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High resolution electron back-scatter diffraction analysis of thermally and mechanically induced strains near carbide inclusions in a superalloy

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Abstract

Cross-correlation-based analysis of electron back-scatter diffraction (EBSD) patterns has been used to obtain high angular resolution maps of lattice rotations and elastic strains near carbides in a directionally solidified superalloy MAR-M-002. Lattice curvatures were determined from the EBSD measurements and used to estimate the distribution of geometrically necessary dislocations (GNDs) induced by the deformation. Significant strains were induced by thermal treatment due to the lower thermal expansion coefficient of the carbide inclusions compared to that of the matrix. In addition to elastic strains the mismatch was sufficient to have induced localized plastic deformation in the matrix leading to a GND density of $3 \times 10^{13}\ m^{-2}$ in regions around the carbide. Three-point bending was then used to impose strain levels within the range ±12% across the height of the bend bar. EBSD lattice curvature measurements were then made at both carbide-containing and carbide-free regions at different heights across the bar. The average GND density increases with the magnitude of the imposed strain (both in tension and compression), and is markedly higher near the carbides particles. The higher GND densities near the carbides (order of $10^{14}\ m^{-2}$) are generated by the large strain gradients produced around the plastically rigid inclusion during mechanical deformation with some minor contribution from the pre-existing residual deformation caused by the thermal mismatch between carbide and nickel matrix.

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1. Introduction

Superalloys have performed well as blade and disc materials in turbine engines due to their exceptional elevated temperature strength, high resistance to creep, oxidation and corrosion as well as good fracture toughness. However, a critical property of these alloys is their resistance to fatigue cracking, particularly at service temperature. The largest single cause of component failures in aircraft turbine engines can be attributed to fatigue [1,2]. Fatigue cracking results in rapid, often unpredictable failure, due to propagation of fatigue cracks, which usually initiate from small defects that are either inherent in a material or build-up as a result of deformation [1].

Among the several different classes of superalloys, nickel-based superalloys have gained an eminent place due to their structural stability in the face-centred cubic (fcc) form, which is both tough and ductile. Much work has been carried out on the initiation and propagation of cracks in nickel superalloys, which, due to complexity of practical applications, depend on several factors [3–7]. In particular, fatigue crack initiation studies in nickel-based superalloys have revealed that inclusions such as carbides [4,8–16], and persistent slip bands (PSBs) [17–23] are major sources of crack nucleation. However, if the deformation and failure processes are to be well understood from a fundamental level,

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it is necessary to study the local strain enhancements and lattice distortions near these microstructural features. The need to know the local state of stress and strain near selected features within a material is of great importance, especially in structural engineering components.

With recent progress made towards elastic strain (and hence stress) mapping using electron back-scatter diffraction [24,25], it is now possible to measure the strain fields around selected microstructural features. Use of the Nye dislocation tensor [26] then allows the measured lattice curvatures to be used to estimate the geometrically necessary dislocation (GND) content, thus providing information about plastic deformation in addition. To date, this technique has been used to study functional materials [24,25,27,28], indentations in Si [29], Fe [30] and Ti [31,32], transformation induced GNDs in steels [33,34], GND accumulation during hot rolling [35] and fatigue [36] in polycrystalline Ti–6Al–4V.

Here we present the measured strains, rotations and estimated GND content around large carbide precipitates (~10–20 μm) in an essentially single crystal directionally solidified nickel-based superalloy (Mar-M-002). We consider both the case of thermal loading and subsequent mechanical loading through three-point bending.

2. Materials and methodology

Mar-M-002 is a high tantalum and niobium-containing variant of the well-known Mar-M-200 superalloy. The composition of MAR-M-002 is given in Table 1. The sample was supplied by Rolls-Royce, in the form of a bar stock (13.8 mm in diameter and 100 mm long) and directionally solidified along its length. Simple beam sections (13.5 mm × 3 mm × 3 mm) were then cut out from the centre of the supplied bar. These beams have large columnar grains containing carbide precipitates distributed randomly throughout the matrix. These carbides are elongated along the solidification direction and act as large hard barriers to slip in the metallic matrix. The sample was given a heat treatment consisting of two steps, the first being 1 h at 1100 °C in order to take the material over the γ' transus to solutionize. This was followed by ageing for 16 h at 870 °C to precipitate the γ' particles. This heat treatment procedure was repeated for a second time, so as to achieve an enlarged grain structure.

After the double heat treatment, all outer surfaces, except ends, of these test specimens were metallographically polished. The surface was first ground on water-cooled silicon carbide paper of successively smaller grit size. This was followed by polishing using successively finer grades of diamond abrasive. A final polish using colloidal silica was performed, as it is known to give exceptional results and the level of finish required to perform electron back-scatter diffraction (EBSD) on the surface of our test pieces.

Preliminary EBSD results showed a single crystal matrix, which was not unexpected due to massive grain growth from the double heat treatment. The nickel matrix was oriented with its cube axes almost aligned with the axes of the bend beam as is illustrated by the wireframe inset in Fig. 1a, and is described by the Euler angles $\varphi_1 = 83.5°$, $\varphi = 91.5°$, $\varphi_2 = 0°$ using Bunge notation [37]. Cooling from such high temperatures combined with the difference in thermal expansion coefficients between the matrix and carbides results in a considerable thermal residual strain around the carbides. Several carbides were identified through the height (i.e. position along the x_2 axis) of this beam and EBSD maps were obtained around each particle to assess these thermal strains.

The beam was then deformed monotonically in a three-point bend rig, with a displacement of 0.5 mm imposed at the centre of the beam supported by two rollers 12 mm apart. EBSD maps were then taken around the same carbides that had been mapped before bending. The axis of the beam (i.e. tensile/compression axis) is along the x_1 axis in all EBSD maps. In addition to these, maps were made in regions away from the carbide precipitates at different heights across the bend beam. A finite element analysis of the three-point bend test was used to establish the strain levels at the sites investigated using EBSD.

In each case, EBSD patterns were recorded using a ~1000 × 1000 pixel, peltier cooled charge coupled device (CCD) camera at full resolution on a tungsten filament JEOL-6500 FEG scanning electron microscope (SEM). The scintillator screen was held at its usual position so as to subtend a large capture angle (~70°) at the sample. Typical SEM conditions used were 20 keV beam energy, and a beam current of about 10 nA, for which exposure time was about a second. Patterns were recorded at full resolution with intensities digitized to 12-bit on hard disk using TSL/EDAX OIM DC 5.3 software for subsequent off-line batch-wise analysis using the strain determination software CrossCourt 3.[1] All maps were obtained using a 250 nm step size, which is well above the spatial resolution of the method.

Lattice rotations and elastic strains within the sample cause small shifts in the positions of zone axes and other features in the EBSD patterns obtained as the electron beam is scanned over the sample. These small pattern shifts are measured using automated image-processing based on cross-correlation analysis and then related to the size and nature of the strains and rotations [24,25]. The shifts at four or more sub-regions widely dispersed across the EBSD patterns are sufficient to directly calculate eight of the nine degrees of freedom. This last degree of freedom is the hydrostatic dilation or contraction of the lattice and can be recovered by making use of the fact that EBSD measurements come from within a few tens of nanometers of the free surface which must remain traction-free. Setting the stress normal to the free surface to zero allows the three normal strains to be fully separated so that all six terms in the strain tensor and all three terms in the lattice rotation tensor are fully determined. In this work, we measure the pattern shifts at many more than the minimum four

[1] From BLG Productions Ltd., Bristol, UK (www.blgproductions.co.uk).

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