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The effects of ruthenium additions on tensile deformation mechanisms of single crystal superalloys at different temperatures

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ABSTRACT

The tensile behavior of two experimental nickel-base single crystal superalloys has been studied from room temperature to 1100 °C. Emphasis is placed on elucidating the effects of ruthenium (Ru) additions on the deformation mechanisms using transmission electron microscopy (TEM). Furthermore, the partitioning behavior of the alloy elements between the γ and γ′ phases for both experimental alloys has been studied using three-dimensional atom probe (3DAP). Detailed analysis demonstrates that at low and medium temperature ranges, the stacking faults present in the γ matrix of the 3Ru alloy but no trace of stacking fault in the γ matrix of the 0Ru alloy have been observed; during high temperature range, as a result of Ru additions, the γ/γ′ interfacial dislocation space of the 3Ru alloy is smaller than that of the 0Ru alloy due to further decreasing the lattice misfit. Apart from that, Ru additions result in more Re partitioning to the γ′ phase, and thus the solution strengthening for the γ phase is decreasing. Thus, during tests below and at the temperature corresponding to the peak strength, the yield strength of the 3Ru alloy is lower than that of the 0Ru alloy. At last, in the light of the TEM observations, the changing trends of the stacking fault energy in the γ matrix and the transformation points (the temperature related to the stacking faults formation) for the two experimental alloys have been drawn. The temperature range of the stacking faults formation in the γ matrix is expanded after Ru additions. The energy conditions of the stacking faults formation of the 0Ru and 3Ru alloys have been analyzed in detail. The changing of lattice misfit with temperature can be considered as one of the principal reasons for the stacking faults formation.

1. Introduction

Nickel-base single crystal superalloys have been widely used for structural components in modern aircraft to withstand ever more arduous conditions of high temperature and loading due to their excellent mechanical performance [1–3]. These alloys contain a high volume fraction (about 65%) of face-centered cubic (fcc) ordered gamma-prime (γ′, Ni_3Al) precipitates with $L1_2$ structure type coherently embedded in a disordered fcc gamma (γ) solid solution. It is well known that the matrix and the precipitates have a cube/cube orientation relationship. As a result of a small difference of the lattice parameter between the two phases, the misfit stresses are present in these superalloys [4]. In addition, it is generally considered that the γ′ precipitate is largely responsible for the strength of the alloys and of vital importance for improving resistance to deformation [5–8]. Nevertheless, as the origin area of the dislocations, the γ matrix also has a significant effect on the deformation. In fact, the mechanical properties of nickel-base single crystal superalloys depend strongly upon the state of the microstructure, which, in turn, is controlled by the chemical composition and heat treatment conditions.

The chemical elements, such as Cr, Co, Mo, W, and Ta have been added into the alloys to improve the deformation resistance at elevated temperatures [9]. To further improve the overall performance of the single crystal superalloys, more refractory elements have also been added, such as Re and Ru. The second-, third- and fourth-generation single crystal superalloys contain about 3, 6 and 6 wt% Re, respectively [9–13]. On the other hand, in order to enhance phase stability 3 wt% Ru has been added to the fourth-generation superalloys [6,10,14–17]. The elements of Cr, Co, Mo, Re and Ru have a preference to partition into the γ matrix and dramatically improve the strength of the γ disordered solid solution have been suggested in early works [18–20]. Recently, some studies of the effect of Ru additions on mechanical proprieties have been carried out [10,16,21–23]. The results show that creep resistance of these superalloys has been potentially increased. Tensile test is a basic test for superalloys, the results can be seen as an

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important reference for creep and fatigue. However, the work focusing on the effects of Ru additions on the tensile behavior has been rarely reported previously and the deformation mechanisms of tensile test remain incomplete. Thus, in order to further investigate these effects, two single crystal superalloys of Ru-containing (3Ru) and Ru free (0Ru) have been prepared. Detailed studies on the microstructure evolution at each temperature have been carried out through TEM analysis and the role of Ru has been established.

2. Experimental details

The nominal chemical compositions of the two superalloys are listed in Table 1. The 3Ru alloy is nominally identical to the 0Ru alloy in compositions with the exception of 3 wt% Ru, which was substituted for nickel. Polycrystalline ingots of the experimental compositions were directionally solidified into single crystal bars along the [0 0 1] direction at a constant withdrawal rate of 6 mm/min using the Bridgman method. The two experimental alloys have nearly the same incipient melting point, thus, the same heat treatments were carried out. The two steps solution treatment given to the alloys consisted of 1315 °C for 16 h plus 1325 °C for 16 h, followed by air cooling. The two steps aging treatment given to the rods were 1150 °C for 4 h, followed by air cooling and 870 °C for 24 h, followed by air cooling.

After heat treatment, the samples for scanning electron microscopy observation were mechanically polished and etched in a solution of 20 g $CuSO_4$ + 100 ml HCl + 5 ml H_2SO_4 + 80 ml H_2O. Examinations of the alloys by an Inspect F50 field emission scanning electron microscope (FESEM), as shown in Fig. 1, indicated the volume fraction is approximately 70% and average size of the γ' precipitates is approximately 300 nm. Here, it should be noted that the standard deviation for the 0Ru and 3Ru alloys is about 0.070 μm and 0.066 μm, respectively. The partitioning behavior of the other alloy elements between the γ and γ' phases is changed after addition of Ru and potentially influences the tensile behavior. Consequently, 3DAP analysis was conducted for the two experimental alloys. Square rods of approximately 0.2 mm × 0.2 mm × 10 mm were cut from the post heat treated single crystal bars of the two experimental alloys along the [0 0 1] direction. These small rods were then electropolished to sharp, needle shaped specimens for the 3DAP analysis.

The cylindrical specimens for tensile test, with gauge length and diameter of 25 mm and 5 mm, were machined from the single crystal bars. The tensile tests for both alloys were carried out at room temperature (RT), 600 °C, 760 °C, 900 °C, 1000 °C and 1100 °C along the [0 0 1] direction to rupture. A constant strain rate of 1.67×10^{-3} s^{-1} was used in this study.

After the tensile test the ruptured samples were cut into thin foils (about 0.6 mm in thickness) perpendicular to the longitudinal axis using spark erosion wire cutting machine. The foils, for TEM observation, were mechanically ground to a thickness of approximately 50 μm, and then electrochemically thinned using a twin jet polisher with a solution of 10% perchloric acid and 90% ethanol by volume. The optimum jet polishing conditions were determined to be a current of 30 mA and temperature of −25 °C. The resulting foils were examined using a JEM 2100 transmission electron microscope operating at 200 kV.

Table 1
Nominal chemical compositions (wt%) of the experimental superalloys investigated.

Alloy	Co	Al	Cr + Mo + W + Ta	Re	Ru	Ni
0Ru	12	6	19.4	5.4	0	Bal.
3Ru	12	6	19.4	5.4	3	Bal.

3. Results and discussion

3.1. Tensile curves

Fig. 2 shows the tensile engineering strain–stress curves of the two experimental alloys at different temperatures. At first glance, the overall trends of the curves of the 0Ru alloy (Fig. 2a) are similar to those of the 3Ru alloy (Fig. 2b). After close inspection, the curve at room temperature of the 0Ru alloy is different from that of the 3Ru alloy. The stress jumps (steps) present in the 3Ru alloy at room temperature tensile test whereas no steps present in the 0Ru alloy. What interests us is that, at 600 °C, the steps present both in the 0Ru and 3Ru alloys. At 760 °C, a strong work hardening but poor plastic deformation has been expressed by the tensile curves. It is noteworthy that the maximum yield strengths of the two alloys both appear at 900 °C. Discussions about the yield strength have been carried out in detail in the next paragraph. Indeed, for the 3Ru alloy, a slight work hardening occurs during tensile test at 1000 °C, whereas it is not the case for the 0Ru alloy. A significant softening was observed during tensile test at 1100 °C for the two alloys. This different tensile behavior of the two alloys might have a close relationship with Ru element. The detailed analysis has been carried out in the following sections.

Fig. 3 shows the variation of the yield strength of two experimental alloys with temperature. It should be noted that the yield strengths of the two alloys at different temperatures are the value of $\sigma_{0.2}$. In fact, before the significant yield points appearing the alloys have already yielded (the difference between $\sigma_{0.2}$ and significant yield point is not significant except of the 3Ru alloy tensile test at room temperature). An abnormal yield behavior is observed from the yield strength of the 0Ru and 3Ru alloys. In other words, the stress necessary for the onset of plastic deformation by dislocation slipping does not decrease significantly with increasing temperature, as is the case for most other alloy systems. Conversely, for many superalloys the yield strength increases with increasing temperature, typically until the temperature of about 800 °C is reached [24]. The peak strength of two experimental alloys at 900 °C is reached, as shown in Fig. 3, which is slightly higher than other superalloys [24]. It should be noted that the yield strength of two experimental alloys slightly decreases from room temperature to 600 °C. Above 600 °C, the yield strength remarkably increases until the peak value. Beyond the temperature corresponding to the peak strength (named peak temperature), the yield strength decreases remarkably. The dislocation cross-slip from the {1 1 1} to the {0 0 1} plane is considered to be the root cause of the positive temperature dependence of the yield strength. This mechanism is the so called Kear–Wilsdorf lock [25–27]. Beyond the peak temperature, it is considered that a preference for the thermally activated slip on the cube plane is responsible for the softening. In fact, below the peak temperature, the yield strength of the 0Ru alloy is higher than the 3Ru alloy. Beyond this temperature, the case is contrary. This difference might be correlated with the addition of Ru and the detailed discussion has been carried out in Section 3.3.

3.2. Deformation microstructures

Fig. 4 shows the morphology of the stacking faults in the 0Ru and 3Ru alloys after tensile tests at room temperature. A remarkable difference of the microstructures between the two alloys is some stacking faults present in the γ matrix of the 3Ru alloy but no traces of them have been observed in the γ matrix of the 0Ru alloy. Thus, it can be confirmed that the stacking fault energy of the γ matrix is significantly reduced by the addition of Ru. Another noteworthy observation is that the stacking faults presenting in the γ' precipitates of the 3Ru alloy are thicker than those of the