Acta Materialia 116 (2016) 200–211

Contents lists available at ScienceDirect

Acta Materialia

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

Full length article

Determining heterogeneous slip activity on multiple slip systems from single crystal orientation pole figures

Darren C. Pagan [a, *], Matthew P. Miller [b, c]

[a] Lawrence Livermore National Laboratory, United States
[b] Sibley School of Mechanical and Aerospace Engineering, Cornell University, United States
[c] Cornell High Energy Synchrotron Source, United States

ARTICLE INFO

Article history:
Received 20 April 2016
Received in revised form
5 June 2016
Accepted 9 June 2016

Keywords:
Diffraction
Pole figures
Plastic deformation
Crystallographic slip

ABSTRACT

A new experimental method to determine heterogeneity of shear strains associated with crystallographic slip in the bulk of ductile, crystalline materials is outlined. The method quantifies the time resolved evolution of misorientation within plastically deforming crystals using single crystal orientation pole figures (SCPFs) measured *in-situ* with X-ray diffraction. A multiplicative decomposition of the crystal kinematics is used to interpret the distributions of lattice plane orientation observed on the SCPFs in terms of heterogeneous slip activity (shear strains) on multiple slip systems. To show the method's utility, the evolution of heterogeneous slip is quantified in a silicon single crystal plastically deformed at high temperature at multiple load steps, with slip activity in sub-volumes of the crystal analyzed simultaneously.

1. Introduction

Crystallographic slip is the primary mode of plastic deformation in ductile, crystalline materials (notably engineering alloys): under application of shear stresses, dislocations move on particular crystallographic planes in prescribed directions. Crystallographic slip remains an important area of research since there are many important material failure modes, such as yielding, fatigue, and ductile fracture, that have been directly linked to slip. Often it is the heterogeneity of slip, the formation of intracrystal regions that experience different slip modes or the localization of slip, that appears to matter most for material failure. An accurate, detailed understanding of slip processes and the manifestation of slip-induced material evolution therefore has the potential for significant impact over a broad range of materials topics and applications. Modern micromechanical models of the plastic behavior of crystalline materials are built to accommodate representations of slip, but a detailed understanding of the complexities of the slip initiation process, driven by the subgrain mechanical environment, has proven illusive. Like many deformation processes, *in-situ* experimental observations of slip inside deforming crystals are difficult to acquire and most of what we know about slip has been inferred from post-mortem micrographs. Measurements of slip traces [1,2] and dislocation structures using electron microscopy [3,4] have helped to further our understanding of slip processes, but if we wish to fully model and predict plasticity driven failure processes, our experimental measures of slip must improve. We need to be able to quantitatively measure the complicated dynamics of slip processes within the bulk of samples to capture the initiation and acceleration of failure processes. Therefore, we need probes with rapid collection times and significant penetration depth used in conjunction with models to interpret these raw data in terms of crystallographic slip. To help meet this need, this paper presents a new experimental method which uses X-ray diffraction data measured at a synchrotron source with a kinematic model of crystallographic slip to quantify the heterogeneity of shear strains on different slip systems within ductile crystals as they plastically deform. Our method relates the heterogeneity observed in distributions of diffracted X-ray intensity to the variation of rotation of a set of crystallographic planes and the underlying cause, plastic slip on a particular slip system. The method is capable of probing slip during *in-situ* mechanical loading in sub-volumes of crystal that are deforming using different combinations of slip systems.

There is a long history of using X-ray diffraction to study crystallographic slip. Slip was inferred from deformation-induced changes in the patterns of diffracted X-ray intensity [5,6], and in

* Corresponding author.
E-mail address: pagan2@llnl.gov (D.C. Pagan).

http://dx.doi.org/10.1016/j.actamat.2016.06.020
1359-6454/

fact served as motivation for long-lasting theories of slip and dislocation motion [7]. Typically when studying slip with X-ray diffraction, models are necessary to relate slip activity to quantities that are measurable through diffraction processes, such as elastic strain or lattice orientation. Recent efforts studying heterogeneous slip activity within individual crystals generally rely on using models to relate diffracted intensity to the density fields of dislocations present in the crystal. Distributions of elastic strain [8,9,10] or misorientation [11,12] produced by the dislocation density fields are measured and subsequently used to determine the dislocation density, dislocation type, and active slip systems from the diffraction data. Approaches to understanding slip through analysis of distributions of lattice strain or orientation each have their own benefits. Strain based methods are sensitive to both statistically stored (SSD) and geometrically necessary (GND) dislocation types, making them well-suited for understanding the development of material strength. A downside of this approach is that there is typically insufficient instrument resolution to associate distributions of strain to sub-volumes of the region probed when analyzing line profile data (intensity versus 2θ). Unlike distributions of strain, distributions of lattice orientation are only sensitive to GND content present in the diffraction volume. However, sub-volumes deforming by different slip systems will have marked differences in GND content and if sufficiently misoriented, can be identified and isolated in distributions of lattice orientation. The ability to differentiate sub-volumes of a crystal makes orientation based methods attractive for studying heterogeneity of slip at sub-crystal length scales, and subsequently, the precursors to material failure.

With the increased use of modern synchrotron sources for studying the mechanical deformation of engineering materials, X-ray experiments to study slip processes are becoming increasingly important. These sources, with their ability to provide high-energy X-ray beams at enormous flux, combined with large, fast area detectors are enabling rapid measurements of complete three dimensional distributions of diffracted intensity. These new diffraction data, along with *in-situ* sample environments are enabling a new generation of mechanical tests in which the evolution of quantities associated with plastic deformation, including distributions of elastic strain [13,14,15] and crystal orientation [16,17,18], can be studied with significantly finer temporal and spatial resolution [19,20]. New experiments and data processing models designed for use at synchrotron sources promise to advance our capability to experimentally measure slip processes and improve our understanding of crystal plasticity in manners not possible using optical or electron microscopy methods.

In this paper, we use the enormous quantity of diffracted intensity data that is possible to acquire at a synchrotron light-source, along with a kinematic model of crystallographic slip, to correlate measured single crystal pole figures with slip system activity. A single pole figure only provides information about the orientation distribution from a set of lattice planes, but from multiple pole figures, heterogeneity of slip can be quantified. We present experimental results from a single crystal silicon compression specimen deformed at high temperature, with silicon serving as a model material for other materials, including metals. In §2, an introduction to single crystal pole figures and how their basic features relate to misorientation distributions within a crystal is given. A kinematic model is introduced to relate the evolution of single crystal pole figures to heterogeneity of slip. Then a description of the experiment geometry and the process to produce single crystal orientation pole figures from area detector diffraction data is in §4. §5 presents single crystal pole figures measured *in-situ* from a plastically deforming silicon single crystal and the evolution of lattice misorientation as the crystal deformed. In §5.1 the slip model is used to determine heterogeneous slip activity from the deforming silicon crystal. In §6, the slip activity results are discussed and extension of the presented experimental method to the study of grains in polycrystals is described.

In this paper, bold characters will indicate vectors and tensors ($\boldsymbol{a}$, $\boldsymbol{A}$). A vector in the undeformed configuration is denoted with an overbar $\overline{\boldsymbol{a}}$. The vectors $\boldsymbol{e_x}$, $\boldsymbol{e_y}$, and $\boldsymbol{e_z}$ will be reserved for basis vectors of rectangular Cartesian coordinate systems. Superscripts will denote the coordinate systems in which vectors or tensors are expressed ($\boldsymbol{a}^S$), except T reserved for a transpose operation. The superscripts S, L, C, and DET denote the sample, laboratory, crystal, and detector coordinate systems respectively. Subscripts will generally identify specific scalars, vectors or tensors (a_0, $\boldsymbol{a_0}$, $\boldsymbol{A_0}$). The dot product of a second order tensor and a vector is $\boldsymbol{A}\cdot\boldsymbol{b}$ ($A_{ij}b_j$), while the dot product of two second order tensors) is defined as $\boldsymbol{A}\cdot\boldsymbol{B}$ ($A_{ij}B_{jk}$). The vector outer product is defined as $\boldsymbol{a}\otimes\boldsymbol{b}$ (a_ib_j). The operation of building a skew-symmetric matrix from a vector is defined as $\boldsymbol{a}\times\boldsymbol{I}$ ($\varepsilon_{ijk}a_k$ where ε is the permutation symbol).

2. Single crystal orientation pole figures and distributions of orientation

Distributions of lattice orientation within individual crystals are a by-product of slip processes, so through measurement of these distributions and their evolution, we can infer information about the underlying slip processes. The measurement of distributions of lattice plane normals with respect to an external coordinate system, or pole figures, is one of the primary tools for quantifying lattice plane orientation non-destructively in crystalline materials [21]. Building a pole figure for a particular family of lattice planes (denoted by Miller indices $\{hkl\}$) begins with determining the intersection of the plane normal $\boldsymbol{n}$ (a pole) with a unit sphere. The sphere represents every possible plane orientation with respect to an external coordinate system. Fields expressed over a pole figure indicate the presence of a plane in a particular orientation or a physical quantity, for instance, lattice strains [22,23,24]. A pole figure associated with a family of lattice planes $\{hkl\}$ ($P_{\{hkl\}}$) is most commonly used to indicate the probability of finding lattice planes in a particular orientation [25,21], and has also been used to find orientation distributions in single crystals [26,27]. There is a close connection between orientation pole figure data and X-ray diffraction measurements: every value on a pole figure represents a crystal volume containing lattice planes with normal $\boldsymbol{n}$, and each crystal volume containing lattice planes with that normal will contribute to the measured diffracted intensity.

In contrast to traditional studies of textures in fine-grained polycrystalline samples, when diffraction peaks from individual crystals can be resolved, pole figures can be generated for specific sets of lattice planes, rather then symmetrically related families. On these single crystal orientation pole figures (SCPFs), plane normal densities are zero for most possible lattice plane orientations (see Fig. 1). As such, most of the SCPF can be neglected and focus is placed on non-zero values clustered within a few degrees of the average plane orientation. In this work, the small region of interest is shown with an aerial view of the pole figure. Fig. 1 illustrates a more familiar complete pole figure and a magnification of the region of interest as an example to acclimate the reader to the data presented. From this point onward, only SCPFs are discussed, so the parentheses indicating a specific set of lattice planes, rather than a family of lattice planes will no longer be included ($P_{(hkl)} = P_{hkl}$).

When studying crystallographic texture using pole figures, all spatial information about the position of a grain with a specific orientation is lost. This is also true for SCPFs: the spatial arrangement of probed crystal volumes does not influence the SCPF and only the angular distribution of lattice plane orientation is measured. However, if some degree of lattice continuity is assumed