

A study on the microstructural property and thermal property of Ti-alloys without Al as biomaterials

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Abstract

Ti-10Ta-10Nb alloys were designed for surgical implants, dental and orthopedic materials without V and Al. Specimens of the Ti-10Ta-10Nb alloy were remelted three times through the consumable VAR process and were made into small rods. Homogenization heat treatment was carried out for 24 hours under a vacuum of 10^{-3} torr and at constant temperature of 1050°C and then the specimens were cooled in water. After that, we observed the microstructure of the alloy by using an SEM. Rockwell (B) hardness, thermal expansion coefficient and specific heat of the Ti-10Ta-10Nb alloy were measured in order to examine the material properties. It was found that the mechanical property of the specimen was altered by the heat treatment, and thermal expansion coefficient and specific heat of the Ti-10Ta-10Nb alloy would be useful data for engineering processing design.

Keywords: Heat treatment; Thermal expansion coefficient; Specific heat; Microstructure

1. Introduction

Although Ti and its alloys were introduced as biomaterials in the 1940's, late in the Second World War, studies on their biomedical application were very late compared to the Co-Cr alloy or 316 L stainless steel, and they were not used as biomaterials until the late 1980's [1]. According to a biocompatibility study of the metallic biomaterials, V and Co are classified as materials having cytotoxicity; and Mo, Al, and 316 L stainless steel are classified as protective membrane forming materials. Therefore, it was necessary to develop new biomaterials which had better biocompatibility characteristics than the widely used Cr-Co alloys and 316 L stainless steels [2, 3].

In the beginning, Ti and its alloys were first found as a form of FeTiO₃ in the late 1700's, and they were extracted as pure metallic elements in the 1800's be-

cause of their extremely strong bonding with oxygen. Then in the early 1950's, as they could be mass-produced by America and Japan, the uses of Ti alloys were expanded. After the 1970's, as forging techniques for Ti alloys showed rapid progress, they were used as a component of products which require high-strength and light weight such as engines, missiles and aircraft. Nowadays, Ti alloys with appropriate mechanical properties required such as tensile strength, ductility, fracture toughness, creep intensity, resistance against fatigue crack propagation have been studied and developed through control of microstructure, heat-treatment and mechanical processing. [4, 5]. The representative Ti alloy developed for these studies is Ti-6Al-4V, which has been used as a component of military equipment, such as aircraft and missiles, sporting goods and widely applied in the medical industry such as in orthopedics and dentistry.

However, in spite of the outstanding biocompatibility of Ti alloy, it has been constantly reported that the elements of Al and V in Ti alloy provide causes of

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bio-synthesis and specific diseases. Therefore, a new alloy system needs to be developed which can replace them. It has been reported that a Ti-10Ta-10Nb alloy was developed by researchers, who needed a new material showing very outstanding biocompatibility, satisfactory processing capability and abrasion resistance condition for use in the medical industry [6]. But its thermal behavior has not been completely identified yet.

In this study the elements of Al and V were excluded from the Ti-6Al-4V alloy, which might be a problem of biocompatibility, and the elements Ta and Nb were added to increase the biocompatibility. In order to find the optimal hot working temperature zone of the Ti-10Ta-10Nb alloy, the characteristics of thermal property change are very important.

2. Method of experiment

2.1. Thermal treatment

In order to decide the homogenization heat-treatment temperature of the Ti-10Ta-10Nb alloy roughly, Ti-Ta and Ti-Nb phase diagrams [7-8] were reviewed, as shown in Fig. 1. Based on these phase diagrams, this study carried out a homogenization heat-treatment at 1050°C for 24 hours under a vacuum of 10^{-3} torr. This holding temperature is far above the T_{β} phase transformation temperature 800°C. After heat treatment the specimens were cooled in water. Microstructure of the homogenized specimens was observed with an SEM (Scanning Electron Microscope). And the mechanical property of the specimens was measured with a Rockwell (B) hardness tester.

Specimens of Ti-10Ta-10Nb alloy were remelted three times in the consumable VAR equipment, and they were cast into small rods.

2.2 Measurement of thermal expansion coefficient

The length change of the specimens was measured to identify the thermal expansion behavior of Ti-10Ta-10Nb alloy at β phase transformation zone using a Dilatometer (DIL type 802, Germany). The shape of the cylindrical specimen was $\phi 3$ mm in diameter and 10mm in length. The temperature ascending speed was selected as 5°C per minute within a test temperature range from 15°C to 1,100°C. To decide a more precise thermal expansion coefficient at the temperature range below T_{β} , this study also controlled the temperature ascending speed to 3°C per minute within 15°C ~700°C. As a test tool, TMA 2940 produced by TA Instrument was used. An expansion probe was put on a specimen and the thermal expansion coefficient was measured by using one.

3. Results of test

3.1 Microstructures according to thermal treatment

Fig. 2 shows microstructures of the resolidified Ti-10Ta-10Nb alloy by using a VAR and the homogenization heat treated structure for 24 hours at 1050°C. There are much more dendritic α -phases in the resolidified Ti-10Ta-10Nb alloy. And it was found that the dendritic α -phase in the homogenization treated one grew bigger in size. In general, for Ti alloys, it has been known that the more β -phase, the better the tensile strength and fatigue characteristics, β -phase

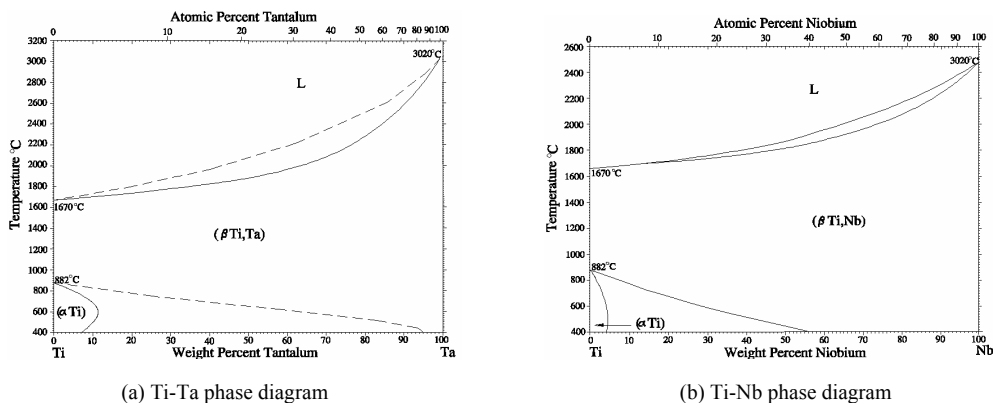


Fig. 1. Binary alloy phase diagrams of (a) Ti-Ta, (b) Ti-Nb [7].

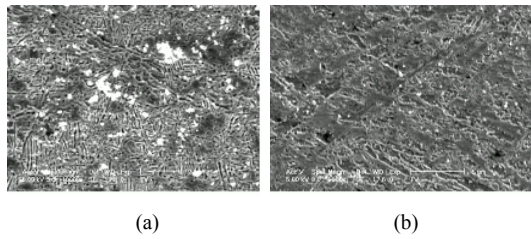


Fig. 2. SEM micrographs of the Ti-10Ta-10Nb alloy, (a) as-cast and (b) homogenization treated at 1050°C.

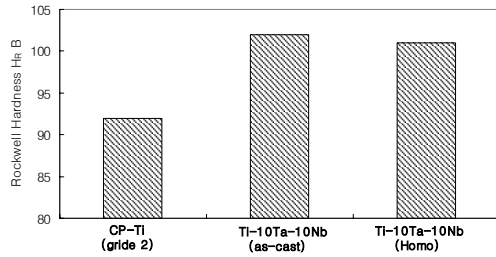


Fig. 3. Rockwell hardness of the Cp-Ti alloy and as-cast and homogenized Ti-10Ta-10Nb alloy.

impedes dislocation movement within the α matrix and at grain boundaries. And it can improve creep strength and tensile strength. In reviewing the Rockwell hardness test results for the as-cast alloy and the homogenized specimens, it was found that the hardness of CP-Ti was about 93 and that of Ti-10Ta-10Nb alloy was about 102~105. It is thought that such results were obtained due to solid solution reinforcement by addition of two alloys (Ta and Nb) to α -phase Ti. Fig. 3 shows the hardness values of the CP-Ti, as-cast Ti-10Ta-10Nb alloy and the homogenized Ti-10Ta-10Nb alloy.

3.2 Thermal expansion property of Ti-10Ta-10Nb alloy

The thermal expansion property of the $\alpha+\beta$ type Ti alloy shows sharp change around the transformation temperature (T_β). Fig. 4 shows the thermal expansion coefficient of the Ti-10Ta-10Nb alloy according to temperatures. The length variation of the alloy according to temperatures is roughly divided into three sections. The first section ranges from normal temperature to around 720°C, where it was observed the length variation inclination had a certain value. The second section ranges from 720 to 910°C, where T_β temperature is included and the amount of length extension decreases. The third section, which is a complete β area (Single phase), shows the same

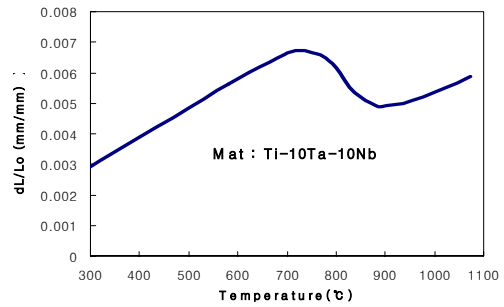


Fig. 4. Variation of Ti-10Ta-10Nb alloy according to thermal treatment.

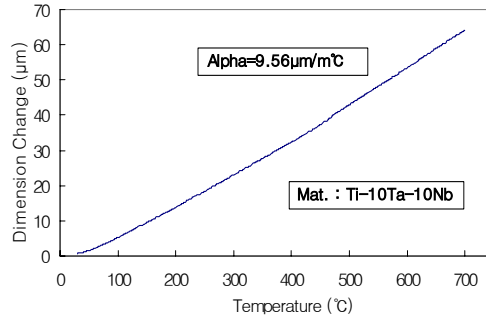


Fig. 5. The dimensional change of Ti-10Ta-10Nb alloy with heating temperature by TMA.

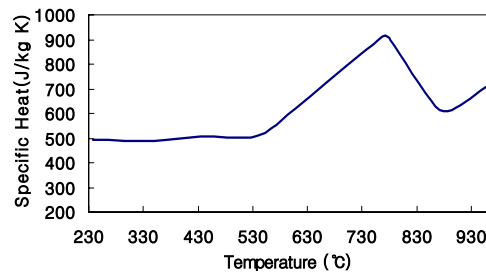


Fig. 6. Variation of specific heat of Ti-10Ta-10Nb alloy according to temperatures

variation behavior as the first section. The same tendency was observed both in the casting structure and the swaging structure. In Fig. 5, the temperature rose to 3°C per minute with the use of TMA (Thermo Mechanical Analyzer, TMA 2940) and the thermal expansion coefficient was measured through more precise temperature control. Temperatures were increased from 20°C to 700°C, and the thermal expansion coefficient of both the casting structure and the swaged structure was about 9.56 $\mu\text{m}/\text{m}^\circ\text{C}$, showing no significant difference, as seen in Eq. (1). And this study measured specific heat of the Ti-10Ta-10Nb alloy as shown in Fig. 6. The specific heat was meas-

ured up to 1000°C where it was thought the microstructure has a complete β -phase. In reviewing specific heat variation curves obtained from this experiment, the specific heat curves can be expressed in different ways based on the β phase transformation temperature (T_β). TTR (transformation temperature range) was found as α phase in the matrix began to transform into the β phase from 550°C and finished around 880°C.

$$\alpha = 9.56\mu\text{m}/\text{m}^\circ\text{C} \text{ (Thermal Expansion Coefficient)} \\ \text{at heating rate of } 3^\circ\text{C}/\text{min} \quad (1)$$

$$C_p = 0.0029T^2 - 2.1266T + 857.52 \text{ J/kg K } (\alpha+\beta\text{-microstructure)} \\ \text{at heating rate of } 3^\circ\text{C}/\text{min} \quad (2)$$

$$C_p = 0.0226T^2 - 40.451T + 18704 \text{ J/kg K } (\beta\text{-microstructure)} \\ \text{at heating rate of } 3^\circ\text{C}/\text{min} \quad (3)$$

4. Conclusion

As a result, it was known for a material property to be changed according to thermal treatment, and the thermal expansion coefficient and the specific heat of Ti-10Ta-10Nb were measured, which would be some fundamental data for engineering processing. This study obtained SEM pictures at different temperatures as it changed, and considered physical characteristics such as volume fraction of α -phase, hardness change, and specific heat change to obtain the following results:

- 1) The temperature of Pure-Ti T_β which is α stabilization element was 882°C according to bibliography [7] and for Ti-10Ta-10Nb which is a target alloy of this study, Ta and Nb, both of which are β stabilizing elements, were added to Pure-Ti base, and it was found that the β stabilized area expanded.
- 2) This study increased temperature at a very slow pace (3°C/min) from the normal temperature of $\alpha+\beta$ -phase area to the complete β -phase area using TMA, a thermal analysis device and a Dilatometer, and measured the dimensional change of the material to find the thermal expansion coefficient of Ti-10Ta-10Nb to be $\alpha = 9.56\mu\text{m}/\text{m}^\circ\text{C}$ at heating rate of 3°C/min.
- 3) For specific heat of Ti-10Ta-10Nb alloy, it was divided into single area and 2-phase area and each area showed the following values:
 $C_p = 0.0029T^2 - 2.1266T + 857.52 \text{ J/kg K}$ ($\alpha+\beta$ Phase) at heating rate of 3°C/min.
 $C_p = 0.0226T^2 - 40.451T + 18704 \text{ J/kg K}$ (β Phase) at heating rate of 3°C/min.

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