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Effect of ruthenium on γ' precipitation behavior and evolution in single crystal superalloys

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Abstract: The effect of Ru on γ' precipitation behavior and evolution in single crystal superalloys with different Ru contents were investigated by scanning electron microscopy with energy dispersive spectroscopy, 3D atomic probing, differential scanning calorimetry. The results show that the solvus of the γ' phase decreases gradually with increasing Ru content in the alloys by casting or by the same solution and aging treatments, the alloy with a larger Ru content yields a smaller γ' phase. The addition of Ru increases the growth rate and coarsening rate of the γ' phase. Ru mainly distributes in the γ phase, which causes more Re and Mo partition into the γ' phase, increasing the absolute value of mismatch and the rafting rate of the γ' phase.

Key words: single crystal superalloy; ruthenium; gamma prime; precipitation; growth rate; coarsening rate; mismatch

1 Introduction

The excellent high-temperature performance of the Ni-based single superalloys is mostly due to the strengthening precipitates, thus, the control of the amount, size, morphology and distribution of phases to improve the strength and plasticity of alloys by heat treatment and tailoring alloy compositions is critical [1−3]. Generally, the volume fraction of the γ' phase is 60%−70% in a single crystal superalloy. The outstanding high-temperature mechanical properties of alloys benefit from the addition of a number of refractory alloying elements. Therefore, γ' precipitation behavior and evolution obtained by adding alloying elements are important for improving the temperature capability of alloys.

Many researchers have studied the effect of Ru on the microstructure of single crystal superalloys; however, there are some controversial points. For example, MA et al [4] found that 3% Ru content has no effect on the suppression of the topological closed packed (TCP) phase of single crystal superalloys with high contents of refractory alloying elements. It was pointed out that Ru could suppress the precipitation of the TCP phase [5,6]. Some researchers [7−9] indicated that the partition behavior of alloying element is not affected by the addition of Ru. CARROLL et al [10] maintained that Ru causes matrix- forming element partitioning in the γ' phase. Reports on the evolution of the γ' phase in Ru-containing single crystal superalloys are infrequent. Therefore, in the present work, the effects of Ru on γ' the precipitation behaviors and evolution in single crystal superalloys are investigated in detail.

2 Experimental

The experimental single crystal superalloys used in the current investigation included three alloys containing different Ru contents (0, 1.5%, and 3% in mass fraction). The nominal compositions of tested alloys are listed in Table 1. The three alloys were designated as 0Ru alloy, 1.5Ru alloy, and 3Ru alloy, respectively. Single crystal bars with dimensions of d16 mm×160 mm were produced in the ⟨001⟩ direction by the screw selection method, with a mould drawing rate of 6 mm/min in a directional solidification vacuum induction furnace. Differential scanning calorimetry (DSC) was performed on the samples to determine the transformation temperatures in the as-cast and heat-treated samples. To investigate the effect of heat treatment on the γ' phase

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Table 1 Nominal compositions of tested alloys

Sample	w(Al)/%	w(Ta)/%	w(Mo)/%	w(W)/%	w(Cr)/%	w(Co)/%	w(Re)/%	w(Ru)/%	w(Ni)/%
0Ru alloy	6	8	1	6	5	12	5	0	Bal.
1.5Ru alloy	6	8	1	6	5	12	5	1.5	Bal.
3Ru alloy	6	8	1	6	5	12	5	3	Bal.

evolution of the three alloys, different solution and aging treatments were proceeded. A 3-D atom probe (3DAP) was used to determine the alloying element partitioning between the γ and γ' phases in the heat-treated samples. In order to lower the alloy element segregation, the 3DAP samples were solution heat-treated for a long time shown in Table 2 and then aged at 1120 °C for 4 h and 899 °C for 20 h. Three samples were analyzed for each alloy. To study the γ' phase coarsening behavior at high temperatures, the specimens were exposed to a temperature of 1100 °C for 200 h. The γ' phase morphologies of the as-cast, heat-treated, and long-term exposed samples were analyzed by scanning electron microscopy (SEM). The mean sizes of the γ' particles were measured by quantitative metallography, and at least 500 γ' particles were measured under each condition. The average sizes of the samples (denoted by "a") were utilized. For cuboidal and global γ' particles, "a" was designated as the edge length or the diameter.

Table 2 Solution treatment conditions of three alloys with different contents of ruthenium

Sample	Solution heat treatment condition
0Ru alloy	(1325 °C, 16 h)+(1335 °C, 16 h)+air cooling
1.5Ru alloy	(1315 °C, 8 h)+(1325 °C, 16 h)+air cooling
3Ru alloy	(1315 °C, 8 h)+(1325 °C, 16 h)+air cooling

3 Results and discussion

3.1 Morphology of γ' phase in as-cast alloys

Under the same solidification condition, the three alloys with different Ru contents solidify as dendrite structures, which consist of dendrite core, dendrite arm, and interdendritic region. Some γ/γ' eutectics are observed in the interdendritic region of the three alloys. Figure 1 shows the γ' phases in the dendrite cores and interdendritic regions in three alloys in the as-cast state. In all three alloys, the size of the γ' phase in the dendrite core is smaller than that in the interdendritic region. The size of the γ' phase decreases slightly with increasing Ru content.

During the directional solidification under steady-state condition, the mushy zone is comprised of the single phase γ dendrites and the liquid phase. The alloying elements addition in the Ni-based superalloys tends to partitioning preferentially into either the γ phase or the γ' phase; thus, the limited solubility of γ'-forming elements exists within the single phase dendrites during the solidification, and elements such as Ta and Al are enriched in the liquid phase. The other alloying element addition, Re, W, Cr, Co, Mo, or Ru, is soluble in the γ phase and tends to partition preferentially into γ dendrites during the solidification. After the termination of solidification, the solid continues to cool. While cooling from the solidus temperature, the supersaturation of the γ phase with Al and Ta could occur as the solubility limits are exceeded. Thus, the γ' phase rich in Al and Ta would precipitate from the supersaturated γ phase.

Figure 2(a) shows the DSC heating curves of the three alloys in the as-cast state with different Ru contents. Three endothermic peaks in the heating curves of each alloy are observed. The first gentle peak near 1250 °C corresponds to the endothermic peak of the γ' phase solution. In single crystal superalloys, the γ/γ' eutectic is the last region to solidify, so the incipient melting is defined as the melting temperature of the γ/γ' eutectic, which corresponds to the second peak in the heating curves. The main peak shows the exothermic peak for the melting of the γ phase.

Due to the segregation of the alloying elements in the dendrite structure, the compositions of the γ' phase in the dendrite core and interdendritic region are not identical, and the precipitation or solution of the γ' phase occurs over a large temperature range. Moreover, the γ' phase precipitation from the γ matrix belongs to coherent precipitation, therefore, the transformation process needs less absorption of heat. Consequently, the slope of the endothermic peak of the γ' phase is gentle and almost unobservable in Fig. 2(a). To study the effect of Ru on the solution temperature of the γ' phase, fully heat-treated samples were analyzed by DSC, as shown in Fig. 2(b). The DSC curves show two endothermic peaks: one is for the solution of the γ' phase, and its peak value is listed in Table 3; the other is for the melting of the γ phase. From the position of the peaks, the solution temperature of the γ' phase decreases with increasing Ru content.

The earlier the γ' phase precipitates, the lower the γ' solubility limit is. From the above results, we can deduce that Ru increases the solubility limits of Al and Ta in the γ phase. In the 0Ru alloy, the γ' phase precipitates earliest, whereas the γ' phase in the 3Ru alloy precipitates latest. Because the γ'phase in the