THE INFLUENCE OF B, P AND C ON HEAT-AFFECTED ZONE MICRO-FISSURING IN INCONEL TYPE SUPERALLOY

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Abstract

The influence of minor elements, viz., C, B and P, present either individually or in combination, on HAZ micro-fissuring susceptibility of Inconel 718 Superalloy was studied. The micro-fissuring susceptibility was evaluated by hot ductility testing in a Gleeble 1500 system and by the measurements of cracks in HAZs around electron beam welds. It was then correlated to the microstructure of the alloys and the segregation of minor elements to the grain boundaries. It was observed that B affected the HAZ micro-fissuring susceptibility very adversely, but the influence of C was only marginal. The addition of P to the B containing alloy made it more susceptible. However, the addition of C to both the B, and B and P containing alloy was beneficial in reducing the HAZ micro-fissuring susceptibility. The influence of minor elements could be reasonably explained by the segregation and/or formation of precipitates on grain boundaries.
Introduction

An understanding of the effect of minor elements to the base composition of Inconel\textsuperscript{1} 718 is essential to improving the mechanical properties of the alloy. The effect of elements such as C, B and P on the properties of Superalloys is complex, with their influence being dependent on the chemical composition of the alloy and heat treatment history. Carbon provides a source of carbides, whereas the role of Boron in Superalloys has been generally believed to be beneficial, especially for mechanical properties such as creep parameters. P is generally considered a tramp element, though concentrations of up to 0.015 wt. % in Inconel 718 have been reported, \cite{1-3}. Allvac has also concluded that the effect of a combined addition of B and P, on creep strength of IN718, is much greater than the sum of individual effect of P and B. The role of the elements B, C and P on weldability has been less clear, though initially Pease \cite{4} considered B to be harmful. C has been considered to be either detrimentally affecting the weldability \cite{5}, or innocuous \cite{6}. The effect of P has been generally reported to be detrimental to weldability. Savage \cite{7} for example indicated a tripling of the total crack length (TCL) values in Inconel 600 for an increase in P from 0.001 to 0.01 wt.%

Previous work by the authors has shown significantly adverse influence of B on the micro-fissuring susceptibility of weld HAZ in IN718 \cite{8-10}, however to-date, no detailed studies have been published on the effect of P on weldability of IN718. In addition, the influence of the combined addition of C, B and P on the micro-fissuring behavior of IN718 has not been studied. This communication, therefore, presents the results of a study of the effect of controlled additions of C, B and P individually, and in combination, on the weldability of 718 type alloys.

Experimental Methods and Materials

16.0 mm diameter hot rolled bars, of Inconel 718 type alloys with varying B, C and P concentrations (Table 1) in a base of 53.16\% Ni, 19.38\% Fe, 17.8\% Cr, 2.87\% Mo, 5.06\% Nb+Ta, 0.92\% Ti, 0.63\% Al, 0.10\% Si, 0.004 \% S, 0.0017\% Mg, were provided by Allvac. The as-received bars were swaged in two steps, with two intermediate annealing treatments of 1 hour each at 1200°C and 1050°C to produce 6 mm diameter rod suitable for testing in a Gleeble thermal simulator. The specimens were given a final solution heat treatment at 1050°C for 45 minutes followed by two cooling rates, viz, air cooling 1°C/sec and water quenching 284°C/sec.

Scanning electron microscopy with energy dispersive x-ray (EDX) analysis and analytical transmission electron microscopy electron microscopy were used for metallography and phase analysis. Secondary Ion Mass Spectroscopy (SIMS) was carried out to determine the grain boundary segregation of the minor elements.

The weldability of all the alloys was evaluated by hot ductility testing in Gleeble 1500 thermo-mechanical simulation system and by electron beam welding. For hot ductility testing 6.00 mm dia.x100mm long specimens were heated to a peak temperature of 1210°C, held there for 5 seconds and then cooled to the test temperature and tensile tested to failure. The ductility of the specimens at the test temperature was measured by the reduction in area. The Ductility Recovery Temperature (DRT) and the Liquidation Cracking Temperature Range (LCTR) was used to evaluate the Heat Affected Zone (HAZ) liquation cracking tendency.

Table 1: Concentration of C, P and B in various IN718 Base Alloys

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C (wt. %)</th>
<th>B (wt. %)</th>
<th>P (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>988 Base</td>
<td>0.008</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>982 Base + C</td>
<td>0.031</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>989 Base + B</td>
<td>0.005</td>
<td>0.013</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>991 Base + C + B</td>
<td>0.031</td>
<td>0.012</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>T91 Base + B + P</td>
<td>0.006</td>
<td>0.010</td>
<td>0.022</td>
</tr>
<tr>
<td>726 Base + C + P</td>
<td>0.030</td>
<td>&lt;0.001</td>
<td>0.022</td>
</tr>
<tr>
<td>727 Base + B + C + P</td>
<td>0.033</td>
<td>0.011</td>
<td>0.022</td>
</tr>
</tbody>
</table>

Electron beam welding was carried out at 40 kV, 30 ma and 77 cm/min. Ten metallographic sections were polished and etched and the length of individual micro-fissures measured on an SEM. The results are reported as the average of the Total Crack Length per section (Av. T.C.L.)

Results

Microstructural Analysis

The microstructure of the base alloy (988) was essentially single phase FCC of about 90 µm grain size with a few particles dispersed within the grains and at grain boundaries, as shown in a typical SEM micrograph in Fig. 1(a).

![Fig 1 (a) S.E.M. Image](image-url)
These particles (78 at. % Nb/16 Ti) were observed to be rich in B and C as shown in an EDX spectrum in Fig. 1b. Due to the software limitation it was not possible to quantify the B and C concentrations. The analysis of selected area diffraction patterns (SADP), obtained by TEM examination of these particles extracted on carbon replicas revealed them to correspond to MC type particles with FCC crystal structure and a lattice parameter of about 0.45 nm. The microstructure of the higher carbon alloy (982), after air-cooling as well as water quenching, was essentially the same as that of 988 except that a few TiN precipitates were also observed.

The microstructure of high B and low C alloy (989) contained two types of precipitate. One was Nb-rich carbide containing B, as was observed in the base alloy (988) and another 38 Nb/31 Mo/23 Cr/5Fe (at. %) containing precipitates; in addition about 3 at. % P was observed in some of the precipitates. The latter particles were also extracted on a carbon replica and a representative TEM micrograph is shown in Fig. 2.

The SADP analysis of these particles revealed them to be tetragonal M3B2 particles with a lattice parameters of $a = 0.57$ nm and $c = 0.22$ nm. The crystal structure and lattice parameter of these particles is the same as was observed by Vincent in the HAZ of Inconel 718 [11]. The alloy containing C and B, (991), also had the same general microstructure with Nb-rich carbide particles containing B, along with some Ti-rich carbides. The EDX spectra revealed the presence of both C and B in the precipitates, but the Nb/Mo/Cr/Fe tetragonal boride was not observed in this alloy.

The microstructure of the high B and high P alloy (T91) was, in general, similar to those of the other alloys, with Nb-rich B containing carbide precipitates being present. However, some P (up to 5 at.%) was also observed in a few Nb/Ni/Cr containing particles that were present in this alloy. In addition, 15-20% of the precipitates examined were of M3B2 type borides whose chemical composition was the same as that of the particles observed in alloy 989. Fig 3 (a) shows an S.E.M. image of a collection of Nb/Mo/Cr/Fe boride particles, along with an E.D.X. spectrum from one of the precipitates, Fig 3 (b) and X-ray maps for B, C, P, Ni and Mo in Fig 3 (c). A few Ti-rich carbides, with less than 5% of the other elements in them, were also observed.
The two remaining alloys, high C and high P (726) and high C, high B and high P alloy (727) also exhibited the same general microstructure. The high C and high P alloy (726) contained mainly Nb-rich B containing carbide particles of varying sizes. Minor amounts of Nb/Ni/Ti/Cr/Fe-rich precipitates were also present. This phase was also found to be FCC with a lattice parameter of 0.45 nm, i.e. it was a compositional variation of the MC phase. Fine carbides (<1.0 μm) were observed in the highly alloyed (B, C, P) material (727), which were of the Nb-rich type. No B was observed in any of the EDX spectra; however, some Ti-rich carbides were present.

Segregation Analysis by Secondary Ion Mass Spectroscopy
The segregation of B, C and P was examined by a Cameca Secondary Ion Mass Spectrometer (SIMS). The analysis of alloy 988 and 982 only showed the presence of carbon as precipitate, Fig 4(a), corroborating the observations by SEM and TEM, while a very small degree of segregation of B at the grain boundaries was also observed in the A.C. alloy.

The high C, high B and high P alloy (727) exhibited the segregation of B at the grain boundaries (Fig. 4d) in the AC specimens but only a few B containing particles were observed. In the water-quenched specimens the segregation of B to carbides was even more limited. This is consistent with the SEM/EDX observations, where only carbon was observed to be present in the particles and B was not observed. The SIMS analysis of P in high P containing alloy (T91) did not reveal any segregation of P either to the grain boundaries or to the precipitate particles, although some P was detected in a few particles by SEM/EDX analysis. It is, therefore, likely that P was generally present as monolayer in the grain boundaries but could not be detected by the SIMS used in this study. Horton et al [3] detected segregation of B, C and P to grain boundaries, but only a few M3P precipitates of < 50 nm in size were observed.

SIMS analysis of alloy 989, containing high B concentration, revealed the presence of B in particles as well as on grain boundaries (Fig. 4 b). However, the degree of segregation of B at grain boundaries was smaller in the WQ specimens as compared to the air-cooled (AC) specimens, as was also observed by the authors in their previous studies [9-10]. The high C and high B alloy (991) exhibited similar images, especially in the AC specimen, though the TEM studies did not reveal the presence of boride particles. The B-ion images in SIMS are thus likely to be from Nb-rich carbide particles instead of boride particles. In the AC high B and high P alloy (T91), segregation of B was observed at the grain boundaries as well as in the precipitate particles (Fig. 4c). However, a significantly small degree of segregation was observed in the water quenched specimen, as would be expected if segregation of B was to occur by non-equilibrium segregation mechanism. It should be noted that in the high C and high P alloy when B concentration was less than 10 ppm, some B segregation was observed at the precipitates. This is corroborated by the SIMS/EDX observations of B and C segregating to the precipitates.
HAZ microfissures during welding usually form because of a lack of ductility in the material near its melting point. Therefore hot ductility measurements in a Gleeble thermo-mechanical simulator are conducted to study the weldability of a material. One of the commonly used parameter is called ductility recovery temperature (DRT). It is defined as the temperature at which a material recovers its ductility on cooling from the melting point or peak temperature[12]. The DRT values of various alloys were measured by hot ductility testing in a Gleeble 1500 system. The 6 mm dia. X 100 mm specimens were heated at a rate of 1500°C/s to 1210°C, held there for 5 seconds then cooled to various temperatures and tested to failure. The effect of C, B and P, individually and in combination with each other, on the weldability was analyzed by plotting the DRT values in four different graphs shown in Fig. 5 a-d. As seen in Fig. 5a the base weldability was highest.

**Fig 4 (c) S.I.M.S. B Image T91 A.C.**

**Fig 4 (d) S.I.M.S. B Image 727 A.C.**

**Hot Ductility Testing by Gleeble 1500**

The addition of C reduced the DRT value by 30-40°C with the value of AC specimens being somewhat lower than that of the WQ specimens. The addition of B to the C containing alloy (991) reduced the value of DRT even further by 60-80°C. Again, the DRT value of the AC specimens was smaller than that of the WQ specimens by a small, but noticeable, amount. It was also observed that the addition of P to the alloy containing C and B (i.e. alloy 727) raised the DRT value of the AC specimens but that of the water-quenched remained almost unchanged. The influence of the addition of P to the C containing alloy (726) is shown in Fig. 5b. It is seen the addition of P had no influence on the DRT value of the air-cooled specimens but reduced it in the WQ specimens from 1180°C to 1100°C. The addition of B to this alloy (727) had, curiously, no influence.

**Fig 5 (a) D.R.T. vs. B, C and P Additions**

**Fig 5 (b) D.R.T. vs. B, C and P Additions**
Fig. 5c shows the effect of B on the DRT value. It is seen that B has the greatest influence reducing the DRT value of the base alloy from 1200°C to about 1090°C in both the air-cooled as well as WQ condition. The addition of C to the B containing alloy improved the DRT values to a small extent. However, as shown in Fig. 5d, when P was added to the B containing alloy the DRT values of the alloy reduced even further (alloy T91). The ternary addition of C to this alloy (727) increased the DRT values in both the WQ and air-cooled specimens, with improvement being greater in the air-cooled specimen (Fig. 5d).

These results suggest that the adverse effect of B on the DRT values and weldability is most severe. The addition of P to B alloys reduces the weldability even further. However, the addition of C to the B, as well as to the B and P containing alloy is beneficial, although when only C is present the weldability of B and P free alloy is reduced to a small extent.

Evaluation of Micro-fissuring in EB Welds by Total Crack Length Measurements

The weldability of various alloys was also determined by measuring the lengths of micro-fissures in cross-sections of EB welded samples in a SEM. The average of the sum of crack length measured in 10 cross-sections was designated as the average total crack length or TCL. The variation in the values of TCL by the additions of B, C and P is shown in Fig. 6 (a-b).

In general, the values of TCL followed the same trend that was exhibited by the DRT values. The lowest TCL values were seen in the base alloy (988) with a TCL of 24 μm. The addition of C (alloy 982) did not significantly affect the TCL in either the AC or WQ condition. In contrast, the addition of high B (989), did increase the TCL to 105 μm and 72 μm in the AC and WQ condition, respectively.

Adding B (991) to the base plus C alloy (982), increased the TCL values in both the AC and WQ conditions with the addition of P

(727) showing varying effect with the cooling rate, Fig. 6 (a). Fig. 6(b) show that in the sequence base, base+C, base+C+P, base +C+P+B, there is a continual increase in TCL with minor alloy addition. On the other hand, in the sequence, base, base+B, base+B+P, base+B+P+C, the detrimental effect of P is evident when C is low, Fig. 6, in both the AC and WQ conditions. With C present, Fig. 6, the detrimental effect of P is somewhat mitigated.

Discussion

To attempt to understand the effects of the additions of C, B, P, individually and in combinations, on weldability through their effect on the DRT and TCL parameters, it is necessary to briefly review current theories on the influence of secondary and tertiary addition on segregation of minor element to grain boundaries. Two main theories have been proposed, one by Guttmann [13] and another by Erhart and Grabke [14]. Guttmann [13] considered a ternary addition to a binary alloy, where the ternary
element was able to influence the solubility of the tramp element such as S and thus increase the grain boundary segregation. It should however be noted that Guttmann’s theory is more likely to be applicable to higher alloying additions (>1%), than used in the present study.

Site competition was however proposed by Erhart and Grabke [14], in a systematic investigation of P segregation in plain C and Cr alloyed steels. They showed that C for example could displace P from grain boundaries. That is, site competition rather than an intensification of the segregation of a minor element by a ternary addition is responsible for the increased concentration of minor elements to the grain boundary. The addition of Cr to a steel, resulted in the Cr reacting with C, which reduced the activity of C, allowing P segregation to occur on the grain boundary.

In analyzing the effect of minor elements on weldability another factor that needs to be considered is the potential effect of increased C additions on the solidification path. Thompson [5] has shown that increasing the C level can influence the solidification path via increased fractions of carbides, ensuring that the liquid film is retained to lower temperatures relative to a smaller amount of liquid. Similarly, DuPont et al [15], have shown that C additions of > 0.1 wt % can increase the initiation of the reaction, L -> y + Laves, and a decrease in the temperature interval of the primary L -> y reaction. Considerable improvements in solidification cracking behavior at higher C levels was observed during Varestraint testing, using the maximum crack length as the failure criterion.

Effect of the Chemical Composition

The discussion on the effect of minor elements on microstructure and HAZ micro-fissuring is divided in three groups: individual effect, interaction of two and of three elements.

Individual effect: The effect of C can be evaluated by comparing the behavior of the base alloy (988) and the higher C alloy (982). A carbon addition to the base alloy increased the volume fraction of carbides, though the overall increase in C level from 0.008 wt. % to 0.03 % had little effect on the TCL values. This is in agreement with Kelly’s observation [6] that C additions up to 0.1 wt % had no effect on the TCL values in cast Inconel 718. Thompson (5) on the other hand found that the TCL values increased by 24% in cast 718 on raising the C level from 0.02 wt % to 0.06 wt.%. In the present situation the increase in C level from 0.008 wt. % to 0.031 wt % has had little effect on the TCL values (Fig 5), though the DRT was reduced by up to 40°C. This is probably related to the amount of carbide particles present and the potential availability of liquated phase during the thermal cycle.

The effect of B on the weldability parameters from the present work is seen by comparing the base alloy (988) to the B alloy (989), (Fig. 5). The addition of B led to the precipitation of boride particles in the solution treated condition and to significant segregation of B to the grain boundaries, especially in the A.C. condition. This modification of the microstructure affected the ductility of the material. The high B alloy (989) showed increased micro-fissures after EB welding (Fig. 5) compared to alloy 982, especially in the A.C. specimen, and a low DRT in Gleeble testing (Fig.5 c & d ). The segregation of B on grain boundaries lowered the solidification temperature of the boundaries and led to the formation of micro-fissures due to cooling stresses after welding [8-10].

From a purely weldability point of view therefore, B is considered detrimental to the weldability of Inconel 718 as demonstrated through its influence on the hot ductility DRT parameter and the TCL values.

Interaction of two elements: 1. Carbon-Boron: This interaction can be evaluated by considering the DRT and TCL values of alloys 988, 989 and 991. Summarizing from the results section, the addition of B to the 0.03 wt. % alloy (982), resulted in a reduction in the value of DRT and a slight increase in the TCL values (i.e. reduction in weldability). The addition of C to the B alloy, i.e. alloy 991, showed a slight improvement in DRT and a moderate improvement in TCL values (i.e. improvement in weldability). The detrimental influence of the addition of B to the 0.03% C alloy on the weldability parameters is related to boron’s well known effect in reducing weldability, as explained by Huang et al [8-10] and outlined in the previous section, i.e., R exacerbates the effect of C additions in the range of from as low as possible to up to about 0.1 wt %, via a combination of equilibrium and non-equilibrium segregation.

In the case of the C addition to the B alloy, since the C addition is not large enough to effect the solidification path (> about 0.1 wt % needed according to DuPont et al, [15]), it is possible that the C can compete with B for grain boundary sites and thus mitigate to some extent the deleterious effect of B. In addition, with more carbides present, some B is incorporated into the carbides and consequently reduces the effectiveness of B on the grain boundaries in influencing weldability of the alloy. Borides were not observed when C was present in the alloys.

2. Carbon-Phosphorous: The interaction of C with P is given by alloys (982) and (726). The effect on the DRT value was variable depending on cooling rate, Fig 4(b), whereas the TCL value was adversely affected by the P addition, Fig 5. The microstructure of alloy 726 did not contain any new precipitates such as phosphide or Laves phase, neither was P detected on the grain boundaries using SIMS. It may be that P segregated to the boundaries as a monolayer, which was difficult to detect by SIMS. From Paju’s research, [16,17], C is evaluated as influencing the segregation of P in austenite by mutual displacement, with both elements

Fig 6(b) T.C.L. va. Alloy. for A.C. and W.Q.
segregating to grain boundaries. Similar results were obtained by Erhart and Grabke [14], where it was proposed that the activity of C was such that C can displace P from grain boundaries, even in a high P material. Consequently in an alloy containing a sufficient level of C as in alloy 726, the effect of P is likely to be mitigated by the 0.03 wt % C present.

3. Boron-Phosphorous: The effect of B and P on weldability is seen by comparing alloys (989) and (T91). The P addition to the high B alloy dramatically increased the TCL values and reduced the DRT values by 40-50°C, to the lowest values observed during the investigation.

In the air-cooled alloy containing B and P, (T91), the Nb/Mo/Cr boride formed, which was observed to be detrimental to micro-fissuring resistance of the alloy (Fig. 4d & 5). A study is to be expected, if non-equilibrium segregation was occurring, the SIMS image for B would be stronger in the AC than in the WQ condition, as was the case; however the SIMS analysis did not show any segregation of P. Again the P may be segregating by equilibrium segregation or the B may be influencing the grain boundary segregation. This has been also observed by Paju [16, 17] where B additions were found to reduce the segregation of P to grain boundaries. In the present set of results however the combination of P and B in a low C alloy was observed to be detrimental to weldability, showing the lowest DRT values and the highest TCL values for the entire experimental set.

Interaction of three elements

The effect of C, B and P, on micro-fissuring in HAZ, is observed in the B, C and P alloy (727). The most dramatic effect of the three element combination is seen in Fig 5 where the addition of 0.03 wt. % C in alloy 727 reduced the TCL values from the B+P alloy (T91) by a factor of about 80%, in both AC and WQ conditions. Similarly the DRT values improved by 50 to 110°C for the AC and WQ conditions (Fig. 4d).

The microstructure of alloy 727 showed only carbides, contrasting with alloy T91 where borides were observed. The location of the borides on the grain boundaries in alloy T91 considerably affected the weldability parameters. The C addition in alloy 727 however eliminated the borides and considerably improved weldability of the alloy.

Following Paju's observations [16, 17], one can assume that the 0.03 wt. % C addition was sufficient to reduce segregation of P on grain boundaries to a level where the precipitation of phosphides was prevented. It is likely, however, that some P segregation still exists in the alloy, but it's effect is severely mitigated by the C activity arising from the 0.03 wt. % addition. As mentioned previously, Horton et al have observed C, B, and P elemental segregation to the grain boundaries of modified alloy 718, using the Atom Probe. [3].

Effect of the Cooling Rate

Some general trends on the interrelation between the effect of the cooling rate and the alloy composition on the DRT and TCL values were noticed.

There was no effect of cooling rate in the base alloy (988). This is to be expected since the alloy did not contain a high concentration of any minor element, which may segregate or precipitate on grain boundaries. The high C alloy (982) had very similar weldability results to alloy 988, after AC or WQ considering inherent errors in the measurement process.

The B containing alloy (989) and the C and B alloy (991) had similar hot ductility results, though the slower cooling rate in air allowed B to segregate to grain boundaries, increasing the micro-fissuring as shown by the TCL values.

In the alloys containing P, (T91, 726 and 727), the general trend in the TCL values indicated an improvement in weldability in the AC rather than in the WQ condition. The DRT values also reflected this trend in two of the three alloys (726 and 727).

Conclusions

1. The addition of minor elements, either singly or in combination, can significantly influence the weldability of IN 718 alloy.

2. C additions of 0.03 wt. % to the base alloy (0.008 wt. % C) had little effect on the TCL, but adversely affected the DRT up to 40°C in the AC condition.

3. B additions to the base alloy had a significantly detrimental effect on the TCL and DRT values.

4. C additions to the B containing alloy somewhat mitigated the detrimental effects of B. This can be explained by site competition as postulated by Erhart and Grabke.

5. B+P additions gave the worst combination of TCL and DRT values through the formation of borides.

6. The addition of C to a P containing alloy reduced the adverse effect of P, which could also be attributed to the site competition.

7. Similarly in a C+P+B alloy, C mitigated the detrimental effects of B and P, with the elimination of borides, the B being incorporated into the Nb rich carbides.

8. Borides were not observed in the microstructure when C was present.

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