High frequency in situ fatigue response of Ni-base superalloy René-N5 microcrystals

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1. Introduction

A fundamental understanding of fatigue mechanisms is essential to predict the usable life of engineering components in aerospace applications. The fatigue life of turbine engine components, for instance, has a major influence on the cost of maintaining an aircraft fleet. The turbine blades experience low cycle thermo-mechanical fatigue loading from their start-up/shut-down cycles. High cycle fatigue is also induced on turbine blades via smaller vibrational loads. To maximize the fatigue and creep resistance of these integral components, turbine blades in modern jet engines are fabricated from predominately single crystal Ni-base superalloys [1].

The fatigue behavior of Ni-base superalloys has been well-characterized at both room temperature and high temperature testing conditions [2–4]. Superalloys, with their characteristic γ–γ′ microstructures, have been shown to exhibit both cyclic hardening and cyclic softening in fatigue [2]. At room temperature the cyclic hardening is attributed to an increase in the density of slip bands [5], while cyclic softening has been attributed to the dissolution, disordering, and the shearing of γ′ precipitates [2,3].

While conventional bulk scale fatigue tests provide a way to quantify the fatigue life of materials, specific microstructural features that result in failure are difficult to ascertain. This motivates researchers to investigate the underlying mechanisms of fatigue failure as opposed to taking a stochastic approach in predicting the fatigue life of engineering components. One approach to accomplish this is to cyclicly test small volumes of materials. Small material dimensions allow for a more fundamental investigation of the microstructural features that contribute to fatigue failures.

Further motivation for micro-scale fatigue testing is to characterize crack growth in ductile materials. Such findings can be used to investigate the rate of crack growth at various crack length scales. Stable crack propagation experiments have proven to be difficult for several reasons. First, notches made using conventional...
microfabrication methods are generally not atomically sharp and have a large plastic zone in ductile materials. Blunting effects inhibit the crack growth and promote yielding behavior at notch tips. Second, inertial effects from large strain bursts make it difficult to control the strain on the sample once a crack initiates which often results in catastrophic fracture.

Several studies have made progress in the field of microscale fatigue testing [8–13]. Schwaiger and Kraft performed a fatigue study on thin Ag films on SiO2 substrates [9]. Cantilever micro-beams were fabricated using a combination of sputter deposition and photolithography. The micro-beams were then tested by imposing a sinusoidal load superimposed over a mean load. This methodology was unique in its utilization of the continuous stiffness measurement (CSM) method built into many nanoindenter systems. However, with this experimental methodology it was not possible to maintain a zero mean stress and the experiments were limited to film-substrate sample geometries.

More recently, Kiener et al. performed in situ low cycle fatigue experiments in a scanning electron microscope (SEM) on micro-beams under bending [10]. In this approach, fully reversed loading (i.e., zero mean stress) was achieved using a custom-made W indenter probe. The probe was able to push and pull on the micro-beam at loading end but in between half-cycles the grip would lose contact with the specimen. The method was able to reveal deformation mechanisms during fatigue; however, it was unable to exceed 100 cycles. Kirchlechner et al. have also performed in situ micro-beam bending while performing Laue diffraction to characterize the dislocation motion during fatigue; however, they were also limited to cycle counts on the order of ~ 10 [11].

Some researchers have taken the approach of performing in situ microscale uniaxial fatigue experiments [12,13]. These experiments have exhibited differences in hardening/softening behavior when compared to their monotonic loading behavior. Possible explanations for this behavior include friction between the testing tip and the sample or perhaps the lateral stiffness of the loading frame. High load frame stiffness perpendicular to the loading direction has been shown to cause lattice rotation and thus alter the active slip systems. This effect has been mitigated using high aspect ratio beams. The method was able to reveal deformation mechanisms during fatigue; however, it was unable to exceed 100 cycles. Kirchlechner et al. have also performed in situ micro-beam bending while performing Laue diffraction to characterize the dislocation motion during fatigue; however, they were also limited to cycle counts on the order of ~ 10 [11].

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Investigations of fatigued materials have found that crack initiation is most commonly observed at high-angle grain boundaries, twin boundaries, and alongside surface extrusions (i.e., intrusions) [6,17,18]. Thin film fatigue studies have found that for larger film thicknesses (≥ 3 μm) the dominant mechanism for crack initiation is surface intrusions [19,20]. When the film thickness is decreased to the submicron level, cracks are almost exclusively found at twin boundaries and grain boundaries. It is argued that for submicron film thicknesses, size-effects inhibit the ability of the micrometals to accumulate plasticity. Less plasticity means less dislocation dipole annihilation which is believed to lead to surface extrusions/intrusions [21]. Also, void formation at the interface between the film and substrate has been found to be a major contributor to crack initiation [22]. Despite the breadth of studies carried out, there is still relatively little known about crack initiation, and further small-volume studies are necessary to elucidate the mechanisms that lead to crack initiation.

The motivation of the current work is to characterize plasticity, crack initiation, and fatigue life of single-crystal, Ni-base superalloy René-N5 cantilever micro-beams. In that attempt, an in situ SEM methodology is developed that uses high frequency actuator dynamics built into a nanoindenter to apply a cyclic load at high frequencies. In the following, the details of the experimental procedure are first presented in Section 2. The experimental results showing the effect of the imposed strain amplitude on the fatigue life and deformation mechanisms are then examined in Section 3. In Section 4 further insights regarding the deformation mechanisms and the fracture surface morphology are discussed. Finally, a summary and concluding remarks are made in Section 5.

2. Experimental setup

2.1. Material and sample preparation

The material used in the current study is a single-crystal René-N5 Ni-base superalloy acquired from General Electric Aviation. The composition of the alloy by atomic percentage is 63% Ni, 7% Cr, 7.5% Co, 1.5% Mo, 5% W, 6.5% Ta, 6.2% Al, 0.05% C, 0.004% B, 0.15% Hf, 3% Re, and 0.015% Y [23]. The alloy has a two-phase microstructure that consists of a γ matrix and an ordered array of cuboidal γ’ precipitates. The γ’ phase constitutes the majority of the alloy by volume fraction and are approximately ~ 300 – 500 nm in size [24]. Rectangular samples were prepared from the as-received bulk single crystal by slicing 6 × 5 × 12 mm² sections using wire electrical discharge machining (EDM), followed by mechanical polishing of the surface using silicon carbide metal-lurgical papers.

2.2. Microspecimen fabrication

Cantilever-like micro-beams were fabricated on the top edge of the single crystal sample such that the side and top surfaces of each micro-beam can be directly viewed in the SEM during testing. The micro-beam was oriented such that the [100] and [001] crystallographic directions of the FCC lattice were parallel to the micro-beam length and loading directions, respectively, as shown in Fig. 1. The cross-section of each micro-beam was rectangular with approximate dimensions of 8.5 × 8.5 μm², while their lengths were
The micro-beam fabrication was conducted in two steps. First, a Clark MXR femtosecond laser was used to fabricate a rough geometry of the micro-beam [25]. A laser energy of ~40 μJ was used with a repetition rate of 1.0 kHz. During the ablation, the laser beam is stationary and the bulk crystal is repositioned using an Aerotech ANT-95 XY nanopositioning stage. Multiple passes were needed to achieve the required dimensions. Initially, the [001] direction was laser-milled to obtain a lamella of approximately 15 μm in thickness and 120 μm in depth. The bulk single crystal was then rotated 90° such that the [100] direction was in line with the laser and the under-side of the micro-beam was milled. Second, after the rough laser-milling, the final dimensions of the micro-beams were attained using an FEI DualBeam 235 focused ion beam (FIB). An ion-beam current of 5 nA was then used to achieve the required dimensions. An ion-beam current of 5 nA was then used to achieve the final dimensions of the micro-beams.

2.3. Testing methodology

The fatigue setup presented here and shown in Fig. 1 combines the use of a Tescan Mira3 scanning electron microscope and a Nanomechanics InForce 1000 nanoindenter [26]. The nanoindenter is held by a stage-mounted cradle inside the SEM. The cradle allows for precise control of the extension of the indenter in the z-direction as well as x and y positioning of the specimen using micro-positioners. In the current setup, the lowest achievable working distance between the electron source and the sample is ~40 mm.

The indenter tip used consists of a tungsten micromanipulation needle with a tip radius of ~20 μm that is glued to a 140 μm diameter SiC fiber with a 30 μm diameter tungsten core, using a hard 5-min epoxy mixed with graphite powder for conductivity. The SiC fiber has a length to diameter ratio of approximately 100:1, which allows for compliance lateral to the direction of loading and reduces any possible misalignment.

Before testing, the indenter tip is inserted into the polymer-based adhesive Kleindiek Nanotechnik SEMGlu [27]. This is done using the cradle extension and micropositioning stage controllers while viewing the process using a low intensity electron beam. Once the tip is wet with ~1,000 μm² of SEM glue, the tip is lowered to be in contact with the end of the micro-beam resulting in a very small downwards deflection of no more than ~2 μm. The SEM raster box is then positioned onto the glue and beam end and the electron beam intensity is increased to cure and harden the SEMGlu within a few minutes. A quasi-static loading cycle is performed on the beam and if the beam does not exhibit any significant sign of compliance lateral to the direction of loading and reduces any possible misalignment.

The InForce 1000 Nanoindenter has a loading capacity of 1.0 N and is capable of imposing large dynamic loads. In the current study, the imposed oscillating force on the micro-beam tip is a periodic sinusoidal load with a driving frequency, ω, and a loading amplitude, Fmax, such that F(t) = Fmax sinωt. Due to the damping effects in the nanoindenter’s electromagnetic coil, the resulting displacement amplitude is strongly dependent on the loading frequency. The nanoindenter load frame and the sample can be modeled as a one-dimensional (1D) forced spring-mass-damper system and the complex representations of the imposed force and the resulting displacement as a function of time can be expressed as follows

\[ F(t) = F_{\text{max}} e^{i\omega t} \]  
\[ d(t) = d_{\text{max}} e^{i(\omega t - \phi)} \]  

where \(d_{\text{max}}\) is the resulting displacement amplitude. The phase difference between the load and displacement response, \(\phi\), is given by Ref. [28]:

\[ \phi = \tan^{-1} \left( \frac{2\zeta \omega}{\omega^2 - 1} \right) \]  

where \(\omega_0\) is the natural frequency of the system and \(\zeta\) is the damping ratio. A schematic of the steady-state imposed load and displacement response are shown in Fig. 2 (a) and 2(b), respectively.

The ratio between the force transmitted to the system and the imposed force is referred to as the transmissibility, T, and it depends on the frequency and damping parameters as follows [28]:

\[ T = \frac{1 + \left( \frac{2\zeta \omega}{\omega^2 - 1} \right)^2}{\left( \sqrt{1 - \left( \frac{\omega}{\omega_0} \right)^2} \right)^2 + \left( \frac{2\zeta \omega}{\omega_0} \right)^2} \]  

Both the transmissibility and phase angle are shown schematically in Fig. 2(c) and (b), respectively. The maximum transmissibility occurs approximately at the resonance frequency, \(\omega_0\). This also coincides with a phase angle of approximately 90°. At very high frequencies the damping effects reduce the transmissibility to zero and the phase angle between the load and displacement reach 180°. If the stiffness of the system was to increase (i.e. the natural frequency increases while the damping ratio decreases), both the transmissibility and the phase angle curves would move to the right. Likewise, a decrease in stiffness would move the curves to the left. Since both curves at the natural frequency are steep, even a small change in stiffness would have a profound impact on the phase angle given that the driving frequency is held constant. Thus, the phase angle of the system provides a very sensitive way to quantify changes in stiffness while applying a harmonic load.

On the other hand, the dynamic stiffness is defined as the real portion of the ratio between the applied load and the resulting displacement and takes the form:

\[ S_{\text{dyn}} = \text{Real} \left( \frac{F(t)}{d(t)} \right) = \frac{F_{\text{max}}}{d_{\text{max}}} \cos \phi \]  

Here, the cosine in equation (5) leads to a high sensitivity of the dynamic stiffness with respect to changes in the phase angle. The dynamic frequency is thus optimized such that the imposed loading frequency results in a phase angle within approximately 3° of resonance (i.e., \(\phi \approx 90^\circ\)). This results in maximum sensitivity to changes in the micro-beam’s stiffness. The resonant frequency for the current system is in the range of 40–50 Hz. The maximum operating frequency is limited by how quickly the actuator can oscillate before damping effects become too restrictive to allow the target displacement amplitude to be reached. In practice, this limit is found to be approximately 300 Hz for a dynamic displacement of 10.5 μm. Major sources of damping are from eddy currents in the electromagnetic coil of the indenter. The beam and the load frame are in parallel and consequently experience the same displacement.

In the context of this paper, the term “dynamic stiffness” is used to refer to cyclic hardening or softening; however, this is somewhat of a misnomer. Stiffness, in the context of solid mechanics, is used
to refer to the elastic region of a material’s response to a load. In this study, dynamic stiffness is calculated by measuring the dynamic properties of materials undergoing cyclic loading. In a given cycle, there are both elastic and plastic contributions to the total deformation. If the material were to harden, then a larger dynamic force would be required to maintain the same dynamic displacement and the dynamic stiffness would increase. Thus, the term “dynamic stiffness” is used to describe changes in the flow stress as well as modulus in this paper.

The nanoindenter setup utilizes a phase lock-in amplifier to extract the displacement signal at the driving frequency and filter out any unwanted noise corresponding to other frequencies. A fast Fourier transform is performed on the filtered displacement signal over a period of 20–100 ms to determine the displacement amplitude along with the phase angle between the displacement and load signals. These dynamic parameters are continuously measured and recorded during dynamic loading. Thus, the dynamic stiffness can be continuously calculated from Equation (5). Also, the error between the target and the measured displacement amplitude is computed and a control loop adjusts the applied dynamic force accordingly to maintain the target displacement amplitude.

It should be noted that while the loading end of the micro-beam oscillates at a high frequency, the fixed end of the micro-beam remains still allowing for continuous monitoring of surface features at this fixed end. The SEM scan rate is 3.13 pixels/second and videos of the micro-beam experiments are generated using a screen capture software with a frame rate of 30 frames per second.

2.4. Strain amplitude calculations

The strain amplitude is determined using a finite element simulation using the ABAQUS software package. An elastic-plastic isotropic material model is assumed with elastic properties $E = 128$ GPa and $v = 0.28$ [23]. For all plastic material behavior, the stress-strain data as obtained from a 10 µm single crystal micro-pillar compression experiment [24] is used to relate the strain deformation to stress values. The model geometries used for each beam were simple rectangular prisms with the dimensions adjusted to match the SEM-measured dimensions for each beam. The length of each beam is measured from the fixed end to the centroid of the indenter tip. The boundary conditions imposed were a fixed boundary on one end of the rectangular prism and a vertical displacement boundary condition in the [001] direction on the opposite end. The vertical displacement is the maximum displacement imposed by the indenter (i.e., the displacement amplitude). The loading-end of the beam was not prescribed any displacement boundary conditions in the [100] or [010] directions to allow for rotational displacement at the tip. All stress and strain data reported are the effective values at the notch of the beam as computed by these simulations.

3. Results

3.1. Fatigue life

For all fatigue tests performed here, failure is defined as the point at which the micro-beam completely fractures from the fixed end. The cycles to failure as a function of the maximum strain amplitude at the notch from all samples tested are summarized in Fig. 3. The results are also fitted to a Coffin-Manson power law [29]:

$$\Delta \epsilon / 2 = \sigma / E \left(2N_f \right)^b + \sigma_f \left(2N_f \right)^c$$

(6)

with a fatigue ductility coefficient $\sigma_f = 1.02$, a fatigue ductility exponent $c = -0.688$, a fatigue strength coefficient $\sigma_s = 2.09$ GPa, and an Basquin exponent $b = -0.067$. It is clear that the predicted fatigue ductility exponent from the current experiments is within the range of values typically reported for metals ($-0.5 \leq c \leq -0.7$).

In addition, the fatigue life measured for different bulk Ni-based single crystal superalloys during uniaxial fatigue experiments [2,30] are also shown in Fig. 3 for comparison. It should be noted that the stress concentration at the fixed end of the beam in the current study plays a big role in determining the fatigue life of the micro-beams. These types of stress localizations are not present in uniaxial bulk fatigue tests, and as such, comparisons with the bulk results can only be made in a qualitative manner. Nevertheless, even with the stress concentration in the present study, the micro-beams show an increase in the overall fatigue life when compared...
to bulk specimens, which can be attributed to sample size effects. This size effect has also been observed in small-scale fatigue tests of wire specimens [31]. Khatibi et al. observed a Hall-Petch like behavior in varying the diameter of fatigue wire specimens [32]. Although there are various mechanisms used to explain the size effect, it is well established that metals having micro-scale dimensions are stronger. This would mean that for a given strain amplitude, there is a reduction in plastic strain for such micron-scale metals, as compared to their bulk counterparts. Since plastic strain is strongly correlated to fatigue life, this would indicate that smaller sizes should yield longer fatigue life.

It should also be noted that in the high cycle fatigue regime there is significantly more scatter than that observed in the low cycle fatigue regime. This can be attributed to the fact that in the high cycle regime the sample life is significantly affected by pre-existing defects (e.g. casting defects, surface roughness, etc.) in the sample [33]. In microcrystal testing, the spacing between defects is expected to be larger than the sample size. Thus, a statistical approach with a larger number of test samples is required to characterize the fatigue life of micro-scale specimens in the high cycle and ultra-high cycle regime.

3.2. Large cyclic strain amplitude response

Fig. 4(a) shows the dynamic stiffness for a fatigue test conducted at a strain amplitude of $9.26 \times 10^{-3}$. During the experiment the evolution of the surface morphology near the micro-beam’s fixed end is continually monitored and the SEM micrographs during testing at different cycle numbers of this test are shown in Fig. 5. A movie of this experiment is also shown in the online Supplementary movie 1. These results are representative for all tested samples at a strain amplitude greater than $7 \times 10^{-3}$.

As clearly observed from the insert in Fig. 4(a), a periodic oscillatory noise is superimposed on a mean dynamic stiffness value. The phase angle does not exhibit this behavior. The oscillatory noise occurs predominantly due to small overshoots of the target displacement amplitude, where the control loop for the load amplitude never settles on a constant value but rather oscillates about the required load amplitude for the entirety of the fatigue test. This noise, although very small, becomes amplified in the calculated dynamic stiffness since the vibrational frequency is very close to resonance. For this reason, a moving average with a time constant of 4 s (equivalent to 200 data points) is used to smooth the dynamic stiffness curves. This is achieved by using a discrete convolution algorithm on the raw dynamic stiffness curves. The result of this procedure is shown in Fig. 4(b). By comparing Fig. 4(a)
and (b) it is clear that all trends are directly captured after the smoothing step.

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2017.10.049.

It is also important to note that at the beginning of the test, there is a temporary spike in the dynamic stiffness due to the transient nature of the damped harmonic oscillators. This transient behavior typically decays quickly within ~10 – 20 seconds.

Fig. 4(b) indicates that for the majority of the fatigue life of the micro-beam, the dynamic stiffness gradually decays as the number of cycles increases. This is an indication of cyclic softening occurring in the sample. Towards the end of the life of the micro-beam (i.e. after ~ 14,620 cycles), a dramatic decrease in the dynamic stiffness is observed. From the surface morphology images as a function of number of cycles it is observed that early on during the deformation visible extrusions form on the top and bottom edges of the micro-beam as shown in Fig. 5(a) after 6840 cycles. Slip trace analysis indicates that these extrusions coincide with \{111\} slip planes. As the number of cycles increases, more extrusions are formed and become longer and more pronounced (Fig. 5(b)). After 14,620 cycles the first stages of crack nucleation become visible at the lower corner of the fixed end (Fig. 5(c)). This lower corner is a preferred site for crack nucleation due to the stress concentration at this point. Once the crack nucleates the dynamic stiffness is observed to dramatically decrease as observed in Fig. 4(b) as the crack propagates along the width of the beam until it spans the entire width resulting in complete fracture at the fixed end (see Fig. 5(d)-(f)). The dramatic decrease in the dynamic stiffness observed during the crack propagation stage is mostly due to the dramatic decrease in the cross-sectional area at the fixed end of the micro-beam. It is interesting to note that the process from visible crack nucleation to complete fracture spans a total of 1,680 loading cycles.

3.3. Small cyclic strain amplitude response

The dynamic stiffness versus cycle number for a micro-beam cyclically deformed with a strain amplitude of $7 \times 10^{-3}$ is shown in Fig. 6(a). This is representative for samples cyclically deformed at a strain amplitude less than $7 \times 10^{-3}$. Unlike the response under larger cyclic strain amplitudes, the dynamic stiffness exhibits a gradual increase by 45% with increasing number of cycles. This indicates cyclic hardening in the micro-beam. After the onset of crack initiation (~ $3.95 \times 10^4$ cycles) the dynamic stiffness decreases gradually over the next $2.28 \times 10^5$ cycles. When the crack reaches a critical length, steady crack propagation commences and the dynamic stiffness dramatically decreases. The dynamic stiffness during the last $1.35 \times 10^5$ cycles is shown in Fig. 6(b) showing the inflection points in the dynamic stiffness curves and indicating two regimes with different rates of decrease after crack initiation. The first regime represents the nascent stages of crack growth, which corresponds to short-crack growth. In this regime the rate of decrease in the dynamic stiffness is relatively small since the crack growth rate is relatively slow. Once the crack reaches a critical size, the rate of decrease in dynamic stiffness increases significantly, which corresponds to a faster crack growth rate. The transition from a short-crack to a large crack response is predicted from an experiment with a strain amplitude of $4 \times 10^{-3}$ to commence when the crack length is ~ 18% of the micro-beam width.

In addition, two discontinuities are observed in Fig. 6(a) and can be explained as follows. The first discontinuity after ~ $1.6 \times 10^5$ cycles is a result of an adjustment of the driving frequency from 40 Hz to 45 Hz to account for the change in the resonance frequency of the system due to the drastic cyclic hardening. This guarantees that the phase angle is always close to 90°, which leads to
maximum sensitivity to changes in the micro-beam’s stiffness. A higher driving frequency results in higher velocities, and thus higher damping effects, which explains the jump in the dynamic stiffness. On the other hand, a second small discontinuity is observed after \( \sim 2.8 \times 10^6 \) cycles; this is commonly observed whenever the experiment is stopped then restarted at the same driving frequency and is due to the transient nature of the damped harmonic oscillators. Nevertheless, after a short period the overall shape of the dynamic stiffness is recovered.

To further quantify the hardening observed in the dynamic stiffness measurement, the high-frequency loading was interrupted regularly during an experiment with a strain amplitude of \( 3.97 \times 10^{-3} \) to conduct quasi-static loading and determine the normalized load-displacement hysteresis loops at different cycles. The hysteresis loops for select cycle numbers are shown in Fig. 7. Up to \( 1.1 \times 10^6 \) cycles, the mechanical response shows a dramatic increase in the peak load. The increase in the peak load between cycles \( 1.1 \times 10^6 \) and \( 2.9 \times 10^6 \) is much less significant but still noticeable. After this point, a crack nucleates and the peak load gradually decreases only on the side of the beam that corresponds to the crack being in tension, as shown in Fig. 7(b). When the crack is in compression, the peak load is almost unchanged since the crack is completely closed. As the crack propagates, the curve becomes more asymmetric with the peak load continuously decreasing until the micro-beam completely fractures.

Fig. 8 shows the surface morphology at different cycles for an experiment performed at a strain amplitude of \( 3.97 \times 10^{-3} \). No significant surface roughening is observed near the fixed end of the beam and the crack nucleates on the bottom of the beam and propagates until the beam is completely fractured. It is worth noting that two separate cracks nucleated during this experiment. One nucleates at approximately \( 2.5 \times 10^6 \) cycles but does not grow more than \( \sim 1 \mu m \) in length. A separate crack nucleates closer to the fixed end at \( 2.90 \times 10^6 \) cycles. This crack ends up propagating along the entire width of the beam and ultimately leads to failure.

4. Discussion

4.1. Mechanisms leading to the observed cyclic hardening/softening at different strain amplitudes

As shown by the surface morphologies in Fig. 5, there is significant plastic deformation accumulating over the course of the cyclic loading in all samples tested at a strain amplitude larger than \( 7 \times 10^{-3} \). In contrast, samples tested below this strain amplitude show very little surface roughening throughout the sample lifetime. Combining these observations with the gradual decline in the dynamic stiffness of the beam for high strain amplitude tests, one can surmise that the plastic deformation is likely accommodated by dislocation shearing of the \( \gamma' \) precipitates. This mechanism has been previously shown to be the main contributor to cyclic softening in ordered alloys [34,35] and is further supported by the following analysis.

The resolved shear stress required to shear a \( \gamma' \) precipitate by a superdislocation is [36]

\[
\tau_a = \frac{\Gamma_{APB}}{2b}
\]

where \( \Gamma_{APB} \) is the anti-phase boundary energy, and \( b = 2.52 \text{ Å} \) is the magnitude of the Burgers vector for Ni\(_3\)Al. It should be noted that Equation (7) is limited to the ideal case of a single infinitely-long
dislocation shearing through an infinitely-long precipitate and represents an upper bound of the actual value of $\tau_a$. Experimentally-determined values for $\Gamma_{APB}$ reported in literature and the corresponding predicted APB shearing stresses based on Equation (7) are indicated on Fig. 9 [37,38]. In addition, the maximum resolved shear stresses as predicted from the FEM simulations of the current experiments are shown in Fig. 9. In the current experiments the $\{111\}[10\bar{1}]$ slip system exhibits the maximum Schmid factor. Different markers are used to indicate whether cyclic hardening or softening was predominant during the sample life.

Comparing the predicted critical shearing stress in Fig. 9 with the critical resolved shear stress required to shear a precipitate, it would seem highly likely that the transition from cyclic hardening to cyclic softening can be attributed to stress required to shear precipitates in the superalloy when $\Gamma_{APB} < 0.2$ J/m². Nevertheless, given that the predictions from Equation (7) represent an upper limit and in real crystals $\tau_a$ can be lower [36], it can be argued that when the applied strain amplitude is larger than $7 \times 10^{-3}$ softening is expected due to precipitate shearing. When the applied strain amplitude is lower than this threshold, shearing is not expected and dislocation hardening would dominate due to dislocation interactions in the $\gamma$ phase and pile up at the $\gamma$/$\gamma'$ interface.

4.2. Crack initiation

All tested specimens contained no pre-existing cracks. The vast majority of the fatigue life for all tested samples was spent nucleating a crack and once the crack was present, the number of cycles to reach complete failure was relatively small. Understanding the micro-scale mechanisms that lead to crack initiation would provide great insight into quantifying the lifetimes of engineering materials.

The slip bands observed for strain amplitudes larger than $7 \times 10^{-3}$ are typical for cyclically loaded Ni-base superalloys [40]. Though these slip bands are distinct from persistent slip bands observed in single phase FCC metals, they still contain a characteristic ladder structure of edge dislocation dipoles [40]. A high concentration of vacancies are expected to generate from the annihilation of edge dipoles in these slip bands [41]. With the gradient in point defect concentration being the driving force, these vacancies diffuse to the interface between the slip bands and the matrix at the free surface, which subsequently results in the formation of intrusions that deepen with the increasing vacancy concentration. Observations in the present study supports this model. Fig. 10 shows the progression of slip bands during a micro-beam fatigue experiment with a strain amplitude of $7.85 \times 10^{-3}$. It is clear that the intrusions do not form until there are distinct extrusions. Presumably, the intrusions do not form until the dislocations have clustered and annihilated to form vacancies. Once the vacancies form and have enough time to diffuse to the matrix, intrusions form as seen in Fig. 10(c). These intrusions are very consequential in the context of fatigue crack initiation because they have been known to deepen, sharpen, and eventually form a crack [18]. The cracks forming from these intrusions are clearly apparent in the post-mortem SEM micrographs of the fractured surface shown in Fig. 11.

4.3. Fracture surface morphology

In all tested micro-beams, the dominant crack that leads to failure always initiated at or near the lower corner of the micro-beam since the corner leads to a stress concentration. Surface extrusions along the $\{111\}$ slip planes were also observed as preferred sites for crack nucleation in micro-beams that failed in less than $10^6$ cycles. In most tested micro-beams, failure was dominated by the propagation of the crack from the bottom corner vertically upward along the $(100)$ fixed end plane. However, in some cases the crack deviated from the fixed end plane and connected to a crack initiating along extrusions on the top surface, as shown by the morphology of the fractured surface in Fig. 11. In this experiment the strain amplitude was $8.28 \times 10^{-3}$ and the micro-beam completely fractured after $9.6 \times 10^4$ cycles. A combination of intrusions along the $(111)$ slip planes on the top surface and a crack approximately along the $(100)$ plane contributed to this failure. Fig. 11(a) reveals the texture of the fracture surface of the beam along its cross-section. Towards the top half of the micro-beam, the fracture surface is relatively smooth since the intrusions correspond to $(111)$ slip planes in the FCC crystal. Towards...

![Fig. 9. Calculated resolved shear stress values on the $(111)[10\bar{1}]$ slip system versus the number of cycles to failure for all tested cases in the current study. Dashed lines denote precipitate shearing stresses as calculated by Equation (7) for various values of $\Gamma_{APB}$.](image)

![Fig. 10. SEM micrographs of slip band formation near the fixed end of a cyclically loaded micro-beam with a strain amplitude of $7.85 \times 10^{-3}$ showing: (a) the early stages of slip band formation; (b) fully developed slip band extrusions; (c) intrusions alongside each slip band.](image)
the bottom half, the fracture surface is relatively rough since this surface does not follow any specific slip system.

The more oblique viewing angle in Fig. 11(b) clearly shows the intersecting slip traces from the (T11) and (111) planes on the top surface. The fracture surface at the top of the beam creates a vertex where the two intrusions intersect. Note also that the sides of the micro-beam show a significant amount of extruded material.

5. Conclusions

In this study, a new microfatigue testing methodology was developed to perform high cycle fatigue tests in situ on small volumes of material. Microfatigue experiments were conducted on single-crystal René-N5 Ni-base superalloy micro-beams at zero mean strain with strain amplitudes ranging from 3.97 × 10^{-3} to 1.98 × 10^{-2}. The method was shown to be able to probe fatigue lives ranging 5 orders of magnitude. The small-scale fatigue data reported here were observed to be superior to bulk superalloy fatigue studies, which can be attributable to an apparent size effect. Real time in situ SEM viewing allowed for observations of the cause and location of fatigue failures. By tracking the dynamic stiffness during cyclic loading, it was found that high displacement amplitude tests exhibited softening from the very start of the test until complete failure. Tests conducted below a strain amplitude of 7 × 10^{-3} cyclically hardened for the vast majority of the fatigue life until eventually a crack formed and caused failure. This is attributed to a transition from dislocation accumulation in γ channels to shear of the γ' precipitates as the dominant hardening and softening mechanisms, respectively. High strain amplitude tests show surface extrusions and slip traces forming near the fixed end. Cracks formed at stress concentrations as well as along surface extrusions. For low amplitude tests, two regimes of softening were observed possibly indicating a transition between short and large crack growth. Such observations could allow further characterization of the nature of short crack growth. In looking towards the future of studying crack nucleation, it is clear that further modifications to testing methodology would be needed to further characterize crack initiation and the nature of short crack growth. For example, the geometry of the test specimen can be redesigned to eliminate any sharp corners or notches that can lead to high stress concentrations. In addition, the stress gradient imposed by the bending loading could be mitigated by imposing a uniform stress field through tension/compression loading. This will lead to crack initiation not dictated by the stress distribution in the sample but purely by physical mechanisms inherent in the material. This would make it easier to draw further conclusions regarding the origins of crack initiation from in situ SEM experiments.

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References


