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Investigation of fatigue crack initiation from a non-metallic inclusion via high energy x-ray diffraction microscopy

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ABSTRACT

Crack initiation at inclusions is a dominant, unavoidable and life-limiting failure mechanism of important structural materials. Fatigue progresses in a complex manner to find the 'weakest link' in the microstructure, leading to crack nucleation. In this study, fully 3-D characterization methods using high energy synchrotron x-rays are combined with in-situ mechanical testing to study the crack initiation mechanism in a Ni-based superalloy specimen. The specimen was produced via powder metallurgy and seeded with a non-metallic inclusion. Two x-ray techniques were employed: absorption contrast computed microtomography (μ-CT) to determine the morphology of the inclusion and its location in the gauge section of the specimen; and far-field high energy diffraction microscopy (FF-HEDM) to resolve the centroids, average orientations, and lattice strains of the individual grains comprising the microstructure surrounding the inclusion. Sequential μ-CT and FF-HEDM scans were carried out at both peak and zero applied stress following schedules of cyclic deformation. The μ-CT data showed the onset and location of crack initiation, and the FF-HEDM data provided temporal and spatial evolution of the intergranular strains. Strain partitioning and the associated stress heterogeneities that develop are shown to stabilize within a few loading cycles. Elasto-viscoplastic fast Fourier transform simulations were utilized to supplement interpretation of the experimental stress distributions and compared with the experimental stress distributions. Appropriate conditions for crack nucleation in the form of stress gradients were demonstrated and created by virtue of the inclusion, specifically the residual stress state and local bonding state at the inclusion-matrix interface.

1. Introduction

Nickel-based superalloys find extensive application in nuclear reactors, gas turbine engines and propulsion systems that are subject to extreme environments. These alloys are produced via a variety of processes, the most prominent of which is a powder metallurgy (PM) route, chosen in order to generate superior properties through greater control over compositional ranges and microstructure [1]. The alloying elements and the manufacturing process, however, inherently introduce unwanted non-metallic inclusions into the otherwise optimized microstructure. Research into processing techniques and improvement in general purity and quality standards for the powder and processing route have had success in reducing, but not completely eliminating, their occurrence in structural alloys.

Crack initiation accounts for a significant portion of the fatigue life [2]. After documenting fracture surfaces in a multitude of specimens subjected to cyclic loading, Caton et al. [3] exposed a clear bifurcation between two competing failure modes: crack nucleation at crystallographic facets and crack nucleation at inclusions. The inclusion driven failures showed correspondence

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with lower lifetimes to failure. Research into the effect of temperature [4], grain size [5], stress level [3], strain range [6] and size of non-metallic inclusions [7] has further demonstrated the prominence of inclusion driven failure for applications in which superalloys find relevance. Conclusively, crack nucleation at inclusions is an unavoidable, dominant and life-limiting failure mode as fatigue progresses to find the 'weakest link' in the material. A robust understanding of the mechanisms and driving forces behind crack initiation at inclusions is, however, still elusive.

Towards the goal of an improved understanding of inclusion driven failures, many mesoscale characterization tools are available today for mapping the underlying deformation and damage in polycrystalline materials. For example, 2-D measurements on a specimen's free surface through diffraction contrast in a transmission electron microscope [8] or high-resolution electron backscatter diffraction (EBSD) [9–12] have been successful in revealing elastic mismatch and plastic deformation through dislocation density accumulation in the vicinity of inclusions at the surface. In most cases, however, crack initiation from inclusions occurs at sub-surface features, where the effect of surface stress relaxation and advantages of compressive residual stresses through surface treatments like shot-peening subside, and the underlying matrix-inclusion interactions become prominent. EBSD can be combined with serial sectioning techniques such as focused ion beam milling [13], femtosecond laser ablation [14] and mechanical polishing [15] to collect data within the bulk of a sample. These processes, however, are destructive and cannot be used in situ to capture evolution of critical state descriptors around sub-surface features.

High energy synchrotron x-ray techniques have enabled the rapid, non-destructive characterization of the internal structure and mechanical state of a test specimen, providing the possibility of tracking the evolution of these quantities under in situ mechanical testing. Absorption contrast μ-CT involves collecting x-rays that have been directly transmitted through a sample [16,17]. During passage, the x-rays are absorbed by the material and local variations in density within the material can be captured as an intensity contrast on the detector. This allows characterization in two very important ways. Firstly, inclusions and other impurities in the material have a different chemical composition and their exact location and morphology can be determined. Secondly, discontinuities in the sample, whether they are pre-existing voids, surface aberrations and most important for this study - cracks, can be observed once they initiate and can be tracked thereafter. The μ-CT measurements are complemented by a diffraction-based technique, FF-HEDM, also known as three-dimensional x-ray diffraction microscopy (3DXRD), which can be utilized to gain information about the microstructure and micromechanical state [18].

FF-HEDM is a non-destructive technique for 3-D characterization of bulk polycrystalline specimens around a millimeter in size using a monochromatic beam of x-rays [18–23]. The FF-HEDM technique is essentially the classic rotation method adapted to high energy x-rays (>50 keV) and polycrystalline samples [18]. The fundamental setup is identical to that required for collimated-beam tomography: a single-axis goniometer perpendicular to the incident x-ray beam with a framing area detector placed downstream of the sample. The two techniques utilize different detectors, however, with the μ-CT requiring high spatial resolution (*i.e.* small effective pixel size and field of view) and the FF-HEDM technique requiring high angular resolution (intermediate effective pixel size and large field of view). Due to the constraints imposed by Bragg's law ($n\lambda = 2d_{hkl}sin\theta_{hkl}$, λ: wavelength of x-ray beam, *hkl*: Miller indices of the diffracting lattice plane, d_{hkl} : interplanar distance, $2\theta_{hkl}$: Bragg angle and n is 1) in the case of a monochromatic beam, a single crystal must be rotated to observe diffraction from different planes. While rotating, images are captured in the continuous rotation mode to form a 3-D image series from the frames integrated over equi-spaced $\Delta\omega \approx 0.25^\circ$ for $\omega \in (0, 360^\circ)$. If we parameterize a diffracted beam signal using polar coordinates $(2\theta, \eta)$ and the ω position in the rotation image series of the measured peak, the coordinates (2θ, η, ω) of all the spots produced by a single grain encode its crystallographic orientation, centroid in the specimen, and elastic strain. In the far-field configuration (detector is ~1 m from the sample) diffraction spots from different families of crystallographic planes lie near the projection of the ideal Debye-Scherrer cones on the detector, and can be pre-sorted by {*hkl*} up to degeneracy in d_{hkl} before associating/indexing them to the different grains that produced them. For more information about FF-HEDM implementation and data reduction strategies, please refer to [19–23]. Note that because full elastic strain tensors are available for each grain indexed by the analysis, the corresponding stress tensors are determined unambiguously via the stiffness tensor using the generalized Hooke's law. The combination of μ-CT, sensitive to density, with FF-HEDM, sensitive to crystal mechanical properties, provides a rich, multimodal characterization of the microstructure and micromechanical state of the sample.

The effect of residual stresses on fatigue lifetimes has been well documented [24,25] but the quantification of these stresses on a grain by grain basis has been difficult until recently. Far-field HEDM measurements can capture the residual stress state in a specimen at this scale [26–28]. Recent studies utilizing FF-HEDM measurements demonstrated that the residual stress state at the beginning of an experiment had a significant influence on the grain level stress state of a specimen [26–29]. The FF-HEDM measurements are utilized in the current work to unravel the internal stress state of the sample, especially around the inclusion-matrix interface. Once measured, these stresses can be used to initialize simulations [27–31] and check the stress evolution due to applied load from an initialized state that incorporates processing and heat-treatments prior to that point. Fast Fourier transform (FFT) simulations [32] are particularly well suited for such studies since voxelized information from FF-HEDM results can be used as a direct input and compared [33] to experimental results. Frameworks have also been established to incorporate the residual state of the material by means of an eigenstress [34].

In this paper we describe an experimental methodology, based on concurrent FF-HEDM and μ-CT, to characterize a large volume of a Ni-based superalloy with an embedded inclusion. The initial state of the sample is fully characterized and subsequently tracked spatially and temporally across an applied deformation schedule by periodically interrupting cyclic loading until a crack initiates. We use the evolution of the local microstructure throughout the volume, especially around the crack, to determine the factors contributing to crack initiation. Various failure metrics and parameters are discussed to understand and rationalize the stress distribution around the inclusion and its role in crack initiation. Finally, a 3-D elasto-viscoplastic (EVP) FFT model is used to comprehend the role of residual stresses and simulate the effect of debonding around the crack initiation site. Qualitative comparisons are performed to aid our understanding of these factors in recreating the observable conditions for crack initiation.

2. Material and specimen design

The material selected for this study is a super solvus heat treated coarse grain variant of RR1000, a polycrystalline Ni-based superalloy with a face centered cubic (FCC) crystal structure