



Surface Recrystallization in Nickel Base Single Crystal Superalloy DD6

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Received 26 August 2009; accepted 17 November 2009

Abstract

The samples of single crystal superalloy DD6 are grit blasted and then heat treated either with the standard heat treatment procedure or in the temperature range of 1 000-1 250 °C for 4-16 h at vacuum atmosphere, then the recrystallization behavior of DD6 alloy is investigated. The results show that the equiaxed recrystallization grains form in the γ phase region where the as-cast γ' phases have been dissolved completely, and cellular recrystallization forms in the region where the as-cast γ' phases have been dissolved partially. The cellular recrystallization area consists of cellular grains, and the cellular grain consists of cubic γ' phase, lamellar γ' phase and $\gamma+\gamma'$. The coexistence of the equiaxed recrystallization zones and cellular recrystallization zones is a recrystallized characteristic of the cold worked single crystal samples which are heat treated at a temperature lower than the solution temperature. When the heating temperature is higher than 1 150 °C, with the increase of heat treating temperature, the equiaxed recrystallization zone expands, whereas the cellular recrystallization zone shrinks. All the deformed regions are consumed by equiaxed recrystallization after annealing at solution temperature.

Keywords: single crystals; superalloys; DD6; recrystallization; equiaxed recrystallization; cellular recrystallization; microstructure

1. Introduction

Single crystal (SX) superalloys possess extremely good elevated temperature capabilities due to the elimination of highly stressed grains boundaries^[1-2]. In particular, the creep strengths of SX superalloys are significantly higher than their polycrystalline counterpart. For this reason, they are widely used as turbine blades in advanced aero-engines and industrial gas turbines^[3-7]. DD6 is a Chinese low cost second generation SX superalloy, with tensile and creep rupture properties comparable to those of the second generation SX alloys such as SC180, René N5, CMSX-4 and PWA1484^[8].

During the manufacturing process of SX blades, a number of processes can occur and result in plastic deformation in the material. These processes are as follows: cooling from casting resulting in residual stresses, shaping processes used to produce the final contour to the blades, surface cleaning (such as shot

peening and grit blasting), and foreign object impact encountered. Then, surface recrystallization (RX) can take place in the SX blades during solution heat treatment^[9]. With the complete removal of grain boundary strengthening elements such as C, B, Zr, the presence of RX grains could be detrimental to the performance characteristics of SX alloy, particularly its creep resistance^[10-12]. It has been reported that the RX is one of the major causes of non-conformance during the processing of SX blades^[11]. However, little information is available in the open literature about the formation and growth of RX grains of SX superalloys. The main purpose of this investigation is to find the microstructural evolution and formation mechanism of RX behavior of SX superalloys.

2. Experimental

The nominal chemical compositions of the DD6 alloy are listed in Table 1^[8]. SX plate of the alloy, 160 mm in length, 24 mm in width and 12 mm in thickness was directionally solidified in Bridgman furnaces. The processing details were described elsewhere^[13]. Longitudinal orientation of the plate is within 15° deviating from [001] direction. In order to avoid additional

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surface deformation, electric discharge machine (EDM) was used to cut the plates into small pieces (11 mm× 11 mm×8 mm), with the 11 mm×11 mm plane parallel to the [001] direction.

Table 1 Nominal chemical compositions of single crystal superalloy DD6^[8]

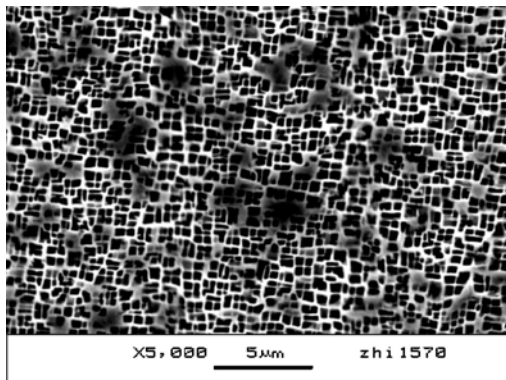
										wt%
Cr	Co	Mo	W	Ta	Re	Nb	Al	Hf	Ni	
4.3	9	2	8	7.5	2	0.5	5.6	0.1	Bal.	

The SX pieces were grit blasted at a pressure of 0.25 MPa on the 11 mm×11 mm plane for 30 s. SiO₂ spheres with a radius of 75 μm were used. The grit blasted pieces were sealed in a vacuum quartz tube, and then the samples were subjected to heat treatment. In order to study the microstructural evolution of RX process, the grit blasted samples were heat treated at 1 000 °C, 1 050 °C, 1 100 °C, 1 150 °C, 1 200 °C and 1 250 °C for 4-16 h respectively or with DD6 alloy standard heat treatment procedures: 1 290 °C/1 h+ 1 300 °C/2 h+ 1 315 °C/4 h (A.C.) + 1 120 °C/4 h (A.C.) + 870 °C/32 h (A.C.).

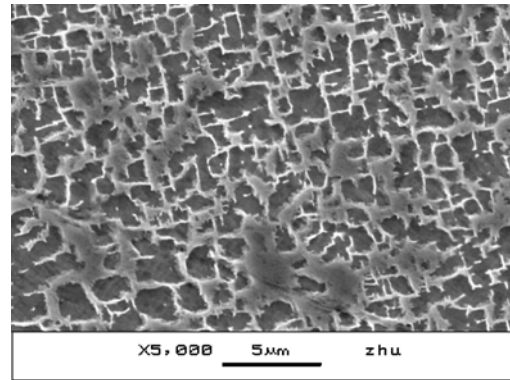
The samples were cut along the section perpendicular to the recrystallized surface by EDM after heat treatment. All the sections were polished and etched using the reagent containing 80 mL H₂O+100 mL HCl+20 g CuSO₄, and were applied for about 10 s. The microstructure of the recrystallized samples was examined by using optical microscopy (OM), scanning electron microscopy (SEM), high resolution scanning electron microscopy (HRSEM) and transmission electron microscopy (TEM). Electron dispersive X-ray spectroscopy (EDS) was used to observe the distribution profile of element near surface. In addition, some recrystallized samples were electrolytically etched in a solution of HCHO+HCL+Cr₂O₃ at a potential of 3 V. The average depth of RX was obtained based on the measurements every 100 μm along the recrystallized surface from at least five photographs for each sample.

3. Presentation of Results

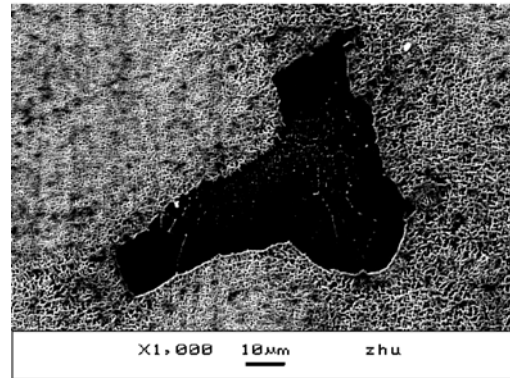
The as-cast microstructure of DD6 alloy is shown in Fig.1. The γ' phase morphology in dendrite core is



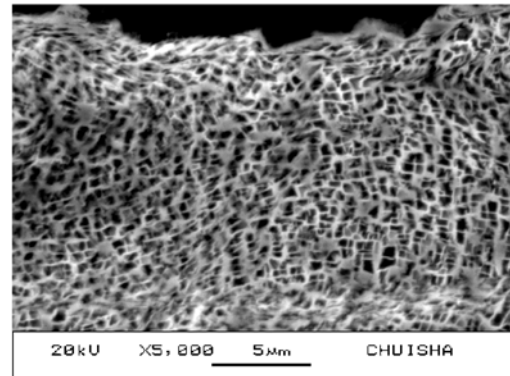
(a) In dendrite core in as-cast



(b) In interdendrite in as-cast



(c) γ+γ' eutectic



(d) After grit blasting

Fig.1 As-cast microstructure of DD6 alloy.

different from interdendritic region for the as-cast samples (see Fig.1(a)-(b)), and γ+γ' eutectic distributes in interdendritic region (Fig.1(c)). In contrast, the γ' phases are severely distorted in the cold worked region by grit blasting (see Fig.1(d)).

Progressive RX behavior has been studied by heat treating of the grit blasted SX samples. Fig.2 illustrates the microstructural evolution of the grit blasted samples heat treated either with the standard heat treatment procedure or in the temperature range of 1 000-1 250 °C for 4 h at vacuum atmosphere, respectively.

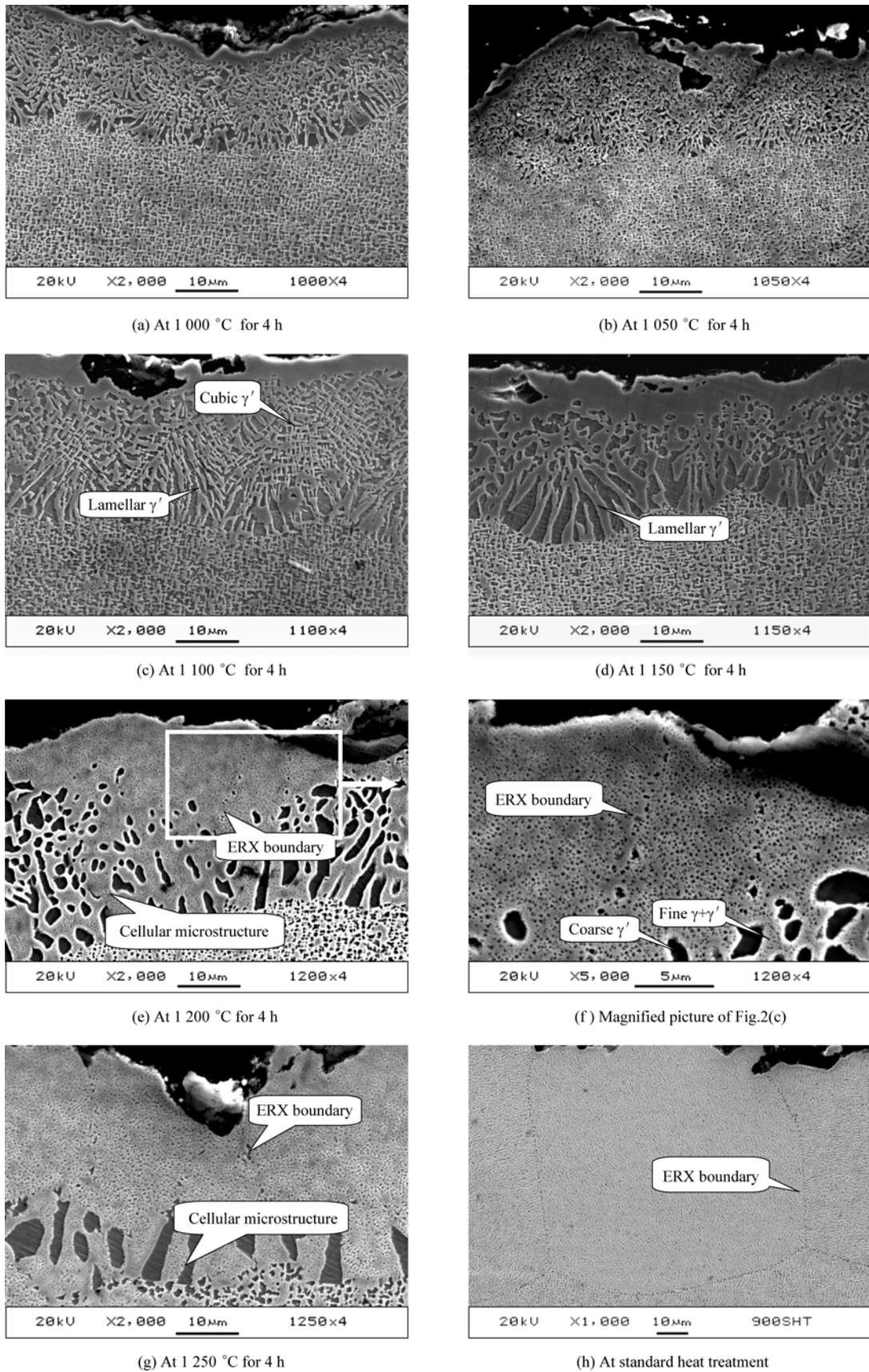


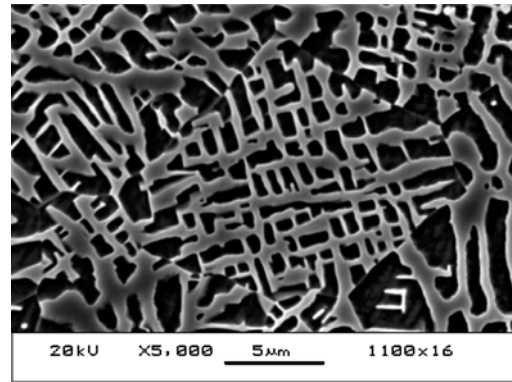
Fig.2 Microstructure of grit blasted DD6 alloy after annealing at standard heat treatment and in temperature range of 1 000-1 250 °C for 4 h.

The microstructure of grit blasted DD6 alloy after heated at 1 000 °C for 4 h is shown in Fig.2(a). Cellular recrystallization (CRX) occurred in the surface layer. The shape of γ' phases in the CRX was equiaxed near the surface of the grit blasted samples and lamellar at the interface between CRX and the original zone, respectively. Similar RX structure was observed when the grit blasted samples were heat treated at 1 050 °C for 4 h (see Fig.2(b)). The particulate γ' phases in the CRX zone partially turned to be cubic after 1 100 °C for 4 h (see Fig.2(c)). However, the lamellar γ' phases became coarser, and the cubic γ' phases partially merged into lamellar ones in the CRX zone at 1 150 °C for 4 h (see Fig.2(d)). After heat treated at 1 000 °C, 1 050 °C, 1 100 °C, 1 150 °C for 4 h, average CRX depth was approximately 16.6 μm , 18.7 μm , 22.0 μm , 23.0 μm , respectively.

Fig.2(e) shows the RX microstructure beneath the grit blasted surface in the sample annealed at 1 200 °C for 4 h. The equiaxed recrystallization (ERX) grains occurred near the surface, and CRX located between ERX grains and the original zone. The depth of ERX and CRX was approximately 12.5 μm , 13.5 μm respectively. The shape of γ' phases in the CRX changed from lamellar to coarse and equiaxed (see Fig.2(e)). Under this condition, the CRX zone consisted of coarse γ' phases and fine $\gamma + \gamma'$ (see Fig.2(f)).

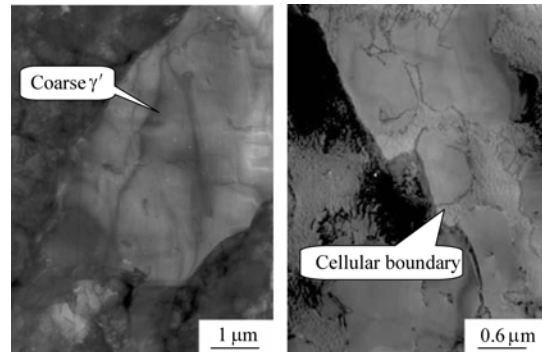
Cross section of CRX region is shown in Fig.3. It can be seen that the CRX region consists of cellular grains (see Fig.3(a)). The cubic and lamellar γ' phase in the CRX grains was coarse (see Fig.3(b)). TEM observation demonstrated the coarse γ' phase in the cellular grain (see Fig.4(a)); few dislocations were found in the new grains. This means that the deformed area is consumed by new grains which nucleate on the surface of grit blasted samples. The boundary between different cellular grains was observed (see Fig.4(b)), and the interface between the CRX and the original region was a high angle grain boundary^[11].

Fig.5 illustrates the ERX and CRX microstructures of the samples electrolytically etched. It can be seen clearly that the CRX consisted of cellular grains, and



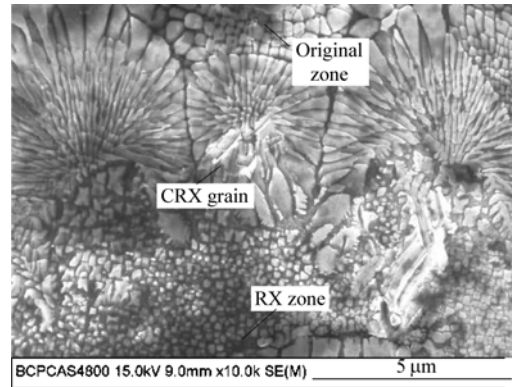
(b) Magnified SEM image of a cellular grain shown in the square of Fig.3(a)

Fig.3 Cross section of CRX of DD6 alloy heat treated at 1 100 °C for 16 h.

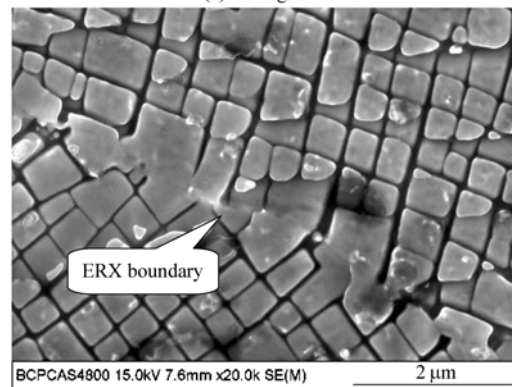


(a) Coarse γ' in cellular grain (b) Cellular grain boundary

Fig.4 TEM micrograph of cellular grain of DD6 alloy.

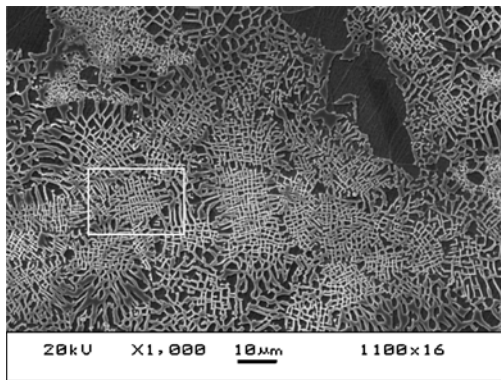


(a) CRX grains



(b) ERX grain boundary

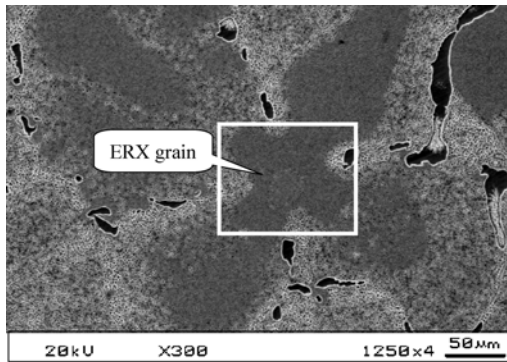
Fig.5 Microstructure of recrystallized samples electrolytically etched.



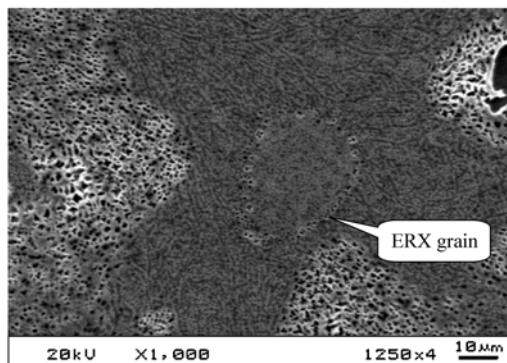
(a) Cross section of CRX

the interface between different cellular grains was obvious. Compared with the γ' phase in the original zone, the γ' phase in ERX grains was smaller. The lamellar γ' phase in the cellular grains was radial and perpendicular to the cellular grain boundary (see Fig.5(a)). Different from cellular grain, the γ' phase in the ERX grains was homogeneous, and there was an obvious misorientation between the γ' phases in different ERX grains (see Fig.5(b)).

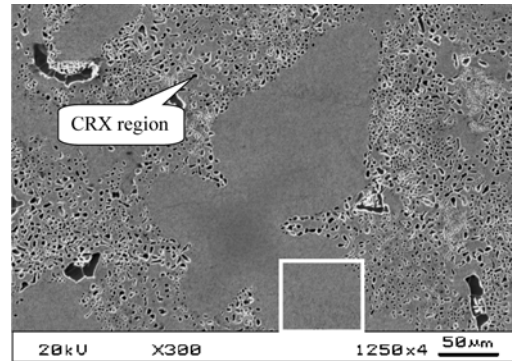
When the grit blasted samples were heat treated at 1 250 °C for 4 h, the ERX zone expanded and the CRX became a narrow zone. Also, the shape of γ' phases in CRX changed into thick lamina (see Fig.2(g)). Discontinuous ERX grains nucleated in the dendritic core (see Figs.6(a)-6(b)), and gradually proceeded to the interdendrite region. Then, the CRX grains formed in the interdendrite region until the entire dendritic cores were consumed by ERX (see Figs.6(c)-6(d)).



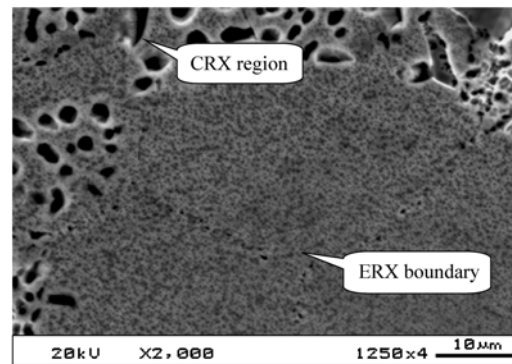
(a) ERX in dendritic core



(b) Magnified picture of Fig.6(a)



(c) ERX in dendritic core and CRX in interdendrite



(d) Magnified picture of Fig. 6(c)

Fig.6 Cross section of RX zone of grit blasted DD6 alloy heat treated at 1 250 °C for 4 h.

The deformed area was fully recrystallized after annealing at standard heat treatment. Compared to Fig.2(e) and Fig.2(g), the grain size was larger than that of the specimen annealed at 1 200 °C and 1 250 °C and the CRX region had not been found (see Fig.2(h)). The SEM observation showed that the ERX layer was approximately 75 μm in depth. The γ' phases were homogeneous and cubic in the ERX grains, but coarse on the ERX boundary.

4. Discussion

The schematic microstructure of grit blasted single crystal samples which were heat treated at a temperature lower than the solution temperature is shown in Fig.7. The surface region of grit blasted samples is divided into three districts, which are defined as District I, District II and District III.

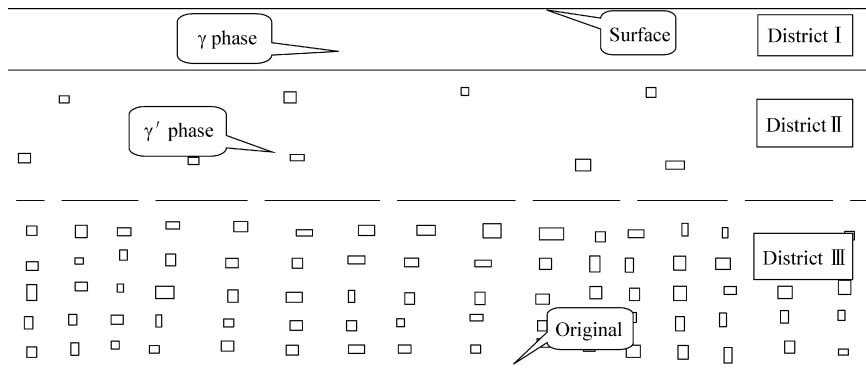


Fig.7 Schematic microstructure of deformation region in single crystal superalloy heat treated lower than solution temperature.

It has been reported that residual stresses assist dissolution of the existing phases^[12]. The surface of the single crystal specimens experienced most serious deformation during grit blasting, where there was an extensive concentration of retained energy. Therefore, the γ' phase in District I changed into solid solution at a high temperature, and then single γ phase formed in the surface region. The plastic deformation reduced as the distance was further away from the surface of specimen, and the center region almost kept as original. Therefore, the retained energy in District II and District III was lower than that in District I, but higher than the unaffected zone. Consequently, the volume of pct of γ' phases in District II and District III is lower

than the unaffected zone during heat treatment, but higher than District I. In addition, the EDS line-scan analysis demonstrated that no segregation was found on the surface of grit blasted sample (see Fig.8). On the contrary, Cr was relatively rich and the γ' phase forming elements (e.g. Al) were poor at the surface of grit blasted sample heated at 1 200 °C for 4 h (see Fig.9). It means that element diffusion is affected by the retained energy at heat treatment, and then the composition is different in District I, District II and District III. This would lead to different volume of pct of γ' phase in different districts on the surface of grit blasted sample after heat treatment.

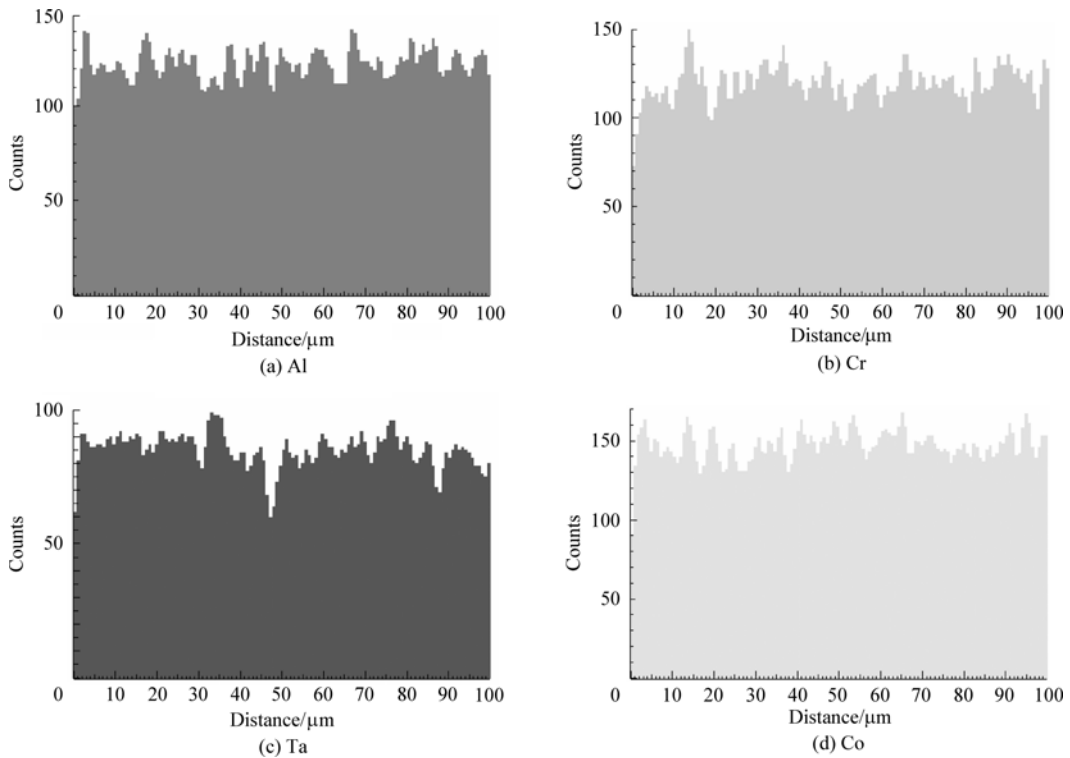
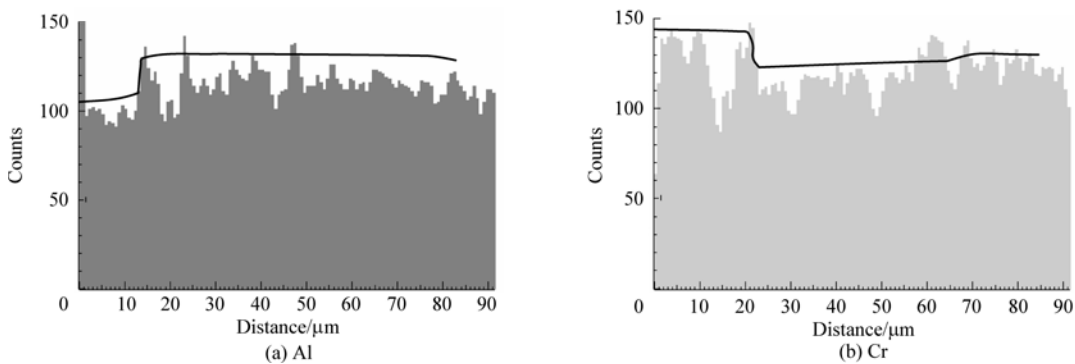


Fig.8 Distribution of Al, Cr, Ta and Co near grit blasted surface layer of DD6 alloy according to EDS.



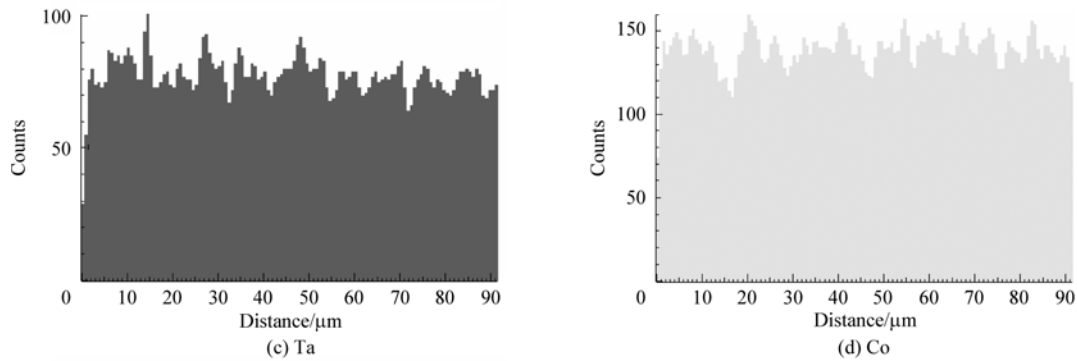


Fig.9 Distribution of Al, Cr, Ta and Co near grit blasted surface layer of DD6 alloy after heat treated at 1 200 °C for 4 h according to EDS.

An extensive concentration of dislocation lied within the deformed surface region, thus the stored energy which provided the driving force for RX was higher on the surface. As a result, the ERX grains originated in District I due to the absence of inhibition of the γ' phase. Furthermore, the new grains would gradually proceed to District II until District I was completely consumed by ERX.

Because the heating temperature did not reach the γ' solution temperature, there was a transition region between γ phase district and the original region. This transition region always located inside the deformed microstructure, which was defined as District II and District III. District II consisted of γ phase and γ' phase, and the fraction of γ' phase in this region was lower than that in the original region. District III also consisted of γ phase and γ' phase, and the fraction of γ' phase in this region was higher than that in District II but lower than that in the original region. With the growth of recrystallized grains, new grains boundary started to migrate to District II. The as-cast γ' phase in the District II would dissolve in the RX boundary. These effects were explained in terms of high solubility and diffusivity in the RX interface^[14-16]. The solute dissolving behavior of as-cast γ' phase in the District II would lead to supersaturation on the RX boundary. Compared with District III, the stored energy was higher near the cold worked surface, and the content of γ' phase was lower in District II, so the RX boundary migrated quickly. The cubic γ' phase would reprecipitates in the region where RX boundaries have migrated. On the contrary, the stored energy was lower and the content of γ' phase was higher in District III, then RX boundary migrated slowly. Therefore, the γ' phases almost reprecipitated in the RX interface at the same time as the as-cast γ' phases were solute dissolved. Consequently, this reprecipitating behavior along with the migrating grain boundary would make the reprecipitated γ' phase grow directionally, and finally formed the lamellar γ' phases. In addition, the lamellar γ' phases turned coarse in the CRX region due to the high reprecipitating temperature. As a result, the

CRX formed in District II and District III, and hence mixed characteristics of the ERX and CRX existed at the same time in the deformed region, as shown in Fig.2. It can be concluded that the ERX grains formed in the single γ phase region where γ' phase dissolved completely, and CRX formed in the region where γ' phase dissolved partially.

The γ' phase is small in the dendritic core, so it is more likely to dissolve during heat treatment. Then, the γ phase formed in the dendritic core, thus the ERX grains initiated. However, the γ' phase is coarser in the interdendrite. It is difficult to dissolve completely with heat treatment. This means that District II and District III locate in the interdendrite region at 1 250 °C. Then, the cellular grains formed in the interdendrite region (see Fig.3(c)).

The depth of different districts in Fig.7 varies with different heating temperatures. With the increasing heat temperature, the total depth of three districts increased. When heating temperature was higher than 1 150 °C, District I expanded, District II shrunk, and when temperature was higher than 1 250 °C, District III began to shrink (see Fig.10). As a result, when the heating temperature was higher than 1 150 °C, with the rising of heat treatment temperature, the ERX region expanded, while the CRX region shrunk (see Fig.11).

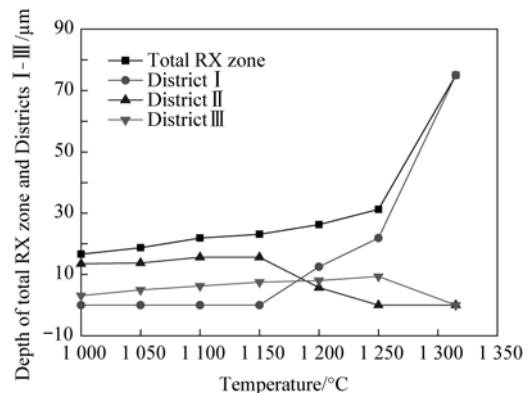


Fig.10 Depth of total RX zone and Districts I-III at different temperatures.

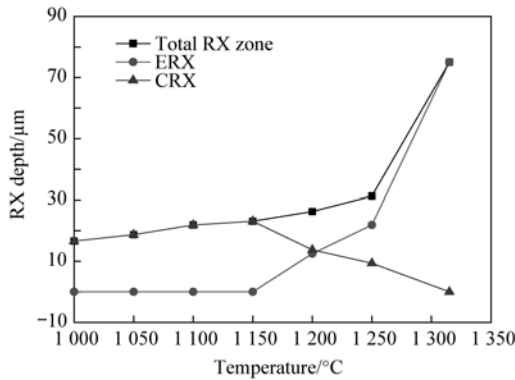


Fig.11 Depth of CRX and ERX at different temperatures.

The single γ phase formed in the deformed region at the γ' phase solution temperature. The ERX boundary was not inhibited by γ' phase during the migrating process, so the ERX grains consumed all the deformed regions and the cellular grains would not occur.

5. Conclusions

The ERX grains form in the γ phase region where the γ' phase has been dissolved completely and CRX forms in the region where the γ' phase has been dissolved partially. The CRX region consists of cellular grains, and the cellular grains consist of cubic γ' phase, lamellar γ' phase and fine $\gamma+\gamma'$. The coexistence of the ERX zones and CRX zones is a recrystallized characteristic of the cold worked single crystal samples which were heat treated at a temperature lower than the solution temperature. When the heating temperature is higher than 1150 °C, with the increase of heat temperature, the ERX region expands, while the CRX region shrinks. All of the deformed regions are consumed by the ERX grains, and the CRX would not occur after annealing at the γ' phase solution temperature.

Acknowledgements

The authors are grateful to Liu Liyu for her experimental assistance in this study. The authors also greatly appreciate their helpful discussions with Chen Rongzhang and Qu Shiyu.

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Biography:

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