

# Crystal plasticity and high-resolution electron backscatter diffraction analysis of full-field polycrystal Ni superalloy strains and rotations under thermal loading

Tiantian Zhang<sup>a,\*</sup>, David M. Collins<sup>b</sup>, Fionn P.E. Dunne<sup>a</sup>, Barbara A. Shollock<sup>a,c</sup>

<sup>a</sup> Department of Materials, Imperial College London, London SW72AZ, UK

<sup>b</sup> Department of Materials, University of Oxford, Oxford OX13PH, UK

<sup>c</sup> WMG, University of Warwick, Coventry CV47AL, UK

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## Abstract

Electron backscattered diffraction (EBSD) has been employed to study a polycrystalline nickel superalloy containing a complex non-metallic agglomerate under thermal loading. Heterogeneous distributions of elastic strains are observed near the inclusion due to its complex geometry and these have been quantified. Lattice rotations were also related to geometrically necessary dislocation (GND) density ( $\sim 10^{14} \text{ m}^{-2}$ ), indicating the development of localized plasticity arising from the mismatch in thermal expansivity between the Ni polycrystal and the inclusion. A crystal plasticity finite-element (CPFE) model which explicitly represents the full detail of the complex microstructure was developed to interpret the experimental measurements, and good quantitative and qualitative agreement has been obtained. However, a limitation of the EBSD technique when investigating polycrystal systems is that full-field, transgranular strain measurement remains difficult due to the necessity to reference a lattice spacing within a grain for strain calculation. An inverse reference shifting methodology has been developed using CPFE modeling to overcome this problem, thereby allowing like-for-like and grain-by-grain strain comparisons to be made. The method, in conjunction with high-resolution EBSD, shows promise for the determination of full-field strains and rotations in polycrystalline materials, and provides key information for fatigue nucleation in these material systems.

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## 1. Introduction

Polycrystalline nickel-based superalloys are uniquely capable of delivering exceptional mechanical properties when subjected to high temperatures within a corrosive environment, enabling their extensive use for gas turbine disc applications. The mechanical performance of these alloys arises from the presence of a multimodal distribution

of coherent,  $\text{L}_{12}\gamma'$  precipitates, which exhibit a cube–cube orientation relationship with the  $\text{Al } \gamma$  matrix. Due to the levels of segregation in conventional cast-wrought processing and the need to impose deformation straining, there is increasing interest in producing superalloy disk components via a powder metallurgy route. Despite the fact that great efforts have been introduced to improve powder cleanliness, contamination from refractories in contact with molten metal are inevitable and result in the formation of inclusions. Commonly observed inclusions in modern nickel superalloys include such non-metallic inclusions. These particles severely degrade the mechanical properties.

\* Corresponding author.

E-mail address: [tiantian.zhang08@imperial.ac.uk](mailto:tiantian.zhang08@imperial.ac.uk) (T. Zhang).

In particular, the resistance to fatigue loading is strongly affected because cracks usually initiate from these inherent features and can rapidly propagate with the aid of loading, sometimes resulting in unpredictable failures [1–3].

Much work has been carried out to study the influence of inclusions on fatigue crack nucleation and propagation in nickel superalloys. Temperature [4,5], grain size [6,7], stress level [8], strain range [9] and size of non-metallic inclusions [10] have been found to affect fatigue performance. These microstructural features (grain boundaries, inclusions) introduce dislocation barriers and thus give rise to accumulation of damage, resulting in crack nucleation. However, if the effects of inclusions are to be well understood, a detailed study of elastic strain and stress accumulation as well as lattice distortion local to the heterogeneity must be conducted at appropriate length scales in conjunction with experimental validation.

Cross-correlation-based high-resolution (HR) electron backscattered diffraction (EBSD) has become an emerging tool for mapping elastic strains at mesoscopic and microscopic scales [11,12] and reveals microstructural mechanisms of deformation. The geometrically necessary dislocation (GND) density can be measured by analysis of lattice curvature measured through Nye's tensor [13] and hence provides information on plastic deformation. To date, this technique has been used to study the anisotropic behavior of hexagonal close-packed (hcp) crystals [14], GND distributions after tensile deformation in Ti [15], macrozones in hot-rolled Ti–6Al–4V [16], residual stresses and GND accumulation in annealed [17,18] and deformed copper [19], and transformation-induced GND development in steels [20]. In particular, a recent application of this technique to obtain strain and rotation fields around a carbide particle in a single-crystal nickel matrix under thermal loading has shown both qualitative and quantitative agreement with gradient-enhanced crystal plasticity finite-element (CPFE) predictions [21,22]. However, the quantitative determination of full-field, absolute elastic strain and lattice rotation distributions in polycrystalline metals using HR-EBSD is currently not available due to the so-called “reference pattern” problem [23].

CPFE is a desirable tool to probe microscopic deformation. A significant driver is to develop a cost-effective way to predict accurately the evolving plasticity in industrially relevant engineering alloys under relevant loading conditions [24,25]. Application of this technique is widespread and can be found in studies of anisotropy in Ti [14,24,26], fatigue crack nucleation in steels [27], and thermal residual stresses in nickel superalloys [22]. A remaining challenge is to capture accurately the full details of microscopic strain, stress, lattice rotation and GND density measured by experiments, and particularly with HR-EBSD [28]. The use of Bragg's law simulation patterns is a potential way to overcome this challenge [29], but the geometric configuration of EBSD camera and sample must be obtained with great accuracy. Common uncertainties in pattern centre lead to errors of  $\sim 10^{-3}$  in strain

measurement [30]. However, the selection of the simulation method may have significant effects on shift measurement [29]. Therefore, the level of fidelity and uncertainty associated with pattern simulation motivates the development of alternative techniques to overcome the “reference pattern” problem.

In this paper, a CPFE model and HR-EBSD are employed to study a non-metallic inclusion in a powder metallurgy produced polycrystalline nickel-based superalloy (RR1000). When subjected to stress relief by heat treatment at 1033 K, significant strains develop due to differing thermal expansivities of the inclusion and nickel matrix. Here, we present elastic strains and lattice rotations determined by both model and experiment. Qualitative and quantitative comparisons are then performed. A reference shifting methodology is introduced to reference correctly the strain and rotation fields, hence enabling detailed, full-field comparisons between the HR-EBSD-measured results and those predicted using CPFE. Correctly referencing polycrystal strains and rotations in HR-EBSD is termed direction correction, but direct experimental methodologies to do this are not currently available. Hence, in this paper, inverse “referencing” of strains and rotations from the CPFE model is performed in order to enable detailed, full-field polycrystal comparisons between experimental measurements and model predictions.

## 2. Materials and methodology

RR1000 samples were supplied by Rolls-Royce plc and were cut down to a dimension of  $6\ \mu\text{m} \times 5\ \mu\text{m}$  for microscopic characterization. The nominal composition of the alloy is given in Table 1. The sample was produced via a powder metallurgy route followed by extrusion, forging and a two-step heat treatment. A sub-solvus solution heat treatment was conducted at 1393 K for 4 h and then forced air quenched. Upon cooling, the  $\gamma$  matrix becomes sufficiently supersaturated that a fine dispersion of intragranular  $\gamma'$  precipitates homogeneously nucleate and grow until the diffusion fields overlap, resulting in soft impingement [31]. The material was next subjected to an aging heat treatment at 1033 K for 16 h where the intragranular precipitates coarsen in a controlled manner to provide precipitate size distributions which are tailored to yield the best overall performance of a turbine disk component. Notably, the resulting fine-grained microstructure, combined with the multimodal distribution of  $\gamma'$  precipitates, results in good low-cycle fatigue properties.

A non-metallic inclusion was detected from an evaluation of microstructure in the RR1000 sample, which was then carefully sectioned to leave the inclusion on the surface. Standard metallographic grinding and polishing were performed to achieve a suitable surface quality for scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis, after which a final light polish with colloidal silica for 10 min was applied to obtain the optimum surface finish required for subsequent EBSD analysis.

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