EFFECTS OF REFINERS ON GRAIN STRUCTURES OF SUPERALLOY IN 718 AT HIGH POURING TEMPERATURE

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Abstract

In order to improve the filling ability of large complex thin wall castings, the pouring temperature should be increased, but this will result in grain size coarsening. Therefore, effects of refiners on grain structures of superalloy Inconel 718 have been investigated. It is shown that, adding mixed refiner consisting of two ternary intermetallic compounds at pouring temperature of 1520°C, the average equiaxed grain size could be refined from 10.56 to 2.71mm and the proportion of equiaxed grains at cross-section could be improved from 10 to 84%. At the same pouring temperature, the amount of porosity in specimens could be greatly decreased by adding refiners into the melt. Moreover, experimental results indicate that trace addition of refiners to the melt neither introduces inclusions nor changes the phase constituents of the alloy. Grain refinement mechanisms are also discussed.

Introduction

With the continuous improvement of the engine thrust weight ratio and the advances in casting technology, the turbine disk and intermediate case become large, complex, and thin-walled, which makes it more difficult to cast such components. Meanwhile, the operating temperature of these superalloy castings is approximately 700°C. Under this temperature, a uniform and fine-grained structure is desired in order to obtain good low cycle fatigue resistance and high tensile strength [1]. However, one of effective methods is to improve the filling ability of castings through the increase of the pouring temperature, leading to a coarser grain size, which can decrease the low cycle fatigue resistance and high tensile strength.

Grain refinement is an effective method to improve the deformability and mechanical properties of metallic materials. Refinement of cast structure requires that nucleation occurs at a large number of sites and that extensive growth of individual crystals be avoided. It follows that grain refinement necessitates both ease of nucleation and inhibition of the continued growth of crystallites in the melts. Fine grain casting techniques of superalloys mainly include the thermal control method [2], the chemical approach [1, 3] and the dynamic method [4, 5]. Among of these, the chemical fine grain process is an efficient method, which neither needs complicated equipment nor has a complex process. For some superalloys, refractory metal oxides, carbides, nitrides and boron were used as refiners [6, 7]. However, this method will introduce inclusions which become crack initiation sites, influencing low-cycle-fatigue property [8]. Especially, addition of boron decreases incipient melting temperature of the alloys, which reduces the hot-working and plastic properties [9]. Liu et al [1] developed two kinds of refiners used in Ni-Fe

based superalloys. These refiners not only have effective refinement capability, but also do not introduce inclusions. However, this refiner is used at the temperature of 1360-1420°C, far below the melting and pouring temperature for most superalloy castings.

In this work, two intermetallic compounds from the ternary system Co-Fe-Nb and Cr-Fe-Nb were prepared as refiners, and their effects on the grain structures of superalloy Inconel 718 under various refining processing were investigated at alloy pouring temperature of 1520°C.

Materials and Experimental Procedures

Preparation of Refiners

An alternative method of refinement of cast structure is nucleation promoting and growth hindering, which is more effective for grain refinement. Ideal refiners for superalloys should have the following characteristics [10]:

1. They are composed of the alloying elements in superalloys, such as Fe, Ni, Cr, Co and Nb.
2. Their melting point should be higher than the pouring temperature.
3. The density of refiner should be close to the alloy, in order to avoid floatation or deposition. Accordingly, the density of refiner should be controlled between 8.0-9.0g/cm³.
4. Good lattice matching between refiner and the alloy should be required to decrease the interfacial energy. Generally, if lattice misfit between refiner and alloy is smaller than 12%, the refiner can act as an effective nucleation substrate.
5. Its must brittle and easy to triturate.

Based on calculation of lattice misfit and extensive experiments, some high melting intermetallic compounds satisfied the above conditions simultaneously. The button ingots of refiners were prepared by melting an appropriate proportion of the constituents in a vacuum arc melting furnace in an argon atmosphere. The raw materials were 99.95% Cr powder, 99.5% Fe powder, 99.9% Co power and 99.97% Nb block. Then they were ground into powders with the different average size. The physical and crystallographic parameters of refiner A1 and A2 are listed in Table 1. The mixed refiner was prepared by mixing A1 and A2 physically with the proportion of 1:1 in weight percentage. The melting point of the refiners was analyzed by differential thermal analysis (DTA).

Superalloy Inconel 718

The alloy had the following chemical composition (wt. %): 0.056 C, 0.01 Co, 52.54 Ni, 19.15 Cr, 3.11 Mo, 0.61 Al, 0.94 Ti, 5.03 Nb, 0.0026 B, 0.028 Zr and with the rest being Fe. The equilibrium liquidus and solidus temperatures of the alloy are 1349 and 1270 °C respectively determined by DTA.

Melting and Casting

A vacuum melting and casting unit and high-purity master alloy ingots were used to cast conventional and fine-grained cylindrical ingots. The melt was firstly superheated up to 1550 °C for 2-3 minutes and then cooled down to the pouring temperature. Then, for the conventional

cast process, the melt was poured. However, for chemical grain refinement, the refiner was added into the melt. The addition amount was 0.3 wt. % of the charge. After that, an appropriate stirring was needed in order that refiner particles can be dispersed in the melt uniformly. Then the melt was held for 30-60 seconds for homogenization and was subsequently poured into a ceramic mould with its inner size of 110 mm long and 20 mm in diameter. The pouring temperature and mould preheating temperature were kept at 1520 °C and 900°C respectively in all cases.

Metallographic Examinations

The alloy ingots were sectioned along the transverse cross-section and samples were ground, polished and subsequently chemically etched with a solution consisting of 15g CuSO₄, 3.5ml H₂SO₄ and 50ml HCl to expose grain structures. The average equiaxed grain size and fraction of equiaxed grains at transverse cross-section were determined by a standard quantitative metallographic technique by the line intercept method. The grain size was also estimated with reference to the ASTM standard. The distribution of the alloying elements was determined with Electron Microprobe (EPMA).

Table 1 Physical and crystallographic parameters of the refiner

Refiner	Crystal structure	Density(g/cm ³)	Composition	Melting point(°C)
A1	Hexagonal	8.8	Co-Fe-Nb	1550
A2	Hexagonal	8.2	Cr-Fe-Nb	> 1650

Experimental Results

Grain Structures

Table 2 illustrates the experimental scheme and processing parameters, together with characteristic parameters of grain structures under various refining processing. The corresponding grain structures are shown in Fig.1. Columnar grains were observed under without additions. On the contrary, adding refiners to the melt can reduce grain size (D) and improve the fraction of equiaxed grains at transverse cross-section (P) at pouring temperature of 1520°C. In addition, adding mixed refiner consisting of two ternary intermetallic compounds at pouring temperature of 1520°C, the average equiaxed grains size could be refined from 10.56 to 2.71mm and the proportion of equiaxed grains at cross-section could be improved from 10 to 84%.

Table 2 Grain structure features under various casting conditions

Sample no	Refiner	Particle size(μm)	D(mm)	ASTM grade	P (%)	SDAS (μm)
1	without	without	10.56		10	72.63
2	A1	100-150	3.02	M-7.5	75	50.75
3	A1	50-75	3.17	M-7	50	72.62
4	A2	100-150	3.83	M-6.5	59	60.12
5	A2	50-75	4.36	M-6	70	69.02
6	A1+A2	*	2.71	M-7.5	84	68.73
7	A1+A2	75-100	2.84	M-7.5	81	63.45
8	A1+A2	50-75	2.98	M-7.5	82	62.87

Note: * the particle size of A1 is 100-150 μm and the particle size of A2 is 50-75 μm .

Fig.1. Grain structures of cylindrical ingots obtain under various refining processing (a-h) correspond to specimen nos.1,2,3,4,5,6,7 and 8, respectively.

Dendrite Structures

The dendritic morphologies of samples 1, 2, 4 and 6 are shown in Fig.2. It can be seen that dendritic morphologies are developed branches under the condition of without additions. In contrast, the average length of the primary dendrite axes decreases with the grain refinement additions. The addition of refiner to the melt causes a large amount of heterogeneous nucleation sites to occur. Therefore, a large number of grains form, which soon impinge on another, resulting in the growth of dendrites being hindered by another dendrites.

Moreover, the secondary dendrite arm spacing (SDAS) of both samples 1 and 6 is about 65 μm , indicating that the grain refinement has little effect on the SDAS, which relates to the melt pouring and mould temperature, i.e. to the cooling rate, which was kept almost constant throughout the present study.

Fig.2. Dendritic morphologies of IN718 under refining processing (a-d) correspond to specimen nos.1, 2, 4 and 6, respectively.

Interdendritic Segregation

The segregation ratio is defined as the maximum concentration in interdendrite areas over the minimum concentration in the center in dendrite arm. Fig.3 illustrates the segregation ratio of elements Fe, Cr, Mo, Nb and Ti. It can be concluded that Mo, Nb and Ti are positive segregation elements and segregate to interdendritic area. Whereas Fe and Cr are negative ones and enrich in dendritic core. Moreover, it can be seen that the segregation ratios decrease gradually with decreasing grain diameter. The results also indicate that segregation alleviates after grain refinement. It is known that spatial scope for growth of dendrites diminishes with grain refinement, resulting in the distance and amplitude for chemical composition to segregate to be reduced, which is the most important reason for the lessening of the element segregation. Moreover, it can be seen that the segregation ratios decrease gradually along with decreasing grain diameter.

Fig.3. Variation of segregation ratio of alloying elements with grain size

Inclusions

Superalloys are very sensitive to inclusions because they can serve as crack origins. Thus, it is necessary to determine if there exist inclusions in the chemically fine-grained specimens. Metallographic and scanning electron microscope observations, as well as composition analysis, show that there exist no metal oxides and other elements different from the alloying elements in the chemically refined specimens. The reason for this is that the high-purity master alloy ingots were remelted at a high vacuum level of 10^{-2} Pa, which is favorable for preventing the formation of metal oxides. Moreover, a trace addition of inoculants whose elemental composition is part of that of IN718 would not produce inclusions.

Porosity

It is known that superalloy Inconel 718 has a wide solidification temperature range. Therefore, porosity is likely to form in its castings. Figure 4 displays the morphology, distribution, and amount of porosity in conventional and fine grained specimens poured at 1520°C. It can be found that there exists some intensively distributed and large-sized porosity in specimen 1. However, there is uniform and little porosity in chemically refined specimen 6. That is, the amount of porosity in the specimen can be remarkably decreased with grain refinement.

Fig.4. Porosity at different cast processing (a) specimen 1, (b) specimen 6.

Discussion

Grain Refinement Mechanisms by the Addition of Refiners

Generally, the effectiveness of refiner should meet the following principles: (1) a fine epitaxial fit between low-index planes of the substrate and the nucleated solid should exist; (2) the interfacial energy between the substrate and the liquid should be higher than the interfacial energy between the nucleated solid and the liquid; and (3) the lower the lattice disregistry, the more effective the refiner is in promoting nucleation is. According to the calculated model of lattice disregistry (δ) between it and the nucleated phase proposed by Bramfitt [11], the value of δ for some specific crystal planes is shown in Table 3 [12].

When the lattice is less than 12%, the refiner will have a good refining effect. The planes matching models and matching orientations were calculated and simulated, which are shown in

Fig.6 and 7 [12]. The results show that (0001) planes of refiners A1 and A2 have $\delta = 4.45$ and 3.34% with (111) planes of γ matrix, respectively. (01 $\bar{1}$ 0) planes of refiners A1 and A2 have $\delta = 6.12$ and 5.90% with (110) planes of γ matrix, respectively. In addition, (01 $\bar{1}$ 0) planes of refiners A1 and A2 have $\delta = 8.38$ and 6.60% with (111) planes of γ matrix, respectively. Specifically, as to the A1, the disregistry of (0001) with (110) is 9.35%. Therefore, the refiners can act as the nucleation substrata of γ matrix and allow its epitaxial growth with the refiners for they have less lattice disregistry between the matrix of the alloy. Presence of a great number of active refiner particles in the melt would cause enormous heterogeneous nucleation of several crystallites, which would impinge on another and restrict further growth. Hence, the formation of numerous nuclei and restriction their further growth result in the refinement of grains. However, due to the higher pouring temperature, refining effect is reduced.

Table 3 Lattice disregistry δ of specific crystal plane between refiners and the alloy

Refiner	Matching crystal plane	Matching orientation $[uvw]_s [uvw]_n$	δ (%)
A1	(0001)//(111)	$[\bar{1}\bar{2}\bar{1}0] [\bar{1}10]$, $[\bar{2}\bar{1}10] [\bar{1}01]$, $[\bar{1}010] [11\bar{2}]$	4.45
	(0001)//(110)	$[\bar{2}\bar{1}10] [\bar{1}12]$, $[\bar{1}\bar{2}\bar{1}] [\bar{1}10]$, $[\bar{1}010] [001]$	9.35
	(01 $\bar{1}$ 0)//(111)	$[\bar{2}\bar{1}10] [\bar{1}10]$, $[\bar{2}\bar{1}12] [\bar{1}01]$, $[11\bar{2}2] [\bar{1}\bar{1}2]$	8.38
	(01 $\bar{1}$ 0)//(110)	$[\bar{2}\bar{1}10] [\bar{1}10]$, $[\bar{2}\bar{1}12] [\bar{1}\bar{1}2]$, $[0001] [00\bar{1}]$	6.12
	(0001)//(111)	$[\bar{1}\bar{2}\bar{1}0] [\bar{1}10]$, $[\bar{2}\bar{1}10] [\bar{1}01]$, $[\bar{1}010] [11\bar{2}]$	3.34
A2	(01 $\bar{1}$ 0)//(111)	$[\bar{2}\bar{1}10] [\bar{1}10]$, $[\bar{2}\bar{1}12] [\bar{1}01]$, $[11\bar{2}2] [\bar{1}\bar{1}2]$	6.60
	(01 $\bar{1}$ 0)//(110)	$[\bar{2}\bar{1}10] [\bar{1}10]$, $[\bar{2}\bar{1}12] [\bar{1}\bar{1}2]$, $[0001] [00\bar{1}]$	5.90

Note: Subscripts s and n are abbreviations of substrate created by refiner and new nucleus, respectively.

Fig.6. Planes matching models of A1: (a) (0001) $A_1 // (111)_\gamma$, (b) (0001) $A_1 // (110)_\gamma$, (c) (01 $\bar{1}$ 0) $A_1 // (111)_\gamma$, (d) (01 $\bar{1}$ 0) $A_1 // (110)_\gamma$.

Fig.7. Planes matching models of A2: (a) $(0001)_{A2} // (111)_{\gamma}$, (b) $(0\bar{1}\bar{1}0)_{A2} // (111)_{\gamma}$,
(c) $(0\bar{1}\bar{1}0)_{A2} // (110)_{\gamma}$.

Mechanism of the Increment in Equiaxed Fraction after Grain Refinement

It can be seen from Table 2 and Fig. 1 that adding refiner to the melt makes the equiaxed fraction increase along with the grain refinement. It is known that the condition that equiaxed grains appear ahead of the columnar front can be expressed in the following equation [13]

$$G < \eta n^{1/3} \left[1 - \left(\frac{\Delta T_N}{\Delta T_C} \right)^3 \right] \left[-8\Gamma m(1 - k_0) \frac{C_0 V}{D} \right]^{1/2} \quad (1)$$

where G is the temperature gradient (K/m) in the melt in front of solid-liquid interface; η a columnar to equiaxed transition coefficient relative to the alloy; n the number of sites per cubic meter (m^{-3}); ΔT_N the undercooling at the heterogeneous nucleation temperature (K); ΔT_C an undercooling equal to that of the columnar growth front (K), there exists $\Delta T_C \propto (VC_0)^{1/2}$; V the growth velocity (m/s), C_0 alloy composition (wt. %); Γ is Gibbs-Thomson coefficient (m K), the ratio of solid-liquid interfacial energy to entropy of melting; m the liquidus slope (K/wt. %); k_0 the distribution coefficient; D the liquid diffusion coefficient (m^2/s). It can be seen from Eq. (1) that lowering G has an advantage for the formation of equiaxed grains.

The addition of refiner can increase n , leading to the increase the right-hand side of Eq. (1). As a result, it is beneficial for the forming of the equiaxed grain zone. Additionally, refiner particles dispersed uniformly in the melt caused a large quantity of equiaxed grains to form and release a great amount of latent heat which prohibits their further growth. In addition, the formation and growth of many equiaxed grains impede the growth of columnar grains.

Conclusions

1. At the pouring temperature of 1520°C , the average grain size of cylindrical ingots decrease obviously after Co-Fe-Nb or Cr-Fe-Nb refiner is used alone.
2. When adding mixed refiner to the melt, the average equiaxed grains size could be refined from 10.56 to 2.71mm and the proportion of equiaxed grains at cross-section could be improved from 10 to 84% at pouring temperature of 1520°C .

3. The segregation ratios of alloying elements such as Fe, Cr, Nb, Mo and Ti decrease along with the decrease in grain size, indicating that grain refinement by addition of refiners can diminish the element segregating in solidification, even though the local solidification time is almost unchanged.
4. Refiner particles of good lattice compatibility with matrix act as substrata of matrix, thereby causing grain refinement.

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