Effect of grit blasting and subsequent heat treatment on stress rupture property of a Ni-based single-crystal superalloy SGX3

Jinshan He¹ · Zhengrong Yu² · Longfei Li² · Xitao Wang^{1,3} · Qiang Feng²

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Abstract

In the present study, the effect of grit blasting and subsequent heat treatment on the stress rupture properties of a third-generation nickel-based single-crystal superalloy SGX3 sheet was studied. It was found that the stress rupture life of alloy SGX3 sheet at 980 °C/250 MPa was reduced by about 60% by only vacuum heat treatment at 1100 °C for 200 h and further reduced by 20% and 70% respectively with grit blasting of 0.3 MPa/1 min and 0.5 MPa/2 min before heat treatment. The microstructure analysis results indicated that the degradation of stress rupture life of alloy SGX3 sheet by vacuum heat treatment was mainly attributed to the variation of γ/γ' microstructure, i.e., the decrease in γ' volume fraction and the coarsening of γ' precipitates. Furthermore, such degradation by grit blasting and subsequent vacuum heat treatment should be attributed to the formation of cellular recrystallization with different thicknesses at the surface of alloy SGX3 sheet, which not only acts as the vulnerable site for cracks to initiate and propagate but also reduces the effective loading area.

Keywords Ni-based single-crystal superalloy · Creep life · Degradation · Recrystallization

1 Introduction

Ni-based superalloys are often used for high-pressure turbine blades of advanced aero engines [1–5]. Among these, single-crystal alloys have been introduced due to their excellent high-temperature mechanical properties by eliminating grain boundaries [3, 6]. Both thermal stress by temperature gradient during casting and mechanical stress caused by surface processing such as shot peening or grit blasting lead to the

local plastic deformation [7–9]. At those sites, the equiaxed recrystallization can occur when Ni-based single-crystal superalloy is further heat-treated at solution temperature [10, 11]. As a result, the transverse grain boundaries introduced by recrystallization lead to the degradation of creep property of Ni-based single-crystal superalloy. Jo et al. reported that the recrystallized grains grew preferentially in the dendritic core and early crack initiation on grain boundaries of surface recrystallization caused a relatively high creep rate in alloy CMSX-2 at 982 °C, 240 MPa [12, 13]. Meng et al. found that creep rupture life shows a sharp decrease with increase in recrystallized depth in a Ni-based superalloy deformed by shot peening [14]. While cellular recrystallization consisting of γ phase, γ' phase and TCP phases may form when the Ni-based superalloy is subjected to lower temperature during service, threatening the safe operation of blades. For instance, it is reported that cellular microstructure of different thicknesses formed on a Ni-based superalloy containing Re and Ru after grit blasting and then heat treatment at 1100 °C [15]. Local stress facilitated cellular recrystallization on single-crystal superalloy accompanied with formation of twin structure and TCP phase during annealing at sub-solvus temperature [16]. Therefore, it is necessary to investigate the effect of cellular recrystallization on stress rupture property for Ni-based single-crystal superalloy.

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✉ Longfei Li
lilf@skl.ustb.edu.cn

- ¹ Beijing Advanced Innovation Center for Material Genome Engineering, Collaborative Innovation Center of Steel Technology, University of Science and Technology Beijing, Beijing 100083, China
- ² Beijing Advanced Innovation Center for Material Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China
- ³ Shandong Provincial Key Laboratory for High Strength Lightweight Metallic Materials, Advanced Materials Institute, Qilu University of Technology (Shandong Academy of Sciences), Jinan 250353, China

More recent studies have identified the degradation effect of recrystallization on creep resistance of Ni-based superalloy [17, 18]. Xie et al. reported an almost linear reduction of the creep rupture life with the increase in the local recrystallized depth for directionally solidified Ni-based superalloy and suggested that the transverse recrystallized grain boundary is a vulnerable site of crack initiation and propagation [19]. In addition, Xiong et al. reported that the stress rupture life of a Ni-based superalloy with cellular recrystallization reduces slightly than that with equiaxed recrystallization after grit blasting and heat treatment by using the round bar specimens. However, most of the turbine engines are thin-walled components, even as 0.5–1.0 mm thickness [20–22]. The ratio of cellular colony area for round bars and sheet specimens should be different even though the manufacturing process is same. Consequently, the effect of grit blasting on the stress rupture properties for the two types is not the same. Furthermore, few studies have distinguished the separate effects of degraded γ' phase (i.e., the decrease in γ' volume fraction and the coarsening of γ' precipitates) and recrystallization induced by the subsequent heat treatment after grit blasting on creep properties quantitatively.

In the present work, to investigate grit blasting effect on the stress rupture property for a third-generation Ni-based single-crystal superalloy SXG3, different grit blasting processes are used to induce various plastic deformation at surface for sheet materials. After heat treatment at 1100 °C in vacuum to mimic the serving temperature, the stress rupture properties for different specimens were tested and the corresponding microstructures before and after creep tests were characterized by scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD). Bare specimens and specimens just heat-treated at 1100 °C in vacuum are also investigated for comparison.

2 Experimental

The nominal composition (wt%) of the investigated third-generation single-crystal superalloy is Ni-12.0Co-4.0Cr-2.0Mo-6.0W-7.0Ta-5.0Re-5.0Al-0.15Hf-0.02C. The single-crystal bars grown along [001] orientation within 5° were cast by Bridgman method and then were subjected to standard heat treatment (1300 °C/4 h, AC + 1100 °C/4 h, AC + 870 °C/16 h, AC (air cooling)). The single-crystal bars were machined into the sheet specimens for stress rupture tests, whose geometry is illustrated in Fig. 1. To investigate the effect of grit blasting on stress rupture properties, specimens with different processing were designed as shown in Table 1. Specimens GH1 and GH2 were grit-blasted at pressure of 0.3 MPa for 1 min and 0.5 MPa for 2 min, respectively. Subsequently, they were sealed in silica glass tubes and heat-treated in vacuum at 1100 °C for 200 h. In addition,

Fig. 1 Geometry of creep rupture testing specimen (mm)

Table 1 Specimens with different grit blasting

No	Standard heat treatment	Grit blasting	Vacuum heat treatment
SH	✓		
VH	✓		1100 °C/200 h
GH1	✓	0.3 MPa/1 min	1100 °C/200 h
GH2	✓	0.5 MPa/2 min	1100 °C/200 h

bare specimens SH with standard heat treatment and specimens VH heat-treated in vacuum at 1100 °C for 200 h after the standard heat treatment were prepared. For each processing, at least two stress rupture tests were conducted at 980 °C/250 MPa.

Microstructures for different grit blasting before and after stress rupture tests were observed by SEM. The orientations for recrystallized grains near the fracture surface were characterized with EBSD. Metallographic specimens were prepared by grinding with 240–2000# grind paper and polishing with Al₂O₃. The polished specimens were etched with the liquid consisting of 1 vol.% HF, 33 vol.% HNO₃, 33 vol.% CH₃COOH and 33 vol.% H₂O.

3 Results

The microstructures and the result of related statistical analysis for different specimens before stress rupture tests are presented in Fig. 2 and Table 2, respectively. As shown in Fig. 2a, 64.0 vol.% cuboidal γ' precipitates distributed regularly in γ matrix for specimen SH. For specimen VH, the γ' precipitates coarsened and connected, as shown in Fig. 2b. The γ' volume fractions of specimen VH was 40.6%, smaller than 60% of specimen SH. For specimens GH1 and GH2, cellular recrystallization consisting of γ phase, γ' phase and TCP phase formed at surface, as shown in Fig. 2c, d. The average thickness of cellular recrystallization in specimen GH2 was 49.6 ± 4.2 μ m, larger than 18.4 ± 1.9 μ m in specimen GH1. The formation of cellular colonies should be attributed to the occurrence of cellular recrystallization below γ' solvus temperature driven by the stored energy of

Fig. 2 Microstructures of different specimens before stress rupture tests: **a** specimen SH; **b** specimen VH; **c** specimen GH1; **d** specimen GH2

Table 2 Statistical analysis of microstructures for different specimens

Specimen	Thickness of cellular recrystallization μ (μm)	Area of cellular recrystallization ($\times 10^5 \mu\text{m}^2$)	Effective loading area (%)	Volume fraction of γ' (vol.%)
SH	0 ± 0	0	100	64.0
VH	0 ± 0	0	100	40.6
GH1	18.4 ± 1.9	1.5	96.1	40.1
GH2	49.6 ± 4.2	4.0	89.4	40.0

plastic deformation caused by grit blasting [21, 22]. Thus, the increase in grit blasting intensity usually leads to an increase in the thickness of cellular recrystallization [13, 23]. The γ' volume fractions of the matrix in specimens GH1 and GH2 were around 40%, similar with that of specimen VH. Since the intrinsic strength of the cellular recrystallization is much lower than the original γ/γ' microstructure leading to the loss of load bearing area, the effective loading area was defined as the ratio of the area without cellular recrystallization over the whole transverse area [23, 24]. With the thickness of cellular recrystallization, the effective loading area fractions for specimens GH1 and GH2 were calculated to be 96.1 and 89.4%, respectively. Both the change of γ/γ' microstructure and the formation of cellular recrystallization would affect the creep resistance of the used single-crystal superalloy.

Figure 3 shows the stress rupture life values for specimens subjected to different grit blasting and heat treatments. The

rupture life for specimen SH was 589.2 h. For specimen VH, it was drastically decreased to 230.5 h due to the additional vacuum heat treatment. Compared to specimen VH, the rupture lives of specimens GH1 and GH2 were decreased by about 20% and 70%, respectively, i.e., 186.9 h and 73.4 h, respectively, due to the formation of cellular recrystallization on the surface.

The microstructures away from and near fracture surfaces for different specimens after stress rupture tests are shown in Figs. 4 and 5. Obviously, the γ' precipitates in specimen SH had turned to rafting due to the long creep life. For other specimens, there was no obvious rafting structure and the γ' precipitates coarsened and linked with each other since rupture lives for those specimens were short, as presented in Fig. 4b–d. As shown in Fig. 5a, there were only 2 cracks at surface within 2 mm away from fracture surface for specimen SH with lots of microvoids inside. Similar microstructure feature was presented in specimen VH with only a

Fig. 3 Rupture life values for specimens with different heat treatments and grit blasting

surface microcrack, as presented in Fig. 5b. However, there were much more cracks, 17 and 18 cracks along transverse direction for specimens GH1 and GH2, respectively, shown in Fig. 5c, d. The length of those cracks were also longer than those in specimens SH and VH. In addition, multiple voids were also formed in specimens GH1 and GH2, which were likely to be induced by the propagation of cracks at surface. To further analyze the initiation and propagation

of cracks at surface, EBSD images with orientation information are presented in Fig. 6. It was found that for the specimen without grit blasting, there was almost no misorientation chaos inside the specimen except for few tips of microcracks, as shown in Fig. 6a, while the misorientations in cellular recrystallization at surface were different with that in the matrix, as shown in Fig. 6b, indicating the existence of recrystallization. Furthermore, most of retained cracks located along the grain boundaries inside the cellular recrystallization or between the matrix and cellular colony, illustrating that the cracks were easy to initiate and propagate at grain boundaries.

4 Discussion

With the above results, the average stress rupture life of specimen VH was about 230 h, which was reduced by about 60% of that of specimen SH at 980 °C/250 MPa. The creep rupture property is largely dependent on the volume fraction, morphology and size of γ' [25, 26]. As indicated, the γ' volume fraction decreased to 41% and γ' precipitates coarsened and linked together in specimen VH. Consequently, the rupture life of specimen VH was much shorter than that of

Fig. 4 Microstructures of longitude sections with 4 mm away from fracture surfaces for different specimens after stress rupture tests: **a** specimen SH; **b** specimen VH; **c** specimen GH1; **d** specimen GH2

Fig. 5 Microstructures of longitude sections near fracture surfaces for different specimens after stress rupture tests: **a** specimen SH; **b** specimen VH; **c** specimen GH1; **d** specimen GH2 (the insets are of small magnifications)

Fig. 6 Morphologies of retained cracks and corresponding loading direction inverse pole figure maps for different specimens after stress rupture tests: **a** specimen VH, **b** specimen GH2

specimen SH and there was no time for γ' phase in specimen VH to rafting.

Moreover, the formation of cellular recrystallization led to the further degradation of stress rupture life of alloy SGX3 sheet. The rupture lives of specimens GH1 and GH2 were reduced by 20% and 70%, respectively, compared to

specimen VH without grit blasting. Although those specimens had similar degraded matrix microstructures, specimens GH1 and GH2 had additional cellular recrystallization consisting of irregular γ' , γ and TCP phases at the surface, which did not have the same strengthening effect as the matrix. As a result, cracks were easy to initiate and propagate along grain boundaries in cellular recrystallization during creep, as shown in Figs. 4 and 5. What is more, the thickness of cellular recrystallization in specimen GH2 was larger than that in specimen GH1. The thicker the cellular recrystallization, the smaller the effective loading area. Accordingly, compared to specimen GH1, specimen GH2 was subjected to larger stress during creep and the stress rupture life was shorter.

By assuming that cellular recrystallization does not bear any stress during creep, the effects of grit blasting and subsequent heat treatment on stress rupture property of alloy SGX3 sheet are summarized in Fig. 7. The additional vacuum heat treatment led to the decrease in γ' volume fraction and the coarsening of γ' precipitates, resulting in the weakened γ' strengthening effect, as indicated by the blue dash line in Fig. 7. Apart from the microstructure degradation, the formation of cellular recrystallization in specimens by grit blasting and subsequent heat treatment led to the further degradation of stress rupture property, as indicated by the red dash line in Fig. 7, since the stress the matrix bear was increased in some extent with the decrease in effective loading area. Moreover, the cracks were easy to initiate and

Fig. 7 Effects of grit blasting and subsequent heat treatment on stress rupture property of alloy SGX3 sheet

Fig. 8 Illustration of surface recrystallization on stress rupture life for different Ni-based superalloys

propagate in those areas during creep. It is worthy to be noted that the intersection of the red line and blue line lies on the right of specimen VH, indicating that the cellular recrystallization at surface may bear some stress during loading.

The recrystallization depth influence on stress rupture life for other superalloys and the current alloy is summarized as shown in Fig. 8, and the related information is presented in Tables 3 and 4. Obviously, the normalized stress rupture life shows an almost linear reduction with the increase in the local recrystallization depth and a decrease of ~5% effective loading area generally leads to a drop of 30% to 40% in the creep rupture life for both directionally solidified Ni-based superalloys and other single-crystal superalloys [8, 14, 17, 19, 27]. This seems that the recrystallization depth has similar influence on the stress rupture properties of different alloys, since the surface recrystallization not only reduces the effective loading area but also acts as the vulnerable site for crack initiation and propagation. In addition, it is also noted that the recrystallization type does not seem to have pronounced influence on the stress rupture properties as the figure shows a similar trend for the creep rupture properties with the increasing depth of the equiaxed or cellular recrystallization. Further research combining with FEM simulation should be conducted to analyze the strength of surface recrystallization in different alloys, for providing a guidance on evaluation of safe operation for turbine blades.

5 Conclusions

In the present study, the third-generation Ni-based single-crystal superalloy SXG3 sheet was surface processed by grit blasting of 0.3 MPa/1 min or 0.5 MPa/2 min and subsequently vacuum heat-treated at 1100 °C for 200 h. The effects of grit blasting and heat treatment on stress rupture life at elevated temperature were investigated. The conclusions can be summarized as follows:

- (1) By vacuum heat treatment at 1100 °C for 200 h, γ' volume fraction decreased and γ' precipitates coarsened in alloy SGX3 sheet, resulting in the reduction of stress rupture life at 980 °C/250 MPa by about 60%, compared to the specimen by the standard heat treatment, i.e., 230.5 h versus 589.2 h.

Table 3 Chemical composition (wt%) of alloys in Refs.[8, 14, 17, 19, 27]

References	Ni	Cr	Co	W	Mo	Al	Ti	Ta	C	B	Nb	Hf
[8]	Bal	8.5	5	9.5	—	5.5	2.2	2.8	0.02	—	—	—
[14]	Bal	8	5	5	2	6	2	3	—	—	—	—
[17]	Bal	8–10	9–11	11.5–12.5	—	4.75–5.25	1.75–2.25	—	0.12–0.16	0.01–0.02	0.75–1.25	1–2
[19]	Bal	8.9	10	11.7	—	5	2	1	—	0.014	1	1.8
[27]	Bal	9	10	7	2	5	3.5	4	0.1	0.01	—	—

Table 4 Creep information of alloys in Refs.[8, 14, 17, 19, 27]. The normalized stress rupture life is defined as the ratio of the stress rupture life time of a specimen over that of the same alloy without recrystallization at the same creep condition

References	Alloy	Creep condition	Recrystallization type	Effective loading area (%)	Normalized stress rupture life
[19]	Directionally solidified	980 °C/235 MPa	Not indicated	1	1 ± 0.18
				0.98	0.99 ± 0.06
				0.96	0.84
				0.88	0.44
				0.8	0.3
[27]	Directionally solidified	955 °C/255 MPa	Equiaxed	1	1
				0.95	0.68
[8]	Single crystal	1000 °C/195 MPa	Equiaxed	1	1
				0.92	0.56
				0.91	0.5
				0.88	0.48
				0.87	0.45
				0.86	0.36
[14]	Single crystal	950 °C/240 MPa	Equiaxed	0.85	0.34
				1	1
				0.96	0.71
				0.93	0.5
[17]	Single crystal	1000 °C/195 MPa	Not indicated	0.89	0.365
				1	1
				0.92	0.5
				0.876	0.474
				0.851	0.34

- (2) By grit blasting of 0.3 MPa/1 min or 0.5 MPa/2 min and subsequent heat treatment in vacuum at 1100 °C for 200 h, cellular recrystallization with thickness of 18 µm and 49 µm, respectively, formed at the surface on alloy SXG3 sheet, resulting in the reduction of stress rupture life by about 20% or 70% at 980 °C/250 MPa, i.e., 186.9 h and 73.4 h, respectively, compared to the specimen only by vacuum heat treatment at 1100 °C for 200 h.
- (3) Cellular recrystallization is detrimental to the stress rupture property of alloy SXG3 sheet, since they are vulnerable for microcracks to initiate and reduce the effective loading area.

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