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# Damage and fracture mechanism of a nickel-based single crystal superalloy during creep at moderate temperature

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ABSTRACT

By means of creep properties measurement, microstructure and fracture morphology observation, an investigation has been made into the damage and fracture mechanism of a nickel-based single crystal superalloy during creep at intermediate temperature. The results show that the deformation mechanism of the alloy is dislocations slipping in the $\gamma$ matrix and shearing into $\gamma'$ phase, the dislocations shearing into $\gamma'$ phase may be decomposed to form the configuration of the partials plus stacking fault and the K–W locking. While in the latter stage of creep, the primary–secondary slipping systems are alternatively activated to shear and twist the cubical $\gamma'/\gamma$ phases, which results in the initiation of the micro-crack occurring in the intersection regions of two slip systems. As creep goes on, the micro-cracks are propagated on the (001) plane along $\langle 110 \rangle$ directions. Because the multi-cracks are formed and propagated on the different cross-section of the sample during creep, which may result in the tearing edge or secondary cleavage plane generating at the tip of the cracks along the direction with bigger shearing stress. When the propagating primary cracks on (001) plane intersects with {111} secondary cleavage plane, the propagation of the primary cracks is terminated to form the square-like cleavage plane on the (001) plane. In further, the multi-cracks propagating on the different cross-section are joined by the tearing edge or secondary cleavage plane until the occurrence of creep fracture, this is thought to be the main reason of the creep fracture displaying the uneven and multi-level cleavage features.



## 1. Introduction

The microstructure of nickel-based single crystal superalloys consists of the cubical $\gamma'$ phase embedded coherent in $\gamma$ matrix [1–3], and they have been widely used to make the blade parts of the advanced aero-engines for their high volume fraction of $\gamma'$ strengthening phase which results in the good creep resistance at high temperature [4–6]. With the increase of service performance, such as the aero-engine power and thermal efficiency, the mechanical and creep properties of superalloys at high temperatures need to be further improved [7–9]. Adding some refractory elements, such as W, Ta, or Mo, is expected to improve the creep resistance of single crystal nickel-based superalloys [10] due to the solution strengthening effects of the elements to slow down the diffusing processes. Although the single crystal nickel based superalloys possess the excellent mechanical and creep properties at high temperature, the centrifugal force originated from high-speed rotation in aero-engine in service still may cause the creep damage of the blade parts, which is thought to be the main model of aero-engine failure [11–13].

The microstructure and creep feature of the single crystal superalloys change with imposed stresses and temperatures. Under the conditions of the applied stress of 552 MPa at 850 °C, the creep of some alloys displays an incubation period, in which some dislocations are piled up in matrix channels [14]. In the primary stage of creep, the activated dislocations overcome the Orowan resistance to slip along the $\langle 110 \rangle$ direction on the {111} planes in the $\gamma$ matrix channel [15]. And the reaction of two sets dislocations slipping in the same channel occurs to form the dislocation networks, which may promote the climbing of dislocations during steady state creep of alloy [16]. But with the enhancement of creep temperature, the interfacial dislocation to induce shearing $\gamma'$ phase becomes progressively easier, because of the decrease of elastic energy and the increase of diffusivity [17]. And the cavities and micro-cracks are formed and propagated along the interfaces of $\gamma'/\gamma$ phases in the latter stage of creep [18].

When no eutectic and initial melting phenomena occurs in alloy, the micro-void formed during solidification may transform into the micro-cracks during creep at high temperature [19], and the disordering micro-cracks may initiate and propagate along the $\gamma'/\gamma$ interfaces vertical to the stress axis up to the occurrence of creep fracture [4,20]. Some investigations on the creep fracture of

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superalloys at high temperature reported [21–24] that the micro-voids formed during solidification may be congregated into the cavities or micro-cracks during creep at high temperature, and the cavities or micro-cracks are initiated and propagated along the interface of $\gamma'/\gamma$ phases, as the creep goes on, up to the occurrence of creep fracture.

Due to the work of aero-engines in service undergoing the processes from intermediate temperature/higher stress to high temperature/lower stress, the blade parts in aero-engine had to bear various work conditions from start to stable course. And the superalloys with various compositions display different creep properties at various temperatures. Although some literatures had reported that the creep and damage behaviors of single crystal nickel based superalloys at high temperature [25–28], few literature reported that the damage and deformation mechanisms of the superalloys during creep at intermediate temperatures, and the effect of the slipping traces on the initiation and propagation of the cracks and the fracture mode of the alloy during intermediate temperature creep is still not clear. Especially, the single crystal blade parts in service at medium temperature/higher stress conditions is easy to failure, and understanding the damage process of the blade parts in service has an important guiding significance on choosing materials and structure design. Therefore, the damage behaviors of single crystal superalloys during creep at medium temperature are to be investigated.

Hereby, in the paper, by means of creep properties measurement and microstructure observation, the damaged and fracture behaviors of a single crystal nickel based superalloy during creep at intermediate temperature are investigated.

## 2. Experimental procedure

The single crystal nickel based superalloy with [001] orientation had been produced by means of selecting crystal method in a vacuum directional solidification furnace under the condition of a high temperature gradient. The nominal composition of the superalloy is Ni–6.0Cr–11Co–9W–*x*Mo–6.0Al–7.0Ta (mass fraction, %), and all samples are within 7° deviating from the [001] orientation. The heat treatment regimes of the single crystal nickel based superalloy are given as follows: 1280 °C × 2 h, AC + 1315 °C × 4 h, AC + 1080 °C × 4 h, AC + 870 °C × 24 h, AC.

After fully heat treated, the bars of the alloy were machined, along the [001] orientation, into the tensile creep samples with the cross-section of 4.5 mm × 2.5 mm and the gauge length of 20 mm, the wider surface of the sample is parallel to the (100) plane. The uni-axial constant loading tensile tests were conducted, in a creep testing machine with GWT504 model, under the applied stress of 800 MPa at 760 °C, 780 °C and 800 °C and applied stress of 750 MPa, 775 MPa an 800 MPa at 800 °C, respectively. The samples after crept up to fracture are grinded and polished, and then the microstructure and fracture of the alloy are observed by using Scanning electron microscope (SEM). The compositions of the eroded solution are 20 g $CuSO_4$ + 5 ml $H_2SO_4$ + 100 ml HCI + 80 ml $H_2O$. The films are prepared by twin-jet electro-polishing method for observing the microstructure under transmission electron microscope (TECNAI20 model), combined to the contrast analysis of the dislocation configuration by means of the double beams technology, for investigating the damage and fracture mechanism of the single crystal nickel-based superalloy during creep at intermediate temperature.

## 3. Experimental results and analysis

### 3.1. Creep features of alloy

Under the conditions of the applied higher stress at intermediate temperatures, the creep curves of the free-Re single crystal nickel-based superalloy are measured, as shown in Fig. 1. The creep curves of the alloy under the applied stress of 800 MPa at various temperatures are shown in Fig. 1(a), indicating that the strain rates of the alloy during steady state creep at 760 °C, 780 °C and 800 °C are measured to be 0.00876%/h, 0.0127%/h and 0.0155%/h, respectively, and creep lifetimes of the alloy are measured to be 323 h, 165 h and 84 h, respectively. It may be understood that when the creep temperature enhances from 760 °C to 780 °C, the strain rates of the alloy increase from 0.00876%/h to 0.0127%/h, and the lifetimes of the alloy decreases from 323 h to 165 h, the decreasing extent of the lifetime is about 96%. The creep curves of the alloy under the applied stress of 750 MPa, 775 MPa and 800 MPa at 800 °C are shown in Fig. 1(b), which indicates that the strain rates of alloy during steady state creep at 750 MPa and 775 MPa are measured to be 0.0067%/h and 0.013%/h, respectively, and creep lifetimes of the alloy are measured to be 260 h and 125 h, respectively. When the applied stress enhances from 775 MPa to 800 MPa, the strain rates of the alloy increase from 0.013%/h to 0.023%/h, and the lifetimes of the alloy decreases from 125 h to 84 h, the decreasing extent of the lifetime is about 48.8%. This suggests that the alloy possesses an obvious sensibility on the applied temperatures and stresses in the ranges of the applied temperatures and stresses.

The transient strain of the alloy occurs at the moment of applying load at high temperatures. And the density of dislocations increases as the creep goes on, which results in the strain strengthening of alloy to decrease the strain rate due to increasing the dislocations moving resistance [29]. As creep time prolongs, the slipping and climbing of dislocations are activated to promote the recovery softening of the alloy, which may relax the stress concentration in local regions. Once the equilibrium of the deformation strengthening and the recovery softening is obtained, the strain rate of the alloy keeps constant for entering the steady state stage of creep. Thereinto, the strain rate of the alloy during steady-state creep may be described by Dorn's law as follows [30]:

$$\dot{\varepsilon}_{ss} = A\sigma_A^n \exp\left(-\frac{Q_a}{RT}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the strain rate during steady state creep, $A$ is a constant related to the microstructure, $\sigma_A$ is the applied stress, $n$ is the apparent stress exponent, $R$ is the gas constant, $T$ is the absolute temperature, and $Q$ is the apparent creep activation energy.

According to the data in creep curves of Fig. 1, the dependences of the strain rates during steady state creep on the applied temperatures and stresses are shown in Fig. 2(a) and (b). In the further, in the ranges of the applied temperatures and stresses, the apparent creep activation energy and apparent stress exponent of the superalloy are calculated to be $Q$=568.3 kJ/mol and $n$=13.8, respectively. This indicates that the single crystal nickel base superalloy has a better creep resistance in the ranges of the applied stresses and temperatures. And it may be deducted according to the calculating stress exponent that the dislocations slipping in the $\gamma$ matrix and shearing into the $\gamma'$ phase are thought to be the main deformation mechanisms of the alloys during steady state creep at medium temperature.

### 3.2. Dislocation configuration during creep

The morphologies of the alloy at different states are shown in Fig. 3, the normal of the observing sample is [100] orientation, the direction of the applied stress is marked by the arrows. After fully heat treated, the microstructure of the alloy consists of the cubical $\gamma'$ phase embedded coherent in the $\gamma$ matrix, is shown in Fig. 3(a), thereinto, the [001] and [010] directions on (100) plane is marked by the arrows, which indicates that the cubical $\gamma'$ phase in the alloy is regularly arranged along 〈100〉 orientation, and the average