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New opportunities for quantitative tracking of polycrystal responses in three dimensions

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ABSTRACT

An important advance in understanding the mechanics of solids over the last 50 years has been development of a suite of models that describe the performance of engineering materials while accounting for internal fluctuations and anisotropies (ex., anisotropic response of grains) over a hierarchy of length scales. Only limited engineering adoption of these tools has occurred, however, because of the lack of measured material responses at the length scales where the models are cast. Here, we demonstrate an integrated experimental capability utilizing high energy X-rays that provides an *in situ*, micrometer-scale probe for tracking evolving microstructure and intergranular stresses during quasi-static mechanical testing. We present first-of-a-kind results that show an unexpected evolution of the intergranular stresses in a titanium alloy undergoing creep deformation. We also discuss the expectation of new discoveries regarding the underlying mechanisms of strength and damage resistance afforded by this rapidly developing X-ray microscopy technique.

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Introduction

Many critical engineering materials, such as metals and ceramics, are polycrystalline. As such, they are inherently heterogeneous over a hierarchy of length scales ranging from the engineered component, to the distribution of different crystalline grains, phases, voids and/or secondary particles, and down to the distribution of lattice defects at the atomic scale. This structural heterogeneity manifests as anisotropy in functional material properties such as stiffness, strength, conductivity, and damage resistance. However, engineered products are almost universally designed using idealized homogeneous representations of the material at the macroscopic scale. While this approximation scheme permits unambiguous linkages between boundary condition constraints and material response for a range of structures such as bridges, aircraft, and electronic devices, it is often quite limited in terms of predictive capability.

More recently, much interest has been paid to materials science at the so called "mesoscale" [1,2]. While not an explicit length scale, the mesoscale can be thought of here as a length scale over which homogenization breaks down, and where materials properties are the result of an ensemble of constituents. In the present context, this length scale encompasses an ensemble of individual crystallites or "grains", possibly including a distribution of voids and secondary phase particles. Each grain or constituent expresses directionally dependent properties (e.g. strength, stiffness, electrical conductivity, etc.) stemming from the atomic compositions and structure along with crystal defects. The metrics of these ensembles, referred to as the *microstructure* of the material, result in the internal forces of a loaded body being distributed quite heterogeneously. In turn, these heterogeneities drive the emergent behaviors of creep, crack initiation and fracture. With few exceptions, mesoscale information has not been incorporated into the design process, not because mesoscale modeling tools do not exist, but rather because of a lack of appropriate experimental data and methods with which to validate such models, leaving an unacceptable level of risk for a design engineer.

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One approach toward capturing the necessary experimental data is integration of *in situ* mechanical testing with advanced characterization methods to provide full 3D microstructural characterization of the test volume, in which ensembles of individually resolved grains are tracked throughout an experiment [3]. Measuring a sufficiently large number of grains is important, not only to characterize the heterogeneities in internal stresses as they develop, but also to facilitate detection of rare events such as void/crack nucleation. This information could be used to validate predictive models that explicitly represent 3D microstructure [4]. In this work, we describe a novel capability to nondestructively characterize the evolving microstructure and micromechanical state of deforming polycrystalline ensembles through concurrent integration of three high energy synchrotron X-ray techniques. We present first-of-a-kind results for a titanium alloy specimen undergoing time dependent creep deformation that reveal a complex redistribution of internal stresses during the creep process.

Methods

High energy diffraction microscopy (HEDM)

The enabling technology for performing the measurements presented herein is high-energy synchrotron radiation. High-energy synchrotron sources provide a unique blend of high brilliance, high-energy (>50 keV) radiation that enables nondestructive evaluation of microstructure and micromechanical state in bulk (penetration depths on the order of millimeters) engineering materials. For reference, the brilliance of these light sources can be 6–10 orders of magnitude greater than that of a laboratory X-ray source. Over the past 15 years, several experimental techniques capable of independently mapping structure and grain-by-grain mechanical response have been developed. In order to study "bulk" phenomena, as well as the relevant statistics to capture critical events (void/crack nucleation), it is necessary to measure on the order of 1000 grains. The most prevalent techniques capable of spatially resolved measurements of microstructure and micromechanical state for aggregates that large are tomography and a class of diffraction-based measurements that are built upon the rotating crystal method [5,6]. These techniques consist of interrogating a sample with monochromatic X-rays while the sample is continuously rotated and images of diffracted beams are collected on area detectors in transmission geometry over discrete angular intervals [7–10], and are alternately referred to as three-dimensional X-ray diffraction microscopy (3DXRD) or high-energy X-ray diffraction microscopy (HEDM). These techniques differ from polychromatic methods such as differential aperture X-ray microscopy (DAXM) [11], which consists of point-by-point measurements and uses micro-focused beams of lower energy polychromatic X-rays (8–35 keV) to probe three dimensional structure relatively near sample surfaces. The polychromatic methods offer excellent intragranular resolution, but are less suited for characterizing large ensembles of interior grains.

The three X-ray techniques we have integrated are referred to as far-field HEDM (ff-HEDM) [12–15], near-field HEDM (nf-HEDM) [8,16,17], and absorption micro-computed tomography (μ-CT) [18]. These techniques result in correlated data that (1) quantify an average elastic strain tensor (stress tensor with known elastic stiffness matrix) for each grain, (2) map the structure and local crystallographic orientation within and between grains, and (3) permit observation of the structure of voids, cracks, and second phase inclusions, respectively. In each case, raw data consists of images of diffracted or transmitted X-ray beams collected on area detectors placed at different working distances while the specimen is rotated and irradiated with a monochromatic X-ray beam. The experiment was conducted at the high energy beamline 1-ID-E at the Advanced Photon Source (APS), Argonne National Laboratory using an X-ray energy of 65.4 keV. While measurement hardware, procedures, and analysis software have been developed independently for each technique, the ability to collect unified correlated datasets yields a more complete view of the evolving material that is greater than the sum of the independent results.

Far field high energy diffraction microscopy (ff-HEDM)

The ff-HEDM technique provides the average elastic strain tensor (from which average stress tensors can be calculated assuming linear elasticity), the average crystallographic orientation, and the center of mass position for individual grains within a deforming polycrystalline sample [12–15]. The data reduction consists of a back-projection method where individual diffraction spots are first identified by segmentation of the detector image, subsequently associated with one or more Debye–Scherrer rings using a specified space group and angular tolerance, then finally associated with a uniquely oriented crystal lattice through an indexing operation. Once a set of orientations is obtained, the 12 degrees of freedom that describe the orientation (3), position (3), and elastic strain (6) of an individual grain are optimized, in a least-squares sense, using the subset of measured spot centroids associated with it from the indexing [15]. The detector is positioned "far" (∼1 m) from the specimen to improve angular resolution of the diffraction pattern and thus provide high elastic strain sensitivity. Typically the strain resolution of this technique is quoted to be $\pm 1 \times 10^{-4}$. This value is directly coupled to experimental conditions and is often conservative [19,20]. It is important to note that these measurements differ from conventional 'aggregate' or so called powder experiments since each diffraction spot originates from and is assigned to an individual grain within the specimen while accounting for precession during the rotation of the specimen.

The selection of the X-ray beam size with respect to the average grain size in the specimen dictates how the ff-HEDM results should be interpreted. If the beam size is large enough such that the grains of interest are fully irradiated, then the center of mass, average orientation, and the average elastic strain tensor represent grain averaged quantities. However, in the current work we used a line focused X-ray beam as wide as the specimen (∼1.4 mm on the diagonal) but only ∼2 μm tall (along the tensile axis). The novel application of the line focused beam for the ff-HEDM technique provides the average elastic strain and stress tensors for the irradiated portion of each grain, i.e. the grain cross-section averaged (GCSA) elastic strains/stresses, resulting in sub-grain resolution in the direction orthogonal to the plane of the beam.

Near field high energy diffraction microscopy (nf-HEDM)

The nf-HEDM experiment nondestructively characterizes the microstructure (crystallographic orientation, size/shape/relative position of each grain) within the diffracting sample [8,16,17]. The measurement presented herein employed the same line focused X-ray beam as for ff-HEDM (∼2 μm by the width of the sample) to scan a 'layer' of the material. A high resolution detector images the shapes of diffraction spots as the sample rotates. A specimen volume is mapped by translating the sample perpendicular to the beam plane to illuminate successive layers. Diffraction measurements from each layer are measured at multiple sample to detector distances such that diffraction spots are seen to radiate away from the grain of origin, thus encoding the grain position as well as the scattering angles. The salient feature of the experimental geometry is that the detector needs to be "near" the specimen (within 10 mm), providing greater sensitivity to grain position rather than diffraction angle. The data reduction consists of iteratively comparing a forward model of potential crystal orientations for each volume element (voxel) within the sample to the measured