

# Quantitative Characterization and Assessment of Served René N5 Ni-based Single Crystal Industrial Gas Turbine Blade

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**Abstract:** A high-pressure 1<sup>st</sup> stage industrial gas turbine (IGT) blade made of René N5 served for nearly 26 000 h was studied to figure out the evolution of microstructure and the degradation degree in different locations of the blade. The morphology and volume fraction of  $\gamma'$  precipitates, the topological inverse degree, and the morphology and composition of carbides were analyzed. Results show that the volume fraction of  $\gamma'$  precipitates and topological inverse degree are applicable to be quantitative microstructural characteristic parameters to characterize the microstructural degradation of the served blade. And the morphology of the  $\gamma'$  precipitates and the precipitation of topologically close-packed (TCP) phase can be employed as the qualitative microstructural characteristic parameters. In terms of these parameters, the microstructural degradation of the served IGT blade can be classified into 3 levels: slight, moderate and severe degradation. These results are expected to lay a solid foundation for the assessment of the degree of the microstructural degradation of single crystal IGT blades and also have practical significance in service safety.

**Key words:** superalloy; René N5; served blade; quantitative characterization

Ni-based single-crystal superalloys are important materials used in industrial gas turbine (IGT) blades, which are usually manufactured by investment cast to produce complex internal cooling passages and shape<sup>[1, 2]</sup>. The blades are exposed to severe environment and subjected to significant rotational and gas bending stresses at extremely high temperatures, as well as severe thermo-mechanical loading cycles as a consequence of normal start-up and shut down operation and unexpected trips<sup>[3, 4]</sup>. It has been found that the microstructure of Ni-based superalloy turbine blades will inevitably degrade after service, including the coarsening, rafting and dissolution of  $\gamma'$  precipitates, the decomposition and transformation of carbides, the formation of topologically close-packed (TCP) phases, etc<sup>[5, 6]</sup>. The evolution of microstructure and the degree of degrada-

tion in different locations of a served turbine blade are various due to various service conditions. The investigation on the microstructure of the IGT blade after service has important practical significance. It is helpful for researchers to figure out the degree of damage in different locations of the blade. And the research can help to provide the theoretical foundation for the assessment of the microstructural degradation of single crystal IGT blades, which is instructive in the field of service safety.

So far, some work has been done on the degradation of microstructure of the IGT blade after service. Chen et al<sup>[7]</sup> investigated the effect of temperature and stress on microstructure of turbine blades made of directionally-solidified superalloy DZ125 after service, and found that the extent of microstructural degradation varies with the service time and

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location of turbine blades. Tong et al.<sup>[8]</sup> assessed the service induced degradation of microstructure and properties of turbine blades made of GH4037 alloy undergone normal and overheating exposures, and the results indicated that overheating exposure can cause the dissolution and re-precipitation of GB precipitates and  $\gamma'$  phase and the reduction of the minimum creep rate and rupture strain. René N5 is a commercial 2<sup>nd</sup> generation single crystal superalloy which has been applied in the advanced IGT blades<sup>[4]</sup>. Little research has been conducted on microstructure evolution of single crystal IGT blades after long time service, to our knowledge. In this study, a René N5 single crystal IGT blade after service was investigated. Several quantitative characteristic parameters such as the volume fraction of  $\gamma'$  precipitate, the topological inverse degree and the chemical component of carbides were studied and analyzed for quantitative characterization and assessment of the microstructure of the served blade.

## 1 Experiment

A high-pressure 1<sup>st</sup> stage blade served for nearly 26 000 h was taken from an IGT. The blade was made of 2<sup>nd</sup> generation single-crystal Ni-based superalloy René N5, and the coating was stripped off. A sample was cut from the blade shank and the misorientation from crystallographic [001] was measured by Laue method to be 4.7°. The measured composition of major alloying elements of the blade is listed in Table 1.

As shown in Fig.1a, transverse sections of the blade, approximately perpendicular to [001] orientation, were cut from sections 1, 2, 3 and 4, which are longitudinally 3, 16,

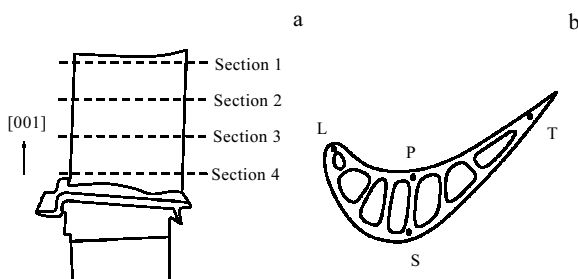
29, 42 mm from the blade tip, respectively. The leading edge, pressure side, suction side and trailing edge of each transverse section were named as L, P, S and T, respectively, as shown in Fig.1b. Then, longitudinal sections parallel to [001] orientation at L, P, S and T were cut from sections 1, 2, 3 and 4. For comparison, a transverse section perpendicular to the [001] orientation of a single-crystal bar after standard heat treatment was prepared for the initial microstructure observation of René N5 alloy.

The metallographic specimens were mechanically polished and then etched with a solution of 1% HF, 33% CH<sub>3</sub>COOH, 33% HNO<sub>3</sub> and 33% H<sub>2</sub>O. The microstructural examinations were performed using an optical microscope (OM) and a ZEISS SUPRA 55 field-emission scanning electron microscope (FE-SEM). The chemical composition of carbides was examined by the energy dispersive X-ray spectroscopy (EDS) equipped in SEM. For transverse microstructure perpendicular to [001] orientation, the volume fraction and average size of  $\gamma'$  precipitates were measured using Adobe Photoshop and Image-Pro Plus software. Five images were used in order to obtain statistically significant results.

It is noticeable that the  $\gamma/\gamma'$  microstructures perpendicular and parallel to [001] orientation in the served blade are different, and the adjacent cubic  $\gamma'$  particles in longitudinal sections parallel to [001] orientation directionally coalesce and transform into flat shape that is called rafts<sup>[9]</sup>. The topological inversion degree ( $R$ ) was used to characterize the topological state of the  $\gamma/\gamma'$  microstructure parallel to [001] orientation<sup>[10-12]</sup>, which is represented by the following equation:

**Table 1 Measured composition of major alloying elements of the IGT blade made of René N5 (wt%)**

Cr	Co	W	Mo	Re	Al	Ta	Hf	C	Ni
6.92	7.32	4.98	1.43	2.80	6.09	6.44	0.16	0.092	Bal.



**Fig.1** Illustration of the locations in the served IGT blade made of René N5 for microstructural investigation: (a) transverse sections 1~4 along the blade longitudinally; (b) locations L, P, S and T in a transverse section of the airfoil

$$R = \frac{\rho}{\rho'} = \frac{N}{N'} \quad (1)$$

where  $\rho$  and  $\rho'$  are area densities of  $\gamma$ - and  $\gamma'$ -terminations;  $N$  and  $N'$  are numbers of  $\gamma$ - and  $\gamma'$ -terminations determined in areas of about 100  $\mu\text{m}^2$  in the current study. Five images were used for statistical results. The value of  $R$  is 0 for the microstructure in alloys after standard heat treatment, as the  $\gamma$  phase is consecutive. The  $R$  value increases with the formation of raft structure, and the critical value of topological inversion occurrence is  $R=1$ . When  $\gamma'$  phase becomes relatively consecutive instead of the  $\gamma$  phase, the value of  $R$  exceeds 1.

## 2 Results and Discussion

### 2.1 Microstructure of René N5 after standard heat treatment

Typical microstructure perpendicular to [001] orientation of René N5 after standard heat treatment is shown in Fig.2. It mainly consists of  $\gamma$  matrix and  $\gamma'$  precipitates. The fine cuboidal  $\gamma'$  precipitates are arranged orderly, and the mean diameter of them is 0.44  $\mu\text{m}$ . The volume fraction of  $\gamma'$  precipitates is 66.2%.

## 2.2 Microstructure in the transverse sections of the served blade

Fig.3 shows the microstructures of  $\gamma/\gamma'$  in transverse sections 1~4. It is clearly indicated that the  $\gamma'$  precipitates are coarsened and coalesced compared with the initial microstructure after standard heat treatment in Fig.2. However, the degree of coarsening and coalescence varies at different locations in the blade, which can be roughly divided into 3 levels. For the first level, most  $\gamma'$  precipitates remain cuboidal such as the suction side of section 2 (Fig.3b<sub>3</sub>); for the second level, the  $\gamma'$  precipitates are connected with each other just like the pressure side of section 1 (Fig.3a<sub>2</sub>); as for the third level, the  $\gamma'$  precipitates are coalesced seriously and needle-like TCP phases precipitate, and typical microstructure refers to the leading edge of section 1 (Fig.3a<sub>1</sub>). This is probably due to the various stresses and temperatures the blade suffered during the long-term service process.

The volume fraction of  $\gamma'$  precipitates in different locations of the served blade is measured and compared in Fig.4. The volume fraction of  $\gamma'$  precipitates in all locations of the

served blade is lower than that after standard heat treatment, implying that besides coarsening and coalescence, the  $\gamma'$  precipitates dissolve to different extents at different locations after the blade has been serving for nearly 26 000 h.

As shown in Fig.4, comparing the  $\gamma'$  volume fraction of the same position in different transverse sections, the largest decrease appears in the blade tip (section 1), indicating that the service temperature is the highest there. Section 3 has the maximum volume fraction of  $\gamma'$  precipitates, implying that the corresponding service temperature is the lowest. The  $\gamma'$  volume fractions of section 2 and section 4 are similar, between section 1 and 3. Consequently, the supposed service temperature at section 2 and 4 is higher than that at section 3 and lower than that at section 1.

Then the  $\gamma'$  volume fractions of different positions in the same transverse sections are compared. In consideration of

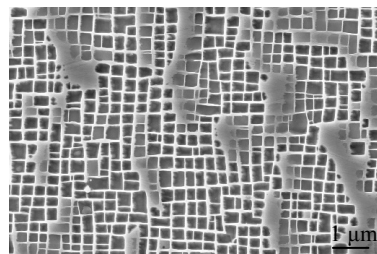


Fig.2 SEM image of the  $\gamma/\gamma'$  microstructure in the dendritic cores of René N5 after standard heat treatment

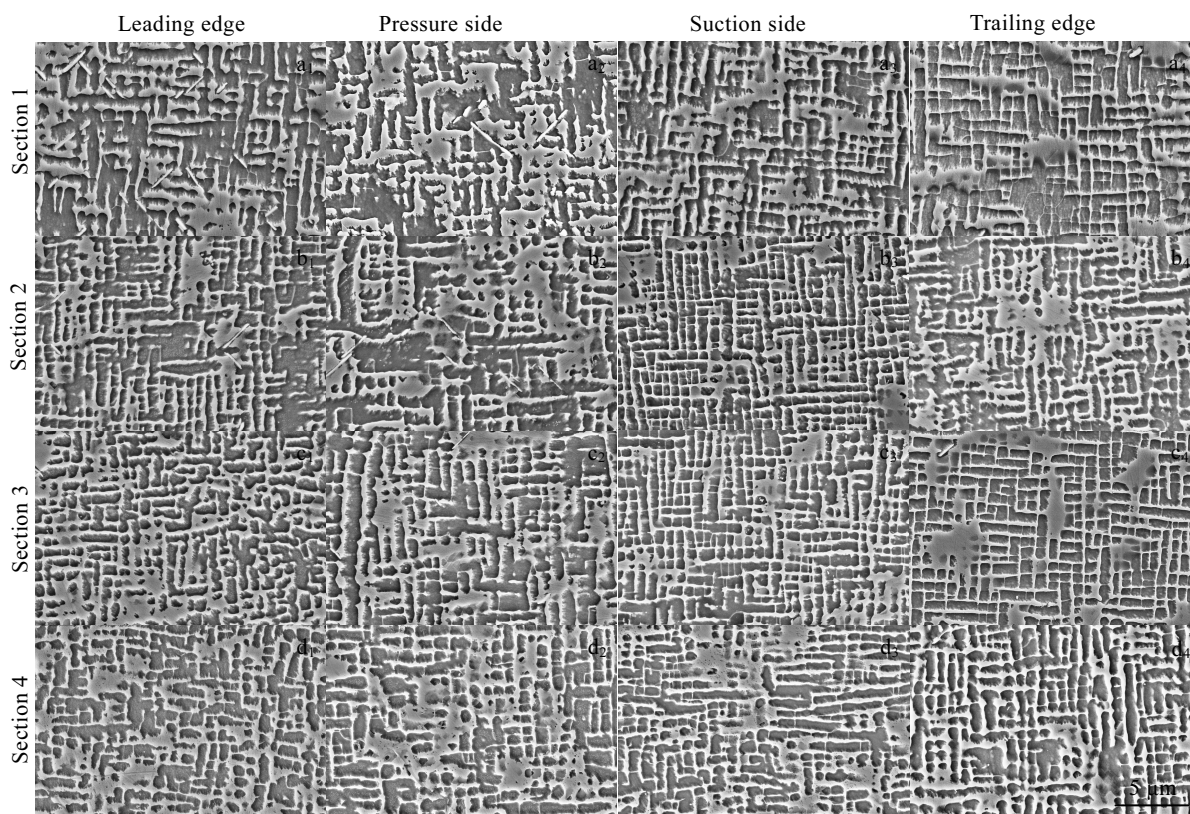


Fig.3 SEM images of  $\gamma/\gamma'$  microstructures perpendicular to [001] orientation at leading edge, pressure side, suction side and trailing edge of transverse sections 1~4 of the IGT blade made of René N5 after service for nearly 26 000 h

four different locations in the same transverse section, according to the data, the  $\gamma'$  volume fraction at pressure side is the lowest except section 3, and the  $\gamma'$  volume fraction at trailing edge is the highest except section 2. Needle-like TCP phase appears in the leading edge of section 1, and the pressure side of section 1 has the minimum volume fraction of  $\gamma'$  precipitates. Therefore, a conclusion can be drawn based on these facts that the highest service temperature is at the leading edge and pressure side of section 1 in the served blade.

### 2.3 Morphology and degradation of carbides

Fig.5 shows the typical morphologies of carbides in the IGT blade served for nearly 26 000 h. Generally, there are two kinds of carbides: isolated blocky and rod-like carbides [13]. EDS analysis reveals that all carbides, no matter what kind of morphology, are typical MC carbides and rich in Ta and Hf. No degradation or transformation of carbides occurs after the long-term service compared with the carbides in the standardly heat-treated René N5 alloy.

### 2.4 Microstructure in longitudinal sections of the served blade

The microstructures of  $\gamma/\gamma'$  in longitudinal sections at the locations of leading edge, pressure side, suction side and trailing edge in sections 1~4 are shown in Fig.6. It shows that the morphology of the  $\gamma'$  precipitates is changed, including coarsening, dissolution and rafting. The  $\gamma'$  precipitates form “rafts” at certain locations. The topological inverse degree ( $R$ ) is measured and the statistics data are shown in Fig.7. The values of  $R$  at all locations are larger than 0, and several  $R$  values exceed 1. The largest  $R$  value appears at the pressure side of section 1, and the  $\gamma'$  precipitates rafted and the rafts connected with each other partially become the matrix instead of  $\gamma$ , and TCP phase forms simultaneously (Fig.6a<sub>2</sub>).

As shown in Fig.7, the values of topological inverse degree  $R$  at the pressure side in all 4 sections are the largest among the 4 locations of L, P, S and T. This implies that the microstructure at pressure side is the most seriously degraded

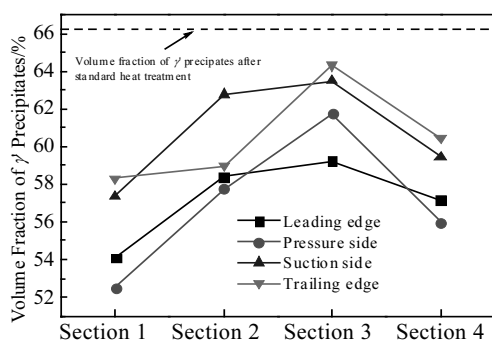


Fig.4 Volume fraction of  $\gamma'$  precipitates at different locations of the IGT blade made of René N5 after service for nearly 26 000 h

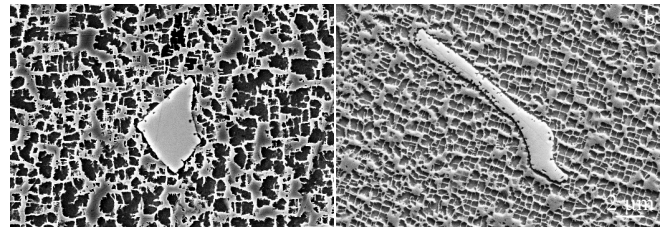


Fig.5 Typical morphologies of carbides in the IGT blades made of René N5 after service for nearly 26 000 h: (a) blocky carbides and (b) rod-like carbides

and corresponding service temperature is the highest.  $\gamma'$  precipitates at the suction side of sections 3 and 4 and the trailing edge of sections 2 to 4 remain almost independent although some of them are elongated and connected slightly. The values of  $R$  at these locations are no more than 0.2, and the service temperatures are relatively low there. Among the 4 sections,  $R$  values at pressure side, leading edge, suction side, trailing edge of section 1 are all larger than those at the same locations of other 3 sections. Therefore, it is suggested that the microstructure is the most seriously degraded in section 1 of the served blade.

It is known that  $\gamma/\gamma'$  lattice misfit is the driving force for the rafting behavior during creep deformation. Ni-based single crystal superalloys that raft perpendicular to the applied tensile stress have a negative lattice misfit. And alloys with a positive lattice misfit raft parallel to the applied tensile stress. If the lattice misfit is eliminated, the rafting structure should not appear [14]. It should be noticed that turbine blades in service are subjected to complex in-homogenous stress, including tensile stress, creep stress, fatigue stress as well as thermal stress, etc. Also, the stress conditions in different locations of the blade are various. Miura et al [15] estimated the stress distribution of a 2<sup>nd</sup> high pressure turbine blade based on the raft structure direction, and the microstructural evidence indicated that the uni-axial tensile stress perpendicular to the blade surface and/or the multi-axial compressive stress parallel to the blade surface are acted on the blade in service [15]. In the current research, the rafting directions at different locations of the served blade are not the same, some are perpendicular to [001] (Fig.6d<sub>2</sub>), while others are parallel to [001] (Fig.6a<sub>3</sub>), and also there are other raft conditions (Fig.6c<sub>2</sub>). These experimental results well verify the complex stress distribution of the blade in service.

### 2.5 Microstructural degradation characteristic parameters

The service condition inferred from microstructural degradation based on the results of  $R$  values coincides well with that based on  $\gamma'$  volume fraction. The locations with low  $\gamma'$  volume fraction correspond to large  $R$  and the microstructure degraded relatively seriously, and the service

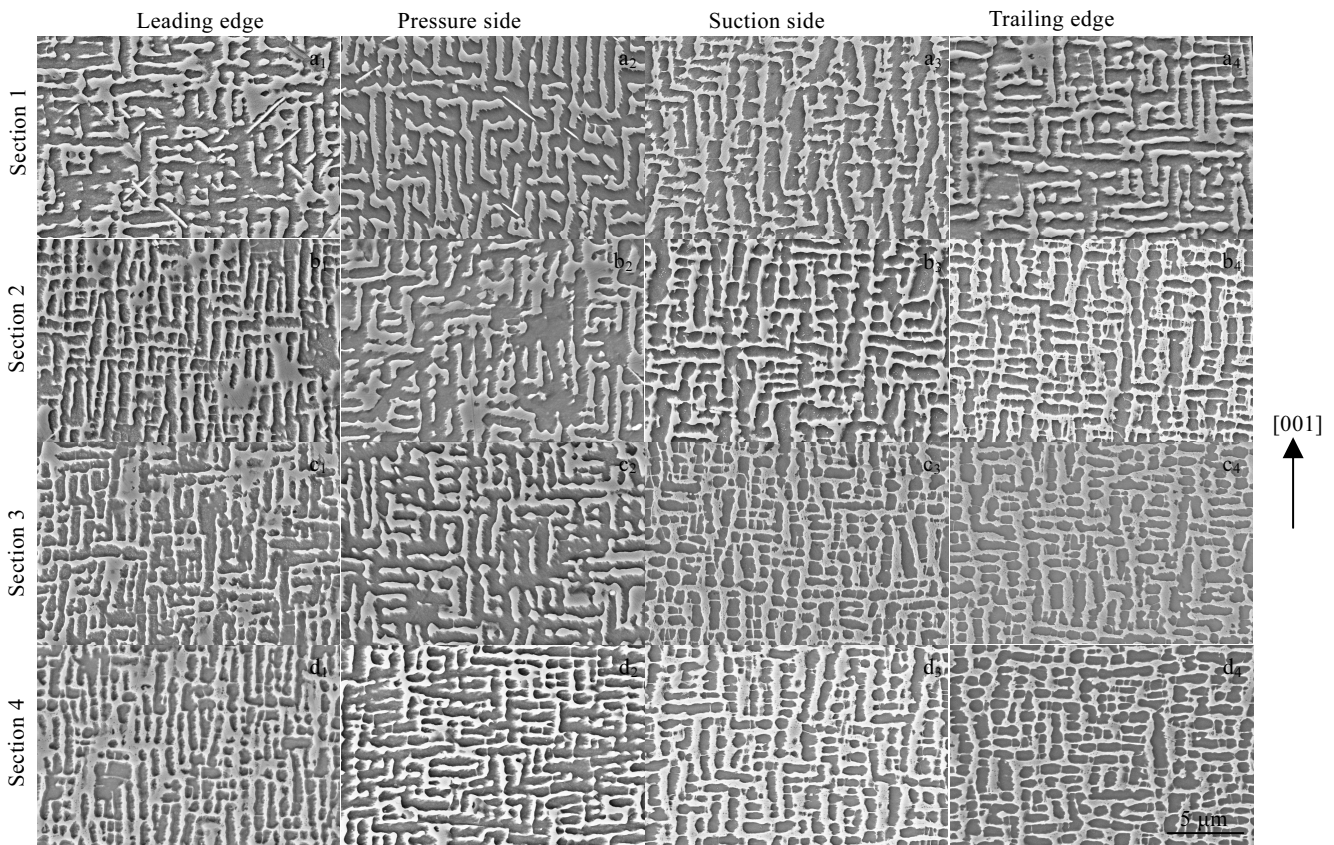


Fig.6 SEM images of longitudinal  $\gamma/\gamma'$  microstructures parallel to [001] orientation at the leading edge, pressure side, suction side and trailing edge in sections 1~4 in IGT blade made of René N5 served for nearly 26 000 h

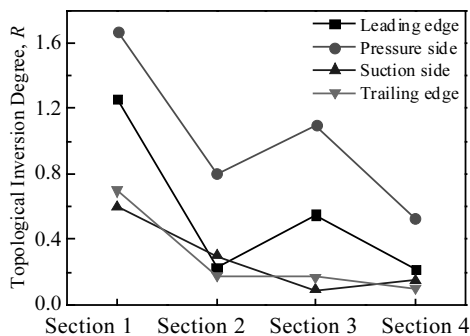


Fig.7 Topological inversion degree ( $R$ ) at different locations of the IGT blade made of René N5 served for nearly 26 000 h.

temperature is deduced to be higher. Based on the investigation above, it is suggested that the volume fraction of  $\gamma'$  precipitates and topological inverse degree are applicable quantitative parameters to characterize the degree of microstructural degradation in the served blade. The cubical  $\gamma'$  is gradually coarsened and coalesced, and TCP phases precipitate when microstructure is degraded seriously. Therefore, the morphology of the  $\gamma'$  precipitates and the formation of TCP phase can be qualitative microstructural char-

acteristic parameters. The microstructure degradation in the served IGT blade can be classified into 3 levels according to the characteristic parameters mentioned above as follows:

(1) Slightly degraded: most  $\gamma'$  precipitates remain cuboidal, the volume fraction of  $\gamma'$  is high (over 60%), the topological inverse degree  $R$  is low (below 0.2), and the microstructure at the suction side of section 3 (Fig.3c<sub>3</sub> and Fig.6c<sub>3</sub>) is the typical microstructure.

(2) Moderately degraded: the  $\gamma'$  precipitates connect with each other, the volume fraction of  $\gamma'$  is 56%~60%, and the topological inverse degree  $R$  is in the range of 0.2~1.0, reference to the leading edge of section 2 (Fig.3b<sub>1</sub> and Fig.6b<sub>1</sub>).

(3) Seriously degraded: the  $\gamma'$  precipitates connect with each other seriously, TCP phases form, the volume fraction of  $\gamma'$  is low (below 56%), the topological inverse degree  $R$  is higher than 1, and the typical microstructure is referenced to that at the leading edge of section 1 (Fig.3a<sub>1</sub> and Fig.6a<sub>1</sub>).

### 3 Conclusions

1) The  $\gamma'$  precipitates dissolve and the volume fraction of  $\gamma'$  precipitates decreases to different extents in the long-term served IGT blade. The volume fraction of  $\gamma'$  precipitates is

an applicable quantitative parameter to characterize the degree of microstructural degradation in the served blade.

2) The  $\gamma'$  precipitates are coarsened and rafted in longitudinal sections to different extents. The topological inverse degree is another applicable quantitative parameter to characterize the degree of degradation of the microstructure in the served blade.

3) The  $\gamma/\gamma'$  microstructure in the dendrite cores is degraded to different degrees. The morphology of the  $\gamma'$  precipitates and the formation of TCP phase can be qualitative microstructural characteristic parameters of the microstructure degradation in served René N5 IGT blades.

4) Based on the quantitative and qualitative microstructural degradation characteristic parameters, the microstructure degradation in the served IGT blade can be classified into 3 degrees: slightly, moderately and seriously degraded microstructure.

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## 服役后的 René N5 镍基单晶高温合金燃机涡轮叶片定量表征与评估

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**摘要:** 对服役近 26 000 h 的 René N5 单晶高温合金燃机高压一级涡轮叶片进行解剖分析, 研究了叶片不同部位的组织演化和损伤程度。分析了  $\gamma'$  相的形貌和体积分数, 拓扑反转程度, 碳化物的形貌和成分。结果表明,  $\gamma'$  相的体积分数和拓扑反转程度可以作为服役叶片显微组织损伤的定量表征参数,  $\gamma'$  相的形貌和拓扑密排相(TCP)的析出可以作为半定量的表征参数。根据以上参数, 服役后的燃机涡轮叶片的组织损伤程度可以分为 3 个等级: 轻度损伤, 中度损伤和重度损伤。本研究结果为燃机用单晶涡轮叶片的组织损伤评价奠定了基础, 并对叶片服役安全具有重要意义。

**关键词:** 高温合金; René N5; 服役叶片; 定量表征

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