Microstructure evolution and mechanical properties of homogenizing treated fully lamellar Ti-46Al-0.5W-0.5Si alloy

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Abstract: The microstructure of as-cast Ti-46Al-0.5W-0.5Si alloy exhibits significant microinhomogeneity due to the non-equilibrium solidification and low atom diffusion rate. In order to reduce the adverse effect of this microsegregation on plasticity, the microstructure evolution of the Ti-46Al-0.5W-0.5Si alloy homogenized at different temperatures from 1,200 ºC to 1,320 ºC was investigated, and the optimized process of homogenizing treatment, i.e., annealing treated at 1,280 ºC and held for 8 h, was determined. Microstructures of both the as-cast and heat treated alloys were observed by means of optical microscope and scanning electron microscopes. Tensile tests at room temperature were conducted on the homogenizing treated fully lamellar Ti-46AI-0.5W-0.5Si alloy with loading axis parallel to the lamellar interface. Results show that, at higher heat treatment temperatures, the W element diffuses sufficiently, the microstructure tends to be more homogeneous, and the profile of the silicide clusters becomes smooth. Heat treating conducted in the α+γ two phase region can keep the columnar grains and the original lamellar orientation within them. The microstructure of the alloy after heat treated in α+γ two phase region exhibits the coexisting morphology of coarse lamellar and thin lamellar. The homogenization process at 1,280 ºC for 8 h can significantly reduce the microsegregation, and the elongation at room temperature can increase from 0.48% (as-cast) to 1.34%.

Key words: γ-TiAl alloy; microsegregation; homogenizing treatment; fully lamellar; plasticity

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The γ-TiAl based alloys have received much attention due to their promising applications in the aerospace and automotive industries. The addition of alloying elements such as W, Nb and Si can improve high temperature strength, creep resistance and oxidation resistance of the TiAl alloys [1-4]. However, the addition of those elements will exacerbate the microsegregation which has already existed in the binary TiAl alloys [5-8]. The as-cast microstructures of solidified TiAl alloys usually have a cored structure caused by regions of different chemical composition. For the Ti-46Al-0.5W-0.5Si alloy in this study, the fast solidification, caused by using a metal mould, leads to more insufficient diffusion during solidification, and more noticeable microsegregation formation in the grains. The former study has shown that these segregations result in the initiation and propagation of microcracks and have adverse effect on the mechanical properties [11]. To eliminate the cored structure, as-cast ingots are heated to elevated temperatures to accelerate the solid-state diffusion [10]. Recently, investigations on mechanical properties of fully lamellar γ-TiAl alloy have shown that valid mechanical properties could be obtained with the loading axis parallel to the lamellar interface [11-14]. Therefore, for as-cast fully lamellar Ti-46Al-0.5W-0.5Si alloy, it is of great importance to investigate the process of homogenizing treatment to reduce the adverse effect of segregation on plasticity, without changing the lamellar orientation. The purpose of this study is to investigate the microstructure evolution of the Ti-46Al-0.5W-0.5Si alloy during homogenizing treatment and to select an optimized process of homogenizing treatment to improve plasticity. In this research, tensile tests were also conducted to evaluate the homogenizing treated fully lamellar Ti-46Al-0.5W-0.5Si alloy.

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1 Experimental procedure

The alloy chosen for this study has the nominal composition of Ti-46Al-0.5W-0.5Si (at.%), which was prepared by using titanium sponge, high purity aluminum (99.99wt.%), silicon (99.9wt.%) and Al-W(55.92wt.%) master alloy. A cylindrical ingot with a diameter of 112 mm and a height of 220 mm was prepared using a water-cooled copper crucible vacuum induction skull melting (ISM) furnace. Specimens were cut from the longitudinal section of the ingot with an electric discharge machine. And the specimens for heat treatment were encapsulated individually in a quartz tube, which was evacuated and back-filled with high-purity Ar (99.99vol.%). Then the specimens were subjected to an annealing treatment at different temperatures from 1,200 to 1,320 °C (α+γ two phase region), and held for 8 h. Both the as-cast and annealed specimens were mechanically polished and chemically etched for observation under optical microscope (OLYMPUS-GX-71) and scanning electron microscopes (HITACHI S-4700 and FEI Quanta 200F). Compositions of the various microstructural constituents were determined by using of an energy dispersive spectrometer (EDS) with SEM.

Flat tensile specimens before and after heat treatment were cut parallel to the axial direction of the longitudinal section of the ingot using an electric discharge machine. The gauge section of the flat tensile specimens had dimensions of 6 mm in width, 2 mm in thickness and 18 mm in gauge length. All tensile specimens were polished before testing. The tensile tests were carried out at room temperature on an INSTRON 5569 testing machine with a strain rate of 5×10^{-4} s^{-1}.

2 Results

2.1 Microstructure of the as-cast Ti-46Al-0.5W-0.5Si alloy

The Fig. 1, taken using metallographic microscope, shows that the macrostructure of the as-cast Ti-46Al-0.5W-0.5Si alloy mainly consists of columnar grains growing parallelly to the center of the ingot with a colony width of 100 to 1,000 µm. Within these columnar grains, the microstructure mainly consists of fully lamellar of α2 phase and γ phase. And the α2/γ lamellar interface is perpendicular to the growth direction of columnar grains.[11]

The SEM micrograph in Fig. 2 was taken using backscattered electron (BSE) mode in order to observe the microsegregation areas, which show different contrast due to different average atomic numbers in composition. Within the α2/γ lamellar microstructure, some bright reticular structure and a large dark grey region have been observed. The bright reticular structure regions are rich in W and poor in Al, which can be verified by Energy dispersive X-ray spectroscopy (EDS) analysis of Point A list in Table 1 in Section 2.2. Dark-grey structures with irregular shape concentrated in the interdendritic regions or along the boundaries of the grains in as-cast microstructure are formed by the eutectic reaction L → γ + Ti5Si3.[11, 15]

Fig. 1: Macrostructure of as-cast fully lamellar Ti-46Al-0.5W-0.5Si alloy

Fig. 2: Segregation of as-cast fully lamellar Ti-46Al-0.5W-0.5Si alloy

2.2 Microstructure evolution during homogenizing treatment

Figure 3 shows the variation of segregation after heat treatment at 1,200 to 1,320 °C and holding for 8 h. The top right high magnification micrographs clearly show the structure of the light color regions after heat treatment. In order to compare with the heat treated structures, Fig. 3(a) provides the as-cast microsegregation. The very clear bright reticular structures indicate that there are serious segregations in the as-cast structure.

Figure 3(b) shows that after heat treated at 1,200 °C and held for 8 h, the original bright color lines become shallow. The high magnification micrograph demonstrates that the W rich phases embedded in the α2 layers are replaced by grey white area. Figure 3(c) shows that when heat treated at 1,250 °C and held for 8 h, the bright color lines become much lighter, and the bright reticular structures cannot be seen any more. In the high magnification micrograph, the bright W rich phase cannot be found, instead of the light-color lamellar structure which contains thinner lamella comparing with the surrounding lamella. Therefore, the microstructure consists of two kinds of lamella, the original lamella coarsened during heat treatment, and the thin lamella transformed after heat treatment. Figure 3(d) shows that the fraction of the light-grey areas increases after heat treated at 1,280 °C. The high magnification
micrograph shows the fraction of thinner lamella is larger than that of the thinner lamella at lower temperature. The same result can be observed in Fig. 3(e) showing the structure heat treated at 1,300 ºC. After heat treated at 1,320 ºC, as shown in Fig. 3(f), the microstructure mainly consists of thinner lamella, and only a small fraction of coarsened lamella remains.

Micrographs in Fig. 3 show that, for the same holding time, the contrast gradually becomes weaker with the increase of heat treated temperature, until it disappears. W element diffuses sufficiently at higher heat treatment temperatures, and the microstructure tends to be more homogeneous.

Semi-quantitative energy dispersive X-ray spectroscopy (EDS) analysis was conducted to evaluate the homogenization effect. Table 1 shows the measured compositions at points A to F in Fig. 3. Points A to E were selected in the light-grey area, while point F was selected in dark-grey area. The data in Table 1 indicate that the W content decreases in order from point A to point E, with the increasing treatment temperature. And the point F has a higher Al content than the other points. The higher treatment temperature provides a better homogenization effect.

### Table 1: Measured compositions at points A–F indicated in Fig. 3

<table>
<thead>
<tr>
<th>Point</th>
<th>Composition (at.%)</th>
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</thead>
<tbody>
<tr>
<td>Ti</td>
<td>Al</td>
</tr>
<tr>
<td>A</td>
<td>54.35</td>
</tr>
<tr>
<td>B</td>
<td>56.07</td>
</tr>
<tr>
<td>C</td>
<td>55.19</td>
</tr>
<tr>
<td>D</td>
<td>52.61</td>
</tr>
<tr>
<td>E</td>
<td>53.97</td>
</tr>
<tr>
<td>F</td>
<td>50.89</td>
</tr>
</tbody>
</table>

### 2.3 Silicide clusters after homogenizing treatment

Figure 4(a) shows the silicide cluster after heat treated at 1,280 ºC for 8 h. The irregular shape of the as-cast silicide clusters consists of edges and corners. After heat treatment, the profile of the silicide cluster becomes smooth, and the shape of particles in the silicide clusters becomes conglomerating. A large number of fine and dispersive particles precipitate along the interface of α2/γ lamellar around the heat treated silicide cluster.
Figure 4(b) shows the high magnification micrograph of the precipitated particles, and the analytical EDS result is given in Fig. 4(c). Combined with the References [16-17] and analysis of EDS, the particles can be identified as Ti$_5$Si$_3$ phase.

Researches have shown that the solubility of Si is limited at both room temperature and elevated temperatures in the $\gamma$-TiAl based alloy [16-17]. Silicon element is rich in the inter-dendritic regions of the as-cast alloy during solidification. The process of homogenization makes it possible for Si to diffuse sufficiently to the $\alpha_2/\gamma$ lamellar around the silicide clusters. Ti$_5$Si$_3$ particles nucleate at the interface of $\alpha_2/\gamma$ lamellar and precipitate during cooling.

2.4 Tensile tests of alloy after homogenizing treatment

It is worth noting that the $\alpha_2/\gamma$ lamellar microstructure of tensile specimens in this study is perpendicular to the direction of columnar grains[11]. According to the cutting method in this study, the loading axis is parallel to the lamellar interface, which has been expected to have better mechanical properties. On the other hand, the above investigation of microstructure evolution during homogenizing treatment has indicated that heat treatment in the $\alpha + \gamma$ two phase region can keep the as-cast columnar grains and the lamellar orientation. The segregation can be reduced to a great extent. Therefore, based on the microstructural study in Section 2.2, for the purpose of keeping the columnar grains and the original lamellar orientation within them, and for reducing segregation as possible, the process of heat treatment at 1,280 °C and holding for 8 h was selected for homogenization.

Figure 5 shows the representative tensile stress-strain curves of the as-cast Ti-46Al-0.5W-0.5Si alloy before and after homogenizing treatment. Curve 1 is for the as-cast Ti-46Al-0.5W-0.5Si alloy, and Curve 2 is for the homogenizing treated at 1,280 °C. The curves show a gradual transformation from elastic to plastic behavior. The length of plastic deformation of Curve 2 is much longer than that of Curve 1, suggesting that Curve 2 has much better ductility than Curve 1. After the as-cast alloy was homogenized at 1,280 °C, the elongation of the alloy at room temperature increases from 0.48% to 1.34%, indicating the homogenizing treatment obtains a good effect. The ultimate tensile strength of the alloy has a slight decline, from 533.7 MPa to 509.2 MPa, which is caused by the elimination of casting stress during heat treatment.

3 Discussion

The top right high magnification micrograph in Fig. 3(a) clearly shows that several W-rich phases are located in the $\alpha_2$ layers, the light grey layers in as-cast Ti-46Al-0.5W-0.5Si alloy. Figure 6 shows the W-rich phase transforms into $\alpha$ phase gradually during heat treatment in the $\alpha + \gamma$ two phase region.

During heat preservation in the $\alpha + \gamma$ two phase region, the positions of the W-rich phase, with higher Ti content, achieve the nucleation condition firstly, and W-rich phase transforms into $\alpha_2$ phase gradually. New $\alpha$ phase grows up continually with the extension of heat preservation time. The point A in Fig. 6 shows that the new $\alpha$ grain nucleus can grow into the adjacent bilateral $\gamma$ phase layers with an arc-shaped growth interface, which can reduce the interface energy and obtain the composition condition required for growth. Several $\alpha$ phases growing perpendicular to the lamellar interface will connect together, as shown at the positions of A and B in Fig. 6. The positions of C and D show that the $\alpha$ phases have connected together. In fact, with the growth of $\alpha$ phases, the adjacent $\gamma$ phases (dark layers in BSE micrograph) will dissolve. While other $\gamma$ phases become coarse gradually during heat preservation to reduce the phase interface energy. The transformed $\alpha$ phases grow up continuously, and change to thinner $\alpha_2/\gamma$ lamella when cooling to room temperature, while the untransformed $\alpha_2/\gamma$ lamella become coarser than before. Therefore, microstructure after heat treated in $\alpha + \gamma$ two phase region usually exhibits the coexisting morphology of coarse lamellar and thin lamellar.
During heat treatment in the $\alpha+\gamma$ two phase region, the macrostructure is still composed of columnar grains, in which the coexisting coarse lamellar and thin lamellar have the same lamellar orientation, perpendicular to the growth direction of columnar grains.

The above investigation on microstructure evolution during homogenizing treatment indicates that heat treatment in the $\alpha+\gamma$ two phase region can keep the as-cast columnar grain morphology and the orientation of $\alpha/\gamma$ lamellar. The diffusion of W element reduces the lacerate role of W-rich phase to the $\alpha/\gamma$ lamellar. Large silicide clusters, spheroidizing and obtuse, also reduces the lacerating role of matrix. These are contributing to the improvement of plasticity.

4 Conclusions

(1) Homogenizing treatment in the $\alpha+\gamma$ two phase region can reduce the microsegregation to a great extent. The W element diffuses sufficiently at higher heat treatment temperatures, and the microstructure tends to be more homogeneous. The microstructure of the alloy after heat treated in $\alpha+\gamma$ two phase region exhibits the coexisting morphology of coarse lamellar and thin lamellar.

(2) After heat treated at 1,280 °C for 8 h, the profile of silicide clusters becomes smooth, and the shape of particles in the silicide clusters becomes conglobating. A large number of fine and dispersive particles precipitate along the interface of $\alpha/\gamma$ lamellar around the silicide clusters.

(3) For as-cast fully lamellar Ti-46Al-0.5W-0.5Si alloy, the heat treatment at 1,280 °C for 8 h can keep the columnar grains and the original lamellar orientation, and at the same time reduce the segregation to a great extent. After this homogenizing treatment, the elongation at room temperature can increase from 0.48% to 1.34%, with loading axis parallel to the lamellar interface. The plasticity of this heat treated fully lamellar alloy has been improved.

References