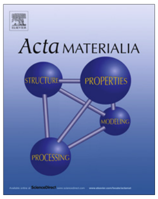
Acta Materialia 97 (2015) 367–379

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat



On the mechanistic basis of fatigue crack nucleation in Ni superalloy containing inclusions using high resolution electron backscatter diffraction

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ARTICLE INFO

Article history:
Received 5 January 2015
Revised 4 June 2015
Accepted 16 June 2015
Available online 14 July 2015

Keywords:
EBSD
Fatigue crack formation
Geometrically necessary dislocations
Residual stress
Ni superalloy with inclusion

ABSTRACT

A series of interrupted three-point bend low-cycle fatigue tests were carried out on a powder metallurgy FHG96 nickel superalloy sample containing non-metallic inclusions. High resolution electron backscatter diffraction (HR-EBSD) was used to characterise the distribution and evolution of geometrically necessary dislocation (GND) density, residual stress and total dislocation density near a non-metallic inclusion. A systematic study of room temperature cyclic deformation is presented in which slip localisation, cyclic hardening, ratcheting and stabilisation occur, through to crack formation and microstructurally-sensitive propagation. Particular focus is brought to bear at the inclusion–matrix interface. Complex inhomogeneous deformation structures were directly observed from the first few loading cycles, and these structures were found not to vary significantly with increasing number of cycles. A clear link was observed between crack nucleation site and microstructurally-sensitive growth path and the spatially-resolved sites of extreme values of residual stress and GND density.

1. Introduction

High performance structural materials drive technology forward and enable us to travel the world, exchange cultures and ideas, and broaden collaboration and understanding. Material scientists and engineers enable progress through innovation in materials design and manufacture, delivering new technologies and making existing technologies more economical for both supplier and consumer. As our demands heighten so must understanding and prediction of materials performance improve, and in particular we must strive to obtain physical understanding and to enable prediction of materials performance at the microstructural level to generate change.

In this work, we focus on mechanistic understanding of fatigue in Ni superalloys used as structural materials in gas turbine applications. These alloys were first developed in the 1940s and evolutionary design since their invention has pushed their performance ever further and significantly improved fuel efficiency and thrust in a modern jet engines, enabling lower cost, more sustainable and safer transport of people and goods across the world. Well understood technical materials, with outstanding structural performance, like the Ni superalloys, are essential in delivery of successful jet engine technology, including blade and disk components [1].

Turbine disks in advanced aero-engine are subjected to very high cyclic stresses over long periods often at elevated temperatures (~550–750 °C) and their structural performance, as well as high resistance to creep and oxidation, are paramount. For Ni-based disk components, powder metallurgy manufacturing routes are often used to achieve more homogenous microstructures and chemical distributions. Of particular interest for this study, the second generation of FGH 96 powder metallurgy (PM) Ni superalloy was designed for damage tolerance.

Non-metallic inclusions are one of the typical defects in Nickel-based PM superalloys. These inclusions are inevitable defects presenting in powder metallurgy alloy processing, as although extreme care has been taken, contamination of powder cannot be avoided completely in melting, powder atomisation and handling, through to consolidation. It is found that non-metallic inclusions significantly degrade FGH96 low cycle fatigue life [2]. This motivates us to improve our understanding of this complex process, and we begin with low temperature fatigue crack nucleation utilising state of the art experimental techniques in order to improve fatigue life predictions, and deliver safer and more economical components.

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http://dx.doi.org/10.1016/j.actamat.2015.06.035
1359-6454/

Many recent reviews have outlined the importance of understanding of fatigue crack formation, and propagation mechanisms at the microstructure scale are critical to facilitate quantitative prediction of fatigue lifetime as a function of microstructure [2–7]. These reviews highlight the need to deliver new experimental insight into deformation processes and failure in fatigue to drive modelling that can accurately capture microstructurally-sensitive effects and use geometrically faithful models. These modelling efforts have focused at a range of length and timescales, using approaches such as molecular dynamic simulations [8,9] up to the grain level using crystal plasticity finite element techniques [10–15]. This range of approaches necessitates ever increasing fidelity of experimental studies that span length and timescales, such as X-ray synchrotron [16–18] and high energy neutron diffraction [19]; as well as electron microscopy [20,21]. Microstructurally-sensitive and physically based modelling approaches necessitate local measurements of defect content and residual stresses to improve the prediction of fatigue crack nucleation and short crack growth.

Modelling efforts using molecular dynamics [8] and crystal plasticity finite element [13] techniques lead to the conclusion that fatigue crack formation criteria should be closely linked to stored energy which, for materials that deform plastically, is related to stored dislocation density and residual elastic stress.

High resolution, cross-correlation based, electron backscatter diffraction (HR-EBSD) enables the evaluation of lattice curvature which may be linked to dislocation content using Nye's tensor [22] to calculate stored geometrically necessary dislocation (GND) densities. These GNDs, which are equivalent to a local accumulation of dislocations with the same sign, can be included as hardening terms in strain gradient plasticity, such as those proposed by Ashby [23], Fleck et al. [24] and Gao et al. [25]. Plastic strain gradients can play a critical role in understanding size effects present in most material systems and mechanical tests, either due to sample geometry or strain gradients developed because of heterogeneous microstructures, resulting in extra hardening effects [26] and can be incorporated into crystal plasticity finite element models [27–29].

At present there is limited work that addresses the relationship between stored GND density in cyclically deformed materials and crack nucleation, likely due to the relatively recent renewed interest in GNDs and the development of measurement techniques. Littlewood et al. [30] have used HR-EBSD to study the development of GND density in Ti–6Al–4V alloy subjected to load controlled dwell fatigue and four EBSD maps were acquired in different samples which were subject to 0, 1500, 3000, 4200 cycles respectively. Jiang et al. [31] used HR-EBSD to study GND density distribution and evolution in cyclically deformed polycrystalline copper samples subjected to 0, 2, 200, 2000 cycles respectively. While these studies provide quantitative analysis of stored GND density and associated structure, no crack formation process was directly observed and comparison of the samples was statistical in nature as different samples and sampled areas were used. Birosca et al. [32] used Hough based EBSD approaches (which have $\sim$100 less GND resolution) to quantitatively study GND density distributions near fatigue crack propagation paths in nickel superalloy subjected to thermo-mechanical fatigue. They found that local microstructural texture influences fatigue crack propagation and fatigue life. However the evolution of GND density and local texture was absent from their study which is required to link the development of GNDs to fatigue crack formation.

The EBSD-based curvature analysis of stored GND density does not enable measurement of statistically stored dislocations (SSDs), as they cause no change in net lattice curvature. SSDs may be closely bonded dipoles or multipoles and from earlier TEM studies on fatigued oxygen free high conductivity copper samples, it can be seen that reversible cyclic deformation generated high proportions of closely bonded ladders of dislocation dipoles in persistent slip bands (PSBs) which are potentially associated with crack formation sites [33–36].

While spatial resolution of SSDs is not currently possible with HR-EBSD based methods, recent work by Wilkinson et al. [37] includes quantitative estimation of the total dislocation (i.e. SSD and GND) content for a map (i.e. not spatially resolved, but still quantitative) through calculations based upon distributions of stress associated with the statistics of probing close to a dislocation core within a dislocated crystal, using an analysis based upon the work of Groma et al. [38,39]. Wilkinson et al. [37] validated with discrete dislocation dynamics calculations and demonstrated that this technique works with HR-EBSD based residual stress maps assessing a series of experimental samples in Cu deformed in tension to increasing levels of plastic strain. The mathematical framework to calculate total dislocation density from HR-EBSD measurement can be found in Appendix of this current work.

The main purpose of this current study is to provide new experimental insights into cyclic deformation in structural materials, with focus on room-temperature deformation of Ni-superalloys. We address correlations of dislocation content with fatigue crack formation, and their experimental study presented provides new information that is required to provide predictive capability in fatigue life, which encompasses both crack nucleation and microstructurally-sensitive crack growth, as well as crack propagation which all contribute to component life.

2. Method

2.1. Sample fabrication and preliminary characterisation

A powder metallurgy (PM) Ni superalloy FGH96 bar-stock sample containing a non-metallic inclusion was supplied by AVIC-BIAM. A bending test sample with rectangular $12 \times 3\ \text{mm}^2$ cross-section and 3 mm width was cut using electric discharge machining (as shown in Fig. 1). It was annealed at 750 °C for 7 h to relax internal stresses and reduce the stored dislocation content induced during manufacture. The sample was ground using 800-grit silicon carbide papers for 20 min at 20 N force and this was followed with finer grinding (1200-, 2000-, and 4000-grit) for increasing durations with the same force. Final polishing was performed using oxide polishing suspension (OPS) for 40 min and high quality EBSD patterns were obtained for mapping (shown in Fig. 2(a) and (b)) and HR-EBSD analysis.

In general, the non-metallic inclusions present in this material vary significantly in terms of size and morphology. The maps in Fig. 2(a) and (b) are illustrative of the FGH96 Ni matrix microstructure and the associated inclusion. While this paper focuses on the behaviour of a particular inclusion, it remains indicative of deformation and failure in generic PM Ni superalloys with inclusions.

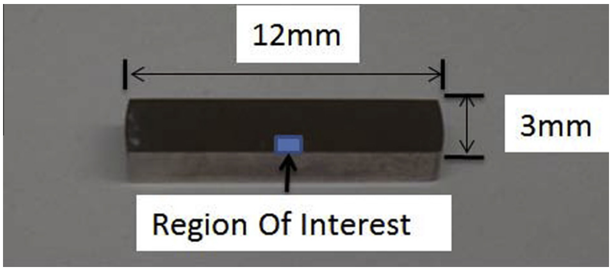

Fig. 1. An image of PM Ni bar specimen with marked dimensions. The region of interest (ROI) indicates EBSD mapping area at the centre base of the bar.