Research on microstructure and properties of Ti-15Mo-3Al alloy with high oxygen content

Zaidong Xu, Yanjing Wang, Rongzheng Xu, Qiuye Hu, Dongyu Shi and Xin Lu
School of Materials Science and Engineering, Shenyang Aerospace University, Shenyang 110136, Liaoning, People’s Republic of China
E-mail: wangyj2006@163.com
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Abstract
In this paper, the near-equilibrium microstructure and properties of Ti-15Mo-3Al-1O alloy treated by solid solution at different temperatures were systematically studied. The microstructure of the alloy was observed and analyzed by optical microscope (OM), scanning electron microscope (SEM) and x-ray diffraction (XRD). The results indicated that the microstructure composed of equiaxial \( \beta \) grains without any precipitate was detected after solution treatment at 1,100 °C/4 h and 1,200 °C/2 h. In addition, the lattice parameters of \( \beta \) phase increased gradually with the increasing of solution temperature. Hardness and room temperature compression test were performed using Vickers hardness tester and universal testing machine. The results showed that the alloy subjected to solution treatment exhibited a high level of hardness value, with the minimum value of 367HV; the alloy subjected to solution treatment at 800 °C/2 h exhibited high compressive yield strength, with the maximum value of nearly 1,650 MPa; the alloy subjected to solution treatment at 1,100 °C/2 h and 1,200 °C/2 h exhibited excellent compressive ductility (The specimens were not crushed, the strain \( \delta > 50\% \)) with high compressive yield strength (YS > 1,100Mpa).

1. Introduction
Owing to high specific strength, excellent corrosion resistance and low elastic modulus, \( \beta \)-type titanium alloys are extensively used in aerospace, petrochemical and biomedical industries [1]. Ti–Mo based alloys with body-centered cubic perform excellent aging strengthening effect due to \( \omega \) and \( \alpha \) phase. Several deformation mechanisms such as \{332\} \{113\} mechanical twinning, dislocation slip and stress-induced martensite (SIM) formation may occur during the deformation of \( \beta \)-type titanium alloys resulting in a wide range of mechanical properties [2–6]. In addition, the mechanical properties of Ti–Mo alloys can be improved by the addition of aluminum. Consequently, Ti–Mo–Al alloys show the potential of performing high strength and toughness. For example, U.S.\( \beta \)21s alloy and China’s TB8 alloy are based on Ti–Mo–Al alloy which exhibits excellent mechanical properties and corrosion resistance [7–13]. A large number of studies of Ti–Mo based alloys with low oxygen content have been conducted by Min et al. [14–17] on the role of oxygen to the alloy showing that desirable mechanical properties is obtainable through utilizing oxygen to control the deformation mode, and oxygen affects phase stability of \( \beta \)-type titanium alloys and precipitated behavior of \( \alpha \) phase. Therefore, it is essential to elucidate the effect of oxygen on \( \beta \)-type titanium alloy.

In the study of Ti-15Mo-3Al alloy, we found that the microstructures of the alloy subjected to solution treatment and its compressive deformation behavior at room temperature changed significantly after Ti-15Mo-3Al alloy was mixed with oxygen content, which was rarely studied. Therefore, the author studied the samples from Ti-15Mo-3Al-1O ingots treated with solution at 800 ~ 1,200 °C for 2 ~ 8 h, followed by water quenching (hereinafter referred to as solution treatment) to obtain a near- equilibrium microstructure at room temperature. In addition, the microstructural evolution of Ti-15Mo-3Al-1O alloy subjected to solution treatment and its compressive deformation behavior at room temperature were analyzed. In conclusion, this
paper can provide a basis for understanding the microstructure and property of oxygen-containing β-type titanium alloy and the experimental evidence for the design of oxygen-containing titanium alloy.

2. Materials and methods

Ti-15Mo-3Al-1O alloy was prepared using pure titanium rod (99.9 mass%), granular pure aluminum (99.99 mass%) and molybdenum powder and TiO₂ powder by non-consuming vacuum arc melting in a water-cooled copper crucible using a tungsten electrode under argon atmosphere. All the ingots were turned over and melted at least three times repeatedly to ensure homogeneous composition. Oxygen content in the alloy was detected using pulse heating inert gas melting infrared absorption method. The ingots were cut into different shapes corresponding to different tests by wire cut electrical discharge machining, placing the smaller specimens for heat treatment into sealed vacuum quartz tubes, while the larger specimens were directly heat treated followed by cutting off the oxide scales. Solution treatment was carried out on the specimens at every 100 °C between 600 °C and 1200 °C.

Samples were subjected to mechanical-chemical polishing to obtain a smooth surface, and then were etched for 60 s with an etchant (10%HF + 30%HNO₃ + 60%H₂O) for observations by OLYMPUS PMG3 inverted metallographic microscope and JSM-6700 scanning electron microscope. Phase identification was made by x-ray diffraction (XRD) using an Ultima IV diffractometer with Cu-Kα radiation and operated at 40 kV and 300 mA, and the XRD spectra were recorded for 2θ values of 10–90° using a step size of 0.02° and scanning rate of 10°/min. The Vickers hardness was measured by HVS-50 Vickers hardness tester with 20 kg load and the holding time of 10 s, and the hardness value was calculated by averaging ten measurements. Compression tests was performed at room temperature in a test machine AI-7000-LA20 with a compression rate of 0.5 mm/min, and the specimens were Φ 4 mm × 6 mm cylinder. In addition, graphite powder was applied to the contact surface between specimen and pressure head in order to reduce the influence of friction.

3. Experimental results

3.1. As-cast microstructures of the Ti-15Mo-3Al-1O alloy

The microstructure of the as-cast specimen was distinctive as there were a large number of α phase (the black area) precipitated in coarse original β grains. Although the alloy was solidified in a water-cooled copper crucible with a great cooling rate, there were still a large number of α phases precipitated in grains because of the addition of 1wt.% oxygen content. According to the SEM micrograph, α phase in grain precipitated in the form of fine needles, as shown in figure 1(b). The x-ray diffraction pattern of the as-cast specimen is shown in figure 2. The results showed that there were only α and β phases in the as-cast alloy. As can be seen from the relative intensity of diffraction peaks, there was mainly α phase in the as-cast alloy. 1% oxygen was completely dissolved in α and β phase and no oxide was formed.

3.2. Microstructures of the Ti-15Mo-3Al-1O alloy after solution treatment at different temperature

Figure 3 shows the micrographs of the Ti-15Mo-3Al-1O alloy subjected to solution treatment at 900 °C/2 h. The microstructure of the specimen subjected to solution treatment at 900 °C/2 h was distributed with grain boundary α phase, intergranular α phase (which were located near grain boundary or were perpendicular to...
Figure 2. X-ray diffraction pattern of the as-cast specimen.

Figure 3. Micrographs of Ti-15Mo-3Al-1O alloy subjected to solution treatment at 900 °C/2 h. (a) OM micrographs; (b) SEM micrographs.
grain boundary) and intragranular α phase. The length of the intragranular α phase was about 2–5 μm and the width was about 0.1–0.2 μm. The intragranular α phase was stripe-like and the corresponding sides of each two triangles were parallel to each other, forming a triangular structure, which was a typical characteristic model of the intragranular α phase in Ti-5553 and Ti-7333 alloys [18, 19]. Figure 4 shows the SEM micrographs of the alloy subjected to the solution treatment at 1,100 °C/4 h and 1,200 °C/2 h. As can be seen from the figure, there was only β phase without α phase or oxide when the alloy was subject to solution treatment at 1,100 °C for 4 h and 1,200 °C for 2 h respectively. The optical micrographs of the Ti-15Mo-3Al-1O alloy under different solution conditions are shown in figure 5. On the one hand, at the same solution treatment time, with the increasing of solution temperature, the number of α phase gradually decreased, and the shape gradually changed from stripe to ellipsoid, and finally it was dissolved into the matrix. On the other hand, under the same solution temperature, α phase at grain boundaries gradually decreased with the increase of solution time.

The x-ray diffraction patterns of Ti-15Mo-3Al-1O alloy subjected to solution treatment at different temperatures for 2 h are shown in figure 6. The vertical lines represent the positions of (110), (200) and (211) standard characteristic peaks of β phase (pure titanium), respectively. In addition, it can be found that the peak...
positions of the β phase (Ti-15Mo-3Al-1O) shifted to the right based on the standard characteristic peaks of the β phase (pure titanium), which was caused by the addition of a large amount of alloying element with an atomic radius smaller than that of titanium. Furthermore, with the increasing of solution temperature, the peak positions of the β phase (Ti-15Mo-3Al-1O) shifted to the left, which was attributed to increased lattice constant. It should be noted that the reason for the change of lattice parameter was because that more oxygen was dissolved in the β phase as the solution temperature increased. Meanwhile, Min et al \cite{15}, reached similar conclusions in their experiments to investigate the influence of different oxygen contents on β phase. The lattice parameters can be calculated by the following formula:

\[ a = d(h^2 + k^2 + l^2)^{\frac{1}{2}} = \frac{\lambda(h^2 + k^2 + l^2)^{\frac{1}{2}}}{2 \sin \theta} \]  

(1)

In formula (2), \( a \) is the lattice parameter of β phase; \( d \) is the crystalline interplanar distance of \((h, k, l)\); \( \lambda \) is the wavelength of x-ray, taking 0.154178 nm; \( \theta \) is the Bragg diffraction angle; \((h, k, l)\) is the Miller indices. According to formula (1), the lattice parameters of β phase (Ti-15Mo-3Al-1O) subjected to solution treatment at 900 °C, 1000 °C, 1100 °C and 1200 °C/2 h is \( a = 0.32774, 0.32880, 0.32894 \) and 0.32902, respectively. It can be seen that the lattice parameters of β phase gradually increases with the increasing of solution temperature.

3.3. Tests and analysis of mechanical properties of Ti-15Mo-3Al-1O alloy

3.3.1. Hardness test and analysis

Figure 7 presents the Vickers hardness curve of Ti-15Mo-3Al-1O alloy subjected to solution treatment at different temperatures for 2 h. As can be seen from the figure, the maximum hardness value of the as-cast alloy could be up to 493HV. And the hardness value of the alloy subjected to solution treatment experienced three stages: Firstly, when the temperature was lower than 900 °C, the hardness values linearly decreased with the increasing of solution temperature, and it decreased by about 10HV for every increase of 10 °C. Secondly, the hardness value of the alloy decreased rapidly in the β-trans temperature range of 900 °C ∼ 1,100 °C, and it decreased by about 70HV for every increase of 10 °C. Finally, the hardness value of the alloy subjected to solution treatment at 1,200 °C/2 h was slightly higher than that at 1,100 °C/2 h. The reason for the three-stage change of hardness can be explained as follows. With the increasing of solution temperature, the content of β phase gradually increased, while that of α phase gradually decreased. At the same time, the content of oxygen in β phase gradually increased, which was attributed to the gradual increase of the lattice disorder of β phase (which could be found from the increase of the lattice parameters of β phase). And the solution strengthening effect was gradually enhanced. Therefore, the β phase hardened gradually with the increasing of solution temperature. Nevertheless, the amount of the α phase gradually decreased with the increasing of solution temperature, thus gradually weakening the strengthening effect of the α phase. When the content of the α phase was large, the hardness of the alloy was mainly controlled by the content of the α phase. Consequently, the hardness value of the alloy decreased as the content of the α phase decreased. Hence, when the solution temperature was lower than 900 °C, the amount of α phase in the alloy gradually decreased slowly with the increasing of the solution temperature, thus resulting in a gradual decline of the hardness value of the alloy. The number of second phase
decreased rapidly in the \( \beta \)-trans temperature range \((900 ^\circ C, 1100 ^\circ C)\) so that the hardness value of the alloy decreased rapidly in such a temperature range. There were few second phases in the alloy when the solution temperature was higher than 1,100 \(^\circ C\), and the hardness value was mainly controlled by \( \beta \) phase, which gradually increased with the increasing of solution temperature. Therefore, the hardness value of the alloy subjected to solution treatment at 1,100 \(^\circ C\) was higher than that at 1,200 \(^\circ C\).

3.3.2. Compression performance test and analysis

Figure 8 shows the compressive stress-strain curve of the Ti-15Mo-3Al-1O alloy at room temperature. The alloy subjected to solution treatment at 800 \(^\circ C\)/2 h exhibited the highest compressive yield strength, \( Y_S = 1650 \text{MPa} \), which was higher than the yield strength of general metastable \( \beta \)-type titanium alloys with aging treatment, which indicated that the \( \beta \)-type titanium alloy with high oxygen content showed the potential of high strength after solution treatment. When subjected to solution treatment at 1,100 \(^\circ C\)/2 h and 1,200 \(^\circ C\)/2 h, the alloy exhibited good compression ductility, specimens were not crushed and the strain exceeded 50%. Furthermore, the specimen subjected to solution treatment at 1,100 \(^\circ C\)/2 h and 1,200 \(^\circ C\)/2 h showed high compressive yield strength, \( Y_S > 1100 \text{MPa} \), indicating a solution strengthening caused by oxygen atoms. Figure 9 shows the variation of compressive yield strength with solution temperature during the three compression tests. It can be seen from the figure that the average value of compressive yield strength decreased gradually, and then increased slightly, which was consistent with the test results of hardness.
4. Analysis and discussion

4.1. Effect of 1% O on the microstructure of the alloy subjected to solution treatment at different temperature

As can be seen from figure 3, the α phases in the microstructure of Ti-15Mo-3Al-1O alloy subjected to solution treatment at 900 °C/2 h exhibited an excellent precipitation state that α precipitation was dispersed and finely distributed within the crystal grains, which was similar to the aging microstructures of general metastable β-type titanium alloy. At the same time, compared with Ti-15Mo-3Al alloy, more α phases were precipitated in the microstructure of the Ti-15Mo-3Al-1O alloy subjected to solution treatment at low temperature. And it can be explained as follows: the transformation temperature of the alloy can be calculated empirically, which is:

\[ T_{β→α+β} = 885 °C + \sum_{i=1}^{n} (C_i \times \eta_i) \]  

(2)

In formula (2), 885 °C is the β-trans temperature of pure titanium. \( C_i \) is the content of alloying elements and impurities; \( \eta_i \) is the impact value of alloying elements on β-trans temperature [20]. According to the calculation, the β-trans temperature of Ti-15Mo-3Al-1O was 1,012 °C. It also can be seen from figure 5 that the β-trans temperature of Ti-15Mo-3Al-1O alloy was between 1,000 °C and 1,100 °C, which showed that the β-trans temperature was significantly raised by 1% oxygen content. Moreover, the α phase region was enlarged due to the addition of oxygen, which was beneficial to the precipitation of α phase. Therefore, there were a large number of primary α phases in the microstructure of the alloy subjected to solution treatment at 900 °C as oxygen reduced the thermal stability of β phase. Furthermore, Ti₆O phase might generate in microstructure when the Ti-15Mo-3Al-1O was subjected to solution treatment. On the one hand, when temperature was lower than 900 °C, Ti₆O phase would precipitate in titanium alloys containing more than 2.8 wt% oxygen element [21]. On the other hand, when solution temperature was lower than 1000 °C, the solubility limit of oxygen element in β phase was extremely low according to the Ti–O phase diagram. Though the α phase in pure titanium could dissolve more oxygen element than β phase, the small amount of α phase was not enough to dissolve all oxygen element, which was why the solubility of local oxygen element was higher than the solubility limit of α phase, and thus Ti₆O phase finally emerged. In addition, it was difficult to distinguish the XRD diffraction peak of Ti₆O phase from α phase because their positions were too close, so it needs to be further verified. Based on the XRD analysis, the lattice parameters of β phase gradually increased from 0.32774 to 0.32902 nm with the increasing of solution temperature. The lattice parameters of β phase in the alloy subjected to solution treatment gradually increased as the content of oxygen in β phase gradually increased with the increasing of solution temperature. The increased lattice parameters affect the lattice strain of β phase, thus affecting the precipitation of α phase during the solution treatment below the β-trans temperature, and further affecting the morphology of the α phase [19]. Therefore, the α phase gradually changed from stripe to ellipsoid and then to a spherical shape with the increasing of solution temperature, as shown in figure 5.

![Figure 9](image-url) Compressive yield strength values of the alloy as the increase of solution treatment temperature.
4.2. Effect of 1% O on deformation behavior of Ti-15Mo-3Al alloy at room temperature

One of the characteristics of this alloy during compressive deformation at room temperature was that the alloy exhibited high yield strength when subject to solution treatment at 800 °C and 900 °C. The reason was that there were a large number of evenly and finely dispersed α phases in the microstructure of the alloy subjected to solution treatment at 800 °C and 900 °C, which played an important role of precipitated-phase strengthening. Moreover, oxygen was dissolved into α and β phase to improve solution strengthening effect. Therefore, the yield strength of the alloy subjected to solution treatment at 800 °C and 900 °C significantly increased. Another characteristic of the alloy during compressive deformation at room temperature was that the alloy performed excellent compressive ductility with high yield strength after being subjected to solution treatment at 1,100 °C and 1,200 °C. This characteristic can be explained as follows: The cold deformation mechanism of β titanium alloy was closely related to the stability of β phase, which was associated with ductility [22]. The number of chemical bonds between atoms (Bo) and d-orbital energy level (Md) of elements in d-electron alloy theory was used to measure the influence of substitution elements on stability of β phase. The values of $\overline{Bo}$ and $\overline{Md}$ can be calculated by equation (3):

$$\overline{Bo} = \sum_{i=1}^{n} X_i (Bo_i); \quad \overline{Md} = \sum_{i=1}^{n} X_i (Md_i)$$

(3)

where $X_i$ is the atomic fraction of component $i$, (Md), and (Bo) are the respective values for component $i$. The values of $\overline{Bo}$ and $\overline{Md}$ for conventional Ti alloys can be shown in a $\overline{Bo}$-$\overline{Md}$ map, in which α, α + β and β phase regions are clearly defined [23–25]. The values of $\overline{Bo}$ and $\overline{Md}$ for conventional Ti alloys are shown in a $\overline{Bo}$-$\overline{Md}$ map, in which α, α + β and β phase regions are clearly defined. The calculated $\overline{Bo}$ and $\overline{Md}$ values of Ti-15Mo-3Al alloy were 2.791 and 2.395 respectively. According to figure 10, mechanical twins and α′ martensite was generated in the plastic deformation process of Ti-15Mo-3Al alloy, which typically exhibited a high strain hardening rate and therefore a greater uniform elongation [26, 27]. In addition, the generation of twins and martensite also improved the ductility of the alloy due to TRIP and TWIP effects during deformation of β-type titanium alloy [28]. In addition, for Ti-15Mo alloy, the addition of oxygen reduced the number of twins in the deformation process, and when the oxygen content exceeded 0.7%, the deformation mechanism of Ti-15Mo alloy was inclined to single dislocation slip [14]. And it was also found that the deformation stability of metastable β phase was improved after the addition of oxygen element [15–17], thus moving the (Bo, Md) value of Ti-15Mo alloy leftward, as shown in figure 10. Similarly, the deformation stability of Ti-15Mo-3Al alloy might also be improved by oxygen content of 1%, forming the trend of deformation mechanism to single dislocation slip. Nevertheless, the elongation of metastable β titanium alloy with single dislocation slip was generally low [28]. In addition, oxygen tended to accumulate at the defect [17], preventing dislocation and further reducing the ductility of the alloy. What’s more, our previous work studied that the compression behavior of Ti-15Mo-0.8O alloy with a single β-phase at room temperature, performing that the compressive yield strength of the alloy was very high, but the specimens were broken during the compression test, which exhibited poor ductility. While Ti-15Mo-3Al-1O alloy with a single β-phase subjected to solution treatment at 1,100 °C/4 h and 1,200 °C/2 h exhibited good compressive ductility during compression, which indicated that
the \(\beta\) phase of Ti-15Mo-3Al alloy performed a lower stability. It can also be illustrated with the predicted position of Ti-15Mo-3Al-1O alloy in figure 10. Therefore, Ti-15Mo-3Al alloy could dissolve more O while maintaining a certain degree of metastability so that Ti-15Mo-3Al-1O alloy had better compression ductility.

5. Conclusion

(1) The as-cast microstructure of Ti-15Mo-3Al-1O alloy is composed of a large amount of \(\alpha\) phase and a small amount of residual \(\beta\) phase. After solution treated at 800 °C, there are a large amount of triangular \(\alpha\) phase precipitated in microstructure. With the increasing of solution temperature or time, the \(\alpha\) phases in grain decrease gradually. There are only \(\beta\) phase in microstructure after solution treated at 1,100 °C/4 h and 1,200 °C/2 h. The lattice parameter of \(\beta\) phase increases gradually with the increasing of solution temperature.

(2) With the increasing of solution temperature, the hardness value first decreases and then increases. The alloy with single \(\beta\) phase subjected to solution treatment at 1,100 °C/2 h and 1,200 °C/2 h perform excellent compressive ductility (\(\delta > 50\%\)), and the alloy subjected to solution treatment at 800 °C/2 h exhibits high compressive yield strength, \(\text{YS} = 1,650\) MPa.

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ORCID iDs

Zaidong Xu https://orcid.org/0000-0002-6665-2699
Qiuye Hu https://orcid.org/0000-0002-1123-6305

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