The Effect of Cooling Rate from Solution Treatment Temperature on Phase Constitution and Tensile Properties of Ti-4.3Fe-7.1Cr-3.0Al Alloy

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Titanium and its alloys are one of very attractive metallic materials for health-care and welfare goods, because these alloys have high specific strength and high biocompatibility. However, high cost of Ti alloys is disadvantage in application to the health-care and welfare goods. To overcome high cost barrier of Ti alloys, Ti-4.3Fe-7.1Cr-3.0Al alloy was developed. This alloy has good tensile properties, i.e. about 1 GPa as tensile strength, about 20% as elongation and about 50% as reduction in area, in solution treated state. It is very important that effect of cooling rate from solution treatment temperature on tensile properties is investigated, because diffusion coefficients of Fe and Cr in beta phase are higher than other beta stabilizers, e.g. V and Mo. When a β stabilizer with higher diffusion coefficient is contained in β phase, isothermal α precipitation that makes the alloy brittle becomes fast. To suppress isothermal α precipitation, it is necessary to set the cooling rate higher than an appropriate value. In this study, the effect of cooling rate from solution treatment temperature on phase constitution and tensile properties was investigated by electrical resistivity and Vickers hardness measurement and tensile test in Ti-4.3Fe-7.1Cr-3.0Al alloy. In Ti-4.3Fe-7.1Cr-3.0Al alloy cooled by a cooling rate of 0.46 Ks⁻¹ or more, only β phase was identified by X-ray diffraction, while β and α phases were identified in the alloy cooled by furnace cooling, i.e. 0.024 Ks⁻¹. Resistivity ratio remained almost constant between 0.46 Ks⁻¹ and 3.47 Ks⁻¹. In specimen cooled by 0.024 Ks⁻¹, resistivity ratio significantly decreased and HV drastically increased because of α phase precipitation. Tensile strength remained about 1020 MPa between 0.46 Ks⁻¹ and 3.47 Ks⁻¹. In specimen cooled by 0.024 Ks⁻¹, tensile strength slightly increased. Elongation remained almost constant between 0.85 Ks⁻¹ and 3.47 Ks⁻¹, and then decreased with decrease in cooling rate below 0.46 Ks⁻¹.

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1. Introduction

Life expectancy in most countries has increased, and elderly population is on the rise.¹) In Japan alone, the elderly population (above 65 years old) will increase by 27% within the next two decades. Therefore, developing supporting equipment for the care of the elderly people is very important. Titanium and its alloys, especially β titanium, are very attractive materials for use in such supporting equipment, due to virtue of their high specific strength and good biocompatibility.²) However, β titanium alloys are relatively expensive, because of the use of high-cost β stabilizing elements such as vanadium.³) Low-cost Ti-4.3Fe-7.1Cr and -3.0Al alloys, developed by the present authors, have an attractive balance of tensile strength and ductility;⁴) i.e., a tensile strength of about 1 GPa and a reduction in area of about 50%. In this alloy, which includes Fe having a high diffusion coefficient,⁵) an isothermal α phase is precipitated by aging, making the alloy brittle.⁶) Therefore, these alloys are highly prone to precipitation of the isothermal α phase upon cooling from solution treatment temperature. An important step for realizing application to supporting equipment; e.g., health-care goods, is investigation of phase precipitation due to cooling at various cooling rates. In order to clarify the phase precipitation during cooling at various cooling rates, the effect of average cooling rate from solution treatment temperature on phase constitution and tensile properties was investigated through measurement of electrical resistivity and Vickers hardness, X-ray diffractometry, and tensile testing.

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2. Experimental Procedure

Ti-4.3Fe-7.1Cr-3Al alloy was prepared by the Plasma Progressive Casting Furnace Process, followed by the Vacuum Arc Remelting Process.⁷) The chemical composition of this alloy is 4.19 mass% Fe, 6.95 mass% Cr, 3.06 mass% Al, and 0.15 mass% O, with Ti accounting for the balance. The obtained ingots were hot-forged at about 1270 K and then hot-rolled at about 1170 K to form 25 mm diameter round bars. Round bars of 20 mm diameter and 100 mm length were prepared from the hot rolled round bar by machining. Each prepared round bar was solution treated at 1123 K for 3.6 ks and then cooled to room temperature or ice water temperature by one of five different cooling methods. The five different cooling methods are as follows: quenching in ice water (WQ), cooling by an electric fan in the atmosphere (EFAC), air cooling (AC), cooling by maintaining the specimen in a Tammann reaction tube in the atmosphere (TAC), and furnace cooling (FC). Average cooling rates of the five cooling methods were measured by following method. A round bar measuring 20 mm in diameter and 70 mm in length for measuring cooling curve was prepared from the hot rolled Ti-4.3Fe-7.1Cr-3.0Al alloy bar by machining. In the round bar for measuring cooling curve, a longitudinal hole was drilled to the center of the specimen. An alumel-chromel thermocouple was inserted in the hole and the hole was sealed by ceramic cement. The bar specimen was maintained at 1123 K for 3.6 ks and then cooled by five different cooling methods. On cooling, time and temperature were measured by a high-speed digital data recorder (YEW Digitalscope). Measured data were analyzed and time-temperature curves were drawn by a personal computer. The time over which temperature dropped from 948 to 473 K was measured. Average cooling rate was calculated by dividing 475 K by the
measured time. Figure 1 shows cooling curves measured by the five different cooling methods, along with average cooling rates obtained by the five cooling methods; i.e. 34.7 Ks$^{-1}$ for WQ, 2.45 Ks$^{-1}$ for EFAC, 0.85 Ks$^{-1}$ for AC, 0.46 Ks$^{-1}$ for TAC, and 0.024 Ks$^{-1}$ for FC. After cooling, a tensile test specimen measuring 6 mm in diameter and 20 mm in gage length was prepared from the round bar. Before tensile testing, the electrical resistivity of each tensile test specimen was measured at room and liquid nitrogen temperatures ($\rho_{RT}$ and $\rho_{LN}$). Tensile tests were performed at room temperature and a crosshead speed of $5 \times 10^{-5}$ m s$^{-1}$. For each cooling method, three specimens were subjected to tensile tests. After tensile testing, Vickers hardness of specimens prepared from the grip portions of tensile test pieces was measured. Phase constitution was identified using X-ray diffractometry (XRD) in the same specimens as used for HV measurement. Fracture surfaces were also observed by SEM.

3. Results and Discussion

Figure 2 shows optical microscope images (OM), X-ray diffraction profiles, and values of average $\beta$ grain sizes of specimens in various cooled states. Only $\beta$ reflection was identified by XRD in specimens quenched by the WQ, EFAC, AC, and TAC methods. In these specimens, $\beta$ grains were observed only by OM, and all cooling methods yielded almost the same average $\beta$ grain size. In the specimen cooled by FC, precipitated $\alpha$ was observed by OM and reflections of the $\alpha$ and $\beta$ phases were identified by XRD. At the right side of the OM, non-recrystallized structure was also observed. Non-recrystallized structure formed by hot-forging and hot-rolling may remain in the alloy after solution treatment. The non-recrystallized structure had no unfavorable effects on tensile properties, as mentioned later.

Figure 3 shows changes in electrical resistivity at room and liquid nitrogen temperatures ($\rho_{RT}$ and $\rho_{LN}$), resistivity ratio ($\rho_{LN}/\rho_{RT}$), and Vickers hardness (HV) versus average cooling rate. Resistivity at RT and LN remained almost constant between 34.7 (WQ) and 2.45 Ks$^{-1}$ (EFAC). At both temperatures, resistivity increased slightly with decreasing average cooling rate until 0.46 Ks$^{-1}$ (TAC), and then drastically decreased at 0.024 Ks$^{-1}$ (FC). The increase of resistivity from 2.45 to 0.46 Ks$^{-1}$ is considered to be due to phase separation of $\beta$ phase. The increase of resistivity due to $\beta$ phase separation has already been reported in Ti-13V-11Cr-3Al alloy by G. H. Narayanan.\textsuperscript{7} The drastic decrease at 0.024 Ks$^{-1}$ (FC) is caused by $\alpha$ precipitation, as shown in Fig. 2. In $\beta$ Ti alloys, content of $\beta$ stabilizing element in $\beta$ phase increases with increasing $\alpha$ precipitation. Generally,
resistivity of β Ti alloys; e.g., Ti-V binary β alloys, decreases with increasing content of the β stabilizing element; e.g., V. Therefore, resistivity of β Ti alloys, including, of course Ti-4.3Fe-7.1Cr-3.0Al alloy, decreased with increasing α precipitation. Resistivity ratio \( \rho_{\text{LN}} / \rho_{\text{RT}} \) remained almost constant between 34.7 Ks (WQ) and 0.46 Ks (TAC) and then decreased drastically at 0.024 Ks (FC) because of α precipitation. HV remained almost constant between 34.7 Ks (WQ) and 2.45 Ks (EFAC) and then drastically increased at 0.024 Ks (FC). HV began to increase at 0.85 Ks (AC) and then drastically increased at 0.024 Ks (FC). β phase separation is considered to cause the increase of HV between 34.7 Ks (WQ) and 0.46 Ks (TAC). The drastic increase of HV at 0.024 Ks (FC) is due to α precipitation, which was identified by XRD and is shown in Fig. 2.

Figure 4 shows changes in tensile strength \( \sigma_B \), 0.2% proof strength \( \sigma_{0.2} \), elongation \( \delta \) and reduction in area \( \phi \) with increase of average cooling rate.

A region of balance between 0.2% proof strength and reduction in area in developed Ti alloys was reported by Kawabe and Muneki. Tensile strength \( \sigma_B \) of about 1000 MPa, 0.2% proof strength \( \sigma_{0.2} \) of about 970 MPa, elongation \( \delta \) above 15%, and reduction in area \( \phi \) above 50%.

In the tensile specimen cooled at 0.024 Ks (FC), an intergranular fracture area was mainly observed, although some dimple areas were observed. Intergranular fracture supports properties of the furnace-cooled specimen such as
From these data. When resistivity ratio is measured in the Ti-4.3Fe-7.1Cr-3.0Al alloy cooled at various cooling rates, tensile properties of the alloy are easily estimated from measured resistivity ratio, by use of the regression line. This method may become useful method for estimating tensile properties of Ti-4.3Fe-7.1Cr-3.0Al alloy.

In Ti-4.3Fe-7.1Cr-3.0Al alloy specimens cooled at average cooling rates faster than 0.46 Ks\(^{-1}\) (TAC), phase transformation; i.e., isothermal \(\omega\) precipitation or \(\alpha\) precipitation, was not clearly observed, and tensile properties; i.e. tensile strength, elongation, and reduction in area, showed almost constant values; i.e. about 1 GPa, about 18%, and about 55%. These results indicate that when cooled at an average cooling rate faster than 0.46 Ks\(^{-1}\) (TAC), this alloy has good balance between strength and ductility, and this balance is in no way inferior to the balance of other \(\beta\) Ti alloys; e.g., Ti-4.5Fe-6.8Mo-1.5Al alloy.\(^\text{11)}\)

### 4. Conclusions

The effect of cooling rate from solution treatment temperature on phase constitution and tensile properties of Ti-4.3Fe-7.1Cr-3.0Al alloy was investigated by measurement of electrical resistivity and Vickers hardness, X-ray diffractometry, and tensile testing. Obtained results are as follows.

In the specimens cooled at average cooling rate faster than 0.46 Ks\(^{-1}\) (TAC), \(\beta\) phase was only identified by X-ray diffraction. Electrical resistivity and resistivity ratio remained almost constant. Vickers hardness increased slightly with decreasing average cooling rate from 2.45 to 0.46 Ks\(^{-1}\). Tensile strength, 0.2% proof strength, and elongation remained almost constant. Reduction in area decreased low elongation and low reduction in area.

Figure 6 shows the relationships between tensile strength \(\sigma_B\), reduction in area \(\phi\) and elongation \(\delta\) versus resistivity ratio \(\rho_{LN}/\rho_{RT}\). Although data for each relationship are clearly separated into two areas, a regression line was calculated.

![Fig. 6 Relationship between tensile properties, \(\sigma_B\), \(\phi\) and \(\delta\) and resistivity ratio \((\rho_{LN}/\rho_{RT})\) in Ti-4.3Fe-7.1Cr-3.0Al alloy cooled by five different cooling rates.](image-url)
slightly with decreasing average cooling rate from 2.45 to 0.46 Ks\(^{-1}\).

In the specimen cooled at 0.024 Ks\(^{-1}\) (FC), \(\beta\) and precipitated \(\alpha\) phases were identified. Resistivity and resistivity ratio decreased drastically and HV increased significantly. Tensile strength and 0.2% proof strength increased significantly, whereas elongation and reduction in area decreased drastically. In a word, by cooling at 0.024 Ks\(^{-1}\) (FC), the specimen became brittle.

The tensile properties of the Ti-4.3Fe-7.1Cr-3.0Al alloy cooled at average cooling rates faster than 0.46 Ks\(^{-1}\) (TAC) are equal to, or more excellent than, those of other \(\beta\) Ti alloys; e.g., Ti-4.5Fe-6.8Mo-1.5Al alloy.

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REFERENCES