Fracture toughness analysis of anisotropic nickel-based single crystal superalloys at high temperature

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Abstract The purpose of this article is intended to study the fracture toughness of anisotropic nickel-based single crystal superalloys at elevated temperature by compact tension (CT) specimen in experiment method. The special attention is put on the orientation and temperature dependence of mechanical properties of single crystal as well as thickness effect of specimen. The experimental results show that crystallographic orientation and temperature have much complex effect on fracture toughness. The difference of fracture toughness of single crystal for different crystallographic orientation at low temperature is much greater than that at high temperature. The nickelbased single crystal specimen may merely become less anisotropic with the increase of the ambient temperature seems as more multi-slip action appears. The fracture mode of the specimens transfers from brittle to ductile as the temperature increases; also, the fracture toughness of single crystal for the same orientation becomes larger with the decreasing of the thickness of single crystal specimen.

Introduction

Nickel-based single crystal superalloys had been widely used in gas turbine blades and jet engineer due to their superior creep and fatigue properties by eliminating the grain boundary, in which micro-crack and void come into being easily in polycrystalline metal at high temperature [\[1](#page-5-0)]. In the past three decades, several researchers had performed many works on the elasto-plastic, creep, and fatigue constitutive behaviors of single crystal superalloys in theory and experiment $[1-5]$. However, the fracture behavior of such components is not yet well understood. A main problem is orientation and temperature dependence of mechanical properties of single crystal, while the thickness effect of single crystal structure may be an other trouble because of the thinness of turbine blade. Fracture toughness, which means the resistance to crack propagation, plays a major role in fracture mechanics analyses of single crystal blades to understand and predict crack initiation.

As deep understanding on the occurrence of fracture accident and improving of design technology of single crystal blade, the new design method, e.g., damage and tolerance limit method, has been suggested. But the lack of experimental data of fracture properties of single crystal material limits the development of the damage and tolerance design method. Consequently, fracture toughness is a crucial property for single crystal to obtain accurate work life of turbine blade.

Recently, study of the fracture behavior of single crystal materials had been almost carried out by analyzing theoretically the stress and strain deformation fields at the tip of a crack in a single crystal $[6–14]$ $[6–14]$. Rice $[6, 7]$ $[6, 7]$ $[6, 7]$ $[6, 7]$ $[6, 7]$ proposed the first asymptotic solution of the crack tip stress field in single crystals. Kysar studied crack tip deformation fields in ductile single crystal media experimentally and compared the result to that predicted by Rice. The kink shear sector boundary was found [[8\]](#page-5-0). Flouriot et al. [[9–11\]](#page-5-0) analyzed strain localization at the crack tip in single crystal compact tension (CT) specimens within the framework of classical continuum crystal plasticity. Zhao et al. [\[12](#page-5-0)] proposed a failure assessment approach to predict the crack initiation time of single crystal notched specimen based on the stress intensity

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factor. However, studies on the fracture behavior of such alloy are lacking from the engineering point of view.

It is well known that anisotropic property is a significant feature of single crystal superalloy compared to traditional casting alloys. The discrepancy of creep or fatigue life can be almost up to six times for single crystal superalloys in different crystallographic orientations [\[13](#page-5-0)]. The high-temperature ambient makes the problem more complex. So it is necessary to study the orientation and temperature dependence to fracture toughness of single crystal. Since the thickness of a typical component (e.g., gas turbine blade) is on the order of millimeters [\[12](#page-5-0)], the thickness effect on the fracture toughness must be taken into consideration to calculate the life of blades accurately.

In the present article, we report on fracture toughness results from CT specimen of single crystal for different orientations and thickness at high temperature. The fracture micro-characteristics of specimen fracture surface have been examined by scan electron microscope (SEM).

Materials and experiment procedures

In this work, the nickel-based single crystal high-temperature superalloy DD3 was used, which has been used commercially in turbine blade. The chemical composition in weight percentage is given in Table 1. It has a highvolume fraction of coherent intermetallic γ' precipitates dispersed throughout nickel matrix γ phase [\[14](#page-5-0)]. The crystallographic structure of nickel-based single crystal alloys DD3 is face-centered cubic (F.C.C). DD3 bars were grown using the Bridgeman technique. DD3 bars were machined into CT specimens using spark cutting, whose geometric shape and dimension is shown in Fig. 1. The notch front direction and loading axis are defined in Fig. 1 as X_1 , and X_2 axes, respectively. The X_3 axis is taken to be normal to the $\langle X_1, X_2 \rangle$ plane. Three kinds of specimen crystallographic orientations for (001)[100], (011)[100], and $(111)[0-11]$ are selected for tests. (001) , (011) , and (111) are normal directions of crack planes and [100] and [0–11] are the crack propagation directions. The crystallographic orientations were determined by Laue backreflection X-ray procedures. The misorientations were $<$ 5 $^{\circ}$ in casting direction. Before fracture testing, the pre-crack was made under cyclic loading using a frequency of 2.5 Hz at room temperature. The length of the pre-crack was about 0.8 mm. All the different orientation specimens were tested

Table 1 The chemical composition of single crystal DD3

			Cr Co W Al Ti Fe S Si Ni	
			9.0 4.5 5.0 5.5 1.7 0.5 0.001 0.2 Bal	

Fig. 1 CT specimen geometric dimension of nickel-based single crystal

in Instron 8802 model servohydraulic testing machine at 630, 760, 850, and 950 °C. Specimen heating was by an induction 10 kW RF induction heater. Temperature was measured using a Pt/Pt10%Rh thermocouple, roped by an asbestos string in the center of the specimen. Two other thermocouples were roped at the ends of the specimen. The temperature was maintained to within ± 1 °C of the test temperature and allowed to stabilize for more than half an hour before each test began. All tests were carried out under fully displacement control. The stretching rate was 1 KN/min. The curves of load versus displacement at the load point were recorded by computer automatically.

The fracture surfaces of all specimens were examined by a scanning electron microscopy (SEM) to characterize the micro-fracture features.

Experiment result and analysis

The stress intensity factor of single crystal CT specimen is calculated by three-dimensional finite element method, because the specimen thickness is different and some small size specimen cannot satisfy the plane strain condition stated by the experiment standard. Then the fracture toughness can be obtained on the elastic limit load.

Thickness dependence of fracture toughness

Since the thickness of a typical nickel-based single crystal component (e.g., gas turbine blade) is on the order of millimeters, the thickness effect on the fracture toughness must be taken into consideration. In this experiment, all the crack orientation of single crystal specimens are (001)[100] and the test temperature is 630 $^{\circ}$ C. Table [2](#page-2-0) and Fig. 2 show that the experimental results of fracture toughness and loading curves of single crystal specimens for different thickness. It is found that the specimen geometry and thickness have

Specimen	Orientation	Temperature/ ${}^{\circ}C$	Load (P_o/kN)	Thickness (B/mm)	Width (W/mm)	Crack (length/mm)	Fracture toughness
10	<001	630	4.31	3.50	20.01	10.36	80.6
11	<001	630	7.58	6.54	20.02	10.34	72.3
12	<001	630	8.82	10.08	19.97	10.56	64.9

Table 2 Experiment result of fracture toughness of single crystal specimen for different thickness

Fig. 2 The curves of load with crack mouth opening displacement of single crystal specimen for different thickness

strong influence on the crack tip fields as well as fracture toughness of single crystal. The fracture toughness of single crystal at the same orientation becomes large with the decreasing of the thickness of single crystal specimen. The maximum difference of fracture toughness between the thin and thick specimen is 27.8%. However, the crack tip opening displacement decreases with the decreasing of the thickness of single crystal specimen. Experimental results are in agreement with that of general metal at room temperature [\[15](#page-5-0)]; so, the thickness effect of single crystal specimen must be taken into consideration in design of single crystal blades. Moreover, it has much influence on the crack propagation direction. For the thin specimen (3.5 mm), the crack propagation direction will deflect from the original crack direction. The experimental result shows that, to the

specimen in (001)[100] crack orientation, the tilt angle from the fracture surface to the crack plane is 45° . The crack growth is caused by the activation and movement of the surface slip systems. For the thick specimen (10 mm), the crack propagation direction is perpendicular to the loading axis. For the middle thickness specimen, the fracture behavior is more complex and between the thin and thick specimens. The fracture surface is composed of the plane region in mid-section and shear lip in free surface. The thick specimen is under plane strain state while thin specimen is under plane stress state, which has strong influence on the activation of slip system as well as critical resolved shear stress of slip system. In general, the octahedral slip system will be activated in $\langle 001 \rangle$ orientation single crystal at 630 $^{\circ}$ C. For thick specimen, there are eight slip systems moving simultaneity. When the thickness of specimen becomes small, initial slip system activates and moves first in loading process. Subsequently, secondary slip system will activate because of rotary movement of crystal lattice. This will lead to the increase of critical resolved shear stress of slip system, also, multiple slip system movement causes more extension displacement of specimen.

Orientation and temperature dependence

In this section, the fracture toughness experiment is performed for the specimen in three crack orientation (001)[100], (011)[100], and (111)[0–11] at 760, 850, and 950 °C to study the crystallographic orientation and temperature dependence of single crystal fracture toughness. From Table 3, it is found that the fracture toughness of

single crystal at high temperature is higher than other metal. The crystallographic orientation and temperature have much effect on the fracture toughness [\[16](#page-5-0)]. It is clear that fracture toughness will remarkably decrease with the increase in temperature. At 760 and 850 $^{\circ}$ C, the fracture toughness in (111)[0–11] orientation specimen is biggest, $(011)[100]$ orientation specimen is middle, and $(001)[100]$ orientation specimen is smallest. The maximum difference is 15.9% between $(111)[0-11]$ and $(001)[100]$ orientation specimens. At 950 \degree C, the fracture toughness decreases in the order of the specimens of $(001)[100]$, $(111)[0-11]$, and $(011)[100]$ orientations, the maximum difference is 4.6% between (001)[100] and (011)[100] orientation specimens. The nickel-based single crystal specimen may merely become less anisotropic with the increase of the ambient temperature. This can be found in the crack tip opening displacement. The maximum difference of displacement at 760 °C is 22.1% between (001)[100] and (011)[100] orientation specimens, while that is only 8.4% at 950 $^{\circ}$ C. Temperature has much more effect on the crack tip opening displacement strongly. The average of displacement at 760 \degree C for three orientations is only 0.98 mm, while the average of displacement at 950 $^{\circ}$ C is up to 4.77 mm. Figure 3 shows the curves of load with crack mouth opening displacement. It can be found that displacements at the load point at 950 \degree C are much bigger than that at 850 and 760 °C for all specimens. This shows that the fracture mode of the CT specimens transfers from brittle to ductile with the increase in temperature. It will be confirmed by the SEM analysis of fracture surface in the follow.

The mechanical anisotropy of single crystal superalloys is dependent on test temperature, stress state, chemical composition, and crystallographic orientation. It has been shown that activation and movement of slip systems are found to be the basic deformation mechanisms of the nickel-based single crystal superalloys. In nature, the activation degree of the octahedral and cubic slip systems strongly affects the anisotropy. Temperature is a main factor that affects on the activation degree of slip systems, so it influences the anisotropy of single crystal. The mechanical performance of single crystal at low temperature is affected by ambient more than that at high temperature. For single crystal DD3 material, the experiment shows that the octahedral slip systems will be activated below $760 °C$ and cubic slip systems will also be activated gradually above $760 \degree C$ [\[17](#page-5-0)]. The anisotropy of single crystal at low temperature is stronger than that at high temperature as more multi-slip action appears. In fact, there may be a little deviation of crystallographic orientation, which also affects on the activation of slip systems. So the crystallographic orientation and its misorientation also have much effect on the anisotropic performance.

Fig. 3 The curves of load with crack mouth opening displacement (a) $(001)[100]$ orientation, (b) $(011)[100]$ orientation, and (c) $(111)[0-11]$ orientation

SEM photos of fracture surface of single crystal specimens at different temperatures and crack orientations are shown in Fig. [4.](#page-4-0) One can find that the fracture surfaces are smooth and cleavage planes at 760 °C for different crystallographic orientations as shown in Fig. [4a](#page-4-0), d, and g.

Fig. 4 SEM photos of fracture surface at different temperature and orientations. (a) $(001)[100]$ orientation, at 760 °C. (b) $(001)[100]$ orientation, at 850 °C. (c) $(001)[100]$ orientation, at 950 °C. (d) (011)[100] orientation, at 760 °C. (e) (011)[100] orientation, at 850 °C.

(f) (011)[100] orientation, at 950 °C. (g) (111)[0-11] orientation, at 760 °C. (**h**) $(111)[0-11]$ orientation, at 850 °C. (**i**) $(111)[0-11]$ orientation, at 950 °C

Fracture mode of these specimens is pure shear fracture on the crystallographic plane {111}. Measure result shows that the angle between the {111} facet and principal stress is about 57°. The deformation of single crystal is provoked by the activation of $\langle 110 \rangle$ [111] octahedral slip systems and occurs only in some prior slip systems. This priority leads to the high slipping in homogenous nature and petrosal feature in fracture appearance [\[18](#page-5-0)]. The thickness of the specimens at the crack tip does not change after fracture. There is no necking phenomenon in specimens for all crack orientations at 760 \degree C. But the fracture surfaces of single crystal specimens at 950 °C in Fig. 4c, f, and i, contain a lot of voids and dimples. It is illustrated that the superalloy is sensitive to the shrinkage porosity. It should be pointed out that a lot of small smooth stair cases at the right of the image in Fig. 4f comes into being because of pre-crack under cyclic loading. From Fig. 4i, we can obviously find that the thickness of the specimen in the fracture cross-section is much thinner than that before fracture. There is obvious necking phenomenon in specimens at 950 °C. In the fracture surface of the specimens at 850 \degree C, the characteristics of fracture surface are between 760 and 950 \degree C. There are micro-voids and cleavage planes in fracture surface showed in Fig. 4b, e, and h.

The mechanical behavior of nickel-based single crystal shows that there are three kinds of slip systems, octahedral slip systems $(\langle 110 \rangle [111])$ and cubic slip systems $(\langle 110 \rangle [100])$, which may be activated and moved in the deformation process of single crystal. The research results reveal that the octahedral slip systems will be activated at low temperature, but the octahedral and cubic systems will be activated, simultaneously. At high temperature, the critical resolved shear stress will decrease. The multi-slip systems action induces cross-section shrinkage at the crack front.

Generally, the initiation and growth of crack in nickelbased single crystal can be divided into two categories: crystallographic crack and non-crystallographic crack. What crack occurring is dependent on the environmental temperature and stress state for the real structure. The crystallographic orientations affect the mechanical properties strongly at low temperature, but it is not obvious at high temperature. At low temperature, the maximum resolved shear stress is a major factor that makes the crack in single crystal to occur on the {111} slip planes; so the crack deflects from the crack orientation and belongs to the crystallographic crack. At elevated temperature, the microstructure occurs to raft and the micro-voids coalescence is major crack source. The specimen will fracture along the plane that is normal to the loading axis.

Conclusions

This work studies the fracture toughness of anisotropic nickel-based single crystal superalloys at elevated temperature by CT specimen, taking the different crystallographic orientations into consideration. The following summaries are necessary:

- 1. Crystallographic orientation and test temperature prove to be the major factors that affect the fracture toughness of single crystal. The difference of fracture toughness of single crystal for different crystallographic orientation at low temperature is much greater than that at high temperature.
- 2. The fracture mode of the CT specimens transfers from brittle to ductile with the increase in temperature. The activation degree of the octahedral and cubic slip systems affects the fracture behavior of single crystal strongly at different temperatures and crystallographic orientations.

3. The fracture toughness of single crystal at same orientation becomes large with the decreasing of the thickness of single crystal specimen. The activation and movement of multiple slip system are main reason that the fracture toughness becomes large in thin specimen.

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