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ARTICLE

Anisotropic Creep Behavior of DD15 Single Crystal Superalloy

Shi Zhenxue, Liu Shizhong, Yue Xiaodai, Li Jiarong

Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, Beijing 100095, China

Abstract: DD15 single crystal superalloy with [001], [011] and [111] orientations was cast by seed method in the directional solidified furnace. The creep properties of the alloy with different orientations were investigated at 980 °C/300 MPa. In order to obtain the microstructure evolution after different creep time, the creep after 50 and 100 h and the creep fracture of three kinds of tests were investigated. The alloy microstructure with different orientations were investigated by SEM and TEM. The results show that the alloy with different orientations has obviously various microstructure characteristics on the section perpendicular to the crystal growth direction. The γ' phase with [001] orientation is regular square, that with [011] orientation is rectangular, and that with [111] orientation is polygon. The creep properties of the alloy exhibit obvious anisotropy at 980 °C/300 MPa. The creep life of the alloy decreases in the sequence of [111], [001] and [011] orientation. The creep strain of the alloy under different conditions decreases in the sequence of [001], [011] and [111] orientation. The common feature of the creep curves is a very short primary creep stage. Compared with creep curves of the [001] and [111] orientation, the creep curve of [011] orientation has a shorter third creep stage. The degree of γ' coarsening is obviously different for alloy with different orientations after creep. The rafting speed of [001] oriented sample is larger than that of [011] and [111] oriented sample. After creep fracture, the dislocation network of [001] or [111] oriented alloy is denser and better than that of [011] oriented alloy.

Key words: single crystal superalloy; DD15; creep behavior; anisotropy

Single crystal superalloys have excellent mechanical properties at high temperature, so they are widely used in the casting of aero-engine turbine blades[1,2]. As a kind of monocrystalline metal material, the single crystal superalloy also has anisotropy[3-7]. Although the stress of single crystal blade is mainly centrifugal load in the [001] orientation, multi-axial stress will still occur due to the complex blade shape and the temperature gradient of each part. Therefore, it is necessary to investigate the effect of crystal orientation on creep properties of the single crystal superalloy. There are a lot of studies on anisotropic creep of single crystal superalloy[8-10]. The creep rupture life of the first generation single crystal superalloy DD407 with [111] orientation at 980 °C/260 MPa is the longest, while the rupture life of [001] oriented alloy is almost identical with that of [011] oriented alloy[11]. The creep properties of DD6 alloy are anisotropic and the alloy with [001] orientation has higher working-hardening tendency than the alloy with [011] and [111] orientations[12]. The creep strength of the second generation single crystal superalloy CMSX-4 with [111] orientation is the smallest in the three orientations at 850 °C[13]. The creep life of the third generation single crystal superalloy at 1100 °C/150 MPa decreases in the sequence of [001], [011] and [111] orientation[14]. DD15 alloy studied in this research was developed for aeroengine turbine blade applications by Beijing Institute of Aeronautical Materials. The properties of the alloy are equivalent to those of other fourth-generation single crystal superalloys[15,16]. In support of the development of the fourth-generation single crystal superalloy, it is necessary to investigate anisotropic creep behavior of the alloy.

1 Experiment

DD15 single crystal superalloy with [001], [011] and [111]

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Corresponding author: Shi Zhenxue, Ph. D., Professor, Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, Beijing 100095, P. R. China, Tel: 0086-10-62498312, E-mail: zhenxue.shi@biam.ac.cn

orientations was cast by seed method in the directionally solidified furnace. The nominal chemical compositions of the DD15 alloy are shown in Table 1. The crystal orientations of the samples were determined by X-ray diffractometer, and the crystal orientation deviations of the samples with [001], [011] and [111] orientations are 2.1°, 2.7° and 1.6°, respectively. The samples received a heat treatment including a solution treatment (1330 °C/6 h, AC) and a two-step aging treatment (1140 °C/4 h, AC + 870 °C/32 h, AC). Then the specimens for creep property test were machined along the original orientation of the sample. Fig. 1 shows schematic diagram of specimen for creep property test. They were tested at 980 °C/300 MPa which is the common test condition for the fourth generation single crystal superalloy. The 980 °C/300 MPa condition is one of the creep tests required by DD15 alloy research projects. In order to obtain the microstructure evolution after different creep time, the creep stopped at 50 and 100 h and the creep fracture of three kinds of tests was carried out. The samples were electrolyzed in a solution of citric acid, ammonium sulfate and water which dissolves γ matrix or etched with $CuSO_4$+HCl+H_2O+H_2SO_4 which dissolves γ' phase. Microstructures were examined by scanning electron microscope (SEM). Foils for transmission electron microscopy (TEM) of the creep ruptured samples were obtained by cutting 0.2-mm-thick discs perpendicular to the tensile axis of the samples using an electric discharge machine. Thin foils were prepared by twin-jet thinning electrolytically in a solution of perchloric acid and ethanol.

2 Results

2.1 Microstructure after heat treatment

The microstructures of DD15 alloy with different orientations after heat treatment are shown in Fig. 2. It can be seen that the microstructures consist of γ' precipitates embedded coherently in the γ matrix phase and the alloy with different orientations has obviously various microstructure characteristics on the section perpendicular to the crystal growth direction. The γ' phase with [001] orientation is regular square, that with [011] orientation is rectangular, and that with [111] orientation is polygon. The γ' phases of the alloy with different orientations are all in cubic shape after heat treatment. The morphology feature of γ' phases with different orientations is related to the section orientation. The vertical growth cross section of [001] oriented alloy is {001} plane, that of [011] oriented alloy is {011} plane and that of [111] oriented alloy is {111} plane. So different morphology features of γ' phase can be obtained because of different sections.

2.2 Creep properties

The creep curves of DD15 alloy with different orientations at 980 °C/300 MPa are shown in Fig.3 and the corresponding creep properties are shown in Table 2. It can be seen that the creep life of the alloy in different conditions decreases in the sequence of [111], [001] and [011] orientation. The creep strain of the alloy in different conditions decreases in the sequence of [001], [011] and [111] orientation. The common feature of the creep curves is a very short primary creep stage. However, there are two different types of creep behavior in the creep curves with different orientations. Compared with creep curve of the [001] and [111] orientation, that of [011] orientation has a shorter third creep stage.

2.3 Microstructure evolution at different creep time

The microstructure on the longitudinal section in the specimens of the alloy at different creep time with different orientations is shown in Fig.4. It illustrates that the coarsening extent of γ' phases obviously varies for alloys with different orientations after different creep time. For [001] oriented sample, the γ' phase is still in cubic shape after creep for 50 h and the higher perfection of rafted structure forms after creep for 100 h. The γ matrix is no longer continuous, forming islands surrounded by the γ' phase after creep for 401 h. For [011] oriented sample, the γ' phase remains cubic even after creep fracture (200.5 h). For [111] oriented sample, the γ' phase remains cubic after creep for 100 h and the severe rafted microstructure forms after creep fracture (1128.9 h). The γ' raft of the alloy is at an angle to stress direction. No topological close packed (TCP) phase can be observed in creep microstructure of the alloy with different orientations. So DD15 alloy has very excellent microstructure stability. The rafting speed of [001] oriented sample is larger than that of [011] and [111] oriented sample.

The cubic γ' phase gradually changes into a raft structure because of the directional diffusion of the elements[17]. It is determined by applied stress and γ/γ' mismatch[18]. In the process, the diffusion and redistribution of the alloying elements in the γ' and γ phases occur[19]. The forming elements Hf, Nb, Ta, Al of γ' precipitates diffuse to the vertical channels at high temperature to promote the γ' phase growth perpendicular to [001] direction under the action of the

Table 1 Nominal chemical composition of DD15 alloy (wt%)

Cr	Co	Mo	W	Ta	Re	Ru	Nb	Al	Hf	Ni
2~4	7~10	0.8~1.5	6~9	7~10	4~6	2~4	0.2~1.0	5~6	0.1~0.3	Bal.

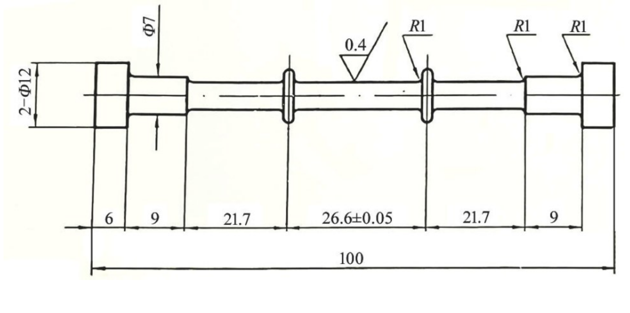

Fig.1 Schematic diagram of specimen for creep property test

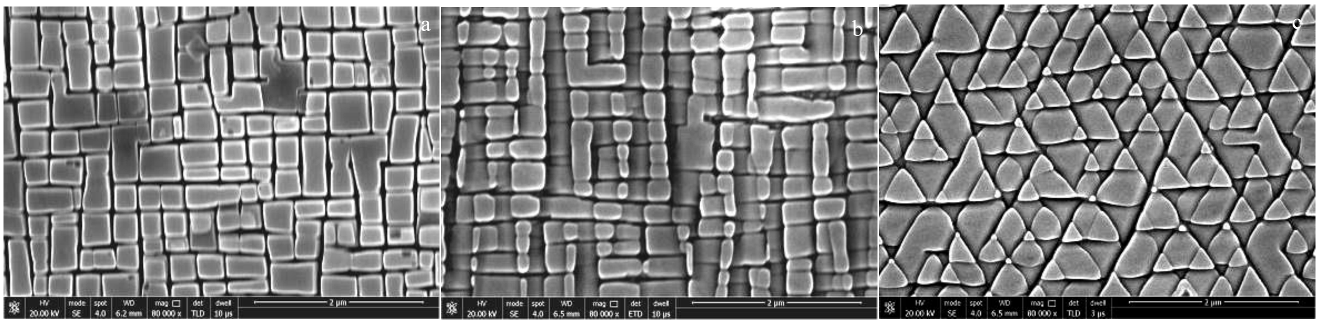

Fig.2 Microstructures of DD15 alloy with [001] (a), [021] (b), and [111] (c) orientations after heat treatment

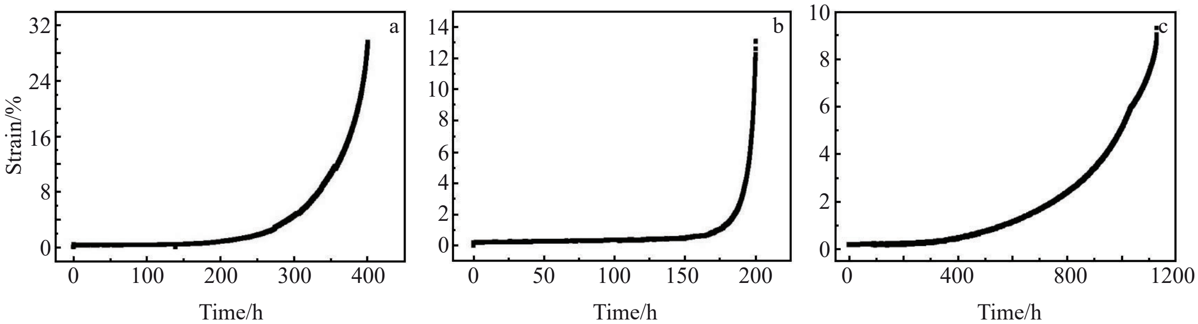

Fig.3 Creep strain curves of DD15 alloy with different orientations at 980 °C/300 MPa: (a) [001], (b) [011], and (c) [111]

Table 2 Creep properties of DD15 alloy with different orientations at 980 °C/300 MPa

Orientation	Total time to 0.5%/h	Total time to 1%/h	Total time to 2%/h	Total time to 5%/h	Rupture life/h	Rupture strain/%
[001]	174.7	217.6	260.0	309.2	401.0	29.5
[011]	174.3	185.4	193.1	195.0	200.5	13.1
[111]	420.1	567.7	752.2	997.3	1128.9	9.3

applied stress and the misfit stress. The forming elements Re, Mo, W, Co, Cr of γ matrix diffuse to the horizontal channels in the reverse direction to enlarge the γ matrix width in the meantime. Under the condition of high temperature and stress, the γ' rafting structure is gradually formed. The higher the stress, the faster the γ' phase grows and rafts. The applied stress on the γ' phase with [001] orientation is the largest in three samples, so its speed of γ' rafting is the fastest.

3 Discussion

Most of single crystal superalloys with [111] orientation have the best creep performance at high temperature, but there are other different results. The [111] orientation exhibits the poorest creep strength due to poor strain hardening and the activation of additional deformation mechanism in the CMSX-4[13]. The [001] oriented specimens display the longest rupture life, [111] oriented specimens show the shortest rupture life, and [011] specimens exhibit the intermediate life in a third-generation nickel-based single-crystal superalloy[14]. It is because for [111] specimens, a large number of crack propagation paths and inhomogeneous deformations caused by irregular rafted structures deteriorate the property and result in the shortest life. The [001] oriented specimen has the best ductility and the [111] oriented specimen has the longest lifetime, while these two properties of the [011] oriented specimen are lower[7]. Therefore, the test temperature, loading stress, chemical composition and microstructure will all affect the anisotropic features.

The DD15 alloy is the fourth generation single crystal superalloy which is newly developed in China. The alloy contains a large number of reinforcing elements, such as Re, Ta, W, Ru. Re, W and Ru are the forming elements of γ matrix and Ta is the forming element of γ' precipitates. During the creep process, the γ matrix and γ' precipitate play the role of solution strengthening and precipitation strengthening, respectively. The octahedral and hexahedral slip system start at the same time at 980 °C. The number of actuated slip systems of the alloy with [001], [011] and [111] orientation is 8, 4 and 6, respectively, and their Schmid factors of slip system are 0.41, 0.27 and 0.41, respectively[12]. The alloy with [001] orientation has a higher creep strength because of most slip system and higher Schmid slip factor. The alloy with [111] orientation has the highest creep strength because more slip system and the highest Schmid slip factor make the alloy evenly deform to produce the strongest deformation reinforcement. However, the alloy with [011] orientation has the minimal slip system and higher Schmid slip factor, which

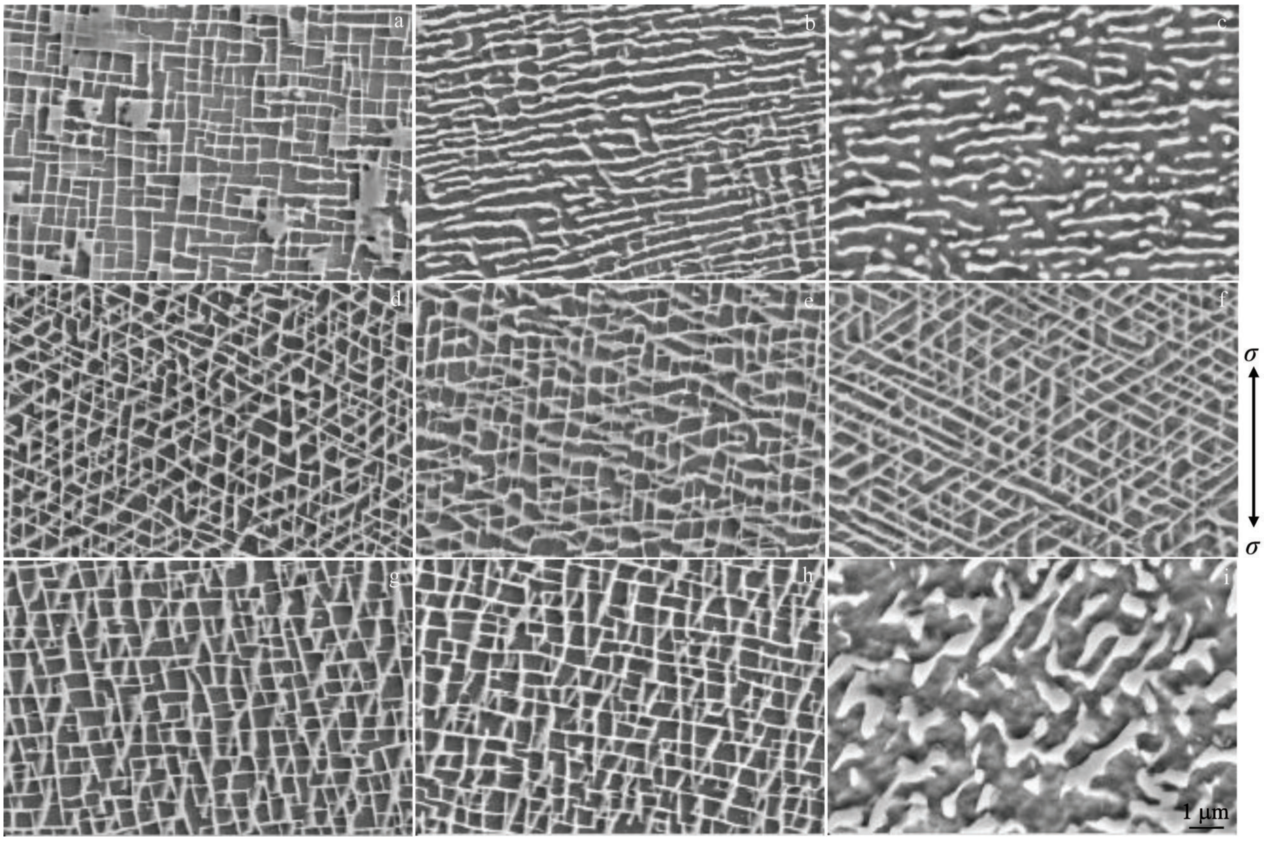

Fig.4 Microstructures of DD15 alloys with different orientations after different creep time: (a) [001], 50 h; (b) [001], 100 h; (c) [001], 401 h; (d) [011], 50 h; (e) [011], 100 h; (f) [011], 200.5 h; (g) [111], 50 h; (h) [111], 100 h; (i) [111], 1128.9 h

result in uneven deformation, lower probability of crossover between different slip systems and the lower resistance to deformation. When the cross section of sample with [011] orientation becomes elliptical, the actual loading stress increases significantly, which in turn increase the creep rate. So the alloy with [011] orientation has a shorter third creep stage and the lowest creep life.

The difference of creep life at high temperature may be due to different degrees of γ' rafting[9]. However, the rafting speed of [001] oriented sample is larger than that of [011] and [111] oriented sample in this study. Fig. 5 shows the γ/γ' interfacial dislocation feature of creep ruptured sample at 980 °C/300 MPa. It shows that the dislocation network is formed at γ/γ' interfaces of the alloy with different orientations. The deformation mechanism of dislocations is by-pass at high temperature[20]. The dislocation networks arise from two sets of dislocation reaction with different Burgers vectors during plastic deformation. The dislocations belong to different slip planes. The networks form by dislocation reaction when the dislocations move to the same slip plane and cross each other. The dislocation cross of different slip systems with [011] orientation may be smaller than that in other directions. So the dislocation network with [001] or [111] orientation is denser and much more better than that with [011] orientation. The dense γ/γ' interfacial dislocation networks can prevent the dislocation from cutting into γ' phase in the creep process[21].

Fig.5 Creep fracture microstructures of DD15 alloy with different orientations at 980 °C/300 MPa: (a) [001], (b) [011], and (c) [111]

In the later creep process, the deformation feature of the alloy is the shearing of γ' phase by dislocations from the interfaces where networks are damaged. It may be one of other reasons that cause short creep rupture life of [011] samples.

4 Conclusions

1) The DD15 single crystal superalloy with different orientations has obviously various heat treated microstructures on the section perpendicular to the crystal growth direction. The γ' phase with [001] orientation is regular square, that with [011] orientation is rectangular, and that with [111] orientation is polygon.

2) The creep properties of the alloy exhibit obvious anisotropy at 980 °C/300 MPa. The creep life of the alloy decreases in the sequence of [111], [001] and [011] orientation. The common feature of the creep curves is a very short primary creep stage. Compared with creep curves of the [001] and [111] orientation, that of [011] orientation has a shorter third creep stage.

3) The degree of γ' coarsening is obviously different in alloy with different orientations. The rafting speed of [001] oriented sample is larger than that of [011] and [111] oriented sample. The dislocation network with [001] or [111] orientation is denser and better than that with [011] orientation.

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DD15单晶高温合金的蠕变各向异性

史振学，刘世忠，岳晓岱，李嘉荣
(北京航空材料研究院 先进高温结构材料重点实验室，北京 100095)

摘　要：在定向凝固炉中制备了［001］、［011］和［111］3种不同取向的DD15单晶高温合金，研究了980 ℃/300 MPa条件下不同取向的蠕变性能。为对比分析不同蠕变时间后的微观组织，蠕变50和100 h后停止试验。采用扫描电镜和透射电镜分析不同取向的合金组织。结果表明，不同取向合金在垂直于生长方向的截面上具有不同的组织特征，［001］取向γ'相为规则的正方形，［011］取向γ'相为矩形，［111］取向γ'相为多边形。合金在980 ℃/300 MPa条件下的蠕变性能呈现明显的各向异性，蠕变寿命按［111］、［001］、［011］取向的顺序减小，应变量按［001］、［011］、［111］取向的顺序降低。3种取向合金蠕变曲线的共同特征为具有非常短的蠕变第1阶段，与［001］和［111］取向相比，［011］取向合金具有较短的蠕变第3阶段。不同取向合金蠕变后的γ'相具有明显不同的筏排化程度。［001］取向合金的γ'相筏排化速率大于［011］和［111］取向合金。蠕变断裂后，［001］或［111］取向合金的位错密度大于［011］取向合金。

关键词：单晶高温合金；DD15；蠕变性能；各向异性

作者简介：史振学，男，1975年生，博士，研究员，北京航空材料研究院先进高温结构材料重点实验室，北京 100095，电话：010-62498312，E-mail：zhenxue.shi@biam.ac.cn