Relationship between Microstructures and Mechanical Properties in Ti–4.5Al–2Mo–1.6V–0.5Fe–0.3Si–0.03C for Next-Generation Aircraft Applications*1

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The effects of various heat treatments on the microstructure and mechanical properties of an (α + β)-type titanium alloy, Ti–4.5Al–2Mo–1.6V–0.5Fe–0.3Si–0.03C (KS Ti-9), were investigated systematically for possible use in next-generation aircraft applications.

The as-received KS Ti-9 shows strong anisotropy of mechanical strength derived from the intense texture (T-texture) of the primary α phase. This anisotropy continues to be observed in KS Ti-9 annealed at a temperature below β transus followed by air cooling, but the anisotropy drastically decreases in KS Ti-9 when subjected to a duplex heat treatment, that is, when the as-received material of KS Ti-9 is sequentially heated to a temperature slightly below β transus followed by water quenching and then annealed followed by air cooling. It is considered that the reduction in the anisotropy in KS Ti-9 subjected to this duplex heat treatment is associated with the difference in the orientation of the precipitated acicular α phase.

Keywords: (α + β)-type titanium alloy, tensile properties, texture, anisotropy

1. Introduction

Research and development have been conducted on the application of an (α + β)-type titanium alloy (Ti–4.5Al–2Mo–1.6V–0.5Fe–0.3Si–0.03C (KS Ti-9)) to next-generation aircraft as this alloy can be rolled into coils of thin sheets, while maintaining a strength equivalent to that of the Ti–6Al–4V alloy (Ti-64), which is widely used for aircraft applications.1) Compared with Ti-64, which requires pack rolling in order to be fabricated into thin sheets, KS Ti-9 can be rolled with considerable ease at much lower cost.1) However, a significant problem with this material is its anisotropy upon hot rolling or hot rolling followed by cold rolling to an arbitrary thickness. The tensile strength, 0.2% proof stress and elongation differ considerably between the longitudinal direction of the rolling (L-direction) and the transverse direction (T-direction),2) although they satisfy the minimum requirements for a Ti-64 sheet under AMS (Aerospace Material Specification) 4911L. This differing degree of strength between the L- and T-directions (the material is weaker in the L-direction) is readily apparent upon both hot and cold rolling. It is reported to result from the formation of a T-texture (a texture characterized by the strongly preferential alignment of the c-axis of the primary α phase in the T-direction), as slabs are hot rolled in a single direction on a continuous rolling mill to a thickness of approximately 4.0 mm.3) It has also been reported that Young’s modulus in an α-phase titanium single crystal, which has a hexagonal close-packed structure and thus a low degree of crystallographic symmetry, depends on crystallographic orientation. The Young’s moduli of parallel and perpendicular to the c-axis show around 145 G and 100 GPa, respectively.3) There is concern that upon forming such as bending sheets of the alloy into shapes for use in aircraft applications. The different strengths and Young’s modulus resulting from the textured structure occurs the change in workability and spring back. As the result, there are some wrinkles on the surface of sheets. Thus, there is significant interest in reducing the anisotropy of the alloy for fabricating the high-quality products for use in such applications.

In this study, the effects of microstructures, particularly anisotropy (texture), on the mechanical properties of KS Ti-9 annealed at various temperatures were systematically examined. On the basis of these results, the effectiveness of heat treatments intended to reduce the degree of anisotropy was then examined.

2. Experimental Procedures

2.1 Material and heat treatments

The material used in this study was a hot rolled plate of Ti–4.5Al–2Mo–1.6V–0.5Fe–0.3Si–0.03C (KS Ti-9) with a thickness of 4.5 mm (referred to as as-received materials), which was fabricated from its ingot with a diameter of 840 mm. This ingot was hot forged at 1473 K and then hot rolled at 1223 K to form a sheet with a thickness of 4.5 mm. Table 1 shows the chemical compositions at the top and bottom parts of the ingot. The sheet was machined into blank materials with a length of 15 mm, a width of 15 mm and a thickness of 4.5 mm. To obtain blank samples having different volume fractions of the primary α phase, a number of the above blank materials were annealed in a vacuum for 3.6 ks at temper-
atures ranging from 973 to 1248 K, after which they were air cooled. These blank materials are collectively referred to as “annealed materials”. Similarly, to obtain blank samples with reduced anisotropic characteristics, a number of the above blank materials were heat treated by solution treatment in a vacuum at 1248 K for 3.6 ks followed by water quenching and then annealing in a vacuum at 973 K for 7.2 ks followed by air cooling (referred to as duplex heat treatment). These blank samples are collectively referred to as “duplex heat-treated materials”.

2.2 Microstructural evaluation

The microstructural observations of the as-received materials, annealed materials and duplex heat-treated materials were carried out by optical microscopy. Samples for microstructural observations were prepared by polishing them to a mirror finish by wet polishing with up to #1500 emery papers, followed by buffing with a silica suspension, and then etching them via immersion in an aqueous solution of 2% HF and 5% HNO₃ at room temperature. Square samples with a size of 5.0 mm × 5.0 mm × 3.0 mm were machined from the as-received materials, annealed materials and duplex heat-treated materials; these samples were then wet polished with up to #1500 emery papers and analyzed by X-ray diffraction along their rolled planes. Pole figures were created with the resulting data. X-ray diffraction was carried out with a Cu–Kα X-ray source under a tube voltage of 40 kV and a tube current of 40 mA.

Other square samples were machined from the as-received materials and annealed materials; these samples were polished to a mirror finish by wet polishing with up to #1500 emery papers, followed by buffing with a silica suspension. The crystal orientations of these samples were then evaluated by electron backscatter diffraction (EBSD) with an accessory mounted on a field emission scanning electron microscopy (FE-SEM) apparatus. Measurements were taken under an electron beam acceleration voltage of 15 kV and a spot size of 80 nm.

2.3 Tensile testing

For tensile testing, plate tensile specimens with a thickness of 1.5 mm and a gauge length of 13 mm were first machined from the as-received materials, annealed materials and duplex heat-treated materials; the other dimensions of the plate tensile specimens are shown in Fig. 1. The surface of the plate tensile specimen was then wet polished with up to #1500 emery paper. Tensile testing was then performed on the plate tensile specimens using an Instron-type testing machine with a capacity of 19.6 kN at a crosshead speed of $8.33 \times 10^{-6} \text{ m s}^{-1}$ at room temperature. Load detection was conducted with a load cell mounted on the machine, and strain was measured by means of a strain gauge attached to the sample at the part in the gauge length. Elongation to fracture was measured by a reading microscope and was calculated the change in the gage length before and after the tensile test. Fractured surfaces were cut away from fractured tensile specimens and examined by SEM after ultrasonic cleaning with acetone.

3. Results and Discussion

3.1 Microstructures after annealing

Figure 2 shows the optical micrographs of the as-received and annealed materials. The rolling direction, the direction of the perpendicular direction to the rolling direction, and the direction to the thickness were defined as L, T and S directions, respectively in this study. Furthermore, the planes consisted of L and T directions, L and S directions and S and T directions were defined as L-T, L-S and S-T planes, respectively. The white and black regions in the microstructures are the α and β phases, respectively. The microstructure of the as-received material was highly elongated in the L-direction (i.e., the rolling direction). The length of the short axis of the primary α phases on the S-T and L-S planes was about 1.0 µm. In the annealed material, the primary α phase tends to be more spheroidized with increasing annealing temperature; the degree of the spheroidization is apparently the greatest in the annealed material at 1223 K, which is close to β transus (1236 K). Apart from the primary α phase, an (α + β) phase is also apparent within the microstructures of the materials annealed at a temperature between 1173 and 1223 K; this phase consists of an acicular α phase precipitated within the β phase, and the length of the short axis of the acicular α phase increases slightly with increasing annealing temperature. The microstructure of the material annealed at 1248 K, above β transus (1236 K), reveals that the primary α phase disappears and coarse prior β grains with a diameter of around 500 µm, where the acicular α phase precipitates and grain boundary α phase formed along the boundaries of the prior β grains appears.

Figure 3 shows the volume fraction of the primary α phase measured on the S-T plane as a function of the annealing temperature. The volume fraction of the primary α phase measured on the S-T plane of the as-received material is
approximately 65%. Furthermore, the volume fraction of the primary $\alpha$ phase measured on the S-T plane decreases with increasing annealing temperature, and remarkably decreases from 35 to 12% in the temperature raising from 1173 to 1223 K.

Figure 4 shows (0002) pole figures for the $\alpha$ phase and relationship between the hot rolling temperature and texture of $(\alpha + \beta)$-type titanium alloys. $^3$ It has been reported that this is caused by the preferential precipitation of the $\alpha$ phase showing one selected variant among the crystallographic-orientation relationship (Burgers orientation relationship) of $\{110\}_\beta // \{0002\}_\alpha$, $\{110\}_\beta // \{1120\}_\alpha$ maintained upon an allotropic transformation from the $\beta$ phase to the $\alpha$ phase. $^3,6)$ Since the hot...
rolling was carried out at 1223 K, which is a temperature within the $\alpha + \beta$ region near $\beta$ transus, where the resistance to the deformation is low so as to maintain equiaxed structure and workability in this study, T-texture mentioned above is considered to be formed.

The crystallographic orientation of the $\alpha$ phase in the material annealed at 973 K exhibits almost no change as compared to those in the as-received material and in the material annealed at 1223 K, near $\beta$ transus. As can be also seen in the microstructural change, the volume fraction of the primary $\alpha$ phase in the material annealed at 1223 K has been only 12%, which is substantially less than that of the as-received material. This suggests that the $\alpha$ phase formed upon air cooling after annealing at 1223 K have the same crystallographic orientation as the primary $\alpha$ phase. On the other hand, the material annealed at 1248 K, which is a temperature above $\beta$ transus, exhibits an $\alpha$ phase that has an entirely different crystallographic orientation from that of the as-received material. According to reports by Shang et al., when ($\alpha + \beta$)-type Ti–Al–Mo–V–Fe–B alloys are annealed at a temperature below $\beta$ transus, an $\alpha$-phase precipitate upon post cooling have the same crystallographic orientation as that of the primary $\alpha$ phase. Contrary to this phenomenon, when the alloys are annealed at a temperature above $\beta$ transus, $\alpha$ precipitate upon post cooling has a crystallographic orientation different from that of the primary $\alpha$ phase. Since no change in texture can be seen in the material of KS Ti-9 annealed at a temperature below $\beta$ transus, the $\alpha$ phase is considered to precipitate with maintaining the abovementioned crystallographic relationship as it undergoes variant selection that is the same as that of the primary $\alpha$ phase. Furthermore, with regard to the material annealed at 1248 K, which is a temperature above $\beta$ transus, showing the change in the texture, since variant selection originated in the primary $\alpha$ phase does not take place, the $\alpha$ precipitate with random crystallographic orientation is considered to form.

3.2 Tensile properties after annealing

Figure 6 shows the relationship between annealing temperature and tensile properties of as-received and annealed materials. The tensile strength in both the L and T directions of the as-received material contained working strain introduced during the manufacturing process (i.e., rolling) is higher than that of the annealed material. The as-received material also shows strong mechanical anisotropy, with a tensile strength in the L-direction of approximately 1000 MPa, which is roughly 300 MPa less than that in the T-direction showing a tensile strength of around 1310 MPa. On the other hand, the elongation of the as-received material is essentially the same in both directions. As mentioned above, the $c$-axis of the primary $\alpha$ phase in the as-received material has T-texture, which is strongly oriented to the T direction. In this case, the main deformation mechanism when tensile stress is applied in the T-direction is considered to be activation of ($c + a$) dislocations and twin formation, as suggested by the observation that the shear stress on ($a$) dislocations, which have slip planes parallel to the basal plane of the $\alpha$ phase is theoretically zero. It has been reported that since the critical resolved shear stress (CRSS) for ($c + a$) dislocations in a single crystal of the Ti–6.6Al alloy is roughly twice greater than that for ($a$) dislocations at room temperature. Therefore, more stress would be required for deformation in the T direction of KS Ti-9 than in the L-direction, to which the ($a$) dislocations can act, in KS Ti-9 similar to the case of the single crystal of the Ti–6.6Al alloy because the strong texture of the $\alpha$ phase is formed in KS Ti-9 although Ti-9 in this study is polycrystal material. However, further investigation is needed on this point.

The tensile strengths in the L and T directions of the material annealed at a temperature between 973 and 1123 K are remarkable decreased due to the grain growth and reduction of the residual strain. However, since the tensile strength in T direction is 300 MPa less than that of the L direction, the effect of the texture on the tensile strength is maintained for the materials annealed at a relatively high temperature. On the other hand, the elongations of the materials annealed at a temperature between 973 and 1123 K are similar in both the L and T directions.

The difference in tensile strength between the L and T directions decreases to around 200 MPa for the material annealed at 1223 K, which is a temperature close to $\beta$ transus. However, the anisotropy recognized in the as-received material remains even for the materials annealed at a temperature near $\beta$ transus. In the case of the annealing at 1248 K, which is a temperature above $\beta$ transus, the tensile strengths in both the L and T directions are nearly the same; that is, anisotropy is considerably reduced. However,
since the microstructure changes to an acicular α structure, the elongation decreases substantially in both directions. Anisotropy of the tensile strength decreases markedly by the annealing at a temperature above β transus. Therefore, the anisotropy of the tensile strength in the material annealed at 1223 K, which is a temperature a little below the β transus is also suggested to be due to the precipitated α phase having the same orientation as that of the primary α phase. Therefore, the anisotropy of the tensile strength and the decrease of strength in the materials annealed at 1248 K, which is a temperature above β transus, because the microstructure is changed to the coarse one having the α precipitate with the β grain by the annealing at a temperature above β transus.

Figure 7 shows the nominal stress–displacement curves obtained from the L and T directions of the as-received and representative annealed materials. Young’s moduli of around 100 GPa in the L direction and around 130 GPa in the T direction are obtained for the as-received material. A marked difference of Young’s modulus is apparent between the two directions. This is because of a difference in crystallographic direction resulting in the formation of texture. For the as-received material, non-uniform elongation increases to a greater extent in the T-direction than in the L-direction, while uniform elongation conversely decreases. The macro-fracture surface of the T-direction on the as-received material exhibited the cup and cones fracture along with local necking. On the other hand, the macro-fracture surface exhibited 45° inclined one to the direction of tensile stress showing little local necking. Therefore, non-uniform elongation due to the local necking is larger in the T-direction than in the L-direction and the elongation in the T-direction is considered to be essentially same as that in the L-direction. Furthermore, many equiaxed dimples were observed on the surface of the T-direction, while on the surface of the L-direction, both equiaxed dimples and many facets parallel to the macro-fracture surface were observed. The fracture in the L-direction is accompanied by little non-uniform deformation, because the facet appears to form corresponding to separation along the specific slip planes.

Materials annealed at temperatures below β transus, under which no change in their texture was observed, also exhibited the same trend as the as-received material mentioned above. However, the material annealed above β transus at 1248 K exhibited fracture surfaces of both directions essentially unaccompanied by non-uniform deformation. Furthermore, their macro-fracture surfaces exhibited large tortuous ones, and micro-fracture surface contained both equiaxed dimples and facets.

From the facets mentioned above, it would be difficult to diminish the anisotropy of the tensile strength by annealing at a temperature below β transus because such efforts are hindered by the formation of an precipitated α phase having the same crystallographic orientation as that of the primary α phase. On the other hand, the anisotropy of the tensile strength diminishes through suppression of the textured structure by the annealing at a temperature above β transus, but the elongation deteriorates markedly. Therefore, it would be difficult to achieve good mechanical properties even by the annealing at a temperature below β transus.

Thus, another suitable heat treatment to diminish the anisotropy of the mechanical properties is required. The heat treatment process comprising solution treatment and stabilizing treatment has been proposed by Fujii et al. in order to prevent the anisotropy of the mechanical properties. It is suggested that the fine α precipitates or martensite (α′), which is transformed from the rapidly cooled β phase after the solution treatment in the (α + β) phase region at a temperature near β transus leading to decreasing the primary α phase having texture (T-texture), has the random crystal orientation different from that of the primary α phase. In this case, the anisotropy of the mechanical properties is considered to decrease because the small amount of the primary α phase having texture and the precipitated α phase coexists in the microstructure.

3.3 Microstructure after duplex heat treatment

Figure 8 shows an optical micrograph of KS Ti-9 subjected to duplex heat treatment to suppress anisotropy
of the mechanical properties. The microstructure consists of a spheroidized primary $\alpha$ phase and $(\alpha + \beta)$ phase containing acicular $\alpha$ precipitates within the $\beta$ phase. As compared with the material annealed at 1223 K (Fig. 2), the lath width of the acicular $\alpha$ phase in this material solutionized at 1223 K is a little narrower.

Figures 9 and 10 show a (0002)$_{\parallel}$ pole figure of the $\alpha$ phase obtained from the rolling plane (L-T plane) and a crystallographic orientation mapping of the primary $\alpha$ phase and $\alpha$ precipitate measured on the same plane by EBSD for the duplex heat-treated material. In Fig. 9, it can be seen that the formation of the $\alpha$ phase with different crystallographic orientations, although that with the T-texture, which have been recognized in the as-received material, also retains as shown in Fig. 4. EBSD measurements show that the precipitated $\alpha$ phase have a crystallographic orientation different from that of the primary $\alpha$ phase. Therefore, the $\alpha$ phase having a T-texture and the other $\alpha$ phase having a clearly different crystallographic orientation from that of T-texture correspond to the primary $\alpha$ phase and the precipitated $\alpha$ phase, respectively. This would confirm that the microstructure of KS Ti-9 can be controlled to have a
random microstructure with no variant selection resulting from the primary \( \alpha \) phase by changing from the fine precipitated \( \alpha \) phase or martensite (\( \alpha' \)) formed by the rapid cooling after solution treatment.  

3.4 Tensile properties after duplex heat treatment

Figure 11 shows the tensile properties of KS Ti-9 subjected to duplex heat treatment (duplex heat treated material) to suppress the anisotropy of the mechanical properties along with corresponding properties for the as-received material and the material annealed at 1223 K, which have been already shown in Fig. 6. The tensile strengths of the L-direction of the duplex heat-treated material are around 60 and 150 MPa greater than those of the as-received material having residual working strain and the material annealed at 1223 K. On the other hand, the tensile strength of the T-direction of the duplex heat-treated material is around 150 MPa less than that of the as-received material and only around 30 MPa greater than that of the material annealed at 1223 K. In the duplex heat-treated material, the tensile strength of the T-direction is around 85 MPa greater than that of the L-direction, but the difference of the tensile strength between the L-direction and the T-direction of the duplex heat-treated material is remarkably decreased as compared to those of the as-received material and the material annealed at 1223 K, whose anisotropy of the tensile strength is slightly decreased. Therefore, although some anisotropy of tensile strength due to a small amount of the retained primary \( \alpha \) phase is recognized in the duplex heat-treated material, it is recognized that the anisotropy of the tensile strength of KS Ti-9 can be reduced by controlling the original microstructure to the microstructure having precipitated \( \alpha \) phase with a random crystallographic orientation. On the other hand, although the elongation in T-direction of the duplex heat-treated material is similar to those of the as-received material and the material annealed at 1223 K, but that in the T-direction of the duplex heat-treated material is around 2% less than those of the as-received material and the material annealed at 1223 K.

Figure 12 shows the nominal stress–strain curve of the duplex heat-treated material along with those of the as-received material and the material annealed at 1223 K, which have been already shown in Fig. 7. The Young’s modulus for the duplex heat-treated material is around 110 GPa in the L-direction and 120 GPa in the T-direction, respectively. Therefore, the difference of the Young’s modulus between these two directions for the duplex heat-treated material is smaller than that for the as-received material. The uniform elongations in both directions of the duplex heat-treated material are less than those in both directions of the as-received material and the material annealed at 1223 K; this trend is particularly apparent in the L-direction. The decrease in elongation mentioned above is considered to be a result of this decrease in uniform elongation. On the other hand, the non-uniform elongation in the L-direction tends to increase. The change in the pattern of uniform and non-uniform elongation seems to be related to the microstructure including the crystallographic orientations of \( \alpha \) precipitate. Further investigation is needed to make clear on this point. The fracture surfaces of both the L- and T-direction of the duplex heat-treated material exhibited local necking, and the local necking was not apparent on the tensile specimen of the
L-direction of the as-received material. The fracture surfaces of the both directions of the duplex heat-treated material exhibited numerous equiaxed dimples, and there was no significant differences between macro- and micro-fracture surfaces of the two directions.

As mentioned above, the KS Ti-9 subjected to duplex heat treatment to reduce anisotropy had a texture different from that in the as-received material, indicating that duplex heat treatment is effective to reduce the anisotropy of the tensile strength between the L- and T-directions. However, considering a reduction in ductility in the L-direction, it is necessary to develop another heat treatment for reducing the anisotropy of the tensile strength by optimizing parameters in the solution treatment and annealing conditions in the duplex heat treatment process.

4. Conclusions

KS Ti-9 was subjected to annealing at different temperatures or to a duplex heat treatment designed to reduce anisotropy. Then, the changes in the anisotropy of associated textures and mechanical properties were investigated. The following results were obtained.

(1) The primary α phase of the as-received KS Ti-9 exhibits a strong T-texture, where the c-axis of the primary α phase aligns strongly to the T-direction.

(2) The tensile strength of the T-direction of the as-received KS Ti9 is around 300 MPa greater than that of the L-direction, indicating strong anisotropy. This anisotropy of the tensile strength is considered to be due to a difference in the deformation mechanism caused by the T-texture as mentioned above.

(3) The strong anisotropy for the tensile strength in as-received KS Ti-9 remains in the annealed KS Ti-9 at a temperature below β transus. On the other hand, since α precipitates having various crystallographic orientations form in the material annealed at a temperature above β transus, the anisotropy of the tensile strength decreases remarkably, but the elongations in both the L- and T-directions decrease as well.

(4) According to the precipitation of the α phase by the duplex heat treatment, the strong anisotropy in the tensile strength in the as-received material decreases remarkably, but the elongation in the L-direction also slightly decreases. Therefore, it is necessary to optimize the heat treatment parameters in the duplex heat treatment in order to improve the elongation with relatively low anisotropy of the tensile strength.

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