A NEW ANALYTICAL METHOD OF γ/γ' MORPHOLOGY IN SINGLE CRYSTAL Ni-BASE SUPERALLOYS: FOR NEW ORIENTATION OF DAMAGE AND REMAINING LIFE ASSESSMENT.

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Abstract

This paper focused on γ/γ' morphological changes in a single crystal Ni-base superalloy, CMSX-4, induced by monotonic plastic straining and cyclic fatigue loadings. It was shown by a series of experiments that an application of the monotonic straining at R.T. and the subsequent heat treatment at high temperature resulted in a directional coarsening of γ/γ' microstructure, depending on the sign, magnitude and history of strain. A critical strain was found to be needed to build up the microstructural changes. Not only monotonic but also cyclic loadings could induce similar changes in the γ/γ' microstructures. These changes have been summarized in tables. In order to quantify the γ/γ' morphologies, a new method, named by Cluster-Method, was proposed, where the degree of the coarsening and the directional coarsening can be displayed on an original 2-dimensional diagram. This method successfully evaluated various types of γ/γ' microstructures. Furthermore, this method may provide us new potentials for both the stress field detection and the remaining life analysis of superalloy components in service.

Introduction

Ni-based superalloys, especially single crystal superalloys, have been inevitable materials for blades and vanes applications in advanced gas turbines, because of their excellent reliability and mechanical properties [1,2 for example]. In general, single crystal Ni-based superalloys have regularly arrayed composite microstructure consisting of cuboidal γ' precipitates surrounded by narrow channels of γ matrix. During the gas turbine's operation period, on the other hand, the microstructure may change accompanying with severe directional coarsening, so-called rafting, of the initially cuboidal γ' precipitates to the plate-like (normal to the stress axis) or needle-like (parallel to the stress axis) structure by the creep stress due to the centrifugal force [for example 3-6]. Many types of experimental and analytical works showed that the direction and extent of rafting were determined by several factors such as the sign and magnitude of the γ/γ' lattice misfit and of the external applied stress [3-8]. For example, in the case of a negative misfit and tensile creep, plate-type rafting is found, whereas needle-type rafting is found for either positive misfit alloys after tensile or for negative misfit alloys after compressive creep [3-8].

Nowadays, several investigations have indicated that the directional coarsening could occur by many types of loading conditions: monotonic loading associated with plastic strain [9,10] and the high temperature fatigue or thermo-mechanical fatigue loadings [11,12]. A series of relating works strongly indicate the morphologies of the γ/γ' microstructure should be formed and reformed under metallurgical kinetics and interactions between internal and external stresses, temperature and loading histories. Accordingly, once the understanding on the kinetics as well as the influencing factors are achieved, it may be possible to estimate the stress state and the remaining life of superalloy components in service, through a quantitative analysis of γ/γ' microstructures.

Primary objective in this work is to make clear experimentally the effects of the monotonic and cyclic (fatigue) loadings on the geometrical changes of γ/γ' morphologies. The second is to propose and explore a new quantitative method to analyze the effects of thermo-mechanical histories from the γ/γ' morphology.

Experimental Procedure

The material used in this work is a second generation single crystal superalloy, CMSX-4. For this material, the heat treatments were given as follows: the 8-stage solution treatments by 1277°C×2h + 1288°C×2h + 1296°C×3h + 1304°C×3h + 1313°C×2h + 1316°C×2h + 1318°C×2h + 870°C×2h in Ar atmosphere, and then the two stages aging treatments by 1140°C×6h + 870°C×20h in air. As the result of the heat treatments the CMSX-4 revealed very regular γ/γ' microstructure, involving cubic γ' precipitates (see Fig. 1). The γ/γ' lattice misfit of this alloy is considered to be negative value [13,14]. The volume

Fig. 1 Initial microstructure of CMSX-4.

Table I Summary of loading conditions employed.

Specimen number	Plastic strain history		Loading Temp.	Loading Mode
	Step 1	Step 2		
M1	+1%	-	R.T.	Monotonic
M2	-1%	-		
M3	+3%	-		
M4	-3%	-		
M5	+3%	-3%		
M6	-3%	+3%		
M7	+3%	-2%		
M8	-3%	+2%		
No.	Stress range (MPa)	Stress ratio	Hold time	Loading Mode
C1	200	0	-	Stress controlled cyclic loading
C2	400	-1	-	
C3	200	0	600(sec) on tension side	
C4	400	-1	600(sec) on tension & compression side

Fig. 2 Schematic illustration for the monotonic and cyclic straining process. The Specimens M1-M8 were subjected to plastic strain at R.T., while the Specimens C1-C4 were to fatigue or creep-fatigue loading cycles at 1000°C. The post heat treatments at 1080°C were performed to all specimens after pre-straining.

fraction and the size of γ' are 63% and 0.45 μm , respectively. The solid cylindrical specimens, of which gauge section is 3.5mm in diameter and 10mm in length, were machined for the monotonic and cyclic loading tests. All specimens axes lie within 5° from [001] crystallographic orientation.

The monotonic and cyclic loadings were applied to the specimen according to the test program summarized in Table I. For easier understanding, schematic illustrations of each test are shown in Fig. 2. Uni-axial plastic strain was given to the specimen M1-M8,

associated with different straining directions, magnitudes and histories at room temperature. On the other hands, the specimens C1-C4 were exposed to fatigue and creep-fatigue loadings with different waveforms. Fatigue loadings were applied under the stress-controlled conditions for 150h at 1000°C in air. After these monotonic and cyclic loadings, a post-heat treatment was given to all the specimens by 1080°C $\times$ 24h, which is a treatment to reconstruct the γ/γ' microstructure depending on loading histories [15]. After the post-heat treatment, the vertical sections were carefully polished and observed by SEM (JEOL 5400).

Fig. 3 SEM micrographs of the Specimen M1-M8 after the post-heat treatment at 1080°C. Arrows in figures represent the uni-axial loading directions

Results

Morphological Changes Induced by Monotonic Loading

Fig. 3 summarizes the γ/γ' microstructures of the specimens M1-M8 after the post-heat treatment. The following characteristics should be pointed out from Fig. 3.

(1) On the effect of sign of monotonic plastic strain.

The γ/γ' microstructures in the specimen M1 and M3 reveal the directional coarsening normal to the loading axis (see Fig.3(a) and (c)) similar to the plate-type rafted structure by creep. On the other hand, in the specimen M2 and M4, the directional coarsening occurred parallel to the compressive strain axis (see Fig. 3(b) and (d)). The relationship between the sign of strain and coarsening direction is same to that of the rafting phenomenon in negative misfit superalloys [4-6]. These results clearly indicate the γ/γ' microstructure was constructed under an influence of the sign of straining, and they agree with the results in Ref. [9], [10].

(2) On the effect of plastic strain magnitude

Comparing between the specimen M1 and M3 (compare Fig.3(a) and (c)), and between the specimen M2 and M4 (compare Fig. 3(b) and (d)), it is found that the extents of the coarsening in the specimen M3 and M4 are larger than those in the specimen M1 and M2. It means that the extents of the directional coarsening are significantly affected by magnitude of plastic strain and suggests that there is a critical strain to be needed to build up the morphological changes.

(3) On the effect of plastic strain history

In the specimen M5 and M6, the final level of plastic strain reached was the same, but along with different loading histories. It is clear from Fig. 3(e) and (f) that microstructure in the specimen M5 (; Fig. 3(e)) directionally coarsens parallel to the strain axis, while that in the specimen M6 (; Fig. 3(f)) is normal to the axis. According to the microstructure in the specimen M1-M4, the coarsening directions in the specimen M5 and M6 might be determined by the final (second) plastic strain directions. These trends also can be seen in the specimen M7 and M8 (Fig.3(g) and (h)). Note that the coarsening directions in the specimen M7

Fig. 4 Microstructures of the Specimens C1-C4. Arrows denote the uni-axial loading directions.

and M8 are depending on the final plastic strains, although they were smaller than the first applied plastic strains. Thus, the γ/γ' microstructure is reconstructed depending on the strain history, especially on the final one, and little on the level of total plastic strain.

Morphological Changes Induced by the High-Temperature Cyclic Loadings

γ/γ' microstructures in the specimen C1-C4 are shown in Fig.4, where both the as-loaded microstructures and subsequent post-heat treated ones are presented.

At first, let's focus on the as-loaded microstructures (Fig.4(a)-(d)), which were not subjected to the post-heat treatment. The γ/γ' microstructures in the specimen C1 and C3 coarsened directionally normal to the stress axis (Fig. 4(a), (c)), while those in C2 and C4 prolonged to both the normal and parallel direction (see Fig. 4(b) and (d)). It must be attributed to the difference of stress ratio. It is also found in Fig. 4(a)-(d) that the extents of coarsening in the specimen C3 and C4 are apparently greater than those in C1 and C2, which might be caused by the holding time during the cyclic loading in C3 and C4. Thus, the γ/γ' morphologies are reconstructed under an influence of stress ratio and stress waveform. It is also worth noting that the fatigue loading could also produce the rafted structure similar to that by creep.

In the γ/γ' microstructures after the post-heat treatments (Fig.4(e)-(h)), it is found that the post-heat treatment promoted the extent of directional coarsening in the specimen C1 and C2 (see Fig. 4(e) and (f)). In the specimen C3 and C4, on the other hand, great difference can not be seen between the γ/γ' before the heat

treatment and after that (compare Fig. 4(c) with (g) and Fig. 4(d) with (h)). These results indicate that the γ/γ' can not reach the kinetic stable state only by the fatigue loadings without the holding time. The post-heat treatment at 1080°C can change the extent of the coarsening, and then make us possible to see the effect of stress ratio and to judge the existence of the hold time during cyclic loading.

Analysis of γ/γ' Morphology by New Method

The investigation in the Section 3 is not beyond phenomenological discussions, thus it becomes inevitable for the engineering application to analyze the γ/γ' morphologies in a quantitative manner. In this work, two analytical methods are tried. One is a so-called *Intercept Method* and the other is an original method named by *Cluster-Method* in this work.

Intercept Method

Intercept Method is a traditional method that is usually used to express grain sizes. In this work, 30 straight lines parallel and perpendicular to the loading axis on the SEM micrograph were drawn by an interval of 0.5 (μm) as schematically shown in Fig.5. The covered area was 15×15 (μm). Then, the number of the intersections between each line and the γ' phases was counted. The γ/γ' morphologies are quantitatively expressed by the following 3 parameters;

H : the number of the intersections between the γ' phases and the lines parallel to loading axis,

V : the number of the intersections between the γ' phases and the lines perpendicular to loading axis,

Fig.5 Schematic illustration of the *Intercept-Method* showing how to analyze the γ/γ' microstructural morphology.

R : value defined by the following equation (1),

$$R = \log \left(\frac{V/V_0}{H/H_0} \right) \quad (1)$$

here, H_0 and V_0 are the values of H and V in the initial γ/γ' microstructure shown in Fig.1.

Table II shows the values, H , V and R , of some γ/γ' microstructures shown in Fig. 3 and 4. Here, the values of H and V are normalized by H_0 and V_0 , respectively. It is found from Table II that the value of R takes negative value in the plate-type microstructures normal to the loading axis, while it takes the positive in the needle-type microstructures. It is also found that the extent of the directional coarsening is expressed by the absolute value of R ; i.e., relatively large absolute value corresponds to the well-directional coarsened γ/γ' (compare the R value of specimen C1 and C2). These results were not so influenced by line intervals; i.e., the intervals directly changed the values of V and H , but not so affected those of V/V_0 , H/H_0 and R . Thus, the *Intercept Method* can evaluate quantitatively the direction and the extent of coarsening by the sign and the absolute value of R . However, unfortunately, the R value in randomly coarsened γ/γ' microstructures such as Fig.4(d), (h) become almost zero, so it fails to distinguish such a morphological difference from the initial γ/γ' .

Cluster Method

In order to make up for the defect of Intercept-Method, an original method named by *Cluster-Method* was proposed in this work. In this method, the actual microstructures were represented by the cluster consisting of minute square elements as schematically shown in Fig.6. Here, the microstructural morphologies are numerically represented by following two parameters;

- s : the number of the elements which construct the cluster (gray region in Fig.6(b); $s=23$),
- t : the number of the elements which surround the cluster (dashed region in Fig.6(b); $t=29$).

Table II Analytical results of the *Intercept-Method*.

Specimen number	Figure Number	Post heat treatment	V/V_0	H/H_0	$R = \log \left(\frac{V/V_0}{H/H_0} \right)$
Initial	Fig.1	Done	1	1	0
M1	Fig.3(a)	Done	0.558	1.23	-0.345
M2	Fig.3(b)	Done	1.03	0.615	0.225
M3	Fig.3(c)	Done	0.371	0.929	-0.399
M4	Fig.3(d)	Done	0.906	0.315	0.459
M7	Fig.3(g)	Done	1.07	0.456	0.370
M8	Fig.3(h)	Done	0.440	1.14	-0.415
C1	Fig.4(a)	No	0.561	1.11	-0.298
	Fig.4(e)	Done	0.345	1.06	-0.488
C3	Fig.4(c)	No	0.321	1.23	-0.585
	Fig.4(g)	Done	0.297	1.21	-0.610
C4	Fig.4(d)	No	0.684	0.711	-1.66×10^{-2}
	Fig.4(h)	Done	0.644	0.651	-4.69×10^{-3}

The values of s and t represent the size of microstructures (e.g. the extent of randomly coarsening) and the extent of directional coarsening, respectively.

Fig. 7 shows some typical morphology and their values of s and t are plotted in Fig.8, where the horizontal and vertical axis takes s and t , respectively. In Fig.8, the square type morphologies such as Fig.7(a), (b), (c) lie on a solid line ($t = 4\sqrt{s}$), whereas a dashed line ($t = 2s + 2$) correspond to such a morphology in which the elements stand in a line. Then the intermediate morphologies such as Fig. 7(g) and (h) are located between the solid and dashed line. Note that well-coarsened clusters (such as Fig. 7(c) or (h)) are located at the area far apart from the origin, while the extent of directional coarsening is described by the deviation from the solid line (compare Fig. 7(c) and (h)).

Fig. 9 shows the analytical results by the *Cluster-Method* for the microstructures in Fig. 3 and 4. Here, the coordinate system of Fig.8 are transformed such a manner to convert the solid and dashed line to the horizontal and vertical axis, and to put the plot of initial microstructure on the origin. The size of cluster element was set for $0.1 \times 0.1 (\mu\text{m})$, which is sufficiently small to get the stabilized analytical result; i.e., the values of s and t were

Fig.6 Schematic illustration for the *Cluster-Method*. The values of s and t are counted by 23 and 29 for this geometry, respectively.

significantly scattered in case larger clusters, especially larger than initial γ' size ($0.5\mu\text{m}$), were employed. In Fig. 9, it is worth noting that the size of the coarsened γ' phase is represented by the distance from the origin, r , and the level of the directional coarsening corresponds to the angle from the s' axis, ϕ . It is found from Fig. 9 that the well-directionally coarsened structures such as Fig. 3(c) or (d) have larger values of r compared with Fig. 3(a) or (b). In addition, the differences between the initial γ/γ' and the randomly coarsened ones (Fig.4(h)) is obviously evaluated, which could not be distinguished by the *Intercept-Method*. These results suggest that the *Cluster-Method* proposed in this work can lead the quantitative evaluation for the various morphological changes in the γ/γ' microstructure. Through this method, the stress field or loading mode of superalloy component can be detected quantitatively, and they may provide us new potentials for the remaining life assessment of components in service.

Summary

Present work experimentally investigated the γ/γ' morphological changes in a single crystal Ni-base superalloy, CMSX-4, and proposed a new analytical method of γ/γ' morphology. It was found that:

1. An application of plastic strain at R.T. and a subsequent heat treatment at high temperature caused directional coarsening of γ/γ' microstructure, depending on the sign, magnitude and history of plastic strain.
2. There was a critical strain to be needed to build up the marked microstructural changes induced by plastic strain.
3. Cyclic loadings could also induce the γ/γ' morphological changes, which depended on stress ratio, stress wave form and holding time on tension and compression side.
4. The *Intercept-Method*, which is a traditional method to express grain size, were successfully applied to describe the γ/γ' coarsening direction and its extent, but failed to distinguish a differences between the initial γ/γ' morphology

Fig.7 Typical morphologies in the Cluster-Method.

Fig.8 Relationship between s and t for some typical morphologies in Fig.7. Remarks (a)~(f) corresponds to the figures in Fig.7.

Fig.9 The analysis of the γ/γ' morphologies by the *Cluster-Method*. On this 2-dimensional diagram, the degree of the γ' coarsening and that of the γ/γ' directional coarsening are represented by the distance from the origin, r , and the argument angle, ϕ , respectively.

and the randomly coarsened ones.

5. An original method, the *Cluster-Method* proposed in this work, successfully evaluate various types of γ/γ' morphologies in a quantitative manner, which could not be evaluated by the Intercept-Method. This method may provide us new potentials for both the stress field detection and the remaining life assessment of superalloy components in service.

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