Technical Report

Development of Ti-AI-Fe Based Titanium Alloy Super-TIX[™]51AF Hot Rolled Strip Products

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Abstract

It has been conventionally considered to be difficult to produce high strength $\alpha+\beta$ type titanium alloy hot rolled strip products due to 1) high strength and low ductility during hot rolling, 2) strong in-plane anisotropy in mechanical properties in the final products, 3) necessity of careful handling in recoiling, coiling, etc. However, hot rolled strip products of Super-TIXTM51AF (Ti-5Al-1Fe), that is a high strength $\alpha+\beta$ type titanium alloy consisting of inexpensive alloying elements, such as iron, have been developed by optimizing hot rolling conditions, oxygen content and heat treatment conditions to solve these problems. In addition to superior hot-workability of this alloy, reheating the alloy at elevated temperature higher than its β -Transus enables us to manufacture hot rolled strip products whose weight amount to several metric tons in a production hot rolling mill for steel products. Although in-plane anisotropy in mechanical properties of uni-directionally hot rolled sheet is enhanced due to development of hot rolling texture (transverse-texture), in which c-axis of hcp a phase is accumulated in the width direction of the sheet, in-plane anisotropy was relaxed and sufficient ductility was maintained by optimizing oxygen content. The strip products can be smoothly coiled and recoiled at around room temperature since ductility in the longitudinal direction of the strip is considerably high. Moreover, in-plane anisotropy in mechanical properties was relaxed and superior strength-ductility balance was obtained by 2-step heat treatment, including solution treatment at high temperature in the $\alpha+\beta$ region, followed by stabilizing heat treatment.

1. Introduction

Sheets of high-strength $\alpha+\beta$ type titanium alloys, including Ti-6mass%Al-4mass%V (hereinafter Ti-6Al-4V), are manufactured primarily by hot sheet rolling or hot pack rolling. However, manufacturing those sheets involves several problems: low productivity, poor sheet thickness accuracy, etc.^{1, 2)} These problems will be solved if uni-directionally hot-rolled sheets (strips) can efficiently be manufactured by using a hot-rolling mill. However, high-strength $\alpha+\beta$ type titanium alloy strips are seldom manufactured on an industrial basis. The reasons for this are as follows.

- (1) Poor hot workability (high deformation stress and low ductility during hot working) makes it difficult to hot-roll wide, and/or thin sheets.
- (2) In uni-directional hot-rolling, a transverse texture (T-texture) develops where the basal plane (0001) of the hexagonal close-packed (HCP) structure that is the crystal structure of α phase—the main phase—is accumulated in the width direction of the strip. In particular, (12 10) [10 10] develops, intensifying the in-plane anisotropy in material properties. Specifically, strength increases and ductility decreases in the width direction of the strip. This is due mainly to the fact that, in a high-strength α + β type alloy containing aluminum and oxygen, twin deformation is suppressed and slip deformation becomes predominant.³⁾ In this case, since the slip direction of the strip is composed of a transverse texture, slip deformation toward the

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width direction of the strip is restrained. As a result, strength increases and ductility decreases in the width direction of the strip.

(3) Difficulty in handling coils (in recoiling, threading coils through descaling process, welding coils) accounts more or less for the low productivity.

To allow for the supply of high-strength $\alpha+\beta$ type titanium sheets that offer higher quality and lower production costs than conventional hot-sheet-rolled or hot-pack-rolled products, it is necessary to solve the above problems and find manufacturing conditions permitting the desired titanium alloy strips to be produced using a powerful hot rolling mill for steel.

Nippon Steel & Sumitomo Metal Corporation has developed a high-strength $\alpha+\beta$ type titanium alloy, Super-TIXTM51AF (Ti-5%Al-1%Fe), utilizing inexpensive alloying elements (Fe, Al, etc.).⁴) This alloy has relatively high hot workability since the amount of aluminum—an element that decreases hot workability—is less than in Ti-6Al-4V, etc.^{4, 5}) In addition, similar to other high-strength $\alpha+\beta$ type alloys, alloy strength can be controlled by adjusting the amount of oxygen that strengthens the α phase.^{4, 5})

Therefore, by taking advantage of the above-mentioned high hot workability alloy, it may become possible to manufacture hot-rolled titanium alloy strips on an industrial basis by discovering optimum reheating conditions prior to hot rolling. In addition, since oxygen strongly influences texture and anisotropy in material properties,⁶⁰ it could become possible to control anisotropy in material properties by optimizing oxygen content, and identifying optimum heat treatment conditions. Therefore, these factors—hot rolling conditions, the amount of oxygen, and heat treatment conditions—were examined with special interest regarding the manufacture of hot-rolled strips of Ti-5Al-1Fe.

2. Optimum Manufacturing Conditions for Unidirectionally Hot-rolled Strip

2.1 Optimization of hot-rolling conditions

In the laboratory, a 100 mm-thick slab of Ti-5Al-1Fe-0.16O (containing the addition of 0.16% oxygen) was reheated to 1050°C, slightly above the β -transus of the alloy (1010°C), and another slab of the same thickness was reheated to 950°C, slightly below the β -transus. Then, each of the reheated slabs was uni-directionally hot-rolled into a 4-mm-thick sheet. Photo 1 shows appearances of the edges of the hot-rolled sheets. In the slab reheated to 950°C prior to hot-rolling, hot deformation stress sharply increased during the latter stage of rolling, making it difficult to reduce sheet thickness. In addition, edge cracks up to approximately 10 mm in depth were observed (Photo 1 (a)). On the other hand, the slab reheated to 1050°C prior to hot-rolling was smoothly hot-rolled to the final pass without trouble. The edge cracks that formed in the sheet were more shallow (5 mm or less in depth (Photo 1 (b)), compared with those formed in the 950°C hot rolled sheet. In view of the critical rolling load of a hot rolling mill for steel, it is considered possible to massproduce on a stable basis several tons of coil up to approximately 1 000 mm in width by reheating the alloy to the β -phase region prior to hot-rolling.

2.2 Examination of oxygen content

Slabs (100 mm in thickness) of Ti-5Al-1Fe containing 0.16%O, 0.26%O, and 0.36%O (0.16O, 0.26O, and 0.36O) were prepared. They were reheated to 1050°C in the β phase region and uni-directionally hot-rolled into 4 mm-thick sheets. **Figure 1** shows the effects of oxygen content on the tensile properties (tensile strength



Photo 1 Effects of RHT (reheating temperature prior to hot rolling) on appearance of edge cracks in uni-directionally hot rolled sheets Ti-5Al-1Fe-0.16O (4.0 mm in thickness), hot rolling reduction: 96%, RHT: (a) 950°C , (b) 1,050°C





gauge width: 6.3 mm, thickness: 3.9 mm).

(TS) and total elongation (T-EL)) of hot-rolled sheets. In tensile tests, JIS 13B 1/2-scale test pieces (gauge length: 25 mm, gauge width: 6.3 mm, sheet thickness: 3.9 mm) machined from hot-rolled and pickled sheets were used. In the tensile test for 0.36O in the T direction (across the sheet width), the specimen fractured before the occurrence of localized deformation that caused it to constrict in the middle during the test. Therefore, the test results for 0.36O are shown in brackets to differentiate from those of the other test pieces. It can be seen in Fig. 1 (a) that, regardless of the amount of oxygen, TS in the T direction is higher than in the L direction (along the sheet length) by 150 MPa or greater, showing strong anisotropy in strength. It can also be seen that TS increases with an increase in oxygen content. As can be seen from Fig. 1 (b), 0.16O and 0.26O have high ductility, with their T-EL exceeding 10% in both the L



Fig. 2 (0002) pole figures of as hot rolled sheets (a) 0.26% oxygen added, (b) 0.36% oxygen added (mass%), RD: Rolling direction, TD: Transverse direction

and T directions. In contrast, 0.36O shows considerably poor ductility in the T direction (T-EL = 3.9%), although ductility in the L direction is high.

Figure 2 shows (0002) pole figures of 0.26O and 0.36O as hot rolled sheets, indicating a considerable difference in ductility in the T direction. Both sheets contain T-texture consisting mainly of $(12\ \bar{1}0)\ [10\ \bar{1}0]$ in which the basal planes of HCP are strongly accumulated in the width direction of the sheet. 036O shows higher peak strength in the (0002) plane, which is parallel to the T direction (Fig. 2 (b)), and has a stronger T-texture than that in 0.26O (Fig. 2 (a)). It is considered that ductility deterioration in 0.36O in the T direction is caused by a considerable development of T-texture due to high oxygen content, with slip deformation in the T direction strongly suppressed by excessive solid solution strengthening of oxygen. The above results indicate that, to obtain a sufficiently practical strength and ductility balance without causing excessive anisotropy in material properties in uni-directionally hot rolled Ti-5Al-1Fe, it is desirable that the amount of oxygen should be 0.26% or less.

2.3 Manufacturing of strips using commercial production equipment

Based on the results described in 2.1 and 2.2, a Ti-5Al-1Fe-0.16O slab (170 mm in thickness, 840 mm in width) was manufactured by hot-forging a 3.7-ton ingot obtained by VAR (vacuum arc re-melting). The slab was then test-rolled in the hot-rolling mill of Hirohata Works. With the slab reheating temperature set at 1050°C, a hot-rolled strip of 3.9 mm in thickness and 835 mm in width was test-manufactured. Deformation stress during hot rolling was sufficiently lower than the upper limit of the rolling load, and a small number of edge cracks were observed. Thus, the slab could be satisfactorily hot-rolled into a strip. In addition, the strip could be coiled and recoiled easily and smoothly at room temperature. **Photo 2** shows a coil of hot-rolled Super-TIX[™]51AF strip manufactured using the commercial production mill mentioned above. At present, several tons of strip products are mass-produced using a hot rolling mill for steel.

3. In-Plane Anisotropy in Mechanical Properties of Uni-directionally Hot-rolled Strip

As shown in Fig. 1, in the case where Ti-5Al-1Fe is reheated in the β single phase region and subjected to uni-directional hot rolling, in-plane anisotropy in mechanical properties is intensified. This is because uni-directional hot rolling causes a T-texture to develop in Ti-5Al-1Fe, as shown in Fig. 2. To reduce in-plane anisotropy in mechanical properties due to the growth of a T-texture, two methods have been proposed for Ti-Fe-O based $\alpha+\beta$ type alloys, in which the α phase is strengthened by interstitial solid solution elements alone.



Photo 2 Hot strip product of Super-TIX[™]51AF manufactured at a production mill 3.9 mm in thickness, 835 mm in width, manufactured from 3.7 ton ingot

One method is applicable to Super-TIXTM800 (Ti-1Fe-0.35O). which can be cold-rolled. In this method, the allov is subjected to cold rolling in order to considerably reduce the thickness, followed by high-temperature annealing to produce recrystallized grains composing split-TD type B (Basal)-texture (in which (0001) of the α phase is accumulated in a direction tilted by 30 to 35 degrees from the normal direction toward the transverse direction of the sheet, mainly $(12 \ 14) [10 \ 10]$, whose orientation is considerably different from that of the T-texture. Recrystallization texture develops as the annealing progresses.⁷⁾ The other method is applied to Ti-1.5Fe-0.5O-0.05N, which can hardly be cold-rolled. It consists of a twostep heat treatment process (solution treatment (ST) in the hightemperature $\alpha + \beta$ region followed by water quenching and stabilizing treatment in the low-temperature $\alpha + \beta$ region).⁶ In this method, a uni-directionally hot-rolled sheet is subjected first to ST at a temperature below the β -transus, and then to stabilizing treatment at a suitable temperature. Ductility in the T direction improves and in-plane anisotropy in material properties becomes weaker after this two-step heat treatment. The reason for this is as follows. When the reversetransformed β that occurs during ST is guenched, fine acicular α or α' martensite having a random grain orientation is formed, whereby the influence of the primary α phase mainly having a crystal orientation of T-texture is weakened.

Ti-5Al-1Fe has a tensile strength higher than the strength level that allows the alloy to be cold rolled with a large reduction in thickness. This means that the former process for controlling anisotropy by cold rolling and annealing can hardly be applied to the alloy. Therefore, for Ti-5Al-1Fe, which contains both Al (α phase substitutional solid solute element) and O (interstitial solid solute element), the effects of a two-step heat treatment process and oxygen content on the in-plane anisotropy of the alloy sheets were investigated. For the two-step heat treatment, the specimens were first heated to between 975 and 1025°C for 1 h during ST, followed by water quenching, and were then furnished with stabilizing heat treatment at 700°C for 2 h, followed by air cooling. The effects of oxygen content and temperature on the volume fraction of β phase in Ti-5Al-1Fe were calculated using ThermoCalc. The results are shown in Fig. 3. Based on the results shown in Fig. 3, 975, 1000 and 1025°C that made the β phase volume fraction 70-80% were selected as the holding temperatures during ST for 0.16O, 0.26O and 0.36O, respectively.



Fig. 3 Effects of temperature and oxygen content on volume fraction of β phase in Ti-5Al-1Fe Calculated by ThermoCalc, 0.160: 0.16% oxygen added, 0.260:

0.26% oxygen added, 0.36O: 0.36% oxygen added (mass%)

3.1 Influence of two-step heat treatment on microstructure and tensile properties

Photo 3 (a)-(c) show the microstructures of longitudinal cross sections of 0.16O, 0.26O, and 0.36O specimens, which were subjected to the two-step heat treatment. All of the microstructures consisted of primary α phase and acicular $\alpha+\beta$ phase (hereinafter, "transformed β phase"). The volume fraction of the primary α phase was 20-30%, which is close to the volume fraction estimated from Fig. 3.

Figure 4 (a) and (b) compare the effect of oxygen content on tensile properties of two-step heat treated specimens and hot-rolled specimens. Tensile tests of the two-step heat treated specimens used JIS 13B half-sized test pieces (gauge length: 25 mm, gauge width: 6.3 mm, thickness: 3.8 mm) machined after their surfaces were polished to #80. Test results are shown in brackets for the test pieces fractured before the occurrence of localized deformation that caused them to constrict in the middle during the tensile tests. From Fig. 4 (a) it can be seen that for both types of test pieces, tensile strength (TS) was higher in the T direction than in the L direction, indicating that they had strong anisotropy in TS. In the case of 0.26O, however, anisotropy in TS became weaker since the TS in the L direction increased as a result of the two-step heat treatment. With the exception of TS in T and L directions of 0.36O, which was subjected to the two-step heat treatment, TS increased with the increase in oxygen content. It can also be seen from Fig. 4 (b) that in the case where oxygen content was 0.26% or lower, the two-step heat treatment caused the total elongation (T-EL) to increase in both the T and L directions, although the T-EL of 0.36O decreased in both directions as a result of the two-step heat treatment.

Figure 5 shows the strength–ductility balance, in the L direction (Fig. 5 (a)) and T direction (Fig. 5 (b)), of hot-rolled sheets and those subjected to two-step heat treatment. With the exception of 0.26O and 0.36O that were subjected to the two-step heat treatment shown in Fig. 5 (a), ductility exhibited a tendency to decrease with the increase in strength (indicated by the data enclosed in the circle). With respect to the above exception, 0.26O indicates a high strength-ductility balance, whereas 0.36O, which is nearly the same in strength as 0.26O, shows noticeably low ductility. With regard to the strength-ductility balance in the T direction presented in Fig. 5 (b), in the case where oxygen content is 0.26% or less, hot-rolled sheets and those subjected to two-step heat treatment show high



Photo 3 Optical micrographs of 2-step heat treated sheets in the longitudinal cross section (a) 0.16% oxygen added, (b) 0.26% oxygen added, (c) 0.36%

(a) 0.16% oxygen added, (b) 0.26% oxygen added, (c) 0.36% oxygen added (mass%), RD: Rolling direction



Fig. 4 Effects of oxygen content on tensile properties of 2-step heat treated sheets (a) TS: tensile strength, (b) T-EL: total elongation, As HR: as hot rolled, 2-step HT: 2-step heat treated, JIS 13B half size specimen was used (gauge length: 25 mm, gauge width: 6.3 mm, thickness: 3.8 mm).

ductility, whereas as hot rolled and two-step heat treateed 0.360 sheets indicate poor ductility. In particular, 0.360, which was subjected to two-step heat treatment, shows noticeably low ductility not



Fig. 5 Effects of oxygen content on strength-ductility relationship of Ti-5Al-1Fe as hot rolled sheets and 2-step heat treated sheets
(a) L-direction: Longitudinal direction, (b) T-direction: Transverse direction, TS: Tensile strength, T-EL: Total elongation, 0.160: 0.16% oxygen added, 0.260: 0.26% oxygen added, 0.360:
0.36% oxygen added (mass%), JIS13B half size specimen was used (gauge length: 25 mm, width: 6.3 mm, thickness: 3.9 mm (as hot rolled), 3.8 mm (2-step heat treated)).

only in T-EL but also in TS.

3.2 Concentration of alloying elements in each individual phase during solution treatment, and microhardness after two-step heat treatment

For 0.16O, 0.26O and 0.36O, the volume fraction of the β phase at each ST temperature and the concentrations of O, Al and Fe in the α and β phases were calculated using ThermoCalc. Calculation results are shown in **Table 1**. For all of the alloys, the concentrations of Al and Fe in the α and β phases are nearly the same, whereas the concentration of O in the primary α phase of 0.36O is significantly high (0.81%). It was considered that in the case where 0.36O was subjected to two-step heat treatment, the hardness value of the primary α phase would rise considerably due to solid solution strengthening by oxygen.

Therefore, the micro-Vickers hardness (HV 0.025: indenting load 25 gf) of the primary α phase and of the transformed β phase were compared in specimens that were subjected to two-step heat treatment. The results obtained from specimens with different oxygen content are shown in **Fig. 6** (a) and (b). With the increase in oxygen content, both the micro-Vickers hardness of the primary α phase (Fig. 6 (a)) and that of the transformed β phase (Fig. 6 (b)) increase linearly. Thus, it is considered that the increase in microTable 1 Effects of oxygen content on volume fraction of β phase at solution treatment temperature and chemical composition of alloying elements in α and β phase Calculated by ThermoCalc, 0.160: 0.16% oxygen added,

0.26O: 0.26% oxygen added, 0.36O: 0.36% oxygen added (mass%)

No.	ST temp. (°C)	V _f of	mass% in α phase			mass% in β phase		
		β phase (%)	0	Al	Fe	0	Al	Fe
0.160	975	71.1	0.340	5.81	0.016	0.099	4.73	1.33
0.260	1 000	79.5	0.575	5.74	0.013	0.175	4.80	1.27
0.360	1 0 2 5	83.9	0.810	5.65	0.011	0.256	4.85	1.23

ST: Solution treatment. V_f: Volume fraction



Fig. 6 Effects of oxygen content on micro-Vicker's hardness number of primary α phase and transformed β phase
(a) Primary α phase, (b) Transformed β phase, L-cross section: Longitudinal cross section, T-cross section: Transverse cross section

Vickers hardness is due to the increase in the amount of oxygen, as estimated from Fig. 3. In the case of 0.36O, in particular, the micro-Vickers hardness of the primary α phase was as high as 460 in the L cross section and 430 in the T cross section (Fig. 5 (a)). Therefore, the poor ductility of 0.36O is considered to be due to the excessive hardening of the primary α phase caused by the high concentration of oxygen.

In the primary α phase, hardness in the L cross-section was higher than in the T cross-section (Fig. 6 (a)), whereas they were nearly the same in the transformed β phase (Fig. 6 (b)). It is consid-

ered that in the primary α phase, anisotropy of the hot-rolled sheets was maintained, while in the transformed β phase a randomization of crystal orientation (or orientation dispersion) occurred, whereby anisotropy in mechanical properties is weakened. It is implied that the purpose of the two-step heat treatment—to weaken anisotropy in mechanical properties—was attained, as expected.

3.3 Textures and orientation image maps of specimens subjected to two-step heat treatment

Figure 7 shows (0002) pole figures of 0.26O (a) and 0.36O (b) after two-step heat treatment. A comparison with the (0002) pole figures of the hot-rolled sheets (Fig. 2 (a), (b)) shows that the accumulation of (0002) planes in TD (transverse direction: mainly (12 $\bar{1}0$) [10 $\bar{1}0$]) became weaker, whereas accumulation of (0002) planes in RD (rolling direction: mainly (0001) [10 $\bar{1}0$]) and split-RD (mainly ($\bar{1}2 \bar{1}8$) [4 $\bar{1}43$]), etc., was also observed. This implies that the accumulation orientation was dispersed by two-step heat treatment.

Figure 8 (a) and (b) shows orientation image maps (OIMs) of parts of the regions from which the (0002) pole figures shown in Fig. 7 (a) and (b) were obtained. The transformed β phase formed colonies having a comparatively random orientation that is different from the main component orientation of the T-texture. Therefore, the T-texture became weaker than the hot-rolled sheet that did not contain any transformed β phase. The colonies were several micrometers in size, finer than the primary α phase whose grain size is 10 μ m or more. It is considered that the high tensile strength of two-step heat-treated sheets is due to the above-described texture refinement.

Figure 9 (a) and (b) show OIMs, in which the portions of trans-



Fig. 7 (0002) pole figures of 2-step heat treated sheets (a) 0.26% oxygen added, (b) 0.36% oxygen added (mass%), RD: Rolling direction, TD: Transverse direction



Fig. 8 Orientation image maps of 2-step heat treated sheets (a) 0.26% oxygen added, (b) 0.36% oxygen added (mass%), RD: Rolling direction





formed β phase shown in Fig. 8 (a) and (b) are enlarged. Colonies formed in the 0.36O are much finer than those in the 0.26O. The colonies in the 0.260 were approximately 10 μ m and the lath width was comparatively large $(2-3 \mu m; Fig. 9 (a))$. In any case, the concentration of O was not high enough to cause tensile strength to increase significantly (Table 1), and the superior plastic deformability is considered to be maintained. Because of all these factors, there is a possibility that the ductility of 0.260 was comparatively high. In contrast, it is considered that ductility in both the L and T directions is poor in 0.36O since slip deformation was restrained by the high oxygen concentration, and tensile strength increased due to an exceptional refinement of texture (lath width 0.5-1 μ m; Fig. 9 (b)). Thus, although two-step heat treatment caused the ductility of 0.36O to be lower in both the L and T directions, in-plane anisotropy in mechanical properties was relaxed in 0.260, where ductility increased and high tensile strength was maintained comparable to that of the hot-rolled sheets in both the L and T directions.

4. Conclusion

Process conditions for manufacturing hot-rolled strips of $\alpha+\beta$ type titanium alloy Super-TIXTM51AF (Ti-5Al-1Fe) were examined and the following results were obtained.

- (1) In the case where Ti-5Al-1Fe was reheated in the β phase region and hot-rolled, the occurrence of edge cracks, which formed in a sheet reheated and hot-rolled in the $\alpha+\beta$ region, was restrained and hot deformation stress was reduced. Hotrolled sheets of the alloy showed anisotropy in mechanical properties. Although anisotropy in ductility of the 0.36O sheet was considerably strong and ductility was low in the T direction, excessive anisotropy was not found in the 0.26O sheet, which showed high ductility in both the L and T directions. Based on these results, a Ti-5Al-1Fe-0.16O strip (from 3.7 ton VAR ingot, 3.9 mm thick and 835 mm wide) was test-produced with a reheating temperature of 1050° C in the β phase region using a production hot-rolling mill. It was found that the alloy could be hot-rolled smoothly and the strips could be coiled and recoiled easily. At present, several tons of hot-rolled strips of Ti-5Al-1Fe are being mass-produced.
- (2) Two-step heat treatment (i.e., high-temperature solution treatment followed by water quenching and stabilizing treatment) not only reduced the anisotropy in mechanical properties of the 0.26O sheet but also improved the tensile strength and ductility

in both L and T directions. This is attributed to the combined effect of the randomization of grain orientation and the enhancement of tensile strength by the improvement of plastic deformability and refinement of texture. On the other hand, the two-step heat treatment caused the ductility of 0.36O sheet to decrease in both L and T directions. This is due to the suppression of slip deformation through an increase in oxygen content in the primary α phase, and also due to an excessive increase in tensile strength caused by formation of an extremely fine acicular microstructure (transformed β phase).

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