

# Interactions Between P and S or B Trace Elements on Creep Property of Inconel 718 Alloy

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**Abstract:** Optimum content of phosphorus in Inconel 718 alloy has a pronounced beneficial influence on creep resistance. Increasing phosphorus to 0.02% causes an increase of the apparent activity energy from 681kJ/ mol to 744kJ/ mol. It has been suggested that the significant beneficial effect of phosphorus be mainly caused by the inhibition of diffusion along grain boundary. It was also proved that there is a strong beneficial interaction between P and B and a weak detrimental interaction between P and S. The preexponential frequency constant is related with the creep activation energy because of the so-called compensation effect. The compensation temperature in Inconel 718 alloy is about 1080K, which in turn corresponds to the transformation temperature from coarsened  $\gamma'$  phase to  $\delta$  phase.

## 1 Introduction

It has long been realized that relatively small changes in composition can cause significant changes in properties of superalloys<sup>[1,2]</sup>. In the crowd of all the trace elements that are still common in modern commercial superalloys, boron, sulfur and phosphorus are somewhat unusual. Earlier in the late 1950s it was recognized that boron markedly improved creep properties and hot-workability<sup>[3]</sup>. Since that time, boron has been used as a universal-strengthening element in almost all nickel-base superalloys. On the other hand, sulfur has been known for more than 100 years to be harmful<sup>[4]</sup> and hence has to suffer strict limitation in superalloys<sup>[5]</sup>. As for phosphorus, the situation is rather complicated, perhaps, a bit controversial. It, like sulfur, has long been regarded as a detrimental impurity in superalloys<sup>[1,2,5,6]</sup>. However, experimental evidences for its beneficial effects are

increasingly found since last decade<sup>[7,8]</sup>. Recent investigations revealed that phosphorus is playing a role similar to boron, significantly increasing the stress rupture life of alloy 718<sup>[9,10,11]</sup> and some other alloys<sup>[12,13]</sup>. As a result, the effects of phosphorus throwback to the original classification presented by Bieber and Decker<sup>[4]</sup> who made the first systematic study of the influences of trace elements and viewed phosphorus as an element “beneficial in small amounts but harmful in larger additions”. This recurrence has a twofold significance: it leads to a new stride in the full understanding of the sophisticated effects of trace elements and it sets an urgent demand for the reexamination of our current knowledge related with phosphorus.

In this work the creep behavior of alloy 718 doped with different contents of trace elements was systematically studied. The three dopants, boron, sulfur, and phosphorus, were chosen, because: 1) they may typify three interesting categories of trace elements according to the above statements, 2) a strong interaction between boron and phosphorus has been found in alloy 718<sup>[14]</sup>, and 3) a possible competition between sulfur and phosphorus has been proposed in Cr-Mo-V steel<sup>[15]</sup>. The purpose of the present work is not emphatically to discuss the respective effect of each element separately, but to approach a possible general mechanism from different aspects.

## 2 Experimental

Seven test materials of 10kg per heat, manufactured from one master ingot of commercial alloy 718, were prepared by induction melting under vacuum. One heat, alloy M, remained the regular trace element level of the master alloy; others were doped with different contents of boron, sulfur and/or phosphorus. Thus, all the test alloys have the same matrix composition (wt%), namely Ni 52.65, Cr 18.36, Nb 5.26, Al 0.55, Ti 1.02, Mo 3.03, C 0.038, Si 0.13, S 0.002, Mg 0.004, and Fe the balance. The boron, sulfur, and phosphorus contents of the alloys are listed in Table 1.

The 10kg ingots were first forged at 1110°C to produce 35mm square bars, then rolled to 16mm diameter bars at the same temperature. Creep specimen blanks cut from the rolled bars were subjected to the following standard heat treatment: solutionized at 965°C for 1 hour followed by air cooling, then aged at 720°C for 8

hours followed by furnace cooling to 620°C and held at this temperature for 8 hours, then air cooled. This yielded an equiaxed grain size within the range of 8.0~10.5µm and a uniform distribution of  $\gamma''$  and  $\gamma'$  precipitates with rod-like grain boundary  $\delta$ -phase particles. Consistent with the results of other investigators<sup>[9,10,11]</sup>, no notable microstructural difference was observed at the scanning electron microscopy (SEM) level and the as heat-treated microstructures of these seven alloys were essentially identical.

Table 1 Boron, sulfur, and phosphorus contents of the test alloys / wt%

Alloy	M	dS	dPS	dP	dB	dPB	dP'B
Boron	0.005	0.004	0.005	0.005	0.011	0.009	0.010
Sulfur	0.002	0.020	0.017	0.002	0.003	0.002	0.002
Phosphorus	0.003	0.003	0.016	0.020	0.004	0.020	0.026

Creep specimens consist of threaded ends and a gage section of 8mm diameter by 100mm long. The test temperatures were checked directly using three chromel-alumel thermocouples attached to the specimen gage section. The creep strain was recorded with respect to time using a dual-dial gage averaging type extensometer attached to the shoulders of the specimens. All tests were performed into the steady state creep regime. After the steady-state was well established, the test was terminated either at a given time or at 1% strain, and then the specimen was allowed to cool down inside the chamber.

The microstructures of the as-crept samples were examined by SEM. All the specimens were mechanically polished first, then electropolished for 20 seconds at 25 volts in a solution of 20% H<sub>2</sub>SO<sub>4</sub>-80% methanol. After that, the specimen were rinsed in hot water and fresh alcohol and then cleaned ultrasonically in fresh alcohol. The electropolished specimens were etched at 5 volts for 5~15 seconds in a solution of 1 part HCl, 3 parts HNO<sub>3</sub>, and 5 parts glycerine. Finally, the specimens were rinsed as after the electropolishing step.

### 3 Results

#### 3.1 The Steady-State Creep Rate

The variation of steady-state creep rate with applied stress and with temperature for all the alloys are presented in Fig.1 and Fig.2 respectively. From

these figures it can be seen that trace amounts of boron, sulfur, and phosphorus have remarkable effects on creep behavior of alloy 718. Consistent with the results in the literature<sup>[14]</sup>, the phosphorus-doped alloys, dP, dPB, and dPS, clearly show higher creep resistance than their counterpart none-phosphorus-doped alloys, M, dB, and dS. The harmful effects of sulfur are evident that the two sulfur doped alloys, dS and dPS, have higher creep rate than their counterpart low sulfur alloys, M and dP.

However, the two high boron alloys, dB and dPB, have lower creep resistance than their counterpart alloys, M and dP with conventional content of boron. This probably means that excessive boron is not beneficial for alloy 718.

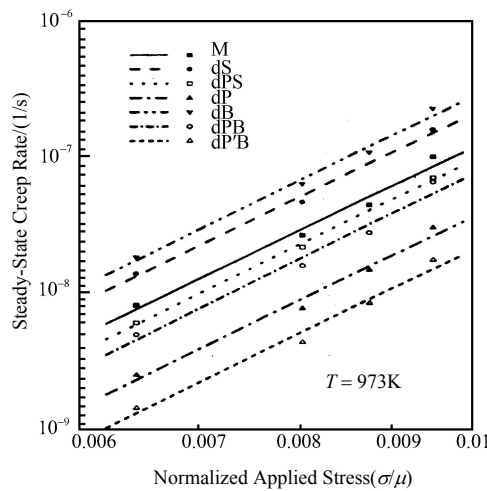


Fig.1 Stress dependence of steady-state creep rate at 973K in a double-logarithm coordinate. also included are the straight lines drawn according to equation (2)

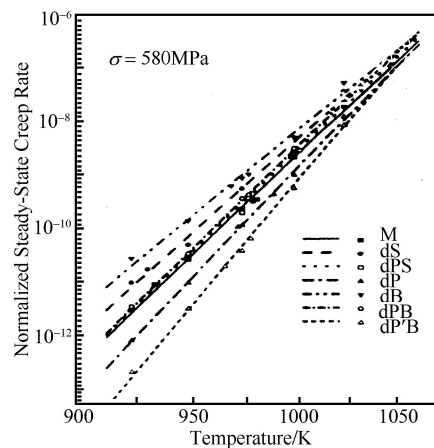


Fig.2 Temperature dependence of steady-state creep rate at 580MPa. The creep rate has been normalized according to equation (4). Also included are the straight lines predicted by Equation (4)

In general, creep behavior usually obeys the following Dorn equation<sup>[16]</sup> where

$\sigma$  is the

$$\dot{\varepsilon} = \frac{AD\mu b}{kT} \left( \frac{\sigma}{\mu} \right)^n \quad (1)$$

applied stress,  $D$  is the lattice diffusion coefficient,  $\mu$  is the shear modulus,  $b$  is the Burgers vector,  $k$  is Boltzmann's constant,  $T$  is the absolute temperature,  $A$  is a dimensionless constant, and  $n$  is the stress exponent. At a given temperature, the stress dependence of steady-state creep rate can be described by an even simpler power-law

$$\dot{\varepsilon} = A' \left( \frac{\sigma}{\mu} \right)^n \quad (2)$$

where the pre-exponential temperature-dependent factor

$$A' = \frac{AD\mu b}{kT} \quad (3)$$

Equation (2) is easy to be transformed into a linear relation through taking logarithm and has been fitted to the data shown in Fig.1. The values of  $n$  and  $A'$  determined by the linear-least-square regressions are summarized in Table 2. The straight lines in Fig.1 are predicted by equation (2) with the parameters listed in this table. The parallelism on Fig.1 between the straight lines is obvious, which suggests that within the experimental error, the slopes of the straight lines are identical each other between the seven alloys. In other words, the apparent exponents of the seven alloys are almost equal to the same value, which is slightly higher than 6 (see Table 2). This indicates that the additions of boron, sulfur, and/or phosphorus appear not to change the stress exponent of alloy 718.

From the above analysis, one can see that the effects of boron, sulfur, and phosphorus likely focus on the pre-exponential temperature-dependent factor,  $A'$ , only. According to equation (3),  $A'$  is a function of  $D$ ,  $\mu$ ,  $b$ , and  $T$ . Since the tensile properties of alloy 718 are not significantly influenced by boron<sup>[17]</sup>, sulfur<sup>[10]</sup>, and phosphorus<sup>[9,10,11]</sup>, it is logical to think that the shear modulus,  $\mu$ , is independent of these trace elements. Because  $b$  is possibly insensitive to trace elements, it can be concluded that the effects of boron, sulfur, and phosphorus predominantly concentrate on the diffusion coefficient,  $D$ . As is known to all,  $D$  is in accordance with Arrhenius formula and thus Dorn equation (1) can be rewritten

as

$$\dot{\epsilon} \left[ \frac{\mu b}{kT} \left( \frac{\sigma}{\mu} \right)^n \right]^{-1} = A'' e^{-Q/RT} \quad (4)$$

where  $A''=AD_0$  is a pre-exponential frequency constant,  $D_0$  is the diffusion frequency factor,  $Q$  is the creep activation energy. The term on the left hand of equation (4) can be regarded as the normalized creep-rate, which has taken the temperature dependence of shear modulus into account. Equation (4) is also easy to be transformed into a linear relation through taking logarithm. Let  $\mu_{\text{[MPa]}}=90190-30T$ , and  $b=0.249\text{nm}$  for all the alloys (the  $T$  dependence of  $\mu$  only slightly influences the following calculations; and  $b$  is employed only to determine the value of  $A''$  which has no any substantive effect on all the calculations), then fit equation (4) to the data shown in Fig.2. The results are also listed in Table 2. The activation energies for creep are found to be higher than the value for self-diffusion in nickel-base superalloy Nimonic 90,  $(364\sim 502)\text{kJ/mol}^{[18]}$ , and significantly higher than that in pure nickel,  $279.5\text{kJ/mol}^{[19]}$ . However, the activation energies obtained in this work are roughly consistent with the results of McKamey et al.<sup>[20]</sup> who found the creep activation energy of commercial alloy 718 was  $(569\sim 769)\text{kJ/mol}$ .

### 3.2 Microstructure

The microstructures of different alloys, which have crept for identical time, are different in a way. Fig.3 gives two examples in which the microstructures of both alloy M and alloy dP were taken from samples crept for 600 hours at 923K and 580MPa. At this time, M had been reached the tertiary stage of creep, whereas dP is still within the secondary creep stage. The characteristics of the precipitates in both alloys are similar, which confirms the observation of Horton et al.<sup>[21]</sup> who found the phosphorus addition did not influence the thermal stability of the precipitates; but the states of grain boundaries exhibit some distinction. There is no indication of deformation in alloy dP (Fig.3(b)), while the deformation is evident at the grain boundary of alloy M where, between the triple point and the tip of a grain boundary phase, a cavity has already been shaped as indicated by the arrow in Fig.3(a). At first sight Fig.3 may be employed as an evidence indicating the grain-boundary-cohesion enhancement caused by phosphorus addition, which mechanism has been proposed by several investigators<sup>[8,9]</sup>. But on a close

examination, this mechanism can not get direct support from Fig.3 alone because the creep strains of the two samples are different. The strain experienced by M is more than two times as high as that by dP, which makes the comparison between their grain boundary states unfair. Fig.3, however, does indicate boundary states unfair. Fig.3, however, does indicate something associated with grain boundary (see below in the discussion).

A reasonable comparison of the microstructures, which are taken from samples terminated at the same creep strain rather than at the same time, shows no notable difference between different alloys. Two examples are presented in Fig.4. It can be seen that, When stopped at 1% strain, the microstructures of alloy dP and alloy M are essentially identical at the SEM level, though the creep resistance of dP is much higher than M due to the addition of phosphorus (see Fig.1 and Fig.2). The only distinction lies in that to reach the same creep strain more time needs to be spent for dP than that for M.

Table 2 Apparent stress exponent  $n$ , the pre-exponential temperature-dependent factor  $A'$ , the pre-exponential frequency constant  $A''$ , and the Creep activation energy  $Q$  determined by the linear-least-square regressions of Equation (2) and Equation (4) respectively

Alloy	M	dS	dPS	dP	dB	dPB	dP'B
$n$	6.21	6.23	6.29	6.23	6.28	6.36	6.24
$A'/(10^5/s)$	3.00	5.75	3.47	1.01	9.86	3.79	0.60
$Q/(kJ/mol)$	681.0	634.8	683.4	744.1	582.7	692.5	843.6
$A''/(10^{26} m^2/s)$	9.011	0.066	14.24	10019	$1.85 \times 10^{-4}$	49.50	$9.13 \times 10^8$

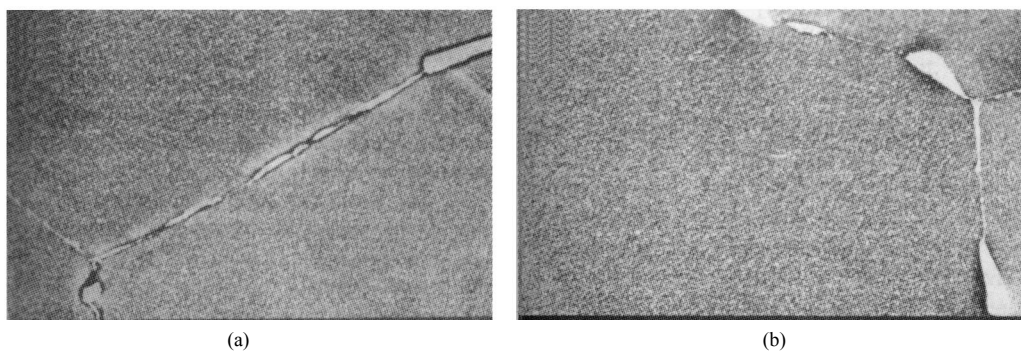


Fig.3 Microstructures of (a) Alloy M and (b) Alloy dP Crept for 600h at 923K and 580MPa. The Corresponding Creep Strains of M and dP are 1.3% and 0.5% Respectively. The Arrow in Photograph (a) Points to a Cavity. The Tensile Axis is Horizontal

A more meaningful comparison can be made between the incubation times for different alloys to reach the same creep stage. For example, the times

corresponding to the inflexion point in the creep curve (strain vs. time) for different alloys, are listed in Table 3. Combining Fig.3, Fig.4, Table 2, and Table 3, an important conclusion can be drawn that the creep of alloy 718 are either retarded or speeded by additions of the trace elements depending on their specific effects on the diffusion coefficient.

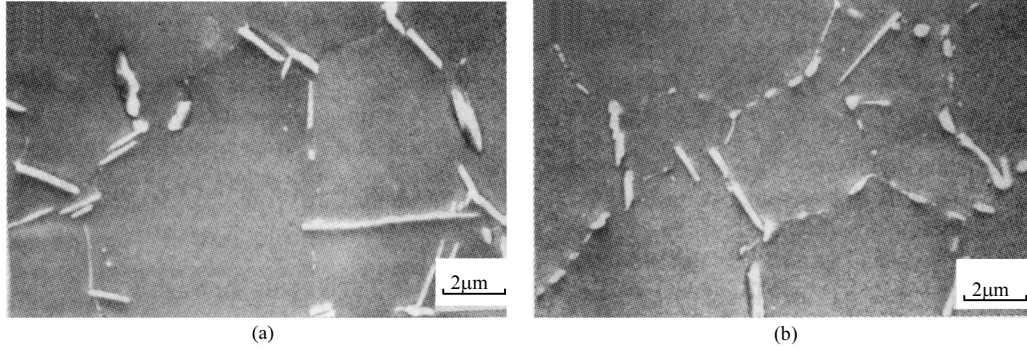


Fig.4 Microstructures of (a) alloy M and (b) alloy dP crept at 973K and 535MPa to 1% strain. The corresponding times for M and dP are 26.8h and 90.5h respectively. The tensile axis is horizontal

## 4 Discussion

### 4.1 Interactions Between the Trace Elements

According to Table 2, the creep activation energy,  $Q$ , varies with trace element content. When two elements, namely  $x$  and  $y$ , are concurrently doped, they produce a synergistic effect on  $Q$ . Denote this effect with sign  $Q(x, y)$ , where  $x$  and  $y$  are the contents of  $x$  and  $y$  respectively. If and only if the effects of  $x$  and  $y$  are independent each other, their individual effects,  $Q(x, y_0)$  and  $Q(x_0, y)$ , are additive and thus the net synergistic effect is equal to the sum of the two net individual effects with respect to the master alloy where  $x_0$  and  $y_0$  are the contents of  $x$  and  $y$  in the master alloy respectively; otherwise, between the net synergistic effect and the sum there must exist a difference  $Q_{x-y}^{\text{excessive}}$ , which quantitatively characterizes the strength of the  $x$ - $y$  interaction. Apparently,

$$Q_{x-y}^{\text{excessive}} = Q(x, y) - Q(x, y_0) - Q(x_0, y) + Q(x_0, y_0) \quad (5)$$

From Table 2, it is easily shown that

$$Q_{P-B}^{\text{excessive}} = Q_{dPB} - Q_{dP} - Q_{dB} + Q_M = +46.7\text{kJ/mol}$$

and



$$Q_{P-S}^{\text{excessive}} = Q_{dPS} - Q_{dP} - Q_{dS} + Q_M = -14.5\text{kJ/mol}$$

This means that within the composition range listed in Table 1, the P-B interaction increases the creep activation energy of alloy 718, which confirms the results of Ref. 14 and 20; whereas the P-S interaction slightly decreases the energy. To the knowledge of the present authors, this is probably the first report in the literature that clearly reveals the detrimental interaction between phosphorus and sulfur on the creep behavior of alloy 718. A simple comparison indicates that the beneficial P-B interaction is slightly weaker than the beneficial individual effect of phosphorus, whereas the detrimental P-S interaction is much weaker than the detrimental individual effect of sulfur.

Table 3 Effect of boron and phosphorus on the time corresponding to the inflexion point in the creep curve (strain vs. time) at 973K and different applied stress levels

Alloy	M	dP	dB	dPB	dP'B
535MPa	5.7	15.0	6.2	17.0	30.2
580MPa	3.0	11.1	3.4	7.3	13.0

## 4.2 Compensation Effect

One significant feature of Fig.2 is that the creep rate differences between different alloys tend to fade gradually as the temperature increases. In other words, all the straight lines tend to converge to a vanishing

$$[Q(x, y) - Q(x_0, y_0)] = [Q(x, y_0) - Q(x_0, y_0)] + [Q(x_0, y) - Q(x_0, y_0)] \quad (6)$$

point. The basic reason for this phenomenon lies in a profound interdependence between the pre-exponential frequency constant,  $A$ , and the activation energy,  $Q$  (see Table 2). This interdependence has been termed "compensation effect" by Shvindlerman and coworkers<sup>[22-24]</sup>. In the present case, the change of  $Q$  caused by the trace elements in turn induces a corresponding change of  $A$  so that a large portion of the effect of the changing  $Q$  on the creep rate is remarkably counteracted. This effect is illustrated in Fig.5, which show a clear linear relationship between  $\ln[A]$  and  $Q$  then the compensation temperature,  $T_c$ , which is corresponding to the vanishing poin in Fig.2, can he determined as  $T_c \approx 1080\text{K}$ . Below 1080K,

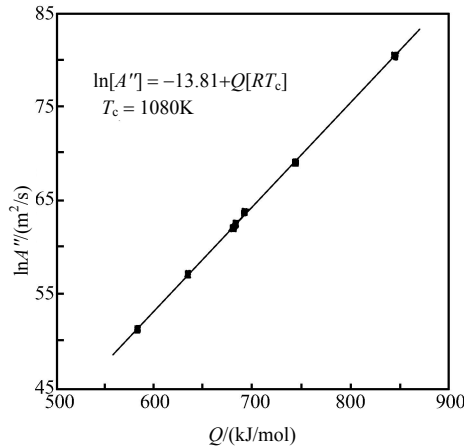


Fig.5 Dependence of the logarithm of the pre-exponential frequency constant  $A''$  on the creep activation energy due to the so-called compensation effect.

The compensation temperature,  $T_c$ , is found to be 1080K

the observed effects of trace elements, such as the harmful effect of sulfur, and the beneficial effect of phosphorus, still hold; theoretically, above 1080K the effects of boron, sulfur, and phosphorus will become reversed. However, the straight lines can not be practically extrapolated beyond 1080K because near this temperature the principal mechanism of alloy 718, mainly due to the presence of  $\gamma''$ , will breakdown.

$$\ln[A''] = -13.81 + 1.117 \times 10^{-4} Q \quad (7)$$

Let

$$\left[ 1.117 \times 10^{-4} Q - \frac{Q}{RT} \right]_{T_c} = 0 \quad (8)$$

### 4.3 Effects on grain boundary

Compensation effect is generally observed in the thermally activated grain boundary processes and represents a more fundamental principle of thermally activated grain boundary phenomenon<sup>[22-24]</sup>. The surprisingly excellent linear-correlation demonstrated by Fig.5 is beyond all expectations. This strongly suggests that the effects of boron, sulfur and phosphorus be surely associated with some thermally activated grain boundary processes. As a matter of fact, it is generally accepted that the trace elements invariably affect grain boundary properties through enrichment at grain boundary<sup>[1,6]</sup>. The segregation of a solute can affect three aspects of the Properties of a grain boundary, namely energetics, mechanics, and kinetics<sup>[25]</sup>. Most of the trace elements significantly reduce the

grain-boundary energy and hence strongly segregate to it, which in turn serves as a prerequisite for this element to effectively act upon grain boundary. In simple terms, presence of a segregant will certainly affect the state of bonding across a grain boundary and consequently will influence both the cohesion and the self-diffusion of it. Auger analyses have firmly proven that boron<sup>[26]</sup>, sulfur<sup>[26]</sup>, and phosphorus<sup>[9,21,26]</sup> do segregate to grain boundaries in alloy 718. Since the remarkable effects of trace elements mainly focus on the diffusion coefficient,  $D$ , the above discussion implies that there must be an inner link between  $D$  and some thermally activated grain boundary processes. Such a link, however, can not be inferred from the original form of Dorn equation (1).

In fact, the weakness of Dorn equation (1) has been demonstrated by Ashby and coworkers<sup>[27,28]</sup>. It may be remedied by replacing the lattice diffusion coefficient in Dorn equation (1) with an effective diffusion coefficient,  $D_{\text{eff}}(D_g, D_{\text{gb}})$ , which has taken both lattice diffusivity and grain boundary diffusivity into account, where  $D_g$  and  $D_{\text{gb}}$  are diffusion coefficients of grain interior and grain boundary respectively. This is equivalent to assume that the transport of matter via grain boundary contributes significantly to the overall diffusive transport of matter. If this is really true, the trace elements, say boron, sulfur, and phosphorus, thus find their way to exert influences on creep behavior. Consequently, at least part of the effects of trace elements can be readily attributed to their indirect-effect on  $D_{\text{eff}}$  by way of their direct-effect on  $D_{\text{gb}}$ .

On the other hand, it is also generally accepted that grain boundary may slide at a wide range of velocities which depend on the magnitude of the driving force, the temperature, the crystallography, and the grain boundary composition. Ashby<sup>[29]</sup> has analyzed this phenomenon in terms of viscosity,  $\eta_{\text{gb}}$ . According to his theory, the sliding rate of grain boundary is given by

$$\dot{\varepsilon}_{\text{gb}} = \delta \tau / \eta_{\text{gb}} \quad (9)$$

where  $\tau$  is the shear stress,  $\delta$  is the effective thickness of grain boundary. Because viscosity  $\eta_{\text{gb}}$  is inversely proportional to  $D_{\text{gb}}$ , any effect of trace elements on  $D_{\text{gb}}$  would tend to influence  $\eta_{\text{gb}}$  and thus  $\dot{\varepsilon}_{\text{gb}}$ . The grain boundary sliding in turn has two important effects on the creep behavior. First, the grain boundary sliding will increase the overall creep rate markedly by increasing the deformation rate of grain interior through an accommodation mechanism<sup>[30,31]</sup>. The value of the

enhancement factor of the total creep rate due to this effect ranges from about 1.5 to 100<sup>[32]</sup>. Secondly, it has well documented that grain boundary sliding plays an important role in the cavity (or crack) nucleation and growth process<sup>[33,34]</sup>. This effect, unfortunately, may be confused with the direct effects of trace elements on the grain boundary cohesion. For example, from Fig.3 alone it can not be judged whether phosphorus directly enhances the grain boundary cohesion because the cavity in Fig.3(a) may be formed by the relatively rapid grain boundary sliding (indirect effect on cohesion) even if the cohesion of alloy M is identical to that of alloy dP. This effect makes the analysis of grain boundary cohesion even more complicated and difficult.

In short, trace elements tend to strongly segregate to grain boundary. They modify the effective diffusion coefficient through affecting the grain boundary diffusivity, individually or cooperatively (through some kinds of interaction), and thus either retard or speed the entire creep process. This mechanism, though favorite at least in interpreting the present experimental results, does not exclude other possible mechanisms, for example, mechanisms special for phosphorus that involve grain boundary cohesion<sup>[8,9]</sup>, grain boundary phase and oxidation<sup>[10]</sup>, and some kinds of elements interactions<sup>[13,14]</sup>. In view of the complexity, the effects of trace elements on high temperature properties surely can not be attributed to a single mechanism. Indeed, it is quite possible that several mechanisms operate concurrently.

## 5 Conclusions

(1) Trace amounts of boron, sulfur, and phosphorus have remarkable effects on creep behavior of alloy 718. The additions of boron, sulfur, and/or phosphorus appear not to change the stress exponent. The influences of these trace elements mainly concentrate on the effective diffusion coefficient.

(2) There exist a beneficial interaction between phosphorus and boron and a detrimental interaction between phosphorus and sulfur. The beneficial P-B interaction is slightly weaker than the beneficial individual effect of phosphorus, whereas the detrimental P-S interaction is much weaker than the detrimental P-S interaction is much weaker than the detrimental individual effect of sulfur.

(3) The pre-exponential frequency constant strongly correlated with the creep activation energy due to the so-called compensation effect, which indicates a

strong effect of the trace elements on the thermally activated grain boundary process (grain boundary diffusivity). The compensation temperature is about 1080K.

(4) It has been proposed that boron, sulfur, and phosphorus segregate to grain boundaries and thus affect the effective diffusion coefficient individually or cooperatively through affecting the grain-boundary diffusivity, which in turn either retard or speed the creep process.

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