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Influence of Heating and Cooling Rate on Dissolution and Precipitation Behavior of Nickel-based Single Crystal Superalloy

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Abstract: The effects of heating rate before solution treatment and cooling rate after solution treatment on the morphological distribution and evolution of precipitation phase of nickel-based single crystal superalloy were studied. The dissolution, precipitation and growth of precipitation phase and matrix phase during heat treatment were analyzed by means of high power scanning electron microscopy. The results show that the morphology of precipitated phase has nothing to do with the distribution of precipitated phase and the heating rate in the heating process, but the cooling rate in the cooling process affects the shape, size and distribution of precipitated phase. The faster the cooling rate, the smaller the precipitated phase is, the more irregular the shape is, the smaller the equivalent edge length is, and the smaller the channel width of matrix phase is.

Keywords: Nickel-based single crystal alloy; Heat treatment; Heating and cooling rate; Precipitation and dissolution of γ' phase

1. Introduction

Nickel-based single crystal has gradually become the preferred material for aeroengine turbine blades because of its excellent mechanical properties at high temperature, especially creep properties, oxidation resistance, corrosion resistance, high toughness and good processing plasticity[1]. Good high temperature mechanical properties were obtained by precipitation strengthening. The molecular structure of $\text{Ni}_3(\text{Al}, \text{Ti})$ as precipitation strengthening phase was L12 face-centered cubic structure. The macroscopic structure was cubic, and the strengthening phase was surrounded by continuous solid solvent matrix phase, which presented a chessboard-like compact and regular arrangement [2]. It has been found that the basic characteristics of the strengthened phase, such as shape, size, distribution and volume fraction, have an important effect on the mechanical properties of superalloys [3–6].

Many scholars have done a series of work on the dissolution and precipitation behavior of precipitation phases of Ni-based alloys at high temperature. The microstructural evolution of precipitation phases has been discussed. The morphological changes and distribution of the precipitation of γ phases during continuous cooling after solid solution treatment have been studied. Good results have been obtained in the recovery heat treatment system of the alloys. Wang [7] mainly studied the effect of solution temperature and holding time on the γ phase dissolution behavior of IN100 and DS Rene 125 alloys during solution treatment, and explored the dynamic factors of precipitation phase dissolution behavior during solution treatment. The results show that the environmental temperature has a greater influence on the dissolution of precipitation phase precipitation than the holding time. At the same time, the morphology of the precipitation phase is strongly influenced by the elastic strain energy in the structure, which is related to the lattice mismatch between the precipitation phase and the γ phase. Grosdidier [8] studied the precipitation and dissolution of γ phase in nickel-based alloys AM1 and CMSX-2 during heat treatment, discussed

the microstructural evolution of precipitation phase just after precipitation, and proposed that precipitation phase would undergo deformation from spherical to cubic to dendritic from nucleation to growth. Gao [9] studied the effect of cooling rate on the microstructural evolution and mechanical properties of Ni-based superalloy Rene 80 during the cooling process after solution treatment. It was found that the higher the cooling rate, the smaller the precipitated precipitation phases during solidification, and the faster the coarsening of the precipitation phases in the subsequent aging process. After a lot of mechanical properties tests on alloys with different cooling rates, it is concluded that air cooling is the most suitable cooling rate for Rene 80 alloy in the cooling stage of heat treatment. Jackson et al. [10] Studied directionally solidified superalloy (DS) Mar-M200+HF. It was found that higher solution temperature could dissolve more coarsened γ' phases and γ - γ' -eutectic structures, and more high-quality gamma-precipitation phases were precipitated in subsequent cooling and aging. Increasing volume fraction of high-quality precipitation phases could greatly improve creep properties of the alloy, When the volume fraction of strengthening phase increases from 30% to 45%, the creep fracture life of the material increases three times as long as the original one. Li et al. [11] found in the study of FGH4096 blade materials that the cooling rate after solution treatment determined the morphology and distribution of precipitated precipitation phases. Faster cooling rate could make the precipitated precipitation phases have better morphology and narrower channel of matrix phase, which improved the precipitation effect of Orowan[12–14] and effectively hindered dislocation movement in matrix phase, thus the material could be improved. The creep properties were improved at 704°C /690 MPa. At present, DD6 is widely used in hot-end components of aeroengine. Besides plastic deformation caused by load, there is no grain boundary, and the evolution law of microstructure of precipitation phase and matrix phase during creep and after heat treatment process is different from that of general superalloys. At high temperature, the microstructure morphology of DD6 will change to a certain extent, which will affect the performance of blades [15–18]. The study of dissolution and precipitation behavior of γ' phase at high temperature has important engineering practical significance.

Starting from different stages of heat treatment system, we focus on many factors, such as heating rate in solution treatment stage, cooling rate in cooling stage after solution treatment, aging temperature and time in aging stage. By designing different experimental conditions, the effects of various factors (heating rate and cooling rate) on the dissolution and precipitation behavior of γ' phase and the number, size, morphology and distribution of final precipitates in the heat treatment system were studied.

2. Materials and Methods

2.1. Material

Nickel-based single crystal was used as experimental material and standard two-phase alloys, which were γ phase and γ' phase respectively. The γ' phase is cubic and arranges in an orderly manner with an equivalent side length of about 530 nm. Figure 1 is the microstructure of the dendrite cadre site of the second generation nickel-based single crystal alloy DD6 after standard heat treatment(1290 °C × 1h+1300°C ×2h+1315°C × 4h/AC+1120°C × 4h/AC+870°C × 32h/AC, AC means air-cooling). The chemical composition of the material is listed in Table 1.

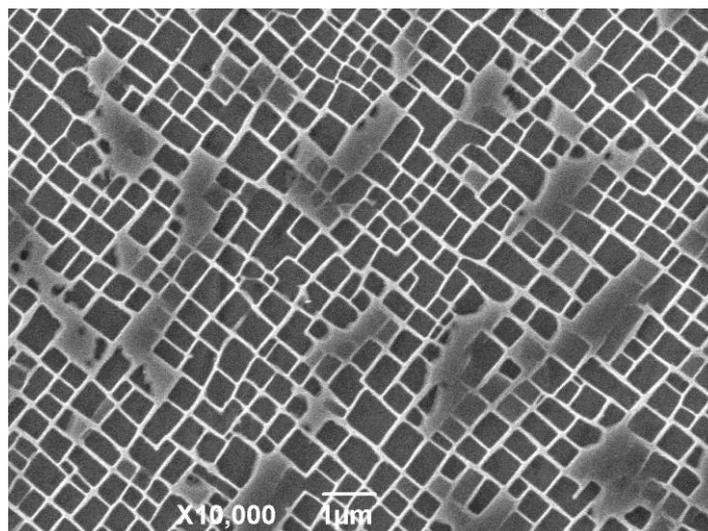


Figure 1. Microstructure of nickel-based single crystal.

Table 1. Chemical constituents of nickel-based single crystal (mass fraction%).

	Al	Cr	Co	Ni	Mo	Ta	W	Re
γ' phase	5.18	4.52	9.21	61.33	0.79	7.16	7.48	2.09
γ phase	4.64	5.21	10.57	60.67	1.73	5.31	8.74	2.13

2.2. Sample test

In order to facilitate the test, cubic specimens were used to prepare the samples by metallographic sample preparation procedure, because this paper focuses on the changes of the microstructures of the specimens during heat treatment without external loads and does not involve the study of macro-mechanical properties. The DD6 material is cut into 6 mm × 5 mm × 3 mm metal blocks, and the 6 mm × 5 mm surface is in the direction of [001] along the 6 mm length direction. After the experiment, one of the 6 mm × 5 mm planes was polished on sandpaper with grain size of 800 mesh, 1200 mesh, 1500 mesh and 2000 mesh, and then polished on the polishing cloth. The planar finish should meet the requirement of no scratches under 50 times magnifying glass. Chemical etching solution was prepared with HNO₃, HF solution and glycerol in a certain proportion. The etching droplets are washed with clean water after 3–5 s on the polished surface, and the operation is repeated 3–4 times until the surface is slightly blue, and the etching operation can be completed. JSM-6390A scanning electron microscopy was used to observe the microstructures and distribution of the strengthened and matrix phases on the polished surface.

3. Experimental Process

The heat treatment process adopted in this experiment can be roughly divided into three distinct stages: solid solution treatment, cooling after solid solution treatment and aging treatment. The effects of heating rate, cooling rate after solution treatment, aging temperature and time on the dissolution and precipitation behavior of precipitation phases in DD6 single crystal alloy and the morphological distribution of precipitation phases are studied. All experiments were repeated at least three times to ensure the reliability of the results.

The whole heat treatment test is carried out in a special high temperature furnace. The device can set up several program sections to control the temperature rising and falling at a specified rate, and can accurately keep the temperature constant.

3.1. Solution Treatment Stage

The solid solution treatment stage is divided into heating process and heat preservation process. The effects of temperature and holding time on the dissolution behavior of materials have been studied in a large number of literatures. In order to explore the influence of heating rate on the final precipitation morphology distribution during the heating process, the following experimental process was designed to explore: the solution temperature of 20°C higher than the standard solution temperature of heat treatment (i.e., 1335°C) was taken as the solution temperature of the new heat treatment system. The samples were heated to 1335°C at different heating rates, which were: ① rapid heating, ② 0.18°C/s, ③ 0.09°C/s, respectively. The test process is shown in Figure 2.

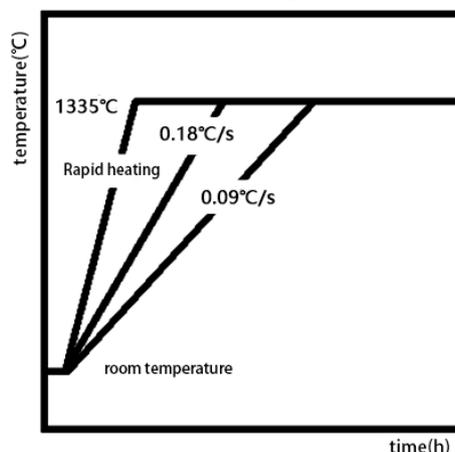


Figure 2. Heating process.

Because the temperature in the early stage of the heating process is very low and far below the dissolution temperature of the precipitation phase, many precipitation phases remain undissolved at the end of the heating process, and because the different heating rates lead to the different amount of dissolution of the precipitation phase at the end of the heating process, the uniform holding of the sample at 1335 °C for 4 h is chosen to ensure the complete dissolution of the phase, and then the air cooling is carried out.

3.2. The Cooling Stage

In the solid solution treatment stage, the precipitation phase gradually dissolves in the matrix phase to form a single-phase solid solution after holding for a long time at high temperature. As the solubility of the precipitation phase in the matrix phase decreases with the decrease of temperature, when the solid solution treatment is finished and the cooling stage is entered, with the decrease of temperature, the single-phase solid solution gradually begins to precipitate the precipitation phase because of its supersaturation. According to the study of Grosdidier [12], the precipitation behavior of precipitation phase is affected by the different cooling rate. In order to study the effect of cooling rate on the shape, size and distribution of precipitated phases, seven different cooling rates were adopted under the same heating rate, solution temperature and treatment time: (a) 0.15°C/s, (b) 0.25°C/s, (c) 0.4°C/s, (d) 0.5°C/s, (e) 0.6°C/s, (f) air-cooled and (g) water-cooled to room temperature at a constant rate. The experimental flow chart of the effect of different cooling rates on precipitation is shown in Figure3.

3.3. Aging Stage

The aging process of single crystal alloys is the growth and re-precipitation of precipitation phases, which essentially depends on the diffusion of solute atoms represented by Al atoms. Therefore, the aging temperature and time control the formation and morphology distribution of precipitation phases. It has been proved that the volume fraction, cubic degree and regularity of phase arrangement of high quality precipitation phases have great influence on creep and fatigue properties of single crystal materials, so aging treatment plays an important role in improving the

mechanical properties of single crystal materials. In order to study the influence of aging temperature and aging time on the shape, size and distribution of precipitates in different aging treatment systems, five different aging treatment systems as listed in Table 2 are adopted. The specific experimental process is shown in Figure 4.

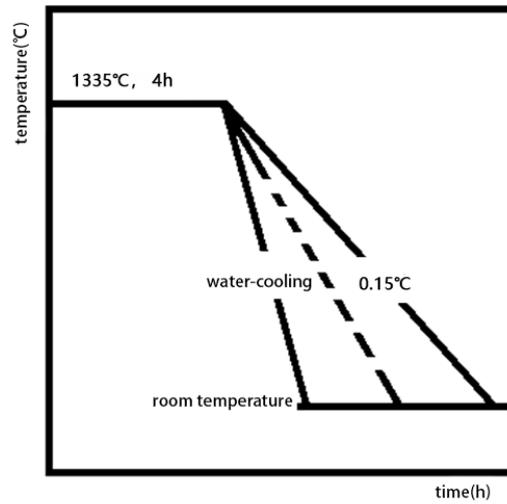


Figure 3. Cooling process.

Table 2. Treatment systems.

Number	Solution Treatment	Aging Treatment
①	1335°C/4h	1120°C/1h
②	1335°C/4 h	1120°C/2h
③	1335°C/4 h	1120°C/3h
④	1335°C/4 h	1120°C/4h
⑤	1335°C/4 h	1120°C/4h+870° C/32h

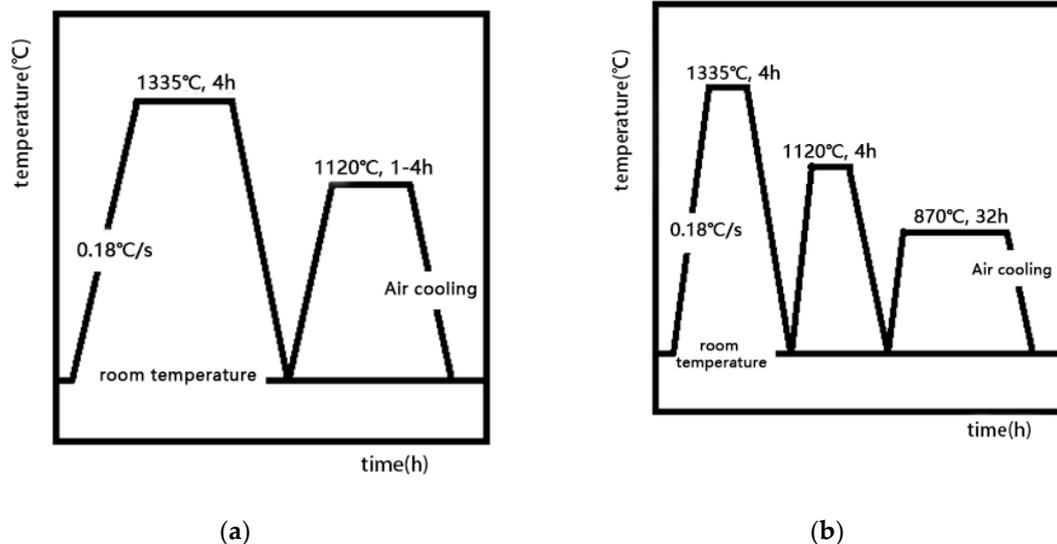


Figure 4. Aging process diagrams (a) The ageing treatment system: ①–④; (b) The ageing treatment system: ⑤.

4. Experimental Results and Analysis

4.1. Heating Rate

Three different heating rates were used to polish and corrode the specimens. The samples are heated from room temperature to target temperature at different heating rates, and after full solution treatment, the temperature is reduced to room temperature at the same cooling rate. The microstructure of the precipitates was observed under high power electron microscopy. The results are as follows: Figure 5. The average equivalent edge length of precipitated phase after cooling are 80 nm (Figure 5a) at rapid heating, 82 nm (Figure 5b) at the heating rate of 0.18°C/s and 79 nm (Figure 5c) at the heating rate of 0.09°C/s. The average equivalent side length of precipitated phase at different rates are shown in Figure 5d.

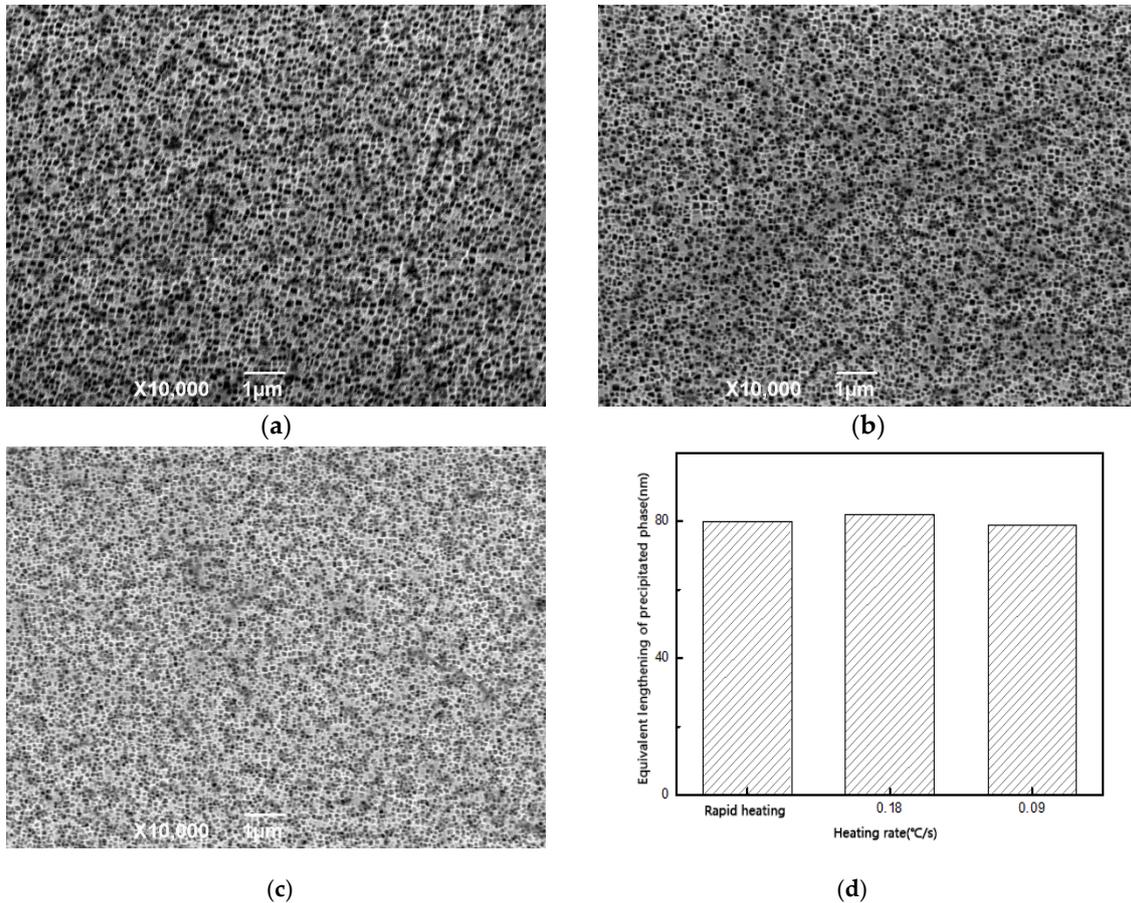


Figure 5. Microstructure of γ/γ' phase after cooling by solution treatment at different temperature rising rates (a) Rapid heating, (b) 0.18°C/s, (c) 0.09°C/s, (d) Average equivalent side length of precipitated phase.

To determine the effects of three heating rate conditions on the distribution of elements in the channel of precipitated phase and matrix phase obtained after cooling, the surface of the sample was analyzed by EDS. The results are listed in Tables 3 and 4.

Table 3. Chemical constituents (mass fraction%) of precipitation phases at three heating rates.

Heating Rate	Al	Cr	Co	Ni	Mo	Ta	W	Re
Rapid heating	4.55	4.57	9.44	60.60	2.12	5.98	9.15	2.59
0.18°C/s	4.61	4.59	9.25	60.93	2.57	5.83	8.41	2.81
0.09°C/s	4.69	4.60	9.30	60.14	2.38	6.34	9.19	2.36

Table 4. Chemical composition (mass fraction%) of matrix phase at three heating rates.

Heating rate	Al	Cr	Co	Ni	Mo	Ta	W	Re
Rapid heating	5.30	4.23	8.93	59.59	3.33	6.03	7.87	2.75
0.18°C/s	5.71	4.71	9.85	58.49	2.25	6.24	8.20	2.56
0.09°C/s	5.18	4.66	9.49	59.21	2.67	6.32	8.36	2.13

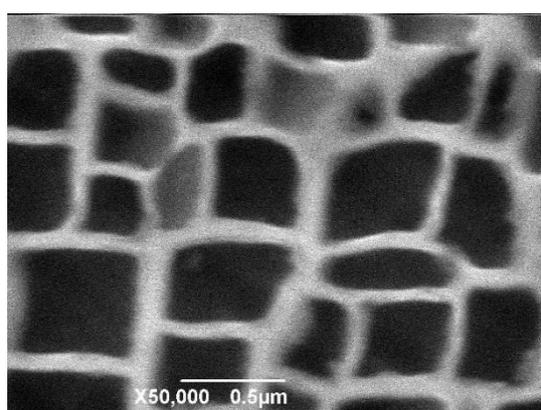
By comparing the phase diagrams obtained under the three heating rates in Figure 5, it can be found that the morphological distribution of precipitated phases is very similar, the cross sections are mostly round, and a few of them show the trend of quadrangle preliminarily. The equivalent side length of the precipitated phase is very close, approximating 80 nm, and the width of the matrix channel between the phases is less than 10 nm.

From the comparison of the results of energy spectrum analysis, it can be seen that the samples were heated to solution temperature at three different heating rates and treated with solid solution for a sufficient time (4h). The samples were lowered to room temperature at the same cooling rate (air cooling). The contents of the main elements in the precipitation phase were basically the same. The contents of Al, Cr, Co, Mo, Ta, W, Re and other elements in the precipitation phase were basically the same under three conditions. Closely approximate, it can be seen that the heating rate conditions have little effect on the migration of elements.

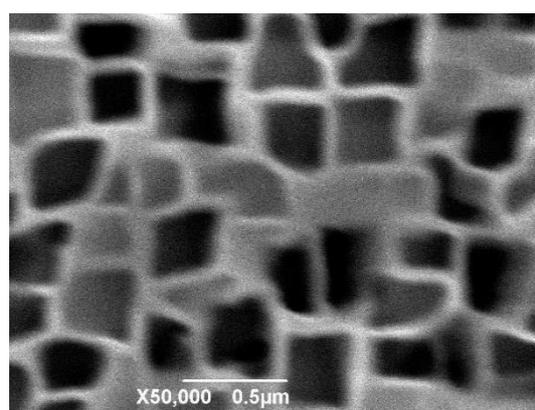
It can be concluded that the morphology of the enhanced phase after precipitation has nothing to do with the distribution and the heating rate during the heating process.

4.2. Cooling Rate

In order to study the effect of cooling rate on precipitation behavior of precipitation phase in the cooling stage after solid solution treatment during heat treatment, DD6 samples were heated to 1335°C and kept for 4 h, so that all phases in the original sample were completely dissolved into the matrix phase to form a uniform solid solution. After cooling operation, seven different cooling rates were taken, namely (a)0.15°C/s, (b)0.25°C/s, (c)0.4°C/s,(d)0.5°C/s, (e) 0.6°C/s and (f) air-cooling and (g) water-cooling, the samples were cooled to room temperature. Figure 7 shows the microstructures of precipitated precipitation phases in the sample matrix at various cooling rates. The average equivalent edge lengths of precipitates in Figure 7 are (a) 375 nm, (b) 273 nm, (c) 187 nm, (d) 169 nm, (e) 132 nm, (f) 86 nm, (g) 12 nm, respectively.



(a)



(b)

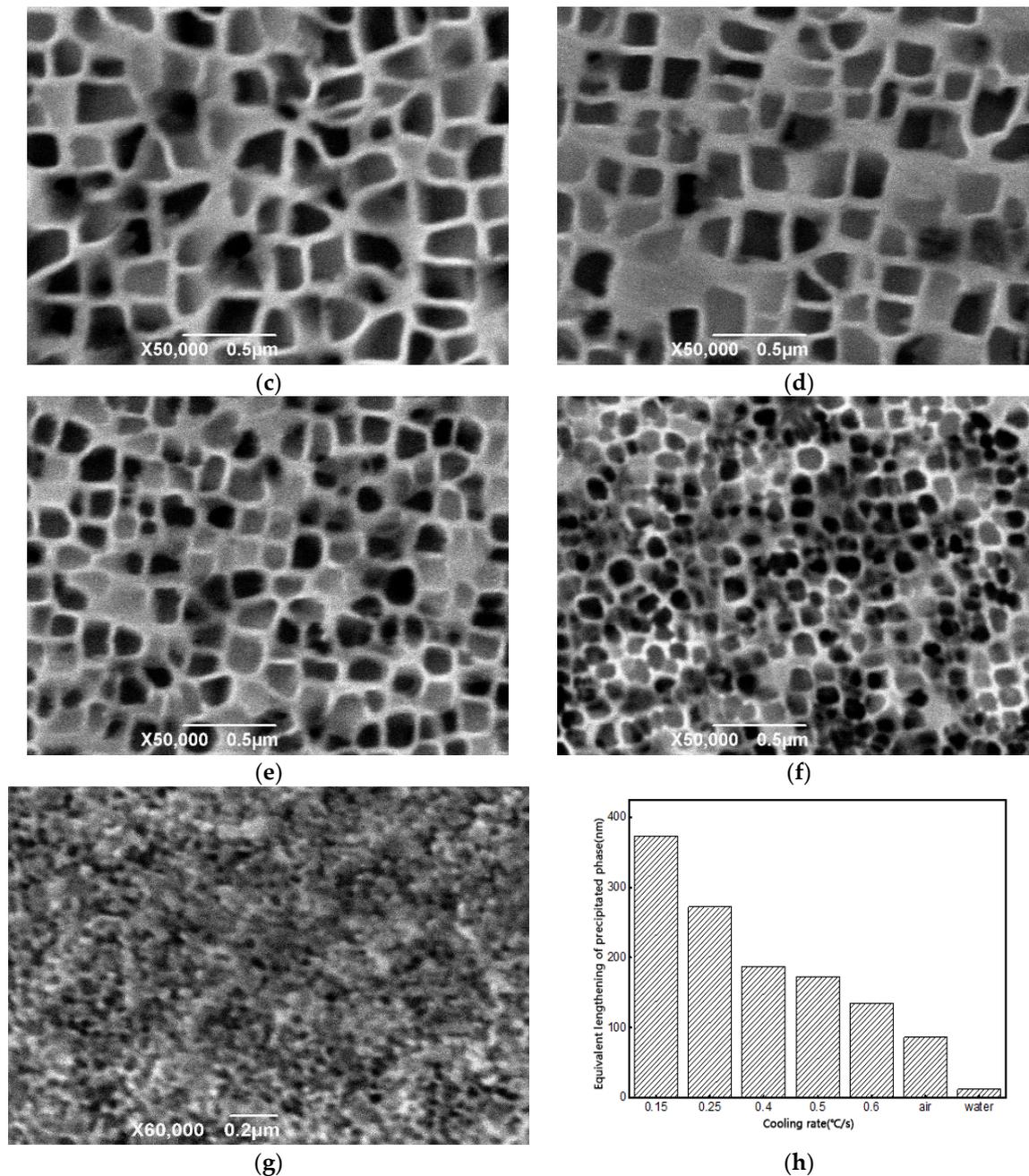


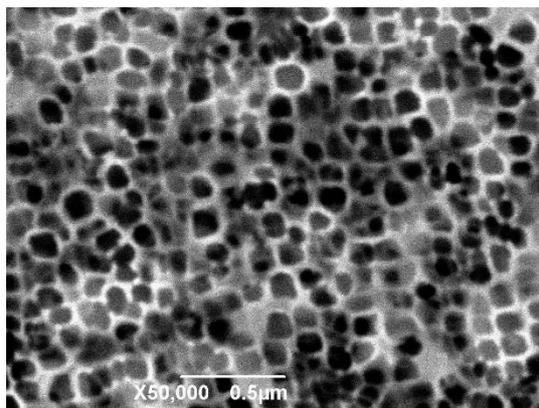
Figure 6. Microstructure of materials obtained by different cooling rates (a) 0.15 °C/s; (b) 0.25 °C/s; (c) 0.4 °C/s; (d) 0.5 °C/s; (e) 0.6 °C/s; (f) Air-cooling; (g) Water-cooling; (h) Average equivalent side length of precipitated phase at different cooling rates.

It can be seen from Figure 6 that the shape, size and distribution of precipitates depend on the cooling rate during cooling. When the cooling rate is more than 300 °C/s, i.e., water-cooled condition, the precipitate phase is very small, its shape is irregular point-like and circular, its equivalent edge length is less than 20 nm, and the width of the matrix phase channel is less than 1 nm; the matrix phase channel is arranged closely and disorderly, and the channel is very narrow, with more fine secondary precipitates. It is difficult to clearly display the micro-structure under the existing electron microscopy conditions. When the cooling rate is greater than 50 °C/s, i.e., air cooling, most of the precipitated phases are circular in shape, and some of them have begun to grow in a quadrangular direction, showing a trend towards a square transition. The equivalent side length is about 86 nm, the width of the matrix phase channel is approximately 8 nm, and the fine secondary precipitated phases in the channel are greatly reduced. Compared with the water cooling condition, the number of precipitated phases is reduced and the volume is increased. The air cooling rate is still very fast,

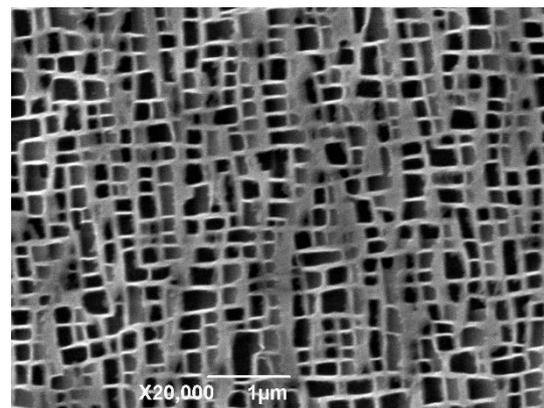
the precipitation time is very short, and the time available for phase growth and merger is very small. When the cooling rate is between 0.4 and 0.6 °C/s, the precipitation phase presents irregular square shape, and the circular precipitation phase decreases or even disappears. Compared with water cooling and air cooling, the cooling rate decreases greatly. The precipitation phase has a certain time to grow up and merge with each other. The circular phase gradually grows into a rectangular outline, the equivalent edge reaches 180 nm, and the distribution gradually becomes regular, and the matrix is interconnected. The channel becomes obvious, the width of the channel is about 15 nm, and the secondary precipitation phase in the channel decreases greatly. When the cooling rate is less than 0.25 °C/s, especially less than 0.15 °C/s, it can be seen from the figure that the precipitated phase presents regular block shape, larger volume, equivalent side length is about 300 nm, more orderly arrangement, and the width of the channel of the matrix phase is about 30 nm. The shape of the precipitated phase is slightly deformed, and there is a tendency to form a butterfly-like structure with four precipitation phases.

4.3. Aging Treatment

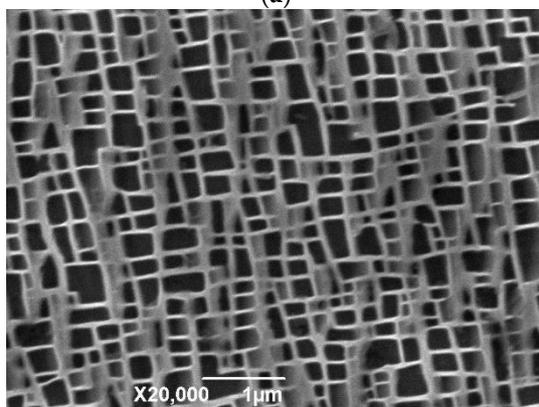
After the sample was heated to 1335 °C and held for 4 h, the air-cooling method was used to cool the sample to room temperature, and then the following aging treatments were carried out respectively: holding (a) 0h, (b) 1h, (c) 2h, (d) 3h, (e) 4h at 1120 °C, and (f) holding at 870 °C for 32 h. After the experiment, the observation plane of the sample was polished. After corrosion with special corrosion solution, the microstructure of the sample was observed under electron microscope. The results show that the equivalent side length of the precipitated phase under each aging regime, and the specific data are listed in Table 5.



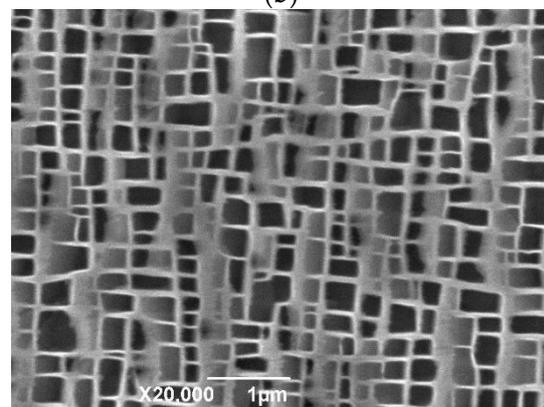
(a)



(b)



(c)



(d)

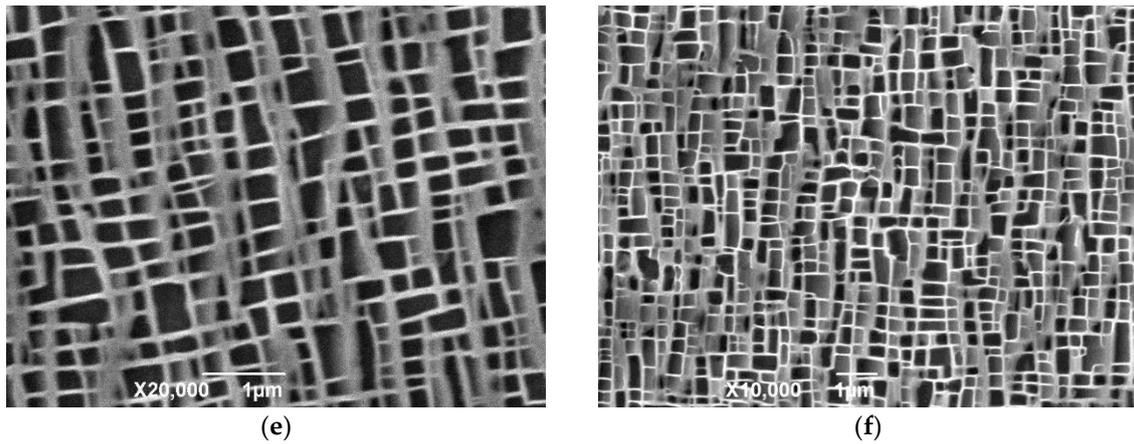


Figure 7. Microstructure of the samples after different aging treatments (a) Non-aging, (b) Aging system ①, (c) Aging system ②, (d) Aging system ③, (e) Aging system ④, (f) Aging system ⑤.

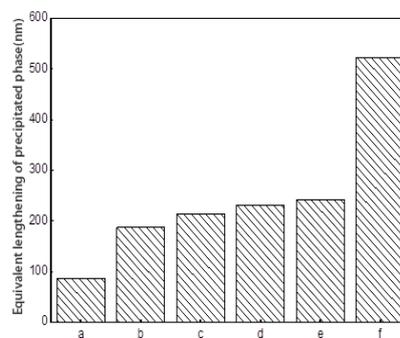


Figure 8. Equivalent side length of precipitated phase under different aging treatment conditions.

Table 5. Side length of precipitated phase under different aging systems.

Number	(a)	(b)	(c)	(d)	(e)	(f)
Length(nm)	86	187	213	231	242	523

It can be seen from Figure 7, the equivalent edge length of precipitated phase increases with aging time. The precipitated phases of the samples treated only by solid solution show irregular spherical shape, and most of them are approximately circular in cross section. Very few of them begin to grow sharp corners where they are close to each other, showing a trend of quadrilateral transformation. However, the distribution of precipitates is disorderly, only in the initial growth stage after nucleation. After initial growth and merger, a large number of precipitates nuclei formed at the beginning of cooling grow into spherical spheres, and the number decreases slightly compared with that at the beginning of cooling. However, due to the growth only in a very short cooling time, the volume is still very small and has not grown to the extent that the arrangement can be changed. From the comparison of Figure 8a,b, it can be seen that the precipitates have obvious growth changes after 1 h aging treatment at 1120 °C. The original spherical precipitation phase grows into a cube shape, which is quadrilateral in cross section, and the quadrilateral is very obvious, and the cubic degree is greatly improved. Compared with the equivalent side length of precipitation phase in Figure 8a, the equivalent side length of precipitation phase in Figure 8b reaches 187 nm. The volume of precipitation phase increases obviously, and the number of corresponding phases decreases greatly. At the same time, the distribution of precipitation phases tends to be regular, and the strengthened cubic phases are arranged tightly and orderly. From Figure 8b–e, it can be seen that the precipitates grow and merge further with the increase of holding time at 1120 °C. The cubic degree of the precipitates increases gradually with the increase of holding time. The magnification of Figure 8e and Figure 8b is 2.8 times as large as that of Figure 8b, the size of the mesophase is larger and the number of the mesophase is smaller, and the number of the mesophase decreases slightly. On the

one hand, the small phase grows gradually because of the continuous migration of elements; on the other hand, the surrounding macrophases also grow continuously. In the process of growing towards each other, the large and small phases break through the barrier of the matrix phase and merge together. It has become a new and larger overall precipitation phase. After holding for 1 h, the equivalent side length of the phase is 187 nm, and after holding for 4 h, the equivalent side length of the phase reaches 242 nm, and the volume of the phase increases obviously. Figure 8f shows that after holding for 4 h at 1120°C and holding for 32 h at 870°C the growth of the mesophase has become complete. Compared with the previous cases, the cubic degree is the highest, the arrangement of the mesophase is the most regular, the volume of the phase is the largest, reaching 523 nm, and the size difference between the precipitation phases is reduced, and there is too large or too small. The reinforcement phase inclusion disappears. Compared with the original sample, the size, shape and arrangement of phases are very close. It can be concluded that in the heat treatment system: 1335 4H + 1120 4H/AC + 870 32H/AC phase precipitation can achieve good results.

After solid solution heat treatment and air cooling, the precipitation phase gradually precipitates. In the process of precipitation and growth, various elements, such as Al, migrate, eventually leading to the differences in the content of elements between the precipitation phase and the matrix. From the comparison of Table 3, Table 4 and Table 1, the elemental content in the hardened phase and matrix phase is different from that of the sample after complete heat treatment only after solid solution treatment. It can be seen that the elements migrate during aging. In order to determine the influence of aging temperature and time conditions on the migration of elements in the precipitated phase and reinforced phase, the energy spectrum analysis was carried out on the surface of the sample. The principle of selecting points was consistent with the above solid solution treatment. The average element contents of each point were obtained at the points of appropriate amount in the reinforced phase and matrix phase, and the chemical composition of the precipitation phase and matrix phase under various aging systems. Listed in Tables 6 and 7.

Table 6. Constituents (mass fraction) of precipitation phases under three aging systems.

Aging system	Al	Cr	Co	Ni	Mo	Ta	W	Re
Non-aging	3.69	5.60	10.30	60.14	3.38	5.34	9.19	2.36
1120°C/4h	5.01	4.55	9.23	59.26	2.57	6.54	9.29	3.55
1120°C/4h, 870°C/32h	5.18	4.52	9.21	61.33	0.79	7.16	7.48	3.09

Table 7. Composition (mass fraction) of matrix phase under three aging systems.

Aging system	Al	Cr	Co	Ni	Mo	Ta	W	Re
Non-aging	5.18	4.66	9.49	59.21	1.67	7.32	6.36	4.13
1120°C/4h	5.01	4.69	9.18	58.28	3.39	6.32	9.52	3.62
1120°C/4h, 870°C/32h	4.64	5.21	10.57	60.67	1.73	5.31	8.74	3.13

It can be seen from Tables 6 and 7 that during aging, with the precipitation, merger and growth of the precipitation phase with Ni₃Al as the main component, Al migrates from the matrix phase to the precipitation phase, with the same migration trend as Ta and Re. It has been mentioned that when the material is treated by solid solution for enough time to form a single-phase solid solution, during the process of rapid cooling, some elements seem to be more easily attached to the nucleus to form precipitated phase and form dendritic structure along a specific growth direction, so there is usually serious element segregation in the dendritic cadre position. In the subsequent aging process, due to the long-term heat preservation at lower aging temperature, enough growth time was given to the precipitation phase. In the process of growth, the precipitation phase exchanges and merges elements with the surrounding matrix phase and fine secondary precipitation phase. The whole system

develops towards a more stable structure and the energy of the system decreases continuously. It can be seen from the above table that elements such as Cr, Co, Mo and W enriched in the precipitation phase migrate from the precipitation phase to the matrix phase during aging.

5. Dissolution and Precipitation Mechanisms

In the dissolution process of the γ' phase, the elastic field related to dislocation still exists in the microstructures, although the sample is not affected by external loads, which affects the dissolution behavior of the γ' phase. Because the channels of cubic reinforcement phase and matrix phase are closely arranged in a chessboard shape, there is elastic interaction between them, thus forming the local stability of the reinforcement phase accumulation group. With the solid solution treatment, the stability of each accumulation group is destroyed due to the influence of temperature, and the first destroyed accumulation group occurs dissolution behavior. Therefore, the overall dissolution behavior of the γ' phase is not a macroscopic phenomenon formed by the simple combination of dissolution of each γ' phase due to the influence of temperature. During the solid solution treatment process, some of these γ' phase stacks connected by elastic interaction become extremely unstable due to the absorption of heat energy, and gradually break away from the bonds between them and enter the matrix phase. That is to say, the dissolution behavior occurs, and the original stable structure is destroyed after the loss of this part. The structure of the whole precipitated phase group becomes more unstable. The lack of stability caused by partial dissolution destroys the integrity of elastic strain field and reduces enough elastic strain field. The resistance to γ' phase dissolution is weakened and the dissolution process becomes smoother and more uniform.

In the cooling stage after solid solution treatment, the solubility of precipitation phase elements in matrix phase decreases due to the decrease of temperature, and then nucleation and precipitation begin to grow. In the process of precipitation, the growth behavior of phase, including the number of nucleation, the size and shape of precipitation, the direction of precipitation growth, and the precipitation of secondary precipitate, are all affected by the cooling rate, which determines the nucleation rate and the kinetics of precipitate growth. Therefore, the morphology, size and distribution of precipitated phases and the overall micro-structure characteristics can be controlled and described by different cooling rates during cooling process. In the cooling process, when the cooling rate is low, as in the case of 0.5°C/s studied previously, due to the slow drop of temperature, low supersaturation in nucleation stage and slow nucleation of precipitated phase, the number of primary nucleation is small and the nucleation density is low, which makes the precipitated phase of primary nucleation have enough time to grow and gradually form dendritic structure. When the cooling rate is accelerated, the precipitate phase is "locked" in the early free growth process.

The microstructures of the specimens after solution treatment were studied by using different cooling rates to reduce the temperature from solid solution to room temperature. When the cooling rate is low, most of the precipitates have undergone relatively full free growth. After a long cooling process, the precipitation phases eventually come close to each other in four groups to form butterfly-like structure knots. The volume of the phase is large and the cube structure with sharp edges and corners is tetragonal in cross section, and the channel of the matrix phase is relatively wide. When the cooling rate increases, the number of phases increases, the volume of the final phase decreases, and the curvature of the corner decreases, and the channel of the matrix phase becomes finer. When air cooling is used, the precipitation phases in the material show irregular spheres, and the volume of the phase decreases obviously compared with the cooling rate at 0.15°C. From the cross section, the phase is irregular circular, a few have the tendency of edge angle, and the channel of the matrix phase is finer, and there are fine secondary precipitation phases precipitated. When the cooling rate is faster than that of water cooling, it is difficult to see the microstructure of the structure under the existing micro-electron microscopy. It can be seen that the micro-spheres are piled up in disorder. Because of the rapid cooling rate, the precipitation phase ends growing and solidifies in a very short time after precipitation.

It has been pointed out that the exact shape of the precipitation phase in the cooling process after solution treatment is affected by factors related to the elastic field and other diffusion dynamics. In

the solid solution treatment process, the precipitation phase and the matrix phase fully melt at high temperature to form a single-phase solid solution. When the temperature decreases, the solubility of the precipitation phase in the matrix phase decreases, and the solid solution is in the supersaturated state. At this time, the surface shape of the precipitated phase presents a depression shape, although from a purely elastic point of view, the phase is not in a balanced state. When the temperature continues to decrease and the precipitation phase continues to precipitate and grow rapidly from the matrix phase, metastable precipitation phase blocks are formed next to a dendritic structure formed along the growth direction. In the subsequent process, when the growth of the precipitate phase makes the surface state of the phase closer to the plane state of the equilibrium state. At this time, precipitated phases continue to precipitate and grow gradually in the decreasing channel of matrix phase, resulting in the continuous increase of volume fraction of γ' phase. At the end of the cooling stage, it can be observed that there are fine precipitation phases of different sizes in the channel of γ' phase and in the low saturation region around the primary precipitation phase. This indicates that during the cooling process, the new nuclei form and grow, and the existing precipitation phases continuously absorb various elements from the surrounding matrix phase for growth. System exists at the same time, and there is competition between them.

6. Conclusions

1. In the process of solid solution treatment of recovery heat treatment, as long as the appropriate solution temperature is selected, and after enough time of solid solution treatment, the precipitation phase will fully dissolve in the matrix phase to form uniform single-phase solid solution. The heating rate has no effect on the structure evolution and element migration of the precipitation phase after cooling.

2. When the cooling rate is very high, reaching the rate similar to air-cooling or water-cooling, the number of primary nucleation of enhanced phase precipitation is very large. The very short cooling time makes the growth degree of enhanced phase very low, and the shape of enhanced phase is inclined to irregular sphere, which shows disorder stacking growth. When the cooling rate is about $0.5^{\circ}\text{C}/\text{s}$, the reinforcement phase merges and reduces the number of reinforcement phases in the growth process, and gradually grows into a cube shape, and the distribution tends to be regular. When the cooling rate was about $0.15^{\circ}\text{C}/\text{s}$, the precipitation phase grew further, and a butterfly structure consisted of four precipitation phases was formed.

3. In the aging process, the precipitation phase has enough time to regenerate and grow, and the shape and size of the phase have been preliminarily adjusted at a higher aging temperature (1120°C). From the approximate sphere after air cooling, it has grown into a more regular cubic structure. Compared with the solid solution treatment, the size of the precipitation phase has increased greatly, and the phase arrangement tends to be compact. The cubic degree of the precipitation phase is further increased at a lower aging temperature (870°C), and the size of the precipitation phases becomes close to each other. The inclusion of too large or too small precipitation phases gradually disappears and the phase arrangement is more orderly. The main directions of elements migration are as follows: Al, Ta and Re elements are more concentrated in matrix phase during the cooling process after solid solution treatment and migrate to precipitation phase during aging process, the migration direction of elements Cr, Co, Mo and W in aging process is from precipitation phase to matrix phase.

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References

1. Z. Wen, D. Zhang, S. Li, et al. Anisotropic creep damage and fracture mechanism of nickel-base single crystal superalloy under multiaxial stress. *Journal of Alloys & Compounds*, 692, 2017, 301-312.
2. Y. Sha, L. Zuo, J. Zhang, et al. Anisotropy of compression creep strength of a nickel-based single crystal superalloy. *Journal of Metals*, 2001, 37(11): 1142-1146
3. Decker RF, Dewitt RR. Trends in high-temperature alloys. *Journal of Metals*, 1965, 17(2): 139-145.

4. Pearcey B J, Kear B H, Smashey R W. Correlation of structure with properties in a directionally solidified nickel-based superalloy. *Asm Transactions Quarterly*, **1967**, 60(4): 634-644
5. Pearcey BJ, Terkelse B E. Effect of unidirectional solidification on properties of cast nickel-base superalloys[J]. *Transactions of the Metallurgical Society of Aime*. **1967**, 239(8):1143-1150.
6. Nowotnik A, Kubiak K, Sieniawski J, et al. Development of Nickel Based Superalloys for Advanced Turbine Engines. *Materials Science Forum*, **2014**, 783-786: 2491-2496.
7. Wang T, Wang X, Zhao Z, et al. Dissolution behaviour of the γ' precipitates in two kinds of Ni-based superalloys. *High Temperature Technology*, **2016**, 33(1):51-57.
8. T. Grosdidier, A. Hazotte, A. Simon. Precipitation and dissolution processes in γ/γ' single crystal nickel-based superalloys. *Materials Science and Engineering A*, **1998**, 256(1-2):183-196.
9. Shuang, Jie-Shan, Kai-Xin, et al. Influences of Cooling Rate After Solution Treatment on Microstructural Evolution and Mechanical Properties of Superalloy Rene 80. *Journal of Metals: English Edition*, **2017**, 30(3):261-271.
10. Jackson J J, Donachie M J, Gell M, et al. The effect of volume percent of fine γ' on creep in Ds Mar-M200 + Hf. *Metallurgical Transactions A*, **1977**, 8(10):1615-1620.
11. Li, Fulin; Fu, Rui; Yin, Fajie. Impact of solution heat treatment on microstructure and creep behavior of a novel cast & wrought FGH4096 turbine disk alloy. *Microstructure and Processing of Properties of A-Structural Materials in Material Science and Engineering*, **2017**, 696: 273-282.
12. Wang B, Wen Z, Liu D, et al. Influence of Precipitate Size on the Strength of Nickel-Base Single Crystal Superalloys. *Rare Metal Materials & Engineering*, **2015**, 44(11):2747-2750.
13. Zhixun Wen, Haiqing Pei, Huan Yang, Yunwu Wu, Zhufeng Yue, A combined CP theory and TCD for predicting fatigue lifetime in single-crystal superalloy plates with film cooling holes, *International Journal of Fatigue*, **2018**, 111: 243-255.
14. Zhixun Wen, Dongxu Zhang, Songwei Li, Zhufeng Yue, Jingyun Gao. Anisotropic creep damage and fracture mechanism of nickel-base single crystal superalloy under multiaxial stress. *Journal of Alloys and Compounds*. **2017**, 692:301-312.
15. Ejaz N, Mansoor M, Salam I. Effect of high temperature exposure on the microstructure of Udimet-500 super alloy. *Journal of Materials Science*, **2010**, 45(16):4507-4517.
16. Pollock T M, Tin S. Nickel-Based Superalloys for Advanced Turbine Engines: Chemistry, Microstructure and Properties. *Journal of Propulsion & Power*, **2006**, 22(2):361-374.
17. Jackson M P, Reed R C. Heat treatment of UDIMET 720Li: the effect of microstructure on properties. *Materials Science & Engineering A*, **1999**, 259(1):85-97.
18. Qin X Z, Guo J T, Yuan C, et al. Effects of Long-Term Thermal Exposure on the Microstructure and Properties of a Cast Ni-Base Superalloy. *Metallurgical & Materials Transactions A*, **2007**, 38(12):3014-3022.