Re-recognition of the Effects of Phosphorus and Boron on the γ'' and γ' Phases in IN718 Alloy

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Abstract

The effects of P and B on the matrix strength and precipitations of γ' and γ'' phases in the grain interior were re-recognized in this study. The combination addition of P and B markedly accelerated the precipitations of γ' and γ'' phases and strengthened the matrix of IN718 alloy when air-cooled from high temperature, while made no difference when water-quenched from high temperature. The effect of single addition of P on the precipitations of γ'' and γ' phases was the same with that of the combination addition of P and B, while the single addition of B had no effect on the precipitations of the two phases. Therefore, it was P rather than B which accelerated the precipitations of γ' and γ'' phases. P could take part in the precipitations of γ' and γ'' phases, which was revealed by electrochemical extraction and quantitative analysis of chemical composition. It also revealed that P atoms were dissolved in the γ matrix to a relatively high degree at the temperature that γ' and γ'' phases began to precipitate, and consequently the precipitations of γ' and γ'' phases were accelerated. The first-principle calculation indicated that P decreased the formation enthalpies of γ' and γ'' phases when it occupied the Ni lattice sites in the two phases, which explained the effect of P on the γ' and γ'' phases.

Keywords IN718 superalloy · Phosphorus and boron · γ'' and γ' phases · Hardness

1 Introduction

Although phosphorus has been taken as a common impurity or a deleterious element in superalloys and steels for a long time [1, 2], it has been well documented recently that P addition in trace quantities could significantly prolong the creep and stress rupture lives of some superalloys including IN718 [3, 4]. Moreover, there is a strong synergistic effect between P and B on improving the stress rupture properties of IN718 alloy, and the effect of P and B co-addition was much greater than sum of the individual effect of the two elements, so it is proposed to modify superalloys by the combination addition of P and B [5]. Essentially, both P and B are the trace elements with strong

segregation tendency and tend to be segregated at the defects such as the grain boundaries [6, 7]. So the previous study mainly focused on the effects of P and B on the grain boundaries. The beneficial effects of P and B in the superalloys have been attributed to the improvement in the grain boundary cohesion [8, 9] and the grain boundary precipitations [3, 9, 10], and the enhancement of the resistance to oxygen intrusion along the grain boundaries [10].

A few investigations have focused on the effects of P and B on the γ'' and γ' phases in the grain interior of IN718 alloy. Liu et al. revealed that P had no effect on the precipitations of γ'' and γ' phases in IN718 alloys undergone standard heat treatment [8]. Horton et al. [11] demonstrated that P and B had no effect on the precipitations and growth of γ'' and γ' phases in IN718 alloy during long-term aging at 704 °C. However, Huang et al. [12] found that P could inhibit the growth of γ'' and γ' phases in DA718 alloy during long-term aging at 680 °C. These inconsistent phenomena indicate that the effects of P and B on the precipitations of γ'' and γ' phases are not clear.

The IN718 alloys with the combination addition of P and B and the single addition of P and B were prepared. In

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order to investigate the effects of P and B on the precipitations of γ'' and γ' phases, the microstructures and micro-hardnesses of the alloys subjected to different heat treatments were characterized and compared.

2 Materials and Experimental Procedures

2.1 Materials

Three master ingots of IN718 alloy were prepared by vacuum induction melting (VIM). The analyzed chemical compositions (mass%) of the alloys are listed in Table 1. Then, the three ingots were cut and remelted into two groups of the specimens with a cylindrical geometry, having a diameter of 15 mm. The alloys were doped with different contents of P and B during remelting. The analyzed contents of P and B in the alloys are listed in Table 2. The alloys A1 and A2 were used to investigate the combination effect of P and B; the alloys B1 and B2 were used to study the effect of B; the alloys C1 and C2 were used to investigate the effect of P.

2.2 Heat Treatments

A homogenizing heat treatment was performed to remove the solidification segregation. A single γ phase microstructure with the homogeneous distribution of P, B and Nb as free atoms in matrix is obtained. The homogenization regime is as the following: 1120 °C/10 h, furnace heating/5 min, 1160 °C/1 h, furnace heating/5 min, 1190 °C/65 h. In order to confirm if the homogenization is efficient, the “Tag” method [13] was subsequently performed.

The homogenized specimens were cooled by two ways, water quenching (WQ) and air cooling (AC). The cooling rate of WQ was much higher than that of AC, which was designed to investigate the effect of different cooling rates on the micro-hardness and the precipitations of γ'' and γ' phases.

2.3 Microstructural Analysis and Mechanical Testing

The microstructures of the alloys were characterized by using scanning electron microscopy (SEM) and

Table 2 Analyzed compositions of the six alloys

Alloy no.	P (mass%)	P (atomic%)	B (mass%)	B (atomic%)
A1	0.001	0.002	0.0046	0.024
A2	0.017	0.031	0.009	0.048
B1	0.001	0.002	0.004	0.022
B2	0.001	0.002	0.010	0.054
C1	0.001	0.002	0.005	0.026
C2	0.015	0.028	0.005	0.026

transmission electron microscopy (TEM). The specimens for SEM were mechanically polished and then electrolytically etched in a solution of (10 ml HNO_3 + 30 ml HCl + 50 ml $\text{C}_3\text{H}_8\text{O}_3$) at 10 V. The sample for TEM was prepared by cutting a thin slice (~ 0.4 mm), grinding it to a thickness of about 0.06 mm and punching a 3-mm-diameter disk. The twin-jet electropolishing was performed in a solution of (10% perchloric acid + alcohol) at -20 °C and 24 V.

To evaluate the effects of P and B on the precipitations of γ'' and γ' phases in the grain interior, the Vickers micro-hardness testing was carried out at room temperature under the loading condition of 200 g for 15 s. The micro-hardness values were an average of at least 5 measurements per sample, and all the micro-hardness values are measured in different grains.

2.4 The Quantitative Analysis of P in γ' and γ'' Phases

To reveal if P can be dissolved in the γ matrix and take part in the precipitations of γ' and γ'' phases, the electrochemical extraction experiment was performed. The γ'' and γ' phases were extracted by dissolving the matrix in an electrolyte of (1.0% ammonium sulfate + 2.0% tartaric acid + water) under the condition of 0.025–0.03 A/cm² and below 5 °C [14]. The content of P in the extractant was measured by chemical composition quantitative analysis. X-ray diffraction (XRD) was carried out for phase analysis.

Table 1 Analyzed concentrations of the three master ingots (mass%)

Master alloy no.	Ni	Fe	Cr	Al	Ti	Nb	Mo	C
A	52.7	Bal.	19.18	0.64	1.11	5.22	3.05	0.058
B	52.6	Bal.	19.12	0.50	0.86	4.99	3.01	0.46
C	52.3	Bal.	19.15	0.48	0.90	5.30	3.08	0.043

2.5 First-Principle Calculations on the Roles of P in γ' and γ'' Phases

In order to study the mechanism of P accelerating the precipitations of γ' and γ'' phases, the formation enthalpies of γ' and γ'' phases when P atoms occupy the substitution sites (Ni, Al and Nb sites) in the lattices were examined by first-principle calculations. As shown in Fig. 1, the crystal structure of γ' phase is face-centered cubic (FCC). Ni atoms are at the face-centered positions, and any Ni site for the calculation is equivalent. Al atoms are at the corner positions, and any Al site for the calculation is also equivalent. The crystal structure of γ'' phase is body-centered tetragonal (BCT). Nb atoms are at the corner and body-centered positions, and any Nb site for the calculation is equivalent. Other sites are occupied by Ni atoms, and there are two types of Ni sites (Ni-1 and Ni-2) in the structure. The formation enthalpy when P occupies the two types of Ni sites has been calculated, respectively.

The total energies were calculated by a plane-wave pseudopotential method based on density functional theory, which were implemented in the VASP. The projector-augmented waves (PAW) potential was adopted to describe the electron–core interaction. The plane-wave cutoff energy was set as 400 eV. The energy tolerance of the electronic minimization was 1×10^{-6} eV. The geometry of the supercells including lattice parameters and atomic positions were fully optimized with the Hellman–Feynman force tolerance of 0.01 eV/Å.

For the calculation of the formation enthalpy, a $2 \times 2 \times 2$ supercell of the conventional Ni_3Al cell and a $2 \times 2 \times 1$ supercell of the conventional Ni_3Nb cell were constructed. Both the two supercells contain 32 atoms, for there were 4 atoms in each conventional Ni_3Al cell while 8

atoms in that of Ni_3Nb . The k-point mesh was set as $3 \times 3 \times 3$ after test. The formation enthalpy was calculated by the following equation:

$$\Delta_f H(\text{Ni}_x\text{Al}_y\text{P}_z) = \frac{1}{(x+y+z)} [E(\text{Ni}_x\text{Al}_y\text{P}_z) - xE(\text{Ni}_{\text{Fm}\bar{3}\text{m}}) - yE(\text{Al}_{\text{Fm}\bar{3}\text{m}}) - zE(\text{P}_{\text{P2/c}})]$$

$\Delta_f H(\text{Ni}_x\text{Al}_y\text{P}_z)$ and $E(\text{Ni}_x\text{Al}_y\text{P}_z)$ denote the formation enthalpy of $\text{Ni}_x\text{Al}_y\text{P}_z$ and the total energy of the supercell, respectively. $E(\text{Ni})$, $E(\text{Al})$ and $E(\text{P})$ mean the energy of Ni, Al and P in its relevant most stable state (eV/atom). The equation for the formation enthalpy of Ni_3Nb is similar to Ni_3Al , but the $\text{Al}_{\text{Fm}\bar{3}\text{m}}$ is replaced by $\text{Nb}_{\text{Im}\bar{3}\text{m}}$.

3 Results

3.1 The Effect of Combination Addition of P and B

As shown in Fig. 2a, the eutectic Laves phases surrounded by the needlelike δ phases are distributed in the interdendritic regions of typical as-cast microstructures. The MC carbides (NbC) with irregular shapes are also observed occasionally in the interdendritic regions. As shown in Fig. 2b, the Laves and δ - Ni_3Nb phases are all dissolved, and the dendritic morphology is disappeared in the homogenized alloys. Only the MC carbides and the naked grain boundaries are left in the homogenized alloys. As shown in Fig. 2c, the δ phases are distributed homogeneously in the grain interior of the alloys treated by “Tag” method, and dendritic morphology cannot be seen, which indicates that the elemental segregation had been removed efficiently. However, the γ' and γ'' phases can hardly precipitate after the specimens are annealed at 910 °C for 14 h based on the phase diagram of IN718 alloy [16].

As shown in Fig. 3, the micro-hardnesses of the alloys A1 and A2 are almost equal when the specimens are water-quenched after homogenization, while the micro-hardness of the alloy A2 is much higher than that of the alloy A1 when the specimens are air-cooled after homogenization.

As a precipitation strengthening alloy, the strength of IN718 alloy is dominated by the γ' and γ'' precipitations [17, 18]. It is reasonable to speculate that the different micro-hardnesses in Fig. 3 result from the different precipitations of γ' and γ'' phases. When the alloys are homogenized and water-quenched, no γ' and γ'' phases precipitate in the γ matrix of the alloys A1 and A2, which is confirmed by the TEM results in Fig. 4. As shown in Fig. 5, when the alloys are homogenized and air-cooled, although no γ' and γ'' phases precipitate in the γ matrix of the alloy A1, a great amount of γ' and γ'' particles distribute

Fig. 1 Sketch maps of γ' and γ'' lattices. Reproduced with permission from [15]

Fig. 2 Typical microstructures of the alloys: **a** the as-cast alloy; **b** the homogenized alloy; **c** the homogenized alloys performed by “Tag” method

Fig. 3 HV micro-hardnesses of the alloys A1 and A2 cooled by WQ and AC after homogenization

in the matrix of the alloy A2 (Fig. 5c, d). It indicates that the combination addition of P and B can accelerate the precipitations of γ' and γ'' phases in IN718 alloy during air cooling, which strengthens the matrix consequently.

3.2 The Effect of Single Addition of B

The results above reveal that the combination addition of P and B can accelerate the precipitations of γ' and γ'' phases during air cooling and strengthen the matrix of IN718 alloy. However, it is still not clear which element is responsible for the accelerating effect. So the effect of single addition of B on the micro-hardness was investigated subsequently. As shown in Fig. 6, the micro-hardnesses of the alloys B1 and B2 are almost equal when the specimens are water-quenched after homogenization, which consists with the results in Fig. 3. When the specimens are air-cooled after homogenization, the micro-hardnesses of the alloys B1 and B2 are equal, which is not consistent with the results in Fig. 3.

As shown in Fig. 7, no particles are observed in the bright-field images of the alloys B1 and B2 cooled by AC after homogenization, and only clear matrix is observed by TEM. This also reveals that no γ' and γ'' phases precipitate in the matrix. It indicates that B has no noticeable effect on the precipitations of γ' and γ'' phases during air cooling.

Fig. 4 Alloys A1 and A2 cooled by WQ after homogenization, dark-field images of γ'' and γ' phases and the SAED patterns with [001] zone axis: **a** Alloy A1, viewed along a (0 1 0) direction; **b** Alloy A1, viewed along a (1 $\frac{1}{2}$ 0) direction.; **c** Alloy A2, viewed along a (0 1 0) direction; **d** Alloy A2, viewed along a (1 $\frac{1}{2}$ 0) direction

3.3 The Effect of Single Addition of P

Although B has no noticeable effect on the precipitations of γ' and γ'' phases, we still cannot determine that the single addition of P can accelerate the precipitations of γ' and γ'' phases. The effect of single addition of P on the micro-hardness was investigated subsequently. As shown in Fig. 8, the micro-hardnesses of the alloys C1 and C2 are almost equal when the specimens are water-quenched after homogenization, while the micro-hardness of the alloy C2 is much higher than that of the alloy C1 when the specimens are air-cooled after homogenization. The results in Fig. 8 totally consist with the results in Fig. 3.

As shown in Fig. 9, when the specimens are homogenized and air-cooled, the bright-field image and corresponding SAED patterns indicate that no γ' and γ'' phases precipitate in the matrix of the alloy C1, and only clear

matrix is observed in the bright-field image. However, a great amount of γ' and γ'' particles precipitate in the alloy C2, which is confirmed by the TEM results in Fig. 9b. Therefore, the accelerating effect on the precipitations of γ' and γ'' phases during air cooling in IN718 alloy results from the effect of single P addition.

4 Discussion

The results in this study reveal that the accelerating effect of P on the precipitations of γ' and γ'' phases during air cooling is remarkable, while P has no effect on the precipitations of γ' and γ'' phases during water quenching. This is because the high cooling rate (WQ) prevents the γ' and γ'' phases precipitations from alloy matrix (Fig. 4), which make the effect of P on the precipitations of γ' and γ''

Fig. 5 Alloys A1 and A2 cooled by AC after homogenization, dark-field image of γ'' and γ' phases and the SAED patterns with [001] zone axis: **a** Alloy A1, viewed along a (0 1 0) direction; **b** Alloy A1, viewed along a (1 $\frac{1}{2}$ 0) direction.; **c** Alloy A2, viewed along a (0 1 0) direction; **d** Alloy A2, viewed along a (1 $\frac{1}{2}$ 0) direction

Fig. 6 HV micro-hardnesses of the alloys B1 and B2 cooled by WQ and AC after homogenization

phases disappear. What is more, the P and B atoms are frozen in the lattice of γ matrix during water quenching, while the micro-hardnesses of the alloys with P and B additions are almost equal (Figs. 3, 6, 8), so the effects of P and B atoms on the matrix micro-hardness can be ignored.

P can accelerate the precipitations of γ' and γ'' phases in the alloy during air cooling; this may result from the dissolution of P in the γ matrix and then takes part in the precipitations of γ' and γ'' phases. The effect of P on the formation enthalpies of γ' and γ'' phases are calculated based on first-principle theory. As shown in Table 3, the formation enthalpy of γ' increases about 3% when P occupies the Al lattice sites but decreases about 7% when P occupies the Ni lattice sites. It also reveals that the formation enthalpy of γ'' increases about 4.6% when P occupies the Nb lattice sites. However, the formation

Fig. 7 Bright-field images and corresponding SAED patterns of the alloys B1 and B2 cooled by AC after homogenization: **a** alloy B1; **b** alloy B2

Fig. 8 HV micro-hardnesses of the alloys C1 and C2 cooled by WQ and AC after homogenization

Table 3 Effect of P on the formation enthalpies of γ'' and γ' phases calculated by first-principle calculations

Phase	Supercell	$\Delta_f H$ (eV/atom)
Ni_3Al (γ')	$\text{Ni}_{24}\text{Al}_8$	- 0.475
	$\text{Ni}_{24}\text{Al}_7\text{-P}$	- 0.461
	$\text{Ni}_{23}\text{Al}_8\text{-P}$	- 0.508
Ni_3Nb (γ'')	$\text{Ni}_{24}\text{Nb}_8$	- 0.329
	$\text{Ni}_{24}\text{Nb}_7\text{-P}$	- 0.314
	$\text{Ni}_{23}\text{Nb}_8\text{-P (Ni-1)}$	- 0.339
	$\text{Ni}_{23}\text{Nb}_8\text{-P (Ni-2)}$	- 0.308

Fig. 9 Bright-field images and corresponding SAED patterns of the alloys C1 and C2 cooled by AC after homogenization: **a** alloy C1; **b** alloy C2

enthalpy of γ'' decreases about 3% and increases about 6% when P occupies the Ni-1 lattice site and Ni-2 lattice site, respectively. So it can be inferred that P decreases the formation enthalpies of γ' and γ'' phases by occupying the Ni-1 lattice sites in the two phases according to the first-principle calculations. The previous first-principle calculation indicated that P preferred to substitute host atoms, especially Ni atoms in γ -block [19, 20], and the recent study also inferred that P atoms occupied the substitution sites in the γ lattice [21]. The atoms distance and interstitial void volumes of γ' , γ'' and γ are similar, so it is reasonable that P tends to occupy the Ni atoms in the γ' and γ'' phases.

The calculated results above are based on the inference that P atoms can take part in the precipitations of γ' and γ'' phases. To further verify the above-calculated results, the inference must be experimentally proved. To precipitate much more γ' and γ'' phases for extraction, the sample of alloy C2 was homogenized and air-cooled, and aged as the following: 720 °C/8 h, furnace cooling/107 min, 620 °C/8 h AC. Then, the second phases, mainly γ' and γ'' phases, were electrochemically extracted with the same method as in previous study [14]. As shown in Fig. 10, the extractants contain γ' , γ'' and MC (NbC), which agrees with the previous studies [14, 22]. The concentration of P in the extractants was chemically analyzed to be 0.019 mass%, which is higher than the analyzed concentration of P in the alloy. The previous study found that P was extremely depleted in the MC carbides [3]. So it is obvious that P in the extractants is mainly dissolved in the γ' and γ'' phases. What is more, the concentration of P in the grain interior is lower than that in the alloy because P is surface-active element and tends to be segregated at the grain boundary [2, 6, 9, 23]. However, the analyzed concentration of P in the γ' and γ'' phases is higher than the analyzed concentration of P in the alloy, which indicates that the most of P

in the grain interior takes part in the precipitation of γ' and γ'' phases. The rationality of the first-principle calculation is partly proved.

The results above imply that it is possible for phosphorus atoms to take part in the nucleation of γ' and γ'' phases and accelerate the precipitations of the two phases during air cooling. The mechanism is that P occupies the Ni site and reduces the formation enthalpies of the two phases during nucleation. However, the content of P doped in the alloy is very low, and the P dissolved in the matrix is not enough for taking part in the whole precipitation progresses of γ' and γ'' phases. So the effect of P on the nucleation (the early stage of precipitation) of γ' and γ'' phases is more remarkable. The previous study revealed that P has no noticeable effect on γ' and γ'' phases after standard heat treatment (the γ' and γ'' phases fully grow) [8], which proves the above discussion.

Furthermore, it is reasonable to speculate that phosphorus might increase the stabilities of γ'' -Ni₃Nb and γ' -Ni₃Al by reducing their formation enthalpies [24], as has been demonstrated that the addition of phosphorus could increase the servicing temperature of IN718 alloy by 20–30 °C [25]. The previous results revealed that P could improve the stability of γ'' phases during long-time aging at 680 °C [12], which strongly proves the above speculation and the rationality of the first-principle calculation in Table 3.

5 Conclusions

The effect of phosphorus on the γ'' and γ' precipitations in IN718 alloy has long been ignored, because P and B have been deemed as typical surface-active elements which tend to be segregated at the grain boundaries and have an extremely low solubility in the γ matrix of the superalloys. However, the essential effects of P and B on the γ'' and γ' phases in the grain interior of IN718 alloy are re-recognized and revealed as follows:

1. The combination addition of P and B accelerates the precipitations of γ'' and γ' phases markedly. The accelerating effects on γ'' and γ' phases are resulted from P rather than B.
2. P can take part in the precipitations of γ' and γ'' phases, which is proved by electrochemical extraction and quantitative analysis of chemical composition. It also reveals that P atoms actually are dissolved in the γ matrix to a relatively high degree at the temperature that γ' and γ'' phases begin to precipitate, and subsequently the precipitations of γ' and γ'' phases are accelerated.

Fig. 10 X-ray diffraction pattern of the extractions of the alloy C2

3. The first-principle calculations indicate that P decreases the formation enthalpies of γ' and γ'' phases when it occupies the Ni lattice sites in the two phases, which explains the effect of phosphorus on the precipitations of γ' and γ'' phases.

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