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## PHASE TRANSFORMATION AND MECHANICAL PROPERTIES OF Ti-12.1Mo-1Fe ALLOY WITH NANO-SIZED PRECIPITATION

### PRZEMIANY FAZOWE I WŁAŚCIWOŚCI MECHANICZNE STOPU Ti-12.1Mo-1Fe Z NANOMETRYCZNYMI WYDZIELENIAМИ

Microstructural characterization and aging hardening behaviors of a new designed Ti-12.1Mo-1Fe alloy during solution treatment and aging were investigated in the present study. It is well known that when  $\beta$ -Ti alloys are generally under solution treatment or aging,  $\alpha$  phases and  $\omega$  phases appear or disappear dependent on heat treatment temperature and holding time. It is very necessary to understand the phase transformation phenomenon and to control the microstructure because these phases can control the drastic changes of the mechanical and physical properties of these alloys. According to the calculated [Mo]eq value and the microstructural observation, the  $\beta$ -transus temperature was about 780°. After the solution treatment, this alloy was composed of the  $\beta$ -phase and the microstructure mainly consisted of the equiaxed  $\beta$  grains with the average size of 25  $\mu$ m.  $\omega$  phases which were precipitated during aging process, played a more important role to the hardening effect than  $\alpha$  phases. The highest hardness value of Ti-12.1Mo-1Fe alloy showed in the condition of the aging temperature of 450°. The hardening due to  $\omega$ -phase precipitation can lead to a high hardness about 480 Hv but the coarse  $\alpha$ -phase result in hardness below 300 Hv.

*Keywords:* Age hardening, Omega phase, Ti-Mo-Fe alloy, Precipitation

## 1. Introduction

Titanium and titanium alloys have been used in many automobiles and vehicles applications due to their excellent mechanical performance and corrosion resistance. The hexagonal  $\alpha$ -phase titanium exhibited a feather-like morphology. The relatively low strength commercially-pure titanium (CP Ti) is currently used in various application fields [1–5]. It is well known that when  $\beta$ -type Ti (BCC) alloys are aged,  $\alpha$  phases and  $\omega$  phases appear or disappear dependent on temperature and holding time. Since these phases cause the drastic changes of the mechanical and physical properties of these alloys, it is necessary to control the microstructure of these phases.

A minimum of 10 wt% of this isomorphous  $\beta$ -stabilizing element is needed to stabilize  $\beta$  -phase at room temperature in a Ti-Mo alloy. More recently, a great deal of effort has been devoted to the study of  $\beta$  and near  $\beta$ -phase alloys, such as Ti-15Mo [6], Ti-13Nb-13Zr [7], Ti-11.5Mo-6Zr-2Fe [8], Ti-Zr-Nb-Ta-Pd and Ti-Sn-Nb-Ta-Pd [9]. Advantages of  $\beta$  /near  $\beta$  -titanium alloys over  $\alpha$ , near  $\alpha$  or  $\alpha + \beta$  alloys include their lower modulus and better formability [10]. However, titanium alloy exhibits the best high strength to density ratio among the materials above but with the highest price at the same time.

Consequently, a new  $\beta$ -titanium alloy was developed with not only good properties, but also lower costs, which

the elements were used to replace some expensive elements for the formulation cost reduction in the present study. And microstructure characterization of the new designed alloy was primarily investigated for understanding of microstructure-property relations. The present study is a continual research of this alloy system with a focus on the effect of  $\omega$ -phase on the deformation behaviors of this particular alloy. The mechanism of the embrittlement that occurred to Ti-12.1Mo-1Fe alloy can be at least partly understood.

## 2. Experimental

The experimental alloy with nominal composition of Ti-12.1Mo-1Fe was fabricated by Induction Skull Melting (ISM, CONSAC, USA). The ISM-melted ingots of the Ti-12.1Mo-1Fe alloys were homogenized in an Ar atmosphere, after which they were subjected to furnace cooling. Molybdenum equivalent ([Mo]eq) of this alloy is about 15 and  $\beta$ -transus were measured to be approximately 780° by metallographic method.

Samples of the alloy were solution-treated at 810° in a box furnace for 1 hour followed by water quenching to retain complete  $\beta$  structure at room temperature. Aging treatment of the solution-treated (ST) samples were carried out at 350°, 400°, 450°, 500°, 550° and 600° for 1 hour, followed by water

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Nominal and analyzed chemical compositions of Ti-12.1Mo-1Fe alloys (wt %)

Element	Ti	Mo	Fe	Cu	Si	C	H	O	N
Ti-12.1Mo-1Fe	Bal.	11.23	1.06	0.088	0.0098	0.042	0.0072	0.070	0.0020

quenching to room temperature to freeze second phases. To minimize the formation of  $\alpha$  cases, all the samples were coated with Acheson's Deltaglaze 151 protective coating before solution treatment. Fig. 1 showed the schematic diagram of heat treatment conditions and process.

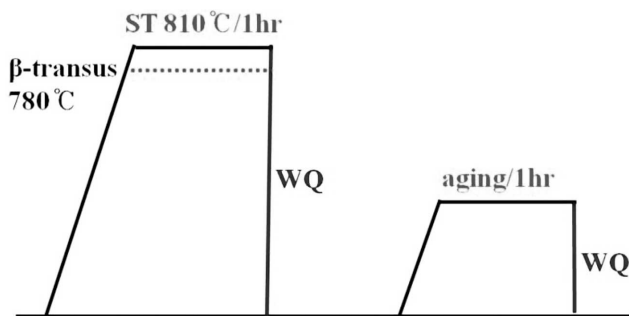


Fig. 1. Schematic diagram of heat treatment process of Ti-12.1Mo-1Fe alloy

For optical micrographs (OM) observations, the specimens were mechanically polished, used SiC sand papers of up to #4000 grit and concludes with a colloidal SiO<sub>2</sub> suspension. In order to reveal the microstructure after polishing, Kroll's etching solution (H<sub>2</sub>O (50ml) + HNO<sub>3</sub> (20ml) + HCl (15ml) + HF (10ml)) was involved. Vickers hardness measurements were done on polished samples with a load of 1 kg force applied for 10 seconds. Ten indentations for each sample were made and averaged to minimize scatter (the measured error is within 5%).

Chemical analyses of many different areas (bulk and surface) were performed, and the actual chemical compositions of the alloys were found to be close to their nominal values (Table 1).

X-ray diffraction (XRD) for phase analysis was conducted using a PANalytical diffractometer (X'pert-PRO, PANalytical Co., Almelo, Netherlands) operated at voltage of 30 kV and a current of 20 mA. An Ni-filtered Cu-K $\alpha$  radiation was used for the study. A silicon standard was used for the calibration of diffraction angles. Scanning speeds of step size 0.02 were used. The various phases were identified by matching each characteristic peak in the diffraction patterns with JCPDS files.

### 3. Results and discussion

#### 3.1. Microstructure and $\beta$ -transus

Fig. 2-(a) showed the typical optical micrographs of Ti-12.1Mo-1Fe alloys. All the samples were primarily solution

treated above the  $\beta$ -transus temperature and the microstructure of the alloy after solution treated at 810° was shown in fig. 2-(b). After the homogenization treatment for 1hr at 810°, the alloy had the microstructure which mainly consisted of equiaxed  $\beta$ -grain with the size of about 25  $\mu$ m.

$\beta$ -transus temperature is basis of selecting temperatures for forging and rolling, and also subsequent solution temperature. So the  $\beta$ -transus temperature of Ti-12.1Mo-1Fe alloy has been measured with a metallography method.

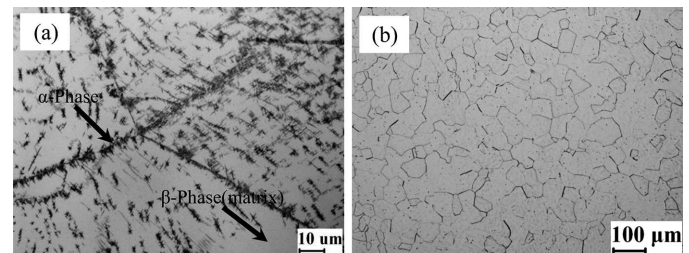


Fig. 2. Optical micrographs of (a) the as-received ingot and (b) the solution-treated at 810° for 1 hour, and followed by water quenching (WQ)

According to the calculated [Mo]eq (Eqn.(1)), the  $\beta$ -transus temperature was predicted about 770.4°. Therefore, 770° and 780° were selected to measuring the  $\beta$ -transus temperature. The relative microstructures were shown in Fig. 3. With temperature increased, both of inter-granular and intra-granular primary  $\alpha$ -phases are getting less before 780°. And at 780°, primary  $\alpha$ -phases were disappeared and transformed into equiaxed  $\beta$ -phase completely. Accordingly, the  $\beta$ -transus temperature could be regarded as about 780°.

$$[\text{Mo}]_{\text{eq}}(T_{\beta}) = 872 + 23.4[\text{Al}] + 32.1[\text{Si}] - 7.7[\text{Mo}] - 12.4[\text{V}] - 14.3[\text{Cr}] - 8.4[\text{Fe}] - 4.3[\text{Zr}] \quad (1)$$

#### 3.2. Phase transformation

Fig. 4 showed microstructures of the alloy after aging at 350°, 400°, 450°, 500°, 550° and 600° for 1 hour. It is quite clear that no  $\alpha$ -phases were found in the aging conditions from 350° and 500° (Fig. 4-(a)~(d)). With increasing aging temperature,  $\omega$ -phases disappeared and  $\alpha$ -phases were formed. Lath-shaped  $\alpha$ -phases were formed parallel to the prior  $\beta$  grain with size of several micrometers (Fig. 4(a)) and lamellar  $\alpha$ -phases were also formed in  $\beta$  grains with a relatively large size of about 25  $\mu$ m, as seen in Fig. 4-(e) and (f).

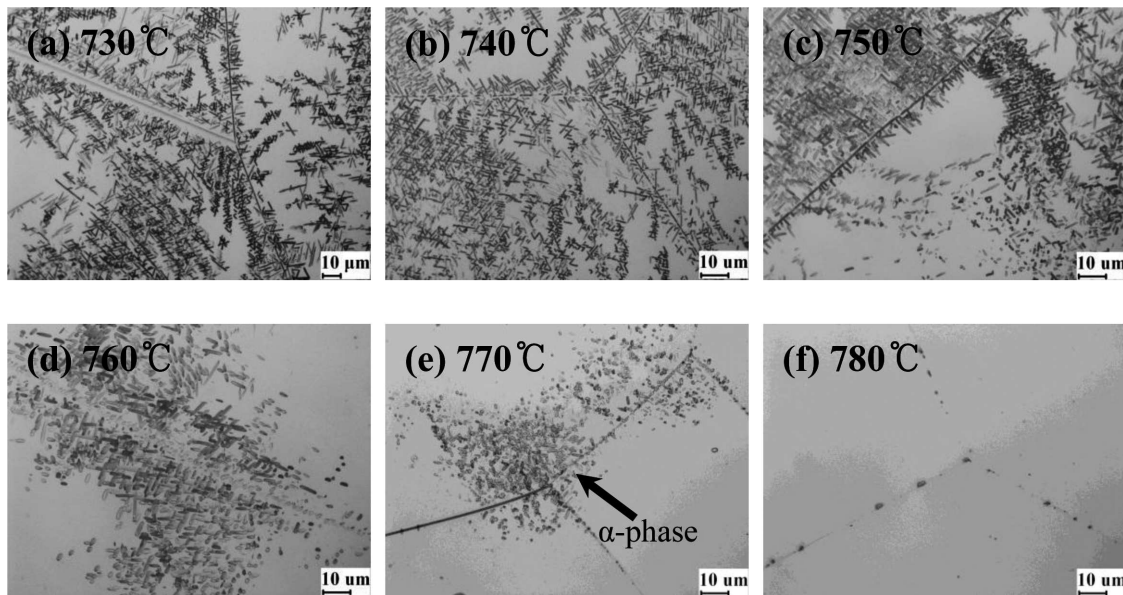


Fig. 3. Microstructural evolution of Ti-12.1Mo-1Fe alloy after various solution treatments; (a) 730°/1h/WQ, (b) 740°/1h/WQ, (c) 750°/1h/WQ, (d) 760°/1h/WQ, (e) 770°/1h/WQ, and (f) 780°/1h/WQ

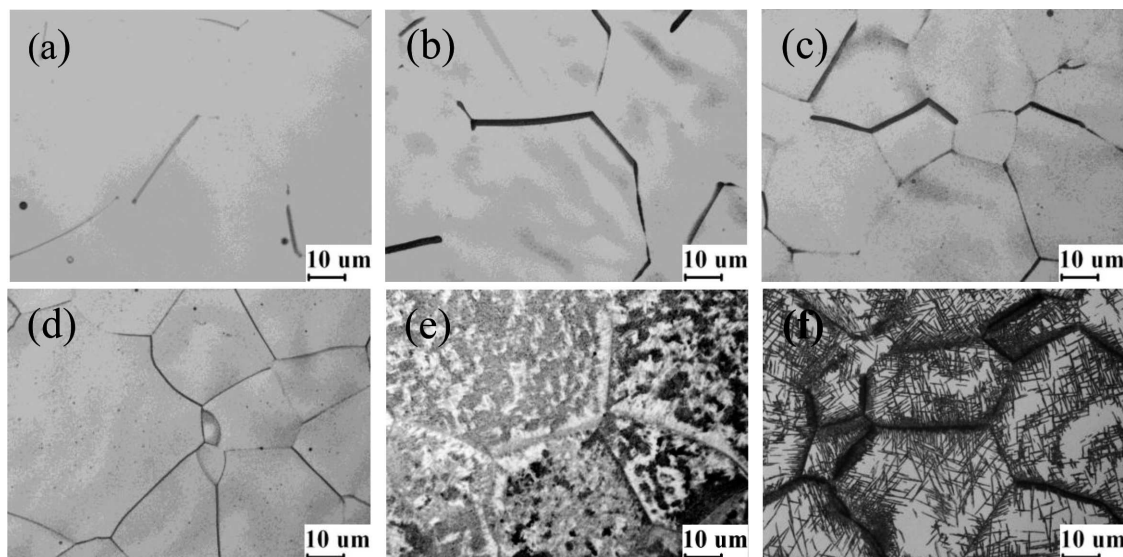


Fig. 4. Microstructural evolution of Ti-12.1Mo-1Fe alloy after various aging treatments for 1 hr, WQ; (a) 350°, (b) 400°, (c) 450°, (d) 500°, (e) 550°, and (f) 600°

Iron has been recognized as a strong  $\beta$ -stabilizing element in a recent study of a series of cast binary Ti-Fe alloys, Lin et al. [11]. As expected, Ti-Fe intermetallic compound was not observed due to the low iron contents in the alloys. Fig. 5 showed the XRD profiles of the Ti-12.1Mo-1Fe alloys. Only peaks corresponding to the  $\beta$ -phase were detected in each solution treatment specimen. No peaks corresponding to the  $\omega$ -phase were detected by XRD and the  $\omega$ -phase formation may be suppressed during water quenching in high  $\beta$  stable alloys.

Ti-12.1Mo-1Fe alloy after solution treatment at 810° has the  $\beta$ -phase microstructure.  $\omega$ -phases and  $\alpha$ -phases were not identified after solution treatment at 810°/1hr. But, after aging treatment from 350° to 500° for 1 hour, the XRD analysis results showed that the  $\omega$ -phases has been precipitated in the beta matrix. On the other hand, from 550° aging treatment,

the  $\alpha$ -phases were observed and the  $\omega$ -phases were diminished based on the XRD analysis (Fig. 5). It means that it was hard to distinguish them in the OM image (Fig. 4).

### 3.3. Vickers hardness

Fig. 6 showed the hardness variation of the alloy after aging at 350°, 400°, 450°, 500° and 600° for 1 hour and water quenching. As mentioned above,  $\omega$ -phases were formed in the matrix at 350°, 400°, 450° and 500° aging conditions. Consequently, higher hardness above 400 Hv was found from 350° to 500° aging conditions, but lower hardness around 300 Hv was shown when aged at above 550° because of  $\omega$  to  $\alpha$  phase transformation. As the content of  $\omega$ -phase decreased, the hardness decreased. This indicated clearly that  $\omega$ -phase is much harder phase than  $\alpha$  or  $\beta$ -phase. So,  $\omega$ -phases can

lead to a drastic increase in yield strength accompanied with severe ductility losses. The hardness values showed differently depending on the amount of  $\alpha$ -phase and  $\omega$ -phase.

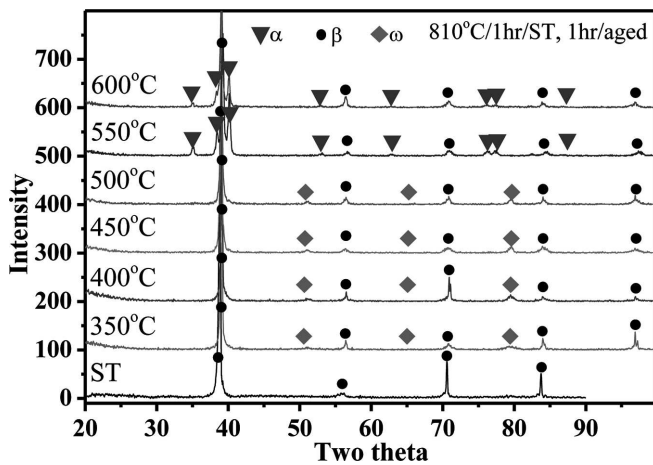


Fig. 5. X-ray diffraction analysis of phase transformation by different aging conditions

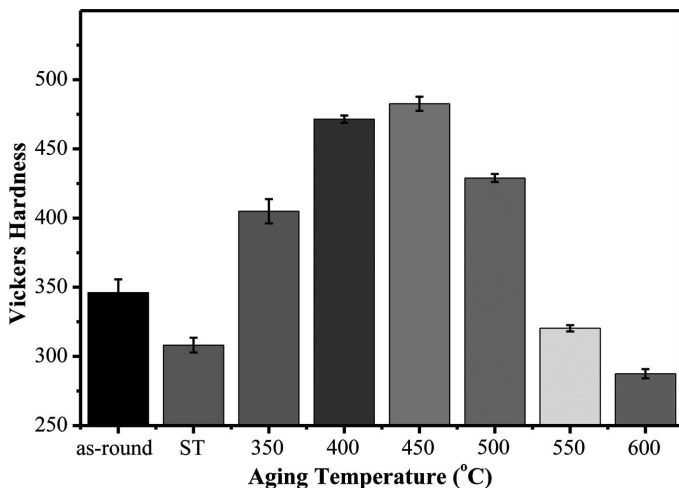


Fig. 6. Vickers hardness changes of the Ti-12.1Mo-1Fe alloy after various aging conditions

From the XRD results in Fig. 5 and the Vickers hardness in Fig. 6,  $\omega$ -phases can play an important role to improve hardness. After solution treatment, the hardness was about 308 Hv but at 350° aging treatment, the hardness increased up to 405Hv. At 450°, the Vickers hardness had maximum value, about 483Hv. This is due to the  $\omega$ -phase precipitation inside the  $\beta$  matrix. On the other hand, since 550°, the hardness values were decreased because of  $\omega$ -phase to  $\alpha$ -phase transformation.

#### 4. Conclusions

A new Ti-12.1Mo-1Fe alloy has been investigated in the present study. The microstructure characterization and hardness in solution treatment and aging condition over the range of 350-600° has been discussed in detail. According to the calculated [Mo]eq value the  $\beta$ -transus temperature was predicted about 770°. From the calculation and the experiments, the  $\beta$ -transus temperature of Ti-12.1Mo-1Fe alloy is from 770 to 780°, which is generally much lower than the pure titanium, 882°. The  $\omega$ -phase precipitation inside the  $\beta$  matrix resulted in the hardness improvement by the aging process, but when the aging was conducted at high temperature over 550°, the hardness values were decreased because of  $\omega$ -phase to  $\alpha$ -phase transformation.

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