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Slip localization and fatigue crack nucleation near a non-metallic inclusion in polycrystalline nickel-based superalloy

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ABSTRACT

Fatigue crack nucleation at a non-metallic agglomerate inclusion has been studied by high spatial resolution Digital Image Correlation (HR-DIC) and high angular resolution Electron Backscatter Diffraction (HR-EBSD). Spatial and temporal characterization and correlation of deformation with underlying microstructures has been performed, with distributions of plastic strain measured from HR-DIC; and residual stress and density of geometrically necessary dislocations (GND) measured from HR-EBSD. Initial residual stress and GND fields, as a consequence of differing thermal expansivities in the metallic and oxide phases, localized around the agglomerate have been quantified using HR-EBSD. The localization of the pre-existing stress and dislocation states appear to lead to early onset of plasticity upon subsequent mechanical loading. Heterogeneous distributions of plastic strain have been observed in the course of the fatigue test by HR-DIC. Crack nucleation via agglomerate/nickel interface decohesion and particle fracture has been demonstrated and this is correlated with the elevation in strain and dislocation density. The measurements of residual stress, strain, and dislocation density provide key information for the mechanisms of fatigue cracking and the development of damage nucleation criteria in these material systems.

1. Introduction

Understanding fatigue life in metallic components used in gas turbine engines is very important so that effective maintenance schedules can be developed, reducing costs and improving safety. In the engine, polycrystalline nickel based superalloys are widely used for turbine disk applications due to their superior mechanical properties and corrosion resistance at high temperatures. The presence of fine γ grains along with a multi modal distribution of γ' renders this class of nickel alloys exceptional properties. Evolution of these alloys has involved careful control of chemistry for a wide variety of chemical constituents and this has driven wrought disk components to be manufactured via powder metallurgy routes. This processing route can lead to the introduction of small non-metallic inclusions, despite best efforts to ensure powder cleanliness. These non-metallic inclusions result in degradation in the mechanical properties and understanding their effects on microstructural crack nucleation and short crack growth is very important [1].

Extensive analyses have been carried out to understand the effects of such defects on fatigue crack nucleation and growth in superalloys under different loading conditions and in various microstructures [2,3]. However, these experimental observations, albeit comprehensive, do not fully provide a physically sound description of microstructurally sensitive damage initiation and evolution associated with the inclusions. This is complicated further as these inclusions are formed in different manufacturing intervals, and have various chemical compositions, morphologies, and neighboring metallic matrices [2,4]. This variance challenges the predictive capability of damage nucleation models and therefore the associated damage process needs to be explicitly addressed using more sophisticated techniques.

The presence of these inclusions, often oxides, in a metallic matrix results in a multiphase material in which damage usually occurs via particle fracture or inclusion/matrix interface decohesion or a mixed mode, depending on the strength of interface, fracture toughness of oxide particle, and plastic response of the matrix [5–7]. This problem is complex and development of physically-based damage nucleation criteria requires microstructurally sensitive characterization of local damage, including plastic and elastic strain distributions, and dislocation content in the vicinity of the inclusion. These quantities evolve during cyclic deformation and lead to crack formation. This necessitates collection and evaluation of high fidelity, high resolution structural

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data over time.

Local strain evaluation can be performed through direct measurement of plastic strains via Digital Image Correlation (DIC), as well as elastic strains and plastic strain gradients via high angular resolution Electron Backscatter Diffraction (HR-EBSD). These techniques provide complementary, but different, components of the deformation and can be used to inform physical models.

DIC compares two or more images of progressively deformed microstructures to measure surface displacement fields which can be interrogated to measure strains. Typically the displacement resolution and subset size limit this technique to measurements of plastic strain fields [8]. DIC has been used to investigate damage nucleation associated with microstructures and has been conducted in Ti alloys [9,10], as well as nickel based superalloy Hastelloy X [11], and recent developments of the technique are reviewed in [8]. Digital Image Correlation follows surface displacements using images and therefore it is only limited by the imaging quality and patterning size, making it suitable for strain measurement at small scales [12]. In practice, strain resolution is limited by marker size, distribution and contrast within the cross-correlated subsets; and spatial resolution is limited by marker size and imaging mode. Correlation of measured strains with EBSD orientation mapping enables evaluation of the effect of microstructure on strain patterning to be undertaken [11]. However, the total plastic strain field is not the only factor that controls crack nucleation and short crack growth and therefore additional techniques are required.

High angular Resolution, cross correlation based, Electron Backscatter Diffraction (HR-EBSD) provides a powerful tool for analyses of residual elastic strain (i.e. stress), lattice misorientation and estimates of the stored geometrically necessary dislocation (GND) density. A recent review provides details of the method [13] and here we describe the method only briefly. This technique measures the relative misorientation and elastic strain state between two or more EBSD patterns. The shifts between zone axes are measured using cross correlation with very high precision and related to the displacement gradient tensor [14,15] with a sensitivity of $\sim 1\times10^{-4}$ in strain and $\sim 1\times10^{-4}$ rad in rotation [15]. Lattice curvatures from maps of these fields can be calculated and used to generate stored GND content [16].

Recent examples of application of the HR-EBSD technique include the study of GND evolution in Ti–6Al–4V deformed in tension and under cyclic loading [17,18], slip band-grain boundary interaction in commercially-pure titanium [19,20], and distribution of intragranular residual stresses and GND density in monotonically deformed copper [21–23]. These fields have been used to compare with high fidelity finite element models, including indents in titanium [24]. More recent work on a directionally solidified and a polycrystalline nickel superalloy has shown that the high resolution EBSD determined thermal residual strains and geometrically necessary dislocations are in good agreement with crystal plasticity finite element predications [16,25–27]. Unfortunately it is only possible to measure the relative deformation state between points within the same grain using HR-EBSD. This has motived a recent study that has involved direct comparison of measurements and crystal plasticity simulations of a thermally induced deformation around an inclusion using the inverse referencing methodology [26].

The present work investigates a polycrystalline nickel base superalloy with a non-metallic agglomerate inclusion surrounded by large nickel grains. The cyclic evolution of strains and dislocation densities are analyzed both qualitatively and quantitatively using HR-EBSD and HR-DIC and correlated with key microstructural features. The results are discussed and provide fundamental information for the development of defect nucleation criteria.

2. Experimental

2.1. Material and sample preparation

A three-point bend cyclic test was conducted on a polycrystalline nickel superalloy (RR1000) sample provided by Rolls-Royce plc. The alloy has a nominal composition as listed in Table 1 [28] and was produced via a powder metallurgy (PM) route. Following extrusion and forging, the alloy was heat treated at a sub-solvus temperature of 1393 K for 4 h and air quenched. This gave rise to the formation of a fine dispersion of intragranular γ' precipitates at γ phase boundaries. The material was subsequently aged at 1033 K for 16 h. This heat treatment process resulted in a fine grain microstructure and multi-modal distribution of γ' precipitates with increased low cycle fatigue properties. An optical image of the alloy is shown in Fig. 1, together with an orientation EBSD map.

During the PM processing, contact and reactions between the lining materials and the molten alloying elements are inevitable and are considered to be the source for non-metallic inclusions [2,4]. In heat treated forgings, these inclusions can be surrounded by large γ grains, as seen in Fig. 1(a) and (b).

The as-received specimen was cut down to a rectangular bar with dimensions of $12.70\times1.94\times3.50\ \text{mm}^3$ as shown in Fig. 2(a) based on the loading capacity of the three-point push–push reversible loading rig. Metallographic grinding and polishing were conducted to obtain a surface finish for scanning electron microscopy (SEM). A final light polish with colloidal silica for 15 min was performed to achieve the required surface quality for EBSD. The sample was carefully machined to locate the agglomerate inclusion at the bottom of the test sample which is the preferential site for crack nucleation under the three-point bend configuration depicted in Fig. 2(b). This region on the front surface is that where the highest macroscopic bending (tensile) stress takes place and hence it is likely to be the location for crack nucleation. The advantage of the three-point bend test over uniaxial or four-point bend testing is that the failure region is localized and identifiable *a priori*. The region to be investigated, marked in black in Fig. 2(a), was located using fiducial Vickers micro hardness indents. Prior to the mechanical test, material characterization by means of EBSD was conducted to identify phase boundaries and obtain crystallographic orientations that fall within the investigated region.

2.2. GND and residual stress by high resolution EBSD

Significant thermal residual strains and stresses develop at the nickel/agglomerate interface following cooling from aging temperature to room temperature due to differences in coefficients of thermal expansion [26]. The thermal strains are large enough to cause the onset of plastic strain gradients at the interface and hence lattice curvatures. The presence of the curvatures is accommodated by an additional dislocation content, namely that of geometrically necessary dislocations (GNDs). In order to capture the elastic strains and GND densities, cross-correlation based high resolution EBSD was conducted on the area containing the inclusion agglomerate with a size of $60\times40\ \mu\text{m}^2$ using a Bruker eFlash^{HR} detector and a Zeiss Auriga SEM. Diffraction patterns at each interrogation point were collected with a probe current of $\sim$13 nA using an exposure time of 200 ms at full detector

Table 1
Nominal composition of the nickel base superalloy RR1000 [28].

Element	Ni	Co	Cr	Mo	Ta	Ti	Al	B	C	Zr	Hf
wt%	bal.	18.5	15	5	2	3.6	3	0.015	0.027	0.06	0.5