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Solidification characteristics and as-cast microstructure of Rhenium-containing single crystal superalloys under high thermal gradient directional solidification

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Abstract. A series of Ni-base single crystal superalloys with increasing contents of Re (3-6 wt. %) were directionally solidified under a high thermal gradient of approximately 250 K/cm. It shows that Re additions slightly increases the liquidus temperature, but decreases the eutectic reaction and γ'-solvus temperature of the experimental alloys. EPMA analysis indicates that Re addition strongly influences the segregation ratios of the constituent elements, particularly for Cr, Mo and W, and thus results in more severe dendrite segregation and large amount of eutectic formation in the as-cast microstructures.

Introduction

As an important high-temperature structural material, Ni-base single crystal superalloys have been widely used in manufacturing the critical hot-section components of aero-engines and industry gas turbines (IGT). In order to promote the high temperature creep strength, single crystal alloys have been added with large amount of refractory elements, such as W, Ta and particular Re, for their great solid-solution strengthening effects [1, 2]. However, the additions of refractory elements with low diffusion coefficient also result in more severe dendrite segregation of the constituent elements. It will promote the detrimental topologically-close-packed (TCP) phase formation [3, 4], and increase the propensity for grain defects formation, such as stray grains and freckles chains [5, 6]. These undesirable grain defects exist in the as-cast microstructures can not be eliminated even by the complicated homogeneous heat treatments, thus degrading the mechanical properties of single crystal superalloys.

Nowadays, the "high rate solidification (HRS)" technique was the prevalent manufacturing process for turbine blade. Unfortunately, because of the relative low thermal gradient (< 100 K/cm) and limited range of withdrawal rates (3-6 mm/min), it was difficult to be used for casting the complicated large IGT blades. Many new technologies, such as gas cooling casting (GCC) [7], fluidized-bed quenching (FBQ) [8], and liquid metal cooling (LMC) [9] processes have been developed. Due to the great advantage over other methods for time consuming and casting quality improving, the most researches was focused on the LMC process for turbine blades casting. However, there are still few researches on the solidification characteristics and as-cast microstructure of single crystal alloys under high thermal gradient directional solidification.

In this investigation, a series of single crystal alloys were prepared, which contained increasing contents of Re (3-6 wt. %). The characteristic phase transformation temperature and segregation ratios of the constituent elements were investigated. Their effects on the as-cast microstructures under high thermal gradient were also discussed.

Experimental procedures

The chemical compositions of the experimental alloys used in this investigation were presented in Table.1. Due to the limited content, the influence of Hf on the as-cast microstructure can be neglected in this investigation. Cylindrical samples with 4 mm in diameter and 70 mm in length were directional solidified in the modified LMC Bridgman apparatus with <001> oriented seeds. A high thermal gradient of approximately 250 K/cm was obtained in this research, which can greatly attributed to the introductions of dual heating zones melting, carefully designed baffles and low melting liquid metal coolant.

Table 1: Compositions (in wt. %) of the experimental single crystal alloys used in this investigation

Alloys	Cr	Co	Mo	W	Al	Ta	Hf	Re	Ni
A	3.01	11.6	1.02	5.90	6.11	7.73	0.04	3.16	Bal.
B	3.18	12.1	1.01	5.95	6.05	7.95	0.08	4.00	Bal.
C	2.99	11.8	1.03	5.85	6.08	7.79	0.09	6.04	Bal.

The thermophysical properties of the experimental alloys were investigated by the differential scanning calorimetry analysis (DSC, Netzsch 409CD). Approximately 250 mg of the master alloy was heated at a heating rate 5 ℃/min in a flowing stream of Ar. Samples were heated to 1460 ℃ and then cooled at the same rate to 1000 ℃. To reveal the microstructure, specimens were etched with a solution of HNO_3 (3 ml) - HF (6 ml) - Glycerin (9 ml). The as-cast microstructure was observed with an optical microscopy (OM, Leica DM-4000M) and a scanning electron microscopy (SEM, JSM-6040). Quantitative calculation of the phase volume fraction was performed by digital image analysis software of SISC IAS V8.0. In order to study the microsegregation of constituent elements, quantitative composition measurements were carried out on the as as-cast specimen in the as-polished condition using electro microprobe analyzer (EPMA) JXA-8100.

Experimental results and discussion

Thermophysical properties. Fig.2 (a) shows the typical DSC curve for alloy A. The characteristic high temperature phase transformations can be easily indicated in the cooling curve from 1410 ℃ to 1200 ℃: (1) $L_1 \rightarrow \gamma$: The first exothermic peak, identified as T_L, associated with the liquidus temperature. (2) $L_2 \rightarrow \gamma/\gamma'$ eutectic: The second exothermic peak, T_E, is related to the γ/γ' eutectic reaction temperature. The solidus temperature, T_S, can be extrapolated from the DSC curve. (3) $\gamma \rightarrow \gamma'$: The third peak, $T_{\gamma'}$, is related with the γ' phase solvus temperature.

The variation of T_L, T_E, T_S, and $T_{\gamma'}$ with the content of Re was shown in Fig.2 (b). It was found that Re slightly increase T_L, but apparently lower the T_s, T_E and particularly $T_{\gamma'}$. The corresponding freezing range (T_L-T_S) increases about 3.4 K with Re addition. As mentioned previously, P. Caron suggested that Re additions apparently increased the γ'-solvus temperature in a series of nickel-base superalloys [10]. It will increase the volume fraction of strengthening γ' phase at elevated temperature, and thus promote the high temperature creep strength of single crystal alloys. Nevertheless, in this investigation, it shows the contradictory result which may be attributed to the complicated interaction behavior of the constituent elements for different alloy systems.

Meanwhile, the eutectic volume fraction can be estimated by the ratios between the eutectic reaction enthalpy and the total enthalpy during solidification. As shown in DSC cooling curve, the eutectic volume fraction is calculated by $Area_1/Area_{1+2}$. The detail analysis of the eutectic volume fraction will be described later.

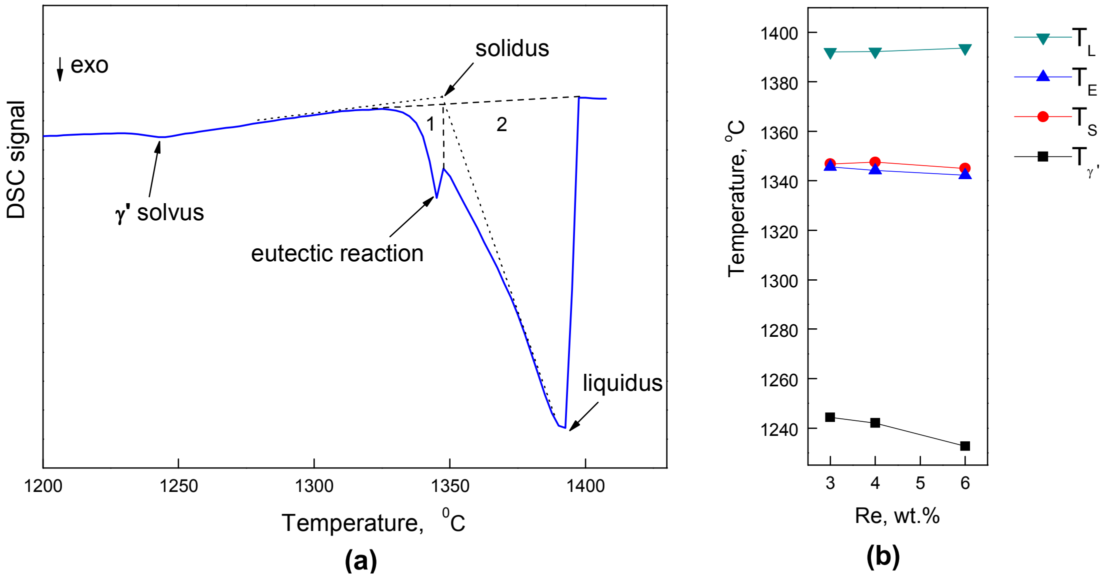

Fig.2 (a) DSC curves for Alloy A at cooling rates of 5 ℃/min. Eutectic volume fraction can be calculated by the enthalpy proportion of $Area_1/Area_{1+2}$. (b) Variation of the characteristic phase transformation temperature with the contents of Re: T_L—liquidus, T_E—"eutectic reaction" temperature, T_S— solidus $T_{\gamma'}$—γ′ solvus.

Microsegregation. The microsegregation of the constituent elements are characterized by the segregation ratios, k', as given by

$$k' = C_{DC} / C_{ID} \qquad (1)$$

where C_{DC} is the concentration of the constituent element in the dendrite core, C_{ID} is that in the interdendritic region. k'=1 indicates the constituent element homogeneously distributes between the dendrite core and interdendritic region. The $k' > 1$ or $k' < 1$ indicate the elements partition preferentially to the dendrite core or to the interdendritic region during solidification. Table 2 shows the segregation ratios of the constituent elements in alloy A and C at the withdrawal rate of 100 μm/s. It reveals that Ta, Al and Ni segregated to the interdendritic region, while Co, Mo, W, and Re segregated to the dendrite core.

Alloy A and alloy C with the Re levels typical of the second and third generation single crystal alloys. Table 2 shows that Re addition results in the more severe segregation for the constituent elements segregate to either the dendrite core (Re, W, Co, Mo) or the interdendritic regions (Al, Ta and Ni). The severe chemical segregation in the as-cast microstructure will be difficult to be eliminated even by the later heat treatments, particularly for the low diffusivity elements Re and W.

Additionally, it is notable that Re has the strongest effects on the segregation ratios of Mo, W, and particular Cr. As shown in Table 2, the segregation ratios of Cr even changed from 0.973 (less than unity) to 1.155 (greater than unity) with Re addition. It demonstrates that there are obvious interactions among the four constituent elements (Cr, Mo, W and Re). Optimizing the content of Cr, Mo, W and Re should be an efficient way to improving the casting performance of single crystal alloys.

Table 2: Segregation ratios (k') of the constituent elements for alloy A and alloy C at the withdrawal rates of 100 μm/s and the thermal gradient of approximately 250 K/cm.

k' Elements / Alloys	Ta	Al	Ni	Cr	Co	Mo	W	Re
Alloy A (3 wt. % Re)	0.517	0.761	0.966	0.973	1.138	1.092	2.041	2.993
Alloy C (6 wt.% Re)	0.482	0.717	0.928	1.155	1.183	1.200	2.134	3.341
δ^*	-0.012	-0.015	-0.013	+0.352	+0.015	+0.036	+0.031	+0.141

δ^* defined as the variation of segregation ratios with per 1 wt. % Re addition.

As-cast microstructure. The as-cast dendritic structures for alloy A, B and C at the withdrawal rate of 50 μm/s were shown in Figs. 4a-c. Due to the stronger dendrite segregation of constituent elements with Re additions, the constitutional supercooling in front of the solid-liquid interface during directional solidification apparently increased. So the primary dendrite arm spacing and the γ′ phase precipitated in the dendrite core were greatly refined with Re addition. In addition, the volume fractions of eutectic in the as-cast microstructures were quantitative calculated by the image digital analysis, as shown in Fig. 5a. It indicated that the volume fraction of eutectic increased with Re addition. The least amount of eutectic (2.62 %) was present in alloy A. With Re addition, the volume fraction of eutectic in alloy B and C significantly increased to 3.54 % and 4.04 % respectively. Furthermore, the influence of Re on the eutectic formation can be confirmed by the enthalpy analysis of the DSC cooling curve, as shown in Fig. 5b, which clearly reveals that Re additions significantly enhance the tendency for eutectic formation.

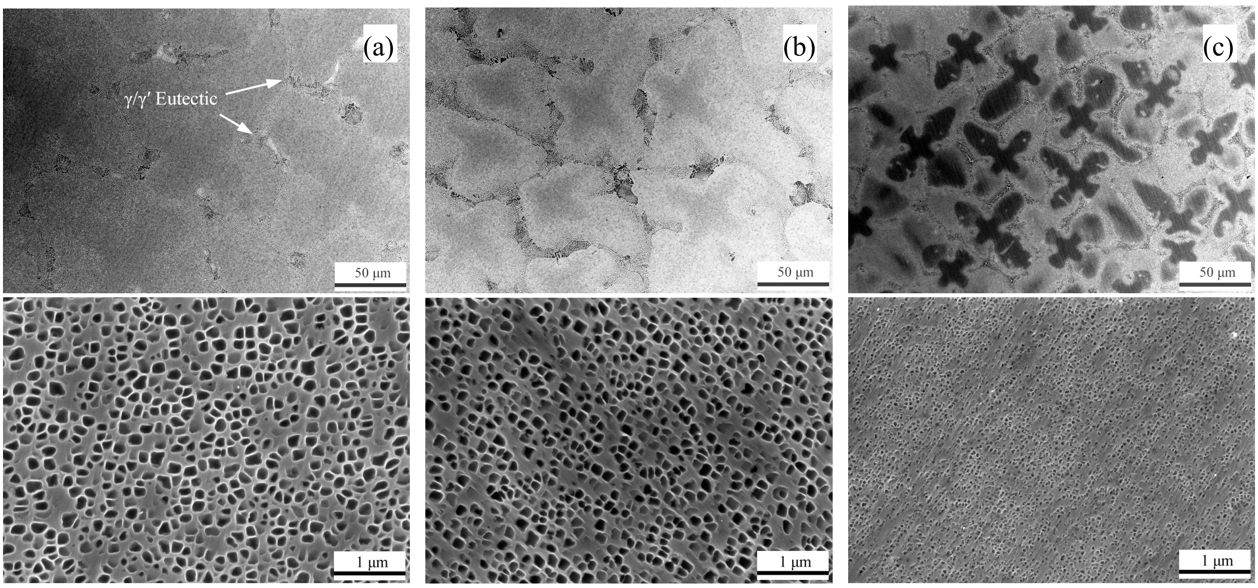

Fig.4. SEM images of cross-sectional as-cast microstructure for the experimental alloys directionally solidified at V=50 μm/s, (a-c) SEM image of the as-cast dendritic structure for Alloy A, B and C; (d-f) γ′ phase precipitate in the dendrite core for alloys A, B and C respectively.

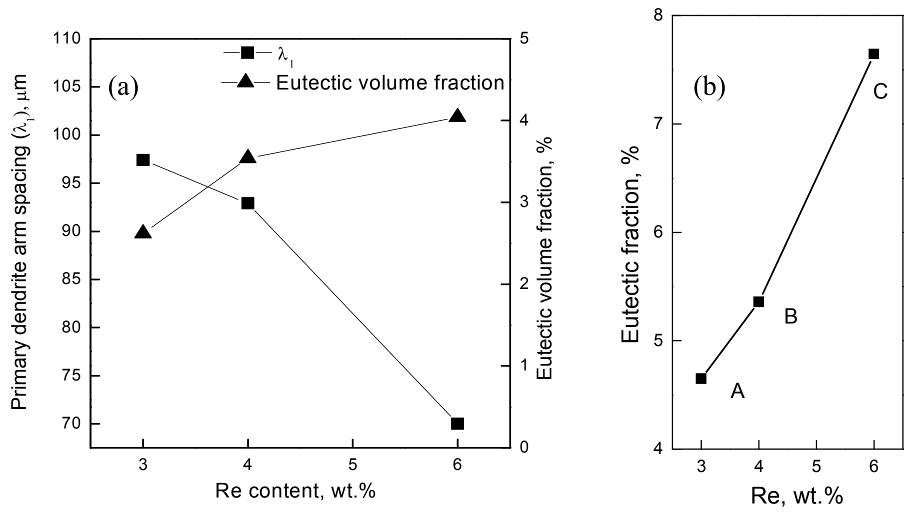

Fig.5 (a) The primary dendrite arm spacing and eutectic volume fraction for alloy A, B and C at V=50 μm/s. (b) Eutectic volume fraction for the master alloys estimated by the DSC enthalpy analysis.

Conclusions

The following conclusions can be drawn from this work:

1. Re addition slightly increases the liquidus temperature, but apparently lower the solidus, γ′-solvus and eutectic reaction temperature of the single crystal superalloys.
2. Re addition results in the more severe dendrite segregation of constituent elements, which partition preferentially to the dendrite core (Re, W and Co et al.) or to the interdendritic region (Ta, Al and Ni).
3. With the increasing Re addition, the as-cast dendritic microstructures were obviously refined at the same withdrawal rates and thermal gradient. Meanwhile, the volume fraction of eutectic in the as-cast microstructure apparently increased with Re addition.

Acknowledgements

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