High-temperature crack-growth behaviour in Nimonic PE16 and Alloy 718

Nimonic PE16 and Alloy 718 are γ'-strengthened nickel-base alloys, and both are being considered for several applications in nuclear reactors, especially for liquid-metal fast-breeder reactors. Specific applications include upper core internals where elevated-temperature fatigue and creep resistance are required. Consideration of Nimonic PE16 is based primarily on its high resistance to irradiation-induced void swelling. On the other hand, consideration of Alloy 718 is based primarily on its high tensile and creep strengths. But recent investigations have shown that, in spite of its high resistance to creep, Alloy 718 has very low resistance to high-temperature crack growth. Knowledge of the crack-growth behaviour of Nimonic PE16 at high temperatures, however, is too limited for making any selection between the alloys for nuclear applications. The present research effort was therefore initiated. In particular, crack growth in Nimonic PE16 was studied at 650°C under cyclic, static, and combined loads, and the results are compared with those of Alloy 718.

Crack-growth data were analysed, mostly in terms of the linear elastic stress intensity parameter K. Since Nimonic PE16 is more ductile than Alloy 718, the usefulness of values of K obtained at high temperature may be questionable. Therefore the data were also analysed in terms of the elastic-plastic parameter J integral, and the results compared with the K correlations. This helps to establish which of the two parameters is better in correlating growth data for fatigue cracks in Nimonic PE16 at 650°C.

Fatigue-crack growth of Nimonic PE16 was studied earlier as a function of temperature up to 650°C, but the tests were carried out at relatively high frequencies where time-dependent effects are small. Crack-growth rates increased with increase in temperature, but the growth rates were not much different from those of Alloy 718. Shahinian has made a comparative study of crack growth in several alloys at a low frequency, but the study of Nimonic PE16 was limited to temperatures up to 593°C. More recently, Michel and Smith have presented growth data for fatigue cracks in Nimonic PE16 at high temperatures under cyclic load, but the decrease of its yield stress and increase of its tensile ductility with time-dependent effects become significant in Alloy 718 and therefore it is a convenient temperature at which to determine the relative performance of the two alloys.

Experimental detail

Nimonic PE16 was obtained from the vendor in the form of 15-2 mm plate. Compact tension specimens of 3/4 7 size with nominal dimensions (see Ref.6), but 12.7 mm thick, were machined with a TL orientation, i.e. with the notch parallel to the final rolling direction. The specimens were side grooved to 5% of the depth on each side and were precracked at room temperature. The compositions of Nimonic PE16 and Alloy 718 are given in Tables 1 and 2, respectively, and typical mechanical properties at 650°C and details of heat treatments for both alloys are given in Table 3. Extended aging times in the modified heat treatment of Alloy 718 cause overaging and contribute to the decrease of its yield stress and increase of its tensile ductility. In contrast, Nimonic PE16 has much lower yield and creep strengths, but its tensile ductility is comparable to that of Alloy 718 under the modified heat treatment. As will be discussed below, the high tensile ductility of Alloy 718 in

<table>
<thead>
<tr>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Si</th>
<th>Co</th>
<th>Mn</th>
<th>Cu</th>
<th>Zr</th>
<th>Others</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>426</td>
<td>18.7</td>
<td>3-16</td>
<td>1.3</td>
<td>1.16</td>
<td>0.22</td>
<td>0.12</td>
<td>0.08</td>
<td>0.08</td>
<td>0.04</td>
<td>0.035</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Table 1 Composition of Nimonic PE16, wt-%
1. Effect of environment on growth rate of fatigue cracks in Nimonic PE16 and Alloy 718

Table 2: Composition of Alloy 718, wt-%

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Cr</th>
<th>Nb</th>
<th>Ti</th>
<th>Al</th>
<th>C</th>
<th>Fe</th>
</tr>
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<tr>
<td>50-65</td>
<td>17-21</td>
<td>50-65</td>
<td>0.05-1.15</td>
<td>0.4-0.8</td>
<td>0.01-0.03</td>
<td></td>
<td></td>
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</tbody>
</table>

Table 3: Typical mechanical properties of Nimonic PE16 (Ref.7) and Alloy 718 (Ref.8) at 850°C, and heat treatments.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>0.2% yield strength, MPa</th>
<th>UTS, MPa</th>
<th>Total elongation, %</th>
<th>100% creep-rupture strength, MPa</th>
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</thead>
<tbody>
<tr>
<td>Nimonic PE16</td>
<td>510</td>
<td>718</td>
<td>22</td>
<td>348</td>
</tr>
<tr>
<td>Alloy 718 standard heat treatment</td>
<td>1004</td>
<td>1198</td>
<td>9.5</td>
<td>780</td>
</tr>
<tr>
<td>Alloy 718 modified heat treatment</td>
<td>946</td>
<td>1085</td>
<td>26.6</td>
<td></td>
</tr>
</tbody>
</table>

Modified heat treatment and of Nimonic PE16 has a significant effect on their time-dependent crack-growth behaviour.

Continuous cyclic tests using load control were conducted in a servo-hydraulic machine, using a triangular wave pattern at a frequency of 0.17 Hz, after the specimens
had been heated by induction. The ratio $R$ of minimum load to maximum load was maintained at 0.05 for all tests. For creep-fatigue tests, a hold time of 1 min was imposed at peak load. Displacement was measured at the notch end of the surface using a strain-gauge extensometer with quartz rods, and load-displacement loops were recorded at intervals.

Static-load tests were conducted using a creep frame. These specimens were heated by a resistance wire-wound furnace with a window. Tests in vacuum were carried out at pressures less than 130 µPa. Crack lengths for all tests were measured using a low-magnification travelling microscope.
Crack-growth rates were determined from the plots of crack length vs. time using a slope-measuring device.

The stress-intensity factor was calculated for $a/W < 0.7$ using the relation:

\[ K = \frac{P}{BW^{\frac{3}{2}}} \left[ 29.6(a/W)^{4.5} - 185.5(a/W)^{3.5} - 655.7(a/W)^{2.5} 
- 1017.0(a/W) + 638.9(a/W)^{-3} \right] \]  

(1)

and for $a/W > 0.7$ using the relation:

\[ K = \frac{P}{2B} \left( \frac{W-a}{(W-a)^{1.5}} \right) \left( 4.0 - \frac{(W-a)}{(W-a)} \right) \]  

(2)

where $P$ is the applied load, $a$ the crack length from the loading line, $W$ the specimen width, and $B$ the thickness.

The stress-intensity range $\Delta K$ was determined from

\[ \Delta K = K_{max} (1-R) \]  

(3)

When comparing the crack-growth rates between the cyclic and static loads, $R$ is assumed to be zero for static load tests.

Growth rates were correlated in terms of both linear and non-linear fracture-mechanics parameters.

### Results and discussion

The growth rate of fatigue cracks, $da/dN$, of Nimonic PE16 is represented as a function of $\Delta K$ in Fig.1, together with data for Alloy 718. In vacuum, crack-growth rates in the two alloys are nearly the same over the range of $\Delta K$ values. But in air, the growth rates differ significantly. The rates in
Crack growth in Nimonic PE16 and Alloy 718

Nimonic PE16 are lower than those in Alloy 718 by a factor of nearly ten. Thus the effect of environment is relatively small in Nimonic PE16, with growth rates increasing generally by a factor of two, whereas in Alloy 718 the increase is by a factor of more than ten.

Figure 2 shows the effect of hold time at peak load. At low load, 8 kN, crack-growth data for both continuous cycling and a 1 min hold fall on two different linear segments. In particular, for continuous cycling the first segment is shallower in slope than the second, but for a 1 min hold, the second segment is shallower than the first. The change in slopes in both cases could be related to crack size. For continuous cycling, the shallow segment, which is short, occurs at small crack lengths. For a 1 min hold, the shallower segment occurs at large crack lengths just before final fracture. If these two segments are excluded, assuming that they are the consequence of geometric effects, a continuous line can be drawn for the remaining data. In addition, the data for high load, both for continuous and hold-time cycling, fall on the same line. In short, the results show that a hold time of 1 min has very little effect on crack growth in Nimonic PE16.

In contrast to Nimonic PE16, Alloy 718 is significantly affected by hold time, as determined earlier. A 1 min hold at peak load at 650°C increases the growth rate by nearly two orders of magnitude, as can be seen in Fig. 3 (which represents the average of several tests). In fact, crack growth in Alloy 718 at 650°C at these low frequencies is essentially time dependent. On the other hand, in Nimonic PE16, the growth is essentially cycle dependent, as indicated in Fig. 2. The large increase in crack-growth rate in Alloy 718 is due primarily to an environmental effect, with oxygen causing the most damage.

The crack-growth behaviour in Nimonic PE16 under continuous and hold-time cycles is next examined on a time basis, $da/dt$, and compared with that under static load in Fig. 4. To compare crack growth under static load with that under cyclic load, the value of $R$ is assumed to be zero for static load. Crack growth under a static load forms a lower bound for the growth under cyclic load with and without hold time. Thus, imposing cyclic load on static load increases the growth rates. Actually there is no time-dependent contribution to the growth, even under a 1 min hold. Since crack growth under cyclic load occurs relatively
Crack growth in Nimonic PE16 and Alloy 718

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Fracture surfaces (SEM) under various conditions

rapidly, the times are too short for creep-crack growth to make any noticeable contribution. In contrast to this, it was shown earlier\(^a\) that crack-growth rates in Alloy 718, on a time basis, fall on a narrow band for continuous, 1 min, and 10 min hold cycles, and static load. In fact, crack growth under 10 min hold was slightly faster than under static or continuous-cyclic load, indicating that synergistic effects, called creep-fatigue interaction effects, are contributing to the acceleration of growth. Thus crack growth in Alloy 718 at 650°C is essentially time dependent, and in Nimonic PE16 it is essentially cycle dependent, at the frequencies and hold times tested. Because of the relatively high growth rates under cyclic load compared to that under static load, crack growth is expected to remain cycle dependent in Nimonic PE16, except for prolonged hold times, i.e. when the contribution of creep to the growth in each cycle becomes significant. Furthermore, creep contributes only when the stress intensity during hold time exceeds the threshold stress intensity for creep-crack growth.

NON-LINEAR FRACTURE-MECHANICS PARAMETER

In comparing the crack-growth rates in Nimonic PE16 and Alloy 718, linear elastic fracture mechanics is assumed to be applicable at 650°C under cyclic, static, and combined loads. Table 3 and Fig. 1 show that Nimonic PE16 is more ductile and less sensitive to environment than Alloy 718. Since plastic strains are likely to be of long range in Nimonic PE16 because of its low yield strength, linear elastic fracture mechanics may not be applicable. So it is important to examine the crack-growth-rate data in terms of a non-linear parameter, such as the \(J\) integral. This helps to resolve which of the two parameters, \(J\) or \(K\), is valid for the alloys at 650°C.

A procedure for determining \(J\) for load-controlled fatigue was developed previously.\(^b\) In particular, it involves measuring the area under the rising part of the load-displacement loop from the minimum to the maximum load, and calculating \(J\) from the Merkle and Cornet equation:

\[
J = 2(\alpha_4 - \alpha_0 \theta_0)Bh
\]

where \(A\) is the area, \(B\) the specimen thickness, \(h\) the length of uncracked ligament, \(P\) the peak applied load, and \(\delta_m\) the displacement along the loading line at the maximum load. The constants \(\alpha_4\) and \(\alpha_0\) are functions of \(W\) and are given in graphical form in Ref.13. It was shown previously\(^c\) that the \(J\) values obtained by the above estimation procedure

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\(a\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

Fracture surfaces (SEM) under various conditions

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\(b\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(c\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(d\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(e\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(f\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(g\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(h\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(i\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(j\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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\(k\) 1 min hold at low load, \(\Delta K = 24 \text{MNm}^{-1/2}\); 1 min hold at low load, \(\Delta K = 50 \text{MNm}^{-1/2}\); c 1 min hold at high load, \(\Delta K = 55 \text{MNm}^{-1/2}\); d and e static load

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are close to the values determined by a more accurate but lengthy data-reduction procedure.

Crack-growth rates in terms of $\Delta$ thus determined are represented in Fig.5 for both continuous and hold-time cycles. The $\Delta$ values are normalized with respect to Young's modulus for Nimonic PE16 at 650°C. By this normalization, values of $\Delta$ and $\Delta K$ can be compared on a one-to-one basis. The dashed lines in Fig.5 correspond to the $\Delta K$ values from Fig.2, while the data points correspond to $\Delta$ values. Clearly, the values of $\Delta$ and $\Delta K$ agree over the range of crack-growth rates. This means that the linear elastic parameter $\Delta K$ may well be adequate to correlate growth data for fatigue cracks at 650°C in Nimonic PE16, although further confirmation should come from tests with different specimen geometries. Importantly, the results imply that a comparison of Nimonic PE16 and Alloy 718 data on the basis of $\Delta K$ is not inappropriate. Note that the segments observed in Fig.2 persist, even in terms of $\Delta$. This may imply that their origin is not due to the limitation of the $\Delta K$ parameter in that range.

**CREEP-CRACK GROWTH**

The crack-growth behaviour of Nimonic PE16 under static load is next examined. The growth rates in air and vacuum are represented as functions of $K$ in Fig.6, along with those for Alloy 718. The data for Alloy 718 with a modified heat treatment are also shown for comparison. In Alloy 718 with standard heat treatment, crack growth in air at 650°C occurred readily, even at stress intensities as low as 17 MN m$^{-2}$. In contrast to this, stress intensities of 60 MN m$^{-2}$ and above are required to induce growth in Nimonic PE16. Thus, the threshold stress intensity for time-dependent crack growth appears to be significantly larger in Nimonic PE16 than in Alloy 718. As in Nimonic PE16, high threshold stress intensities were observed previously for Udimet 700. Thus, large differences in threshold stress intensities can exist among the alloys, even though they may all be similar in respect of being y'-strengthened. The most significant difference between Nimonic PE16 and Alloy 718, however, is in growth rates. The growth rates in Nimonic PE16 are nearly three orders of magnitude lower than in Alloy 718 with standard heat treatment.

The high crack-growth rates in Alloy 718 have been shown to be due predominantly to an environmental effect. The growth rates in vacuum are two orders of magnitude lower than in air. In contrast, the growth rates in vacuum of Nimonic PE16 are not different from those in air. Therefore, environment has a negligible effect on crack growth under static load in Nimonic PE16. Consequently, the time-dependent crack growth is due essentially to a creep process.

A recent study has shown that modifying the heat treatment for Alloy 718 could decrease significantly the growth rate in vacuum. The improvement in crack-growth resistance was accomplished primarily by eliminating the environmental contribution. The growth rate in air for Alloy 718 with the modified heat treatment is nearly the same as that in vacuum for the standard heat treatment. However, it is important to note that the growth rates, even with the modified heat treatment, are higher than in Nimonic PE16, as shown in Fig.6.

These differences in the time-dependent crack-growth behaviour of the alloys contribute to the differences under hold-time fatigue. In Alloy 718, time-dependent behaviour dominates to the extent that crack growth at low frequencies or hold times is essentially time dependent. In Nimonic PE16, the time-dependent crack growth is negligible to the extent that crack growth at the same frequencies and hold times is essentially cycle dependent.

Thus, the present results indicate that there is a pronounced difference in crack-growth behaviour between Nimonic PE16 and Alloy 718 at 650°C, Nimonic PE16 possessing the higher resistance to time-dependent crack growth. On the other hand, previous results indicated very little difference in their fatigue-crack growth behaviour. But, as mentioned above, these results pertain to high frequencies where time-dependent effects are minimal. Therefore, the present analysis provides further indication that, in order to evaluate fully the crack-growth behaviour of high-temperature structural materials, tests need to be carried out at low frequencies or hold times that are representative of the creep-fatigue conditions found frequently in service.

**FRACTURE FEATURES**

Fracture surfaces of Nimonic PE16 were next examined under SEM to investigate the mechanisms of crack growth. Figure 7 shows the surface features of the specimens tested under continuous cycling in air and vacuum (Fig.1); Fig.7a and b show specimens in air, and Fig.7c-e show specimens in vacuum. Note that crack growth is predominantly transgranular in Fig.7a, which is at a low stress intensity. The fraction of intergranular failure increases with increasing $\Delta K$.

In contrast to this, crack growth in vacuum is predominantly transgranular at all stress intensities. In the precrack region (see Fig.7c), crack growth appears to be of a crystallographic faceted mode with surfaces covered by high-density cleavage steps, with different orientations in each grain. In the high-temperature crack-growth region, the crack follows the normal striation mode, as shown in Fig.7d, where the stress intensity corresponds to 40 MN m$^{-2}$. The crack-growth rate computed from the striation spacing in the figure is $1.5 \times 10^{-8}$ mm/cycle, which agrees reasonably well with the data in Fig.1. At still higher stress intensities, $64 \text{MN m}^{-2}$, occasional intergranular damage can be noticed (see Fig.7e).

Therefore, the indications are that the predominant intergranular crack growth in air is due mostly to environmental effects. In fact, fracture is completely intergranular in Alloy 718 at 650°C, under all the conditions imposed in the tests, because of the environmental effect.

Fracture surfaces of Nimonic PE16 specimens subjected to hold-time fatigue and creep are shown in Fig.8. Figure 8a and b are from a specimen tested at a 1 min hold at low load, while Fig.8c shows a specimen tested at high load. With increase in stress intensity from $24 \text{MN m}^{-2}$ (Fig.8a) to $60 \text{MN m}^{-2}$ (Fig.8b), there is an increase in the fraction of intergranular crack growth. Figure 8c corresponds to a region close to the precrack. Transitions from a purely faceted mode in the precrack region to predominant intergranular crack growth are indicated by arrows in Fig.8c. Thus, there appears to be no significant difference in fracture mode between continuous and hold-time cycling.

Finally, Fig.8d and e show the fracture surfaces of a creep specimen. Crack growth is essentially intergranular. The surface, however, was rougher under creep than under fatigue. Some amount of tearing is occasionally noted on the fracture surfaces. The crack growth was also intergranular in vacuum.

**Summary and conclusions**

The crack-growth behaviour of Nimonic PE16 has been
studied at 650°C under cyclic, static, and combined loads, and the growth rates are compared with those of Alloy 718. Nimonic PE16 has lower strength and higher ductility than Alloy 718 at 650°C.

The results show that the crack-growth rates under continuous cycling are the same in both alloys in vacuum, but that the rates in air differ significantly. Crack-growth rates are lower by a factor of ten in Nimonic PE16. The difference between the two alloys is due to the effect of environment. The increase in crack-growth rate being only a factor of two for Nimonic PE16 but more than a factor of ten for Alloy 718.

A hold of 1 min has no effect on crack growth in Nimonic PE16, but increases growth rate by a factor of 100 in Alloy 718. The crack growth is essentially cycle dependent in Nimonic PE16, whereas it is mostly time dependent in Alloy 718 at 650°C.

Crack-growth data are analysed in terms of both the linear elastic parameter $K$ and the non-linear elastic-plastic parameter $J$ integral. The growth rates correlate similarly for $K$ and $J$. This further indicates that $K$ can adequately describe the growth rates under high-temperature fatigue in Nimonic PE16.

The most significant differences in crack-growth rates in the two alloys were observed under static load. In Nimonic PE16, very high stress intensities are required for crack growth. Furthermore, the growth rates are nearly three orders of magnitude lower than in Alloy 718. Again, the difference is due mostly to the environmental effect on crack growth. In Alloy 718, the growth rates in air and vacuum differ by a factor of 100, while in Nimonic PE16 the growth rates are equal. Thus, time-dependent crack growth in Nimonic PE16 is due purely to creep. The most significant differences in crack-growth rates in the two alloys were observed under static load. In Nimonic PE16, very high stress intensities are required for crack growth. The results thus indicate that Nimonic PE16 performs better than Alloy 718 at 650°C under low-frequency or hold-time fatigue, as well as under static load. Even at high frequencies Nimonic PE16 appears to be slightly better than Alloy 718.

Acknowledgments

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References