensile test conducted in this study. **Figure 3.** (**a**) is a diagram indicating the sampling locations from the heat-treated specimen for

was measured using a nitro-vickers nardness tester with a 0.00 kg1 load. The nardness<br>value for each structure represents an average of 5 measurements. Tensile tests were *3.1. Hardness Profile*  at from temperature with a test speed of 0.001/3. The tensile samples were prepared<br>as proportional specimens in compliance with the ASTM E8/E8M [\[30\]](#page-15-17). The machining location of tensile samples is shown in Figure 3a, and the sample dimensions are provided in Figure 3b. Following the tensile test, fractographies were analyzed using SEM. The hardness of each phase in the duplex structure under various aging conditions was measured using a micro-Vickers hardness tester with a 0.05 kgf load. The hardness conducted 3 times using a universal testing machine (5982, Instron, Norwood, MA, USA) at room temperature with a test speed of 0.001/s. The tensile samples were prepared

# **3. Results and Discussion**

# <span id="page-3-1"></span>[13,21–23]. Subsequent short-term aging (1 min) further elevates the hardness of the Ti-*3.1. Hardness Profile*

The hardness profile before and after STA heat treatment is presented in Figure [4.](#page-4-0) In Figure 4, the Ti64 alloy before heat treatment is indicated as the 'initial' alloy. After ST, the hardness of the initial alloy increases significantly to 366.7  $(\pm 7.4)$  Hv. In several studies, this increase in nardness is attributed to the transformation of the  $p$  to  $\alpha$  during quenching [\[13](#page-15-2)[,21–](#page-15-8)[23\]](#page-15-10). Subsequent short-term aging (1 min) further elevates the hardness of the Ti-6Al-4V alloy slightly. The hardness of STA samples increases with increasing aging time, until it reaches a maximum (peak hardness), after which it begins to decrease. The highest hardness, measured at 393.6 ( $\pm$ 3.9) Hv, is achieved at the sample aged at 530 °C for of  $372.4 \pm 5.97$ ) Hv is observed in the sample aged for 2 min, after which it decreases. The sample aged at 630 °C for 480 min has 351.27 ( $\pm$ 3.26) Hv, which is the lowest value  $\alpha$  after STA. studies, this increase in hardness is attributed to the transformation of the  $\beta$  to  $\alpha'$  during 120 min. Moreover, the holding time required for an STA sample to reach peak hardness decreases with an increasing holding temperature. At  $630\degree C$  aging, the peak hardness after STA.

Therefore, in the hardness profiles among the solution treatment and several ag-<br>in a sendition sure these four sonditions which are the solution treatment (CT) and three conditions of aging  $(A_480-1, A_530-120, A_630-480)$ .  $A_480-1$  is the short aging step representing the case of holding at the lowest temperature (480 °C) for the shortest time (1 min); A\_530-120 corresponds to holding at 530 °C for 120 min, which is the highest ing conditions, we chose four conditions, which are the solution treatment (ST) and hardness value; and 630–480 represents holding at the highest temperature (630 °C) for the longest duration (480 min), which is the lowest hardness value in all aging conditions.

<span id="page-4-0"></span>

ple, and various aging conditions for a selection of conditions to analyze microstructural and mechanical properties  $v_{\rm r}$  and  $v_{\rm r}$  selection of conditions to analyze microstructural and mechanical and mechanica **Figure 4.** Micro-Vickers hardness profile of initial state of alloy, solution-treated (ST) sammechanical properties.

# properties. *3.2. Microstructure Model*

*3.2. Changes during STA, schematically shown in Figure [5,](#page-5-0) which is based on several* on a gray background, the  $\alpha$  phase is light gray, and the β phase is dark gray. The red dot is the V element, and the black dot is Ti<sub>3</sub>Al, which is a representative precipitated phase in α during aging [\[11,](#page-15-18)[13\]](#page-15-2). The initial Ti64, which consists of equiaxed α and β in Figure 5a, changes its microstructure during STA heat treatment as follows: While holding sufficient<br>time in ST, the misraetwature consiste of more than half of θ and μ (Figure 5b), and Misthe Herry are interesting the black of these than that or  $\beta$  and  $\alpha$  (rights  $\epsilon$ e), and  $\tau$  is diffused into β at the temperature [\[13,](#page-15-2)[18\]](#page-15-5). During rapid cooling by water, β is transformed into α', a type of martensite phase, and V is supersaturated in α' because diffusion from β to α is impossible due to the fast cooling rate (Figure 5b) [11,13]. Before discussing the microstructure, we proposed the microstructure model that studies [\[13–](#page-15-2)[28\]](#page-15-15). We illustrated that the  $\alpha'$  phase is a granite-like pattern that has black dots time in ST, the microstructure consists of more than half of  $\beta$  and  $\alpha$  (Figure [5b](#page-5-0)), and V is

During aging, the secondary step,  $\alpha'$  is transformed into the plate-like α and β by amount of interfacial area between  $\alpha$  and β plates is larger, the resistance of dislocation movement increases, so the alloys become harder and more difficult to deform. Also, Ti<sub>3</sub>Al, which is the hard phase, can be precipitated in  $\alpha$  during aging [9,[11](#page-15-0),32[\].](#page-15-20) So, we expected that for Ti64 to have the highest strength, the microstructure should be composed of the sufficiently fine α, β plates and Ti<sub>3</sub>Al phase, as shown in Figure [5c](#page-5-0). The Ti<sub>3</sub>Al phase fraction<br>during a sing is annuarimately 2%, calculated using the DAptDre ver 7.0, [32]. If the aging temperature and time are increased, the α and β plates with high V may become coarse, leading to the potential dissolution of Ti<sub>3</sub>Al. This can result in a decrease in hardness and strength while increasing ductility. However, if this change is moderate, toughness may be expected that for Tigure [5d](#page-5-0).<br>The microstructure shows the microstructure shows the microstructure shows that communications of the microstr redistributing the V element into β [\[11](#page-15-18)[–13,](#page-15-2)[20\]](#page-15-7). According to X. Shi et al. [\[31\]](#page-15-19), when the during aging is approximately 2%, calculated using the JMatPro ver.7.0. [\[33\]](#page-15-21). If the aging

propose the two models for the strongest case and the toughest in<br>crostructures, as shown in Figure [5c](#page-5-0),d. Figure 5c, which is predicted to have the highest hardness and strength, consists of equiaxed α, plate-like α and β with concentrated V, and some Ti<sub>3</sub>Al. Figure 5d represents the example with the highest ductility and toughness, with a microstructure consisting of equiaxed α, coarse α and β plates and the highest V concentration in the  $\beta$  plate. Therefore, we propose the two models for the strongest case and the toughest miconcentration in the β plate.

<span id="page-5-0"></span>

Figure 5. Schematic diagrams of microstructural models before heat treatment ((a) initial state), and during solution treatment ( $(b)$  ST) and aging heat treatment  $(c,d)$ . In the diagram,  $(c)$  corresponds to the state of the highest strength and (d) corresponds to the state of the highest toughness.  $\alpha'$  phase is a granite-like pattern, α phase is light gray, and β phase is dark gray. V element is shown as a red dot and  $Ti<sub>3</sub>Al$  is a black dot.

# *3.3. Microstructure Characterization 3.3. Microstructure Characterization*

3.3.1. X-ray Diffraction and Phase Evolution 3.3.1. X-ray Diffraction and Phase Evolution

Figur[e 6](#page-6-0) displays the XRD results for ST and several aging conditions (A\_480-1, Figure 6 displays the XRD results for ST and several aging conditions (A\_480-1, A\_530- 120, A\_630-480). Peaks in XRD patterns were indexed with reference to the Inorganic ganic Crystal Structure Database (ICSD) cards for α-Ti (ICSD 01-089-5009), β-Ti (ICSD 01- Crystal Structure Database (ICSD) cards for α-Ti (ICSD 01-089-5009), β-Ti (ICSD 01-089- 4913) and Ti<sub>3</sub>Al (ICSD 03-065-4565) phases. The ST sample predominantly exhibits  $\alpha/\alpha'$ peaks in the entire theta range. In the magnified images of 37 to 42.5 $^{\circ}$  and 75 to 85 $^{\circ}$ , some separated, inflected and shifted peaks are observed near the closet peak of the  $\alpha$  phase peaks. Here, α is the primary α formed during isothermal holding in the solution treatment.  $\alpha'$  is formed by rapid cooling; due to the residual stress caused by the quenching process, it exhibits a slightly shifted peak compared to  $\alpha$  [\[32\]](#page-15-20). Additionally, the ST sample shows a (211) β peak, suggesting the presence of retained β. This could be because a small amount of β that has not yet been transformed into  $\alpha'$  is retained due to rapid cooling. Therefore, the ST sample contains  $\alpha'$ , α and retained β phases.

During aging, as studied by Morita et al. [\[21\]](#page-15-8) and other studies [\[22](#page-15-9)[–25\]](#page-15-12),  $\alpha$  is precipitated in  $\alpha'$ , so  $\alpha'$  is decomposed to widmanstätten  $\alpha + \beta$ , and subjected to a longer aging process, and Ti<sub>3</sub>Al can be precipitated. Ti<sub>3</sub>Al peaks are similar to  $\alpha$ , but due to the lattice distortion, the peaks are also shifted, and the peak information is in the Ti $_3$ Al ICSD card. The A\_480-1 condition comprises α and β peaks, with more β peaks detected than in the The A\_480-1 condition comprises α and β peaks, with more β peaks detected than in the ST condition, and there are some retained  $\alpha'$  peaks in the magnified area. Regarding this phenomenon, we calculated the evolution of phase fraction according to the holding time phenomenon, we calculated the evolution of phase fraction according to the holding time at each aging temperat[ure](#page-7-0) after solution treatment at 950 °C (Figure 7a); this was based on the time–temperat[ur](#page-7-0)e–transformation (TTT) diagram (Figure 7b) using JMatPro ver. 7.0. [\[33\]](#page-15-21). According to the graph, in the case of aging at 480 °C for 1 min, the transformation of  $\alpha'$  into α starts, but does not reach the equilibrium fraction of α. Regarding Ti<sub>3</sub>Al precipitation in this condition, as shown in the Ti $_3$ Al curve of the TTT diagram,  $0.1\%$  precipitation can occur when held for more than 35 min at 480 °C. However, because of the short holding time of only 1 min, Ti<sub>3</sub>Al was not observed in A\_480-1. A\_530-120 exhibits peaks corresponding to α, β and Ti<sub>3</sub>Al. In Figure 7a, it can be observed that holding at 530 °C for more than 90 min completes the precipitation of α from α'. Also, regarding Ti<sub>3</sub>Al precipitation, in Figure [7a](#page-7-0),b, 0.1% Ti<sub>3</sub>Al is precipitated for more than 15 min; so, holding at 530 °C for

120 min allows for A\_530-120 to be sufficiently precipitated in that phase, and the fraction is about 2%. Therefore, in the XRD results of A\_530-120, the α, β and Ti3Al peaks without  $α'$  observed are valid results. A\_630-480, which has the largest aging degree, consists of  $\alpha$  and β, but without Ti<sub>3</sub>Al peaks. As shown in the 630 °C curve in Figure 7a, the higher the aging temperature, the faster the precipitation and transformation onset, as well as the transformation rate. At 630 °C, the precipitation of α from α' is completed within just 10 min. Furthermore, it can be confirmed from Figure [7a](#page-7-0) and b that even after holding for 10<sup>3</sup> at 630 °C, the precipitation of T<sub>i3</sub>Al does not occur. So, in this study, for A 630-480, only the α and  $β$  peaks exist. peaks without α′ observed are valid results. A\_630-480, which has the largest aging de- $\frac{120 \text{ H}}{1 \times 200 \text{ F}}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  peaks. As shown in the  $\frac{1}{2}$  curve in the  $\frac{1}{2}$  curve in the  $\frac{1}{2}$  curve in  $\frac{1}{2}$  curve in  $\frac{1}{2}$  curve in  $\frac{1}{2}$  curve in  $\frac{1}{2$ 

and the fraction is about 2%. The fraction is about 2%. The  $\bar{X}_5$  and Ti3Al-30-120, the a,  $\bar{B}_5$  and  $\bar{B}_6$ 

<span id="page-6-0"></span>

heat treated samples aged at 480 °C; for 1 min (A\_480-1), at 530 °C; for 120 min (A\_530-120), and at  $heta^2$  of the 480 min (A\_630-480), including peaks for each phase,  $\alpha'$  is dotted line. **Figure 6.** XRD results for the entire and some magnified ranges of solution treatment (ST) and STA heat treated samples aged at 480 °C; for 1 min (A\_480-1), at 530 °C; for 120 min (A\_530-120), and at 630 °C for 480 min ( 630 °C for 480 min (A\_630-480), including peaks for each phase,  $\alpha'$  is dotted line.

<span id="page-7-0"></span>

(a) evolution of the  $\alpha$  and Ti<sub>3</sub>Al fractions (at.%) according to holding temperature and time after soluevolution of the and Ti3Al fractions (at. 8) according to holding temperature and time after solution treatment at 950 °C during aging and (**b**) the time–temperature–transformation (TTT) diagram. tion treatment at 950 ◦C during aging and (**b**) the time–temperature–transformation (TTT) diagram. Figure 7. Calculation results for Ti64 used in this study adapted from JMatPro ver. 7.0. [\[33\]](#page-15-21);

#### 3.3.2. Microstructure 3.3.2. Microstructure

Figure [8a](#page-7-1)-d present the OM images of ST and several aging conditions (A\_480-1, 1, A\_530-120, A\_630-480). Each sample exhibits a duplex microstructure consisting of  $\alpha$  (α<sub>E</sub>), represented by a bright phase, α' and/or a basketweave structure, which is relatively dark and resembles an intertwined structure [11,12]. According to the image analyzer, the amount of equiaxed  $\alpha$  in the ST is approximately 30%, similar to that in other samples. The appearance of  $\alpha'$  and/or basketweave structures is not significantly different samples. The appearance of α′ and/or basketweave structures is not significantly different when observed using OM. when observed using OM.

<span id="page-7-1"></span>

(b) A\_480-1, (c) A\_530-120, and (d) A\_630-480; the area; the dotted line in ST represents  $\alpha'$ , and the dotted line in the aged samples represents  $\alpha'$  and/or basketweave structure. **Figure 8.** OM images of the microstructures of (**a**) solution treatment (ST) and STA samples aged at **Figure 8.** OM images of the microstructures of (**a**) solution treatment (ST) and STA samples aged at

Therefore, in the duplex microstructure, especially  $\alpha'$ , and/or a basketweave structure, were observed at high magnification using SEM, as shown in [Fi](#page-8-0)gure 9a–h. [F](#page-8-0)igure 9b,d,f,h are magnified from Figure [9a](#page-8-0),c,e,g, respectively. In Figure [9b](#page-8-0), transformed β in the ST sample consists of intertwined, very fine (average 50 nm thickness) acicular  $\alpha'$  and some nano-sized, retained β β<sub>re</sub> between α'. During aging, the growth in equiaxed α and the transformation of α' into α and β occ[ur \[](#page-15-18)11]. Fig[ur](#page-8-0)e 9d demonstrates that A\_480-1 exhibits both a basketweave structure with fine α and β plates, and a much finer α'. Similar to the XRD result and JMatPro calculation (Figure[s 6](#page-6-0) and [7\)](#page-7-0), for the extreme early aging condition 480-1, the transformation of  $\alpha'$  into  $\alpha$  and  $\beta$  basketweave structures begins, but this condition is insufficient to complete the transformation. In the case of A\_530-120 shown in [Fi](#page-8-0)gure 9f,  $\alpha'$  is fully transformed into α and β plates, which is also confirmed in the XRD result [in](#page-6-0) Figure 6. Ti<sub>3</sub>Al is too small to be clearly distinguished from the surroundings in Figure 9f, but because of the XRD results (Figure 6) and regardi[ng](#page-8-0) Lütjer[ing](#page-15-18) [11], in A\_530-120, Ti<sub>3</sub>Al clearly exists in the α phase, both of equiaxed α and α plates, with a coherently elliptical shape. A\_6[30-](#page-8-0)480 in Figure 9h has much coarser α and β plates than those of A\_530-120 because of the higher growth rate of each plate under the higher temperature and longer aging duration. Also, Ti<sub>3</sub>Al does not precipitate under  $A_630-480$ ; this was not detected in [XR](#page-6-0)D (Figure 6).

<span id="page-8-0"></span>

Figure 9. SEM microstructures of duplex microstructure (a,c,e,g) and enlarged  $\alpha'$  and/or basketweave structure  $(b,d,f,h)$  in the area marked in red box; (a,b) are ST, (c,d) are A\_480-1, (e,f) are A\_530-120, A\_530-120, and (**g**,**h**) are A\_630-480. and (**g**,**h**) are A\_630-480.

Average grain size and plate thickness from Figures  $8$  and  $9$  were measured and the results are shown in Figure [10.](#page-9-0) The average grain size increases by 15% with increasing aging temperature and time after ST. During aging,  $\alpha'$  in ST is transformed into  $\alpha + \beta$ , and the plates become coarser at the higher aging condition. As shown in Figure 5, when the aging temperature is increased, the rate of the α'-to-α + β transformation accelerates. Moreover, according to J. Chen et al.  $[34]$ , at the higher temperature, the growth rate of transformed plates significantly increases. When aged at the 630–480 condition, the longest holding time at the highest temperature in this study, the thickest  $\alpha$  plate is exhibited, which is more than five times thicker than ST. This growth in grain size and plate thickness during STA shows a similar trend, as shown in Figure [5b](#page-5-0)–d.

 $\mathcal{A}$  and plate thickness from Figures 8 and 9 were measured and 9 were measured and 9 were measured and the  $\mathcal{A}$ 

<span id="page-9-0"></span>

**Figure 10.** Average value of grain size and  $\alpha$  and/or  $\alpha'$  plate thicknesses of ST and STA samples.

### **Figure 3.3.3. Element Redistribution**

across the duplex phases under various STA conditions. Al is one of the  $\alpha$ -stabilizing elements, and V is one of the β-stabilizing elements [11,18]. Therefore, V tends to exist more in the β phase rather than in the α phase. In the case of equiaxed α, the Al concentration in Figure 11a–d ranges from 5.5 to 6 wt.%, similar to the average element composition. However, the V content in equiaxed  $\alpha$  ( $\alpha_E$ ) is low, at 2 to 2.5%, which is significantly lower than the average V content of 4.15 wt.% for the material. In addition, when comparing the Al and V concentrations in equiaxed  $\alpha$  after aging, there is little difference in concentration even if the aging temperature and time increase. Figure [11a](#page-10-0)–d present the line mapping results of EPMA for Al and V composition

The  $\alpha'$  phase observed in the ST (Figure 11a) contains approximately 5.5 to 6 wt.% of V and 4 to 4.5 wt.% of Al. Because  $\alpha'$  is transformed from β, and due to the fast cooling of WQ (approximately 100 °C/s), it was difficult for the V distribution during cooling, so the V concentration in  $\alpha'$  (average 5.5 wt.%) is higher than the average V concentration of the material (4.15 wt.%). The gradient of V concentration in  $\alpha'$  phase is 0.8 wt.%, which is calculated as the difference between the maximum and minimum values of V concentration ( $V_{Max}^{\alpha'} - V_{Min}^{\alpha}$ ). During the aging,  $\alpha'$  is transformed into  $\alpha$  and β phases and V is redistributed to the β plate [\[20\]](#page-15-7). According to Kherrouba et al. [20], as the holding temperature and time are increased, the V concentration in the  $\beta$  plate is increased. Therefore, as shown in Figure  $11b-d$  $11b-d$ , when the aging condition increases the temperature and time from A\_480-1 to A\_630-480, the redistributed amount of V to the β plate is larger, so the difference in V concentration in the basketweave structure,<br> $\frac{1}{h}$  $V_{Max}^b - V_{Min}^b$  is increased. The maximum difference in the basketweave structure of A\_630-<br>420  $\frac{1}{2}$  and 11.1 and 220  $\frac{1}{2}$  (comparison) in the state of the sta 480 (Figure [11d](#page-10-0)) is 1.98 wt.%, which is about twice higher than the value of  $\alpha'$  of ST. The changes in *V* composition in *θ*, plates according to the aging condition about a similar trand changes in V composition in β plates according to the aging condition show a similar trend,  $\epsilon$ as shown in Figure [5a](#page-5-0)–d, which can affect the mechanical properties.

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**Figure 11.** The microstructures with the EPMA test line, which is yellow arrow, and the variation in **Figure 11.** The microstructures with the EPMA test line, which is yellow arrow, and the variation in Al and V profiles; (**a**) ST, (**b**) A\_480-1, (**c**) A\_530-120 and (**d**) A\_630-480. Al and V profiles; (**a**) ST, (**b**) A\_480-1, (**c**) A\_530-120 and (**d**) A\_630-480.

# *3.4. Mechanical Properties*

3.4.1. Change in Phase Hardness

Figure [12](#page-11-0) presents the average Vickers hardness results for equiaxed  $\alpha$  and  $\alpha'$  and/or basketweave structure in the duplex microstructure. Regarding the change in the hardness value of equiaxed  $\alpha$ , the difference between ST and aging conditions is not very large. But in the case of aging at  $A_530-120$ , the hardness is the highest, which is attributed to  $Ti<sub>3</sub>Al$ precipitation. In a similar study, it was found that when  $Ti<sub>3</sub>Al$  is precipitated, the hardness is increased because of the higher resistance of dislocation movement at the precipitated phase [\[11,](#page-15-18)[35,](#page-15-23)[36\]](#page-15-24).

<span id="page-11-0"></span>

**Figure 12.** Average micro-Vickers hardness value of each phase consisting of a duplex microstructure.

The hardness of  $\alpha'$  in ST is higher than that of equiaxed  $\alpha$ . This is because of the formed by WQ [\[30\]](#page-15-17). The hardness of  $\alpha'$  or the basketweave structure of A\_480-1 showed a  $\frac{1}{2}$  structure of A\_530-120 is the hardest, not only because the transformation from  $\alpha'$  to fine  $\alpha$  $+ \beta$  is completed, but also because of the presence of Ti<sub>3</sub>Al [\[35,](#page-15-23)[36\]](#page-15-24). In other words, in the  $\alpha$  p is completed, but also because of the presence of  $T_3$ . The specific words, in the case of A\_530-120, many  $\alpha/\beta$  grain boundaries and precipitated phases blocked dislocation movement. Conversely, the basketweave structure in A\_630-480 has the lowest hardness movement. movement. Conversely, the basketweave structure in  $Y_{\text{1000}}$  -100 has the lowest hardness value due to the large size of  $\alpha$  and  $\beta$  plates and the absence of Ti<sub>3</sub>Al. Regarding the plate presence of the fine acicular plate of about 50 nm in size, with high dislocation density similar hardness with  $\alpha'$  of ST due to insufficient transformation time. The basketweave thickness and mechanical properties, it is known that when the plate thickness is coarse, the amount of  $\alpha/\beta$  boundary becomes smaller, making it easier to move dislocations, and hardness is decreased [\[31\]](#page-15-19). For this reason, in this study, the basketweave structure with the coarsest  $\alpha$  plate of A\_630-480 showed the lowest hardness value compared to other conditions. Consequently, in the case of A\_630-480, the hardness values of the equiaxed  $\alpha$  and the basketweave structure are the most similar. This similar hardness of phases in A\_630-480 allows for relatively uniform deformation under tensile loading and can suppress stress localization, which can have a positive effect on the tensile behavior.

### 3.4.2. Tensile Properties

Figure [13a](#page-12-0) shows the engineering stress–strain curves obtained during the tensile test and Figure [13b](#page-12-0) shows the yield strength, elongation and modulus of toughness for each STA condition obtained from the curves; the specific values are in Table [2.](#page-12-1) Compared with ST, all of the aged samples reveal higher yield strength and elongation due to the transformation of α<sup>'</sup> into α and β. A\_530-120 has the highest yield strength and slightly higher elongation than ST, not only because of the transformation from  $\alpha'$  to fine  $\alpha + \beta$  [\[21\]](#page-15-8), but also because of precipitated T<sub>13</sub>Al [\[35,](#page-15-23)[36\]](#page-15-24). A<sub>-6</sub>30-480 has a 6.84% higher yield strength than ST and the yield strength is similar with A\_530-120. In addition, A\_630-480 has the highest elongation, and its value is 19.10%, which is 21.66% higher than ST, and 16.46% higher than A 530-120. This is because A 630-480 has coarse  $\alpha$  and  $\beta$  plates as well as none of the  $Ti<sub>3</sub>Al phase$ , so it has the least obstacles to dislocation movement, making it easier for dislocation movement [\[31](#page-15-19)[,37\]](#page-15-25).

Also, the area under the stress–strain curve of Figure [13a](#page-12-0) indicates the modulus of toughness according to the combination of strength and elongation, and refers to the energy required to fracture a material. As can be seen in Figure [13b](#page-12-0) and Table [2,](#page-12-1) the modulus of toughness for aged samples is higher than that of ST. Among the several aging conditions, A\_630-480 has the maximum modulus of toughness, which is 26.18% higher than that of ST, and 13.70% higher than that of A\_530-120. The microstructure of A\_630-480 has a basketweave structure with coarse plates, so the amount of  $\alpha/\beta$  interface is the smallest; therefore, the resistance of dislocation movement is relatively easy. As can be seen in

Figure [12,](#page-11-0) because the hardness values of equiaxed alpha and basketweave in the duplex microstructure are similar, it is also believed that during the tensile test, stress was not concentrated on a specific phase and was deformed relatively uniformly over the entire area of the specimen.

<span id="page-12-0"></span>

strength, elongation and modulus of toughness of each condition obtained from (**a**). **Figure 13.** (**a**) Engineering stress–strain curves of STA during tensile test and (**b**) graph for yield



<span id="page-12-1"></span>**Table 2.** Tensile properties of ST and various aging conditions.

For the safety of the UAM industry using Ti64, toughness is one of the most important properties. Therefore, aging at 630 °C for 480 min (A\_630-480) is the optimal aging condi $r_{\text{ion}}$  because it increases toughness by  $26\%$  with adapted bigh strength and tion because it increases toughness by 26% with adequate high strength and elongation<br> $\frac{1}{2}$ compared to the ST condition.

### $\overline{A}$ 3.4.3. Fractographies

Figure [14](#page-13-0) shows fractographies of ST and STA. In Figure [14a](#page-13-0)–d, both cleavage and dimple structures are observed, indicating a mixture of brittle and ductile fracture patterns.

Figure [14e](#page-13-0)–h are enlarged specific areas in Figure [14a](#page-13-0)–d. In Figure [14e](#page-13-0), the ST exhibits some cleavage structures surrounded by cracks. The high dislocation density hinders dislocation and slip movements in  $\alpha'$ , causing it to be unable to withstand deformation under tensile load, which leads to cleavage fracture [\[31,](#page-15-19)[38,](#page-15-26)[39\]](#page-16-0). A\_480-1 has a very fine basketweave structure and  $\alpha'$ ; so, Figure [14f](#page-13-0) shows a mixed fracture pattern similar to that of ST, as shown in Figure [14e](#page-13-0). In Figure [14g](#page-13-0), the fractography of A\_530-120 exhibits various sizes of dimple structures, and small dimples are observed even within the area surrounded by cracks and cleavage facets. These small dimples are made from fracturing  $\alpha$  and  $\beta$  plates in a basketweave structure. In Figure [14h](#page-13-0), A\_630-480 exhibits some tear ridge patterns with various sizes of dimple structures [\[40\]](#page-16-1). This tear ridge patterns were formed from β plates between α. The β plate in A\_630-480 has an excessively high V concentration, as shown in Figure [11d](#page-10-0), and that β plate is surrounded by coarse α plates. According to *Metals* **2024**, *14*, x FOR PEER REVIEW 15 of 17 J.M. Oh et al. [\[38\]](#page-15-26), the hardness increases as the concentration of the contained elements increases. This means that in A\_630-480, the β plate with a higher V concentration is stronger than the surrounding  $\alpha$  plate with a lower V concentration in the basketweave structure. Therefore, during the tensile test, the  $\alpha$  plates in the basketweave structure of A\_630-480 are sufficiently deformed, while the β plates between the α plates act as a tough tissue, which suppresses failure due to severe deformation. Consequently, A\_630-480 has the highest modulus of toughness with adequate high strength and high elongation. the **ingle** 

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Figure 14. Fractographies of STA after tensile test; (a-d) are ST, A\_480-1, A\_530-120, and A\_630-480, respectively, and  $(e-h)$  are enlarged views of the indicated area in  $(a-d)$ , respectively.

# **4. Conclusions**

This study investigated the microstructural and mechanical properties according to heat treatment conditions in various STA (ST, A\_480-1, A\_530-120, A\_630-480) conditions to determine the optimal STA heat treatment conditions to obtain high toughness with high strength of Ti64 for the UAM industry. The following conclusions were obtained:

All selected STA conditions exhibited a duplex microstructure consisting of equiaxed α, and  $\alpha'$  and/or  $\alpha + \beta$  basketweave structures. As the aging conditions increase, the thickness of the  $\alpha$  plate and concentration difference in the V element become larger. In the case of aging at 630 °C for 480 min, the thickness of the  $\alpha$  plate is approximately five times coarser than that of ST, and the difference in V concentration between β and  $\alpha$  plates in the basketweave structure is 1.98%; this value is approximately 2.5 times higher than ST.

Regarding the hardness of equiaxed  $\alpha$  and  $\alpha'$  and/or basketweave under STA conditions, when aged at 530 °C for 120 min after ST, both phases showed the highest hardness. After aging at 630  $\degree$ C for 480 min, the hardness of the basketweave structure decreased rapidly, so the hardness difference according to each phase is insignificant.

Aged Ti64 showed improved yield strength and elongation than the solution-treated sample. The highest strength was exhibited in A\_530-120 due to fine  $\alpha + \beta$  and T<sub>13</sub>Al precipitation. Toughness, which is the highest combination of strength and elongation, was highest in A\_630-480; this value was approximately 26.18% higher than just ST. The β plates between coarse  $\alpha$  plates with sufficiently high V in A 630-480 appeared in a tear ridge pattern, and played a role in withstanding large deformation and suppressing the fracture.

Aging Ti64 at 630 °C for 480 min (A\_630-480) is the best condition as it enhances toughness by 26% with sufficiently high yield strength (1053.40 MPa) and elongation (19.10%) compared to the ST condition.

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