



Effect of phosphorus segregation on as-cast microstructure and homogenization treatment of IN706 alloy

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Abstract: The distribution of phosphorus in IN706 alloy was studied, with particular emphasis on the as-cast microstructure and homogenization treatment. It is found that phosphorus is segregated in Laves phase and markedly influences the as-cast microstructure of IN706 alloy. Phosphorus retards the formation of Laves phase with the eutectic form, but promotes the Laves formation with the blocky form. Phosphorus refines the dendritic structure and noticeably enlarges the interdendritic area. The reason for phosphorus influencing the as-cast microstructure is attributed to its extremely low solubility in γ matrix and great retarding effect on γ matrix solidification. In addition, phosphorus slightly decreases the melting temperature of Laves phase, and retards the dissolution of Laves phase and the element diffusion between dendrite core and interdendritic area during the homogenization treatment. And it is thought that phosphorus delays the homogenization process by inhibiting the element diffusion.

Key words: phosphorus; segregation; IN706 alloy; Laves phase

1 Introduction

IN706 alloy is derived from IN718 alloy by removing Mo, reducing Nb, Al, Cr, Ni, and increasing Ti and Fe [1–3]. The excellent balance of chemical composition results in superior characteristics to IN718 alloy in the segregation tendency, hot workability and machinability. Therefore, IN706 alloy is suitable for fabricating large forgings used at high temperature [4–8], which have been used for “F” series gas turbine disc with a diameter of about 2000 mm [9,10]. In order to manufacture these large gas turbine components, the large-size ingots more than 800 mm in diameter are necessary.

Phosphorus (P) is a common impurity element, which noticeably aggravates the segregation in superalloys [11–13]. The solidification segregation must be properly controlled for making large parts of IN706 alloy. However, the effect of P on the as-cast microstructure IN706 was seldom discussed in the previous literature, despite the importance of its effects on large-size superalloys. XIN et al [14] studied the effect of P content on (Laves + γ) eutectic reaction during

IN706 alloy solidification and presented that when P increased from 0.004% to 0.016%, the morphology of Laves phase and η -phase was markedly changed. But the interval from 0.004% to 0.016% is too wide to determine the critical addition of P for as-cast microstructure in IN706 alloy. During solidification, microsegregation is usually unavoidable. To eliminate the elemental segregation and obtain the uniform microstructure, homogenization treatment must be performed by high temperature diffusion before hot deformation, which plays a key role in the overall thermomechanical processing of nickel-based superalloys.

In the present work, in order to effectively control the chemical composition for large-size ingots, different contents of P are added to IN706 alloy. The segregation of P and its effect on the as-cast microstructure and homogenization process are studied, with a detailed discussion regarding the underlying mechanisms.

2 Experimental

The master IN706 ingot was fabricated by vacuum induction melting (VIM). The chemical composition of the ingot is 41.3Ni, 16.21Cr, 1.92Ti, 3.0Nb, 0.37Al,

0.022C, <0.001B, 0.002P and Fe in balance (mass fraction, %). The master ingot was then remelted via VIM to manufacture six 13 kg ingots doped with different levels of P. The P content of each ingot is listed in Table 1.

Table 1 P content of IN706 alloys

Alloy No.	w(P)/%
1	0.002
2	0.004
3	0.007
4	0.010
5	0.014
6	0.025

The samples at centre, 1/2 radius and edge of all six ingots were cut from a transverse slice at the same corresponding position of the ingots. The microstructure was observed with optical microscope (OM) and scanning electron microscope (SEM). The element distributions and micro-zone composition analyses were measured using electron probe micro-analysis (EPMA).

The melting temperature of Laves phase was determined by checking the microstructure variation of the samples heated at temperatures ranging from 1160 °C to 1190 °C for 0.5 h followed by water quenching. The dissolution of Laves phase was studied by soaking the samples for 1 to 5 h at 1160 °C. Vickers microhardness

was measured at different regions of the sample with Laves phase dissolved to evaluate the effect of P on the homogenization process.

3 Results

3.1 As-cast microstructure and segregation behavior

Figure 1 shows the dendrite microstructures at the centre of the ingots. The as-cast microstructure is constituted by the dendrite core (the light area) and the interdendritic region (the dark area). It can be seen that as the content of P is less than 0.007%, P does not have noticeable effect on the microstructure. However, as the content of P exceeds 0.010%, the dendrite arms become narrower.

Figure 2 shows the detailed microstructures of the interdendritic region at the centre of each ingot. Laves particles with a light color are precipitated in the interdendritic γ matrix with a dark color, which is called segregated area in the text. With the P content increasing from 0.002% to 0.025%, the morphology of Laves phase is changed noticeably. In Alloy 1 (0.002% P), both the eutectic and blocky Laves phase are observed (Fig. 2(a)), and each kind of the morphology accounted for about 50%. Figures 2(b) and (c) present the eutectic and blocky Laves phase observed by SEM at higher magnification. In Alloy 2 (0.004% P), the amount of the eutectic Laves phase is obviously reduced and that of the blocky Laves phase is significantly increased (Fig. 2(d)). In Alloy 3

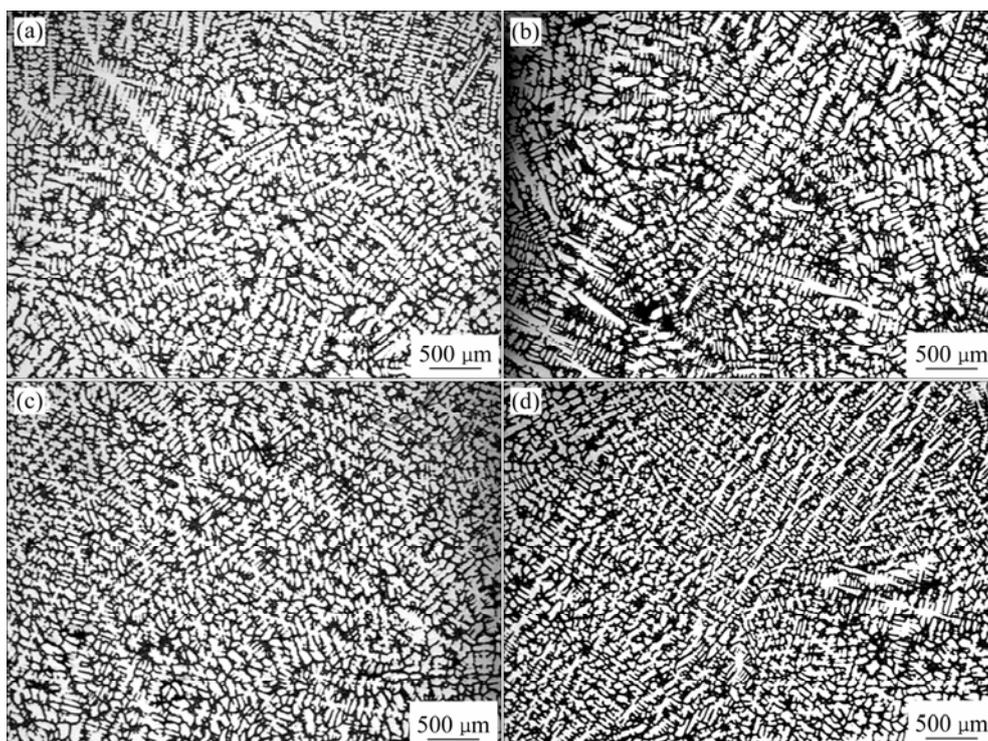


Fig. 1 Dendritic microstructures at centre of IN706 ingots: (a) Alloy 1 with 0.002% P; (b) Alloy 3 with 0.007% P; (c) Alloy 4 with 0.010% P; (d) Alloy 6 with 0.025% P

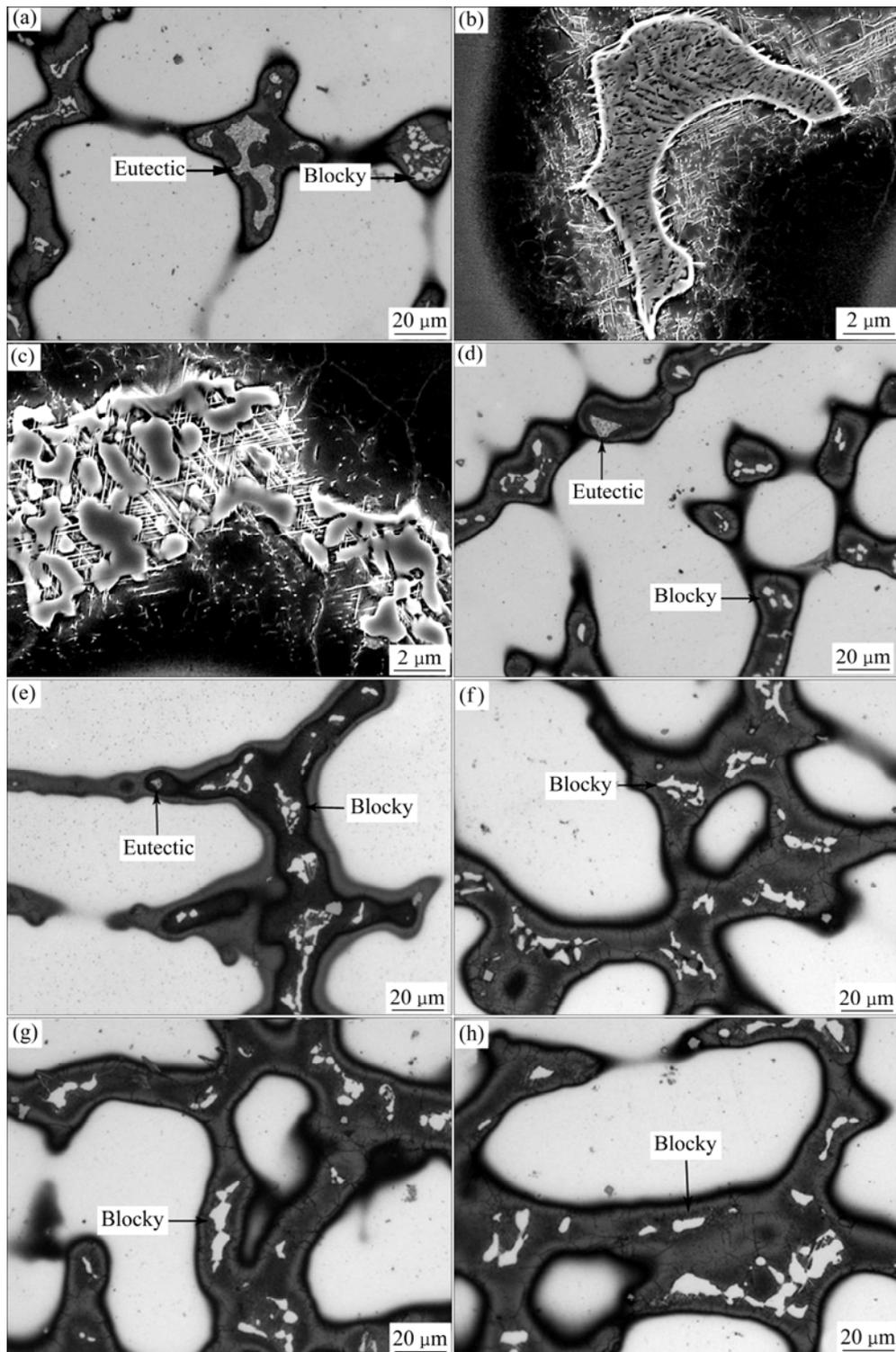


Fig. 2 Precipitation of Laves phase in interdendritic area at center of ingot: (a) Alloy 1; (b) Eutectic Laves phase in Alloy 1; (c) Blocky Laves phase in Alloy 1; (d) Alloy 2; (e) Alloy 3; (f) Alloy 4; (g) Alloy 5; (h) Alloy 6

(0.007% P), almost all of the Laves phases appear as blocky particles and the eutectic Laves phase forms as very small island which is seldom observed (Fig. 2(e)). When the content of P is more than 0.010%, only the blocky Laves phase is found (Figs. 2(f)–(h)). It should be noted in Fig. 2 that both the size of Laves particles and

the area of the interdendritic region are increased with the increasing P content.

The effect of cooling rate on the microstructure can be deduced from Fig. 3 by comparing the microstructures from the edge to the centre in the ingot of Alloy 1 (0.002% P) and Alloy 6 (0.025% P). At the edge of the

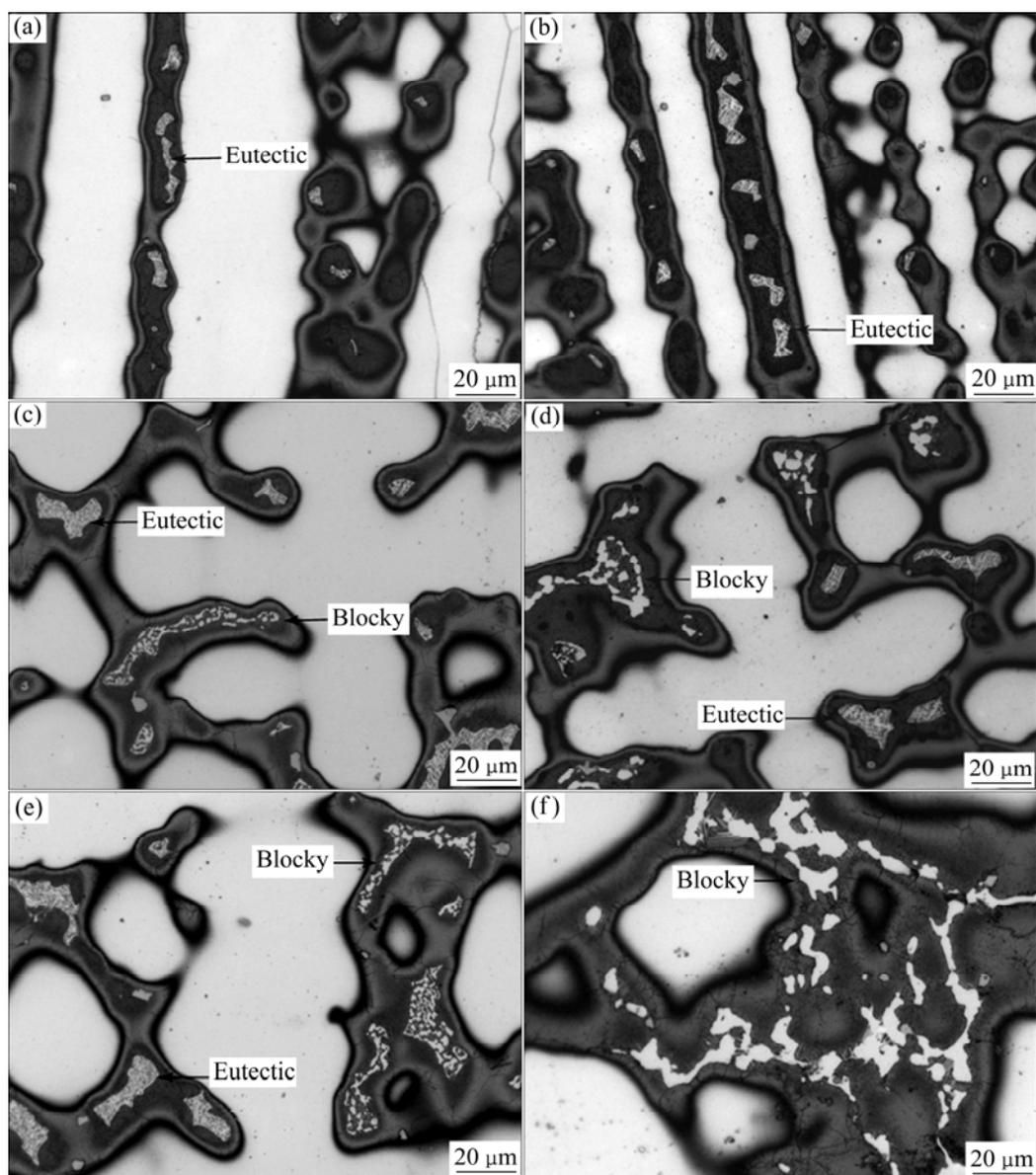


Fig. 3 Laves precipitation from edge to centre in ingot of Alloy 1 and Alloy 6: (a) Edge of Alloy 1; (b) Edge of Alloy 6; (c) 1/2 radius of Alloy 1; (d) 1/2 radius of Alloy 6; (e) Centre of Alloy 1; (f) Centre of Alloy 6

ingots, there is only eutectic Laves phase in both alloys (Figs. 3(a) and (b)). At the 1/2 radius of the ingots, the eutectic Laves phase takes a larger fraction in Alloy 1 (Fig. 3(c)), while more blocky Laves phases are observed in Alloy 6 (Fig. 3(d)). At the centre of the ingots, consistent with the results in Fig. 2, only blocky Laves phase exists in Alloy 6 (Fig. 3(f)), while half of the Laves phase appears as eutectic form in Alloy 1 (Fig. 3(e)). It is also shown in Fig. 3 that P increases the interdendritic area from the edge to the centre of the ingot.

Figure 4 shows the distribution of elements in Alloy 6 (0.025% P) obtained by EPMA. The distribution maps reveal that P, Nb and Ti are seriously segregated in Laves phase, but the segregation behaviors of these

elements are different from each other. P is highly segregated in Laves phase, but hardly dissolved in dendrite core and segregated area with a dark color. Nb is enriched in Laves phase and also slightly dissolved in segregated area. However, Ti is enriched in both Laves phase and segregated area.

The compositions of Laves phase, dendrite core and segregated area near Laves phase in Alloy 1 (0.002% P), Alloy 4 (0.010% P) and Alloy 6 (0.025% P) measured by EPMA are listed in Table 2. It is shown that the content of P in Laves phase is increased with the increase of P content in the alloys, which is 0.059% in Alloy 1 and 0.929% in Alloy 6. Furthermore, it seems that P does not noticeably influence the compositions of γ matrix in dendrite core and segregated area near Laves phase.

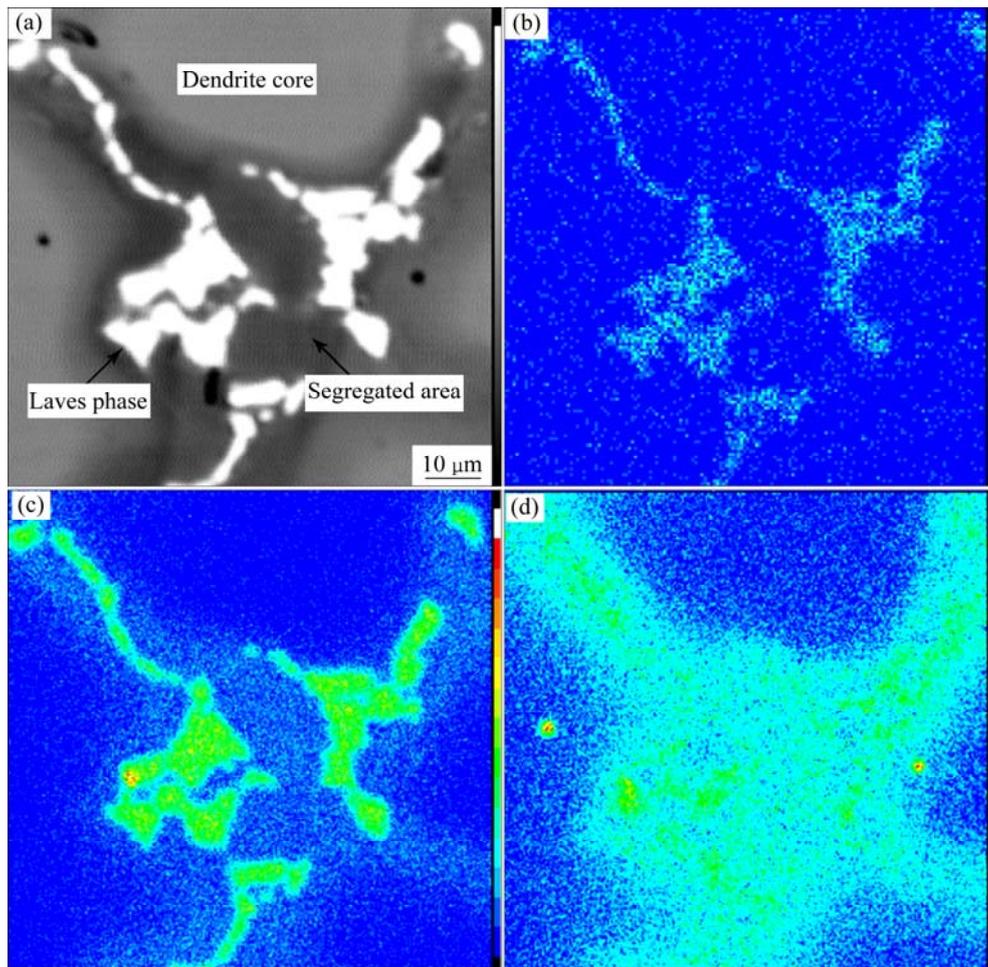


Fig. 4 Distribution of P, Nb and Ti in Alloy 6: (a) Microstructure of analysis area; (b) P map; (c) Nb map; (d) Ti map

Table 2 Compositions of test region in alloys with varied phosphorus additions (mass fraction, %)

Alloy No.	Region	Fe	Ni	Cr	Al	Ti	Nb	P
1	Laves phase	23.65	31.78	10.01	0.12	4.29	30.09	0.059
	Segregated area	28.72	47.35	13.67	0.54	4.15	5.57	0
	Dendrite core	40.60	40.12	17.24	0.28	0.84	0.92	0
4	Laves phase	23.76	31.93	9.87	0.11	4.32	29.56	0.458
	Segregated area	27.17	48.36	13.27	0.51	4.73	5.96	0
	Dendrite core	40.31	40.04	17.53	0.32	0.90	0.91	0
6	Laves phase	23.16	32.13	10.14	0.12	4.23	29.29	0.929
	Segregated area	27.72	48.12	13.04	0.48	4.63	6.02	0
	Dendrite core	40.22	40.09	17.40	0.30	0.98	1.02	0

3.2 Melting temperature of Laves phase

As shown in Fig. 5, the eutectic Laves phase in Alloy 1 is dissolved and a few blocky Laves particles are remained after heating at 1180 °C for 0.5 h (Fig. 5(a)). When the soaking temperature is raised to 1190 °C, the whole Laves phase is melted and the melting pool is formed (Fig. 5(b)), implying that the melting temperature of Laves phase in Alloy 1 is between 1180 °C and

1190 °C. And the melting temperature of Laves phase is also determined between 1180 °C and 1190 °C in Alloy 2 and Alloy 3.

For Alloy 6, as shown in Fig. 6(a), Laves phase is remained after soaking at 1170 °C, and the melting pool occurs when the soaking temperature is increased to 1180 °C (Fig. 6(b)). Therefore, the melting temperature of Laves phase in Alloy 6 is determined to be between

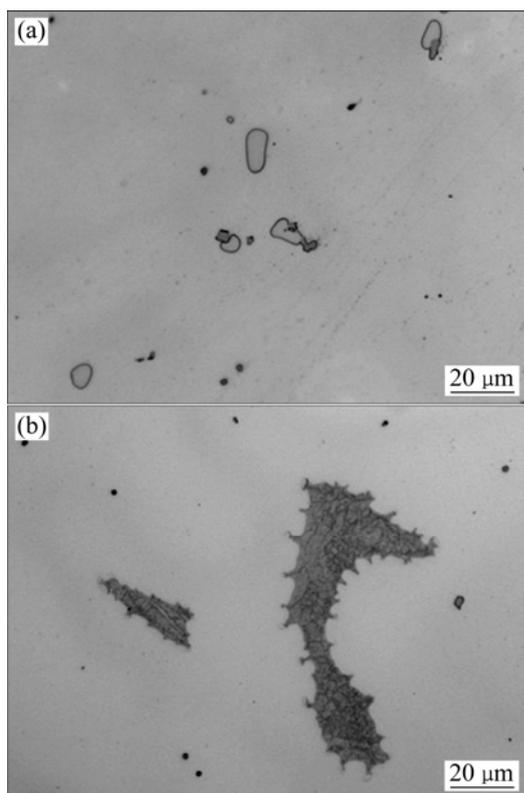


Fig. 5 Microstructures of Alloy 1 soaked at 1180 °C (a) and 1190 °C (b) for 0.5 h and then water quenched

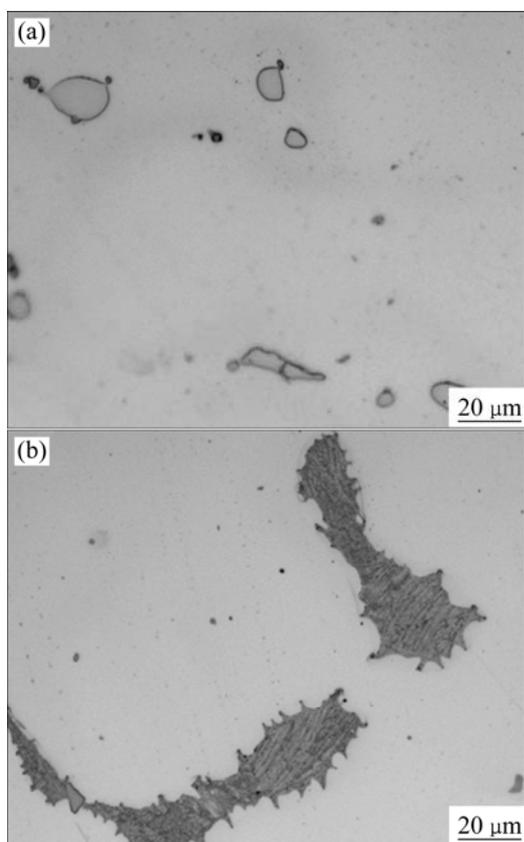


Fig. 6 Microstructures of Alloy 6 soaked at 1170 °C (a) and 1180 °C (b) for 0.5 h and then water quenched

1170 °C and 1180 °C. And the melting temperature of Laves phase in Alloy 4 and Alloy 5 is also determined to be between 1170 °C and 1180 °C. It is obvious that the melting temperature of Laves phase is slightly decreased with the increasing P content in IN706 alloy.

3.3 Dissolution of Laves phase

As the melting temperature of Laves phase has been determined to be higher than 1170 °C, it is safe to soak the alloys at 1160 °C for dissolving the Laves phase. Microstructure evolutions of Alloy 1 and Alloy 6 during soaking at 1160 °C are presented in Fig. 7. After soaking for 1 h, there are still a few Laves particles remained in both alloys, but the Laves particles in Alloy 6 are more than that in Alloy 1 (Figs. 7(a) and (b)). After soaking for 3 h, the Laves particles in Alloy 1 are completely dissolved (Fig. 7(c)), while there are still a few Laves particles in Alloy 6 (Fig. 7(d)). It is obvious that P inhibits the dissolution of Laves phase. The Laves phase in Alloy 6 is completely dissolved until soaking for 5 h (Fig. 7(e)).

There is “dark area” in Alloy 6 after soaking at 1160 °C and then air cooled (Figs. 7(b), (d) and (e)), while no “dark area” is found in Alloy 1 (Figs. 7(a) and (c)). Furthermore, as shown in Fig. 8, “dark area” is not observed both in Alloy 1 and Alloy 6 after soaking at 1160 °C and then water quenched (Figs. 8(a) and (c)). Therefore, it can be deduced that the “dark area” in Alloy 6 is formed during air cooling. As shown in Fig. 9, the Vickers hardness of the “dark area” is obviously higher than that of the matrix at the dendrite core in the homogenized and air cooled Alloy 6. Moreover, the darker area has higher hardness.

4 Discussion

As shown in Fig. 4 and Table 2, in the as-cast alloy, P is highly segregated in Laves phase but depleted in the segregated area and dendrite core. This result is in consistent with the earlier study [11] that P is extremely depleted in γ matrix and severely segregated in the residual liquid. During the solidification of the alloy with higher P addition, the P concentration in the residual liquid will increase gradually due to the low solubility of P in the matrix. The growth of dendrite would make the P atoms highly concentrate at the solidification front and hence the solidification rate of the alloy is slowed down markedly. As a result, P strongly hinders the solidification of IN706 alloy. The hindering effect of P on solidification of γ matrix is the main reason for the change of the as-cast microstructure of the alloys. Under the same solidifying condition, more residual liquid will be left in the alloy with higher P content. With the decrease of temperature, the residual liquid is decreased,

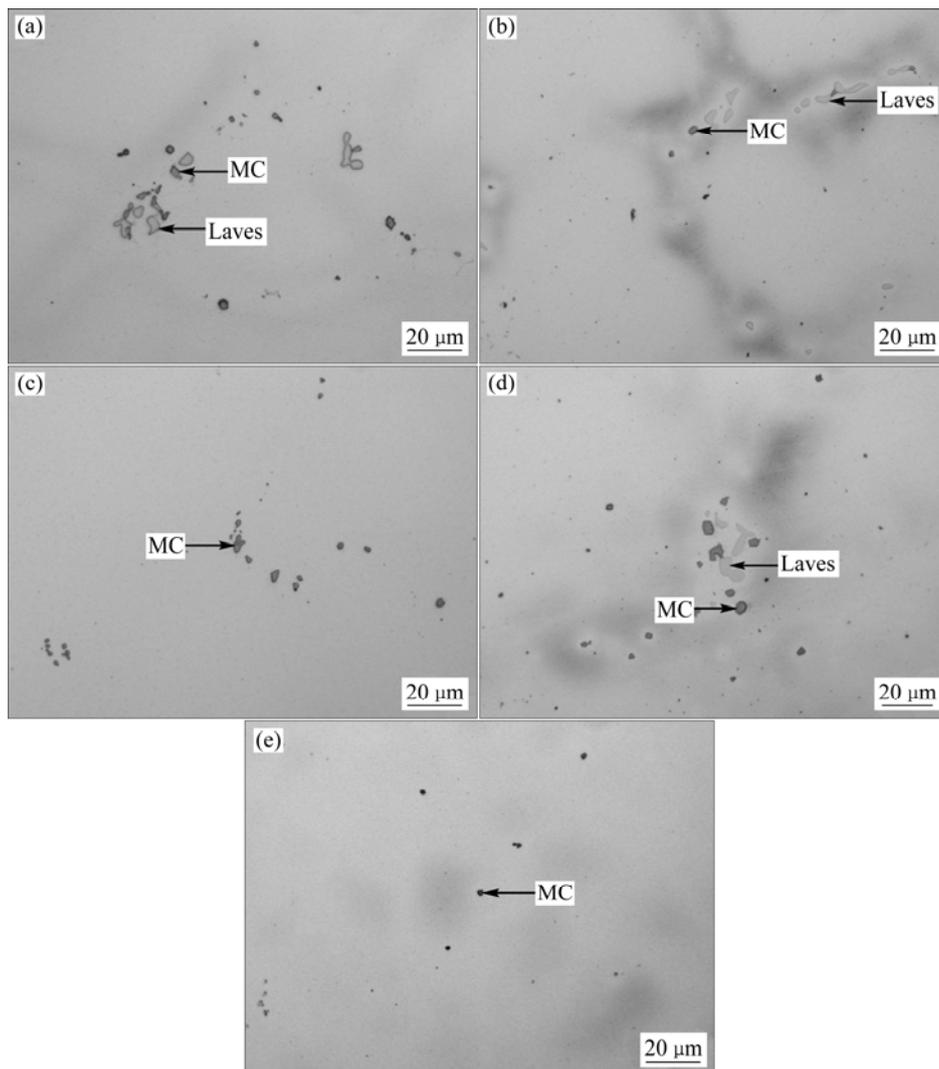


Fig. 7 Microstructures of Alloy 1 and Alloy 6 soaked at 1160 °C for different time and then air cooled: (a) 1 h for Alloy 1; (b) 1 h for Alloy 6; (c) 3 h for Alloy 1; (d) 3 h for Alloy 6; (e) 5 h for Alloy 6

the P content in the residual liquid will be increased and its effect on retarding the solidification of γ matrix will be stronger.

When the temperature is decreased to the level that Laves phase begins to precipitate, the P in the residual liquid will be largely absorbed, and then the solidification of γ matrix will proceed quickly again. The result in this work shows that P does not significantly lower the precipitate temperature of Laves phase. When Laves phase starts to precipitate, more residual liquid is remained in higher P addition alloy, and consequently, larger interdendritic area is formed (Figs. 2 and 3).

For lower P addition alloy, the precipitation of Laves phase absorbs much Nb and Ti which elevates the melting temperature of γ matrix. Then, the dramatic increase of melting temperature around Laves phase leads to the solidification of γ matrix. On the other hand, the solidification of γ matrix propels much Nb and Ti, which promotes the precipitation of Laves phase around

γ matrix. Finally, the (Laves + γ) eutectic is formed in lower P addition alloy (Fig. 2(a)). For higher P addition alloy, although the precipitation of Laves phase also absorbs much Nb and Ti, the P content in the residual liquid is still high enough to prevent the solidification of γ matrix. Only when Laves phase grows into larger particle, the segregation of P in the residual liquid around the Laves phase will be drastically reduced, and then the γ matrix will begin to solidify. Therefore, P promotes the blocky Laves phase formation but tends to prevent the precipitation of eutectic Laves phase (Figs. 2 and 3).

The precipitation of Laves phase is not only related with the P content in the alloy, but also determined by the cooling rate. At the edge of the ingot, the nucleation rate of Laves phase is greatly improved due to the chilling by the cold mould. The dramatic increase of nuclei results in the decrease of P, Nb and Ti contents around the nuclei, thus causes the solidification of γ matrix and forms (Laves + γ) eutectic. However, at the centre of the ingot,

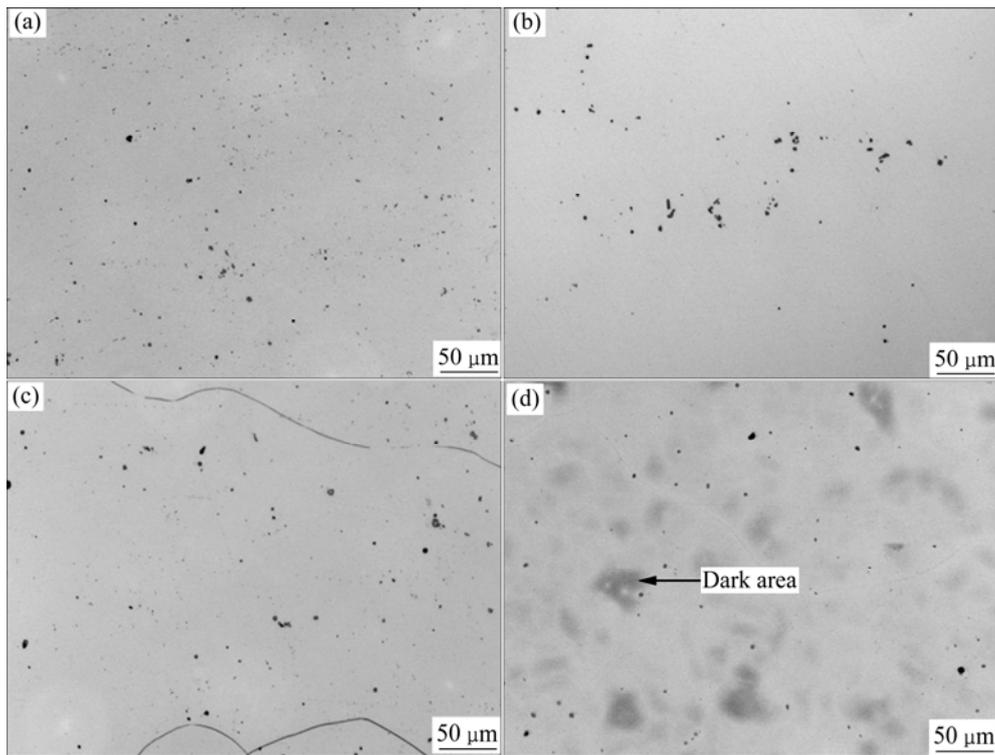


Fig. 8 Microstructures of test alloys soaked at 1160 °C for 10 h: (a) Water quenched for Alloy 1; (b) Air cooled for Alloy 1; (c) Water quenched for Alloy 6; (d) Air cooled for Alloy 6

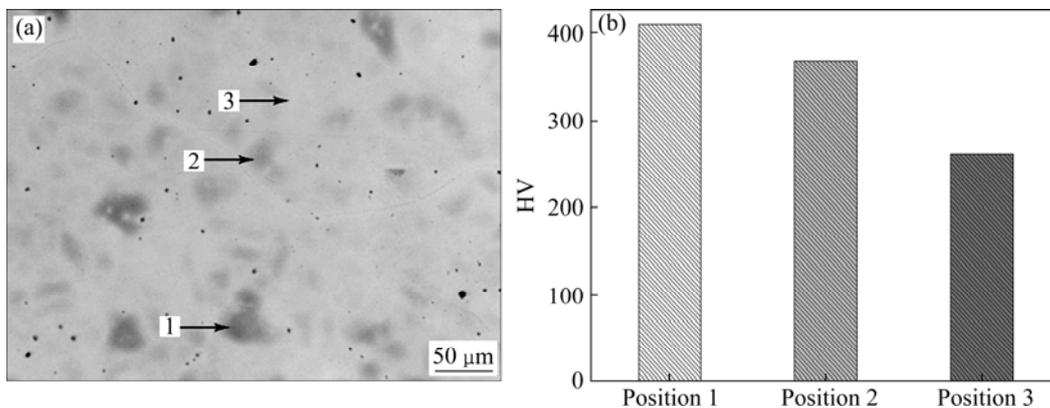


Fig. 9 Microhardness of Alloy 6 soaked at 1160 °C for 10 h and then air cooled

only blocky Laves phase is formed because of the low nucleation rate of Laves phase.

The result in this work reveals that P does not noticeably influence the chemical composition of the dendrite core. Compared with lower P addition alloy, the content of Nb and Ti in the residual liquid in higher P addition alloy is lower, because there is more residual liquid at the same temperature. Therefore, when Laves phase starts to precipitate in lower P addition alloy, higher P addition alloy must be further cooled and subsequently Laves phase could begin to precipitate. This is the reason why P reduces the melting temperature of Laves phase in IN706 alloy.

P is co-segregated with Nb and Ti in Ni-based superalloys [15–17], which will prevent the diffusion of Nb and Ti atoms during the dissolution of Laves phase. Consequently, high concentrations of Nb and Ti atoms are still remained around the dissolved Laves phase. During the cooling process after the dissolution of Laves phase at 1160jk °C, lower cooling rate may lead to the formation of γ' and γ'' phases in the high content area of Nb and Ti atoms. As a result, the “dark area” is formed in the air cooled higher P addition alloy at the site that Laves phase once precipitated. And the result in Fig. 9 verifies that the precipitation of γ' and γ'' phases in “dark area” is heavier than that at the neighbor area.

5 Conclusions

1) P refines the dendritic microstructure, enlarges the interdendritic area, promotes the blocky Laves formation and prevents the formation of Laves phase with the eutectic form in the as-cast IN706 alloy, because it is not dissolved in the γ matrix and significantly retards the solidification of γ matrix.

2) P lowers the melting temperature of Laves phase, inhibits the dissolution of Laves phase, and prolongs the homogenization time, because it retards the diffusion of Nb and Ti atoms.

3) The critical P content is between 0.007% and 0.010% in IN706 alloy. When P exceeds the critical content, the dendritic microstructure and melting temperature of Laves phase are changed.

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磷偏聚对 IN706 合金铸态组织及均匀化处理的影响

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摘要: 研究磷偏聚对 IN706 合金铸态组织及均匀化处理的影响。结果表明, 磷偏聚于 Laves 相中, 显著影响合金的铸态组织。磷抑制 Laves 相以共晶形态析出, 但促进 Laves 相以块状形态析出。磷细化枝晶组织, 并且扩大枝晶间面积。磷对铸态组织的影响是由于其在 γ 基体的低溶解度和对 γ 基体凝固的阻碍作用引起的。此外, 磷稍微降低 Laves 相的熔点, 在均匀化过程中, 磷阻碍 Laves 相溶解以及枝晶干与枝晶间的元素扩散。磷通过阻碍元素扩散来推迟均匀化过程。

关键词: 磷; 偏聚; IN706 合金; Laves 相

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