Suppression of Grain Boundary α Formation by Addition of Silicon in a Near- β Titanium Alloy

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The effect of Si addition on the microstructure and mechanical properties of a near- β titanium alloy Ti-17 with fully lamella microstructure was investigated. It was found that the microstructure of Ti-17 with silicon exhibited the absence of a continuously thick α layer along prior- β grain boundaries (grain boundary α), while the grain boundary α was distinctively formed in Ti-17 without Si. The formation of (Ti, Zr) silicide particles at the prior- β grain interior and its grain boundaries were observed, and the presence of these particles were related to the disappearance of grain boundary α similar to the oxygen scavenging effect of boride particles reported previously. With regard to mechanical properties, Ti-17 with silicon exhibits higher strength and lower ductility compared with Ti-17 without Si. Ductile transgranular fracture morphology was observed even on the fracture surface of Ti-17 with Si after tensile test. [doi:10.2320/matertrans.ME201920]

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1. Introduction

Ti-5Al-2Sn-2Zr-4Mo-4Cr alloy (Ti-17) is a β -rich two-phase ($\alpha + \beta$) titanium alloy (near- β titanium alloy) used for fan and compressor discs in jet engines.¹⁾ These components are spun with blades to meet both high strength and high fracture toughness requirements. In this case, a thermo-mechanical processing route to form fully lamella microstructure, which is advantageous for obtaining high fracture toughness,^{2,3)} is often utilized for Ti-17 material fabrication.⁴⁾ In the case of fully lamella microstructure, it is generally known that the most influential microstructural parameter of mechanical properties is the α colony size because it determines the effective slip length; yield stress and ductility are likely to decrease, while crack propagation rate decreases with increasing α colony size.⁵⁾ Further, the continuously thick α layer at prior- β grain boundaries (grain boundary α) cannot be ignored because it has a negative effect on the mechanical properties of Ti-17 with fully lamella microstructure. The grain boundary α enhances the deterioration of ductility⁶⁾ and fatigue properties⁷⁾ for Ti-17.

Grain boundary α is difficult to eliminate completely; hence, fabricators make an effort to form discontinuous and zigzag-shaped grain boundary α by thermomechanical processing.⁶⁾ A unique method using oxygen scavenging by boride particles has also been proposed for inhibiting the formation of grain boundary α in titanium alloys.^{8,9)} The solid solubility of boron (B) is limited to a low concentration at any temperature, and thus almost all B included in titanium alloys is precipitated as boride particles. These boride particles are formed during solidification by a eutectic reaction with Ti. Then, they scavenge the oxygen solute, which is an inevitable impurity and is classified as a strong

α stabilizer in titanium alloys. In a usual case, the grain boundary α can form at the beginning of β to α phase transformation along prior- β grain boundaries.¹⁰⁾ However, if boride particles are present before phase transformation, the oxygen distribution is inhomogeneous and it has high concentration near the boride particles. In particular, the boride particles can scavenge the oxygen solute from prior- β grain boundaries more strongly than from β grain interior because atomic diffusion tends to be faster at the grain boundary than in the matrix. As a result, local α stability increases near boride particles and decreases at prior- β grain boundaries; this enhances α phase precipitation near boride particles instead of the grain boundary α formation.

Silicon (Si) could play a role similar to B in inhibiting grain boundary α formation in titanium alloys because of its low solid solubility limit, and silicide forms during solidification by a eutectic reaction with Ti. However, because Si addition is effective in improving creep resistance at elevated temperature, previous studies have focused on the effects of Si addition on the microstructure and mechanical properties of α and near- α titanium alloys.¹⁾ Therefore, the effects of Si addition on microstructure and mechanical properties, especially grain boundary α formation, and its effect on tensile properties were investigated for one of the practical near- β titanium alloys, Ti-17, in this study.

2. Experimental

2.1 Samples preparation

Ingots of Ti-17 including 0, 0.3, and 0.6 mass% Si (Ti-17-0Si, Ti-17-0.3Si, and Ti-17-0.6Si, respectively) were prepared by cold crucible levitation melting using an as-received Ti-17 hot-forged disc and pure silicon grains, which were added at the appropriate proportion in high-purity argon atmosphere. The obtained ingots were homogenized at 1473 K for 21.6 ks in an argon atmosphere, and subsequently

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Fig. 1 Preparation procedure of samples of Ti-17 alloys including Si in this study.

quenched in ice water as shown in Fig. 1. However, Ti-17–0.6Si was broken during this quenching, while the other ingots did not crack. Therefore, only Ti-17–0Si and Ti-17–0.3Si were available for the following experiments. It has been reported that appropriate amounts of Si addition to titanium alloys increases the tensile strength while maintaining moderate ductility.¹¹⁾ However, excess Si addition decreases the ductility of titanium alloys drastically when the silicide particles form a network along the prior β grain boundaries, inducing intergranular embrittlement.^{12,13)} Therefore, some researchers optimized the Si content in each titanium alloy, but the reported optimum contents are not unified in the range of less than 1 mass%,^{12–14)} indicating that it probably depends on the type of alloys. The Ti-17–0Si and Ti-17–0.3Si ingots were heated in argon atmosphere at 1188 K and then hot forged into cuboid and hot rolled into plates with approximately 80% reduction in thickness in air. It is reported that the $\beta/(\alpha + \beta)$ transformation temperature (β -transus) of Ti-17 is 1163 K¹⁵⁾. Therefore, the samples were cooled through the β -transus of this alloy during hot forging and rolling. The hot-rolled plates were solution-treated at 1073 K for 14.4 ks, and then aged at 908 K for 28.8 ks followed by air cooling.

2.2 Microstructural analyses

The microstructures of Ti-17–0Si and Ti-17–0.3Si were observed using an optical microscopy. The constituent phases and alloying elemental distributions were analyzed by X-ray diffraction (XRD) and field-emission electron probe microanalysis (FE-EPMA), respectively. The specimens for the observation and analysis were polished mechanically using SiC waterproof emery papers with grit numbers of up to 2400, and subsequently buff-polished by a colloidal SiO₂ suspension in order to obtain a mirror surface. After polishing, the specimens were etched using HF + HNO₃ aqueous solution.

2.3 Mechanical tests

Vickers hardness tests were carried out under constant load of 4.9 N and holding time of 15 s. Tensile tests were performed on each alloy at room temperature using an Instron-type machine at a cross-head speed of $8.33 \times 10^6 \text{ m s}^{-1}$. The specimens for the tensile tests were 1.5 mm thick, 3 mm wide, and 13 mm long in the section, and were cut using a wire electric-discharge machine. The tensile

specimens were polished using SiC waterproof emery papers with grit numbers of up to 2400 before the tests. After the tensile tests, the fracture surfaces were observed and analyzed by a scanning electron microscope equipped with an energy-dispersive X-ray analyzer (SEM/EDX).

3. Result and Discussion

3.1 Microstructures of Ti-17 including Si

Figure 2 shows the microstructures of Ti-17–0Si and Ti-17–0.3Si observed by optical microscopy. Both alloys exhibit fully lamella microstructure. The prior- β grain sizes of these alloys were not significantly different, while the size homogeneity for Ti-17–0Si was relatively lower than that for Ti-17–0.3Si, as shown in low-magnified micrographs. Further, the grain shapes were not elongated for both alloys. These results indicate that dynamic recrystallization of β -grains occurred during hot forging and hot rolling; thus, as-solidified microstructures disappeared but silicide slightly affected nucleation during this recrystallization. According to the high-magnified micrographs, the greatest difference between the microstructures of these alloys is the prior- β grain boundary condition. A continuously thick α layer along the prior- β grain boundaries, that is grain boundary α , was formed in Ti-17–0Si, while the α layer was not observed in Ti-17–0.3Si. Further, α colony was not clearly observed; hence, the effective slip length is probably dominated by the prior- β grains for both alloys. It is also difficult to distinguish the presence of silicide in Ti-17–0.3Si by this observation.

Figure 3 shows the XRD profiles of Ti-17–0Si and Ti-17–0.3Si. Both α and β phases were detected, and no significant difference between these alloys was observed. Silicide was not detected in Ti-17–0.3Si by XRD.

Figure 4 shows the alloying element distributions in Ti-17–0Si and Ti-17–0.3Si analyzed by FE-EPMA. The α stabilizing elements such as Al and Ti were enriched, while the β stabilizing elements such as Mo and Cr were depleted in intragranular α lamellae and grain boundary α . Sn is a neutral element; hence, its distribution was homogenized in both alloys. On the other hand, Zr and Si distributions in Ti-17–0.3Si are distinctive; Zr was distributed at locations where Si exists inside prior- β grains and at prior- β grain boundaries in Ti-17–0.3Si, while Zr was not enriched anywhere in Ti-17–0Si. This result indicates that small (Ti, Zr) silicide

Fig. 2 Optical micrographs of (a) Ti-17-0Si and (b) Ti-17-0.3Si.

Fig. 3 XRD profiles of Ti-17-0Si and Ti-17-0.3Si.

particles were probably formed inside prior- β grains and at prior- β grain boundaries. Further, Zr and Si concentrations were continuously high along prior- β grain boundaries. However, the (Ti,Zr) silicide particles were not continuous at prior- β grain boundaries. According to the XRD results (Fig. 3), only α and β phases of titanium alloys were identified; no other phase was detected. Therefore, the amount of (Ti,Zr) silicide is not large, and the type of (Ti,Zr) silicide was not identified in this study. In previous reports, two types of silicides were identified in near- α titanium alloys: S1 type $(\text{Ti,Zr})_5\text{Si}_3$ ^{11,16)} and S2 type $(\text{Ti,Zr})_6\text{Si}_3$.^{16,17)} These silicides formed depending on the Zr content of the particles.¹⁶⁾

It has been reported that the grain boundary α disappears in cast Ti-6Al-4V with B addition because boride particles

were formed during solidification and play a role in getting oxygen, which leads to high oxygen concentration locally near the boride particles and scavenging oxygen from the prior- β grain boundaries. As a result, the primary α phase may start to form near boride particles during cooling from the β phase region at high temperature above general β -transus for Ti-17, because oxygen is a strong α stabilizing element.^{8,9)} In general, scavenging effect can be obtained by the addition of alloying element whose affinity for oxygen is stronger than base element. In the case of titanium alloys, rare earth elements are employed as the alloying element for scavenging oxygen.¹⁸⁾ Further, it has been reported recently that the rare earth elements were introduced using unstable rare earth compounds such as hydride, boride, silicide, etc.¹⁹⁾ The results of *in situ* synchrotron radiation experiments revealed that yttrium hydride decomposed to metallic yttrium and then yttrium oxide was formed by scavenging oxygen simultaneously in pure Ti at 1373 K and Ti-6Al-4V at 1573 K.²⁰⁾ Further, according to the Gibbs free energies for formation of various metallic oxides, the affinity of Zr for oxygen is stronger than that of Ti.²¹⁾ The result of thermodynamic calculation also indicated that Zr can form oxide preferentially at lower oxygen partial pressure than Ti and Si in Ti-5Zr-(0–0.1)Si system at 1093 K.²²⁾ Therefore, a part of (Ti,Zr) silicides could decompose and then Zr is possible to play a role as a scavenger in Ti-17-0.3Si in this study. As another possible method to obtain scavenging effect, using metastable or substoichiometric oxygen-stabilized titanium compound such as Ti_2C was proposed;^{23,24)} The Ti-C phase diagram suggests that TiC is the equilibrium phase while Ti_2C is not. The Ti_2C particles in titanium alloys

Fig. 4 Alloying element distributions in (a) Ti-17-0Si and (b) Ti-17-0.3Si analyzed by FE-EPMA.

Fig. 5 (a) Vickers hardness and (b) tensile properties of Ti-17-0Si and Ti-17-0.3Si.

could scavenge oxygen via the formation of oxygen-stabilized Ti_2C particles. As the results, local oxygen concentrations at prior- β grain boundaries were reduced, resulting in the suppression of grain boundary α formation. The role of titanium boride in scavenging oxygen seems to be similar to that of Ti_2C . In the Ti-Si phase diagram, α and β phases are equilibrium with Ti_3Si_3 below 1444 K,²⁵⁾ Ti_3Si_3 is a metastable phase so that it is possible to work as a scavenger in Ti-17-0.3Si in this study.

3.2 Mechanical properties of Ti-17 including Si

Figure 5 shows the Vickers hardness and tensile properties of Ti-17-0Si and Ti-17-0.3Si. The hardness of Ti-17-0.3Si is slightly higher than that of Ti-17-0Si. The 0.2% proof stress and tensile strength are higher for Ti-17-0.3Si than for Ti-17-0Si. The elongation of Ti-17-0.3Si is lower than that of Ti-17-0Si.

Figure 6 shows the SEM fractographs of Ti-17-0Si and Ti-17-0.3Si after tensile tests. Ductile transgranular fracture

Fig. 6 SEM fractographs of (a) Ti-17-0Si, (b) Ti-17-0.3Si (low magnification), and (c) Ti-17-0.3Si (high magnification) after tensile tests, and (d) result of EDS analysis at an analyzed point shown in (c).

morphologies were observed in the fracture surfaces of both alloys, while some particles were observed inside dimples only in Ti-17-0.3Si. The result of EDX analysis reveals that Zr and Si concentrations were high at such the particles in the dimples so that these particles are considered to be the (Ti,Zr) silicide. Therefore, these (Ti,Zr) silicide particles may contribute to strengthening Ti-17-0.3Si. With regard to Ti-17-0Si, transgranular fracture occurred in the tensile tests, although continuously thick grain boundary α was formed. It has been reported that grain boundary α dominates ductility for high-strength β titanium alloys with unrecrystallized and elongated β grains including α lamellae; tensile ductility decreases drastically when stress is applied at 45° orientation from the longitudinal axis of the elongated β grains.²⁶⁾ It is believed that such ductility anisotropy is caused by both the softer grain boundary α than the matrix and larger effective slip length due to the elongated β grain geometry. According to microstructural observations (Fig. 2), the recrystallization of β grains probably occurred and then prior- β grain shape was not elongated in this study. Therefore, fracture did not occur at grain boundary α , specifically in Ti-17-0Si.

4. Conclusion

The effect of Si addition on the microstructure and mechanical properties of the near- β titanium alloy Ti-17 with fully lamella microstructure were investigated. The following results were obtained.

- (1) The microstructure of Ti-17 with Si showed the absence of continuously thick α layer along prior- β grain boundaries (grain boundary α), while the grain boundary α was distinctively formed in Ti-17 without Si.

- (2) (Ti,Zr) silicide particles formed in the prior- β grain interior and its grain boundaries. The presence of these particles was related to disappearance of grain boundary α , similar to the oxygen scavenging effect of boride particles reported previously.
- (3) Ti-17 with Si exhibits higher strength and lower ductility compared with Ti-17 without Si. Ductile transgranular fracture morphology was observed even in the fracture surface of Ti-17 with Si.

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