

## Isothermal oxidation behavior of single crystal superalloy DD6

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**Abstract:** The isothermal oxidation behavior of the second generation single crystal superalloy DD6 was studied at 1050 °C and 1100 °C in ambient atmosphere. Morphology of oxides was examined by SEM and their composition was analyzed by XRD and EDS. The experimental results show that DD6 alloy obeys subparabolic rate law during oxidation of 100 h at 1050 °C and 1100 °C. The oxide scale exposed at 1050 °C is made up of an outer NiO layer with a small amount of Al<sub>2</sub>O<sub>3</sub> and an inner Al<sub>2</sub>O<sub>3</sub> layer. The oxide scale exposed at 1100 °C is made up of an outer Al<sub>2</sub>O<sub>3</sub> layer with a small amount of NiO, an intermediate layer, mainly composed of Cr<sub>2</sub>O<sub>3</sub> and TaO<sub>2</sub>, and an inner Al<sub>2</sub>O<sub>3</sub> layer. The  $\gamma'$ -free layer was formed under the oxide scale at two temperatures.

**Key words:** single crystal superalloy; DD6 alloy; high temperature oxidation

### 1 Introduction

Nickel-base single crystal superalloys have been widely used as turbine blade materials for advanced aircraft engines, owing to their superior performance which includes both mechanical strength and oxidation resistance. At high temperature, the oxidation resistance behavior of the materials becomes one of the major life-limiting factors [1]. It is usual to protect the alloy using coating technology. However, for various reasons, it is not always possible to apply coating to the superalloys [2]. And it is necessary to know the intrinsic behavior of the substrate in oxidizing atmosphere. Therefore study on the oxidation behavior of single crystal superalloys becomes important. There have been several studies on the oxidation behavior of single crystal superalloys in recent years [3–7].

DD6 alloy was developed for aeroengine turbine blade applications by Beijing Institute of Aeronautical Materials, China. The properties of the alloy are equivalent to and more than those of other second generation single crystal superalloys widely used in the world [8]. DD6 alloy has an approximate 40 °C improvement of creep strength relative to the first generation single crystal superalloy DD3 and can be made into complex shaped hollow turbine blades which work under 1100 °C [9]. In this study, the high

temperature oxidation behavior of DD6 alloy was investigated at 1050 °C and 1100 °C. Post-oxidation investigations were carried out to characterize the microstructures and compositions in the oxide scales.

### 2 Experimental

The nominal chemical compositions of DD6 alloy are shown in Table 1. The single crystal superalloy rods with [001] orientation were cast by means of crystal selection method in a directionally solidified furnace with high temperature gradient. The heat treatment of the specimens was carried out according to following heat treatment regime: (1290 °C, 1 h)+(1300 °C, 2 h)+(1315 °C, 4 h, AC)+(1120 °C, 4 h, AC)+(870 °C, 32 h, AC). The square specimens with the dimensions of 30 mm×10 mm×1.5 mm were machined from the alloy rod by an electro-spark machine. The surface of the specimens was ground with SiC abrasive paper (1000 grid), and ultrasonically cleaned in acetone and ethanol, respectively.

The isothermal oxidation tests were conducted at 1050 °C and 1100 °C in static air. During the oxidation the specimens were cooled to room temperature at an interval of 25 h for mass measurement by an electrobalance with the precision of 0.1 mg. Three specimens were used to obtain the average of the mass gain. The surface and cross sectional morphology of oxides were examined by scanning electron microscopy

with energy dispersive X-ray analysis (SEM/EDX) and their phase composition was analyzed by X-ray diffraction (XRD).

**Table 1** Nominal chemical compositions of DD6 alloy (mass fraction, %)

Cr	Co	Mo	W	Ta	Re	Nb	Al	Hf	Ni
4.3	9	2	8	7.5	2	0.5	5.6	0.1	Bal.

### 3 Results and discussion

#### 3.1 Oxidation kinetics

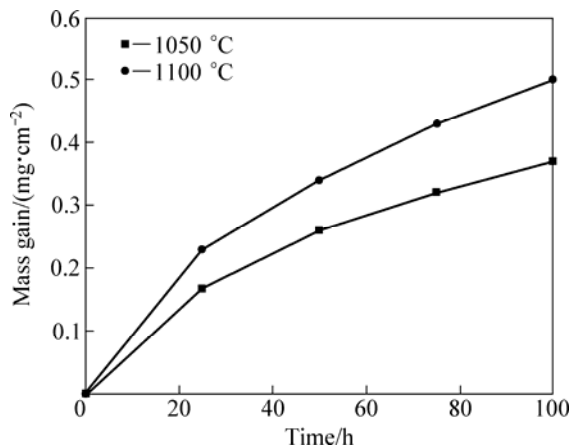
The isothermal oxidation kinetics of DD6 alloy at 1050 °C and 1100 °C is shown in Fig.1. The results show that oxidation resistance of the alloy at 1050 °C is better than at 1100 °C. It can be seen that the oxidation speeds up in initial period of 25 h. This is the formation period of oxide film. Then, the oxidation speeds down, and enters the oxide film growth period. During the oxidation process, a surface reaction changes into diffusion gradually. The kinetics can be described by examining the growth rate—time constant, which is found as the exponent in the following rate equation [10]:

$$(\Delta m/A)=Kt^n+C \quad (1)$$

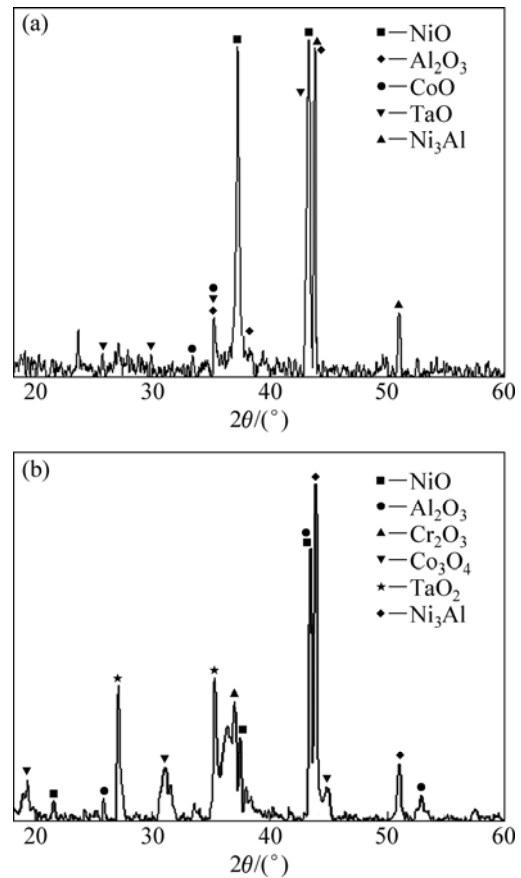
where  $\Delta m$  is the mass change,  $A$  is the specimen surface area,  $K$  is the oxidation rate constant,  $t$  is the exposure time, and  $C$  is a constant. The slope of the  $\lg$ — $\lg$  plot of the above equation gives the  $n$  value, which provides an indication of the oxidation mechanism. The  $n$  values of about 0.21 and 0.26 were obtained for the alloy oxidation at 1050 °C and 1100 °C, respectively, indicating a subparabolic time dependence of the growth rate. Similar oxidation kinetics has been reported for other single crystal superalloys [1, 10].

#### 3.2 Phase constitution of oxidation layer

Figure 2 presents the XRD patterns of the surface of



**Fig. 1** Isothermal oxidation kinetics curves of DD6 alloy samples at 1050 °C and 1100 °C in air



**Fig. 2** XRD patterns of DD6 alloy samples after oxidation for 100 h at different temperatures: (a) 1050 °C; (b) 1100 °C

DD6 alloy after 100 h oxidation at 1050 °C and 1100 °C, respectively. After oxidation for 100 h at 1050 °C, the oxidation products formed on the alloy are mainly NiO, Al<sub>2</sub>O<sub>3</sub>, CoO and TaO. After oxidation for 100 h at 1100 °C, the oxidation products formed on the alloy are mainly NiO, Al<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, Co<sub>3</sub>O<sub>4</sub> and TaO<sub>2</sub>. Compared with the oxidation products at 1050 °C, the Cr<sub>2</sub>O<sub>3</sub> is detected and the TaO<sub>2</sub> and Co<sub>3</sub>O<sub>4</sub> are in place of TaO and CoO in the oxide layer at 1100 °C.

#### 3.3 Morphologies of oxide scale

Figure 3 shows SEM images of surface of DD6 alloy samples oxidized for 100 h at 1050 °C and 1100 °C. It can be seen that the surface morphology of the scale shows considerable difference at different temperatures. The blocky morphology of oxides at 1050 °C tightly piles each other on the surface. No spallation of the scale is observed on the surface. The fine flaky oxides at 1100 °C loosely pile each other. And several cavities are clearly visible. Locally, a little spallation is found on the oxide scale. The compositions of the oxide layer measured by EDS are shown in Fig. 4. According to EDX analysis, O, Ni, Al, Co and Ta are the major constituents of oxide layers at 1050 °C and 1100 °C. This is in accordance with the XRD results.

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