Observations of Microstructure Changes in Used Turbine Blade by IP-XRD SEM and TEM

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ABSTRUCT

In this study, microstructure changes of the first stage of turbine blade which had been operated for 30,000 hours or more in the gas turbine were investigated by means of the imaging plate X-ray diffraction method (IPXRD), scanning electron microscope (SEM) and transmission electron microscope (TEM). According to IPXRD data, two dimensional diffraction image indicates the information of both crystallography and microstructure morphology in the reciprocal lattice space. Debye-Scherrer ring pattern was observed from the root position of the cooled turbine blade, it was agreed with the fine grain polycrystalline microstructures observed by SEM. The pattern observed from the hottest position of the turbine blade was spotty. In addition, the SEM observation revealed that fine γ' (Ni_3Al) precipitates coalesced into each other to become large one. Diffraction profile from (111) plane of γ' (Ni_3Al) changes from ring pattern to intense spots, depending on the position of the turbine blade. TEM observations revealed the dislocation activities in γ/γ'interfaces.

Introduction

Lifetime evaluation of the first stage turbine blade in the industrial gas turbine engine is one of the most important issues among the gas turbine technologies. Recently, both the fundamental understanding of creep behaviors of Ni base superalloy and the accumulative experimental data have led to some lifetime prediction of these materials. Creep behaviors of Ni based superalloy single crystal have been investigated extensively not only in the macroscopic view point but also in the microscopic observations(1, 2). Pollock and Argon(3) studied the creep resistance of CMSX-3 in detail, observing three dimensional dislocation arrangements in γ and γ' phases by the transmission electron microscope (TEM). Based on the TEM observations, the contributions of dislocation network on creep stresses have been quantified. It is known that cuboidal γ' precipitates change their morphology, namely rafting(4,5). Several mechanisms have been proposed about the rafting of Ni base superalloys (6,7). However, discussion of the rafting mechanism has continued and not reached conclusion. Apparently, microstructure changes during the creep deformation strongly correlate with the creep conditions such as temperatures and stress levels. In order to evaluate practical creep conditions and the material durability, direct observations of inside microstructure changes from the variety of positions in the turbine blade were the most appropriate method. However, that method is a destructive method, therefore turbine itself is not reusable. It is important to develop a nondestructive method as a method of detecting a microstructure change.

In the industrial gas turbine, the stresses and temperatures are high and more steady conditions, life limiting parameter is likely to be creep elongation during the high temperature operation(8). Apparently, creep behaviors depend on the stress conditions and grain microstructure. Using the synchrotron energy source, γ' phase in the Ni based superalloy have been investigated by X-ray locking method quantitatively(9). Surface damage in superalloys has been investigated by electron back scattered diffraction (EBSD) and synchrotron x-ray diffraction techniques.(10) However, these methods are not suitable to apply with an actual turbine blade in the power plant because of the limit of apparatus. Therefore, the developments of the non-destructive evaluation method to examine the operated turbine blade are necessary. In this study, the microstructure changes of the first stage of the turbine blade which had been operated in the gas turbine for 30,000 hours or more were investigated by means of the imaging plate X-ray diffraction method (IPXRD), the scanning electron microscope (SEM) and TEM.

Experimental Procedure

Specimen preparation

The first stage turbine blade in the gas turbine power generator has been supplied by Toshiba Ltd... This blade had been operated

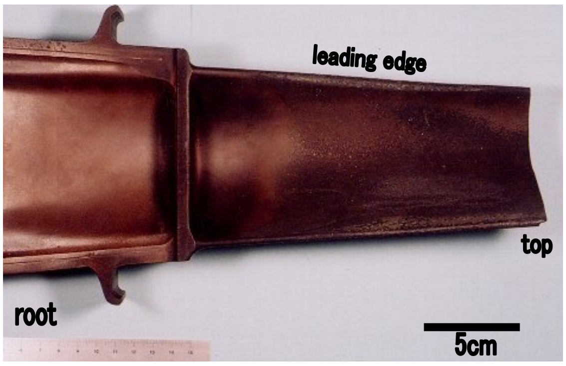

Fig. 1 First stage turbine blade

for 30,000 hours in an actual power plant. Since gas turbine was not the newest generation type, it was made from cast Ni base polycrystalline superalloy . Cooling holes whose diameter was 3mm were penetrated through from the root part to the top of the blade. Figure 1 shows a photograph of the used turbine blade. Center position of leading edge shows surface damage. Table 1 lists the alloy compositions of Ni based superalloy.

Table 1 alloy compositions (mass%)

Ni	Cr	Co	Mo	W	Ti	Al	Ta	C
60.0	14.0	9.5	1.5	3.8	4.9	3.0	2.8	0.1

The hottest position of the turbine blade was examined by surface color and roughness. A 20x20mm^2 block which was 48.5mm distant from the top was cut by the electro discharge machine (EDM). For X-ray diffraction analysis, the block was further cut to approximately cube shape (5x5x5mm^3). Surfaces of cube were polished by emery papers and alumina powder. Polished specimens were etched using conventional etching solution consisting of hydrofluoric acid, nitric acid and distilled water.

IPXRD technique

The X-ray diffraction technique is a nondestructive testing technique that has been established to sophisticated level. Recently, profile of the diffraction intensity is analyzed more quantitatively on the two dimensional space, utilizing the imaging plate (IP) as a recording device. Figure 2 shows the IP and specimen stage of IPXRD equipment. Incident X-ray beam was positioned accurately by the goniometer stage using CCD camera. Focused position of CCD image was coincided with X-ray optics. Collimated beam diameter was 0.3mm that was small enough to examine the local area of the turbine blade. Table 2 list the experimental conditions of XRD measurements.

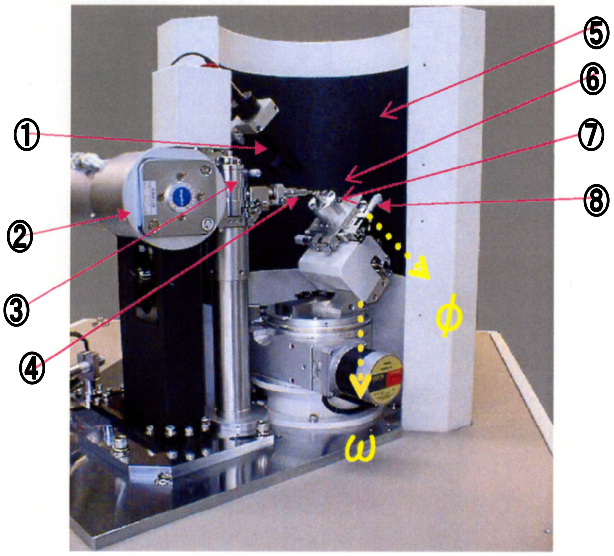

Fig. 2 IPXRD equipment; ①CCD camera, ②X-ray source, ③monochromator, ④collimator, ⑤imaging plate, ⑥specimen holder, ⑦spacer, ⑧specimen stage

Table. 2 Experimental conditions of IPXRD

X-ray	Target	Cu K α
	Voltage /kW	40
	Current /mA	30
	Exposure time /min	120
Goniometer	Camera length /mm	127.4
	2θ	-47 to 163
	Collimator /mm	0.3
Specimen	ω axis	20° fixed
	ϕ axis	0° fixed

Sample preparation for SEM TEM

After the X-ray diffraction measurements, specimens observed by SEM. For TEM observations, thin foil discs are prepared from conventional twin jet electro-polishing technique, using electrolyte of perchloric acid 15% and methanol 85%. TEM observations were carried out using JEM2000FX electron microscope operated at 200keV.

Results and Discussion

IPXRD measurements

Although X-ray diffraction technique is a nondestructive method, a small block cut from the first stage turbine blade was examined, because of limitation of specimen stage in the X-ray instrument. Figure 3 shows the cross section of the block. Surface coating layer whose thickness was 2mm was observed.

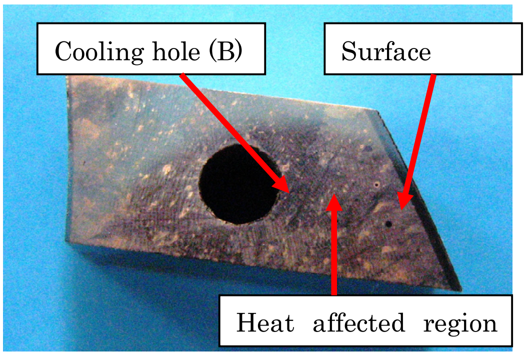

Fig. 3 Cross-section of specimen

At the root position of the turbine blade, the effect of heat was reduced comparing with other positions of the blade, since it was cooled by air flow and attached to the turbine disc, during operation. Figure 4a demonstrates the two dimensional (2D) diffraction image from the root position of the blade. Continuous Debye-Scherrer ring pattern was observed. From the beam center, the first inner ring is indicating diffraction of Ni (111) planes, the second inner ring is Ni(200) planes, the third inner ring is Ni(220) planes, respectively. This diffraction image is elucidating that this observed area (0.3mm diameter) is consisting of randomly oriented fine polycrystalline aggregates. Intensity distribution of Ni(200) plane along ring is slightly heterogeneous due to the texture.

Figure 4b shows the SEM photograph from the same area of

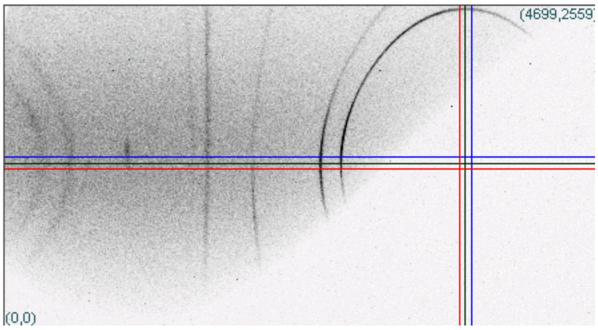

Fig. 4a IPXRD image from root position of cooled blade

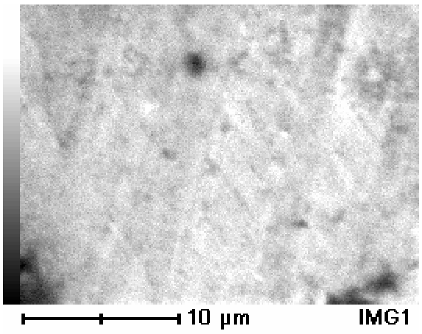

Fig. 4b SEM image of root position of cooled blade

Fig.4a. Average grain size was 30 μ m and γ' precipitate size was 0.5 μ m, respectively.

Figure 5a shows the 2D diffraction image at the vicinity of the cooling hole in the block as seen in Fig.3 (B). No ring pattern was observed. Diffused spot image was recorded at the 2 θ angle of γ' (111) plane. It is suggesting that γ' precipitates are almost aligned toward the same direction but their size is very small. Figure 5b shows the SEM image of the same position of Fig.5a. Clearly, cuboidal γ' precipitates having almost the same crystal orientation are observed. Since average grain size of this position was 0.3mm, diffraction spot from γ phase seems to be hardly detectable. Analysis of the diffraction pattern in the reciprocal lattice space coincides with the SEM observations in the real space, each other . Other diffraction such asγ' (200) and γ'(220) planes are so weak that they are invisible.

Figure 6a demonstrates the 2D diffraction image from the hottest region of the current blade (Fig.3 A). More clear spots were observed at γ'(111) plane. Strong intensity are suggesting that larger size of γ' precipitate than those of Fig.5a. Furthermore, observed three spots in the γ'(111) plane are indicating that γ' precipitate is not aligned to one direction but is scattering. Figure 6b shows SEM image of the same hottest region of the blade. More sphere like γ' precipitates were observed because γ' precipitates were coalesced by creep deformation. If the turbine blade operates long period, γ' precipitates in this region would become more coarsened, therefore, durability of this material would be reduced drastically. These observations demonstrate the possibility in the detection technique of the microstructure changes by means of IPXRD.

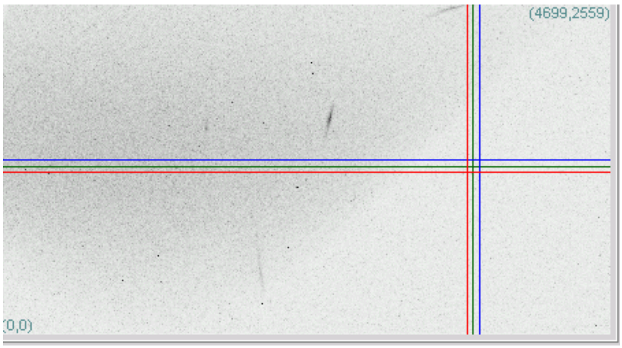

Fig. 5a IPXRD image at the vicinity of cooling whole (region B)

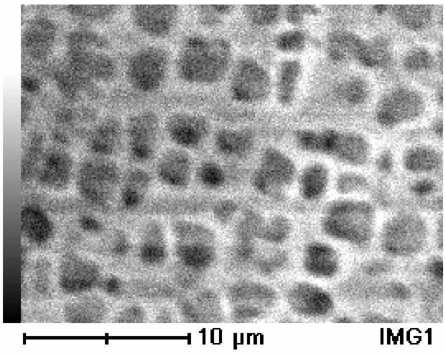

Fig. 5b SEM image at the vicinity of cooling whole (region B)

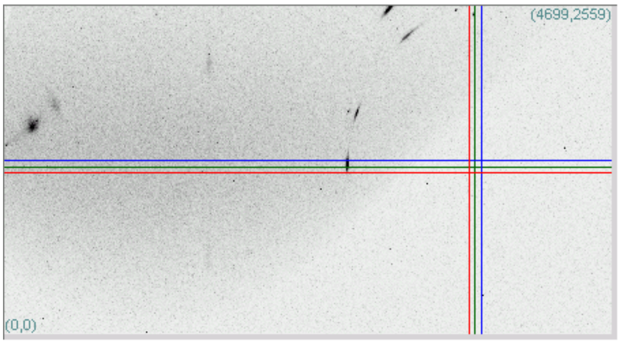

Fig. 6a IPXRD image from the hottest area of blade

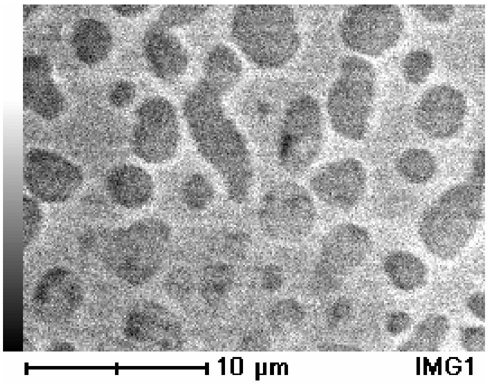

Fig. 6b SEM image of the microstructure in the hottest area of blade

Profile analysis

In order to quantify the morphology of the turbine blade, diffraction from γ'(111) plane was selected for the further analysis. Ring pattern (Fig.4a) , diffused spot (Fig.5a) and intense spot (Fig.6a) are implying the information of crystal morphology in the reciprocal lattice space. If a crystal is consisting of the perfect single crystal with isotropic atomic arrangement, sphere shape diffraction spots would appear in the reciprocal lattice space. Therefore, the cross section of the diffraction spot should be a circle when Laue's diffraction condition is consists $^{(11)}$. If an ellipse is observed in the reciprocal lattice space, it is demonstrating that crystal contains a kind of the anisotropy, such as existing of polygonization , imperfection, elongated grain and so on. Figure 7 shows the three dimensional (3D)profile of (111) ring pattern (see Fig.5a). Figure 8 shows the 3D profile of (111) spot at the vicinity of cooling hole (see Fig.6a). Figure 9 shows the 3D profile of (111) spot from the hottest region of the blade (see Fig.7a).

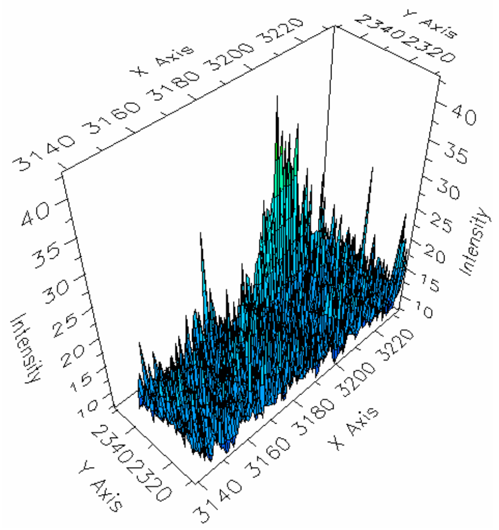

Fig.7 3D diffraction profile of Fig.5a ring pattern

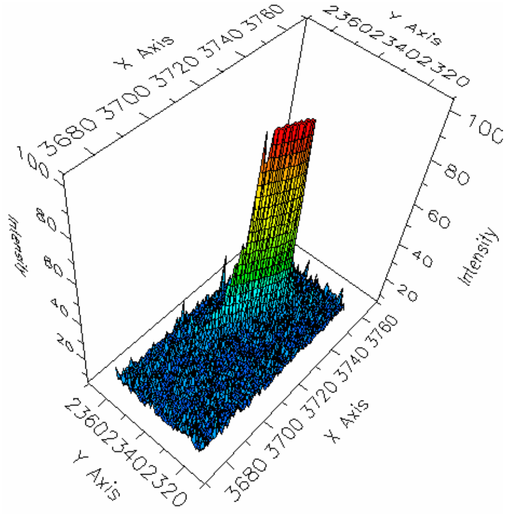

Fig. 9 3D diffraction profile of Fig.7a (111) intense spot

Although many structural parameters affect on the shape of diffraction spot, both **a** value (the width of radial direction of spot) and **c** value (the width of radius direction) were measured at the half value of intensity peaks of Fig.7-9, respectively. Then the aspect ratios **a/c** were calculated. Table 3 lists **a**, **c** and **a/c** values from these IPXRD measurements.

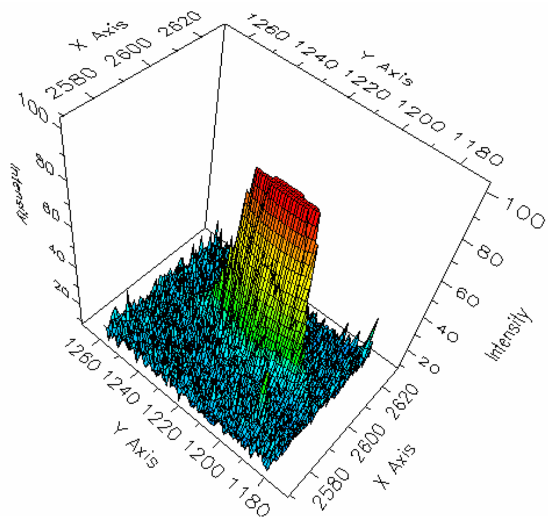

Fig. 8 3D diffraction profile of Fig.6a (111) diffused spot

Table 3 Quantitative data

place	a	c	a/c
Root (A)	316	16.4	19.3
Near hole (B)	162	19.5	8.33
Hottest region(C)	142	23.1	6.15

According to Table 3, an agreement between the aspect ratio **a/c** and tendency of the microstructure changes from SEM observations are evident. Consequently, the aspect ratios of diffraction from γ' precipitates would become the first estimation of the microstructure changes for the nondestructive evaluation.

TEM observations

In order to examine the microstructure changes of the turbine blade in detail, TEM observations were carried out. Figure 10 shows the γ matrix and γ' precipitate of the root position of the blade, where it seems as-cast microstructure would be maintained. In Fig.10, (110) ordered diffraction spot appeared in the electron diffraction pattern. It is indicating that γ' precipitates have an ordered atomic arrangement.

Figure 11 shows the TEM image of γ matrix and sphere γ' precipitate. In the γ matrix, dislocation network was observed. This observation was found out the macroscopic creep deformation was caused by the dislocation generations and propagations at high temperatures during the gas turbine operation. High dislocation density in γ phase could strongly contribute to the microstructure changes by enhancing the atomic diffusion and vacancy generation $^{(12)}$. Detail analysis would be necessarily to clarify the microstructure changes in Ni base superalloy in conjunction with the rafting mechanism .

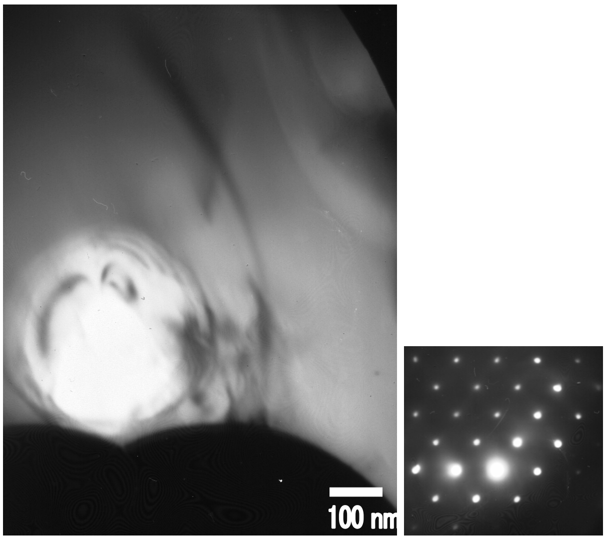

Fig. 10 TEM observation from the root position of the blade. Bright field image (γ' precipitate) and diffraction Pattern

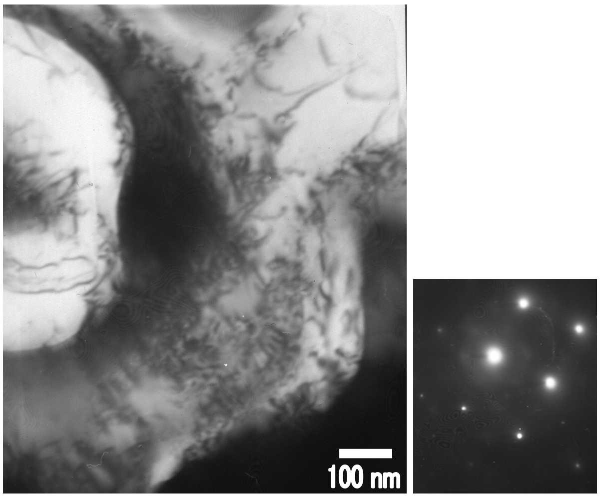

Fig. 11 TEM observation from the hottest position of the blade. Bright field image of dislocation network in γ phase and diffraction pattern.

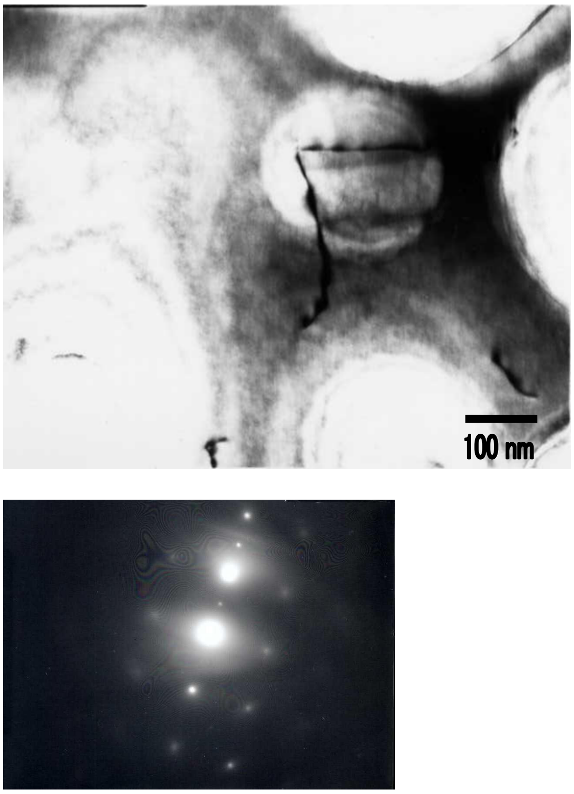

Fig. 12 TEM observation from the hottest portion of blade. Bright field image of dislocation at the γ/γ' interface and diffraction pattern.

Figure 12 shows the bright field image and the diffraction pattern from the thin foil specimen of relatively hot region of the blade. Dislocation is observed at γ/γ'interface. Lattice misfit angle between γ and γ' phases is one of the most important parameter to determine the high temperature strength of superalloy. γ/γ'interface become a strong obstacle to dislocation movements. Anchored dislocation was frequently observed in this used turbine blade.

Debye-Scherrer ring pattern was observed from the root position of the cooled turbine blade having the fine polycrystalline microstructures. Spot pattern was observed from the hottest region of the turbine blade since recrystallization of γ grains took place and grew to large grains. Accordingly, diffraction profile from (111) plane of γ'(Ni_3Al) precipitates changes from the ring pattern to spot, depending on the distance from the cooled root to hottest region of blade.

For quantitative analysis, it was assumed that the cross-section of diffraction spot was as an ellipse and a/c ratio was measured. Measured a/c ratio was 6.1 at the highest temperature region in the blade. Large spherical γ' grains coalesced into each other since recovery and recrystalization took place under the high stress conditions. Dislocation network was observed in the γ phase and γ/γ' interface. These observations were revealed that creep deformation was enhanced by the dislocation activities. On the other hand, a/c was 8.3 at the vicinity of cooling hole where 5mm distant from hottest point. The array of cubic γ' precipitates and the γ grain growth were observed. Measured a/c ratio was 19.2 at the root position of the blade where microstructure supposed to be unchanged as maintaining the original morphology. TEM observations showed that fine grain microstructure of γ' and γ phases and a very few dislocations were observed in the thin foil specimen prepared from the root of blade. Using IPXRD, two dimensional diffracted beam profiles were analyzed to the three dimensional digital figures. Based on the detail analyses of three dimensional diffraction profiles, the microstructure changes of Ni base superalloy can be understood more quantitatively. In order to apply IPXRD method as the practical non destructive testing method, further improvements of the IPXRD instrument would be essential.

Conclusions

In this study, the microstructure changes of the first stage in the turbine blade which had been operated for 30,000 hours or more, were investigated by means of IPXRD, SEM and TEM. According to IPXRD data, two dimensional diffraction images indicate the information of both crystallography and the microstructure morphology in the reciprocal lattice space.

1 Spot pattern was observed from the hottest area of the turbine blade in which fine grains were grown to large grains. Measured a/c ratio was 6.1 at the hottest region in the blade. Large spherical γ' grains coalesced into each other since recovery and recrystalization took place under the high stress conditions at the elevated temperatures. Dislocation network was observed in γ phase and γ/γ' interface which is suggesting that creep deformation was enhanced by the dislocation activities.

2 Diffused spot was observed at the vicinity of cooling hole where 5mm distant from the hottest point. a/c was 8.3. Microstructure shows array of cubic γ' and moderate grain growth.

3 Debye-Scherrer ring pattern was observed from the root position of the turbine blade. Measured a/c ratio was19.2 at the root position of blade where microstructure supposed to be unchanged. TEM observations revealed that a very few dislocations were observed in both γ' and γ phases.

Based on the detail analyses of three dimensional diffraction profiles, the microstructure changes of Ni based superalloy can be understood more quantitatively. In order to apply IPXRD method as the practical non destructive testing method, improvements of the X-ray instrument as well as developments of more sophisticated analytical methods are essential. This paper suggests a possibility of the life time evaluation of the turbine blade by means of IPXRD.

Acknowledgements

Authors would like to thank to Dr. Yoshioka and Mr. Saito who provide us a valuable turbine blade.

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