STUDY ON FORMATION OF \( \beta \) SINGLE PHASE OF A Ti-10at\%Mo-10at\%Cr ALLOY ON SINTERING AND SOLUTION TREATMENT

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Abstract: In this study, the formation of the \( \beta \) single phase of the Ti-10at\%Mo-10at\%Cr alloy on sintering and solution treatment is investigated. A powder metallurgy method is applied as a fabrication method. Alloying of metallic powders occurs on sintering. Formation of a mixture phase such as \( \alpha' \), \( \alpha'' \) and \( \beta \) is promoted with increasing the sintering time. Moreover, microstructural change to coarse plate-like microstructure and diffusion of alloying elements to Ti matrix occur when the sintering time prolongs. On the other hand, the \( \beta \) single phase can be obtained when the sample is subjected to solution treatment at 1373K for 3.6ks and water-quenching. The rapid cooling from the elevated temperature can retard the formation of the \( \alpha' \) and \( \alpha'' \).

Key-Words: \( \beta \) phase, sintering, diffusion, solution treatment, plate like structure, equiaxed structure

1 Introduction

The demands for metallic implant and biomaterials are very high in recent years. In the United State, titanium, steel and other metals for biomaterial will reach $212.8 million in 2008 [1]. Moreover, it is projected that approximately 272,000 total hip replacements will annually be performed by 2030 [2]. Since Ti alloys have higher specific strength and excellent corrosion resistance...
resistance than other alloys such as Co-Cr-Mo alloys and stainless steels [3, 4], the commercially pure (cp) titanium and Ti-6Al-4Al alloys have been used as metallic orthopaedic implant materials. On the other hand, the Ti alloys still suffer from a large degree of biomechanical incompatibility, due to their relatively high elastic modulus (about 120 GPa), compared with that of the bone (max. 30 GPa). They will cause stress shielding, eventually causing bone loss and premature failure of the artificial hip. Moreover, the Ti-6Al-4V alloy can also release toxic ions (e.g. V and Al ions) into the body, leading to undesirable long-term effects [5]. A number of researchers have developed beta type of Ti alloy. Since bcc (β) structures have lowered the elastic modulus, the β structure type alloys are designed as new Ti alloys. However, such kinds of alloys contain high concentration of alloying elements such as Hf, Ta, Nb and Zr [6-8]. Thus, the cost of the alloys increases.

On the other hand, authors have developed a new β Ti alloy, i.e. Ti-10at.%Mo-10at%Cr alloy by powder metallurgy method. It is found that addition of 10% Cr will stabilise the β phase and also will decrease the hardness of the Ti-10%Mo alloy. Since the previous investigation have applied the powder metallurgy method, which pure metal powders are applied as raw materials, it is important to clarify change in phase and microstructure during fabrication. In this study, formation of β single phase of the Ti-10at%Mo-10at%Cr alloy on sintering and solution treatment is investigated as an initial step to develop a new β type Ti alloy.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Bo and Md values of the element used.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
<td>Bo</td>
</tr>
<tr>
<td>Ti</td>
<td>2.79</td>
</tr>
<tr>
<td>Mo</td>
<td>3.063</td>
</tr>
<tr>
<td>Cr</td>
<td>2.779</td>
</tr>
</tbody>
</table>

**Figure 1** A phase-stability map in which the area of α, α-β and β type alloys are separated clearly.

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Purity and particle size of powder used.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powders</td>
<td>Purity (%)</td>
</tr>
<tr>
<td>Ti</td>
<td>99</td>
</tr>
<tr>
<td>Mo</td>
<td>99.5</td>
</tr>
<tr>
<td>Cr</td>
<td>99</td>
</tr>
</tbody>
</table>

**Figure 2** OM and SEM micrographs of Ti-10%Mo-10%Cr alloy, which is sintered and solution treated.

2 Experimental Procedures

2.1 Alloy Design

Employing a molecular orbital method, electronic structures were calculated for β Ti-10%atMo-10%Cr alloy, while two alloying parameters were theoretically determined. One is the bond order (Bo), which is a measure of the covalent bond strength between Ti and an alloying element. The other is the metal d-orbital energy level (Md), which correlates with the electronegativity and the metallic radius of alloying element. For Ti-10%Mo-0%Cr alloy, the average values of Bo and Md are defined by taking the compositional averages of the parameters and denote them Bo and Md, respectively. Bo and Md values of the elements used in this study are given in Table 1. Figure 1 is a phase-stability map (called the Bo–Md map) in which the areas of α, α-β and β type alloys are separated clearly. The stability region of the β type alloys extends to the higher Bo and to the lowerMd region. The position of the Ti-10%Mo-10%Cr is shown in solid circle plot within the map. Since position of the Ti-10%Mo-10%Cr alloy is within β phase region, hence the alloy is suitable as a sample in this study.

2.2 Experimental Procedures

The materials used in this study were commercial pure Ti, commercial pure Mo and commercial pure Cr powders. Table 2 shows purity and particle size of used powders. The powders were mechanically mixed for
To fabricate the sample using the cold compaction process, the mixed powder were filled in the die cavity and compressed under the pressure of 1000MPa for 3.6ks using a 50 tones capacity cold press machine at ambient temperature. In order to initiate a solid-state bonding of the particles, all samples were sintered at temperature of 1573K for 0.6ks ~ 14.4ks and the furnace-cooled under argon gas atmosphere after vacuumed. The sample was subjected to solution treatment at 1373K for 3.6ks and the water-quenching.

Microstructures were observed by optical microscopy and Scanning Electron microscopy. Samples for microstructural observation were etched using a Kroll reagent. Distribution of elements in sample was analyzed using SEM/EDS. Phase characterisation was evaluated by means of X-ray diffractometry with Cu-Kα radiation. Hardness was represented by the average of five measurements in Vickers hardness testing (load 19.6N).

3 Results and Discussion

3.1 Microstructure of the Ti-10%Mo-10%Cr alloy

Figures 2 represents an optical and SEM micrographs of the Ti-10%Mo-10%Cr alloy, which is sintered and solution treated. The alloy exhibits equiaxed grains with grain size of 50µm and pores. It is found that solid-state bonding have occurred during sintering. Since Plate-like structures, which indicate α phase are almost not observed, it is thought that the alloy has a β single phase. Hardness of the alloy is Hv 326 and the values is higher than the hardness of other β Ti alloys [9],[10].

3.2 Change in phase and microstructure on Sintering

Figure 3 shows XRD diffractograms of Ti-10%Mo-10%Cr alloys, which are subjected to sintering at 1573K with various sintering times. XRD results shows that sintered samples reveals a mixture of hexagonal α’, orthorhombic α’’ and β phases. It is found that formation of the α’ and α’’ is obviously observed with increasing sintering time. Figure 4 shows SEM micrographs of the samples, which are sintered for 0.6s.

In the samples, there are two kinds of microstructures are observed. The first is a microstructure having different contrast within the microstructure (Fig. 4-a) and the second is a plate-like structure within equiaxed microstructure (Fig. 4-b). In order to clarify the difference between the two microstructures, element analysis and mapping were performed using an SEM/EDS. Figure 5 shows mapping of elements for the microstructure having different contrast. From the mapping results, it is found that there is significant difference in distribution of the alloying element with the matrix. Cr atoms are almost distributed within the sample (Fig. 5-b). On the other hand, Mo atoms are

![Figure 3](image-url) XRD diffractograms of Ti-10%Mo-10%Cr alloys, which are subjected to sintering at 1573K with various sintering times.

![Figure 4](image-url) SEM micrographs of the samples, which are sintered for 0.6s. with various sintering times.

![Figure 5](image-url) mapping of elements for the microstructure having different contrast for 0.6ks sintered sample.

![Figure 6](image-url) mapping of elements for the fine plate-like microstructure for 0.6ks sintered sample.
concentrated in the light area (Fig. 5-c). Besides, Figure 6 shows Figure 5 shows mapping of elements for the plate-like structure (Fig. 4-b). It is found that all of alloying elements are distributed within the sample. On the other hand, the sample, which is subjected to sintering for 5.4ks reveals three kinds of microstructures, e.g. a microstructure having different contrast (Fig. 7-a), a fine plate-like microstructure (Fig. 7-b) and a coarse plate-like microstructure (Fig. 7-c). In order to clarify the microstructures and the diffusion of the elements on sintering, the 5.4ks-sintered sample is observed by SEM/EDS. Figure 8 represents the mapping of the alloying element in the microstructure having different contrast. It is found that the microstructures having different contrast in 0.6ks- (Fig. 5) and 5.4ks-sintered (Fig. 8) samples are similar in the distribution of, e.g. the Cr atoms are distributed within the samples (Fig.8-b), but the Mo atoms are concentrated in the light areas (Fig. 8-c). On the other hand, Figure 9 shows the mapping of elements in the coarse plate-like microstructure. Although the Mo and Cr atoms are slightly concentrated in the light area, the alloying elements are distributed within the samples. On the other hand, the main element, Ti atoms are slightly concentrated in the dark area. Table 3 shows average concentrations of elements in all of microstructures of the 0.6ks- and the 5.4ks-sintered samples.

![Figure 7](image7.png) SEM micrograph of 5.4ks-sintered sample showing three kinds of microstructures; microstructure having different contrast (a), a fine plate-like (b) and a coarse plate-like microstructures (c).

![Figure 8](image8.png) Mapping of elements for the microstructure having different contrast for 5.4ks sintered sample.

![Figure 9](image9.png) Mapping of elements for the coarse plate-like microstructure for 5.4ks sintered sample.

### Table 3 average concentrations of elements in all of microstructures of the 0.6ks- and the 5.4ks-sintered samples.

<table>
<thead>
<tr>
<th>Time</th>
<th>Microstructure</th>
<th>Concent. of element (at%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6ks</td>
<td>Microstructure having contrast</td>
<td>Ti 74.1 Cr 8.1 Mo 16.7</td>
</tr>
<tr>
<td>0.6ks</td>
<td>Fine plate-like within equiaxed</td>
<td>Ti 84.9 Cr 9.8 Mo 4.3</td>
</tr>
<tr>
<td>14.4ks</td>
<td>Microstructure having contrast</td>
<td>Ti 75.7 Cr 9.6 Mo 14.7</td>
</tr>
<tr>
<td>14.4ks</td>
<td>Coarse plate-like</td>
<td>Ti 77.3 Cr 11.2 Mo 11.5</td>
</tr>
</tbody>
</table>

On the other hand, the concentrations of the Mo atoms differ in both microstructures. The Mo concentration in the microstructure having different contrasts is higher than the added Mo concentration (10at%). In the 5.4ks-sintered sample, there are also significant differences in the concentration of the elements. The Cr concentrations are almost the same in the whole microstructures of the 5.4ks-sintered sample. Moreover, the concentration of the Cr reaches the added Cr concentration, i.e. 10at%, in the coarse plate-like microstructure. On the other hand, it is found that the microstructures in the 5.4ks-sintered samples contain various Mo concentrations. The Mo concentration in the microstructure having different contrast, the fine plate-like microstructure and the coarse plate-like microstructure are 14.7at%, 6.0at% and 11.5%, respectively. Therefore, it can be concluded that the sintering process can allow diffusion of alloying element from the pure metal powders to Ti matrix especially the Mo atoms, which the atom size is much bigger than that of the Ti atoms. However, the β single phase could not be obtained because the formation of the α’ or α’’ can occur on slow cooling from the sintering temperature to ambient temperature.
3.3 An effect of Solution Treatment and Water-Quenching on Phase

Since the sintering process reveals the mixture of the phases, the solution treatment with water-quenching is performed to obtain the β single-phase.

Figure 10 represents XRD diffractograms of the 14.4ks-sintered and the solution treated samples. It is found that the β single-phase are obtained in the solution treated sample. Figure 11 shows the mapping of the elements for the microstructure in Fig. 1. It is found that the all of alloying elements are well-distributed within the β-single phase. Therefore, it can be concluded that the solution treatment and water-quenching can stabilize the β single-phase to ambient temperature. It is also thought that the distribution of the elements is almost the same within the sample.

4 Conclusion

The formation of β single phase of the Ti-10at%Mo-10%atCr alloy on sintering and solution treatment is investigated, and following results are obtained.

1. The β single phase of the Ti-10%Mo-10%Cr are obtained through powder metallurgy and sintering and solution treatment.
2. The diffusion of element and change in phase occur during sintering. When the sintering time prolong, a coarse plate-like microstructure with designed chemical composition can be obtained.
3. The β single phase can be obtained by rapid cooling after solution treatment. The rapid cooling can retard the formation of the α’ or α’’.

References:


Figure 10 XRD diffractograms of the 5.4ks sintered sample and the solution treated sample.

Figure 11 SEM micrograph and mapping of the solution treated sample.


