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# Effects of stacking fault energy on the creep behaviors of Ni-base superalloy

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#### **ABSTRACT**

Cobalt in a 23 wt.% Co containing Ni-base superalloys was systematically substituted by Ni in order to study the effects of stacking fault energy (SFE) on the creep mechanisms. The deformation microstructures of the alloys during different creep stages at 725 °C and 630 MPa were investigated by transmission electron microscopy (TEM). The results showed that the creep life increased as the SFE decreased corresponding to the increase of Co content in the alloys. At primary creep stage, the dislocation was difficult to dissociate independent of SFE. In contrast, at secondary and tertiary creep stages the dislocations dissociated at  $\gamma/\gamma'$  interface and the partial dislocation started to shear  $\gamma'$  precipitates, leaving isolated faults (IFs) in high SFE alloy, while the dislocations dissociated in the matrix and the partials swept out the matrix and  $\gamma'$  precipitates creating extended stacking faults (ESFs) or deformation microtwins which were involved in diffusion-mediated reordering in low SFE alloy. It is suggested that the deformation microtwinning process should be favorable with the decrease of SFE, which could enhance the creep resistance and improve the creep properties of the alloys.

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# 1. Introduction

Ni-base superalloys are an important class of high temperature structural materials, which have been widely used as turbine blades and discs in aircraft engines and industrial gas engines owing to their excellent high temperature mechanical properties, such as good tensile and creep properties  $[1,2]$ . In order to increase the performance of the engines, the engineers explore more challenging materials by improving their creep resistance at elevated temperature. Therefore, the high temperature creep deformation mode is a major concern for the researchers to design new alloys. As we know, Ni-base superalloys mainly consist of  $\gamma$  matrix and  $\gamma'$  $(Ni<sub>3</sub>Al)$  precipitates which are coherent, ordered L1<sub>2</sub> crystal structure embedded in the disordered solid-solution face centered cubic (fcc)  $\gamma$  matrix. In Ni-base superalloys the  $\gamma'$  precipitates contribute to their unique high temperature creep properties through acting as effective barriers to the moving dislocations. Dislocation bypassing  $\gamma'$  precipitates via Orowan loop, cooperative climbing, and dislocation shearing  $\gamma$  precipitates [\[3,4\]](#page--1-0) are three main processes during creep deformation depending on microstructure, temperature and loading stress. When dislocations shear  $\gamma$  precipitates during plastic creep deformation, there will produce different high energy configurations such as anti-phase boundary (APB) and complex stacking fault (CSF) which are energetically unstable. These high energy configurations would convert into superlattice stacking fault (SSF) (including superlattice extrinsic stacking fault (SESF) and superlattice intrinsic stacking fault (SISF)) which possess lower energy in  $\gamma'$  precipitates during creep deformation [\[5\].](#page--1-0) Deformation microtwin running across the whole grain could also be created after the shearing of  $\gamma'$  precipitates during creep deformation [\[6–8\]](#page--1-0).

The previous studies  $[6-11]$  indicated that Ni-base superalloys retained high creep resistance depending on several microstructural factors, including the volume fraction, particle size, distribution of  $\gamma'$  precipitates, and the chemical composition of  $\gamma'$ precipitates and the matrix. In polycrystalline Ni-base superalloys, the grain sizes also played a vital role in affecting their high temperature creep properties [\[12,13\]](#page--1-0). Furthermore, SFE affected by Co content also had crucial effect on the creep deformation of Nibase superalloys [\[14,15\]](#page--1-0). However, due to the complicated chemical compositions of Ni-base superalloys, the creep deformation mechanisms might be influenced by other compositions and  $\gamma'$ content in the studies [\[14,15\].](#page--1-0) Thus, it is necessary to investigate the influence on the creep mechanisms by SFE relating to Co content when other compositions and  $\gamma'$  content are relatively stable in Ni-base superalloys.





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In our previous study [\[16\]](#page--1-0), the SFE of Ni-base superalloys with 5 wt.%, 15 wt.% and 23 wt.% Co content were measured to be 40.1, 33.3 and 24.9 mJ/m<sup>2</sup>, which indicated that the SFE decreased with the increase of Co content in the alloys under certain conditions. The result was in agreement with the previous studies [\[17–20\].](#page--1-0) Then in the present paper the three alloys respectively marked as Alloy1, Alloy2 and Alloy3 were prepared in order to study SFE on the creep mechanisms at 725 °C/630 MPa.

## 2. Experimental details

The nominal compositions of three Ni-base superalloys for creep tests are listed in Table 1. For all tested three alloys, 20-kg ingots were cast using vacuum induction melting (VIM). These ingots were then hot extruded into 35 mm bars at about 1160 °C. The extruded samples were heat-treated at 1100 °C/4 h (air cooling) followed by aging at 650 °C/24 h (air cooling) and 760 °C/ 16 h (air cooling). Constant load tensile creep experiments were performed at 725 °C/630 MPa. The samples before creep tests for optical microscopy (OM) were etched in a solution of modified Kalling reagent (100 ml HCl, 100 ml methanol and 50 g CuCl<sub>2</sub>). TEM disks with thickness of about 300 µm were cut from the samples perpendicular to the stress axis. Then the disks were manually ground down to 50 µm and perforated by twin-jet electro-polisher in a solution of 10% perchloric acid and 90% ethanol at about 16 V and –20 °C. TEM observations were carried out on a JEOL 2100 TEM operated at 200 kV.

# 3. Results

#### 3.1. Initial microstructures before creep tests

[Fig. 1](#page--1-0) shows the heat-treated microstructures of the alloys with various Co contents. It can be seen that all the alloys had a similar grain size of about 35  $\mu$ m, as shown in [Fig. 1\(](#page--1-0)a)–(c). The high magnification microstructures of the three alloys all revealed a sparse distribution of primary  $\gamma'$  precipitate ( $\geq$ 300 nm), secondary  $\gamma'$  precipitate (about 100 nm) and tertiary  $\gamma'$  precipitate (typically less than 30 nm) which were shown in [Fig. 1\(](#page--1-0)d)–(f). The  $\gamma'$  volume fractions of the three alloys calculated by Thermo-Calc software were about 46% at 760 °C. These results indicated that altering Co content had little effect on the phase constituents and microstructures of the alloys, which were well consistent with the previous studies [\[21,22\]](#page--1-0).

## 3.2. Creep curves

[Fig. 2](#page--1-0)(a) shows the creep curves of three alloys tested at 725 °C/ 630 MPa. The curves consisted of primary, secondary and tertiary creep and most of the creep strain accumulated in the tertiary stage. Obviously, the rupture life for the three alloys increased with the decrease of SFE in the figure. The result agreed with the previous work by Jarrett and Tien [\[23\]](#page--1-0) who concluded that the creep rupture life decreased sharply when Co content was reduced below about eight weight pct in the disk alloys.

[Fig. 2](#page--1-0)(b) displayed creep strain rate vs time for three alloys. The curves exhibited that both the primary and secondary creep stages

Table 1 Nominal chemical compositions of the three alloys (wt.%).

				Alloy Ni Co Al Ti W Mo Cr C B		Zr
					Bal. 5 2.3 5.6 1.2 2.8 14 0.02 0.015 0.03	
					Bal. 15 2.3 5.6 1.2 2.8 14 0.02 0.015 0.03	
					Bal. 23 2.3 5.6 1.2 2.8 14 0.02 0.015 0.03	

prevailed longer as the SFE of the alloy decreased. It also could be found that the steady creep rate of the three alloys decreased with the decrease of SFE in the figure.

#### 3.3. Microstructures after creep tests

In order to fully understand the creep deformation mechanisms of the three alloys, the creep tests for Alloy1 and Alloy2 were interrupted at 2 h and 20 h while the creep tests for Alloy3 were interrupted at 2 h and 50 h. The deformation microstructures of the interrupted samples at shorter and longer time respectively represented the microstructures of primary creep stage and secondary creep stage for three alloys according to the curves in [Fig. 2\(](#page--1-0)b). The TEM samples for the two stages were cut from the middle of the interrupted specimens. The deformation microstructures related to the tertiary creep stage were investigated by cutting TEM samples near the fracture surface after creep tests.

#### 3.3.1. Alloy1 with high SFE

At primary creep stage, in most grains the dislocation activities in  $\gamma'$  precipitates could be observed scarcely, as shown in [Fig. 3](#page--1-0)(a). As we know, at this stage the plastic strain is mainly accumulated to the matrix because the strain is very small, meanwhile the creep strain rate is relatively higher due to the motion of the perfect a/2  $\langle 110 \rangle$  matrix dislocations in the matrix. The effective stress is sufficient to lead the  $a/2 \langle 110 \rangle$  matrix dislocations to slip in the matrix and bow around  $\gamma$  precipitates. But in few grains IFs were formed in primary  $\gamma$  precipitates because these grains had enough effective stress due to their grain orientation to force partial dislocations to shear primary  $\gamma'$  precipitates, as shown in [Fig. 3\(](#page--1-0)b).

At secondary creep stage, numerous IFs were observed in primary  $\gamma'$  precipitates and IFs started to be formed in secondary  $\gamma'$ precipitates, as shown in [Fig. 3](#page--1-0)(c). The formation of the stacking faults (SFs) morphology at this stage was related to the increase of the effective stress caused by the reduction of cross section area.

At tertiary creep stage, the density of IFs in primary and secondary  $\gamma$  precipitates became relatively higher in [Fig. 3](#page--1-0)(d). In some primary  $\gamma'$  precipitates, IFs were operative on two different  $\{111\}$  slip planes, as illustrated in [Fig. 3](#page--1-0)(e). As the creep strain continued to increase, more and more  $\gamma'$  precipitates were sheared and even some  $\gamma'$  precipitates were repeatedly cut. Lots of shearing processes would sharply accelerate the creep rate and cause the specimen to rupture in short time.

#### 3.3.2. Alloy2 with intermediate SFE

At primary creep stage, in most grains the SFs were not observed in  $\gamma'$  precipitates, as shown in [Fig. 4\(](#page--1-0)a). However, in few grains there were two different types of SFs: one was IF which was similar to the SF in Alloy1, as shown in Fig.  $4(b)$ . The other cut through  $\gamma'$  precipitates and the matrix as shown in [Fig. 4](#page--1-0)(c), generally called ESF. At secondary creep stage, IFs and ESFs were also found and their densities became larger because more  $\gamma$  precipitates were sheared, as shown in Fig.  $4(d)$ . Moreover, the deformation microtwins were observed in some grains, as illustrated in [Fig. 4](#page--1-0)(e). At tertiary creep stage, three different deformation modes including IF, ESF and deformation microtwin were also observed, as shown in Fig.  $4(f)$ –(h), but microtwinning was the dominant deformation mode. Besides the densities of the SFs and deformation microtwins both increased at tertiary creep stage.

## 3.3.3. Alloy3 with low SFE

At primary creep stage, the deformation microstructures were similar to that of this stage of Alloy2. But IFs could not be observed, as shown in [Fig. 5\(](#page--1-0)a) and (b). At secondary creep stage, both ESFs and deformation microtwins were found in the deformation microstructures, as shown in Fig.  $5(c)$  and (d). At tertiary creep stage,

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