Acta Materialia 98 (2015) 29–42

Contents lists available at ScienceDirect

Acta Materialia

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

High resolution mapping of strain localization near twin boundaries in a nickel-based superalloy

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ARTICLE INFO

Article history:
Received 4 December 2014
Revised 3 July 2015
Accepted 5 July 2015
Available online 17 July 2015

Keywords:
René 88DT superalloy
Strain localization
High resolution SEM DIC
Plastic straining
Coherent twin boundary
In situ testing
Elastic anisotropy

ABSTRACT

Damage during cycling loading of polycrystalline metallic alloys involves localized plastic straining at the scale of individual grains. To better understand damage accumulation processes and to build models for material behavior there is a need for quantitative assessment of the heterogeneous strain fields at the grain and even more microscopic scales. In the present study, a digital image correlation (DIC) approach has been developed to measure the strains at the grain level and at finer scales where plastic strain localization is manifested as physical slip bands. Strain fields have been measured in situ and ex situ on a René 88DT polycrystalline nickel-based superalloy to assess the grain-scale deformation processes during monotonic straining in tension and compression. DIC analysis and transmission electron microscopy demonstrate that slip occurs in a highly localized manner. The highest localized strains developed in slip bands that formed on {1 1 1} planes parallel to, and slightly offset from, annealing twins. Enhanced local straining below yield was observed during compression loading. The degree of strain concentration caused by slip bands impinging on grain boundaries was also analyzed. The results are compared to predictions of plasticity models.

1. Introduction

Damage during cyclic loading of polycrystalline metallic alloys involves accumulation of plastic strain at the scale of individual grains, particularly in the vicinity of grain boundaries and interfaces. In materials that form annealing twins during processing, these special interfaces are often locations of particular interest for their role in damage accumulation. Miao et al. [1,2] reported for the nickel-based alloy René 88DT under very high cycle fatigue loading conditions that cracks initiated in high Schmid factor grains on planes parallel to and slightly offset from coherent twin boundaries. Crack initiation during low cycle fatigue has also been observed along twin boundaries for other nickel-based superalloys [1–3], stainless steels [4] and copper [5,6]. Heinz and Neumann [7] first suggested that elastic anisotropy causes a local stress concentration that strongly enhances glide at twin boundaries. For the case of a fcc coherent twin, the boundary is always parallel to a slip plane, so dislocations can travel across the entire diameter of a grain, creating a stress concentration, particularly if the slip is localized on a single system. Heinz and Neumann [7] emphasized that twins would lead to stronger strain localization than a general grain boundary, though the degree to which local strains are concentrated relative to the macroscopic imposed strains was not examined.

To develop more quantitative, predictive models for monotonic and cyclic loading there is a need for quantitative assessment of the heterogeneous strain fields at the microscopic scale, to better relate the local mechanical behavior to the global loading conditions. While crystal plasticity simulations based upon either molecular dynamic or finite elements are currently being developed to address these microstructural-influenced phenomena [8–15], little experimental data on local strain at the microscale is available for validation [16–18,39–41].

Digital image correlation (DIC) has recently emerged as a robust method for experimental quantification of 2-D in-plane strain fields at the microstructure scale [16,17,19–22,38]. However it is still challenging to obtain spatial resolutions high enough for the measurement of the strain field at the micron and submicron scale. In several studies [16,23,24], grain-scale spatial resolution has been achieved in materials with mm- or cm-scale grains. However, since many high strength structural materials have grain sizes of 100 μm or smaller, it is necessary to refine the DIC

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http://dx.doi.org/10.1016/j.actamat.2015.07.016
1359-6454/

measurements to finer scales. Tatschl et al. [28] succeeded in measuring strain heterogeneities by DIC using a submicron scale speckle on grains with an average size of about 100 μm. Moreover Tatschl et al. [28] demonstrated the combination of DIC measurements and electron backscatter diffraction (EBSD) measurements in order to relate crystallographic data with strain fields that develop during tensile straining. While heterogeneities were detected at the grain-scale, the spatial resolution was insufficient to quantify strains induced by localized slip within grains. To gain insights on the role of grain-scale microstructure, including twin boundaries, higher resolution observations are required [38].

When coherent twins are present in a fcc material, based on the geometry of the slip system activated in the twin and parent grain, there are two possible scenarios: (1) slip transmission across the twin: the plane of the activated slip system crosses the twin boundary, with dislocation transmission across the boundary; (2) parallel slip with slip systems activated parallel to the twin boundary without a requirement for crossing the twin. In the parallel configuration, since dislocations do not cross the twin boundary, they can travel across the entire diameter of a grain. Stein, Rollett, and Ingraffea et al. [14,15] using crystal plasticity models report very high stresses near twin boundaries for superalloys when there is no slip transmission across the twin boundary.

In the present paper, a high resolution DIC measurement approach [37] has been developed for a nickel based superalloy with an average grain size of 26 μm, to obtain insights to the straining processes at the grain-scale, but also at a finer scale where plastic strain localization can be directly correlated with physical slip bands. A strong effect of twin boundaries on the local strain field has been observed and related to deformation mechanisms observed by transmission electron microscopy. Moreover, enhanced local plastic straining near twin boundaries below yielding is reported for straining in compression and strains associated with slip transmission from grain to grain are quantified. The results are compared to predictions of plasticity models.

2. Material and experimental procedure

2.1. Material

The material examined in this study is a polycrystalline nickel-based superalloy, René 88DT. This commercial alloy was processed through the powder metallurgy route and has a nominal composition of 13% Co, 16% Cr, 4% Mo, 4% W, 2.1% Al, 3.7% Ti, 0.7% Nb, 0.03% C, 0.015% B (weight percent) [25,27]. The microstructure of the alloy consists of a γ matrix and two populations of gamma prime (γ') precipitates: larger secondary and nm-scale tertiary γ' within the γ grains. Due to the super-solvus nature of the solution anneal, the microstructure of René 88 DT contains no sub-solvus γ', which is usually termed primary γ' [27]. The size of the secondary γ' phase is about 100–200 nm, while tertiary γ' precipitates are several nanometers in diameter. Crystallographic features have been previously studied using electron backscatter diffraction measurements [29]: the material possesses very weak crystallographic texture, a large population of $\Sigma 3$ grain boundaries (58% of the total boundary fraction), an average grain size of 26 μm, and a low fraction of large grains on the order of two to five times of the average grain size.

To remove any residual surface deformation layer, specimens were ground with SiC papers and chemical-mechanically polished with 0.05 μm colloidal silica for 12 h. Finally, the gauge length of the specimen was etched with a Fe(III) chloride + HCl solution in order to produce a nm-scale speckle pattern which is favorable for digital image correlation without interfering with EBSD measurements [26,28].

2.2. Mechanical tests

Tensile tests were performed at room temperature in situ using a Kammrath & Weiss ±5000 N stage within a Hitachi SU-70 Schottky-SEM on flat dogbone-shaped specimens with a cross-section of 1×3 mm^2. The loading stage was tilted to 50° within the microscope chamber. The specimen itself was tilted to 20° relative to the stage, leading to a sample tilted to 70°, which enables simultaneous acquisition of DIC and EBSD data. The high-resolution images were obtained at 1000× magnification, to avoid distortion associated with low magnification Scanning Electron Microscopy (SEM), and by using Back Scatter Electron (BSE) detectors. The corresponding EBSD maps were taken with an Oxford F + detector with a step size of 0.25 μm. Diffraction patterns were acquired using the EDAX OIM-Hikary XM4 electron back-scatter diffraction detector, with an accelerating voltage of 20 keV, a 4×4 binning and a beam current typically about 0.2 nA. Prior to deformation, initial images and EBSD maps were acquired. During tensile testing, several interruptions at various strain levels were performed (0.35%, 0.5%, 1%, 1.5%, 2%, 2.5%, 3% and 3.5% total strain). SEM images were acquired under load and after unloading. A mechanical extensometer was attached to the back of the specimen to record the macroscopic strain.

The compressive tests were performed at room temperature ex situ on an electromechanical MTS machine, with cylindrical samples with a section of 6 mm in diameter. Flat zones were machined on the gauge length to perform DIC and EBSD measurements. High-resolution SEM images and EBSD maps were obtained at 1000× magnification prior to deformation using. After applying the desired compressive strain to the specimen, the specimen was placed in the SEM microscope once again and images of the deformed state were captured at the same locations and magnification as the reference images. Interrupted tests were performed at −0.35%, −0.5%, −1%, −1.5%, −2%, −2.5%, −3% and −3.5% total strain. Two zones containing more than 50 grains were investigated within each sample with analysis of the strain field during compression and tension at the grain-scale.

2.3. Digital SEM image correlation

The in-plane displacement fields at the microscopic scale were obtained using DIC open source software (OpenDIC) [26]. The SEM images (5120×3840 pixels) were divided into custom sized subsets of 27×27 pixels regularly spaced by 17 pixels in both horizontal "x" and vertical "y" directions. The correlation itself was based on the zero-normalized cross-correlation (ZNCC) criterion [30]. The correlation of each subset is fully independent from the correlation of neighboring subsets. Deformed images were interpolated by a factor of 10 using a biquintic polynomial interpolation algorithm. The interpolation led to a theoretical accuracy of 0.1 pixel (~6.2 nm at 1000× magnification) for the displacements within each subset. A companion application was implemented in MatLab to calculate and plot at each point of the image the in-plane strain fields (ε_{xx}, ε_{yy} and ε_{xy}) from the displacement fields U_x and U_y in the x (loading) direction and y (transverse) directions, respectively. The strain calculation was based on a isoparametric 2D finite element formalism using subset centers as nodes and introducing four Gauss bilinear interpolation points per element. Direct DIC measurements (cumulative) were constructed by comparing after each deformation step the micrograph of the deformed specimen with the micrograph of the undeformed specimen. Fig. 1 presents a typical SEM image used for the measurements. Grain boundaries were correlated using EBSD measurements.

Scanning electron microscopy-based DIC requires careful consideration of error induced by distortions that are inherent to SEM imaging [21]. In order to minimize the error, specific