Current Opinion in Solid State and Materials Science 18 (2014) 286–299

Contents lists available at ScienceDirect

Current Opinion in Solid State and Materials Science

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

Understanding local deformation in metallic polycrystals using high energy X-rays and finite elements

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ARTICLE INFO

Article history:
Received 22 May 2014
Revised 24 August 2014
Accepted 9 September 2014
Available online 26 September 2014

Keywords:
Elastoplastic deformation
X-ray diffraction
Finite element models
Stress state
Material design

ABSTRACT

A methodology for understanding the stress and elastoplastic deformation responses within a loaded polycrystal is presented along with illustrative examples. High energy synchrotron X-rays are used to penetrate bulk metallic samples and produce diffracted intensity from each deforming crystal – revealing the evolving internal structure. A virtual representation of the microstructure is constructed using the finite element method (FEM) to simulate the evolution of the elastoplastic deformations, stress fields, and lattice orientations within the deforming crystals as the polycrystal is loaded. Simulations are compared directly to experimental diffraction data. In the case of powder experiments, lattice strain pole figures (SPFs) measured experimentally are compared to SPFs calculated by projecting X-rays through the finite element mesh. During *in situ* loading experiments, the stress states are found to differ from one crystal to the next and to vary from the stress being applied at the macroscale. A SPF/FEM-based methodology for quantifying residual stress fields within processed polycrystalline components is described. SPFs were measured at many points within a shrink-fit sample. Finite element discretizations of both the sample and orientation space of each diffraction volume were used to formulate an optimization for the distribution of the stress tensor within the sample. A different experiment, one in which the X-ray beam and the crystals are closer to the same size, is used to investigate the aggregate crystal by crystal. The Debye–Scherrer rings reduce to a set of spots associated with each crystal within the diffraction volume. This method is demonstrated by tracking deformation of four grains within a deforming BCC titanium aggregate loaded *in situ* within the elastic regime to determine the single crystal elastic moduli. Plastic deformation can also be investigated by monitoring the size and shape of individual diffraction spots. Each spot contains geometrically exact information regarding the internal structure of the crystal. Instead of reconstructing the crystal structure by inverting the diffraction data, virtual diffraction experiments are performed on the finite element mesh and the resulting simulated diffraction patterns are compared directly to the experimental results. Once the experimental/simulation methodology is validated, the approximation of the subgrain distribution of stress and lattice orientation from the finite element model can be used to construct theories for failure phenomena such as microcrack initiation. As opposed to other methods of discretizing a polycrystalline aggregate, the finite element framework enables a seamless transition to analyses associated with mechanical design.

1. Introduction

Stress is the most pervasive quantity used in the design and implementation of structural materials. Stress serves as the basis for most mechanical failure models. Distributions of stress are carefully computed using sophisticated simulations – often finite element models – before a bridge, building, aircraft or automobile design is deemed safe. The models are carefully calibrated using experimental data and, when possible, experimental stress analysis techniques are used at key locations to verify the computed stress values. The structural mechanics and forensic mechanics literatures are rife with examples where the actual stress state (computed or measured) within a complex structure was very different than what "intuition" might suggest. When it comes to strength of a structure or the safety of passengers, accurately quantifying stress is of upmost importance. At the microscale (the size scale within a macroscopic "material point"), stress drives nearly all of the relevant physical behaviors for a structural material including elastic and plastic deformation, fatigue and fracture. However, due to the seemingly inaccessible nature of the relevant structures at the size scale of the individual crystal lattice, the discussion and

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http://dx.doi.org/10.1016/j.cossms.2014.09.001
1359-0286/

descriptions of stress are often transformed into overly simplistic arguments that are generally lacking in mechanistic rigor. Stresses are often described as being either long or short range; upon yielding, crystals are said to "shed" their loads and transfer some of their stress to surrounding "stronger" crystals; stress is often identified with the load divided by area quantity of a uniaxial test specimen and anisotropic crystals inside a uniaxial test specimen are assumed to also be under a uniaxial stress state; the three-dimensional nature of Hooke's law is often ignored, a component of stress is determined by multiplying a single lattice strain by Young's modulus. Remarkable progress has been made in the science of structural materials in the last century using such plausible yet simple notions of stress. However, in applications where failure initiates on the crystal scale such as fatigue or fracture, significant gains could be realized in a vast expanse of mechanical designs with a more mechanically correct and, more importantly, experimentally verifiable understanding of crystal-scale stress fields, stress fields that are fully consistent within a rigorous theoretical framework.

Images and imaging have played the dominant role of enabling discovery in material science. Advances in the understanding of deformation and damage processes in structural alloys have largely paralleled the development of experimental characterization tools such as light or electron microscopy, designed to observe the internal structure of material. Clearly, important understanding of the *structure* of a polycrystalline alloy has emerged from imaged-based data. The time-honored method of correlating mechanical properties of an alloy such as strength or ductility, with a structural model enabled by a particular image or set of images of the microstructure has been possible only through the advances in microscopy, with ever more powerful microscopes enabling finer resolution and increased detail.

Using serial sectioning or near-field X-ray techniques, it is now possible to create three-dimensional models of the microstructure, possibly including information such as lattice orientation and chemical composition. Regardless of its detail, however, an image-based model of the microstructure cannot provide direct information about stress state or other mechanically relevant quantities on its own. It is up to the investigator to create the micromechanical descriptions that link the microstructure to mechanical properties. Most images are taken forensically (*ex situ*) so now that story must involve the material evolution that preceded the image; the challenge of understanding the micromechanical response of a polycrystal on all its relevant size scales is made more complex by the microstructural evolution that takes place during elastoplastic deformation, for instance. Traditionally, the image-based model of a microstructure is paired with a set of mechanical properties but truly *predicting* the mechanical properties of a polycrystalline alloy from a thorough set of micrographs is only possible in a few simple cases of interpolation between microstructural states. Extrapolation is not possible. This fact is the primary reason that the design of new structural materials remains a time consuming, resource intensive, trial and error process. The goal of rethinking how alloys are designed and chosen in design served as partial motivation for the Integrated Computational Materials Engineering (ICME) [1] study done by NAE and the Opportunities for Mesoscale Science document authored by the BESAC committee of DOE [2]. Accelerating the material design process also served as the primary motivation for The Material Genome Initiative (MGI) [3].

This paper describes methods of enhancing the structural description that comes from traditional material characterization approaches by combining high energy X-ray diffraction data with finite element models to produce a representation of the real time **response** of a deforming polycrystal; one that, for a given set of boundary conditions, can be used to quantitatively understand relationships between descriptors of the microstructure such as grain[1] morphology and orientation as well as mechanically-relevant response variables such as stress. It is our opinion that we must focus on stress and its evolution during deformation if we are to improve the performance of load-bearing materials and to do this, we must augment material structure information with its response to loading. Further, there needs to be a rigorous theoretical framework used to interpret these data in the context of behaviors that define stiffness, strength and ultimately failure.

2. High energy X-rays and finite elements

X-rays have been used for interrogation and characterization of structural materials for a century. The discoveries behind X-ray diffraction and the overarching principles that basically enabled the field of modern crystallography are all based on the fundamental relationships that exist between diffracted X-ray intensity and the underlying material structure. The tremendous utility of X-ray diffraction experiments is the ability to utilize a *diffraction model* to convert a collection of diffracted X-ray intensity into a quantitative, often geometric description of the material structure that produced it. The earliest of these models, Bragg's law and the Laue equations, continue to be used as the building blocks for data reduction (inversion) models that contain more comprehensive representations of materials as instruments advance and understanding deepens.

A high energy synchrotron light source produces well-characterized, concentrated beams of monochromatic high energy (short wavelength) X-rays that are capable of penetrating through bulk metallic samples. Capturing large patterns of diffracted X-rays is now possible using modern area detectors with increasingly fast (sub-second) detection times and ever smaller pixel size. By employing ancillary equipment that can precisely control external loads, temperature and other environmental conditions, diffraction patterns can be obtained in real time from a test specimen subjected to conditions that closely approximate those experienced during processing or in service. Fig. 1(a) depicts a schematic of a high energy X-ray diffraction (HEXD) experiment at a synchrotron light source experimental station. A relatively small (hundreds or thousands of microns square) monochromatic beam of X-rays passes through the polycrystalline specimen diffracting from suitably oriented lattice planes. The specimen can be loaded and heated *in situ* to mimic processing or service conditions. To adequately interrogate all crystals within the diffraction volume, the sample is rotated (ω in Fig. 1(a)) using a precision diffractometer. The sample to detector distance (D in Fig. 1(a)) dictates the character of the diffraction data. A highly resolved detector placed near the sample (millimeters) can be used to construct three-dimensional grain maps. A larger detector placed farther from the sample (meter) collects data that are much more sensitive to the geometry of the unit cell; changes in the distributions of orientation and strain can be discerned from the evolution of such "far-field" diffraction patterns, making these data particularly attractive for understanding mechanical response. Coupled with the capability of capturing diffracted X-rays from every part of an evolving polycrystal many times every second, comes the challenge of constructing diffraction models with the same level of sophistication as the experimental apparati and techniques. To be of most utility, these models must contain accurate representations of (i) the material, (ii) the diffraction conditions and (iii) the loading/heating/environmental conditions. Accuracy includes the appropriate representations of the spatial and temporal heterogeneities of all three. In the work

[1] Within this manuscript the terms crystal and grain are used interchangeably.