

**EFFECT OF P,S,B AND Si ON THE SOLIDIFICATION
SEGREGATION OF INCONEL 718 ALLOY**

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Abstract

The effect of P,S,B and Si on the solidification process of Inconel 718 has been studied in laboratory tests. It essentially revealed the tendency of P,S,B and Si to greatly aggravate the solidification of Inconel 718 . A low segregation Inconel 718 ingot developed based on the laboratory study and a conventional Inconel 718 ingot were parrallel melted in industrial scale for segregation comparison, which confirmed the laboratory results.

Introduction

Severe solidification segregation, a common problem in today's advanced superalloys, is one of the key obstacles to further explore their properties. Much effort has been paid on alleviating the solidification segregation throughout the world. But these researches are largely in technological aspects and less attention has been focussed on composition study, especially trace elements.

The Institute of Metal Research started studying the effect of trace elements on the solidification segregation of superalloys at 1979. By using a new method[1] different from the commonly used thermal differential analysis, it became possible for us to discover the existence of some structures with low melting points during the late stage of solidification which could keep unsolidified at rather low temperature.

According to microprobe determination, in spite of very low average contents in alloys, P, Zr, B and Si contents were extremely high in this type of low melting point structure[1] (as much as several hundreds or even a thousand times higher than their average contents in alloys) and resulted in the enlarging of solidification range up to 50–100°C. Based on this discovery, a series of low segregation cast, wrought and directionally solidified superalloys with improved properties have been developed[2,3]. This achievement was patented in China[4], U.S. A. [5] and West Europe[6].

Present paper is to study the effect of these trace elements on the solidification process and segregation in Inconel 718 alloy.

Experimental Procedure

Two conditions are necessary for a trace element to severely promote the solidification segregation of superalloys: one is its lack of solubility in solid solution; the other is the ability to greatly drop the melting point of alloy. In superalloys, Bi, Ti, Sb etc. meet these two needs, but their contents are always controlled to extremely low levels and have little effect on solidification segregation, while those elements such as P, Zr, B and Si have comparatively high upper limits in superalloy specifications and have the ability to form eutectics with Ni with the melting points of 880°C (Ni_3P), 1172°C (Ni_5Zr), 1045°C (Ni_3B) and 1154°C (Ni_3Si) respectively, therefore, they give the necessary conditions to greatly aggravate solidification segregation. S has a very limited solubility in Ni solid solution and an eutectic reaction with Ni at 600°C, so it should have a stronger effect than P, Zr, B or Si in promoting solidification segregation, but high Ti contents in most superalloys greatly weaken its damage by form-

ing a high melting point compound. In Inconal 718 alloy, there is no Zr and Ti content is not high, resulting in the necessity of studying the effect of S element. The present paper is to study the effect of P, S, B and Si on the solidification segregation of Inconel 718 alloy.

In order to avoid the disturbance of other elements, a master alloy ingot was first melted via VIM with the analytical composition (wt%) of 5.15Nb, 0.94Ti, 0.45Al, 18.80Cr, 17.16Fe, 3.03Mo, 0.02C, 0.0023B, 0.0026S, 0.001P, 0.02Si and Ni bal. Then 10Kg ingots with different P, S, B and Si contents were melted via VIM with the same process. Their compositions are listed in Table 1.

Table 1. Contents of trace elements of test alloys (wt%)

Alloy No.	1	2	3	4	5
Adding	—	0.007B	0.25Si	0.010S	0.010P
Analytical	—	0.009B	0.26Si	0.012S	0.010P

Specimens were taken from the central sections of the ingots and their Laves fractions were analysed with electrochemical separation method, the extraction solution is 1050ml methanol, 50ml HCl and 100ml glycerine. Specimens of about $10 \times 10 \times 10$ mm were cut from the ingots for the measurement of the solidification process. The specimens were first heated to 1420°C , held 5min to make sure that they were melted and homogenized, then furnace cooled to a desired temperature, held 10min and water quenched.

Micro-zone composition analysis was conducted with a Camebax-Micro microprobe device.

Metallographic specimens were etched in an electrolyte of $\text{H}_3\text{PO}_4(40\%) + \text{H}_2\text{SO}_4(35\%) + \text{H}_2\text{O}(25\%)$ or $\text{HCL}(20\%) + \text{ethanol}(80\%)$.

Experimental Results

Laves fractions of the test ingots

Laves fractions in the central sections of the test ingots analysed with electrochemical separation method are given in Table 2. It shows P, S, B and Si obviously increase Laves fraction.

Table 2. Laves fractions in the central sections of the test ingots

Alloy No	1	2	3	4	5
Laves, wt%	0.22	0.34	0.36	0.37	0.45

Table 3. Electron microprobe data of Nb contents (wt%)

Position	No.1	No.2	No.3	No.4	No.5
initially solidified γ	2.3	2.1	1.8	1.8	2.0
γ solidified at T_B	11.3	9.7	8.7	9.6	10.0
Liquid at T_B	20.2	23.8	23.0	24.2	24.6

Table 4. Structure changes during solidification.

No.1	No.2	No.3	No.4	No.5
L	L	L	L	L
1350 ^o C L+ γ	1350 ^o C L+ γ	1350 ^o C L+ γ	1350 ^o C L+ γ	1350 ^o C L+ γ
1150 ^o C L+ γ +Laves	1140 ^o C L+ γ +Laves	1170 ^o C L+ γ +Laves	1175 ^o C L+ γ +Y	1150 ^o C L+ γ +Laves
1140 ^o C γ +Laves	1110 ^o C γ +Laves	1150 ^o C γ +Laves	1160 ^o C L+ γ +Y+Laves	1100 ^o C γ +Laves
			1110 ^o C γ +Y+Laves	

Measurement of solidification process

Measuring the solidification process of an alloy can essentially reveal its tendency to solidification segregation. Table 3 shows microprobing Nb contents in the initially solidified γ solid solution and in the γ solid solution and liquid at solidifying frontier at eutectic reaction beginning temperature (T_B). As we can see, in spite of very low adding amounts, P, S, B and Si significantly lower the Nb solubility in γ solid solution. The reason is that these elements enriched in the liquid at solidifying interface so

heavily that they change the equilibrium concentration. In the early stage of solidification, S has the largest effect. By considering that Si has an order of magnitude higher addition, its effect is after that of P and B. In the late stage of solidification, the promotion effect of S to segregation weakens and Nb solubility in γ solid solution has some rise (No.4 alloy). In addition, P,S,B and Si also obviously raise the Nb content of liquid at eutectic reaction temperature.

Table.4 shows the structure changes of the test alloys during solidification. It can be seen that the minor additions of these elements have little influence on the beginning solidification temperature, but more obvious on the final solidification temperature(T_f). Among them, P,S and B decline T_f , but Si slightly raise it. In addition, Y phase occurs in the high S containing alloy (No.4).

Discussion

In Inconel 718 alloy, P,S and B lower T_f , whereas Si raise T_f , this does not mean that P,S and B aggravate solidification segregation and Si reduces it. Both the Laves fraction analysis and microprobe results show that all these four elements greatly promote solidification segregation. The reason is that Si is a strong Laves-forming element, its severe segregation leads to the early forming of Laves phase which dramatically lowers the Si content in the residual liquid, resulting in the rise of melting point of the residual liquid.

S promotion to solidification segregation weakens in the late stage of solidification (No.4 alloy) . It can be explained that Ti and S enrichment favors Y phase formation at higher temperature (1175 $^{\circ}$ C) which lowers S content in the residual liquid and relieves its damage.

According to the microprobe data and structure change during solidification, the qualitative effect of P,S,B and Si on Inconel 718 pseudo-binary phase-diagram can be shown in Fig. 1. Some important conclusions can be reached.

a)P,S,B and Si aggravate dendritic segregation by decline the solidus, i.e. lowering the Nb content in γ solid solution at equilibrium with the liquid at the solidifying interface.

b)P,S,B and Si move point M leftward, i. e. decrease the largest Nb solubility in γ solid solution. That will flatten the Nb concentration gradient between interdendrite and dendrite arm, and hinders Nb homogenization during heat treatment.

c) The leftward motion of point M and rightward motion of point O by P,S,B and Si will favor the formation of isolated, blocky Laves instead

of eutectic Laves. In the present test, the Nb contents of points M and O in No.1 alloy with low P,S,B and Si contents are about 11% and 20% respectively, while those in No.2, No.3, No.4 and No.5 with high P, S, B or Si are about 10% and 24% respectively. Therefore, the liquid composition of No.1 alloy at T_B lies at the middle section of the eutectic reaction line (MO / ON = 9 / 10) instead of the deviation toward Laves phase side (MO / ON = 14 / 6) of other alloys. Moreover, rightward motion of point O will increase Laves fraction.

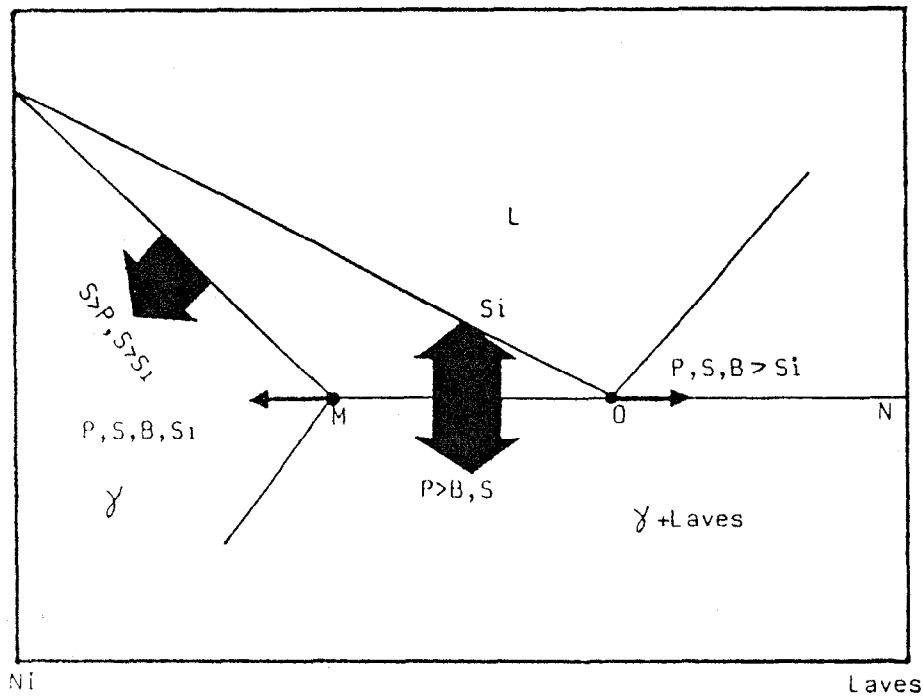


Fig.1. Effect of P,S,B and Si on Inconel 718 pseudo-binary phase diagram.
Segregation of Φ 406mm Industrial Ingots

Based on the above results, the significant deterioration effect of P,S,B and Si even at very low levels has been confirmed. Their contents should be controlled as low as possible. But the technical feasibility and cost prevent them from unlimited decrease. Our research shows that the following specification is acceptable: $P < 0.001$, $Si < 0.05$ and $B 0.002 - 0.004\%$. S effect was paid little attention in the past because of the presence of high Ti in most cast or wrought superalloys. Now, how to greatly drop the S content under the P and Si content requirement and to figure out a proper S content specification are still under study.

The low segregation Inconel 718 alloy developed according to the above composition requirement is now going to be applied in industry. The fol-

lowing is the segregation comparison of the low segregation and conventional $\Phi 406$ mm Inconel 718 ingots.

Table 5 lists the analytical composition of these two ingots. Fig.2 shows the distribution of Laves phase fraction from ingot centre to edge obtained by electrochemical separation method (ESM) and image analysis system (IAS). It can be seen that the results of these two methods are very consistent. It shows that the Laves fraction of the conventional Inconel 718 is much higher than that of low segregation one from centre to edge.

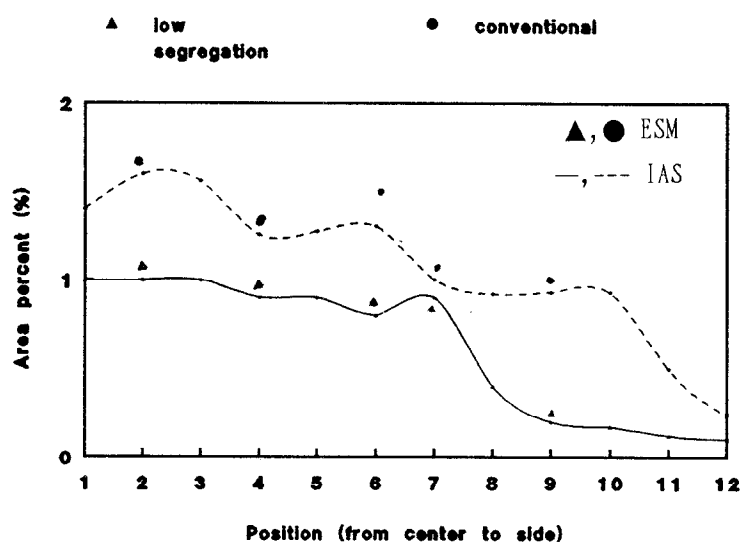


Fig.2. Distribution of Laves fraction in $\Phi 406$ ingots.

Fig.3 and 4 respectively illustrate the distribution of blocky and eutectic Laves fractions obtained with IAS. It can be seen that most of the blocky Laves in low segregation alloy are within $2/3R$ and eutectic Laves within $1/3 \sim 2/3R$, while the existence of the blocky Laves in conventional alloy obviously widens and the presence of the eutectic Laves plateau greatly deviates to ingot edge. It can be explained with fig.1. Laves morphology depends on two factors. One is the cooling rate. The quick cooling rate favors the formation of eutectic Laves phase, but the slow one favors the formation of blocky Laves. The other is the liquid composition location (point O) on the eutectic reaction line. The eutectic Laves is easy to form if point O situates at the middle section of the eutectic reaction line, and the blocky one is easy to form when it deviates towards Laves side. Therefore, the low segregation alloy is easier to form eutectic Laves at the same cooling rate or its eutectic can form at slower cooling rate. This is the reason why eutectic Laves of low segregation alloy devi-

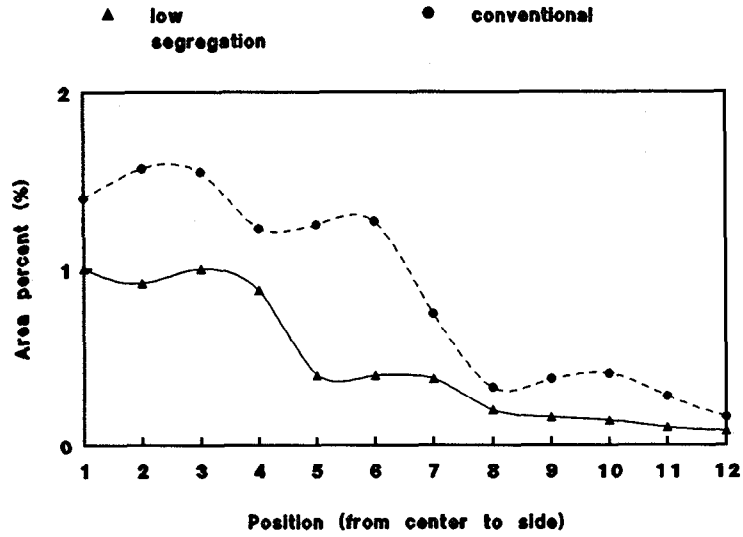


Fig.3. Distribution of blocky-Laves fraction in Φ 406 ingots.

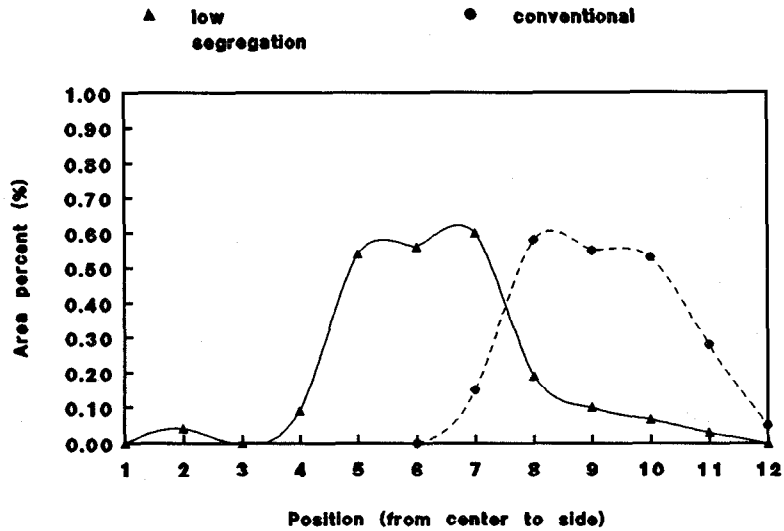
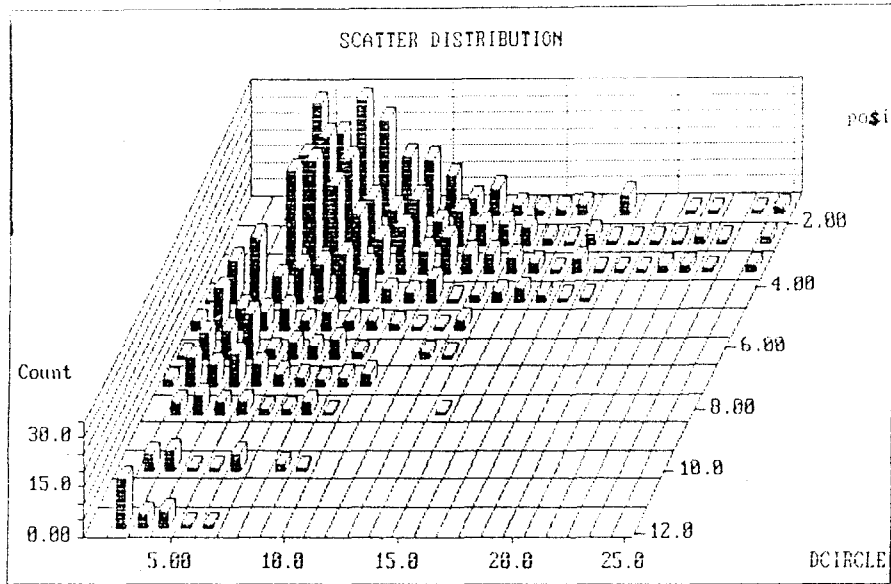
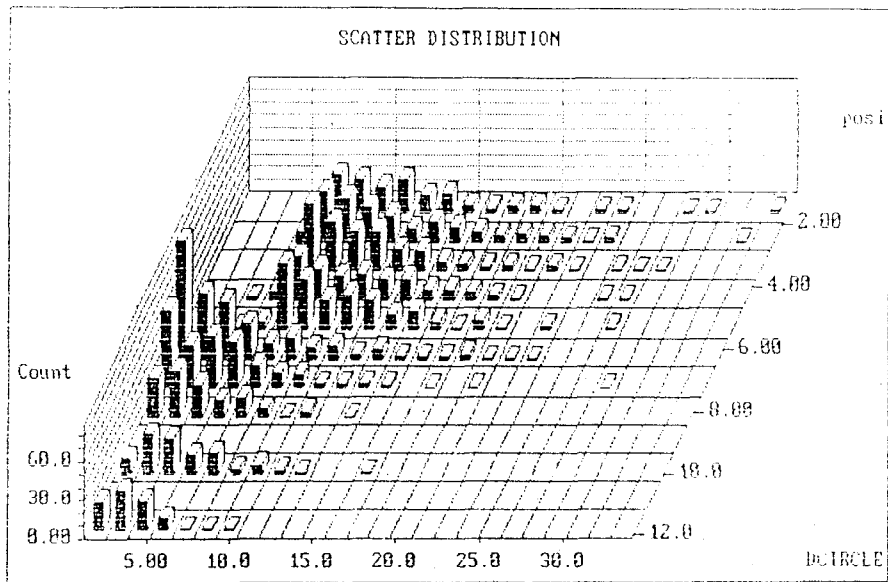


Fig.4. Distribution of eutectic-Laves fraction in Φ 406 ingots.



(a)



(b)

Fig.5. Intensity distribution of blocky-Laves particle perimeter.
 (a) Low segregation alloy, (b) Conventional alloy

ates toward ingot centre and the blocky Laves region becomes narrow.

Table 5. Analytical compositions of $\Phi 406\text{mm}$ ingots.

Alloy	C	Si	S	P	Ni	Cr	Nb	Mo	Ti	Al	B
Low segregation	0.033	0.04	0.0035	0.0007	52.52	18.23	5.15	2.97	1.02	0.53	0.004
Conventional	0.036	0.11	0.0035	0.005	52.35	18.47	5.15	3.06	1.03	0.51	0.004

Fig.5. shows the intensity distribution of blocky Laves particle size. It can be seen that most of blocky Laves particles in low segregation alloy are 4–10 μm compared with 6–15 μm in conventional alloy. The obvious increase of Laves particle size in conventional alloy also results from the rightward deviation of eutectic reaction liquid composition, which leads to the increase of the Laves portion in eutectic reaction product.

Conclusion

In Inconel 718 alloy, P, S, B and Si exhibit obvious promotion effect to solidification segregation. Dislike other high Ti superalloys, its insufficient Ti content for the formation of high melting point compound with S makes its segregation be significantly aggravated by S. The segregation promotion effect of these elements reflects in the following aspects:

- a) Deviate the solidus of γ phase toward low Nb side in phase diagram;
- b) Increase the fraction and particle size of Laves phase significantly.

References

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