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The effect of inclusions on the high temperature low cycle fatigue performance of cast MarBN: experimental characterisation and computational modelling

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Abstract

The presence of inclusions is a known source of crack initiation and component failure in cast materials. In this work, the role of inclusions is investigated via a combined program of high temperature low cycle fatigue testing and computational modelling of a martensitic-ferritic steel, MarBN. Microstructural analysis has shown that manufacturing-induced oxide inclusions are a key source of fatigue crack initiation. A fully-coupled, critical-plane life prediction and damage model is implemented in a unified cyclic viscoplastic user-material subroutine and applied to predict damage and crack initiation for inclusions. It is deduced that more careful control of the development of inclusions in the manufacturing process will provide enhanced material and component performance for highly flexible and ultra-supercritical plant conditions.

Keywords: MarBN, fatigue crack initiation, high temperature low cycle fatigue, critical-plane, inclusions

Nomenclature

$b_i$, isotropic hardening constant; $C$, life prediction constant; $C_k$, kinematic hardening constant; $D$, damage; $d\Delta p$, iterative increment in accumulated effective plastic strain; $E$, elastic modulus; $k$, initial cyclic yield stress; $N_f$, cycles to failure; $\dot{\rho}$, accumulated effective plastic strain-rate; $Q_i$, isotropic hardening constant; $R$, isotropic hardening; $\alpha$, viscoplastic constant; $\beta$, viscoplastic constant; $\gamma_i$, kinematic hardening constant; $\Delta \rho$, accumulated effective plastic
1. Introduction

The rapidly increasing levels of adoption of renewable power generation has led to a reduction in the demand for electricity generated through burning of fossil fuels. The impact of this can be seen in the UK in particular, where coal production has declined by 52.5% to its lowest ever level in the past two years.\(^1\) In Europe, an agreement to increase the share of renewables to 27% by 2030 as a method of reducing CO\(_2\) emissions, as well as the global requirements of the Paris Agreement, will lead to further reductions in terms of fossil fuel dependence.\(^2\) This will have a significant impact on traditional base-load fossil fuel power plants, due to the high frequency of plant shut-downs and start-ups to accommodate the increased contribution from renewable energy sources.\(^3-5\) As a result, component performance is directly affected as complex operational cycles and more rapid load fluctuations lead to an increase in thermo-mechanical fatigue (TMF) and creep-fatigue (CF) degradation, and reduced component lifetime.\(^6,7\) Advanced 9Cr steels have been identified as a candidate material for next generation power plants, as an alternative to more expensive stainless steels and nickel-based superalloys\(^8,9\), but there remains a need to investigate their capability under flexible loading conditions.

MarBN is a martensitic-ferritic steel, originally developed to address the need for advanced materials with improved creep performance at elevated temperatures. Significant research into the effect of varying alloying elements, such as boron and nitrogen, indicates that strict control of such elements provides a thermally-stable microstructure for increased periods of time, relative to current 9Cr candidate steels (e.g. P91 and P92).\(^10\) The hierarchical microstructure of 9Cr steels contributes different strengthening mechanisms, including high angle grain boundary strengthening and solute and precipitate strengthening. Each feature acts as a barrier to dislocation motion, and, hence, reduces the rate of plastic deformation during loading. Maintaining this hierarchical structure for as long as possible is vital for improved component performance. Welded components are particularly important due to Type IV cracking of plant components, as the welding process results in the hierarchical microstructure being replaced with a heterogeneous substructure.\(^10-12\) MarBN has been shown to exhibit improved performance compared to P91 and P92 in terms of creep loading, while also maintaining a
hierarchical microstructure post-weld.\textsuperscript{13} The authors have also recently presented on the cyclic performance of cast MarBN, which is also critical for fitness-for-service assessment.\textsuperscript{14,15}

Inclusions occur in metallic alloys as a result of chemical reactions with the environment during manufacture and substantial financial resources have been invested in reducing such discontinuities in steels. The presence of inclusions leads to void formation during loading and results in premature cracking of components. The strength and toughness of steels, and benefits of microstructural refinement, are detrimentally affected, and increased plastic deformation and stress and strain localisation occur at the interfaces between inclusions and the matrix.\textsuperscript{16–21} These regions have been found to influence the direction of crack propagation, causing a crack to preferentially grow from the matrix towards an inclusion. It is important to understand the effect of inclusions and voids for various in-service loading conditions via experimental and computational methods.\textsuperscript{21–25}

In order to facilitate fitness-for-service of candidate next generation materials, a robust material model that can be calibrated and validated from relatively simple tests, and which can be applied to multi-axial component analysis and design, is required. Furthermore, in order to design against premature failure, a robust multi-axial fatigue damage methodology is required. The use of the critical-plane method as a post-processing tool has been widely implemented\textsuperscript{26–28}, but has not typically been applied for damage calculation within a continuum damage mechanics method. In this paper, we implement a critical-plane method in a user-material (UMAT) subroutine in Abaqus, in conjunction with Ostergren\textsuperscript{29} life prediction and a Chaboche\textsuperscript{30} damage law, coupled within a non-linear isotropic and kinematic hardening, cyclic viscoplasticity constitutive equation set. Calibration and validation is performed via comparison with high temperature low cycle fatigue (HTLCF) and CF tests. Transmission electron microscopy (TEM) of pre- and post-test samples allows evaluation of the effect of cyclic loading at the nano-scale, through examination of low angle boundary annihilation and dislocation motion. Scanning electron microscopy (SEM) and fractography of post-test samples have identified inclusions as key to fatigue crack initiation and propagation in MarBN under HTLCF and CF conditions. Based on this, the UMAT is applied to a matrix containing single and multiple spherical inclusions to identify the micro-scale effects on MarBN during cyclic loading, particularly in terms of damage accumulation and cycles to fatigue crack initiation.

2. Materials & Methods
2.1. Experimental Testing

An experimental test program of fully reversed (triangular), strain-controlled HTLCPF and CF testing was previously published for MarBN at 600 °C and based on this experimental data, material parameters were identified. 14,31 The material has been developed as part of an industry-academic collaborative UK government funded project, IMPACT 32 and heat treatment was performed at NUI Galway. In this work, a similar test program has been performed on MarBN at 650 °C, as per Table 1, to identify the effect of temperature on the cyclic response of the material. **Figure 1 presents an image of the sample geometry manufactured and tested at NUI Galway; a similar geometry is used for testing at GE Power.** HTLCPF testing at NUI Galway is performed using an INSTRON 8500 and a Denison Mayes Group cyclic test machine is used at GE Power (UK) with. An INSTRON 8800 controller is used in all cases. Strain-controlled CF testing is also performed, with a one hour strain-hold period in tension, to identify the effect of dwell periods on failure mechanisms and life. The macroscale crack initiation criterion is defined as a 20% drop in load after the first 150 cycles of testing.

2.2. Microstructural Analysis

After testing is completed, the samples are fractured at room temperature and sectioned using a low-speed saw. MarBN samples are analysed via TEM using two sample preparation methods; carbon replica and twin jet polishing. The carbon replica technique specifically allows the precipitate distribution to be analysed. For the production of thin foils, a portion of the MarBN material is polished to a finish of P4000, and a thickness of approximately 50 to 100 µm., A punch is used to extract a thin foil section and twin jet polishing is performed using a methanol-based etchant on 3 mm discs removed from the larger gauge length disc. TEM of carbon replica samples is performed using a Hitachi H7000 TEM and thin foil specimens are examined using a JOEL JEM-2100F TEM. For SEM analysis, samples are set in resin and polished, using a Buehler EcoMet 300 with Automet 250, to a 0.06 µm finish and etched with Vilella’s reagent. Analysis is performed using a Hitachi S2600N Variable Pressure SEM. Post-test MarBN samples are examined to identify mechanisms of strengthening and degradation.

2.3. Material Model

The constitutive behaviour of the material is defined using a unified cyclic viscoplastic material model with combined non-linear isotropic and kinematic hardening, as described in more detail by Barrett et al. 33 This has been implemented in an implicit UMAT subroutine in the commercial finite element code Abaqus 34 and modified in this work to include life prediction
and fatigue damage effects. A flowchart of the constitutive-damage model is presented in Figure 2. The plastic strain-rate tensor, $\dot{\varepsilon}^{pl}$ is defined as:

$$\dot{\varepsilon}^{pl} = \frac{3}{2} \alpha \sinh \beta \left( J_2 \left( \tilde{\sigma} - \chi \right) - R - k \right) \frac{\tilde{\sigma} - \chi}{\sigma_e}$$

(1)

where $\alpha$ and $\beta$ are the viscoplastic material constants, $\sigma_e$ is the equivalent stress, $\tilde{\sigma} = \frac{\sigma}{(1-D)}$ is the damaged stress tensor and $D$ is fatigue damage. The $\chi$ tensor is the kinematic back-stress accounting for the Bauschinger effect; isotropic softening behaviour is described by the scalar $R$, and $k$ is initial cyclic yield stress. The initial and later strain hardening stages are described through the use of two hardening terms in the kinematic hardening evolution model of Armstrong-Frederick$^{35}$, such that $\chi = \chi_1 + \chi_2$. The evolution equation for the Armstrong-Frederick model, including damage, is:

$$\dot{\chi}_i = C_i \dot{\varepsilon}^{pl} (1-D) - \gamma_i \chi_i \dot{\rho}$$

(2)

where $C_i$ is the hardening modulus, $\gamma_i$ is a recall parameter and $\dot{\rho}$ is the accumulated effective plastic strain-rate. Two Chaboche isotropic softening terms are used to simulate the primary and secondary stages of softening, such that $R = R_1 + R_2$. The evolution of $\dot{R}_i$ is defined as follows:

$$\dot{R}_i = b_i Q_i (1-D) \dot{\rho} - b_i R_i \dot{\rho}$$

(3)

where the rate of decay is controlled by $b_i$, and $Q_i$ is the saturated cyclic softening stress. The accumulated effective plastic strain increment, $\Delta \rho$, is defined as:

$$\Delta \rho = \Delta \rho + d\Delta \rho$$

(4)

where $d\Delta \rho$ is the iterative increment in accumulated plastic strain, defined as:

$$d\Delta \rho = \frac{\varphi - \Delta \rho}{\Delta t}$$

(5)

$$\varphi = \alpha \sinh \beta \left( \sigma_e^{tr} - 3G\Delta \rho - R - k \right)$$

(6)
and

\[ Z = \alpha \beta \cosh \beta \left( \sigma_{e}^{tr} - 3Gp - R - k \right) \]  \hspace{1cm} (7)

where \( \sigma_{e}^{tr} \) is the effective trial stress\(^{36} \) and \( G \) is shear modulus. Life prediction is performed based on the critical-plane approach for multi-axial geometries with the Ostergren equation.\(^{26–28} \)

\[ N_f (N) = C \left[ \max_{n} \left( \Delta \varepsilon_{n}^{pl} (N) \sigma_{\text{max},n} (N) \right) \right]^\delta \]  \hspace{1cm} (8)

where \( N_f \) is number of cycles to failure, \( N \) is cycle number and \( C \) and \( \delta \) are material parameters. The maximum with respect to the plane orientation, \( \mathbf{n} \), is identified for the maximum stress, \( \sigma_{\text{max},n} \), and plastic strain-range, \( \Delta \varepsilon_{n}^{pl} \), over a cycle. The cycle time is defined in the UMAT based on (a) the time for initial fatigue loading in tension and (b) the time for one cycle, within which the maximum and minimum stress and strain components are identified and stored as state variables. The critical-plane type approach involves calculation of the direction cosines for a range of candidate planes, spanning a 180° half-space in 10° increments, with the normal stresses and strains calculated on each candidate plane. The direction cosines are given by:

\[ n_1 = \sin \theta \sin \theta_R \]
\[ n_2 = \sin \theta \cos \theta_R \]
\[ n_3 = \cos \theta \]  \hspace{1cm} (9)

where \( \theta \) and \( \theta_R \) are two angles which uniquely define the candidate planes.\(^{27} \) The associated normal stress and strain are given by:

\[ \sigma_n = \sigma_{11}n_1^2 + \sigma_{22}n_2^2 + \sigma_{33}n_3^2 + 2\tau_{12}n_1n_2 + 2\tau_{23}n_2n_3 + 2\tau_{31}n_3n_1 \]  \hspace{1cm} (10)

\[ \varepsilon_n = \epsilon_{11}n_1^2 + \epsilon_{22}n_2^2 + \epsilon_{33}n_3^2 + \gamma_{12}n_1n_2 + \gamma_{23}n_2n_3 + \gamma_{31}n_3n_1 \]

This method allows calculation of a number of cycles to failure, via Equation 8, for the identified critical Ostergren product. In this paper, the approach adopted to accumulate damage is to assume a Chaboche non-linear evolution\(^{30} \) based on the current prediction of cycles to failure, \( N_f (N) \):
\[ D = 1 - \left[ 1 - \left( \frac{N}{N_i(N)} \right)^{\frac{1}{\phi_1}} \right] \]  

where \( \phi_1 \) and \( \phi_2 \) are damage constants. The use of an incremental damage term,

\[ \frac{dD}{dN} = \frac{D(N)}{N_f} \left( 1 - \frac{N}{N_f} \right)^{\frac{1}{\phi_1}} - N \delta \left( C \left( \frac{d}{dN} \Delta \varepsilon_{pl} \right) \sigma_{max} + C \left( \frac{d}{dN} \sigma_{max} \right) \Delta \varepsilon_{pl} \right) \left( \frac{1}{N_f C \Delta \varepsilon_{pl} \sigma_{max}^{\delta}} \right) \]

was investigated and compared to the method described above. Almost identical damage evolution is predicted by both methods and based on this, the former method is used here to reduce computational expense and provide more rapid output of results for larger geometries.

### 2.4. Material Parameter Identification

The complete constitutive and damage model requires identification of 16 material parameters; Young’s modulus, \( E \), Poisson’s ratio, \( \nu \), four isotropic (\( b_i \) and \( Q_i \)) and kinematic (\( C_i \) and \( \gamma_i \)) hardening parameters, two viscoplastic constants, \( \alpha \) and \( \beta \), and four life prediction (\( C \) and \( \delta \)) and damage (\( \phi_1 \) and \( \phi_2 \)) constants. Initial identification of the cyclic and viscoplastic material parameters follows the method described by O’Hara et al.\(^{14,31} \) However, these initial values are then optimised using the Levenberg-Marquardt algorithm, as part of the \textit{lsqnonlin} non-linear optimisation toolbox in MATLAB, in conjunction with a uniaxial sinh model implementation in MATLAB to predict the cyclic viscoplastic and stress-relaxation response of MarBN at 650 °C. The overall error is minimised with respect to (i) maximum stress evolution, (ii) cyclic response at initial and half-life cycles and (iii) stress-relaxation data. The resulting final identified cyclic viscoplastic material parameters for MarBN at 650 °C are shown in Table 2. Identification of the life prediction constants is performed via plotting the Ostergren product \( \left( \Delta \varepsilon_{pl}^{n} \sigma_{max,n} \right) \) against cycles to failure for a set of fatigue tests across a range of strain-ranges. They have been identified as \( C = 167.3 \) and \( \delta = -0.69 \) at 600 °C, and \( C = 152.8 \) and \( \delta = -0.72 \) at 650 °C, respectively. The damage constants are identified from uniaxial HTLCF data, as presented previously by O’Hara et al.\(^{14} \)

### 3. Results

#### 3.1. Experimental Testing
Figure 3(a) shows the low-cycle fatigue strain-life plot for cast MarBN at 600 °C and 650 °C. There is a small reduction in fatigue life with an increase in temperature. In Figure 3(b), the evolution of maximum stress as a function of cycles is examined, including MarBN under fatigue loading, as-received (AR) rolled P91 (\( \dot{\varepsilon} = 0.03 \% / s \) and \( \Delta \varepsilon = \pm 0.5\% \)) and MarBN under CF loading (one hour hold period, \( \dot{\varepsilon} = 0.1 \% / s \) and \( \Delta \varepsilon = \pm 0.5\% \)). The maximum stress evolution and cyclic softening of MarBN at 650 °C is found to be almost the same for P91 at 600 °C. The additional effect of creep is seen to be detrimental to life and cyclic strength. Although the results presented for CF loading are at a higher strain-rate, the material response has been shown previously to be very similar under pure fatigue conditions to the strain-rate presented here.\(^{14}\) In Figure 3(c) to Figure 3(e), the cyclic evolution of MarBN (600 °C and 650 °C) and P91 (600 °C) is presented for the initial, half-life and final cycles. MarBN at 600 °C has a significantly increased cyclic strength compared to P91 at 600 °C, with comparable constitutive performance at 650 °C for the duration of the test.

### 3.2. Microstructural Analysis

In Figure 4(a) and Figure 4(b), TEM of MarBN is presented for AR and post-test carbon replica samples, and the hierarchical microstructure with carbides distributed along boundaries can be seen. A histogram of the Feret diameter is measured in Figure 4(c), whereby the images are converted to a binary format and watershed segmentation is applied to identify individual particle dimensions using image processing software ImageJ.\(^{37}\) The frequency of particles post-test is found to be approximately double that for pre-test. In Figure 4(d) and Figure 4(e), low angle boundary annihilation and dislocation motion in post-test thin foil MarBN samples are identified, indicating the effect of cyclic loading on the microstructure. Figure 5 shows the fracture surfaces of the MarBN fatigue and CF samples. Inclusions can be seen in regions of cracking, as well as fatigue striations. Ductile dimples can be seen on the fatigue sample (Figure 5(c)) and cleavage facets (Figure 5(d)) are observed on the CF sample, with voids around inclusions in both cases. Figure 6(a – c) shows the effect of spherical (MnS) and non-spherical (Ca-Al-O) inclusions on crack initiation and propagation in MarBN. In Figure 6(d) and Figure 6(e), images of the sectioned gauge length of fatigue and CF samples are shown and secondary cracking and oxidation of the internal cracks is observed. The tensile hold period allows deeper penetration of the oxide within the sample and inclusions are again shown to influence crack initiation and propagation, as well as oxide pit formation. Backscatter electron (BSE) SEM has been used to identify Laves phase in tungsten containing steels\(^{38-40}\), due to the high atomic weight of tungsten resulting in a high contrast between the particles and matrix. It is conjectured
that regions of Laves phase have formed along grain boundaries in the post-test MarBN samples as a result of the high temperature loading. Thus, the significant increase in particle density observed via TEM (Figure 4(c)) is attributed here to Laves phase formation.

### 3.3. Material Modelling

Figure 7(a) shows calibration of the material parameters using MATLAB for MarBN at 650 °C, in terms of the maximum stress and plastic strain evolution with cycles. Validation is shown in Figure 7(b) and (c) at different loading conditions (strain-rates and ranges) to those used for calibration for the initial and half-life cycles. A single-element 2D axisymmetric model, with axisymmetric stress elements (CAX4), is used for calibration and validation of the UMAT under uniaxial cyclic loading conditions across a range of strain-rates and strain-ranges at 600 °C and 650 °C. The UMAT predicts almost identical results to the uniaxial MATLAB code used for calibration and validation of the material parameters. Figure 8 shows a comparison of the UMAT-predicted critical-plane lives for MarBN at a range of loading conditions and temperatures against measured lives.

In order to investigate the micro-scale effects of inclusions on damage and cracking, the cyclic viscoplastic damage model is applied to micro-scale models of single and multiple inclusions in a MarBN matrix. The single inclusion model assumes a spherical inclusion using a 2D axisymmetric approach and axisymmetric stress elements (CAX4), with a 30 µm diameter inclusion in the matrix, as described previously by O’Hara et al. The material properties of the inclusion are obtained from experimental measurements by Melander et al.; a harder inclusion type was chosen as inclusion cracking is not observed experimentally. Frictionless, hard contact is defined between the matrix and inclusion and to accurately model decohesion, separation between the inclusion and matrix is allowed after contact. Figure 9 shows the predicted effect of strain-range around the single inclusion in MarBN at 650 °C in terms of the maximum principal strain distribution after 200 cycles, respectively, with similar results at 600 °C. Ratchetting is predicted to occur as strain accumulates in the region of the inclusion at different rates, depending on the applied strain-range. Increased decohesion can also be seen at the higher loading condition. In Figure 10, the evolution of damage with cycles is presented for an applied strain-range of ±0.5%. Figure 11 shows a comparison of the model predicted number of cycles to micro-crack initiation around the inclusion against the measured total life. Figure 12 depicts a multi-inclusion 2D geometry, based on the SEM image of a sectioned HTLCF sample post-test (Figure 6(a)), modelled using the same conditions as applied to the
single inclusion model, at both 600 °C and 650 °C. **A combination of 3-node (CPE3) and 4-node (CPE4) plane strain elements are used.** The inclusions are assumed to be circular and the influence of out-of-plane inclusions, that may affect the cracking behaviour, is neglected. Figure 13 presents the predicted distribution of von Mises stress, maximum principal strain and damage at 40 cycles, focussing on the inclusions. Stress and strain concentrations can be seen around each inclusion causing an increase in damage in this region. Figure 14 examines the effect of zero strain, compression and tension, in terms of the maximum principal strain distribution and decohesion of the inclusion from the matrix, around inclusion I1 at 600 °C and 650 °C after 40 cycles.

4. Discussion

A program of HTLCF testing is presented for a range of loading conditions at 650 °C to characterise the cyclic behaviour of MarBN steel, with comparisons to the widely used material, P91. Microstructural analysis is performed to investigate the effect of cyclic loading and a hold period on MarBN. Laves phase particle formation has been identified and inclusions are found to heavily influence crack initiation and propagation. Multiaxial modelling of MarBN at 600 °C and 650 °C is performed to further understand the effect of inclusions on the microscale behaviour of the material under cyclic loading.

The HTLCF response of MarBN at 650 °C (Figure 3(a)) indicates that there is little reduction in cycles to failure compared to 600 °C. As discussed previously,14,31 MarBN at 600 °C has a significantly higher cyclic strength than P91, with comparable performance at 650 °C, as shown in Figure 3(b); this is attributed to thermally stable boron-enriched M23C6 carbides along boundaries, providing barriers to dislocation motion, and enhanced W strengthening. However, CF loading is found to reduce the cyclic strength and fatigue life of the material; this is attributed to the introduction of the hold period.42 MarBN and P91 exhibit similar rates of cyclic softening, particularly in the early stages of loading, with an increased rate due to the addition of a hold period and creep of the material. Cyclic softening is a known phenomenon in 9-12Cr steels and is attributed to dynamic recovery of the microstructure. This involves a reduction in dislocation density, and low angle boundary annihilation due to dislocation annihilation, resulting in lath coarsening, the extent to which depends on temperature and strain amplitude. Therefore, even small-amplitude cycling of plant components (e.g. warm starts, hot starts or fluctuations) can cause microstructural degradation and reduce strength.7,43,44
The effect of cyclic loading on the nano-scale mechanisms of degradation in MarBN has been examined in this work via TEM, as per Figure 4. Dislocation motion and low angle boundary annihilation have been observed in both carbon replica and thin foil samples; these are characteristic features contributing to cyclic softening. In terms of micro-scale mechanisms of degradation, inclusions have been identified as playing a key role in crack initiation under both fatigue and CF loading (Figure 5(a) and (b)). This is further promoted when decohesion of the inclusion from the matrix occurs; sufficiently large inclusions, in conjunction with clustering, are found to have detrimental effects under cyclic loading. The size, shape, location and distribution of such inhomogeneities are all key factors, with increased likelihood of cracking closer to the surface.\textsuperscript{45,46} Fatigue striations are also observed in these regions, indicating the rate at which the crack propagates through the material. The main difference between the fatigue and CF fracture surfaces is the presence of cleavage facets and cracking between adjacent crystals as a result of CF loading (Figure 5(c) and (d)). The density of cracks, both at the surface and internally, is found to increase due to the tensile hold period. Ductile dimples are observed on both samples, due to localised plastic strain and dislocation accumulation, and void growth is observed around inclusions, as decohesion from the matrix occurs during loading.

Sectioned images of these samples (Figure 6) provides further evidence of the role of inclusions in fatigue crack initiation and propagation. The shape of inclusions, as per Figure 6(a), indicates that complex-shaped Ca-Al-O inclusions have a greater influence on the cracking behaviour of MarBN under cyclic loading, compared to spherical MnS inclusions. The effect of inclusions in the relatively small samples examined here is likely to be much greater than for thick section components in real plant, particularly as the loading conditions examined in this work are also much higher than for typical plant. Nevertheless, it can be inferred that improved control of inclusions and voids during the manufacturing process would further improve the cyclic response of the material and be advantageous for more prolonged life. The size of secondary cracks in the CF sample (Figure 6(e)) is larger than for fatigue loading alone (Figure 6(d)). Internal oxidation of secondary cracks initiating from the gauge length can be seen, as well as oxide pit formation around inclusions. Inclusions are sites of high strain and dislocation accumulation, and appear to promote oxide pit formation; therefore, the likelihood of crack initiation and propagation in such regions of weakened matrix material is increased.

Evidence of Laves phase particles (Fe\textsubscript{2}W) along boundaries has also been observed in MarBN (Figure 6(d) and Figure 6(e)), as a result of the high temperature cyclic loading. These are
observed close to the surface and near regions of cracking and oxidation; the high concentration of W in these particles, which commonly grow along grain boundaries, indicates that absorption into the oxide layer would result in reduced strengthening. The precipitation hardening effect of boron-enriched M23C6 carbides along boundaries is considered to be the primary strengthening mechanism in modified 9Cr steels, and is further enhanced through the addition of W47,48; however, Laves phase particles (Fe2W) are prone to coarsening during prolonged high temperature exposure40. In P92 steel, these particles have been identified as significant contributors to precipitate strengthening, as particles reach a similar saturated size to M23C6 carbides in less than 10,000 hours.49,50 This may be an indication as to why the cast MarBN examined here has equivalent cyclic performance and fatigue life, compared to a rolled P91 steel. The saturated particle size of Fe2W in cast MarBN, as a result of HTLCF loading, cannot be concluded from this work alone, but presents an interesting focus for future work on the possible effects of Laves phase in modified 9Cr steels.

The improved strength and similar softening behaviour of MarBN in relation to P91 is a significant result for the material in terms of implementation in highly flexible plant operation at further elevated temperatures. MarBN currently satisfies the creep weld strength reduction criterion for operation at 650 °C13; hence, a logical next step is the identification of an optimised weld metal for further cyclic and CF testing. However, in terms of the parent material, initial results look promising for application in ultra-supercritical plant conditions, particularly with the manufacture of forged and rolled components presumably adding to current fatigue performance.

To describe the response of such materials at high temperature, a modified unified cyclic viscoplastic model with damage and multi-axial life prediction has been developed. The hyperbolic sine flow rule allows the strain-rate effect at high temperature to be predicted from a single set of material parameters (Figure 7), as well as allowing extrapolation to more representative plant loading conditions (e.g. lower strain-rates). The damage model does not require a damage initiation threshold as implemented in alternative models51,52 and the material parameters required to predict cycles to failure and damage can be identified from the existing test data for calibration and validation of the cyclic viscoplastic material parameters. This model has been shown in Figure 8 to accurately predict the uniaxial fatigue life of MarBN at 600 °C and 650 °C, generally within ±15%.
The ability to accurately model inclusions provides a greater understanding of what causes crack initiation and failure around these discontinuities. Figure 9 depicts the predicted maximum principal strain distribution around a single inclusion at the centre of a MarBN matrix, across different applied strain-ranges at 200 cycles. Substantial strain accumulation, due to ratchetting, and strain redistribution is predicted to occur as the strain-range is increased. A strain concentration factor, defined here as the ratio of maximum local plastic strain-range to nominal applied strain-range after 200 cycles, is approximately 2.5 in all cases. The evolution of damage up to 200 cycles is examined in Figure 10. Damage is predicted to initiate at the root of the inclusion-matrix interface and propagate perpendicular to the primary loading direction. A number of factors influence the complex distribution of damage around the inclusion, namely (i) the distribution and accumulation of strain, varying during tension and compression loading, (ii) shear band formation, (iii) cyclic softening and (iv) decohesion. Similar results are predicted at lower strain-ranges, but the area over which damage occurs is reduced. Micro-crack initiation is predicted to occur in less than 150 reversals in all cases (Figure 11). There is little difference in the predicted behaviour of the material at 600 °C and 650 °C; the main difference is a reduced maximum stress and an increase in plastic strain at higher temperatures, ultimately resulting in similar predictions for cycles to micro-crack initiation.

The same model was run as a 2D planar model, with plane strain elements, as a comparison to the axisymmetric model. Increased stresses and strains were predicted to occur, spanning a larger region around the inclusion, as well as throughout the matrix. This led to a more conservative prediction of number of cycles to micro-crack initiation. Similar results have been observed elsewhere\textsuperscript{33}, highlighting the importance of three-dimensional effects in more accurately predicting the localised micro-mechanisms of degradation around discontinuities. Current work is focussed on more realistic 3D characterisation of inclusions and future work will examine the associated effects on predicted stress and strain distributions, and hence, on predicted fatigue crack initiation in 3D geometries containing single and multiple inclusions; however, the current methodology indicates the significant localisation effect of inclusions in terms of stress and strain accumulation.

Extensive secondary cracking (greater than 0.5 mm) is observed in a post-HTLCF test MarBN sample around multiple inclusions (Figure 12(a)) and although SEM only provides a 2D slice of the sample, it is a valuable tool in understanding the influence of such discontinuities in the absence of 3D analysis methods. 2D material modelling of a similar geometry (Figure 12(b))
is performed to provide a greater insight into the influence of inclusions, decohesion, clustering and location. The high stress and strain concentrations (Figure 13(a) and (b)) around each of the inclusions result in significant damage accumulation (Figure 13(c)). Figure 13(a) and Figure 13(c) predict interaction between inclusions I₁ and I₃ in terms of stress and damage distribution. The close proximity of I₁ to I₂ leads to a further increase in stress and strain concentrations in this region at 600 °C. The stresses are reduced in the model at 650 °C, but the strains are further increased due to a higher rate of ratchetting, resulting in a similar damage distribution that both propagates towards the surface and predicts a link between inclusions I₁ and I₂. Experimentally, significantly longer crack propagation is observed around inclusion I₃ after nearly 850 cycles. This may be due to the complex shape of the inclusion and interaction between out-of-plane inclusions in the matrix indicating the significance of shape on crack behaviour.

The effect of different loading cases on inclusion I₁ (Figure 14) predicts that, under zero strain after 40 cycles, decohesion of the matrix perpendicular to the primary loading direction will produce strain accumulation and redistribution. Although, quantitatively, similar predicted concentrations are observed for compression and tension loading (Figure 14(b) and (c)), the levels of strain and decohesion are significantly greater under tension, slightly more so at 650 °C, as the material becomes more ductile. The predicted levels of strain accumulation are substantially higher than for the single inclusion case, exceeding the experimentally measured uniaxial failure strain (18%) in all cases after 40 cycles, indicating the effects of location and clustering of inclusions in a matrix. This also leads to increased void formation and reduced effective matrix area capable of withstanding damage. Similar trends around inclusions I₂ and I₃ indicate the detrimental interaction effects of a limited number of inclusions, with dimensions even as small as 20 µm.

5. Conclusion

The main conclusions are:

- **Under high temperature low cycle fatigue strain-controlled loading conditions, cast MarBN is shown to have comparable cyclic strength at 650 °C compared to rolled P91 at 600 °C, demonstrating the capability of the material to endure a 50 °C increase in operating temperature for equivalent cyclic strength.**

- **Characteristic cyclic softening features e.g. low angle boundary annihilation and lath widening, are observed at the nano-scale; however, inclusions distributed throughout**
the cast material have been identified as the primary cause for crack initiation and sample failure under cyclic loading.

- A finite element critical plane fatigue life and damage accumulation methodology was successfully validated with respect to strain-rate and strain-range for MarBN under high temperature low cycle fatigue loading conditions. The method was then applied to investigate fatigue damage at inclusions in cast MarBN, leading to predicted cycles to micro-crack initiation of approximately 5% of total fatigue life.

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**References**


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Tables

Table 1. HTLCF test program for MarBN at 650 °C.

<table>
<thead>
<tr>
<th>Test Type</th>
<th>Strain-rate (%/s)</th>
<th>Strain-range (%)</th>
<th>Waveform</th>
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<tr>
<td>HTLCF</td>
<td>0.1</td>
<td>±0.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.03</td>
<td>±0.5, ±0.4, ±0.3</td>
<td>( R_e = -1 ) (Triangular)</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>±0.5, ±0.4, ±0.3</td>
<td></td>
</tr>
<tr>
<td>CF</td>
<td>0.1</td>
<td>±0.5</td>
<td>1 hr hold period</td>
</tr>
</tbody>
</table>

Table 2. MarBN cyclic viscoplastic material parameters at 650 °C.

<table>
<thead>
<tr>
<th>( E ) (GPa)</th>
<th>( k ) (MPa)</th>
<th>( \alpha ) (s(^{-1}))</th>
<th>( B ) (MPa(^{-1}))</th>
<th>( Q_1 ) (MPa)</th>
<th>( b_1 ) (MPa)</th>
<th>( Q_2 ) (MPa)</th>
<th>( b_2 ) (MPa)</th>
<th>( C_1 ) (MPa)</th>
<th>( \gamma_1 ) (MPa)</th>
<th>( C_2 ) (MPa)</th>
<th>( \gamma_2 ) (MPa)</th>
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</thead>
<tbody>
<tr>
<td>133.1</td>
<td>50.0</td>
<td>( 3.8e^{-7} )</td>
<td>( 4.4e^{-2} )</td>
<td>-70.9</td>
<td>4.8</td>
<td>-100.1</td>
<td>0.2</td>
<td>181,747.0</td>
<td>2,428.9</td>
<td>40,912.3</td>
<td>545.9</td>
</tr>
</tbody>
</table>

Figures

Figure 1. HTLCF specimen geometry for testing at NUI Galway. All dimensions are in millimetres.
Figure 2. Flowchart for implicit implementation of the unified hyperbolic sine cyclic viscoplastic model with life prediction and fatigue damage in a UMAT.
Figure 3. (a) Strain-life plot for cast MarBN at 600 °C and 650 °C at various strain-rates and strain-ranges, (b) comparison of the maximum stress evolution for MarBN and P91 under pure fatigue (\(\dot{\varepsilon} = 0.03 \% / s\)) and creep-fatigue (CF) loading (\(\dot{\varepsilon} = 0.1 \% / s\)) at an applied strain-range of \(\Delta \varepsilon = \pm 0.5\%\), and cyclic evolution for the (c) initial, (d) half-life and (e) final cycles.
Figure 4. TEM of carbon replica samples of (a) as-received MarBN and (b) post-fatigue test of MarBN (red arrows denote lath boundaries), (c) a histogram of the most frequent Feret diameter of particles pre- and post-test, and (d, e) TEM images of thin foil MarBN post-fatigue test at 600 °C (dashed circles are M23C6 carbides and arrows are pinned dislocations within a lath).
Figure 5. SEM images of the fracture surface of post-HTLCF MarBN samples under (a, c) fatigue and (b, d) creep-fatigue loading. Inclusions and voids (a, b, red circles), and ductile dimple (c) and cleavage facet (d) formation is observed (red arrows).
Figure 6. Secondary cracking in a post-HTLCF MarBN sample highlighting (a, b, c) the effect of non-spherical inclusions along the gauge length. Secondary cracking due to (d) fatigue and (e) creep-fatigue loading with regions of dense Laves phase formation (red arrows). Oxide layer formation is also observed.
Figure 7. (a) Material parameter calibration and (b, c) validation at the initial and half-life cycles, for MarBN at 650 °C. The black line represents the model and the symbols correspond to the experimental data.

Figure 8. Predicted reversals to failure versus experimental data for MarBN at 600 °C and 650 °C under uniaxial cyclic loading.
Figure 9. Maximum principal strain contour plots for MarBN across different applied strain-ranges at 650 °C, at a strain-rate of 0.1 %/s up to N = 200. Inset is the stress-strain evolution in the direction of loading at the location of maximum principal strain.
Figure 10. Contour plots of damage evolution with increasing cycles for MarBN at 650 °C, (\( \dot{\varepsilon} = 0.1 \%/s, \Delta \varepsilon = \pm 0.5\%, \) and \( N = 200 \)).

Figure 11. Comparison of model predicted cycles to fatigue crack initiation (FCI) at an inclusion, compared to the bulk experimental failure (Figure 3(a)) for various applied strain-ranges.
Figure 12. (a) SEM image of secondary cracking as a result of inclusions in a post-fatigue test MarBN sample and (b) 2D Abaqus model.
Figure 13. (a) Von Mises stress, (b) maximum principal strain and (c) damage distribution around inclusions in MarBN at 600 °C (left) and 650 °C (right) after 40 cycles.
Figure 14. Maximum principal strain distribution around inclusion $I_1$ at 600 °C and 650 °C under (a) zero strain, (b) compression and (c) tension loading at $N = 40$. 