THE EFFECT OF PHOSPHORUS, SULPHUR AND SILICON ON SEGREGATION, SOLIDIFICATION AND MECHANICAL PROPERTIES OF CAST ALLOY 718

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Abstract

Effects of trace elements P, S and Si on the microstructures and mechanical properties in cast alloy 718 have been studied. The results show that P, S and Si promote the segregation of Nb and formation of Laves phase. It was found that P and Si are significant Laves formers, with P enrichment in the Laves phase of about 1.0wt%, and Si enrichment of 2.06wt%. DTA results demonstrate that, as P, S contents in the alloys increased, the solidification temperature range increased obviously and the complete solidification was further delayed. On the contrary, Si raised the γ / Laves eutectic reaction temperature. Tensile testing indicated that P, S and Si had harmful effects on the tensile strength and ductility at RT and 650℃. When P, S and Si content exceeded 0.013wt%, 0.014wt%, 0.34wt% in the alloy, respectively, smooth stress–rupture life and elongations were reduced markedly.

Introduction

One of main problems in as–cast superalloys is the segregation of elements . How to reduce segregation is an important subject. In alloy 718, Nb is a primary element associated with Laves phase and carbides. It strongly segregates to interdendritic regions and promotes the formation of Laves phase, significantly affecting microstructure stability and mechanical properties[1]. Given the same cooling rate, the higher the Nb content of the alloy, the greater the volume fraction of Laves phase formed in the solidification microstructure. P, S and Si are impurity elements in superalloys, and are generally regarded as harmful, although little has been reported on the effect of phosphorus on the properties of superalloys. Recent studies showed P determined the Laves forming behavior of Nb [2]. R. G. Thompson et al reported that P segregated uniformly to grain boundary surfaces when it was present as an intentional additive [3]. In the solidification of alloy IN738, it has been shown that P increased the segregation of Al and Ti to the interdendritic region and the formation of γ / γ' eutectic which reduced stability of alloys[4]. Much attention has focused on sulphur in high–titanium–content alloys. The formation of Ti_2SC phase could reduce the effect of sulphur to some extent, but in alloy 718, titanium content is lower (about 1.0wt–%), and more work is necessary on the problem . Silicon may be used as a refining addition during melting, but its presence in the final alloys is considered detrimental and therefore upper limits are usually fixed at a low level in most alloy specifications. This research was aimed at the behavior of trace elements P, S and Si and their effects on the segregation of Nb during solidification , and the mechanical properties under different P, S and Si contents.

Materials and Experimental Proceeds

First, in order to eliminate the disturbance of other main elements by variation, a low impurity master heat IN718 was melted in vacuum induction furnace with the composition shown in Table 1.

Table 1 Chemical Composition of Master Heat IN718 (wt–%)

C	Ni	Cr	Mo	Nb	Ti	Al
0.031	53.19	19.07	3.04	5.20	1.05	0.53

B	Si	S	P	Mn	Fe
0.0055	<0.05	0.0038	0.0008	<0.03	bal.

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This heat was divided, remelted and doped with different P, S and Si contents(in the form of Ni–P, FeS and elemental Si, respectively), and subsequently cast by lost wax process to mechanical property specimen ingots. Casting temperature were 1420℃, with a shell–model preheat temperature of 900℃. P, S and Si contents in the alloys are shown in Table 2.

Table 2 P, S and Si Contents in the Cast Alloys (wt–%)

Alloy	P	S	Si
1	0.0008	0.0038	<0.05
2	0.0055	0.0038	<0.05
3	0.008	0.0038	<0.05
4	0.013	0.0038	<0.05
5	0.032	0.0038	<0.05
7	0.0008	0.014	<0.05
8	0.0008	0.051	<0.05
9	0.0008	0.0038	0.34
10	0.0008	0.0038	0.95

The above cast specimen ingots after heat treatment were machined into mechanical property specimens with gauge diameter of 5mm and gauge length of 25mm. Tensile properties at room temperature, 650℃ and 650℃ / 620MPa rupture life were measured. The heat treatment process was: 1090℃ / 1h / AC + 950℃ / 1h / AC + 720℃ / 8h / FC 50℃ / h → 620℃ / 8h / AC. The microstructures were studied by metallographic methods and an electron microprobe was mainly used to observe and measure element segregation. Fracture surfaces were studied by SEM.

The DTA experiments were performed on a Setaram HTC1800 instrument with a α–Al_2O_3crucible. The DTA cell was calibrated using pure Ni(>99.99%). The precision of this system, using Pt–Rh thermocouples, was determined to be better than 5℃. Specimens weighing 0.3g were heated at a fast rate of 100℃ / min to 1450℃ under a flowing helium atmosphere and were held for five minutes before them were cooled at a rate of 15℃ / min to 1000℃. The solidified samples were subsequently cooled at a fast rate(150℃ / min) to ambient temperature. The DTA curves were recorded for the temperature range from 1450℃ to 1000℃. Reaction temperatures were determined by finding the temperature at which the DTA curves deviated from the local baseline.

Results

Microstructure and segregation

The microstructures of specimens in this study varied with composition, with the P, S and Si contents increasing in the alloy. The amount of Laves phase precipitating from liquid increased, especially for P–doped alloys and Si–doped alloys, giving the alloys a tendency to form well defined dendritic structures(Fig.1).

P and Si were heavily enriched in the final solidified γ / Laves eutectic phase or Laves phase during solidification process(Figures 2, 3). In the high–P–content alloy(0.032wt%P), the P content in the Laves phase reached 0.982wt%, about 30 times over average of that doped in the alloy. Nb content in the Laves phase also increased with the increasing P content(Table 3). In the high–Si–content alloy(0.95%Si), the Si content in the Laves phase reached 2.06%. Table 4 shows that S promoted the segregation of Nb, with the more S doped in the alloy, the more heavily Nb segregated in the interdendritic regions and Laves phase.

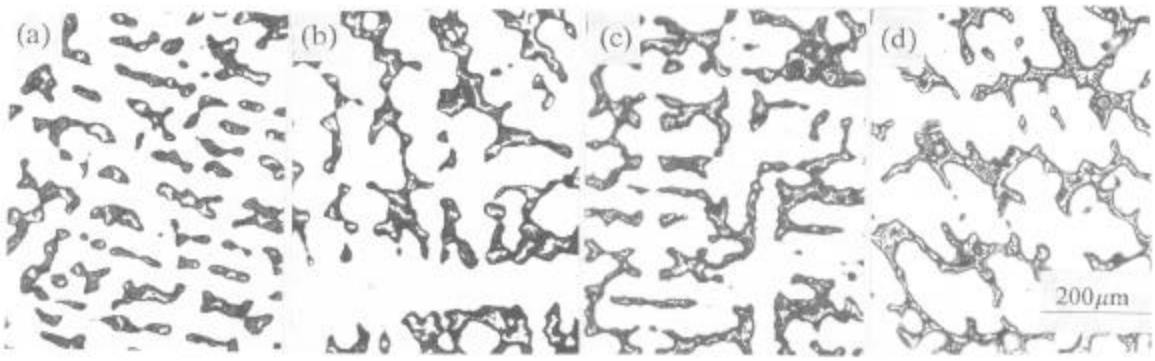

Figure 1: Effect of P, S and Si contents on microstructures of cast alloy 718
(a) No.1, base–alloy (b) No.5, 0.032wt%P (c) No.8, 0.051wt%S (d) No.10, 0.95wt%Si

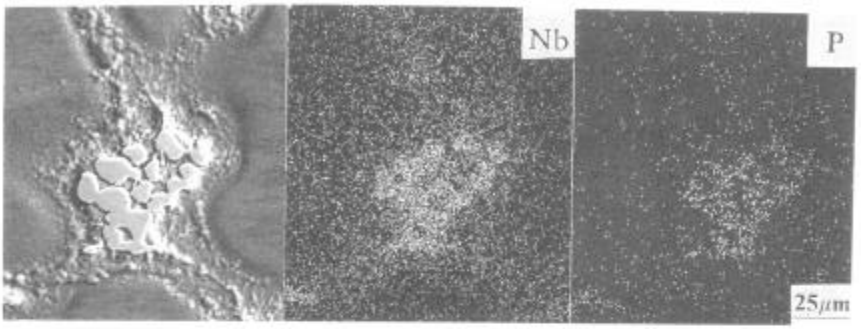

Figure 2: Elemental scanning images in alloy No.5 (0.032wt%P)

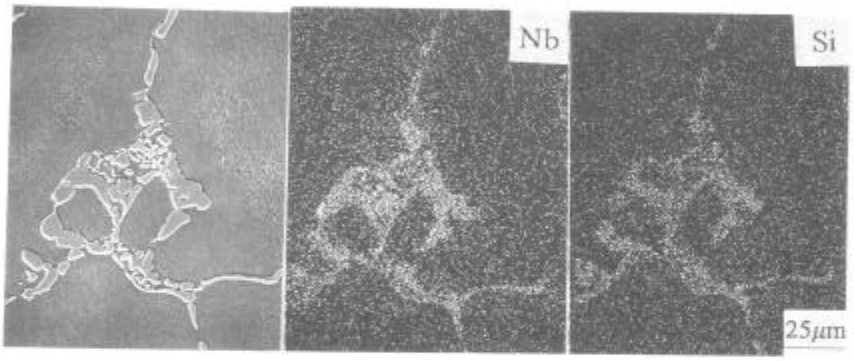

Figure 3: Elemental scanning images in alloy No.10 (0.95wt%Si)

Table 3 Average Composition of Laves Phase in the Alloys Doped with P (wt-%)

Alloy	P	Nb	Fe	Ti	Cr	Mo	Ni
1	0.036	26.619	9.957	1.608	10.649	3.558	bal.
3	0.243	29.904	9.455	1.927	10.059	3.792	bal.
5	0.982	30.886	11.391	0.950	12.818	4.738	bal.

Table 4 The Analysis of Nb Segregation in Alloys Doped with S

Alloy No.	S Wt-%	Dendritic Core	Interdendritic Regions	Laves
1	0.0038	3.16	5.00	15.98
7	0.014	1.71	5.70	20.88
8	0.051	1.55	6.36	21.68

DTA

The DTA cooling curves of cast alloy 718 with different P, S and Si contents have been determined. The data obtained from the DTA cooling curves were tabulated in table 5. It was found that P or S had little effect on the incipient solidification temperature($\gamma_{liquidus}$) and solidification temperature of MC, while Si obviously reduced $\gamma_{liquidus}$. The γ / Laves eutectic reaction temperatures varied with P, S and Si contents in the alloys. With increasing of P and S contents, the final solidificatio n(γ / Laves eutectic reaction) temperature was reduced and the solidification temperature range increased. In contrast, Si raised the γ / Laves eutectic reaction temperature and reduced the solidification temperature range. The S-doped alloy solidified at following sequence: L $\rightarrow$ L +γ $\rightarrow$ L +γ+ Sulphide $\rightarrow$ L +γ +Sulphide + MC $\rightarrow$ γ +Sulphide + MC + γ / Laves(Eutectic).

Table 5 Characteristic Temperatures Obtained from the DTA Cooling Curves

Alloy	$\gamma_{liquidus}$	MC	Sulphide	Eutectic	$\triangle T^{1)}$
1	1329	1229	–	1157	172
3	1321	1225	–	1139	182
5	1321	1225	–	1129	192
8	1329	1225	1200	1143	186
10	1307	–	–	1186	121

1) $\triangle T$ Solidification temperature range (℃)

Mechanical properties

Tensile testing at room temperature and 650℃ with different P, S and Si contents was conducted. The results showed that P, S and Si were harmful to tensile strengthand reduced ductility in the alloys. The smooth stress–rupture life and elongation with different P, S and Si contents have also been determined at the conditions of 650℃ / 620MPa. It showed that smooth stress–rupture life and elongation were reduced with increasing of P, S and Si contents. When phosphorus, sulphur, silicon contents in the alloys are in excess of 0.013wt%, 0.014%, 0.34%, respectively, life and elongation dropped rapidly.

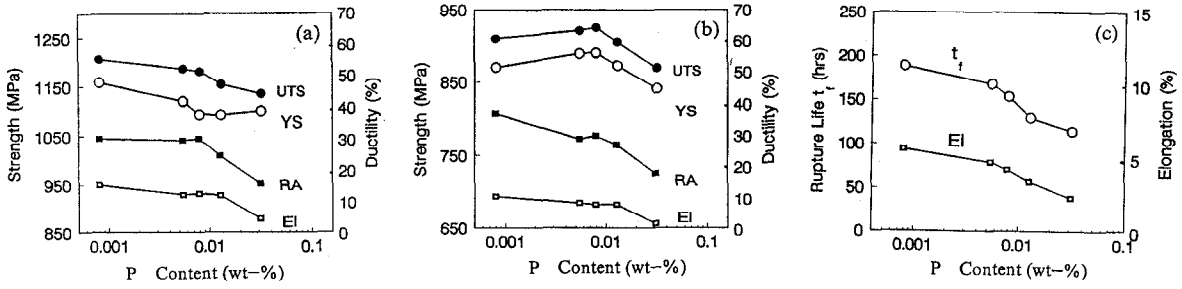

Figure 4: Effect of P content on mechanical properties of cast alloy 718
(a) at room temperature (b) at 650℃ (c) at 650℃ / 620MPa

Table 6 Effect of S Content on Tensile and Rupture Properties of Cast Alloy 718

Alloy	S	YS, MPa		UTS, MPa		El, %		RA, %		SR[1)]	
No.	Wt–%	RT	650℃	RT	650℃	RT	650℃	RT	650℃	Life,h	El,%
1	0.0038	1159	870	1207	910	16.0	10.8	30.6	37.0	188.5	5.7
7	0.014	1102	823	1164	864	8.7	8.7	24.0	36.0	78.0	3.6
8	0.051	1040	808	1105	841	7.3	8.2	21.0	21.5	19.0	2.0

1) SR Stress–rupture properties

Table 7 Effect of Si Content on Tensile and Rupture Properties of Cast Alloy 718

Alloy	Si	YS, MPa		UTS, MPa		El, %		RA, %		SR	
No.	Wt–%	RT	650℃	RT	650℃	RT	650℃	RT	650℃	Life,h	El,%
1	<0.05	1159	870	1207	910	16.0	10.8	30.6	37.0	188.5	5.7
9	0.34	1100	878	1179	889	8.7	6.7	24.0	32.0	136.5	4.2
10	0.95	982	795	988	858	2.0	1.8	2.6	7.8	57.0	2.4

Discussion

The alloy IN718 solidifies in the following sequence: L → L + γ → L + γ + MC → γ + MC + γ / Laves. This is verified by DTA curves of the alloys. The solubility of P, S, Si in nickel is very low with a maximum of about 0.3wt%[5], 0.005wt%[6], <0.5wt%, respectively, so that P, S and Si should mostly enrich in the final solidification regions and result in heavily segregation. The higher the P, S and Si contents doped in the alloys, the more they would be enriched in interdendritic areas.

The segregation of P, S and Si elements promotes the segregation of Nb, this could be seen from above. Nb is a primary element in the Laves phase and the Laves phase is generally accepted to be the form (Ni, Fe, Cr)$_2$(Nb, Mo, Ti) [1]. Therefore, the more Nb segregated in interdendritic regions, the higher volume fraction of Laves formed.

The effects of P, S and Si on the mechanical properties mainly lie in the following: P, S and Si segregate in the final regions, and promote the segregation of Nb to form Laves phase. Laves phase has been generally accepted as being deleterious to the mechanical properties of the alloy. It was associated with reduced tensile strength and ductility, because it is a brittle phase, even at elevated temperature, it is likely to act as a preferred crack initiation and propagation site [1]. The greater volume fraction of Laves phase in the alloys presents a higher probability for the crack to grow through the brittle phase or γ / Laves interfaces. In addition, Laves consumes large amounts of Nb depleting the matrix of Nb which is the principal hardening element. Stress–rupture fracture surfaces were studied by SEM. It was found that the fracture surfaces included grain boundary, eutectic interface, γ / MC interface. With the increasing of P, S and Si contents in the alloys, they showed greater tendency to brittle fracture. That would explain why the high P, S and Si alloys with gross Laves showed reduced rupture strength and ductility.

Conclusions

1 P, S and Si are enriched mostly in the final solidification regions, and P, Si become formers of Laves phase.

2 P, S and Si promote the segregation of Nb and the formation of Laves phase.

3 The solidification temperature range is widened with increasing of P and S contents in the alloy. On the contrary, Si raised γ / Laves eutectic reaction temperature and reduces solidification temperature range.

4 Tensile strength and ductility at room temperature and 650℃ are reduced with increasing of P, S and Si contents in the alloy.

5 Smooth stress–rupture life and elongations are reduced obviously markedly with increasing of P, S and Si contents in the alloy.

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