

# Influence of Pre-aging on Microstructure and Mechanical Properties of Coarse Grained $\beta$ titanium Alloy

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**Abstract:** The influence of pre-aging heat treatment on microstructure and mechanical properties of coarse grained  $\beta$  titanium alloy (Ti-3.5Al-5Mo-6V-3Cr-2Sn-0.5Fe) was studied. The results show that the pre-aging heat treatment can serve as a precipitation step to provide uniform dispersed precursors for secondary  $\alpha$  phase precipitation, and then leads to the dense, fine and homogenous distribution of secondary  $\alpha$  phase during the higher temperature heat treatment. Prolonging of the pre-aging time results in the finer scale of secondary  $\alpha$  phase. The pre-aging heat treatment strongly affects the mechanical properties of  $\beta$  titanium alloy. Within a coarse grain condition, the strength tested in this work is about 1340 MPa when single aging at 550 °C. However, the strength achieves to 1760 MPa after pre-aging at 350 °C prior to 550 °C aging, which increases by more than 30% compared to that of single aging. The micro hardness of the alloy is improved markedly by pre-aging. The effect of pre-aging time on mechanical properties is that the prolonging of the pre aging time increase strength and micro hardness, but the ductility is sacrificed, which is mainly due to the refinement of secondary  $\alpha$  phase.

**Key words:**  $\beta$  titanium alloy; pre-aging; microstructure; refinement; mechanical properties

$\beta$  titanium alloys have received considerable attention due to their high strength-to-weight, and are used in a wide range of structural aerospace, chemical industry, biomedical, etc<sup>[1-4]</sup>. An outstanding feature of the  $\beta$  titanium alloys is their especially high strength which can be achieved by forging and heat treatment. The heat treatment strengthening is an effective method of improving mechanical properties of  $\beta$  titanium alloys. The conventional heat treatment strengthening of  $\beta$  titanium alloys includes solution treatment at near  $\beta$  phase transition temperature and aging treatment at a lower temperature. The fine scale secondary  $\alpha$  phase precipitated in aging treatment process is the main reason for increasing of strength<sup>[5]</sup>.

Recently, a duplex aging (DA) method which is different from conventional heat treatment of  $\beta$  titanium alloy attracts the attention of researchers. Santhosh et al<sup>[6,7]</sup>, carried

pre aging heat treatments out at a lower temperature of 250 and 300 °C before normal aging at a high temperature of 500 °C on Ti15-3 alloy, and the results show that the DA causes higher hardness, strength and high cycle fatigue compared to single aging (SA) at 500 °C. Schmidt et al<sup>[8]</sup> show that DA treatments lead to a fine and homogeneous precipitation in  $\beta$ -C alloy without any precipitate free zones (PFZs) and result in an enhancement of fatigue life. Work done by Ivasishin et al<sup>[9]</sup> show that the improvement both in strength and elongation can be achieved after two step aging (DA) compared to one step aging (SA). Boyer et al<sup>[10]</sup> reported that the strength and fatigue life of Ti15-3 alloy could be improved by duplex aging.

However, there is not much research about the influence of pre-aging on microstructure and mechanical properties of  $\beta$  titanium alloy. Aforementioned researches were mainly

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focused on the pre-aging temperature, while the effect of pre-aging time has not been yet studied. In addition, the forging, rolling or heat treatment of  $\beta$  titanium alloys are usually carried out in  $\beta$  region, which leads to the coarse grain size and reduces the strength. Therefore, the objective of this research is to investigate the influence of pre-aging on microstructure and mechanical properties of  $\beta$  titanium alloy based on the coarse grain size, and mainly focuses on the effect of the pre-aging time.

## 1 Experiment

The as-forged Ti-3.5Al-5Mo-6V-3Cr-2Sn-0.5Fe alloy was used in the present work, and the forging process has been described in our previous articles<sup>[5]</sup>. The  $\beta$  transus temperature of the alloy determined by metallographic analyses is about  $(815\pm 5)$  °C. All the heat treatment experiments in the present study were carried out in an air box furnace. Specimens cut out of the as-forged alloy were firstly solution treated at 830 °C for 30 min followed by water quenching (WQ). Two groups of aging heat treatments were carried out on solution treated specimens for comparative study. The duplex aging (DA) heat treatments were carried on the first group in which solution treated specimens were pre-aged (PA) at a low temperature of 350 °C followed by aging at a high temperature of 550 °C for 4 h. The pre-aging time ranged from 5 min to 8 h. The single aging (SA) heat treatment was carried on the secondary group in which solution treated specimens were only aged at 550 °C for 4 h.

The microstructural evolution was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and scanning transmission electron microscope (STEM) techniques. The tensile specimens with gauge length of 18 mm and thickness of 2 mm were tested on an Instron 5500R testing machine at room temperature with the speed of 1 mm/s.

## 2 Results and Discussion

### 2.1 Microstructure

In this study, the alloy was solution treated in  $\beta$  region and water quenched (WQ) to obtain coarse grain size. Fig.1 shows the optical microstructure of the alloy under  $\beta$  solution treated condition (830 °C/0.5 h/WQ). It can be seen that the  $\beta$  grains exhibit the equiaxed shape with the large grain size. The pre-aging heat treatments were carried out at 350 °C with the time ranging from 5 min to 8 h. Fig.2a shows the diffraction peaks of  $\beta$ ,  $\alpha$  and  $\omega$  phases under PA conditions, which indicates that both the transformation of  $\beta \rightarrow \omega$  and  $\beta \rightarrow \alpha$  both happened at this temperature. Fig.2b shows the diffraction peaks of  $\omega$  phase with different PA time in the same coordinate system, which exhibits the variation of  $\omega$  phase with the change of PA time. It can be seen that the  $\omega$  phase is precipitated when the PA time

comes to 5 min, and the highest peak value of  $\omega$  phase is achieved at 0.5 h. The peak value of  $\omega$  phase decreases when the PA time continues to increase, which indicates that the  $\omega$  phase precipitated at early stage transforms to the  $\alpha$  phase with the prolonging of PA time. This may be due to that more PA time provides sufficient time for transformation of  $\omega \rightarrow \alpha$ .

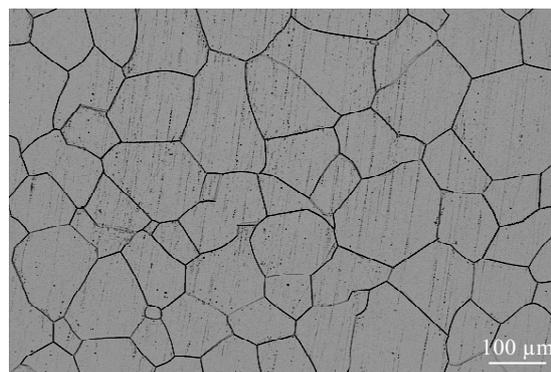


Fig.1 Metallurgical microstructure of the alloy solution treated at 830 °C

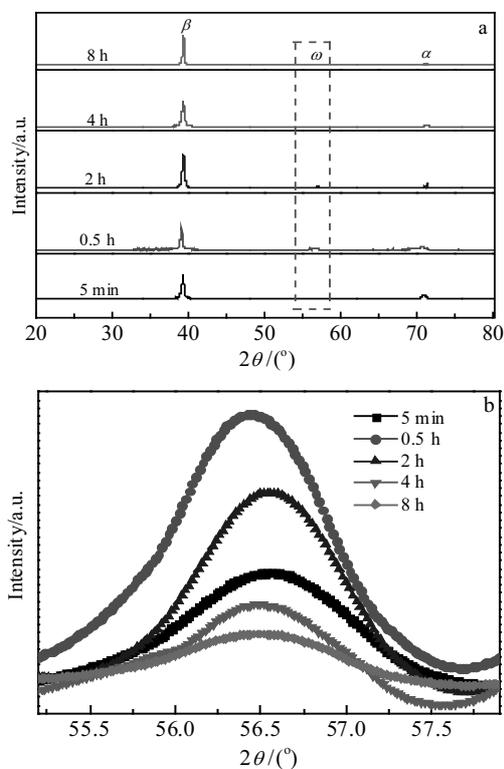


Fig.2 XRD patterns of PA conditions: (a) the diffraction peaks of  $\beta$ ,  $\alpha$  and  $\omega$  phases; (b) the diffraction peaks of  $\omega$  phase

The duplex aging was carried out at 350 °C followed by aging at 550 °C. Fig.3 shows the XRD patterns of the alloy under DA and SA conditions. Results show the pattern indicative of a microstructure composed of  $\beta$  phase and  $\alpha$  phase. In terms of phase composition, there is no difference between SA and DA. From the XRD results of DA conditions, the  $\omega$  phase precipitated at PA stage has been disappears, which confirms the transformation of  $\omega \rightarrow \alpha$  during the aging heat treatment at the higher temperature.

The microstructure of the alloy under DA conditions was analyzed in order to understand the influence of the pre-aging heat treatment on the  $\beta$  titanium alloy. Fig.4 shows the SEM microstructures under the SA and DA conditions. From Fig.4a, it can be seen that the acicular secondary  $\alpha$  ( $\alpha_s$ ) phase precipitates in matrix (as indicated by the arrow). Fig.4b shows the microstructure under DA condition of pre-aging at 350 °C for 5 min, which exhibits the shorter and narrower scale of secondary  $\alpha$  phase compared with that under SA. The scale of  $\alpha$  phase under DA conditions becomes smaller with continuous growth of pre-aging time, which can be due to more precursors provided by the more pre aging time.

Fig.4 illustrates that the scale of the secondary  $\alpha$  phase can be significantly refined by pre-aging heat treatment. Many works have proved that the finer scale and more homogeneous distribution of secondary  $\alpha$  phase can be obtained by precipitation of  $\omega$  phase which may serve as the nucleation site for  $\alpha$  phase<sup>[11-14]</sup>. However, refinement of secondary  $\alpha$  phase in this study seems to be not entirely related to  $\omega$  phase. In conjunction with the results of Fig.2 and Fig.4, the maximum peak value of  $\omega$  phase appears at 0.5 h, which means that the relative content of  $\omega$  phase should be

the greatest at this time. But the scale of secondary  $\alpha$  phase at 0.5 h is larger than that of 2 h, as shown in Fig.5. Such phenomena may be caused by the least stable matrix composition after solution treated in  $\beta$  region. Based on Fig.2, the secondary  $\alpha$  phase can precipitate directly from matrix, which proves the transformation of  $\beta \rightarrow \alpha$ . When aging a low temperature, the  $\beta$  phase will transform to  $\alpha$  phase in two ways:  $\beta \rightarrow \omega \rightarrow \alpha$  and  $\beta \rightarrow \beta' \rightarrow \alpha$ <sup>[15,16]</sup>. The first way has been proved and discussed above. For the second way, the solute-depleted formed in the least stable matrix and could act as the precursors for secondary  $\alpha$  phase precipitation at the low temperature for short aging time. The growth of  $\alpha$  phase is limited by the lack of driving force due to the low temperature. But more precursors are provided with the prolonging of aging time at the low temperature. Therefore, the fine scale and homogenous distribution of secondary  $\alpha$  phase are achieved.

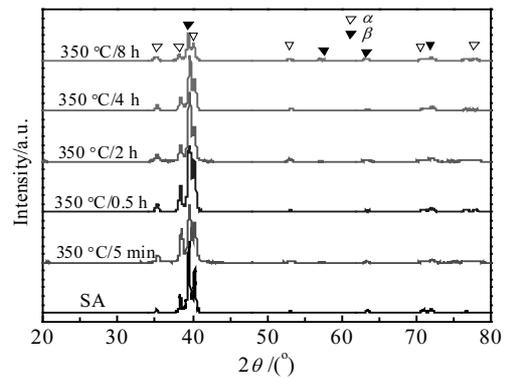


Fig.3 XRD patterns of the alloy under SA and DA conditions

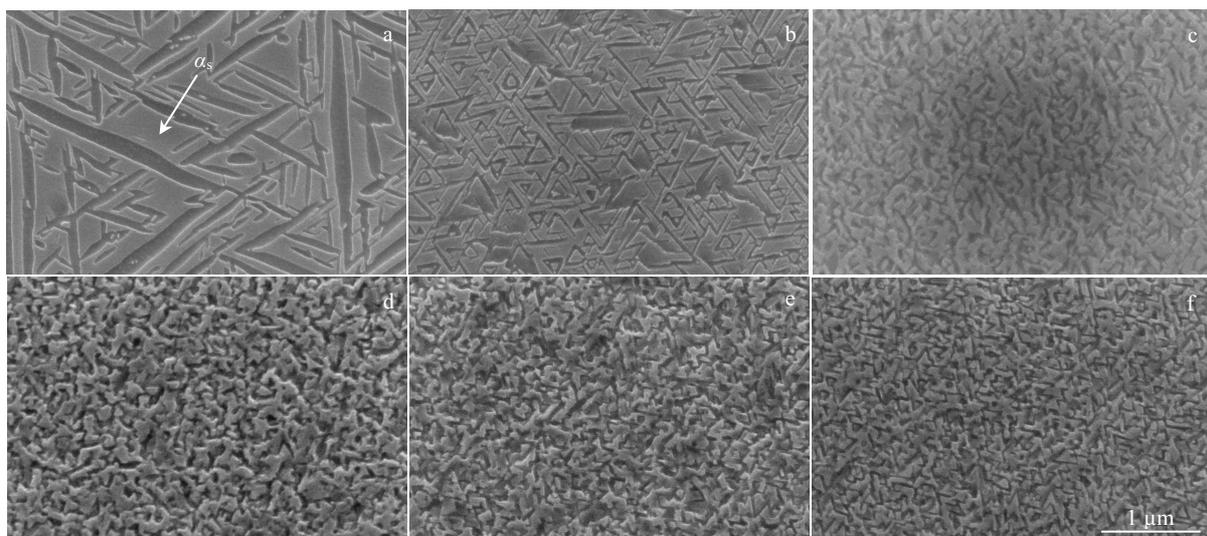


Fig.4 Comparison of microstructures between SA and DA conditions: (a) 550 °C/4 h; (b) 350 °C/5 min+550 °C/4 h; (c) 350 °C/0.5 h+550 °C/4 h; (d) 350 °C/2 h+550 °C/4 h; (e) 350 °C/4 h+550 °C/4 h; (f) 350 °C/8 h+550 °C/4 h

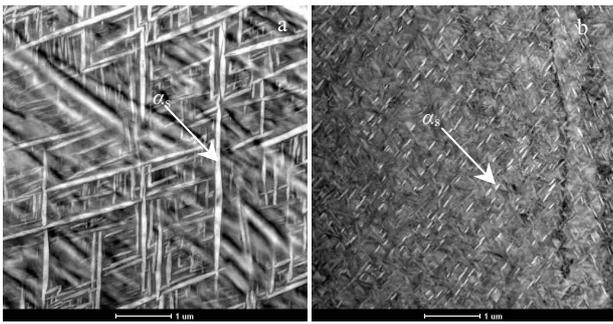


Fig.5 STEM images of DA conditions: (a) PA for 0.5 h and (b) PA for 2 h

Anyway, pre-aging heat treatment could serve as a precipitation step to provide uniform dispersed precursors for secondary  $\alpha$  phase precipitation, and then lead to the dense, fine and homogenous distribution of secondary  $\alpha$  phase. And the finer scale of secondary  $\alpha$  phase can be obtained by prolonging of the pre aging time.

### 2.2 Mechanical protective property

Fig.6 is the comparison of tensile properties between SA and DA conditions. As can be seen from Fig.6, the alloy shows a moderate strength of 1340 MPa under SA condition. The tensile strength of the alloy under DA condition is greatly improved compared with the SA condition, but the ductility decreases. The strength of DA increases gradually with the prolonging of the pre aging time, and the highest tensile strength of 1760 MPa is obtained.

The increase of the strength can be explained by the effect of the secondary  $\alpha$  phase. For  $\beta$  titanium alloys, distribution of fine secondary  $\alpha$  phase in the  $\beta$  matrix causes a microstructure condition similar to a dispersion strengthened system<sup>[17]</sup>. This is because that the small size and the high volume fraction of the secondary  $\alpha$  phase in DA conditions can lead to the occurrence of a large number of  $\alpha/\beta$  phase boundaries which act as effective dislocation barriers<sup>[18]</sup>. In the present work, the strength can be

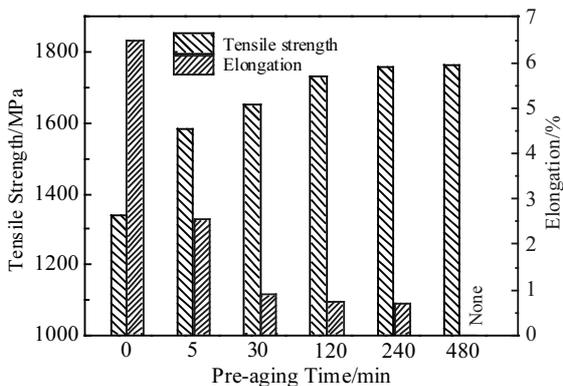


Fig.6 Comparison of tensile properties between SA and DA

expressed as the function of the volume fraction and the size of the secondary  $\alpha$  phase, and the increase of the volume fraction or decrease of the size of the secondary  $\alpha$  phase will improve the strength<sup>[19]</sup>. That is why such a high strength of 1760 MPa was obtained with the large  $\beta$  grain size in this study. Results of Fig.4 show that the scale of secondary  $\alpha$  phase can be significantly refined by PA, which explains the higher strength of DA than SA. The content and scale of the secondary  $\alpha$  phase are the more important factors affecting the ductility of  $\beta$  titanium alloy. Results of Fig.4 and Fig.6 illustrate that the refinement of the secondary  $\alpha$  phase can increase the strength obviously, but the smaller size of the secondary  $\alpha$  phase will lead to the lower ductility of  $\beta$  titanium alloy. Therefore, in this study, the decrease of ductility of DA condition can be induced as the excessive refinement of secondary  $\alpha$  phase. Similar results were obtained in our previous researches that the ductility of  $\beta$  titanium alloy decreased with the refinement of secondary  $\alpha$  phase<sup>[5,20]</sup>.

The refinement of secondary  $\alpha$  phases also leads to the improvement in micro hardness of  $\beta$  titanium alloy. Fig.7 shows the variation of the micro hardness as a function of pre-aging time during DA. The micro hardness when DA condition is significantly higher than that of SA, and increases with the increase of PA time. The increase of micro hardness is mainly due to the increase of volume fraction of secondary  $\alpha$  phase<sup>[21]</sup>. Fig.4 has shown that the longer pre aging time leads to the greater density of secondary  $\alpha$  phase, and the density reaches maximum when the pre aging time comes to 8 h, which corresponds precisely to the changing trend of micro hardness.

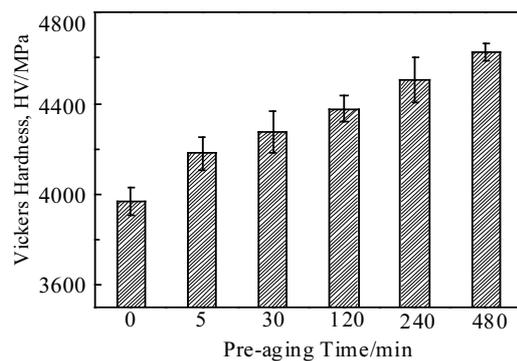


Fig.7 Comparison of micro hardness between SA and DA conditions

### 3 Conclusions

1) Pre-aging of  $\beta$  titanium alloy could serve as a precipitation step and leads to the dense, fine and homogenous precipitation of secondary  $\alpha$  phase.

2) Compared with the single aging, the duplex aging heat treatment greatly increases the strength and micro hardness of  $\beta$  titanium alloy, especially a maximum strength value of 1760 MPa obtained by pre-aging under the coarse grained condition, but the ductility is sacrificed.

3) The finer scale of secondary  $\alpha$  phase can be achieved by prolonging the pre-aging time, and the strength and micro hardness of  $\beta$  titanium alloy increase with the prolonging of the pre-aging time.

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## 预时效对粗晶 $\beta$ 钛合金组织性能的影响

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**摘要:** 研究了预时效时间对粗大晶粒  $\beta$  钛合金 (Ti-3.5Al-5Mo-6V-3Cr-2Sn-0.5Fe) 组织与力学性能的演变。结果表明: 预时效可以辅助次生  $\alpha$  相形核, 形成细小均匀的次生  $\alpha$  相, 随着预时效时间延长, 次生  $\alpha$  相变得越细小;  $\beta$  钛合金对预时效处理很敏感, 在粗晶条件下, 经过 550 °C 的单级时效, 强度仅为 1340 MPa, 而经 350 °C 预时效处理后, 再经 550 °C 的双级时效处理, 强度可达到 1760 MPa, 和单级时效相比提高了 30%; 维氏显微硬度由于次生  $\alpha$  相的细化, 随着预时效时间的延长而增大。

**关键词:**  $\beta$  钛合金; 预时效; 微观组织; 细化; 机械性能

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