Microstructure evolution of DD6 during different heat treatment conditions

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Abstract

[001] oriented DD6 alloy was prepared by directional solidification in this experiment. $\gamma'$ phase precipitation phase structure of different morphology and size was obtained by different high temperature ageing heat treatment processes. Through this experiment, the feasibility of improving high temperature performance by adjusting the heat treatment process to change the microstructure of the single crystal was explored. The as-cast DD6 is first subjected to standard heat treatment (SHT). The single crystal sample after the standard heat treatment was further kept at 900 °C–1300 °C for 10 min, 30 min and 1 h, finally air-cooled respectively. Heat treatment process of DD6 was simulated using Imatpro software. The individual phase transition temperatures of the alloy were determined by DSC experiments. And the size and volume fraction of precipitated phase of the alloy were estimated using Image-Pro Plus software. It is concluded that the microstructure of the alloy obtained after holding at 1100 °C for 30 min after standard heat treatment has the most uniform microstructure distribution.

1. Introduction

Turbine blades are also called blades, and are known as the jewels of the crown of modern industry. It is the most difficult part of the working environment in a turbine engine and the most important rotating part. Nickel base superalloys are popular used in engine turbine blades owing to their excellent high temperature, pressure resistance, corrosion resistance and oxidation resistance [1–6]. The performance requirements for alloys are also increasing because of the increasingly harsh working environment. Nickel base single crystal superalloys have gradually evolved from the first generation to the fifth generation characterized by the gradual addition of a high melting point element Re and a corrosion resistant element Ru to the alloy [7–13]. DD6 is the second-generation superalloy that has been independently studied by China and has high tensile strength, excellent creep resistance, oxidation resistance and hot corrosion resistance.

The solute elements are distributed during the solidification process, eventually forming dendritic segregation. The existence of solidification segregation leads to a low degree of alloying between the dendrites of the alloy, which becomes a channel for crack propagation and significantly reduces the mechanical properties of the alloy [14, 15]. The presence of more $\gamma'$-$\gamma'$ eutectic and coarse $\gamma'$ phase in the as-cast microstructure of single-crystal superalloys reduces the properties of the alloy [16]. Therefore, it is necessary to eliminate or reduce the eutectic and coarse $\gamma'$ phase by solution heat treatment to improve alloy properties. The superior mechanical properties of Ni-based single crystal superalloys at high temperatures are mainly due to the precipitation of larger volume fractions of the $\gamma'$ phase, which are completely coherent with the $\gamma$ phase [17]. Since the $\gamma'$ phase is the main strengthening phase of the single crystal superalloy, its quantity, size,
morphology and distribution have a decisive influence on the properties of the alloy [18–22]. The single crystal superalloys are subjected to aging heat treatment after solution heat treatment to adjust the morphology and size of the $\gamma'$ phase [23, 24]. Therefore, the as-cast alloy must be subjected to subsequent heat treatment to obtain more excellent chemical and physical properties. In this paper, DD6 was heat-treated at different temperatures after complete heat treatment to simulate the service temperature of alloy, the correspondence between the operating temperature and the organization will be established. The microstructure of the blade during service can be roughly analyzed, and the maximum temperature of the turbine blade can be roughly estimated to provide technical support for the designer.

### 2. Experimental procedure

Table 1 shows the nominal composition of the DD6 alloy. The crystal orientation difference of each of the as-cast single crystal sample rods was measured by the Laue back-reflection technique, and was preferably used for research in the extent of 5° from the orientation of the principal stress axis with the $\langle 001 \rangle$ direction. The as-cast sample is first subjected to standard heat treatment. The standard heat-treated single crystal sample was further kept at 900 °C–1300 °C for 10 min, 30 min and 1 h, respectively, and followed by air cooling (AC), as shown in table 2. There is only one sample per heat treatment process. Figure 1 demonstrates a standard heat treated system. The heat treated sample was mechanically ground and polished, and then the sample was electrolytically etched at 5 V for 5–10 s at room temperature with 150 ml $\mathrm{H}_3\mathrm{PO}_4 + 15 \text{ g CrO}_3 + 10 \text{ ml H}_2\mathrm{SO}_4 + 10 \text{ ml H}_2\mathrm{O}$ [15]. The microstructure of the alloys treated by different heat treatment procedures was then observed using an optical microscope and scanning electron microscope. Differential scanning calorimetry determines the phase transition temperature of the alloy. The volume fraction and size of $\gamma'$ were counted by Image-Pro Plus. Thermodynamic simulation of the alloy was analysed by JMaPro software.

![Figure 1. Standard heat treatment procedure of DD6.](image-url)
3. Results and analysis

3.1. Thermodynamic calculation and DSC analysis

Figure 2 shows the thermodynamic simulation results of the DD6 alloy. DD6 alloy mainly consists of $\gamma$, $\gamma'$ phase and a small amount of carbon from 900 to 1300 °C. As shown in Figure 2, solidification of the DD6 alloy begins at 1384.17 °C to form $\gamma'$ phase. The mass fraction of the $\gamma$ phase increased rapidly as the temperature dropped from 1384.17 to 1333.48 °C. The $\gamma'$ phase is the main precipitate at 1288.20 °C, and the volume fraction of the $\gamma'$ phase increases as the temperature decreases from 1288.20 to 500 °C, reaching about 70% at 600 °C. As the temperature is lowered from 1333.48 to 500 °C, the mass fraction of the $\gamma$ phase gradually decreases. When the DD6 alloy is exposed for a long time between 600 and 1150 °C, the $\mu$ phase is gradually formed in the alloy, and the mass fraction of the $\mu$ phase gradually decreases as the temperature increases.

Figure 3 shows the DSC heating curve of DD6 alloy after complete heat treatment. As shown, exothermic process is in the positive direction of the $y$-axis. The high and low fluctuations of the curve are related to the dissolution of the $\gamma'$ phase and the alloy. The $\gamma'$ phase began to dissolve at 895.6 °C, 1282.5 °C corresponds to a large amount of dissolution temperature of the $\gamma'$ phase. The complete re-dissolution temperature of the $\gamma'$ phase was 1297.1 °C. On further heating up to 1356.2 °C, the alloy begins to melt. 1393.1 °C is the peak melting temperature of the alloy. The last peak is the end melting temperature of the alloy above which the alloy will completely become liquid.
3.2. Microstructures of as-cast alloy after standard heat treatment

Figure 4 demonstrates the macroscopic and microscopic images of the sample No. 1 after standard heat treatment. The dendrite segregation of the as-cast alloy is severe. The $\gamma'$ phase is coarse and its size and distribution are not uniform. Figure 4(a) shows that the dendrite segregation of the alloy is substantially eliminated and dendrite and dendrite are homogenized after complete heat treatment. The $\gamma'$ phase in figures 4(b) and (c) also displays a good cubic morphology and a suitable size. No secondary $\gamma'$ phase precipitation in the matrix channel. The $\gamma'$ phase formed well, with size of 0.43 $\mu$m and volume fraction of about 63%. After complete heat treatment, the average matrix channel width was 67 nm.

3.3. Microstructures of DD6 alloy after being kept at different temperatures for 10 min

Figure 5 shows the microstructure of the alloys 2, 5, 8, 11, and 14 which were held at 900 °C–1300 °C for 10 min. The degree and size of the $\gamma'$ phase in the No. 2 alloy increased slightly, the matrix channel was flatter, and there was no secondary $\gamma'$ phase precipitation in the channel. Observing the No. 5 alloy, we found that degree of cubicization of $\gamma'$ phase is slightly reduced, the edge of $\gamma'$ phase is somewhat passivated, the distribution of $\gamma'$ phase is not uniform, the matrix channel is obviously widened, and there is no secondary $\gamma'$ in the channel. It can be seen from the microstructure of the specimen 8 that the $\gamma'$ phase is not uniform in size and a small portion is connected, and the degree of cubicization continues to decrease. The fine secondary $\gamma'$ phase begins to appear in the base channel. When the alloy is kept at 1200 °C, the degree of cubicization, size and distribution uniformity of $\gamma'$ phase begin to increase. The base channel became flatter and similar to the microstructure of sample 2, but...
the edges of a portion of the $\gamma'$ phase began to become serrated. The size of the secondary $\gamma'$ phase in the channel is increased. Figure 5(e) indicates that the tissue completely becomes irregular, small and new precipitation $\gamma'$ phase. No secondary $\gamma'$ phase is separated in the matrix channel.

3.4. Microstructures of DD6 alloy after being kept at different temperatures for 30 min
Figure 6 is the microstructure maintained at 900 to 1300 °C for 30 min. It can be seen from the figure that after the No. 3 alloy is treated at 900 °C, the degree cubic and size of the $\gamma'$ phase continue to increase compared with the No. 2, but the matrix channel becomes insufficiently straight, and there is still no secondary $\gamma'$ in the channel. The degree of cubicization and size of $\gamma'$ phase in sample No. 6 has no obvious change. The matrix channel becomes wider and flat, and there is no secondary $\gamma$ in the channel. A small portion of the $\gamma'$ phase appears to be connected. As can be seen from the microstructure of the sample 9, the size of the $\gamma'$ phase became finer and uniform, and the degree of cubicization increased. A small spherical secondary gamma prime phase begins to appear in the matrix channel. When the alloy is kept at 1200 °C for 30 min, the edges of most $\gamma'$ phase become jagged. The size of the $\gamma'$ phase increases compared with the low temperature, and the size of secondary $\gamma'$ phase increases significantly. The microstructure of the sample No. 15 was still a small spherical $\gamma'$ phase which was re-precipitated after dissolution.

3.5. Microstructures of DD6 alloy after being kept at different temperatures for 1 h
Figures 7(a)–(e) are the microstructures obtained after the alloy was held at 900 to 1300 °C for 1 h. Figure 7(a) shows that after processing No. 4 alloy at 900 °C, the degree of cubicization of $\gamma'$ is improved, but the size and matrix channel are not significantly changed compared with No. 3. And there is still no secondary $\gamma'$ in the channel. The $\gamma'$ phase of sample No. 7 became slightly finer and more evenly distributed, and the $\gamma'$ phase remained in a cubic form but not uniform in size. The matrix channel is significantly broadened and there is no secondary $\gamma'$ phase precipitation in the channel. It can be seen from the microstructure of the sample 10 that the size of the $\gamma'$ phase continues to increase, and the degree of cubicization is significantly reduced. The matrix channel becomes wider. Small globular secondary $\gamma'$ phases appear in matrix channels. When the alloy is held at 1200 °C, the edges of all $\gamma'$ phases become jagged, the size of the $\gamma'$ phase continues to increase, and the size of the secondary $\gamma'$ phase in the matrix channel is significantly increased and the shape is irregular. The microstructure of sample No. 16 is Butterfly-like $\gamma'$ phase which precipitates after dissolution, which is uneven in size and irregular in distribution.
4. Discussion

4.1. Microstructure evolution mechanism of as-cast alloy during standard heat treatment

The formation of γ represents the beginning of solidification of the liquid alloy. However, the solute undergoes concentration redistribution during solidification, so the alloy eventually forms a dendritic segregation pattern and the secondary γ′ phase appears in the matrix channel. When the as-cast alloy is subjected to multi-stage solution treatment, the kinetics of solute diffusion and homogenization are directly proportional to the holding time and the solution temperature. Therefore, dendrite segregation and γ/γ′ eutectic are substantially eliminated. Subsequently, at a higher primary aging temperature, the diffusion rate of the solute element increases, and therefore, the γ′ phase begins to grow as the holding time increases during the aging process. The morphology of γ′ phase is controlled by strain energy and interfacial energy. Due to lattice mismatch, the γ′ phase is subjected to compressive stress, and the γ matrix is subjected to tensile stress and has a negative mismatch [26]. From the center to the edge of the γ′ phase, the lattice mismatch stress is enhanced, so the elastic strain energy is increased, resulting in an increase in the mismatch stress gradient. The elastic strain energy in this direction is higher due to the lower Young’s modulus and higher elastic strain gradient along the ⟨100⟩ direction. Thus the γ′ phase grew along the von Mises stress reduction direction to decrease the stress gradient of γ matrix. The elastic strain gradient at the γ'/γ interface promotes the directional diffusion of the γ′ forming element to the elongated edge of the cubic γ′ lattice, preferentially entering the expanded γ′ lattice, so the lattice strain is reduced, and the γ′ phase cube is reduced. On the other hand, the higher elastic strain energy at the γ'/γ interface inhibits the growth of the γ′ phase along the direction perpendicular to the γ'/γ interface, resulting in the growth of the γ′ phase along the ⟨100⟩ direction and the side of the step through the step mechanism. Two mutually perpendicular steps grow and collide with each other, and the corner is the place where the elastic strain energy is the lowest and the interface energy is the highest. This makes the γ′ phase eventually appear cubic [27]. During the cooling process, the γ′ forming element does not have enough time to diffuse to the γ'/γ interface due to the faster cooling rate. The secondary γ′ phase re-nucleates in the γ matrix channel due to the increase in thermodynamic driving force, causing a large number of spherical γ′ phases to appear in the γ matrix channel. In the secondary aging process, a γ′ phase is roughened to reduce the total interfacial energy. The secondary γ′ disappears at the expense of coarsening and growth of the γ′ phase [28]. At 870 °C, the γ′ phase of the DD6 alloy equilibrium is 65%.

Figure 7. SEM images of microstructure of DD6 alloy at 900 °C–1300 °C for 1 h.
4.2. Microstructure evolution mechanism of DD6 alloy under different heat treatment steps

Figure 8 shows that the microstructures of the alloys have similar trends under different holding times and the same temperature gradient. When the alloys were kept at 900, 1000, 1100, 1200 °C, respectively, the temperature is lower than large dissolved temperature of $\gamma'$. Thus, two processes are performed simultaneously during the heat preservation process, namely the growth of the $\gamma'$ phase and the re-dissolution of the $\gamma'$ phase. The growth of the $\gamma'$ phase follows the Ostwald Ripening $^{[29-31]}$.

In the formula, $t$ is aging time, $k$ is the coefficient related to temperature, $r_0$ is the average radius of the precipitated phase before aging, and $r_t$ is the average radius of the precipitated phase after aging.

In addition, there is another theory for the coarsening of $\gamma'$, which is the Trans-interface diffusion controlled (TIDC) roughening model $^{[30, 37, 38]}$. The model considers that the coarsening kinetics are not related to the volume fraction of the $\gamma'$ phase, but to the diffusion of solute atoms between the $\gamma/\gamma'$ interface $^{[39]}$. Its expression is:

$$r_t^2 - r_0^2 = k_i \cdot t$$  \hspace{1cm} (2)

where $k_i$ is the coefficient related to temperature.

In the light of the Arrhenius equation $^{[40]}$:

$$D = D_0 \exp \left(\frac{-Q}{RT}\right)$$  \hspace{1cm} (3)

where $D_0$ is solute diffusion constant, $D$ is diffusion coefficient, $T$ is thermodynamic temperature, $Q$ is diffusion activation energy and $R$ is molar gas constant.

Equation (3) demonstrates that the growth rate of $\gamma'$ phase and diffusion rate of the element are proportional to the temperature. Thence, when the heat treatment time is constant, the size of $\gamma'$ is proportional to the heat treatment temperature.

The dissolution rate of the $\gamma'$ phase is smaller than the growth rate of the $\gamma'$ phase obviously. At this time, the growth of the $\gamma'$ phase is dominant. Figure 8(a) shows that only a small amount of $\gamma'$ dissolved, and the matrix...
channels are narrowed at 900 °C. The size of γ′ phase grows. When the temperature is further raised to 1100 °C, the γ′ phase continues to dissolve, thus matrix channel begins to widen. The constituent elements of γ′ phase in the matrix will reach saturation slowly. During the air cooling, the fine secondary γ′ phase separate out finally and the size will gradually increase.

When the alloy is heat treated at 1200 °C, the temperature is higher than the large dissolution temperature of the γ′ phase. Thence, the γ′ phase began to dissolve in a large amount at 1200 °C. The width of matrix continues to increase. The precipitation of γ′ is not the same with the precipitation at lower temperatures during the cooling process. There are two ways to precipitate γ′ phase, one is attached to the γ′ that has been precipitated. This growth mode is easier to advance due to the uneven nucleation of the growth mode. From previous literature [41], the γ′ phase has a high elastic strain energy in a localized region. In order to reduce the interfacial energy and elastic strain energy of γ′, the reprecipitated γ′ will settle along the (011) plane. Thus, γ′ phase will grow faster along the (011) plane, eventually forming a serrated γ′/γ′ phase interface as shown in figures 5(d), 7(d) and 8(d). Another method of precipitation is the same as at lower temperatures. Since the γ′ phase forming element is far away from the originally precipitated γ′ phase, it is too late to diffuse to the γ′/γ′ phase interface, so the fine secondary γ′ phase is precipitated in the matrix channel.

When the alloy is heat treated at 1300 °C, the temperature is between the complete dissolution temperature of the γ′ phase and the initial melting temperature of the DD6 alloy. At this temperature, γ′ phase will completely dissolve. The irregular and fine γ′ phase will precipitate during air cooling. When the mismatch between the two phases is small, the γ′ phase tends to form spherical particles to obtain a minimum interfacial area. Since the γ′ phase is completely re-dissolved and re-precipitated, the secondary γ′ phase is not formed.

4.3. Microstructure evolution mechanism of secondary γ′ phase under different heat preservation steps

From figures 5, 6 and 7, it is apparent that the size of the γ′ phase increases as the holding time increases at the same temperature. The larger the holding time, the more forming elements of γ′ phase in the matrix will diffuse to promote the growth of the γ′ phase. And it can be concluded from the LWS roughening theory that the larger temperature is, the larger the time is, so the γ′ phase gradually coarsens with time and the size increases. Figure 8 illustrates that as temperature increases and holding time increases, the γ′ phase gradually dissolves back into the matrix, and forming elements of the γ′ phase gradually become saturated, so that particles are formed in the matrix channel during cooling. The secondary γ′ phase will grow gradually with the increase of temperature, and the volume fraction also shows an upward trend. The size and volume fraction of secondary γ′ reached the maximum at 1200 °C, but both disappeared after 1300 °C. Since 1300 °C is higher than 1297.1 °C, the primary and secondary γ′ phases are completely dissolved back into the γ matrix during the heat preservation process, and then the new γ′ phase is re-precipitated in the subsequent cooling process. This caused the secondary γ′ phase to completely disappear after being kept at 1300 °C.

5. Conclusions

1. After standard heat treatment, the morphology and size of the γ′ phase are better than those in the as-cast state. When the holding time of the alloy is the same, the size of γ′ increases as the temperature increases gradually. The size of γ′ increased to a maximum at 1200 °C, and the edge of γ′ phase is serrated. The γ′ phase dissolves into the matrix completely at 1300 °C and then re-precipitates the fine irregular γ′ phase during subsequent cooling. Through cross-comparison, it was found that when the alloy was kept at 1100 °C for 30 min, the γ′ precipitate had good morphological stability.

2. After the standard heat treatment of the sample, the volume fraction of the γ′ increase and decrease first, then increase from 900 to 1300 °C. Compared to the standard heat treatment, the growth rate of γ′ is faster than dissolution rate at 900 °C, and the content of γ′ phase is increased. At 1000 °C–1200 °C, the percentage of dissolved γ′ phase gradually increases, and thus the volume fraction of γ′ phase begins to decrease. And after holding at 1100 and 1200 °C, the elemental super-saturation of the γ′ phase in the matrix causes a particulate secondary γ′ phase to precipitate in the matrix channel during the cooling process. At 1300 °C, γ′ is completely dissolved, and new irregular and small γ′ phase are re-precipitated during cooling. The matrix channel width is reduced and volume fraction of γ′ is increased.

3. Although the coarsening kinetics appear to fit well with the LSW model, the experimental data also demonstrates the applicability of the TIDC model in this context and therefore does not rule out the possibility of rate limiting diffusion of solute atoms through this interface.
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