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ARTICLE

Dislocation Structure in a Single Crystal Nickel Base Superalloy During High Cycle Fatigue at 870 °C

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Abstract: Dislocation structure and its impact on the mechanical behavior of a γ' strengthened nickel base single crystal alloy under high cycle fatigue (HCF) at 870 °C was studied. The results indicate that HCF lifetime declines with increase of applied stress amplitude. In the early stages, the fatigue deformation occurs by forced bowing of dislocations through the narrow γ matrix channels on {111} planes. During mid-term fatigue stages, most of the dislocations formed in the matrix are located in the γ/γ' interfaces, whose intersection and reaction produce new dislocation segments and three-dimensional dislocation networks. The Burgers vectors of the dislocations in the network are 1/2<110>. The interaction of cyclic stress with high temperature induces the precipitation of homogeneous globular γ' particles, which is beneficial to fatigue strength. At the end of fatigue test, the cyclic stress leads to the formation of persistent slip bands moving through the γ matrix channels and γ' particles. Dislocation shearing through the γ' phase is found occasionally. The extrinsic stacking faults is also observed.

Key words: nickel base single crystal superalloy; high cycle fatigue; dislocation

Single crystal nickel-base superalloy, which contains the large volume fraction usually more than 65%~70% of intermetallic $Ni_3(Al, Ti)$ phase, are widely used as blade and vane materials in modern gas turbine and aeroengine[1-3]. In practice, turbine blades experience complex thermo-mechanical loading including long periods of isothermal creep and creep with superimposed vibrations, i.e. interaction of high cycle fatigue with high temperature creep[4-6]. Hence, high cycle fatigue has been an area of ever-growing interest for the past several decades because of its involvement in numerous industry applications[7,8]. Turbine blades undergo two processes during cycling: the strain hardening and softening and the nucleation and propagation of a microcrack. The cyclic hardening and then softening occur over the whole deformation process. Although the macroscopic behaviors of the turbine blade alloy in fatigue have been extensively studied, no careful study has been devoted to the elementary mechanisms of deformation. In the present study, the objective was to investigate the microstructural evolution behaviour of the test alloy during high cycle fatigue. The emphasis will be put on the behaviour of dislocations produced during HCF in a single-crystal nickel-base superalloy.

1 Experiment

The single crystal nickel-base superalloy with [001] orientation was prepared by a crystal selection method in vacuum directional solidification furnace under a high temperature gradient. All specimens were within 10° deviation from [001] orientation. The nominal chemical composition (wt%) of this alloy is given in Table 1. As cast bar received a solution treatment at 1300 °C for 4 h, and two-step aging treatment, consisting of heat treatment at 1080 °C for 5 h and at 870 °C for 16 h, both followed by air cooling. The process of heat treatment produced a precipitation of regular cuboidal γ' particle aligning along <100> in the γ matrix. The cuboidal γ' possessed a volume fraction of about 65%, and an average grain size of about 450 nm. The microstructure after heat treatment is shown in Fig.1, which is composed of γ matrix

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Table 1 Nominal composition of superalloy (wt%)

Co	Cr	W	Mo	Al	Ti	Ta	Ni
5.5	8.1	5	2.2	6.1	2	3.4	Bal.

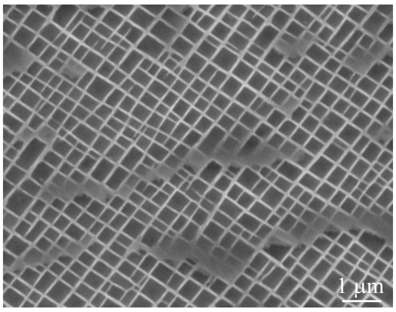

Fig.1 Microstructure of the test alloy after heat treatment

and cuboidal γ' precipitates.

High cycle fatigue tests at 870 °C were carried out after heat treatment. All the specimens were fatigue tested under load control at a stress ratio R=0.1 at a frequency of 115 Hz. Tensile-tensile loading mode was adopted and sinusoidal wave form was performed on the specimens. The stress axis is parallel to the crystal growth direction [001]. The thin foils for observation by transmission electron microscopy (TEM) have been prepared by the twin jet polishing method. Dislocation structure has been examined in a PHILIPS EM 420 microscope using a double tilt goniometer in diffraction and imaging modes.

2 Results and Discussion

2.1 *S-N* curve

The relationship between applied stress and life to failure is shown in Fig.2, in which indicates that the specimens do not fail during tests. As can be seen from Fig.2, the S-N curve shows a general trend that the fatigue life increases with decreasing cyclic stress amplitude, which is normally observed in other alloys. Fatigue strength is usually defined as

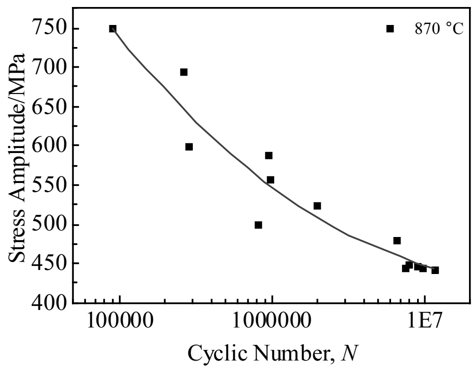

Fig.2 Variation of stress amplitude as a function of the cyclic number of the alloy fatigued at 870 °C

the stress amplitude for a specimen not to fracture after 10^7 cycles[2]. Therefore, the fatigue strength of the test alloy at 870 °C is determined as 443 MPa from Fig. 2. In Fig. 2, the selected time point marks cycle index correspond to those at which the foil specimens for TEM examination were prepared.

2.2 Dislocation structure in matrix

Strain is generally localized in the γ matrix phase. The nature of the active slip systems observed in all deformation microstructures with a [001] stress axis is consistent with the Schmid and Boas criterion i.e[9] with a preferential activation of eight systems among the 1/2{111}<110> possible ones. TEM observation reveals that strain distribution in HCF specimens is quite inhomogeneous, and dislocations are found to be generated in the matrix between γ' particles preferentially. In many areas, the sample (870 °C/443 MPa, N=88 cycles) resembles the aged samples, showing many coherent cuboidal precipitates with only a few isolated dislocations present (Fig.3a). During the early stage the grown-in dislocations serve as sources, and cyclic fatigue dislocations spread from these areas into the previous dislocation free areas. A higher magnification view of the bowing dislocations is shown in Fig.3b. In the area marked "B" there are several dislocation loops which appear to have taken a 90° change in direction during the process of traveling through the matrix. Stereo pairs show that these left angle turns in the dislocations (b=a/2[0$\bar{1}$1] must have resulted from the cross gliding of the leading screw dislocation segments from the (11$\bar{1}$) plane to the (111) plane, and the [001] is normal to plane of the micrograph). During mid-term fatigue stage, the material continues to fill with dislocations and most of γ' particles keep coherence with γ phase. All fatigue deformation continues to be accomplished by bowing the dislocations through the matrix channels on {111} planes. In Fig.3b nearly all of the dislocations have the same Burgers vector, showing a higher density of dislocations, and the γ matrix has become completely filled with dislocations (Fig.3c)

The dislocation networks have been developed by absorbing moving dislocations crossing a few tens of γ' precipitates. After the alloy fatigue cyclic number is increased to 10^6, and the 1/2<110> mobile dislocation are found to be formed in the alloy matrix, as shown in Fig.4a~4d. The normal line direction of the foil is roughly [100] orientation. Fig.4a shows triple dislocation network, where b1, b2 and b3 represent the Burgers vectors and directions of the three kinds of dislocation (Fig.4e). Two sets of g(020) and g($\bar{2}$02) diffraction conditions have been used to form images under two-beam condition, and the dislocations in the b1 set is invisible, as shown in Fig.4b. Therefore, the Burgers vector in the b1 set is a/2[101]. Similarly, contrast analyses show that dislocation b2 is out of contrast in g=002 and 11$\bar{1}$ operation reflection in Fig.4c and 4d; therefore, its Burgers vectors of dislocation b2 and b3 are a/2[1$\bar{1}$0] and a/2[011], respectively. The direction of dislocation lines in the b1 set is parallel to its

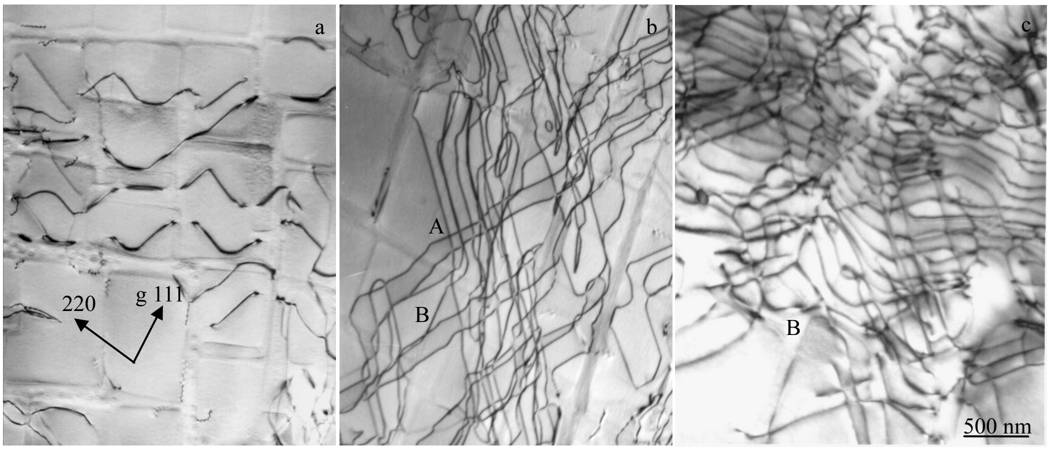

Fig.3 TEM micrographs showing the dislocations structure: (a) 870 °C /443 MPa, N=88 cycles; (b) 870 °C/443 MPa, N=1000 cycles; (c) 870 °C/700 MPa, N=1000 cycles

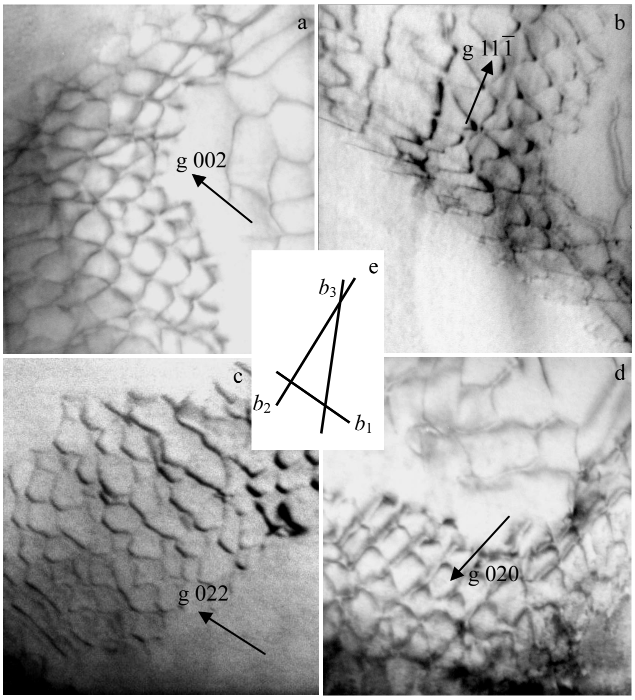

Fig.4 Contrast analysis of 1/2<110> dislocation networks formed in mid-term fatigue at 870 °C/443 MPa and N=10^5 cycles: (a) g= 002, (b) $g = 11\overline{1}$, (c) g = 022, and (d) g = 020

Burgers vector, indicating that the dislocations in the $b1$ set are of the screw type. The dislocations in the other two sets are of the screw type also. According to different operation reflection, it is shown from above analysis that under the applied stress at 870 °C the deformation feature of the alloy is the movement of 1/2<110> dislocations on the octahedral slip systems in the matrix channels. The dislocation networks in matrix result from the reaction of two sets dislocations with different Burgers vectors during mid-term fatigue stages.

2.3 Interaction of dislocation with the ordered γ' phase

In general, there are a large number of dislocations in the matrix in deformed specimens of the single crystal nickel-base superalloy. However, during the end stage of the fatigue test the γ' precipitates are occasionally sheared. A micrograph from a sample which was tested to fracture at 870 °C/550 MPa (N=1 789 000 cycles) is shown in Fig.5a. The slip bans of a new type of dislocation structure are observed. At the arrow a long and straight persistent slip bands (PSBs) penetrates in a single horizontal channel spanning across a number of γ' particles. The PSBs seem to be filled with large amount of dislocations. The γ' precipitates have also coarsened to some extent. With the rise of stress amplitude, the plastic deformation is quite inhomogeneous in the form of asymmetric distribution of dislocations. For the specimen deformed cyclicity at 690 MPa for 92 000 cycles, some shearing of the γ' precipitates also occurs, according to a mechanism that seems to produce extrinsic stacking faults. This type of fault is generally expected for this alloy after dynamic compression [10]. Fig.5b shows the clustering of dislocations in the matrix (lower right corner) and the formation of an extrinsic stacking fault. The clustered dislocation networks roughly align along the direction 45° to stress axis and the orientation of the extrinsic stacking fault is also parallel to the same direction also. It is fascinating to note that the strain induces a phase transformation in the specimen fatigued at 500 MPa for 50 000 cycles, as shown in Fig.6, where a large number of fine globular γ' precipitates are formed in the alloy matrix. It is suggested that the fine globular γ' particles would retard the dislocation bowing by preventing the dislocations from gliding in the matrix channel.

3 Discussion

The dislocation structure observed during initial and mid-term fatigue stages at 870 °C involves bowing dislocation at a

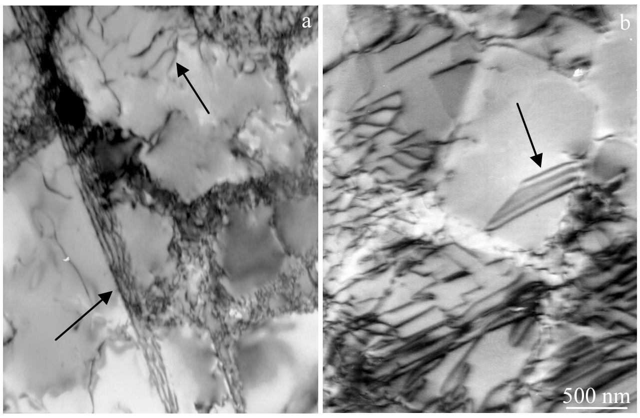

Fig.5 Micrographs of dislocation configuration after HCF tested at 870 °C under 550 MPa (a) and 690 MPa (b)

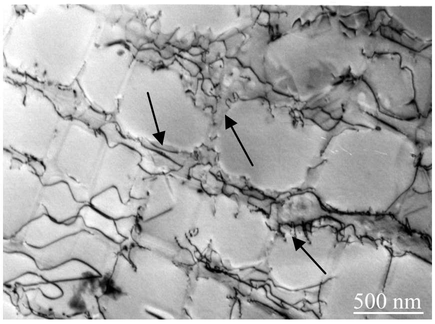

Fig.6 Reprecipitation of γ' phases induced by strain and dislocation under 870 °C/500 MPa

defined (111) interface, hexagonal dislocation networks. As the cyclic number is increased, the dislocation density in the matrix channel is increased and the dislocation networks tend to become homogeneously arranged. The homogeneous distribution of the interfacial dislocations in the alloy can effectively prevent the glide dislocations from cutting the γ/γ' rafted structure. Repeated cross slip of interfacial screw dislocations during fatigue cycles (Fig.3b, 3c), which were produced by the Orowan by-passing mechanism [11], on two {111} planes was also observed exclusively in [110] orientation as schematically shown in Fig.7. An expanding dislocation loop in a matrix channel deposits long segments with screw segments are glissile by cross slip to a second {111} type slip plane. The mechanism is possible due to equal shear stresses on the two possible {111} slip planes of the screw dislocation segments and explains the multiple loops bowing through the channels on {111} planes (Fig.3a~~-~~ 3c).

The dislocation PSBs and cutting of the γ' particles structures still have a close relation to the damage of the specimen at the end fatigue stage. It is found that the γ' particles have significantly coarsening and irregularly arrange after a long-time fatigue deformation, and the dislocation

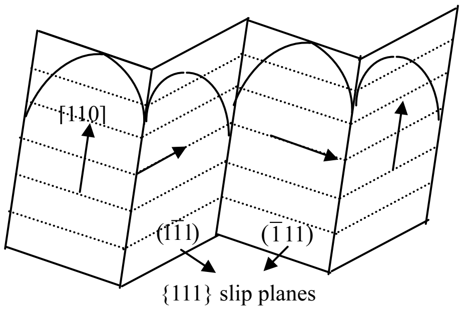

Fig.7 Schematic sketch of repeated cross slip of interfacial screw dislocation in {111} plane

coplanar slip is inhibited because of the lost coherence. This leads to a formation of the persistent slip bands running through the γ matrix and the γ' particles. Consequently, at the sites of the PSBs during fatigue deformation, once the local stress increases, this stress concentration would not be relaxed because the noncoplanar slip is difficult to operate. This promotes the initiation and early propagation of fatigue cracks. Another type shearing of the γ' particles also occurs (Fig.5b) under higher stress amplitude, stacking fault associated with the clustering of dislocations in the matrix and the loss of coherency of γ' precipitates with γ matrix[10-12]. It is evident that the γ' precipitate can efficiently obstacle the glide of dislocations over a long distance and enhance the fatigue strength.

4 Conclusions

1) During the HCF of the alloy at 870 °C, the fatigue strength is 443 MPa, and applied stress amplitude has a significant effect on the fatigue strength. With the rise of loading level, the fatigue strength decreases.

2) The fatigue deformation mechanism is closely related to temperature and stress Level. In the early and mid-term stages of high cycle fatigue test of the alloy at 870 °C, the main deformation feature of the alloy is the dislocation movement in matrix. Two sets of 1/2<110> fatigue dislocations with different Burgers vector move to the same slip planes to form the three-dimension networks by reaction.

3) Another feature of dislocation structures produced in the end fatigue stage is the formation of persistent slip bans moving through the γ matrix channels and the γ' precipitates. The interaction of the cyclic plastic deformation and high temperature induce the formation of the globular fine secondary γ' phase in matrix channel, which is beneficial to suppress dislocation moving in the matrix and enhance the fatigue strength.

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一种镍基单晶高温合金 870 ℃高周疲劳变形微观结构研究

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摘　要：研究了一种镍基单晶高温合金在 870 ℃时的高周疲劳性能及其变形组织结构。结果表明：该合金的疲劳寿命随着应力水平的升高而减小，870 ℃时光滑试样的疲劳强度为 443 MPa；利用透射电镜（TEM）观察疲劳循环试样的位错组态，发现在疲劳变形的初始和中期阶段，位错组态主要为界面位错，位错在基体通道中{111}面运动，并交互反应形成三维位错网络结构。当应力水平提高到 550 MPa 以上时，在变形的末期，观察到高密度位错集中于位错滑移带及位错切入γ'相现象。在循环应力和高温叠加作用下，基体通道中诱发析出大量圆形细小二次γ'相。二次γ'相的析出有益于阻止基体位错的滑动，抑制位错切入γ'相，有利于提高合金的疲劳强度。

关键词：镍基单晶高温合金；高周疲劳；位错

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