

Effect of Heat Treatment Conditions on Mechanical Properties in High Strength Titanium Alloy Super-TIX™ 523AFM

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Abstract

To grasp the fundamental properties needed for proper control of wide range of mechanical properties in high strength β rich $\alpha+\beta$ type titanium alloy “Super-TIX™ 523AFM” (Ti-5Al-2Fe-3Mo), effects of heat treatment conditions on mechanical properties at room temperature were investigated. Mechanical properties such as 0.2% proof stress: 400-1,350 MPa, tensile strength: 1,050-1,700 MPa and Young’s modulus: 78-120 GPa could widely change depending on solution treatment and aging conditions. In particular, the specimens solution treated at 900 and 930°C followed by water quenching exhibited distinctive two-step work hardening on the stress-strain curves, accompanied with quite low 0.2% proof stress: 400-600 MPa, high tensile strength: 1,250-1,400 MPa and low Young’s modulus: about 78 GPa which was almost the same as that of β type titanium alloys. Those characteristic behaviors were considered to be attributed to the extremely low phase stability and the high volume fraction: more than 70% of the transformed β phase.

1. Introduction

Super-TIX™ 51AF (Ti-5Al-1Fe) and Super-TIX™ 52AF (Ti-5Al-2Fe) are $\alpha+\beta$ type titanium alloys that are comparable in strength to the most widely used $\alpha+\beta$ type titanium alloy Ti-6Al-4V, and substitute inexpensive Fe for the rare and costly element, V, to reduce manufacturing costs.¹⁻³⁾ Since their development in the 1990s, they have been applied in diverse fields, including consumer products such as automotive parts and golf clubs. Meanwhile, Super-TIX™ 523AFM (Ti-5Al-2Fe-3Mo) is a β -rich, $\alpha+\beta$ type titanium alloy that contains as much as 5-mass% β stabilizing element of Fe and Mo. Developed in the early 2000s, Super-TIX™ 523AFM is superior in strength, ductility, and cost performance to Ti-6Al-4V and the Ti-Al-Fe based alloys mentioned above. Therefore, because of these characteristics, Super-TIX™ 523AFM is mainly used for intake valves of motorcycles and several high-class models of four-wheeled vehicles. The scope of its application is expected to further expand in the future.⁴⁻⁶⁾

The FeTi intermetallic compound formed in those Fe-added titanium alloys is in equilibrium phase in the low- to medium-temperature range including room temperature. Because of the slow rate of its formation, however, when they are used at a temperature near

room temperature after annealing, they contain metastable β phase rather than FeTi phase. Therefore, in practice, it is possible to handle them as $\alpha+\beta$ type titanium alloys. Even so, depending on the heat treatment conditions, ambient temperature, etc., there is a possibility that equilibrium FeTi phase is formed, causing the ductility, toughness, corrosion resistance, etc. of the alloys to decline. However, in Super-TIX™ 523AFM, the β phase is more stable than Super-TIX™ 52AF, which does not include Mo, and the FeTi phase is not formed despite the alloy being exposed to temperatures around 500°C for many hours. Thus, there is little concern about the deterioration of their properties.^{4,5)}

In addition, since Super-TIX™ 523AFM is a β -rich, $\alpha+\beta$ type titanium alloy, it is considered possible to control its mechanical properties by solution treatment and aging conditions. The reason for this is explained by using a schematic illustration of the titanium alloy phase diagram (Fig. 1). In Fig. 1, the horizontal axis is concentrations of β -stabilizing element, while the vertical axis is the temperatures. When a β -rich, $\alpha+\beta$ type titanium alloy, such as Super-TIX™ 523AFM, is subjected to solution treatment in the $\alpha+\beta$ high temperature region, the amount of β phase becomes larger than that in Ti-6Al-4V and the stability of β phase becomes higher. Therefore,

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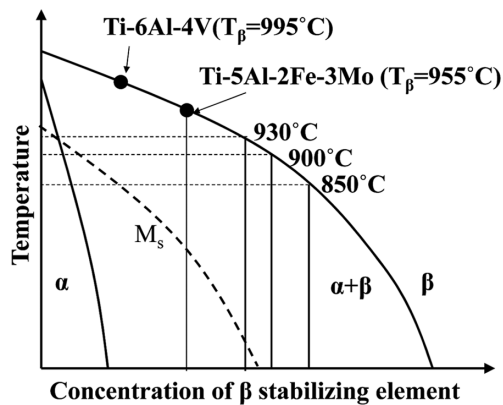


Fig. 1 Schematic illustration of phase diagram in titanium alloys

by rapidly cooling the alloy after solution treatment, it is possible to make the β phase remain in the alloy without causing the α phase to precipitate in the β phase during the cooling process. It is also possible to make the β phase undergo martensitic transformation during cooling. Therefore, it is expected that mechanical properties can be considerably changed by solution treatment at high temperatures in the $\alpha+\beta$ region followed by rapid cooling.

In the present study, we grasped the changes in mechanical properties of Super-TIX™ 523AFM brought by solution treatment and aging and examined the influence of microstructure on those properties.

2. Experimental Procedure

2.1 Material used

The chemical composition of Super-TIX™ 523AFM used in the present study is shown in **Table 1**. The β transus is at approximately 955°C.^{4,7)} A 200-kg ingot prepared by vacuum-arc double remelting was hot-forged into a 100 mm diameter bar. The bar was then hot-rolled into round bars 15 mm and 20 mm in diameter, which were then used as specimens. All of the hot-rolled bars were essentially identical in microstructure. Thus, the difference in diameter made no special difference in their microstructures. Short test pieces 100 mm in length were cut out from the above specimens and subjected to solution treatment (ST) and aging (A). The ST was carried out at 850, 900, and 930°C (all in the $\alpha+\beta$ high temperature region) for 1 h followed by air cooling (AC) and water quenching (WQ). The A treatment was carried out at 500 and 550°C for 4 h followed by AC. In the following sections, each of the specimens subjected to ST shall be designated by the appropriate holding temperature/cooling condition. Example: 850°C/WQ for the test piece that was subjected to ST at 850°C followed by WQ.

2.2 Tensile test

The mechanical properties of the specimens at room temperature were evaluated by tensile test. Heat-treated round bars were machined into test pieces for the tensile test (parallel portion 32 mm in length and 6.25 mm in diameter). The tensile test conditions were as follows: gauge length 25 mm; strain rate from 1×10^{-4} to $5 \times 10^{-4} \text{ s}^{-1}$

(up to 0.2% proof stress) and from 5×10^{-4} to $5 \times 10^{-3} \text{ s}^{-1}$ (until fracture). The Young's modulus of each test piece was measured by a strain gauge attached to the parallel portion of the test piece. In addition, to examine the change in microstructure of each test piece during deformation, the tensile tests consisted of a test load stopped halfway before the test piece fractured.

2.3 Microstructure observation

In the as solution treated specimens and specimens deformed by tensile testing, the microstructure was investigated under an optical microscope and a transmission electron microscope (TEM), and by an X-ray diffractometer. The L-section of the parallel portion of each tensile test piece was examined. For each fractured test piece, the parallel portion away from the part where the necking occurred was used. First, the L-section was polished by machining, etched in a nitric hydrofluoric acid solution, and then observed under the optical microscope. However, since the microstructure of transformed β phase could not be clearly observed under the optical microscope, it was examined under the TEM. In the TEM observation, a thin film specimens were prepared by electropolishing in 90 perchloric acid + 525 butanol + 900 methanol solution at about -30°C . In addition, to identify the constituent phases of each test piece after ST and during each tensile test, X-ray diffraction analysis (CuK α) was performed.

3. Experimental Results

3.1 Mechanical properties

Figure 2 shows the nominal stress–nominal strain (elongation) curves (S-S curves) of the ST specimens. The S-S curves of the ST/AC specimens (**Fig. 2 (a)**) did not differ according to the ST temperature. Interestingly, the S-S curves were very similar to those of typical $\alpha+\beta$ type titanium alloys that show very little work hardening after plastic deformation. Meanwhile, the S-S curves of the ST/WQ specimens (**Fig. 2 (b)**) differ noticeably according to the ST temperature. However, while the 850°C/WQ specimen showed an S-S curve very similar to those of the ST/AC specimens, the 900°C/WQ and 930°C/WQ specimens showed two stages of work hardening.⁷⁾

Figure 3 shows major mechanical properties of the ST specimens. Those mechanical properties change markedly according to heat treatment conditions. In the ST/AC and 850°C/WQ specimens, the 0.2% proof stress is 950–1,000 MPa (**Fig. 3 (a)**), the tensile strength is 1,050–1,100 MPa (**Fig. 3 (b)**), and the elongation is 16%–23% (**Fig. 3 (c)**). Meanwhile, 900°C/WQ and 930°C/WQ specimens show a very low 0.2% proof stress (400–600 MPa), and their tensile strength is very high (1,250–1,400 MPa). The Young's modulus of the specimens is about 115 GPa for ST/AC, about 92 GPa for 850°C/WQ, and about 78 GPa for 900°C/WQ and 930°C/WQ. Thus, it ranges from the value of ordinary $\alpha+\beta$ type titanium alloys to the value of β type titanium alloys.^{4,7)}

Figure 4 shows the relationship between strength (tensile strength) and ductility (elongation), obtained with the STA specimens. Tensile strength varies widely from 1,350 MPa–1,700 MPa. Thus, the tensile strength increased to a maximum of 1,700 MPa. With the increase in strength, however, the elongation decreased. For the specimen that showed the maximum tensile strength of 1,700 MPa (ST at 930°C, followed by WQ and aging at 500°C for 4 h), the elongation was 2.4%. It was clear that the Young's modulus increased by aging. For example, the Young's modulus of the ST/WQ specimen that was approximately 78 GPa before the aging increased to approximately 120 GPa—comparable to the values of ordinary $\alpha+\beta$ type titanium alloys—after the aging.⁴⁾

Table 1 Chemical compositions of material used (mass%)

Al	Fe	Mo	O	N	C	H	Ti
5.1	1.9	3.1	0.18	0.002	0.002	0.0049	Bal.

3.2 Microstructure

For the 900°C/WQ and 930°C/WQ specimens that showed characteristic tensile properties (i.e., low 0.2% proof stress, high tensile strength, and small Young's modulus), the changes in their microstructures that occurred during and after the tensile tests were examined by X-ray diffraction and TEM observation. During the tensile tests, specimens obtained by interrupting the tensile test at an elongation of about 3%, which was the end point of the first stage of

work hardening, and at an elongation of about 6%, which was the end point of the second stage of work hardening, were used for microstructure observation.

Figure 5 shows the X-ray diffraction analysis results. In the 900°C/WQ specimen (Fig. 5 (a)), the diffraction peaks corresponding to both the hcp phase (primary α phase or α' martensitic phase) and the β phase were observed in the as solution treated condition. Those diffraction peaks remained almost the same even after the

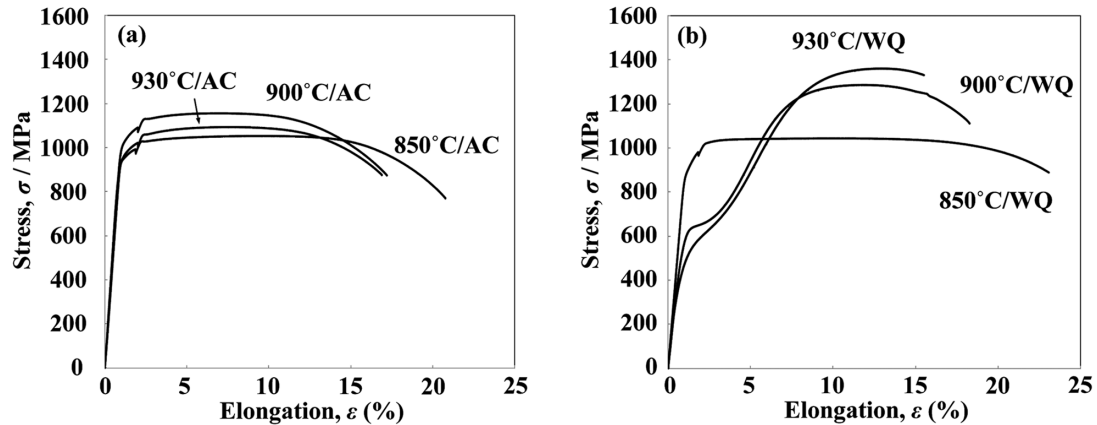


Fig. 2 Stress-elongation curves of as solution treated Ti-5Al-2Fe-3Mo
Solution treatment (ST) was conducted at 850, 900 and 930°C followed by (a) air cooled (AC) and (b) water quenched (WQ).

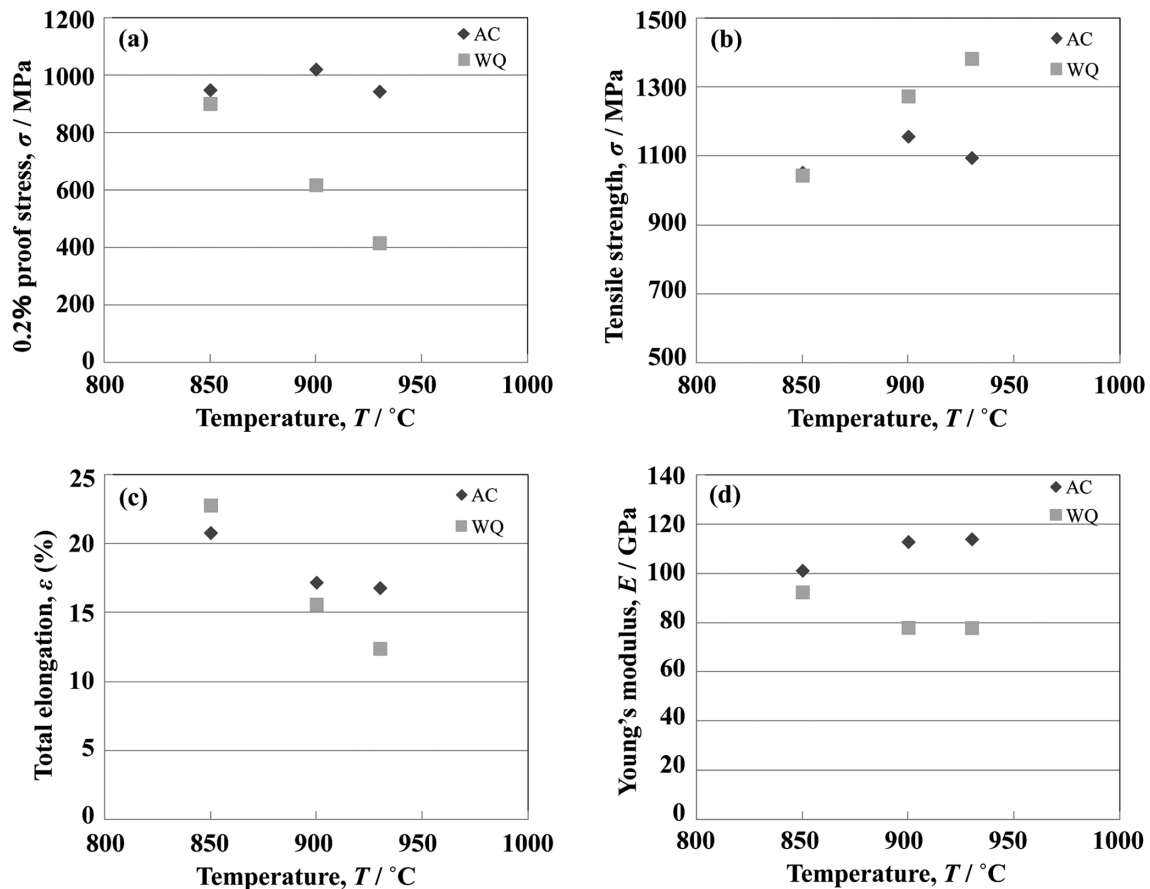


Fig. 3 Relationship between solution treatment temperature and mechanical properties in Ti-5Al-2Fe-3Mo
(a) 0.2% proof stress, (b) Tensile strength, (c) Elongation and (d) Young's modulus

tensile deformation at 3% elongation. After tensile deformation at 6% elongation, the diffraction peak of β phase disappeared and the diffraction peak of the α'' phase (orthorhombic martensite phase)

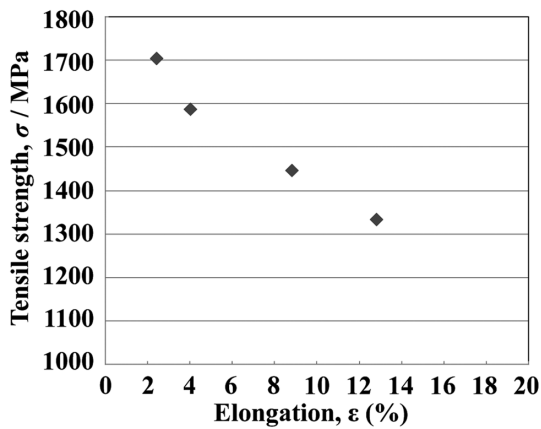


Fig. 4 Relationship between tensile strength and elongation for the specimens solution treated at 900 and 930°C followed by water quenched and subsequently aged at 500 and 550°C in Ti-5Al-2Fe-3Mo

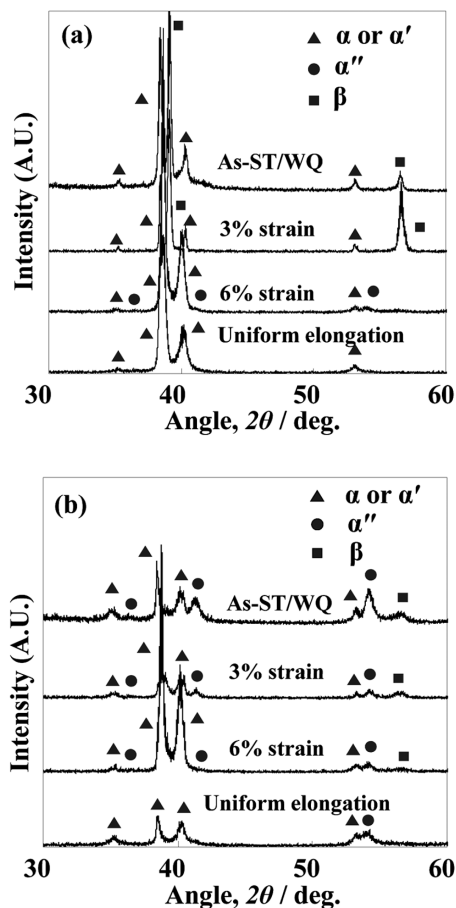


Fig. 5 X-ray diffraction analyses for as solution treated specimen and specimens subsequently tensile deformed at 3%, 6% strain and up to uniform elongation at room temperature (a) STed at 900°C and (b) STed at 930°C followed by water quenching (WQ)

appeared. At the time of a tensile fracture, the α'' phase diffraction peak also disappeared in the parallel portion (subjected to a strain corresponding to the uniform elongation), and only the diffraction peak of the hcp phase was detected.

In the 930°C/WQ specimen (Fig. 5 (b)), diffraction peaks of the hcp phase and the α'' phase were confirmed in the as solution treated condition. At the same time, a slight diffraction peak of β phase was detected. With the increase in amount of tensile deformation (elongation from 3% up to 6%), the diffraction peaks of the α'' and β phases decreased. At the time of a tensile fracture, only the diffraction peak of the hcp phase was confirmed in the parallel portion.

Photos 1 and 2 show TEM microstructures of the specimens that were subjected to the X-ray diffraction analysis shown in Fig. 5.

In the 900°C/WQ specimen (Photo 1, (a)), the transformed β phase between primary α phases is a retained β phase in the as solu-

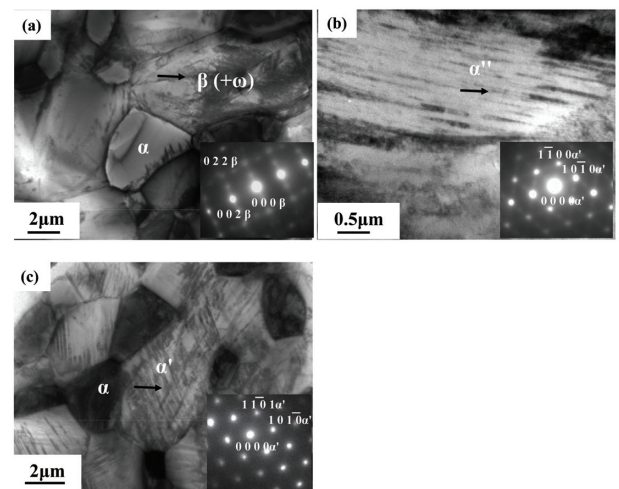


Photo 1 TEM microstructures for the specimens solution treated at 900°C followed by water quenched (WQ) and plus tensile deformed (a) as solution treated, (b) tensile deformed at 3% strain, (c) tensile deformed up to uniform elongation

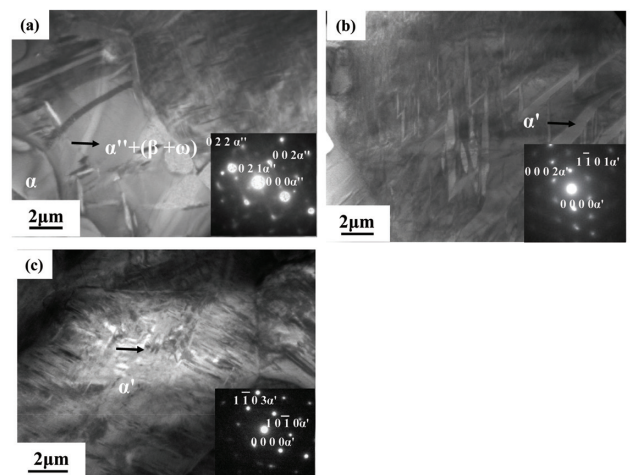


Photo 2 TEM microstructures for the specimens solution treated at 930°C followed by water quenched (WQ) and plus tensile deformed (a) as solution treated, (b) tensile deformed at 3% strain, (c) tensile deformed up to uniform elongation

tion treated condition. In this retained β phase, a diffraction pattern of ω phase was also observed. After the tensile deformation at 3% elongation, a small amount of the α'' phase was observed locally in the transformed β phase (Photo 1, (b)). After the tensile deformation at 6% elongation, the β phase disappeared completely. Instead, a large amount of the α'' phase was observed together with a slight amount of the α' phase. In the parallel portion after tensile fracture, only the α' phase was observed in the transformed β phase (Photo 1, (c)). These results are aligned with the X-ray diffraction results.

In the 930°C/WQ specimen (Photo 2 (a)), the transformed β phase was largely the α'' phase, although a small amount of the retained β phase was confirmed in the as solution treated condition. In the retained β phase, ω phase was also confirmed. With the increase in tensile strain—elongation from 3% up to 6%—the α'' phase and the retained β phase in the transformed β phase decreased and the α' phase increased (Photo 2 (b)). In the parallel portion after the tensile fracture, as in the 900°C/WQ, the transformed β phase consisted entirely of the α' phase (Photo 2 (c)).⁷⁾

4. Discussion

As has been described above, the mechanical properties of Super-TIXTM 523AFM can be changed markedly by varying the heat treatment conditions. In particular, the specimen solution treated at 900 and 930°C in the $\alpha+\beta$ high temperature region, followed by water quenching, showed characteristic mechanical properties such as low 0.2% proof stress, high tensile strength, and low Young's modulus. As shown in Fig. 4, the strength level of this alloy can also vary widely. The reasons why those material properties could be obtained shall be discussed below.

4.1 Mechanisms for developing low 0.2% proof stress and high tensile strength

First, the reason for obtaining a low 0.2% proof stress and high tensile strength is discussed. In the 900°C/WQ specimen, the transformed β phase was the retained β phase, which transformed to the α'' phase by the 6% tensile deformation and then to the α' phase by the tensile fracture. These phenomena indicate that a two-step deformation-induced transformation ($\beta \rightarrow \alpha'' \rightarrow \alpha'$) ultimately occurred, in which the α'' phase acts as the intermediate phase. In the 930°C/WQ specimen, the transformed β phase was the α'' phase, which was transformed to the α' phase by tensile deformation. As a result, a deformation-induced transformation, $\alpha'' \rightarrow \alpha'$, is considered to have occurred. Thus, in the 900°C/WQ and 930°C/WQ specimens, the transformed β phase was unstable and caused a deformation-induced transformation. This is considered the reason why the phase transformation was induced by an unusually small external stress, thereby reducing the apparent 0.2% proof stress. Meanwhile, the α' phase, which was the final phase produced by a deformation-induced transformation, had good work hardenability and is considered to have developed high tensile strength.

4.2 Mechanisms for developing a low Young's modulus

In both 900°C/WQ and 930°C/WQ specimens, the Young's modulus are nearly the same as that of β -type titanium alloys. Generally speaking, the Young's modulus of commercially pure titanium, α type titanium alloys, and $\alpha+\beta$ type titanium alloys is about 110 GPa. Meanwhile, the Young's modulus of β type titanium alloys in the as solution treated condition is about 80 GPa. In the special β type titanium alloys that have been developed mainly as biocompatible materials in recent years, the Young's modulus is as low as 60–65 GPa. It has been known that an extremely low Young's modulus is developed when the composition of the β phase is such that

Table 2 Volume fraction and Mo equivalent of transformed β phase for the specimens solution treated at 850, 900 and 930°C followed by water quenched

Specimen	Volume fraction of β (%)	Mo equivalent* of β (%)
850°C/WQ	55	11.3
900°C/WQ	71	7.0
930°C/WQ	92	4.9

$$* [\% \text{ Mo}] + 2.9 \times [\% \text{ Fe}] - [\% \text{ Al}]$$

the M_s and M_f temperatures are in the neighborhood of room temperature.^{8,9)} For titanium alloys, the Mo equivalent ($= [\% \text{ Mo}] + 2.9 \times [\% \text{ Fe}] - [\% \text{ Al}]$)¹⁰⁾ is often used as one of the stability indexes of β phase. When the Mo equivalent is in the range of about 5% to 8%, the M_s and M_f temperatures approach room temperature as mentioned above. **Table 2** shows the volume fraction and Mo equivalent of β phase in 850°C/WQ, 900°C/WQ, and 930°C/WQ specimens. The Mo equivalent in the 850°C/WQ specimen, which differed slightly in properties from the ST/AC specimens and other $\alpha+\beta$ type titanium alloys, was as large as about 11.2%; whereas, the Mo equivalents in the 900°C/WQ and 930°C/WQ specimens, both of which showed a low Young's modulus, were about 7.0% and 4.9%, respectively. Thus, they fall within the Mo equivalent range that shows an extremely low Young's modulus. In addition, the volume fraction of transformed β phase in the 930°C/WQ specimen (retained β phase in the 900°C/WQ specimen and α'' phase in the 930°C/WQ specimen) was about 71% in the 900°C/WQ specimen and about 92% in the 930°C/WQ specimen. Thus, the composition of the β phase was in the range that possessed an extremely low Young's modulus. Besides, the volume fraction of β phase was large. This is why these specimens showed a low Young's modulus comparable to that of β type titanium alloys.

4.3 Increasing strength by aging

Lastly, mechanisms for increasing the alloy strength by STA are discussed. Generally speaking, β -rich $\alpha+\beta$ type titanium alloys and β type titanium alloys increase in strength when fine particles of the α phase precipitate in the β phase by aging after ST. However, in the 900°C/WQ and 930°C/WQ specimens, there is a peculiar phenomenon, wherein hardening occurs rapidly by aging at 450°C for a few minutes after ST/WQ.^{11,12)} It has been observed that the rapid hardening of the alloy may be attributable to the isothermal martensitic transformation of β phase, rather than the precipitation of α phase in β phase. Although the aging temperature used in the present study was on the high side (500 to 500°C), there is the possibility that the alloy strength was increased by the above mechanisms.

5. Conclusion

With the aim of obtaining basic knowledge for effectively controlling the mechanical properties of the Super-TIXTM 523AFM (Ti-5Al-2Fe-3Mo) titanium alloy, the effects of solution treatment and aging on the mechanical properties of the alloy at room temperature were investigated. The study results are as follows.

- 1) By varying heat treatment conditions, it is possible to control mechanical properties at room temperature over a wide range: 400–1,350 MPa for 0.2% proof stress, 1,050–1,700 MPa for tensile strength, and 78–120 GPa for the Young's modulus.
- 2) In the specimen solution treated at 900 and 930°C followed by water quenching (WQ), an S-S curve indicating two-step work hardening is obtained. It is possible to obtain properties widely

different from those of ordinary $\alpha+\beta$ type titanium alloys, such as exceptionally low 0.2% proof stress (400-600 MPa), high tensile strength (1,250-1,400 MPa), and low Young's modulus comparable to that of the β type titanium alloy (approximately 78 GPa).

- 3) The transformed β phase produced by solution treatment at 900 and 930°C followed by water quenching (retained β phase in the 900°C/WQ specimen; α'' phase in the 930°C/WQ specimen) is so unstable that it undergoes a deformation-induced transformation under even a small external stress during deformation at room temperature ($\beta \rightarrow \alpha'' \rightarrow \alpha'$ in the 900°C/WQ specimen; $\alpha'' \rightarrow \alpha'$ in the 930°C/WQ specimen). This is considered the reason why the apparent 0.2% proof stress of the alloy decreased. Meanwhile, the α' phase, which is the final product of the stress-induced transformation, has good work hardenability and is considered to impart high tensile strength to the alloy.
- 4) The low Young's modulus obtained by solution treatment at 900 and 930°C, followed by water quenching, is considered attributable to the unstable transformed β phase and its large volume fraction (70% or more).
- 5) Super-TiX™ 523AFM whose mechanical properties can be controlled flexibly as described above is expected to find various new applications in the future.

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