

Development of High Performance Ti-Fe-Al Alloy Series



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Abstract

To reduce manufacturing cost of $\alpha+\beta$ titanium alloys, ternary Ti-Fe-Al alloy compositions showing wide range of strength levels and excellent ductility were investigated, and alloy series having 650 to 850MPa of tensile strength were developed. In addition, by controlling the concentration of interstitial impurities, an alloy having up to 1,100MPa of tensile strength in an annealed condition was developed. Among the alloys in the series, Ti-3.5Al-1Fe and Ti-5.5Al-1Fe were proposed as substitute alloys for most widely used Ti-3Al-2.5V and Ti-6Al-4V, respectively. Ti-5.5Al-1Fe possesses unique features such as high fatigue property, strengthening by heat treatment, superplasticity, in addition to excellent strength-ductility relationship. Furthermore, it was made clear that some attention must be paid for the brittle FeTi phase formation when the alloy is used at high temperature like 450 °C, but FeTi is not easily formed at lower temperature like 300 °C or lower.

1. Introduction

Titanium alloys basically consisting of the three elements Ti, Fe, and Al have been developed since 1980¹⁻³⁾. Many of them have been developed as substitutes for Ti-6Al-4V which is the most commonly used titanium alloy, and so are designed to have the same or slightly higher strength than that of Ti-6Al-4V. One typical Ti-Fe-Al alloy, Ti-5Al-2.5Fe, was developed in the early 1980s. This alloy was developed to be used as implant material and Fe, which is not toxic for the human body, was substituted for V which is toxic for the human body in Ti-6Al-4V¹⁾. Ti-6Al-1.7Fe-0.1Si is known as a low cost alloy in which an expensive β stabilizing element V is replaced by Fe which is inexpensive and has similar performance to V, and a small amount of Si is also added to improve the heat resistance. The use of this alloy in automotive engine parts, etc. is under consideration³⁾.

The concept of substituting Fe, which is inexpensive and gives the alloys high-performance, for part or the whole of expensive V or Mo, which are commonly used in many commercial alloys, has been discussed for a long time. In fact, however, actual development has been limited to substitution in Ti-6Al-4V, and there are almost no reports on applying the approach to Ti-3Al-2.5V or its improved alloy which is used for consumer goods such as spectacle frames and connecting rods in automotive engines⁴⁻⁵⁾.

The author and his colleagues therefore changed the contents of Al and Fe over a wide range and investigated the effects on the mechanical properties, and have developed a series of Ti-Al-Fe based alloys having different strength levels⁶⁾. The objective of the development is to reduce the manufacturing cost. This report introduces the development details of the alloy series, the mechanical properties and the strengthening heat treatment of Ti-5.5Al-1Fe which is a

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typical alloy in the series⁷⁾, and discusses the attention needed in heat treatment and actual use.

2. Alloy design of Ti-Fe-Al based Titanium alloy series

2.1 Materials used

10 kg columnar ingots (90 mm in diameter) with varying contents of Al and Fe were prepared by plasma arc remelting and hot rolled to bars of 15 mm in diameter after refining the ingot surface. The heating temperature for hot rolling was in the $\alpha + \beta$ region below the β transus. Annealing at 750°C for 1h followed by air cooling was then carried out and the tensile properties, etc. were evaluated. All alloys in the series have typical mill-annealed microstructures in which some elongated grains are mixed in equiaxed ones. Since the objective of this development was to develop alloys having various strength levels ranging from Ti-3Al-2.5V to Ti-6Al-4V, Al, an α phase strengthening element, was added up to 6% maximum, and Fe which is prone to cause segregation during solidification was added to 2% or less by taking the experiences of the segregation in large ingots manufactured with an actual mill into account. The ingots used in the present experiments were so small that segregation was not significant and had nearly uniform compositions. Also, since titanium sponge with comparatively low oxygen was used as a raw material and interstitial elements such as O were not added intentionally, the content of O was 0.06 to 0.08 mass%, which was a little lower than that in conventional titanium alloys.

1 kg and 200 kg ingots containing various contents of O and N were manufactured by vacuum arc remelting in addition to plasma arc remelting to examine the effect of O and N. 1kg ingots were forged in the $\alpha + \beta$ temperature region while 200 kg one was forged and then hot rolled as done for the plasma arc remelted materials, and then both were annealed at 750°C for 1h followed by air cooling.

2.2 Test results

2.2.1 Effect of Al and Fe contents on strength of Ti-Fe-Al alloy series

Fig. 1 shows the tensile strength of Ti-Fe-Al alloy bars having various compositions. The alloys in the figure were initially plasma arc remelted and the contents of Al and Fe were taken as horizontal and vertical axes, respectively, in the figure. The effect of Al and Fe contents on the strength was similar, and alloy compositions exhibiting the strength from 650 to 850 MPa were clarified from this figure. Meanwhile, all the studied alloys had high ductility, and even the alloy having the highest strength was confirmed to have 15% elongation or more. Consequently, it has become possible to provide alloy compositions having various levels of strength and high ductility, based on this figure, in accordance with the required strength. As mentioned above, in the case of a large ingot the concentration of Fe

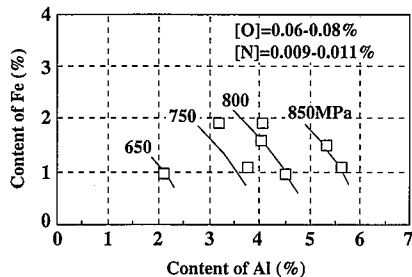


Fig. 1 Tensile strength of Ti-Fe-Al based titanium alloy bars of 15 mm in diameter which were manufactured by plasma arc remelting, hot rolling and annealing (O: 0.06 to 0.08%, N: 0.009 to 0.011%)

was limited to 2% or less in view of its segregation, and in fact a small content of Fe is preferable in order to obtain a homogeneous material. Therefore, among the various alloys having some strength levels, alloys such as Ti-3.5Al-1Fe and Ti-5.5Al-1Fe in which the content of Fe is about 1% are recommended for ease of manufacturing.

2.2.2 Effect of O and N contents on tensile properties of Ti-Fe-Al alloy series

The content of O in the alloy series enumerated in Fig. 1 was lower than that of conventional titanium alloys, as mentioned above. Therefore, if the alloy series are manufactured using the raw materials and manufacturing process similar to those for the conventional titanium alloys, the concentration of interstitial impurities such as O may become somewhat high. O and N are strong strengthening elements and can be used to adjust the strength level of alloys. From this point of view, the effects of interstitial impurity elements in two representative alloys, Ti-3.5Al-1Fe and Ti-5.5Al-1Fe, were examined. Both alloys have the compositions that we propose as substitutes for Ti-3Al-2.5V and Ti-6Al-4V respectively.

Fig. 2 shows the results of the examination. The oxygen equivalent, [O]eq, is defined as $[O]eq = [O] + 2.77 [N]$, in which [O] and [N] are the concentrations of O and N respectively. This equation was derived by examining the strengthening capability of O and N in alloys containing about 1 mass% of Fe⁹⁾. In Fig. 2, 0.2% proof stress and tensile strength of each alloy rose by about 100 MPa when [O]eq increased by 0.1%, which was almost the same as the strengthening capability of O in conventional titanium alloys. On the other hand, ductility did not depend on [O]eq but remained almost constant, and no distinct ductility loss was observed within the current evaluation as [O]eq was increased. Thus, Fig. 1 presents the optimum compositions taking the effect of interstitial impurity elements on strength into consideration.

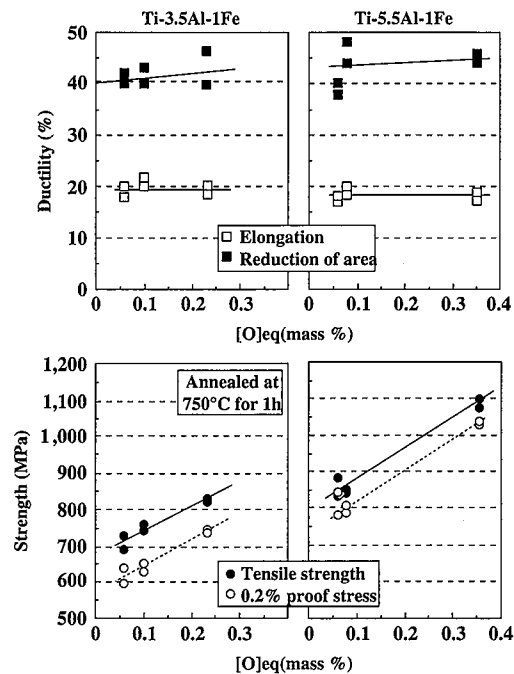


Fig. 2 Effect of interstitial impurity elements on tensile properties of Ti-3.5Al-1Fe and Ti-5.5Al-1Fe (The oxygen equivalent, [O]eq, is defined as $[O]eq = [O] + 2.77 [N]$, in which [O] and [N] are the concentrations of O and N respectively)

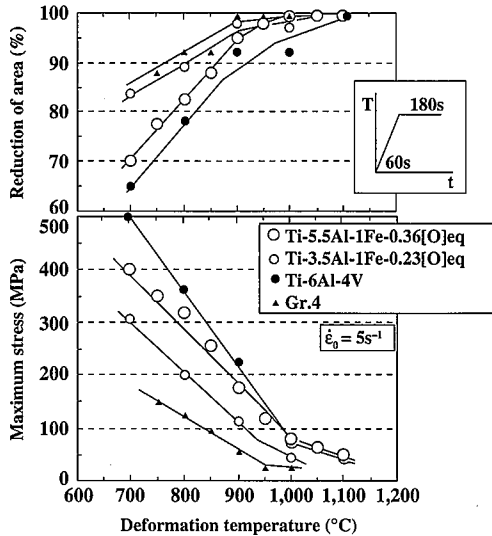


Fig. 3 Hot deformation characteristics of the β annealed Ti-Al-Fe based alloys (Gleeble test). Those of Ti-6Al-4V and ASTM grade 4 are also shown for comparison.

2.2.3 Hot workability of Ti-Fe-Al alloy series

Some compositions of Ti-Fe-Al alloy series have been designed based on the tensile properties at room temperature. However, Al, which is the biggest factor that lowers hot workability in conventional alloys, is also contained at the level of several percent in the alloy series, as in conventional alloys. If hot workability is poor, the advantage of the low cost compositions cannot be fully utilized, so the hot workability of some representative compositions was evaluated by Gleeble test.

Fig. 3 shows the test temperature dependency of reduction of area and maximum deformation stress of two kinds of Ti-Fe-Al alloys, Ti-5.5Al-1Fe(-0.36[O]eq) and Ti-3.5Al-1Fe (-0.23[O]eq). The test results for Ti-6Al-4V and ASTM grade 4 are also shown in the figure for comparison. Since the purpose of the test was to evaluate the workability during hot rolling, microstructures of the materials used were converted to the β annealed ones (fine acicular) by heating the materials in the β region and cooled, and relatively high strain rate 5 s^{-1} was employed.

As shown in Fig. 3, the higher the Al content was, the higher the deformation stress was. The deformation stress of Ti-3.5Al-1Fe and Ti-5.5Al-1Fe was approximately in the middle between grade 4 and Ti-6Al-4V. In other words, although the hot deformation resistance of the developed alloy series was not equivalent to pure Ti (Grade 4), it was smaller than that of Ti-6Al-4V, and it is judged that the hot workability of the alloy series is not a problem in terms of mill power for hot rolling. On the other hand, the hot ductility of Ti-3.5Al-1Fe was nearly equivalent to that of ASTM grade 4, while that of Ti-5.5Al-1Fe was higher than that of Ti-6Al-4V. In other words, it has been confirmed that the hot ductility of the developed alloy series was higher than that of conventional high strength titanium alloys and that the hot workability of the alloy series is not a concern.

3. Material properties of Ti-5.5Al-1Fe hot-rolled bar

Among the Ti-Fe-Al based alloys presented in section 2, Ti-5.5Al-1Fe is a substitute composition for Ti-6Al-4V, and is thought to have a wide range of potential applications. We therefore studied the fatigue properties, the strengthening heat treatment and matters which need some attention in the actual use of this alloy.

3.1 Material

The material used for the experiment was a bar of 15 mm in diameter which was manufactured by double vacuum arc remelting, hot forging, and hot rolling, and its composition was Ti-5.5Al-1Fe-0.15%O-0.075%N (0.36[O]eq). Microstructures of the annealed (750°C, 1h, air cooled) bar are shown in Photo 1, and are the same mill-annealed microstructures as those in the $\alpha + \beta$ processed conventional $\alpha + \beta$ type titanium alloys such as Ti-6Al-4V.

3.2 Fatigue properties

Fig. 4 shows the rotational bending fatigue properties. The S-N curve of hot-rolled and annealed bars of Ti-5.5Al-1Fe-0.08[O]eq, which has different [O]eq from Ti-5.5Al-1Fe-0.36[O]eq, is also shown in the figure. The tensile strength of Ti-5.5Al-1Fe-0.36[O]eq was about 1,100 MPa, which was nearly equivalent to that of conventional Ti-6Al-4V bars (Fig. 2). However, Fig. 4 shows that the fatigue strength of Ti-5.5Al-1Fe-0.36[O]eq is positioned above the reported fatigue strength range of Ti-6Al-4V, and the ratio of fatigue strength to tensile strength is relatively high. It is also shown that in Ti-5.5Al-1Fe-0.08[O]eq whose tensile strength is lower by about 250 MPa, the fatigue strength is not so low as the lower portion of fatigue strength band of Ti-6Al-4V. Those results mean that the developed compositions have excellent fatigue properties.

3.3 Strengthening heat treatment

Strength levels of $\alpha + \beta$ type titanium alloys can be further raised by solution treatment and aging (STA) in which the alloys are heated and maintained in the high $\alpha + \beta$ temperature region, quenched, then aged at lower temperature around 500°C. Since Ti-5.5Al-1Fe is an $\alpha + \beta$ type alloy, the strength of the alloy can be further increased by a similar STA process. If an STA process under optimum conditions is executed, strength can be raised by up to 200MPa. That is, tensile strength of about 1,100 MPa in an annealed material can be increased to about 1,300 MPa by applying solution treatment at 940°C, water quenching, then aging at 500°C for 2 to 4 h.

However, some attention must be paid when applying the STA

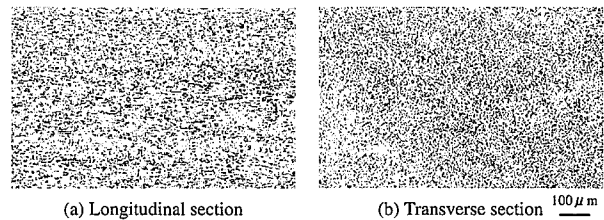


Photo 1 Optical microstructures of Ti-5.5Al-1Fe-0.15%O-0.075%N (0.36[O]eq) hot-rolled and annealed bar (15 mm in diameter)

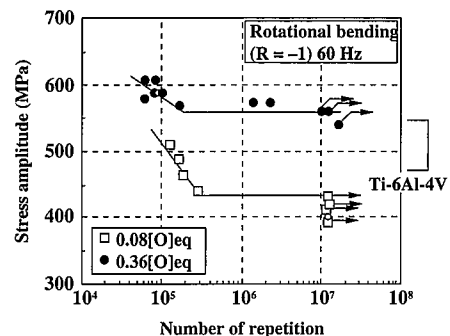


Fig. 4 Rotational bending fatigue properties of hot-rolled and annealed bars of Ti-5.5Al-1Fe

process for this alloy. Fig. 5 shows the tensile properties of the alloy solution treated at 940°C, water quenched, then aged at 500°C. Fairly high levels of strength and ductility could be attained by solution treatment and water quenching alone, but the strength was increased further by aging, and strength as high as 1,300 MPa could be attained by aging for about 4 h with attaining about 10% elongation at the same time. Aging for 8 h or longer led to over-aging and reduced the strength. Therefore, unnecessarily longer aging should be avoided. Furthermore, since it was confirmed that aging for 8 h or longer formed the FeTi intermetallic compound⁷⁾, unnecessarily longer aging should be avoided from this point of view as well.

Photo 2 shows microstructures of an STA processed specimen, which consists of the equiaxed α phase and the transformed β phase. In the transformed β phase, the majority of the β phase transformed into fine acicular martensite (α'') when it was quenched from the solution treatment temperature (part of the β phase was retained), and the martensite phase was converted to the mixture of the stable lath type α phase and the β phase by subsequent aging⁷⁾. As shown in Photo 3, the FeTi compound which is formed by over-aging is extremely fine and formed along the lath interface.

3.4 Phase stability

It was mentioned in the previous section that when the alloy was exposed to a certain environment, the FeTi phase was formed. Although the alloy is classified into $\alpha + \beta$ titanium alloy having the α and β phases as the main phases, it consists of two phases of the α and the FeTi phases in an equilibrium condition at room temperature. However, the rate of formation of FeTi is generally considered to be sluggish, thus making it possible to use the alloy in the state of the α and β two phases. It is thus necessary to clarify whether FeTi may be



Photo 3 FeTi phase formed in the transformed β phase of Ti-5.5Al-1Fe (-0.36[O]eq) over-aged in the STA process

formed during actual use as well as the mechanical properties when it is formed, with regard to not only STA processed materials but also general annealed ones. In this respect, the annealed specimens of the alloy were exposed to temperatures of 300°C and 450°C for a long time, and the change of tensile properties at room temperature was examined.

When the materials were exposed at 300°C for a long time, no remarkable change of tensile properties at room temperature was observed in a specimen exposed for 2,048 h. Examination with an X-ray diffractometer also revealed no formation of FeTi. Consequently, it is considered that at temperatures below 300°C, FeTi would not form and deterioration of the material properties would not occur.

On the other hand, when the specimens of Ti-5.5Al-1Fe (-0.36[O]eq) bar were exposed at 450°C for a long time, an apparent change in both the strength and ductility at room temperature was observed, as shown in Fig. 6. At the initial stage up to 100 h of exposure time, 0.2% proof stress and tensile strength increased with exposure time, while ductility gradually decreased slightly. It is well known that in general, when an alloy containing a high concentra-

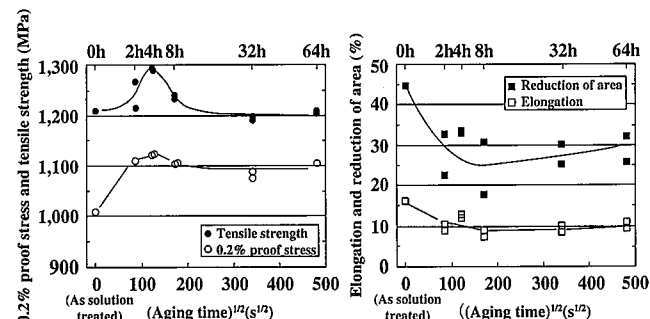


Fig. 5 Effect of aging at 500°C on tensile properties of hot rolled bars of 15 mm in diameter of Ti-5.5Al-1Fe (-0.36[O]eq) which was waterquenched after being solution treated at 940°C

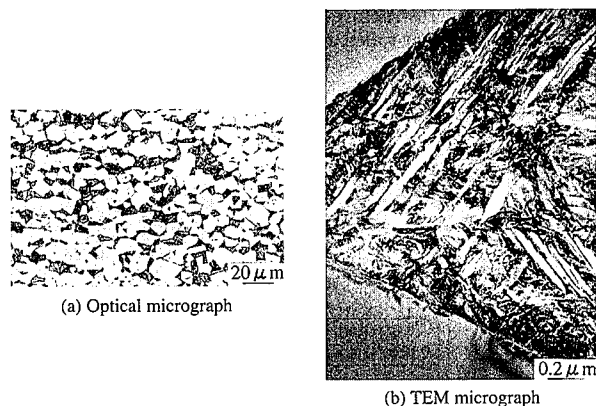


Photo 2 Microstructures of Ti-5.5Al-1Fe (-0.36[O]eq) solution treated and aged (940°C, 1h, water quenching + 500°C, 4 h, air cooling)

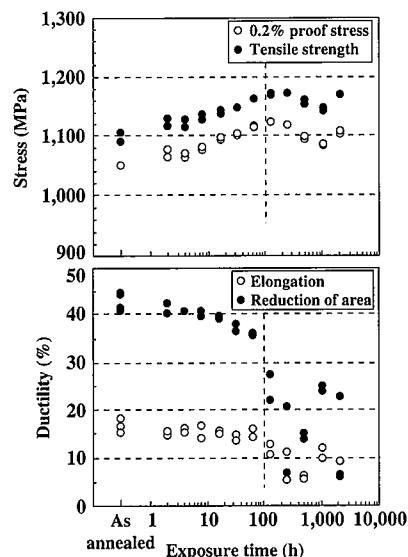


Fig. 6 Effect of long time exposure at 450°C on tensile properties at room temperature of Ti-5.5Al-1Fe (-0.36[O]eq) hot rolled and annealed bar

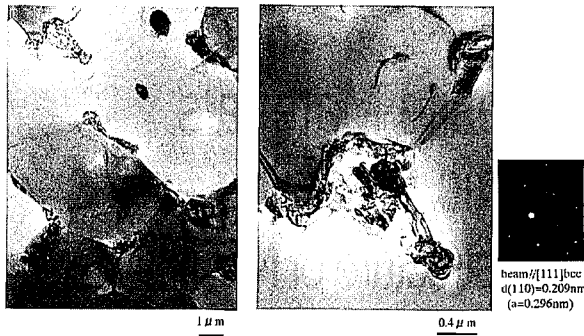


Photo 4 TEM microstructures of Ti-5.5Al-1Fe (-0.36[O]eq) hot rolled and annealed bar exposed at 450°C for 1,024 h (Since FeTi has the same bcc structure as the β phase, the FeTi phase has been identified by comparing the lattice parameters)

tion of Al is exposed to the temperature of 400 to 600°C for a long time, short-range ordering of Ti_3Al or α_2 precipitation occurs. Since this alloy also contains 5.5% Al, and the content of oxygen which helps promote the ordering is high, the change of tensile properties is thought to be due to the short-range ordering of Ti and Al.

However, after the exposure for 100 h or longer, strength decreased, and ductility also decreased suddenly. In the X-ray diffraction analyses, formation of FeTi was observed. Therefore, there is a high possibility that the change in properties in the specimens exposed for 100 h or longer is related to the formation of FeTi. The FeTi is the transformation product in the Fe-concentrated β phase, as shown in **Photo 4**, and the volume fraction of the former β phase is about 10%. If the majority of the former β phase is presumed to have transformed to FeTi, this is very likely to have caused the embrittlement.

As described above, the developed alloy series are not necessarily appropriate alloys when used at a high temperature such as 450°C for a long time, at which temperatures it is desirable to use other alloys. In the case that the alloy series must be used, it is necessary to clarify the conditions causing the embrittlement phase and to avoid using the alloys in this range where embrittlement occurs.

3.5 Superplasticity

The alloy series exhibit superplastic behavior similar to that of some other $\alpha + \beta$ type titanium alloys when equiaxed microstructures are developed⁹⁻¹¹⁾. In that case, the alloy series have the characteristics that superplastic behavior appears over a wide temperature range, and so are easy to use industrially. Refer to referenced literatures 9) to 11) for the details of superplasticity.

4. Conclusion

To reduce the manufacturing cost of $\alpha + \beta$ type titanium alloys containing expensive β stabilizing elements such as V, Ti-Fe-Al based titanium alloy series having various strength levels were developed. In particular, Ti-3.5Al-1Fe and Ti-5.5Al-1Fe were proposed as substitutes for the conventional workhorse alloys Ti-3Al-2.5V and Ti-6Al-4V respectively.

Ti-5.5Al-1Fe has excellent fatigue properties in addition to high strength and high ductility. Furthermore, higher strength can be attained by solution treatment and aging, and the alloy has superplasticity. This alloy may be widely used in applications where Ti-6Al-4V has been used conventionally.

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