Microstructural aspects of fatigue in Ni-base superalloys

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Nickel-base superalloys are primarily used as components in jet engines and land-based turbines. While compositionally complex, they are microstructurally simple, consisting of small (50–1000 nm diameter), ordered, coherent Ni$_3$(Al,Ti)-type L1$_2$ or Ni$_3$Nb-type DO$_{22}$ precipitates (called γ$'$ and γ$''$, respectively) embedded in an FCC substitutional solid solution consisting primarily of Ni and other elements which confer desired properties depending upon the application. The grain size may vary from as small as 2 µm for powder metallurgy alloys used in discs to single crystals the actual size of the component for turbine blades. The fatigue behaviour depends upon the microstructure, deformation mode, environment and cycle time. In many cases, it can be controlled or modified through small changes in composition which may dramatically change the mechanism of damage accumulation and the fatigue life. In this paper, the fundamental microstructural, compositional, environmental and deformation mode factors which affect fatigue behaviour are critically reviewed. Connections are made across a range of studies to provide more insight. Modern approaches are pointed out in which the wealth of available microstructural, deformation and damage information is used for computerized life prediction. The paper ends with a discussion of the very important and highly practical subject of thermo-mechanical fatigue (TMF). It is shown that physics-based modelling leads to significantly improved life prediction. Suggestions are made for moving forward on the critical subject of TMF life prediction in notched components.

1. Introduction: overview of superalloy microstructure

Superalloys are widely used for rotating components in jet engines. One of the main reasons for these applications
is that they possess excellent high-temperature strength, excellent resistance to oxidation and
good creep resistance. These materials provide one of the most striking examples of the way
in which properties can be controlled through both compositional adjustment and processing
to obtain desired levels of those properties which are critical for a given application. Some
representative compositions are given in table 1.

In broad overview, Al and Ni are the basis of Ni-base superalloys. Other elements are added for
specific reasons. Ti is added to increase the stacking fault energy (SFE) \( \gamma_0 \) of the \( \gamma' \) precipitate and
to increase the \( \gamma' \) lattice parameter. Chromium is added to provide additional oxidation resistance
and other elements are added to control the mismatch between the precipitate and matrix (e.g.
Mo), to slow down solid-state diffusion and increase the stability (e.g. Re), to provide solid
solution strengthening of the matrix (W, Ta, Mo, Cr), and to provide resistance to intergranular
oxidation (e.g. B). More detailed information is provided elsewhere [2].

While somewhat complex compositionally, Ni-base alloys are microstructurally simple
consisting of:

- an ordered, coherent \( \gamma' \) precipitate based on Ni\(_3\)Al;
- an FCC solid solution primarily Ni but with other substitutional elements;
- carbides of the type MC, M\(_6\)C or M\(_{23}\)C\(_6\) depending on the temperature and composition;
- the carbides are usually found on boundaries or in the interdendritic regions but
  sometimes in the grain interiors; and
- other minor phases such as borides.

While the main topic of this paper is directed towards Ni-base alloys, some discussion will be
devoted to Fe–Ni–Al-base alloys which find use in the nuclear power industry in addition to
their more common use in jet engines. These alloys are also microstructurally simple consisting
of ordered body-centred tetragonal (BCT) Ni\(_3\)Nb \( \gamma'' \) precipitates (DO\(_{22}\)) which appear as needles
or plates. In some situations, \( \gamma' \) may also be present, usually as a result of over-ageing.

Both the precipitates and the grain size play important roles in determining the mechanical
properties including the fatigue properties. Depending upon the application, desired properties
and processing, typical grain sizes for given applications are shown in table 2.

The grain, dendrite and carbide structures are shown in figure 1 for these processes.
As can be seen, the grain size alone varies over three orders of magnitude.

Superalloys are heat-treated to produce a desired distribution of precipitates. The usual
heat treatment results in large \( \gamma' \) precipitates (of the order of 0.5–1 \( \mu \)m), smaller or so-called
strengthening \( \gamma' \) precipitates of the order of 0.1 \( \mu \)m diameter and an extremely fine distribution
of cooling \( \gamma' \) which is about 5 nm. A typical \( \gamma' \) precipitate microstructure is shown in figure 2.

The \( \gamma' \) precipitates have very unusual properties in that the yield strength increases with
increasing temperature over a significant range of temperature and these precipitates are largely
responsible for the excellent high-temperature strength of these alloys. Their mechanism of
hardening with increasing temperature will be discussed in the body of this paper.

Very detailed information on the physical and mechanical metallurgy of Ni-base superalloys
may be found in the quadrennial series of conference proceedings on superalloys (e.g. [7]).
A dated, but nevertheless important, source of information on superalloys is also available [8],
as is a more up-to-date primer on superalloys [2].

In the following sections, fundamentals of deformation are briefly reviewed since the
deformation behaviour is fundamental to any treatment of fatigue. The effects of deformation
mode on fatigue are first discussed for fatigue at room temperature where effects of environment
can be ignored. Next, the effects of temperature are considered and it is at high temperatures
where the effects of environment, deformation mode, creep and microstructural stability as well
as their complex interactions become important. The paper ends with a discussion of the more
realistic yet extremely complex case of thermo-mechanical fatigue (TMF),\(^1\) in which interactions
that never occur in isothermal fatigue act in concert to determine the overall life.

\(^1\)TMF refers to the variation in temperature and mechanical strain, which are not necessarily in phase with each other and
which, in extreme cases, may be independently applied.
Table 1. Compositions of typical Ni-base superalloys [1].

<table>
<thead>
<tr>
<th>Product Form</th>
<th>Element/(wt.%)</th>
<th>Ni</th>
<th>Fe</th>
<th>Co</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Ta</th>
<th>Nb</th>
<th>Al</th>
<th>Ti</th>
<th>V</th>
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<td>15</td>
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</table>
Table 2. Grain size variation of Ni-base superalloys in jet engine components.

<table>
<thead>
<tr>
<th>application</th>
<th>processing</th>
<th>typical grain size and structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>jet engine disc</td>
<td>powder metallurgy (PM)</td>
<td>3–5 µm/equiaxed</td>
</tr>
<tr>
<td>high-pressure turbine blade</td>
<td>conventional casting (CC)</td>
<td>60–200 µm diameter/equiaxed</td>
</tr>
<tr>
<td>high-pressure turbine blade</td>
<td>directional solidification (DS)</td>
<td>20–300 µm dendrite diameter × 3–10 cm long. Grains of the order of millimetres. Aligned in the [001] direction.</td>
</tr>
<tr>
<td>high-pressure turbine blade</td>
<td>directional solidification with grain selector helix to produce a single crystal</td>
<td>single crystal the size of component. Primary dendrite spacing similar to DS alloys</td>
</tr>
</tbody>
</table>

2. Monotonic deformation of Ni-base superalloys

At the most fundamental level, the deformation behaviour is controlled by the interaction between dislocations and precipitates. As previously mentioned, the precipitates are coherent with the lattice and the orientation relationships are \( \{100\}_p\parallel\{100\}_m \) and \( \langle 100 \rangle_p\parallel\langle 100 \rangle_m \). The difference in the lattice parameter plays a major role in determining both the initial morphology of the precipitate and its stability under stress at high temperatures. It is defined as the mismatch parameter or \( \delta \) parameter and is given by

\[
\delta = 2 \left( \frac{a_p - a_m}{a_p + a_m} \right),
\]

where the \( a \)'s represent the lattice parameters of the precipitate and matrix.
Figure 2. Dark-field TEM micrograph showing the $\gamma'$ precipitate structure of René 77 [4].

Figure 3. Micrograph showing splitting of $a/2\langle110\rangle$ dislocation pairs upon emerging from the $\gamma'$ precipitate in [001] oriented PWA 1480. Tested at 705°C at a strain rate of $8.3 \times 10^{-5}$ s$^{-1}$. Diffraction vector $g = \langle200\rangle$. Note especially the splitting behaviour of a pair of dislocations with segments in both the $\gamma'$ and $\gamma$ (white arrow) [9].

Deformation may take place either by shearing or by looping depending on the volume fraction, mismatch (equation (2.1)) and spacing of precipitates. If deformation takes place by shearing, a common mode of deformation at low temperatures for stable precipitates, then the dislocations travel through the ordered L1$_2\gamma'$ precipitate in pairs, the first creating anti-phase boundary energy (APBE) and the second removing the APBE and restoring the original order. A striking example of close dislocation association in $\gamma'$ and subsequent separation in the matrix $\gamma$ is shown in figure 3 for a slip band in PWA 1480 [9].

A very simple theory for the hardening of Ni-base superalloys was developed by Copley & Kear [10] in which it was assumed that $\delta$ was small, so as to give spherical precipitates of radius $R$, and the volume fraction $V_f$ was high so as to prevent Orowan looping. These assumptions are frequently true for superalloys such as Waspaloy (discussed later) and others. Based on TEM evidence, they assumed that the critical step in yielding was to drive the dislocation into the ordered precipitate to create APBE. The result was

$$\tau_{ys} = \frac{\gamma_0}{2b} - \frac{\Gamma}{bR} + \frac{k}{2}(\tau_m + \tau_p),$$  

(2.2)
where $\gamma_0$ is the APBE, $b$ is the Burgers vector, $\Gamma$ is the dislocation line tension, $k$ is a constant and the $\tau$’s on the right-hand side of the equation represent the resistances to deformation provided by the $\gamma$ matrix and $\gamma'$ precipitate, respectively. The first term represents the strengthening due to the stacking fault; the second term represents the force of curvature tending to pull the dislocation through the precipitate; and the third term represents the average resistance to deformation from the matrix and particle. These authors determined that over 80% of the strength of MarM 200, a Ni-base superalloy which closely follows the assumptions made in developing the theory, is attributable to the APBE.

Other, more detailed, theories for the strength of superalloys have been developed. For example, Brown & Ham [11] developed a theory, related to that proposed by Gleiter & Hornbogen [12,13], for relatively small volume fractions of widely spaced precipitates in which Fleicher–Friedel statistics were valid. Shearing was still assumed to take place and a force balance type of calculation resulted in the following expression for the strength increment due to shearing:

$$\Delta \tau = \frac{\gamma_0}{2b} \left[ \frac{4\gamma_0 RV_f}{\pi \Gamma} \right]^{1/2} - V_f,$$

where $V_f$ is the volume fraction and all other symbols are as previously defined.

This equation was developed only for small volume fractions ($V_f < 0.2$) of $\gamma'$ so it really is not quite applicable to the usual high volume fractions found in modern alloys. However, it does show the effect of volume fraction and in terms of its qualitative predications is largely in agreement with the simpler theory of Copley and Kear. Other authors have considered the effect of strong and weak coupling of the dislocations [14–17]. As shown in figure 3, the two ⟨110⟩-type superpartials appear to be strongly coupled in the precipitate, which influences the effect of particle size on the strength. However, these details, while important for a complete understanding, do not significantly affect the topics considered in this paper. A more complete review of deformation in Ni-base superalloys is available elsewhere [18]. Above some critical precipitate radius, the particles will be sufficiently widely spaced so that deformation can take place by the familiar Orowan looping and the strength will decrease for increasing precipitate radius (i.e. wider precipitate spacing). Examples of deformation by precipitate shearing and by Orowan looping are shown in figures 4 and 5 for Waspaloy under different conditions.

The character and effects of deformation mode during fatigue will be addressed in those sections dealing explicitly with fatigue.
3. Deformation of $\gamma'$

Any understanding of the deformation behaviour of Ni-base superalloys, including cyclic deformation, requires knowledge of the unusual deformation mechanisms of the $\gamma'$ precipitate, which confers so many unique properties on this class of materials. Basically, the yield stress of this material increases rapidly with temperature to about 800°C and then decreases with a change in mechanism. Certain aspects of this behaviour along with a very brief explanation of that behaviour are briefly discussed below:

— Increasing strength with increasing temperature. Explained initially by Takeuchi & Kuramoto [20] as being due to cross slip of extended screw dislocations onto $\{100\}$-type planes which have a high Peierls force but a low SFE. In essence, the cross-slipped segments act as locks making continued $\{111\}\langle110\rangle$-type deformation much more difficult. The higher the resolved stress on the cube slip ($\{100\}\langle110\rangle$) system compared with that on the $\{111\}\langle110\rangle$ system, the greater the amount of cross slip and the stronger the $\gamma'$. Also, since slip is thermally activated, the higher the temperature, the higher the strength since the concentration of such locks will increase. Webb et al. [21] applied thermodynamic concepts, critically accounting for the configurational entropy of these segments as a function of their concentration. These researchers demonstrated that the strength increase of $\gamma'$ followed an Arrhenius relationship with temperature,

$$\tau_{ys} = \tau_0 \exp \left(-\frac{\Delta H_n}{kT}\right), \quad (3.1)$$

where $\Delta H_n$ is the enthalpy of formation of a cross-slipped pinning segment of length $nb$, $\tau_0$ is a constant and the other symbols have their usual meaning. Equation (3.1) lends itself to straightforward evaluation of the parameters $\tau_0$ and $\Delta H_n$.

— Tension and compression asymmetry, which has been attributed as arising from the different constriction length for the Shockley partials of the leading super-dislocation in tension and in compression [22]. This is fully compatible with equation (3.1), as discussed in [21].

— While the yield stress is strongly temperature dependent, the effect of strain rate is very small for strain rates typically used to determine properties. This effect has been explained on the basis that the cross-slipped segments are small (a few Burgers vectors in size), which allowed for detailed calculations of the rate of production of cross-slipped segments [23]. It was found that, for any reasonable size of cross-slipped segments, the concentration came to equilibrium virtually instantaneously. Thus, the test time (i.e. strain rate) is
not a factor in the range of strain rates normally obtainable. Therefore, the seeming contradiction of a strong temperature effect and virtually no strain rate effect is explained.

— The yield stress is recoverable when the temperature is changed [24]. That means that a small pre-strain at an elevated temperature would not affect the yield stress at a lower temperature. It was shown that the cross-slipped segments were thermally reversible. In essence, a material with a kind of reverse memory effect.

Clearly, the nature of the precipitates has a major effect on the deformation mechanisms and strength properties of Ni-base superalloys and in fact is responsible for the high-temperature strength of these alloys.

4. Cyclic deformation of Ni-base superalloys

As will be discussed in subsequent sections, the mode of deformation is intrinsically important in determining damage mechanisms and the fatigue life and also in influencing the severity of environmental damage and interactions. In this section, some of the most important aspects of cyclic deformation are reviewed. A recent general review on this and other topics [25] as well as a detailed review of cyclic deformation in Ni-base superalloys [18] is available.

(a) Cyclic deformation at ambient temperature

From a simplistic perspective, one can consider alloys with low volume fractions of precipitates and with low mismatch parameters. One such alloy is Waspaloy, an alloy that has been widely used for discs in jet engines. When heat-treated to contain very small particles, the particles are necessarily close together and deformation takes place by shearing. During cyclic deformation in strain control, the dislocation density will initially increase and eventually shear the precipitates. This regime of ‘accommodation’ shows increasing stress to the point of shearing. After shearing, the $\gamma'$ particles are less effective obstacles and the stress decreases. A detailed study was performed to elucidate the effects of precipitate size on the cyclic behaviour and deformation substructures [26]. Cumulative glide behaviour is shown in figures 6 and 7 along with the deformation substructures. Figure 8, taken from a different study [27] but for the same material, is a very clear illustration of reverse precipitate shearing by dislocations during cyclic deformation.

The dislocation structure of the slip bands formed during the cyclic deformation of Ni-base superalloys is seen to be very different from the persistent slip bands (PSBs) so extensively studied for Cu. Classical PSBs have been described [28–30] and reviewed [25,31,32] in detail elsewhere.
Suffice it to say for the current discussion, in classical PSBs, the cyclic strain is carried by screw dislocations in channels made up of edge dislocation dipoles and other dislocation debris. It is the author’s opinion that many studies reporting dislocation substructures make unambiguous interpretation very difficult owing to the projection of a three-dimensional structure onto two dimensions. Important insights can be obtained by applying the technique of stereo TEM as reported elsewhere to obtain precise three-dimensional information about the nature of fatigue substructures in Ni-base alloys [33]. This technique, used in a study of the low cycle fatigue (LCF) of the Ni-base alloy René 95, resulted in a completely new interpretation from the traditional two-dimensional micrograph. This stereo image is shown in figure 9 and, using a stereo viewer,[2] it can be seen that what appears to be random dislocation tangles are actually

[2] In fact, a stereo viewer is not necessary provided that the left eye focuses on the left image and the right eye on the right image. The eyes can be trained to do this by placing two holes in the pictures centred precisely on the same feature and then viewing a distant object through these holes. Since the rays coming from the distant object are virtually parallel, the left and right eyes are now in position for the brain to construct the three-dimensional image, exactly the same process that is forced in a stereo viewer. One advantage to this technique is that, by changing the distance from the eye, the degree of magnification can be controlled. The closer the image, the greater the apparent magnification.

Figure 7. (a) Cumulative glide curves for Waspaloy containing 90 nm diameter γ′ particles strained at 25°C at different plastic strain amplitudes and (b) dislocations looping precipitate particles after 40 cycles at 0.50% plastic strain amplitude [26].

Figure 8. Dark-field TEM image of Waspaloy fatigued at Δε_p = 4 × 10^{-3} at 25°C. The spherical precipitates have been sheared by the to-and-fro motion of the dislocations. Note the reversed deformations on different planes [27].
three overlapping, very distinct, slip bands. In fact, similar to the findings for Waspaloy discussed earlier, the bands are very narrow and there is strong evidence for particle shearing in the paired dislocations.

Precipitate shearing and slip band formation have further implications on the deformation behaviour. It was shown, not unexpectedly, that in Nimonic 80A [34], heat-treated to contain shearable precipitates, the slip bands acted as effective barriers and there was reasonable correlation with a Hall–Petch kind of relationship, as shown in figure 10. The change in the slope of the curve for the Nimonic 80A was associated with the onset of multiple slip bands. The deformation substructure via optical microscopy and SEM is shown in figure 11. Also, the form of the stress versus cycle number was exactly as seen in Waspaloy [26] for all strain levels and precipitate sizes, indicative of precipitate shearing.

**Figure 9.** Stereo pair of dislocations in fatigued René 95 [33]. Each individual micrograph gives the appearance of wavy dislocation glide or wide slip bands. However, the stereo pair clearly shows that there are four slip bands, three of which are overlapping and all of which are very narrow, perhaps about one atomic plane. Obviously, such three-dimensional information provides detailed information from which physically accurate models can be developed.

**Figure 10.** Effect of slip band spacing on the maximum stress during cyclic straining of Nimonic 80A [34] and Waspaloy [19] of various precipitate diameters at 25°C.
Figure 11. Optical micrographs of etched specimens revealed a decrease in the slip band spacing with increasing plastic strain range $\Delta \varepsilon_p$. (a) $\Delta \varepsilon_p = 0.05\%$, (b) 0.24\%, (c) 2.90\% and (d) 2.90\%. (d) is an SEM enlargement of (c) [34].

Figure 12. Cyclic stress–strain curve for specimens in which the $\gamma'$ precipitates were 13 nm in diameter and the grain diameter was approximately 60 $\mu$m. The stresses reported were the maximum stresses observed during fatigue loading [34].

Since the slip band density is related to the plastic strain, the graph of maximum stress versus plastic strain should also show a change in the slope at the strain level at which multiple slip bands are formed. Such behaviour is shown in figure 12.

It is also interesting that Lerch & Gerold [35] observed what appears to be a classical PSB at a surface grain. To the best of this author’s knowledge, it is the only time such a deformation substructure was observed in a Ni-base alloy. At this point, the reasons for this structure are not clear. However, looking at the contrast and recalling that high dislocation contrast is expected when the diffracting vector $g$ is parallel to the Burgers vector $b$, the dislocations between the ladder rungs in figure 13 do indeed appear to be screw dislocations, very much in agreement with the classical PSBs.
Figure 13. PSB-type fatigue deformation structure as seen via TEM with $\mathbf{g} = [111]$ [35]. Taken from a near-surface grain with $\Delta \varepsilon_p = 0.10\%$. Same structure as noted in figure 12.

Figure 14. Bright-field (a) and dark-field (b) TEM micrographs. In (a), $\mathbf{g} = [111]$ and planes normal to $\mathbf{g}$ are viewed edge on and are visible due to residual contrast. In (b), the dark-field image was formed using a carbide reflection and shows dynamic precipitation of carbides on slip bands. The relative rotation between (a) and (b) is due to a difference in magnification in the bright- and dark-field images [19,36].

(b) Cyclic deformation at high temperatures

Most of the features mentioned for ambient temperature also occur at high temperature. However, important new features are introduced when cycling at high temperature and these features bear importantly on damage accumulation and cyclic life. Some of the key features which appear at elevated temperature are briefly discussed in this section.

(i) Formation of new phases

In a study of Waspaloy, cyclically deformed over a range of temperatures, one important observation was a kind of dynamic strain ageing in which carbides precipitated upon newly formed slip bands as well as on existing grain boundaries (figure 14) [19,36]. An additional driving force for the formation of carbides is the reduction in strain energy associated with dislocations. Presumably, edge dislocations are favoured sites since they exhibit a higher net dilatation than
Figure 15. Dark-field TEM micrograph showing deformation-induced twinning. It was also shown that carbide particles were precipitated on the twins [36].

Figure 16. Example of the effect of stress on $\gamma/\gamma'$ morphology in Udimet 700 [37]. The sample was stress annealed at 976°C for 100 h at 157.5 MPa tension applied in the [001] direction (vertical direction).

screws. In addition to slip band formation and carbide precipitation, fatigue-induced twinning has also been observed (figure 15) in Nimonic 90. It was shown by dark-field TEM that carbides also precipitated at the twin boundaries.

(ii) Dynamic coarsening of $\gamma'$ precipitates

Tien and Copley [37,38] analysed the effects of applied stress, mismatch and elastic constants on the equilibrium shape of $\gamma'$ precipitates. Further contributions were made by Pineau [39] and Nabarro [40]. In broad outline, these researchers reasoned that the coherency strains between the $\gamma$ matrix and $\gamma'$ precipitates contribute to the internal energy, which can be minimized for certain morphologies depending upon the details. They showed that, for negative mismatches (equation (2.2)) and tensile loading in the [001] direction, plates normal to the stress axis would form upon stress annealing at high temperatures, as shown in figure 16. It is customary to refer to these plates as ‘rafts’ and the phenomenon of stress-induced morphological change as ‘rafting’.

It has been shown that for creep-dominated situations, for certain orientations of single crystals, depending on whether the rafts are parallel or perpendicular to the stress axis, the life
Figure 17. Dark-field image of René 77 showing coarsening of the $\gamma'$ precipitates during LCF at $927^\circ$C and $\Delta \varepsilon_p = 0.24\%$ [4]. Compare this structure with that seen in figure 2, the as-heat-treated precipitate microstructure.

may be improved significantly [41,42]. These authors found that, in their particular experimental alloys, rafting occurred normal to the load axis (N-rafts) at about 60% $\gamma'$ and parallel to the load axis (P-rafts) at about 80% $\gamma'$ and that the creep properties improved with increasing $\gamma'$. However, their conclusion that improvements were associated with narrow $\gamma$ channels was not supported by a model and does not take into account the effect of the morphology (N-rafts or P-rafts). Furthermore, as the volume fraction of $\gamma'$ increases, the mismatch changes, which determines the morphology. In fact, it is known (see [43]) that the raft morphology at a given volume fraction of $\gamma'$ has a major influence on the creep life and that negative mismatch alloys producing N-rafts have the best creep properties. In thermo-mechanical fatigue (discussed in §7), it is shown that high temperature holds, even at low strains, can produce a rafted structure which undoubtedly influences the life as discussed below.

During cyclic loading, it was shown that coarsening occurred very rapidly without rafting. Instead, the small precipitates were rapidly consumed by the larger ones, which had a somewhat irregular form. The irregular form undoubtedly is due to both tension and compression being applied with insufficient time in a cycle to attain the equilibrium morphology. Examples are shown in figure 17 for René 77 [4] and in figure 18 for René 80 [44], both second-generation polycrystalline alloys that were used for turbine blades and continue to be used. A structure similar to that shown in figure 17 was observed after only 50 cycles under the conditions given in figure 17.

As would be expected, for strain-controlled LCF testing, a stress-coarsened precipitate structure would be expected to show a decreasing stress range as coarsening continues and in fact this was indeed observed. Figure 19 shows a continuous decrease in the stress range during continuous cycling. The effects discussed so far are intrinsic for high-temperature deformation. In the next section, the major extrinsic effect, oxidation, is briefly discussed.

(iii) Oxidation of Ni-base superalloys at high temperature

Not only are there phase changes and precipitate coarsening, there is also a very strong environmental effect that is critical to consider. In general, oxides form at various boundaries such as grain boundaries, twin boundaries and slip bands. Oxidation of all of these features is shown in figure 20 for René 77. For many decades, the effects of oxidation appear to not have been fully appreciated, with high-temperature damage in these alloys being assumed ipso facto to be creep/fatigue. Without minimizing the effects of creep during high-temperature fatigue, it should be pointed out that both oxidation and creep rates obey equations of the same general form and
Figure 18. Precipitate/dislocation microstructure of René 80 continuously cycled at 871°C at 0.50%/min and $\Delta \varepsilon_p = 0.25\%$ [44]. The specimen was removed after the load had dropped by 75% (732 cycles). $\mathbf{g} = \{111\}$. Note the extensive $\gamma'$ coarsening, the interfacial dislocations (verified by stereo analysis) and the relatively few matrix dislocations.

Figure 19. Continuous stress decrease for René 80 during continuous cycling at 0.50%/min at 871°C [44].

from an analytical perspective it would be easy to carry out the analysis as if creep were the main damage mode. In the next section on damage and fatigue life, it will be shown that in some cases creep may actually reduce the severity of environmental degradation. In any case, environmental damage is certainly a very important component in assessing the fatigue performance of these materials.
Figure 20. SEM micrograph showing surface oxidation of boundaries and slip bands in René 77 at 714 °C and $\Delta e_p = 0.30\%$. The specimen was stopped after 50 cycles and the expected life $N_f$ was approximately 250 cycles [4].

Figure 21. Slip band and twin cracking (arrows) in Waspaloy fatigued at 25°C [18]. (Online version in colour.)

5. Fatigue of Ni-base superalloys at low temperature

In this section, fatigue deformation will be considered at temperatures where damage accumulation is dominated by cyclic deformation for both LCF and fatigue crack propagation. This temperature could be several hundreds of degrees Celsius since the precipitate structure is very stable and creep and environmental effects may not become dominant until still higher temperatures.

(a) Effects of $\gamma'$ and grain size

The grain size and $\gamma'$ diameter are the two microstructural parameters most amenable to control and which have the largest effect on mechanical properties, as already discussed in the section on deformation. One might infer from the cyclic stress–strain curves for Waspaloy (figures 6 and 7) that the behaviour in LCF and in fatigue crack propagation (FCP) is different. For example, the well-defined slip bands associated with precipitate shearing would be expected to give rise to early crack initiation, as shown in figure 21.

However, although this kind of deformation would lead to early crack initiation, it would tend to be associated with a lower crack propagation rate because of the reduced stress under strain.
Figure 22. Effect of slip reversibility \((1 - \phi^2)\) on the fatigue threshold stress intensity \(\Delta K_o\) for the Ni-base superalloy M4K [46].

Cycling as shown in figure 6. This notion fits in very well with the Tomkins model for cracking during LCF [45]

\[
\frac{da}{dN} = \frac{\pi^2}{8} \left( \frac{\Delta \sigma}{2\bar{T}} \right)^2 \frac{\Delta \varepsilon_f}{(2n + 1)^a},
\]

(5.1)

where \(a\) is the crack length, \(n\) is the strain hardening exponent, \(\bar{T}\) is the effective flow stress of the material and the other symbols have their usual meaning. In fact, \(\bar{T}\) is frequently regarded as a fitting parameter. This equation shows, in qualitative agreement with linear elastic fracture mechanics crack growth correlations, that for a given plastic strain the FCP rate increases with \(\Delta \sigma\). On the other hand, for deformation by Orowan looping (figure 7b), there is a plethora of literature which shows that crack initiation for disbursed slip is more difficult and the initiation life should be longer. Thus, the mechanical behaviour and corresponding dislocation substructure indicate very different effects of microstructure on various aspects of fatigue, which means that alloy design needs to be done with the actual failure criteria in mind. Another interesting example of the effect of slip mode is found in a recent study in which the reversibility of slip was correlated with the threshold stress intensity parameter \(\Delta K_o\) for a Ni-base alloy as a function of temperature as shown in figure 22 [46]. This correlation demonstrates that \(\Delta K_o\) increases with increasing slip reversibility, which clearly is related to the slip mode through the morphology and character of the \(\gamma'\) precipitates as well as thermal activation, which could produce non-planar deformation and reduce the reversibility. Modelling [47] and experimental [48] work pointed to the critical importance of slip mode. Assuming a kind of microscopic crack tip LCF process, the following equation resulted:

\[
\frac{da}{dN} = 4 \left( \frac{0.7A}{E(\sigma'_{ys})^{1+\delta \varepsilon_f'}} \right)^{1/\beta} \frac{1}{\ell^{1/\beta-1}} \cdot (\Delta K)^{2+\delta/\beta},
\]

(5.2)

where the primed symbols are fatigue properties, \(\ell\) is a process zone ahead of a propagating crack, \(S\) is a slip mode/slip reversibility factor, \(A\) is a plastic zone-related constant, \(\beta\) is the Coffin–Manson exponent and all other symbols have their usual meaning. The most important implications of the model were that large grain sizes, low SFE and a high degree of slip reversibility should result in lower crack growth rates. In an extensive review of fatigue [49], it was noted that equation (5.2) describes FCP behaviour for a wide range of industrially important alloys. The preceding work on model materials motivated an initial study to examine the feasibility of improving FCP rates through microstructural control of an alloy used in jet engines, in this case Waspaloy. The results are shown in figure 23 [50].
Figure 23. Fatigue crack propagation of Waspaloy at constant yield strength but with different microstructures at 25°C. The FCP rates order in terms of slip mode with planar reversible slip having the lowest rates and wavy slip having the highest rates [50]. GS, grain size.

From the results shown in figure 23, it is clear that more planar glide (strain concentration) is associated with dramatically lower crack growth rates. Planar glide is associated with a large process zone, equation (5.2), which would lower the FCP rate. It is also likely to be associated with a higher degree of slip reversibility, as shown in figure 22, where at lower temperatures the slip is more planar. The importance of slip reversibility in fatigue damage has also been pointed out by Mughrabi [31], who used shearing of γ’ particles demonstrated by Clavel & Pineau [27] to measure the degree of reversibility (figure 24).

A very detailed study in which the degree of slip planarity/reversibility was controlled by compositional adjustment and heat treatment was reported for a series of model Ni-base alloys [51,52]. Typical results are shown in figure 25 for crack growth behaviour (a) and for deformation substructures (b,c).

From the da/dN versus ΔK plots, it is clear that alloy 1S (low SFE, low δ and low Vf) is most resistant to crack advance. When normalized as described above, the FCP rate of this alloy system is at least two orders of magnitude slower than the others. The low volume fraction of precipitates results in a larger mean free path between obstacles for the mobile dislocations. The imposed plastic strain is therefore more easily accommodated, resulting in less damage accumulation and thus greater resistance to crack advance. The efficiency of the γ’ as obstacles to dislocation motion is reduced further in this alloy by the low values of APBE and δ. Details of the compositions, microstructures and physical properties are provided elsewhere [51,52].
Measurements [31] of slip reversibility for planar-slip PSBs in Waspaloy fatigued to failure at \( \Delta \varepsilon_p = 4 \times 10^{-3}, N_f = 550 \) cycles and \( T = 650^\circ C \) [27]. By algebraically measuring the net slip offset, it was determined that the slip was about 95% reversible.

Figure 25. FCP response of small \( \gamma' \) (0.06 \( \mu \)m) materials for \( R = 0.1 \). Data are normalized with respect to yield strength and elastic modulus [52]. Planar glide (b), alloy 2, and wavy glide (c), alloy 4.

The major findings and observations were as follows.

— Crack closure concepts could not explain differences in the FCP rates for both near-threshold and Paris regime crack propagation in the model Ni-base alloys studied here.
— FCP rates were dramatically low for those compositions and treatments that promoted planar, reversible slip.
— Alloys having high volume fraction, low APBE and low mismatch exhibited FCP rates that were approximately 50 times lower than other alloys.
— Internal resistance to damage ahead of a crack is achieved by low volume fraction of precipitates, low lattice mismatch and low anti-phase boundary energy. FCP resistance is increased by a planar deformation mode. However, in a planar slip material, on a
strength/modulus normalized basis, restricting dislocation motion decreases the alloy’s ability to accommodate damage and increases the FCP rate.
— At the same strength level, it has been demonstrated that the FCP rate can be reduced by at least a factor of 50.
— The implications of this study are that FCP rates in alloys of practical interest can be significantly reduced by heat treatment and modest compositional changes.

A detailed study of crack paths during FCP of Waspaloy was carried out in the short and long crack regime [53]. Crack extension mechanisms were studied in detail via surface replication and SEM, and the mechanisms were subjected to a statistical analysis for short and long cracks. It should be noted that only one microstructure was studied and natural variations in this microstructure were used to characterize interactions with the crack. Importantly, it was noted that most FCP data in the literature were obtained for ‘smoothed’ data as per ASTM or other recommended procedures. Such smoothing obviously masks the effects of crack deviations, retardations, re-initiations, etc., in favour of presenting ‘average’ behaviour, which is useful in design but which tends to mask microstructural effects. Of course, surface observations in themselves give only limited information but in this study they were augmented by post-mortem SEM analysis. Some of the important findings and issues raised by this study are as follows:

— Crack arrests occurred only at points on the crack front where the direction of crack growth was deflected, from which it follows that the more persistent arrests occurred at high-angle corrugated grain boundaries, as pointed out in a very recent review of the effects of grain boundaries [54].
— The likelihood of a deflection event causing an arrest decreased with increasing $\Delta K$, which is probably a reflection of the increased plastic zone size and increased driving force.
— Small grain sizes and increased boundary tortuosity increase the short crack growth resistance of a Ni-base superalloy. The grain size effect is in contradiction to other studies which reported a decrease in FCP rate for increased grain sizes [50,55]. However, the comparison is questionable since the previous studies were for long cracks; mechanisms of short and long crack growth can be, and frequently are, very different.
— For crystallographic cracking, FCP was higher and similar to rates seen in single crystals for other alloy systems.
— For long cracks, the threshold occurred because the whole crack front became pinned by crack arrests, consistent with the second major point.
— Perhaps the single most important result of this study was that the statistics of crack growth and arrest as a function of microstructure were characterized in detail (fig. 11 of that study). The statistical modelling of actual mechanisms could be used to improve high cycle fatigue life predictions. Another important aspect of this study was that the deterministic reasons for ‘scatter’ were appreciated and discussed.

For example, even though there is considerable evidence in Ni-base alloys that microstructural effects on FCP are present in the high $\Delta K$ regime for long cracks (cf. figures 22, 23 and 25), other explanations for microstructural effects have been put forward. For example, it has been posited that the regime of strong microstructural effect depends upon the ratio of the plastic zone to a characteristic microstructural dimension [56,57]. The gist of this idea is that the effects of microstructure become averaged out as this ratio increases since the larger the plastic zone, the smaller the tendency for crack deflection at microstructural barriers. In certain systems that may well be the case. However, in most Ni-base superalloys, the strengthening precipitates are of submicrometre size and, as they become larger, the fundamental deformation mode (which is presumably related to damage accumulation) changes and this effect is manifested within the plastic zone regardless of the ratio of the plastic zone to the microstructural dimension. This mechanism does not depend upon crack deflection so the preceding discussion is not meant
Table 3. Estimated and calculated confidence levels for pre-determined fuzzy rules [58].

<table>
<thead>
<tr>
<th>variable</th>
<th>rule</th>
<th>estimated confidence</th>
<th>calculated confidence</th>
</tr>
</thead>
<tbody>
<tr>
<td>test-type</td>
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<td>0.50</td>
</tr>
<tr>
<td></td>
<td>test is ( K_{max} ) then ( \Delta K_{th} ) is LOW</td>
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<td>0.81</td>
</tr>
<tr>
<td>( R_{th} )</td>
<td>( R_{th} ) is LOW then ( \Delta K_{th} ) is LOW</td>
<td>0.7</td>
<td>0.64</td>
</tr>
<tr>
<td></td>
<td>( R_{th} ) is HIGH then ( \Delta K_{th} ) is LOW</td>
<td>0.95</td>
<td>0.89</td>
</tr>
<tr>
<td>( T )</td>
<td>( T ) is LOW then ( \Delta K_{th} ) is HIGH</td>
<td>0.6</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td>( T ) is HIGH then ( \Delta K_{th} ) is LOW</td>
<td>0.9</td>
<td>0.85</td>
</tr>
<tr>
<td>environment</td>
<td>environment is VACUUM then ( \Delta K_{th} ) is HIGH</td>
<td>1.0</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td>environment is AIR then ( \Delta K_{th} ) is LOW</td>
<td>0.65</td>
<td>0.62</td>
</tr>
<tr>
<td>grain size (GS)</td>
<td>GS is SMALL then ( \Delta K_{th} ) is LOW</td>
<td>0.7</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td>GS is LARGE then ( \Delta K_{th} ) is HIGH</td>
<td>0.8</td>
<td>0.89</td>
</tr>
<tr>
<td></td>
<td>GS is VERY LARGE then ( \Delta K_{th} ) is LOW</td>
<td>0.7</td>
<td>0.68</td>
</tr>
<tr>
<td>( N:GS ) ratio</td>
<td>( N:GS ) is LOW then ( \Delta K_{th} ) is LOW</td>
<td>0.7</td>
<td>0.53</td>
</tr>
<tr>
<td></td>
<td>( N:GS ) is HIGH then ( \Delta K_{th} ) is LOW</td>
<td>0.6</td>
<td>0.53</td>
</tr>
<tr>
<td>yield stress</td>
<td>yield stress is LOW then ( \Delta K_{th} ) is LOW</td>
<td>0.8</td>
<td>0.81</td>
</tr>
<tr>
<td></td>
<td>yield stress is HIGH then ( \Delta K_{th} ) is LOW</td>
<td>0.9</td>
<td>1.00</td>
</tr>
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</table>

to negate the deflection argument but to point out other possibilities that have experimental and analytical foundations. Crack closure and roughness-induced crack tip shielding have been suggested and have gained considerable support (see [57]). However, the trend shown in figure 22 suggests that this is probably not operative in most Ni-base alloys. It has also been shown that microstructural effects persist in the mid-to-high \( \Delta K \) regime for FCP of IN 718 [55]. A more complete discussion of the pros and cons of crack closure on FCP is presented elsewhere [25].

(b) Applications of computational materials science to Ni-base superalloys

The kind of microstructural information discussed earlier served as input for advanced computational techniques such as neural network modelling. An example of this kind of modelling has been published with the goal of developing a neural network model for predicting the threshold in Ni-base superalloys [58]. In overview, this approach is a reality-based technique for dealing with complexity and relies heavily upon having a rich database. Various parameters are fed into the model with appropriate weighting factors and probabilities in a kind of fuzzy logic environment and the network is ‘trained’ by constant comparison with actual data. Microstructural parameters such as the volume fraction and other properties of \( \gamma' \) are fed into the model, and in this way the fundamental understanding gained over the years is part and parcel of the neural model. Table 3 illustrates the process.

Reasonable predictions of the threshold \( \Delta K_o \) were made with limited microstructural input. This paper provides a clear overview of the technique as applied to materials science problems and also gives several useful caveats which may be summarized as follows:

— The integrity of the dataset is of vital importance and must be accurate with respect to the problem being modelled.
— The data must cover a wide range of important variables and be highly reproducible.
— A neural network or neurofuzzy program should always be used in partnership with an expert from the problem field to avoid gross errors.
— Care must be taken such that the size of the dataset and the numbers of inputs are compatible.

A more advanced and detailed application of this method was used in a very complex application, life prediction of the fatigue life of notched components in Ni-base superalloys with variable microstructural features [59]. In this study, an artificial neural network (ANN) was used to correlate the material parameters in a macroscale internal state variable cyclic visco-plasticity model with microstructural attributes. A combination of limited experiments and polycrystal plasticity calculations on virtual microstructures was used for the range of experimental variables. The trained model was applied for notch root analysis with peak load dwells as a proof of concept, which, importantly, included exploring the possible impact of microstructure-sensitive constitutive models on life prediction for notched structures subjected to realistic load histories. These authors pointed out two important results [59, p. 524]:

— “Microstructure-sensitive ‘macro’ material parameters informed using an ANN based on computational polycrystal plasticity can be used to explore effects of a range of different microstructures on service behaviour of components. The response of such ‘engineered’ microstructures can be projected before such materials are physically realized. Such studies can enable setting target volume fractions and precipitate sizes in the case of Ni-base superalloys, facilitating component specific tailoring of the material.

— The sensitivity study of notch root response over the range of microstructures ... provides insight into the potential of the method outlined here for linking polycrystal plasticity calculations to macroscopic constitutive relations for use in component notch root analyses.”

It was interesting that the predicted trends for increasing dwell exhibited cross-over behaviour which could be indicative of intergranular fracture. One important use of such simulations is to link component-level dimensions to microstructural details. In this case, the methodology links length scales that vary by 6–7 orders of magnitude!

6. High-temperature fatigue: effects of stability, deformation mode, environmental damage and interactions

Microstructural instability may be manifested as changes in the form of precipitates (figures 16 and 17) and/or the appearance of new phases such as carbides (figure 14) and oxidized twin boundaries, grain boundaries and slip bands as shown in figure 13. In figure 26, oxidized slip bands impinging upon oxidized grain boundaries and nucleating cracks are seen [19].

Figure 26 is an example of the interaction between at least two damage mechanisms: (i) cyclic deformation and (ii) oxidation. Such interactions are discussed in more detail later. The stability of the precipitate structure is critically important in determining the high-temperature fatigue life. An example of this was shown by Ott & Mughrabi [60], who took advantage of the interaction between stress and mismatch to produce γ′ microstructures in single-crystalline variants of CMSX, in which the plates were either parallel to the applied stress or perpendicular. Single-crystal (SX) superalloys in the [001] orientation are widely used in turbine blade materials because of the superior creep resistance, and low modulus in this direction. This morphology was obtained using tensile or compressive holds at 1100°C (CMSX-4) or 1050°C (CMSX-6) in vacuum at stresses between 110 and 120 MPa, limiting the creep strain to no more than 0.4%. The results of fatigue testing are shown in figure 27 for CMSX-6, which is broadly representative of the results for CMSX-4.

The interactions of the crack with the different plate morphologies are shown in figure 28.

This study shows the critical importance of the microstructure and potential microstructural changes during the life of a component. These results may be understood in terms of the easier deformation in the channels at high temperatures (recall that the precipitate actually hardens at
Figure 26. Slip-band-induced cracking of oxidized grain boundaries in Waspaloy tested in LCF at 800°C. Microcracks are indicated by the dark arrows [19]. (Online version in colour.)

Figure 27. Effect of the $\gamma'$ precipitate morphology on the fatigue life of MCSX-6 at $T = 950^\circ$C and $\Delta\varepsilon_t = 0.9\%$ [60].

Figure 28. Crack interactions for CMSX-4 tested at $T = 1050^\circ$C and $\Delta\varepsilon_t = 0.9\%$ [60]. In (a), the crack moves easily through the $\gamma/\gamma'$ channels and in (b) it is deflected by the less deformable $\gamma'$. 
Potential damage mechanisms at high temperature are creep, fatigue and the environment. The effect of environmental damage in Ni-base alloys is critical and frequently dominant. In some cases, creep may actually reduce the effects of environment and be beneficial to the overall life. An example of such an effect is found in the work of Chasaigne [61] and discussed in an interpretive review [1]. In this work, the effects of creep and environment were studied for specimens taken from different sections of an N18 superalloy disc. The precipitates in the interior were coarser, and the creep or relaxation rate was higher than for specimens taken from the near-surface or ‘skin’ area which had very fine precipitates. The crack propagation rates are shown in figure 29 for various conditions. The nomenclature 10-300-10, for example, means loading and unloading times of 10 s with a 300 s hold. Firstly, note that under vacuum for all conditions and in air for the 10-10 cycles there was no difference in the FCP rates. These two sets indicate that creep effects are either not present (e.g. 10-10 cycle) or have only minimal importance as damage mechanisms. However, strikingly, for hold times in air the difference is very pronounced and associated with stress relaxation at the crack tip, rendering the effect of the environment (oxidation) less aggressive.

A model for the crack growth rate in a Ni-base superalloy was developed by considering the interaction between a growing oxide and the metal substrate during a compressive hold in which detailed stress analyses of the oxide and crack tip were calculated via finite-element analysis (FEA) [62]. Basically, the strains imposed by the oxide are accommodated by creep in the metal and the crack ‘follows’ the oxide. The basic physical aspects of the model are shown in figure 30. Properties of René 95 were used in carrying out the computations. A major result of the analysis was that the crack growth rate was found to be proportional to the hold time. It would have been useful to have provided a comparison with experimental data. However, the approach and reasoning appear to provide a useful approach in considering interactions during hold time fatigue. This approach was a natural extension of the initial work on developing a model for cracking during LCF under compressive holds. The model was developed with the following principal features [63]:

— Tensile stresses develop during the de-straining phase of the cycle after relatively few cycles because of creep effects.
— The tensile stress opens cracks, exposing the surfaces to the atmosphere, thus promoting growth of the oxides.
— As a result of oxidation, there is a dilatational field with an associated strain rate that induces a compressive growth stress.
Figure 30. Crack tip processes for one LCF cycle indicating the effects of creep and oxidation interactions [63]. TGO, thermally grown oxide.

— During the re-straining cycle, transverse extension of the substrate produces bi-axial tension in the oxide, which forces the oxide into the substrate along the crack front.
— Finite-element simulations predicted local crack growth rates comparable to somewhat limited experimental observations.

Since fatigue, creep, oxidation and their interactions under mechanical loading are all possible damage (or damage-limiting!) modes it is important to actually identify the rate-controlling process. In this regard, examination of the fracture surface alone is really insufficient since prominent features (e.g. large creep voids on the fracture surface may have formed only towards the end of life, at which point tertiary creep dominates failure but may have little or nothing to do with the final failure). This was recognized by Sorby in the late nineteenth century. He stated that [64, p. 255]

‘Compared with what can be learned from good sections, the study of mere fractures teaches very little respecting the ultimate structure: because a fracture occurs along a line of weakness between the constituent crystalline grains, whilst a section shows the true relation and ultimate constitution of the constituent crystals.’

In an effort to address the prediction of critical rate-controlling mechanisms in Ni-base alloys during high-temperature fatigue, a graphical method was proposed in which various processes are compared on the basis of the ‘flux’ (essentially like a diffusion coefficient) versus $1/T$ [65]. The results are shown in figure 31 for In 718,3 where it was suggested that the slowest process would control the actual rate.

The so-called strain flux is a reasonable quantity to plot in this way given the form of creep curves with respect to temperature. However, it is essentially a first approximation in that it appears to ignore stress effects on the activation energy (slope of curve). The authors point out that it is in agreement with the idea that a rapid relaxation will impede environmental degradation as discussed previously. It is also in agreement with detailed studies of cracking in In 718. In 718 is well known to be very sensitive to environmental damage, as shown in figure 32 [66]. The details of such damage were elegantly explained using the hypothesis that porous spinels of NiO and FeO initially form at the grain boundary at atmospheric pressure. Oxygen diffuses to the base metal at the crack tip to continue growth of this oxide at decreasing partial pressure because of the tortuous path that oxygen must follow. After sufficient growth, the oxygen partial pressure is so low at the crack tip that stable Cr$_2$O$_3$ forms which is dense and protective, resulting in a

3In 718 is an Fe–Ni-base superalloy (table 1) strengthened by $\gamma'$ DO$_{22}$ precipitates.
Figure 31. Rates of various processes in In 718 which are proposed for use in determining which mechanisms will be rate controlling [65]. (Online version in colour.)

Figure 32. Effects of environment on the FCP behaviour of In 718 [66].

decreased crack growth rate [67,68]. These ideas can be understood in terms of an Ellingham–Richardson diagram for oxide formation [69]. There was an incubation time for such passivation which increased with increasing oxygen partial pressure (figure 33).

Compositional modification may also play an important role in the fatigue behaviour of Ni-base superalloys to the degree that compositional changes affect life-determining mechanisms. As an example, consider In 718 where the environmental effect is often related to grain boundary oxidation. This appears to be a case in which small chemical modifications could greatly reduce
boundary attack without degrading other properties. Studies were carried out to examine the effects of B on FCP behaviour [70] and the results are shown in figure 34.

Boron, because of its small size, tends to segregate at grain boundaries. The results shown in figure 34 showed that B shifted the ‘weakest link’ from oxygen-embrittled grain boundaries to fracture by planar, crystallographic slip, resulting in a significantly reduced FCP rate. SEM examination of the fracture surfaces showed that the fracture mode became increasingly transgranular with increasing B. In reducing the diffusion rate and concentration of O, B could both slow down grain boundary diffusion and limit the formation of complex oxides.

The effects of Re on the morphological stability of Ni-base alloys have been investigated [71] for the Ni-base superalloys shown in table 4.
It was shown that

— the $\gamma'$ precipitates were more stable as seen in TEM and as evidenced macroscopically by a higher, more constant mean stress during strain-controlled LCF testing;
— the addition of Re caused segregation of other elements to the $\gamma$ matrix, thereby causing a more homogeneous slip distribution; and
— there was a modest but discernible improvement in the LCF life.

Typical results are shown in figure 35.

These results contradict those of Ott & Mughrabi [60], who documented the beneficial effects of rafting, and those of Antolovich et al. [44] and Tomkins [45], who showed high mean stress during cycling reduced the life. Clearly, some additional work is required to understand the full range of Re effects and provide a mechanistic basis for these differences.

7. Environment–fatigue interactions in thermo-mechanical fatigue

The ultimate practical goal of all of the fatigue studies reviewed in this paper is to be able to predict and extend the fatigue lives of components used in aeroengines. The great majority of fatigue studies have been carried out isothermally. However, in operation the temperatures are not constant and the applied loads and strains are time-dependent. The loads are not in phase with the strains and this situation is referred to as TMF, as explained earlier. In recent years, research aimed ultimately at developing a life prediction methodology has been carried out in our laboratories and the evolution of this work has been reported in numerous publications [72–80]. The most important findings are reprised in this section but it is appropriate to note that the work was motivated, in part, by simple experiments designed to simulate TMF in René 80

In this section we refer to ‘loads’ in a generic sense, meaning it can refer to stresses or strains depending upon the context.
Figure 36. (a) Schematic of model for prediction of out-of-phase TMF in a single crystal. (b) Observations of slip band impingement in the Ni-base superalloy PWA 1484 [78,80].

[81]. In these experiments, a specimen was first cycled at 760°C to about half of its projected life (approx. 270 cycles) at a strain range of 0.05%. It was then cycled at this strain range at 25°C. In this so-called hi-lo (HL) cycle, the specimen broke on the first loading cycle at 25°C even though, according to isothermal fatigue data, the sample would have been expected to last for many hundreds of additional cycles. It was determined that slip bands at 25°C caused cracking of the previously formed oxides. When the loading sequence was reversed (lo-hi cycle (LH)) with no change in the loading parameters, fracture occurred at the ‘normal’ number of cycles at the higher temperature for the imposed loading conditions. In essence, prior damage at 25°C was annealed out. Furthermore, when the experiment was repeated by cycling for about one-quarter of the life at high temperature, the room temperature life was over 6000 cycles, indicating that damage accumulation was not only sequence dependent but nonlinear as well.

The TMF model was first developed using the semi-empirical model proposed by Neu & Sehitoglu (NS) [82], which works reasonably well for practical life prediction and which includes the damage processes of oxidation, creep and fatigue. It was found that the tuned NS model worked fairly well (about 70% correlation) in making life predictions. However, it was felt that by including the actual physical processes a model could be developed that not only was more accurate but also could be used for interpolation and also for extrapolation. Based on all of the preceding, and noting that out-of-phase TMF is generally most damaging in Ni-base superalloys, the model refined and extended a previous environmental-based model for LCF in polycrystalline René 80 [44]. The basis of the model is shown in figure 36 [78,80]. The fundamental notion is that oxides formed at the high-temperature/low-strain part of the cycle are cracked by impingement of slip bands formed at the low-temperature/high-strain part. The effects of temperature on coarsening and slip band formation are taken into account, as is the effect of surface roughness on oxidation rates. With suitable modification, the model is also able to account for imposed high-frequency testing [79].

The model may be expressed in analytical form as

\[
Ni = \left( \frac{A r_0 (\Delta \gamma_{sl}^{in})^{-m}}{\exp(uT_{eff})} \right)^{1/3} \exp \left( \frac{Q_{sl} + 2Q_{ox} - B(\sigma_{max} + |\tau_{min}|)}{RT_{eff}} \right) \\
\times \left( \frac{v}{v + t_h + 1/2} \right)^{-\frac{n+d}{8}} \left( \frac{R_a}{8} \right)^{-h \cdot p}, \tag{7.1} \]

where \(A\) and \(u\) are constants determined from precipitate coarsening, \(t\) is the total precipitate coarsening time, \(r_0\) is the initial equivalent precipitate size, \(\Delta \gamma_{sl}^{in}\) is the inelastic strain on the slip system of interest, \(T_{eff}\) is the effective temperature (defined below), \(Q_{sl}\) is the slip activation energy, \(Q_{ox}\) is the oxidation activation energy, \(\sigma_{max}\) is the maximum tensile stress in the stabilized cycle (occurring at the cycle minimum temperature), \(\tau_{min}\) is the shear stress for slip band
formation, $B$ is a constant, $R$ is the gas constant, $m$ is the strain dependency exponent, $n$ is the
time exponent determined from the non-stressed $\gamma'$ depletion experiments, $d$ is an experimentally
determined parameter related to precipitate coarsening kinetics, $\nu$ is the cycle frequency, $t_h$ is
a high-temperature hold time (if any), $h$ is the exponent determined from $\gamma'$ depletion surface
roughness experiments, and $R_a$ is the specimen surface roughness (arithmetic average) and $p$
is a combined parameter. The term $T_{eff}$ is the Arrhenius-averaged temperature in a TMF cycle
defined as

$$\exp\left(\frac{-Q}{RT_{eff}}\right) = \frac{1}{\Delta t} \int_{t_1}^{t_2} \exp\left(\frac{-Q}{RT(t)}\right) dt,$$

where $Q$ is the activation energy for the process, $\Delta t$ is the cycle time, $R$ is the gas constant and $T(t)$
is the temperature–time profile of the cycle.

Using experimentally determined values for single-crystalline PWA 1484, life predictions were
made and compared with experiment. The results are shown in figure 37.

It can be seen that the predictive capability of the model is excellent, correlating the data to
about 92% accuracy. The precipitate morphology was examined for both in-phase and out-of-
phase loading as shown in figure 38 [80]. The morphological changes were driven by the sign of
the stress and the high temperature and are fully consistent with a negative mismatch parameter.
Connecting with the work of Ott & Mughrabi [60], the alignment of the plates in the direction of
loading is expected to be beneficial for the TMF life.

As a further test of the model, it was applied to the case of notches with a notch severity of
$K_t = 1.72$ and computing crack tip stresses and strains using Neuber’s rule. Agreement with
experiment was reasonable (within about 20%) but it was pointed out how a more fully developed
crystal visco-plasticity (CVP) model along with detailed FEA would have the potential to
significantly improve the notch predictions [80]. An idealized go/no-go path for moving forward
in making life predictions was also presented and is reproduced in figure 39 for completeness.

Of critical importance in TMF are the facts that: (i) oxidation is generally a critical damage
mechanism for most Mi-base alloys, (ii) damage mode interactions that may not occur in
isothermal fatigue are frequently of critical importance in TMF, (iii) no explanation for TMF
which is applicable to all Ni-base alloys is possible since the damage depends on the details
of deformation and environmental attack, which depend on compositional and microstructural
Figure 38. Effect of a TMF cycle on the precipitate microstructure of single-crystalline PWA 1484 loaded in the vertical direction [80]. Out of phase (a) and in phase (b). TMF cycling at 1.3% strain with the load axis in the vertical direction. The endpoint temperatures were 550°C and 1050°C. The morphological changes are consistent with a negative mismatch parameter.

Figure 39. Suggested path for development of improved notch TMF life prediction [80]. (Online version in colour.)

features, and (iv) while out-of-phase cycling is usually the most damaging cycle type, depending on the system and details of damage mode interactions, in-phase cycling can be life limiting. Thus, development of life prediction models in Ni-base alloys must be based on the underlying physics of the damage process, which can be quite different for different alloys and/or heat treatments. These points are amply illustrated in various studies that have shown, depending on the alloy system, the following damage modes and interactions:

— Twinning [83–85] and oxidation.
— Recrystallization, twinning slip bands and oxidation [86]. In fact, these authors state an essential feature of TMF [86, p. 357]:

‘…deformation and damage mechanisms occurring during TMF are rather different from those traditionally reported for creep or isothermal fatigue. In all cases examined, the
deformation is localized within a rather small number of deformation bands. While these bands were found to consist mainly of micro-twins in some alloys, in others they might be better described as slip or shear bands. Furthermore, in some circumstances these bands are prone to recrystallization.

— Modest compositional changes (e.g. 0.25% Si) to inhibit deformation band formation and recrystallization [87].
— Oxidation/coarsening/deformation mode interactions which produce in-phase lives significantly lower than the corresponding out-of-phase lives [88].
— Crack formation from initial defects [89,90] such as pores [91].
— Coatings, used to protect the base alloy from environmental attack, can exhibit the same TMF life as the base metal. These authors found that ‘results indicate that the coating does not significantly affect the fatigue lives of Mar-M247’.
— Other studies confirm the point that each material, heat treatment and test condition must be considered in terms of the basic damage mechanisms which may be unique to the material/loading profile in question [92]. As evidence of this, it has been stated that [93] ‘To date, no definitive test program systematically covering the different variables has been published in the scientific literature’.

8. Concluding remarks

In this paper, the linkage between microstructure, deformation mode, environmental damage, compositional modifications and life prediction have been reviewed and discussed. It was demonstrated how a fundamental understanding of these processes and their interactions can be used to develop accurate life prediction methodologies. It was also pointed out how this basic understanding is being incorporated into advanced computational techniques. It is anticipated that it is in this area that spectacular future advances will be seen.

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