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ScienceDirect
Acta Materialia 78 (2014) 103–113
Acta MATERIALIA
www.elsevier.com/locate/actamat

Mapping deformation in small-scale testing

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Received 18 April 2014; received in revised form 13 June 2014; accepted 14 June 2014
Available online 16 July 2014

Abstract

A method is demonstrated for mapping the displacements in small-scale test samples using digital image correlation. The deformation associated with crystallographic slip and with the lattice rotation has been determined in a copper micropillar oriented for single slip, in areas as small as $0.16 \times 0.16\ \mu m^2$. It is shown that gradients of slip accompanied the curvature of the lattice at the ends of the pillar, allowing the lattice to rotate. The spacing of slip bands was also seen to decrease in these regions. The observed deformation was associated with gradients of compressive stress across the section of the pillar given by the superimposition of bending moments, caused by the constraint at both ends of the pillar. The curvature of the lattice observed at the base of the pillar was found to be the same as that at the end of a protruding slip step, although the deformation gradients were different. This provides experimental evidence of the non-unique relationship between lattice curvature and deformation gradients.

Keywords: Crystal plasticity; Electron backscatter diffraction; Geometrically necessary dislocations; Slip band; Digital image correlation

1. Introduction

Microcompression, originally developed to study the effects of volume on plastic flow in face centred cubic metals [1–3], is becoming widely used to study both plastic flow and cracking in a much wider range of materials, in particular brittle materials, such as ceramics [4,5], intermetallics [6,7] and hard coatings [8], as well as high strength steels [9], superalloys [10] and even dried colloidal dispersions [11].

Strain measurement is most often carried out by measuring the displacement of a punch corrected for the compliance of the indenter frame, where the sample is treated in essentially the same way as a bulk sample. Much useful information has been obtained in this way. However, such techniques have the potential for studying the effects of microstructural features on deformation, using site-specific milling techniques to isolate areas of interest. Realizing this requires that that local strain measurements can be made. Such measurements must give a quantifiable description of the deformation, which is often large, in particular measuring the contributions of crystallographic slip and lattice deformation.

Crystal plasticity theories provide a well-established kinematic model for describing crystal deformation. Take a crystal that undergoes some deformation given by the tensor $\mathbf{F}$ and consider this deformation to occur in two stages [12]. In the first, elements within the crystal deform individually by the movement of dislocations through the lattice, $\mathbf{F}^p$. If such intermediate deformation is incompatible, i.e. the elements detach or overlap, a further deformation is required to ensure that the material remains continuous, $\mathbf{F}^e$, which includes elastic strains and lattice rotation, $\mathbf{R}^e$. If the plastic deformation is sufficiently large, the elastic strains can be neglected [13], so that $\mathbf{F}^e \approx \mathbf{R}^e$ and $\mathbf{F}$ can then be expressed as

$$\mathbf{F} = \mathbf{R}^e\mathbf{F}^p \quad (1)$$

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http://dx.doi.org/10.1016/j.actamat.2014.06.033
1359-6454/

For a detailed discussion of this decomposition, see Ref. [14].

In recent years, diffraction techniques, such as electron backscatter diffraction (EBSD) and X-ray Laue diffraction, have been used in small-scale testing to map $\mathbf{R}^e$ [15–20]. However, experimental mapping of $\mathbf{F}^p$ has not been achieved. This has limited the ability to characterize the distribution of slip and validate crystal plasticity models, implemented into finite element analysis [21–23], and dislocation dynamic simulations [24]. In this paper, we show that digital image correlation (DIC) may be used to measure $\mathbf{F}$ and $\mathbf{R}^e$, confirmed here using EBSD, allowing $\mathbf{F}^p$ to be obtained.

1.1. Pattern application for DIC in small scale testing

In DIC, a displacement field is calculated which describes the movement of the pixel subsets of a reference image [25,26]. The displacement field can be differentiated to obtain $\mathbf{F}$ as described in Appendix A. Identifying the different subsets is achieved by applying a speckle pattern onto the surface of the sample. The density of speckles therefore defines the useful resolution, with a high density increasing the resolution.

Fine speckle patterns can be made using either electron beam or focused ion beam (FIB) assisted deposition [27]. In these methods, the pattern is reproduced from a bitmap image, giving control over speckle size spacing and distribution. However, the time required for the point-by-point deposition limits high density speckle patterning to preselected areas of $\sim 50 \times 50\ \mu m^2$. This is too small to be of use in polycrystals containing large grains, for which suitable patterning techniques have been developed [28], but is sufficient for micropillar compression and similar small-scale tests.

Speckle patterns applied on polished stainless steel using both Ga-FIB and electron beam deposition have been compared elsewhere [29], although their deposition on the FIB-milled structures has not. Preliminary experiments here showed that particles deposited using FIB tended to smear as shown in Fig. 1, but this did not occur where the patterns were deposited using the electron beam. Electron beam deposition was therefore used.

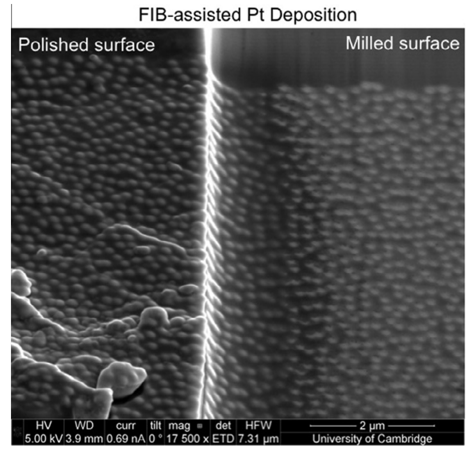

Fig. 1. Degradation of the speckle pattern following Ga-FIB on a milled surface of copper. The milling direction was parallel to the milled area.

2. Experimental

2.1. Focused ion milling and electron beam patterning

A polycrystalline annealed cube of oxygen free high conductivity (OFHC) copper, with 300 μm grain size, was polished on two adjacent facets. An initial 1200 grit paper was used to remove material that might have been plastically deformed during previous machining. The faces were then polished using 6 μm and 2 μm diamond particles, followed by a dispersion of colloidal silica (OPS) for ~5 min.

FIB milling, electron beam assisted Pt deposition and EBSD mapping were carried out using a FIB scanning electron microscope (SEM) (Helios Nanolab, FEI, USA). Because the SEM stage could not be tilted to 90°, the sample was mounted on an inclined stub in order to position the electron beam normal to the side of the pillar for electron beam assisted deposition. A square section pillar with a side length of 5 μm and an aspect ratio of 1:4 was milled at the polished edge to allow imaging normal to the external side of the pillar. The pillar was positioned at ~5 μm from the detected edge to further minimize the possibility of having previously deformed material within the pillar.

Initially, material was milled rapidly, at a current of 9 nA and an accelerating voltage of 30 kV, to shape the pillar and remove surrounding material. The top of the pillar was then milled to ensure that this was flat and normal to the pillar axis. A second milling step was carried out at a beam current of 0.9 nA, which reduced the taper to ~2°. In particular, the face on which the pattern was to be deposited was milled last to remove any material that had been laid down during previous milling. After this, the face was oriented so that it was normal to the electron beam and the pattern reproduced from a bitmap image. Deposition was completed in a single pass to avoid the spreading of deposited material that follows possible shifts in beam position due to sample charging. As the electron beam causes negligible damage to the sample surface [29], the dwell time can be set independently to determine the amount of deposited material per point. The conditions used here are given in Table 1.

A random distribution of speckles might seem the most appropriate because a periodic pattern could introduce ambiguity in the correlation [26]. However, a random distribution can result in relatively large areas free from speckles and others where speckles agglomerate to form larger features. This would reduce the effective density of speckles and so require the use of larger subsets for DIC. In the present study, an algorithm has been scripted to increase the distance between the closest speckles and progressively achieve a uniform but non-periodic distribution.