Rotating bending fatigue property of the Ni₃Al-based single crystal superalloy IC6SX at 900°C

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Abstract: The high cycle fatigue behavior of a Ni₃Al base single crystal superalloy IC6SX has been investigated at 900°C in this work. The specimens used for the fatigue tests were prepared by screw selection crystal method in a directional solidification furnace. The rotating bending fatigue tests were carried out at 900°C in air, the stress ratio of R (σmax/σmin) was -1, and the rotating speed of the fatigue tests was 6500r/min (108Hz). The stress-fatigue cycle life (S-Nf) curve was obtained based on the fatigue tests, and the fracture surfaces were examined using scanning electron microscopy (SEM). It has been found that the median fatigue strength is 457.5MPa and the safety fatigue strength is 413.93MPa. And the fatigue fracture was composed of three different characteristic regions.

Keywords: Ni₃Al; Single crystal superalloy; Rotating bending fatigue; Intermetallics

1. Introduction
As we know that the single crystal superalloys were used widely for the turbine blades and guide vanes which were assembled into the advanced aircraft engine. Single crystal superalloys are critical to the development of the advanced aircraft engine. Because Ni₃Al base single crystal superalloy has higher temperature, excellent resistance to creep, fatigue resistance, high temperature strength [1], lower density and cost. So that the Ni₃Al based single crystal alloys could meet the needs of the aircraft engines with higher thrust-weight ratio. Generally speaking, the high pressure turbine blades bear both the higher aerodynamic force and greater thermal stress due to the sharp air environment temperature changing during their operation. During the processing of the long-term service, the fatigue damage of the turbine blades often happens due to the sudden change of both the temperature and load [2-7]. Previous research [8] showed that the turbine blades bear not only the bending force but also the distorting force when the aerodynamic load is applied on them. And the stress situation of blades is similar with that in the rotating bending fatigue test. Therefore, in this work, the rotating bending fatigue test of a Ni₃Al base single crystal superalloy IC6SX was carried out to analyze the loading condition of the turbine vanes.

2. Experimental Materials and Methods
The Ni$_3$Al base single crystal superalloy IC6SX bars used in this work with the nominal composition of Ni-(7.4~8.0)Al-(13.5~14.3)Mo-(0.02~0.03)B(wt%) were prepared in the DZG-0.025 directional solidification furnace. And the crystal growth method used for the bars is the screw selection crystal method. The crystal orientations were determined with Lauemethod by X-ray back scattering. And the bars which orientation deviating from [001] orientation within 15 degrees were used for fatigue tests in this work. Figure 1 described the dimension of the specimens for the rotating bending fatigue tests. The fatigue tests were carried out on a PV-6500 type cantilever bending fatigue testing machine with the rotary speed of 6500r/min. According to the standard of HB5153-96 [9], the rotating bending fatigue properties of alloys at 900°C were measured and the data were analyzed using the material fatigue statistical analysis method in the standards manual of HB/Z112-86 [10].

![Figure 1. Dimensions (in mm) of specimen for the rotating bending fatigue test.](image)

3. Results and Discussion

3.1. Rotating bending fatigue properties of the IC6SX alloy under 900°C

The rotating bending fatigue properties of the IC6SX alloys were analyzed by the up-and-down method as mentioned in the standard of HB5153-96 [9]. And the up-and-down curve of the rotating bending fatigue for IC6SX alloys at 900°C was shown in figure 2.

![Figure 2. Up-and-down curve of rotating bending fatigue properties for IC6SX alloys at 900°C.](image)

The rotating bending fatigue data for the single crystal alloy IC6SX are given in table 1. It can be found that there were 12 samples were tested. With the change of test stress from 435MPa to 480MPa, there were 6 specimens with their fatigue lives more than 10$^7$ cycles without fracture. However,
another 6 samples were all failure not reaching the appointed life 107 cycles. In addition, the shortest fatigue life was only $2.145 \times 10^6$ cycles. The S-N curve of the IC6SX alloy at 900°C is illustrated in figure 3. As can be seen in figure 3, the fatigue life of IC6SX alloy at 900°C increased as the stress decreased.

Table 1. Data of rotating bending fatigue for single crystal alloys IC6SX at 900°C.

| No. | Stress (MPa) | Time       | Fatigue cycle life(N) | Fracture |
|-----|--------------|------------|-----------------------|----------|
| 1   | 450          | 5h30min    | 2145000               | Yes      |
| 2   | 435          | 25h39min   | $>10^7$               | No       |
| 3   | 450          | 13h21min   | 5206500               | Yes      |
| 4   | 435          | 25h39min   | $>10^7$               | No       |
| 5   | 450          | 25h39min   | $>10^7$               | No       |
| 6   | 465          | 25h39min   | $>10^7$               | No       |
| 7   | 480          | 5h52min    | 2288000               | Yes      |
| 8   | 465          | 25h23min   | 9899500               | Yes      |
| 9   | 450          | 25h39min   | $>10^7$               | No       |
| 10  | 465          | 25h39min   | $>10^7$               | No       |
| 11  | 480          | 14h33min   | 5674500               | Yes      |
| 12  | 465          | 16h38min   | 6487000               | Yes      |

Figure 3. Rotating bending fatigue S-N curve of the single crystal alloys IC6SX at 900°C.

In this work, the median fatigue strength can be calculated using the following equation (1) [8] as used in our previous studies [11]:

$$\hat{\sigma}_{50} = \frac{1}{n} \sum n_i \sigma_i^*$$  \hspace{1cm} (1)

Where, $\hat{\sigma}_{50}$ is median fatigue strength, $\sigma_i^*$ is stress level of the class i, and the stress level can be divided according to the stresses which were used in the fatigue tests by up-and-down method, $n_i^*$ is the matching number of tests which have been carried out under the stress level of $\sigma_i^*$, and $n^*$ is the total matching number under all stress levels of the fatigue tests.
According to the up-and-down curve shown in figure 2 and table 1, the matching numbers of rotating bending fatigue specimens tested with different stress level are given in table 2. It can be found that there are total 6 matching numbers with the three different stress levels. As shown in table 2, it can be found that the matching numbers \( n_i^* \) equal 2 for the stress levels of 472.5 MPa and 457.5 MPa, respectively. And the matching number \( n_i^* \) equal 1 was only for the stress level of 442.5 MPa.

**Table 2.** Data of rotating bending fatigue for single crystal alloys IC6SX at 900°C.

| \( \sigma_i \sim \sigma_{i+1} \) (MPa) | \( \sigma_i^* \) (MPa) | \( n_i^* \) |
|----------------|----------------|-----|
| 480~465         | 472.5         | 2   |
| 465~450         | 457.5         | 2   |
| 450~435         | 442.5         | 2   |

Therefore, it can be calculated that the median fatigue strength \( \hat{\sigma}_{50} \) was 457.5MPa for the single crystal alloy IC6SX at 900°C.

The sample standard deviation for fatigue strength can be calculated approximately by the equation (2) in the following [10]:

\[
S^* = \sqrt{\frac{\sum (\sigma_i^* - \hat{\sigma}_{50})^2 n_i^*}{n^* - 1}}
\]

(2)

Where \( S^* \) is the sample standard deviation for fatigue strength.

In addition, the estimated values of safety fatigue strength \( \hat{\sigma}_p \) and variation coefficient \( C_V \) can be calculated by equation (3) and equation (4) [10], respectively.

\[
\hat{\sigma}_p = \hat{\sigma}_{50} + u_p \beta S^*
\]

(3)

\[
C_V = \frac{S^*}{\hat{\sigma}_{50}}
\]

(4)

According to the standard manual of HB/Z112-86, it can be found that the safety fatigue strength estimator \( \hat{\sigma}_{99.9} \) is 413.93 MPa. And the results of the rotating bending fatigue properties for the single crystal IC6SX at 900°C were listed in table 3. Compared with the attachment in the standard manual of HB/Z112-86, it can be seen that the effective samples in these tests have reached the requirement.

**Table 3.** Results for rotating bending fatigue properties of single crystal alloys IC6SX at 900°C.

| \( N_0 \) | \( n^* \) | \( \hat{\sigma}_{50} \) (MPa) | \( S^* \) (MPa) | \( S^*/\hat{\sigma}_{50} \) | \( \hat{\sigma}_{99.9} \) (MPa) |
|-----|-----|----------------|-------|----------------|----------------|
| 10^7 | 6   | 457.50         | 13.41641 | 0.02933        | 413.93         |

3.2. Microstructure analysis of rotating bending fatigue fracture of single crystal alloys IC6SX at 900°C

The fatigue fracture surfaces of the single crystal alloys IC6SX were investigated after the rotating bending fatigue tests at 900°C with different stresses and fatigue cycle life as shown in figure 4, figure 5 and figure 6, respectively. As can be seen, these fatigue fracture surfaces were roughness and basically perpendicular to the principal stress as shown in figure 4(A and B), figure 5(A and B) and figure 6(A and B). Because the temperature at 900°C is higher for the single crystal alloys IC6SX so that the strength of the alloys IC6SX decreased and the atomic diffusion rate was faster in the alloys. Due to the atomic diffusion ability enhancing in the alloy, the dislocation climb can be promoted so that the cracks propagated easily perpendicular to the principal stress until the alloys ruptured. Therefore, the deformation mode of the single crystal alloys IC6SX was not only the dislocation glide...
but also the dislocation climb. Under the stress of 450Mpa, fatigue crack source was only at one end of the test specimens. The cracks propagated from one end of the test specimens to the other at 900°C /450Mpa until the specimens fractured instantly resulting in unstable propagation of cracks as shown in figure 4 (B). As the stress increased to 465Mpa and 480MPa for the rotating bending fatigue tests at 900°C, fatigue cracks source was not only at one end of the test specimens but also on the surface of fatigue specimens, such as shrinkage cavity, porosity, inclusion, phase boundary, second-phase particle, dislocation and so on. The cracks propagated from surface of the test specimens to the center until the specimens fractured as shown in figure 5 (B) and figure 6 (B).

Figure 4. SEM images of fracture surfaces for IC6SX alloy fatigued under 900°C/450MPa after the fatigue life 2145000 cycles. A. side view of fracture, B. macro profile of fracture, C. rivers pattern, D. fatigue stripes.

According to the view of previous studies [12], because of the alternating of the valid slip system, the initiation of the fatigue micro-cracks occurs after several times. Since the alloys with face-centered cubic system, it can be found that the small class cleavage planes are in the area of fatigue cracks initiation. And the patterns for the class-cleavage facet were different, including annual ring, sector, rivers pattern. As shown in figures 4(C), 5(C) and 6(C), at the initiations area of fatigue cracks, the typical rivers pattern was found. This kind of class-cleavage facet with rivers pattern mentioned above is quite different from the cleavage plane formed in the brittle fracture of the alloys which belong to the body centered cubic system and the hexagonal system. Compared with the cleavage plane, it could be found that the class-cleavage facet formed slowly during the processing of slip, and the cleavage plane formed instantaneously at the time of fracture along the crystal plane with the lowest binding energy.
Figure 5. SEM images of fracture surfaces for IC6SX alloy fatigued under 900 °C/465 MPa after the fatigue life of 9899500 cycles. A. side view of fracture, B. macro profile of fracture, C. rivers pattern, D. fatigue stripes.

Figure 6. SEM images of fracture surfaces for IC6SX alloy fatigued under 900 °C/480 MPa after the fatigue life of 2288000 cycles. A. side view of fracture, B. macro profile of fracture, C. rivers pattern, D. fatigue stripes.
Cracks growth moved into the steady-state propagation stage after propagating a certain distance. At the steady-state propagation stage, the fatigue stripes appeared which were arc-shape and parallel to each other basically. And it is easier to form fatigue stripes in the ductile materials than that in the brittle materials. However, as shown in figure 4(D), 5(D) and 6(D), it can be found that there were some arc-shape strips vertical to the direction of cracks propagation at some areas. The fatigue cracks grew continuously under the action of alternating stress between propagating and arresting. The fatigue cracks propagated along the glide plane with the action of tensile stress. When the tensile stress turned into compressive stress, the fatigue crack growth stopped and blunting occurred at the crack tips. So that fatigue strips were left due to the stress concentration at the cracktips.

4. Conclusion
1. Ni$_3$Al single crystal alloy IC6SX has excellent rotating bending fatigue property at 900°C. The median fatigue strength is 457.5MPa and the safety fatigue strength is 413.93MPa.
2. The typical fatigue stripes appeared at the stage of fatigue cracks steady state propagation due to the stress concentration at the cracktips under the action of alternating stress between propagating and arresting.

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References
[1] Han Yafang and Li Sunhua 1994 Directional solidification cast Ni$_3$Al-based superalloy IC6 Gas Turbine Experiment and Research 1 48–51
[2] H. U. Hong, B. G. Choi, I. S. Kim, Y. S. Yoo and C. Y. Jo 2011 Procedia Engineering Deformation behavior during thermo-mechanical fatigue of a nickel-based single crystal superalloy 10 281–286
[3] Alexander Staroselsky, Brice N. Cassenti 2011 International Journal of Solids and Structures Creep, plasticity, and fatigue of single crystal superalloy 48(13) 2060–2075
[4] Johan J. Moverare, Sten Johansson and Roger C. Reed 2009 ActaMaterialia Deformation and damage mechanisms during thermal–mechanical fatigue of a single-crystal superalloy 57(7) 2266–2276
[5] M. Okazaki and M. Sakaguchi 2007 International Journal of Fatigue Thermo-mechanical fatigue failure of a single crystal Ni-based superalloy 30(2) 318–323
[6] Wu Meiling, GuoFengwei, Li Ming and HanYafang 2016 Materials Science Forum-Special and High Performance Structural Materials Effect of trace strontium addition on microstructure and room temperature fracture toughness of Nb-12Si-22Ti alloys 849 603–608
[7] H. Zhou, H. Harada, Y. Ro and I. Okada 2004 Materials Science & Engineering A Investigations on the thermo-mechanical fatigue of two Ni-based single-crystal superalloys 394(1) 161–167
[8] H. S. Bai, H. Cao, L. F. Liao and B. Z. Zhou 2009 Aeroengine 35(3) 30–31
[9] HB5153-96, The rotating bending fatigue test method of metal at high temperature[S]
[10] HB/Z112-86, Thestatistic analysis method of material fatigue test [S]
[11] L. W. Jiang, M. L. Wu, S. S. Li and Y. F. Han 2015 Materials Research Innovations Rotating bending fatigue behaviour of a Ni$_3$Al based single crystal alloy IC6SX at 760°C 19(S4) 163–169
[12] W. Z. Jiang, S. X. Zhao, C. S. Wang and Z. Zhang 2000 The mechanical properties of engineering materials 88–125