Sub-grain orientation resolution during continuous loading using only far-field HEDM

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Abstract. A far-field data reduction algorithm for high-energy X-ray diffraction microscopy (HEDM) has been adapted to extract sub-grain orientation descriptors in a polycrystalline material during continuous \textit{in situ} loading experiments. Previously, the standard data reduction algorithm would only extract grain-averaged strains, orientation, and the centroid position. A new descriptor, the grain orientation envelope (GOE), is introduced as a measure of the intragranular microstructure extracted from every grain during a continuous loading experiment. Initial results showing the evolution of a GOE from one grain in the titanium alloy, Ti-7Al, during a tension test is presented as a demonstration of the nature of the data.

1. Introduction

Establishing the relationships between microstructure and the thermomechanical response of complex engineering alloys is critical for alloy selection, development, and adoption. Historically, these relationships have been based on decades of empirically derived behaviors, rather than fundamental mechanistic understandings. Even with modern advances in characterization techniques and computational tools, there are still hurdles to reaching this mechanistic understanding such as: bridging characterization techniques between multiple length-scales, simultaneously assessing macroscopic response and microstructural evolution, and identifying significant features that should directly inform predictive models.

Titanium alloys are a favorite among a diverse group of industries for their excellent mechanical properties, relatively low density, and outstanding corrosion resistance even at moderate temperatures. In service, these alloys are often subjected to extreme conditions, such as the complex thermomechanical cycles within a gas turbine engine. These complexities, in addition to those imposed intrinsically by the material response - including the elastic and plastic anisotropies associated with hexagonal metals - make titanium a particularly challenging system for investigating the underlying relationship between microstructures and thermomechanical properties. One response that notably proves challenging for both material performance and characterization is the susceptibility of many titanium alloys to creep and stress relaxation at room temperature. This phenomenon is thought to be one of the primary sources leading to a significant failure mode known as cold dwell fatigue, where cracks nucleate and grow in low temperature environments. In order to understand the fundamental connections between failure modes like cold dwell fatigue and microstructure, the kinetics of deformation and microstructural

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response must be understood, ideally by obtaining both sets of information simultaneously. Unfortunately, room temperature creep (and stress relaxation) not only affects the performance of the material, but the ability to measure microstructure under an applied load. Traditional microstructure characterization methods, which encompass spatial mapping techniques and analysis of defects and chemistries, either require the loading to be paused during data acquisition or are destructive in nature. The collection times of these traditional techniques are on the order of minutes to days. For non-destructive techniques, the acquisition time constraint combined with the room temperature creep of titanium alloys leaves two options for \textit{in situ} experiments: (i) hold the applied load during the measurement, or (ii) reduce the load away from the yield surface to take the measurement. Neither option is ideal considering that inelastic deformation proceeds at any non-zero stress level. High energy X-ray diffraction during continuous loading overcomes these constraints and offers an opportunity for the simultaneous extraction of stress state and microstructural evolution.

High-energy X-ray diffraction microscopy (HEDM) performed at a synchrotron light source enables \textit{in situ} bulk analysis of crystal-scale stresses and orientations for all crystals in a diffraction volume (hundreds to thousands of grains) [1, 2]. HEDM has two primary experimental modes: far-field, which is sensitive to orientation and strain, and near-field, which is sensitive to position [3]. As their names suggest, a detector is placed far from the sample at \( \approx 1 \) m downstream for the far-field technique, and a detector is placed near to the sample at \( \approx 5 \) mm downstream for the near-field technique. While the near-field technique produces 3D spatially resolved grain maps, collection times are on the order of hours for relevant volumes and therefore this technique cannot be performed \textit{during} continuous loading. The shorter acquisition times for far-field HEDM experiments, however, enable sufficient data collection for some continuous loading experiments, admittedly at relatively slow strain rates. From these experiments, the real-time grain-scale stress evolution is readily extracted using data processing algorithms, which for far-field HEDM are optimized to extract grain-averaged data, with the primary output being an elastic strain tensor, the grain centroid, and the grain orientation. This strain tensor can then be used to calculate a grain-averaged stress, capturing the stress evolution during continuous loading without ever pausing the experiment, albeit with slight change in load.

In this manuscript, the standard far-field HEDM data reduction algorithm [1, 4] is modified to capture the evolution of intragranular orientations. A quantity we call the grain orientation envelope (GOE) is introduced. Results for a Ti-7Al alloy are used as the example throughout the paper, but the GOE is discussed broadly for application in any polycrystalline metal.

2. Far-field HEDM: Experiments

General aspects of a far-field diffraction experiment are presented below, followed by details of the Ti-7Al alloy experiment used for analysis. As can be seen from the schematic in Figure 1, an area detector is placed \( \approx 1 \) m from the sample while a relatively large box-beam (achieved through orthogonal beam-defining slits) is transmitted through a large volume of the material. We note that it is imperative that diffraction peaks be \textit{distinct} and not overlap one another for this technique. To capture the behavior of each crystal in the volume, diffraction events from multiple families of lattice planes (\( \{\text{hkl}\} \)) are required. This is achieved through rotation of the crystal, with each revolution of 360 degrees capturing the average response over the duration of the revolution. To avoid halting the experiment for data collection, the sample is continuously loaded and rotated while the detector collects diffraction images.

2.1. Ti-7Al Experiment

The Ti-7Al alloy was machined into the geometry shown in Figure 1(a). A schematic of the experiment is presented in Figure 1(b). Two Dexcela 2923 detectors (74.8 \( \mu \)m effective pixel pitch, 3888 pixels x 3072 pixels) were positioned 884 mm downstream of the sample. An example image
from one of the detectors, Figure 1(b), shows the necessary discrete diffraction peaks. The sample was continuously loaded in uniaxial tension at a constant cross-head displacement velocity of $1 \times 10^{-5}$ mm/s. The experiment was stopped at a final macroscopic strain of 0.03. Throughout loading, the sample was continuously rotated, with diffraction images taken every 0.25 degrees for a total of 1440 frames per 360 degree revolution. Each revolution, or measurement point, took 5 minutes, averaging over an $\approx 0.0005$ spread in strain. A total of 54 total measurements were collected throughout the duration of the experiment. The macroscopic strain was measured using digital image correlation in the gauge region and the load was monitored using a load cell placed above the specimen.

3. Far-field HEDM: Data Reduction
The HEXRD data reduction package was used in this study, details of which can be found in [4]. This section will first discuss the standard data reduction scheme for obtaining the grain-averaged data, followed by the presentation of our new intragranular orientation extraction method.

3.1. Grain Averaged Orientation, Position, and Stress
After calibration, the HEXRD processing algorithm begins with an indexing routine, followed by a peak fitting routine. In the indexing step, the alloy crystal structure, experimental geometry, and X-ray energy are used to simulate diffraction events expected on the detector; for a given lattice orientation, the positions and angles of individual diffraction peaks are simulated. A large number ($\approx 1$ million) of possible discrete orientations, seeded from crystallographic fibers of orientation determined from peaks on the detector, are generated. The peaks simulated using the seeded orientations are compared to the experimental data. A ‘completeness’, defined by the ratio of how many expected peaks from the simulation were measured in the experimental data, is assigned to each orientation. Orientations that are closely clustered together are considered one orientation. In this study, a completeness of 0.98 indicated a high likelihood that the crystal orientation was present in the diffraction volume. After all of the orientations within the diffraction volume have been identified, a peak fitting algorithm identified and optimized the peak centroids to refine the grain averaged orientation, grain centroid, and elastic strain tensor.

Figure 1. Schematic of continuous-loading far-field X-ray diffraction experiment.
This procedure is repeated for every load step, with the refined values of grain orientation, grain centroid, and elastic strain tensor used as the starting values for refinement of each subsequent step.

The data reduction has been limited to extracting *grain-averaged* data since the peak-fitting algorithm is designed to track peak centroids. To illustrate this point, Figure 2 shows a schematic of two diffraction peaks on a dashed Debye-Sherrer ring. Figure 2(a) represents a specific peak for a set of lattice planes which have almost no misorientation, such as would be the case for a well annealed grain before plastic deformation. As metal alloys plastically deform, misorientations develop within the grain. This spread in orientation manifests as an azimuthal spread in the diffraction spot of the same peak in Figure 2(b). Of course, although these spots are spreading, the peak centroid is still what is measured in the peak fitting routine, indicated by purple cross-hairs in Figure 2. Therefore, any information about the morphology of the peak and intragranular spread in misorientation is being ignored. It is precisely this peak spread and identifying the contributing additional orientations, whose peak centroids are indicated by white cross-hairs in Figure 2, that is addressed in the following section.

![Figure 2](image.png)

**Figure 2.** Schematic of diffraction peak for (a) undeformed and (b) plastically deformed metal. Dashed line indicates Debye-Sherrer ring, dark cross-hairs indicate centroid of overall peak, white cross-hairs indicate centroids of slightly offset (hkl) planes, such as would arise from misorientations within a grain.

### 3.2. Intragranular Orientations

To quantify the misorientation developing within a grain during plastic deformation, an additional indexing routine was developed to find all orientations present within a grain, not just the average. The method begins by first running the grain-averaged indexing and fitting routines to identify all unique grains in the diffraction volume. Then, for each grain, a series of new test orientations is generated by discretizing continuous points in orientation space (angle-axis parameterization) around the average orientation. The bounds are defined by the orientation spread in the material at the final load of the experiment, in this case 5 degrees. This new list of possible orientations for each grain is simulated in the indexing algorithm, and once again a ‘completeness’ score assigned based on the comparison to experimental data. Unlike the previous algorithm, no clustering is performed, and ‘completeness’ scores for trial orientations are recorded. We assume the orientations have a probability of existing in the grain commensurate with their completeness value. Similar to the grain-averaged dataset, a threshold completeness is decided upon to generate a list of orientations most likely to exist within the grain. A threshold of 0.98 was used in the Ti-7Al experiment. It is important to mention here that this analysis does not take into account the intensity of the peaks, and therefore only produces a list of unique orientations. In other words, no frequency of occurrence for each orientation is obtained.
Figure 3. Grain orientation envelope (GOE) in Rodrigues space with all of fundamental region (left) and magnified region (right) for Grain No. 143 at a macroscopic strain of 0.015.

Figure 4. Macroscopic stress strain curve with circles corresponding to start of far-field diffraction measurements. GOEs of grain No. 143 plotted for incremental measurements, colored according to their corresponding macroscopic strain. Inset: GOEs for grain No. 143 are plotted in Rodrigues space.
3.3. Grain Orientation Envelope
The list of orientations identified as belonging to a grain is defined as the grain orientation envelope (GOE). While related to a grain-scale orientation distribution function (ODF), the GOE contains only unique orientations that exist within the grain and therefore is the envelope of the distribution function. An important caveat of the GOE is that it is obtained entirely from far-field data and therefore is not spatially resolved - there is only knowledge of which grain the orientations belong to, but not where the orientations reside within the grain.

An illustration of the GOE in orientation space for grain No. 143 at 0.015 macroscopic strain in the Ti-7Al diffraction volume is plotted in Figure 3. The left side of this figure shows the GOE in the cubic fundamental region in Rodrigues space; the right side of the figure shows a magnified view of the GOE region. Each orientation is plotted as a point with the same color and value to emphasize that this is a single measurement as compared to a montage of measurements depicting the evolution of the misorientation which is shown next.

This GOE is extracted for every measurement point taken during the continuous loading experiment. Figure 4 shows the evolution of the GOE for grain No. 143. In this Figure, the macroscopic stress-strain curve is plotted, with open circles indicating the start of far-field scans. For a subset of these measurement points, the GOE is shown next to the stress-strain curve to demonstrate the size and morphological changes with increasing deformation. Each GOE is colored based on its macroscopic strain state; the colorbar is shown along the strain axis. A GOE is obtained for every measurement point, but only 15 of the 54 are presented for illustrative purposes. The size and shape of the GOE is shown to be relatively constant throughout the elastic region, followed by immediate expansion at yield, and then by growth and shape changes as the plastic deformation proceeds. The inset of Figure 4 shows an overlay of the GOE for each macroscopic strain measurement plotted in Rodrigues space. The centroid of the GOE is shifting over time, corresponding to an average rotation of the grain. These GOE shape changes and translations are measures of the microstructural evolution, not only providing a snapshot of orientation content at each time step, but providing a means to measure rotation rate changes.

The enormous utility of this method is that every grain in the diffraction volume has a corresponding GOE evolution history. For this Ti-7Al alloy, 792 grains were illuminated in the diffraction volume. As shown in Figure 5, during a continuous loading experiment every grain in the diffraction volume now can be probed for the evolution of its grain-scale stress tensor and orientation envelope. The far-field technique can be used to simultaneously examine deformation kinetics and to resolve microstructural evolution without the need to pause the experiment.

Figure 5. Spatial map of the diffraction volume (from near-field HEDM) and example stress tensor vs. macroscopic strain and orientation evolution for grain No. 143.
4. Discussion

A far-field data reduction algorithm was introduced which extracts a measure of the microstructure in the form of intragranular orientation spread. This modification is an expansion of existing data reduction routines already used to extract grain-scale strains and requires no modification of the HEDM experiment. The significance of the GOE measurements extracted from these data are now discussed.

4.1. Identifying important behaviors and regions of interest and informing models

Identifying which microstructural features are most important for understanding mechanical response and informing constitutive models is of great importance. With the increasing interest in utilizing big data sets and machine learning to independently identify behaviors and trends, it is important to have robust and representative data. Unfortunately, many characterization techniques can only capture small regions of interest at a time, with the added problem that identification of these regions is highly subjective. The GOEs provide a complete history of evolution for not only an isolated grain, but subsets of grains within a polycrystal including all grain neighborhoods. Because of the simultaneous extraction of stress and intragranular orientation evolution, the combined kinetic and microstructural response can be investigated directly for the hundreds of grains interrogated in a diffraction volume. This type of analysis can be used to identify regions of interest by qualifying which behaviors are representative, anomalous, or linked to particular material responses. These features can then be incorporated into modeling efforts.

HEDM datasets are already frequently paired with crystal plasticity models [5, 6] and the GOE can be readily computed from a simulated deforming polycrystal. This connection provides a directly comparable experimental and simulated result that can be used both to interpret the experimental GOE and validate the crystal plasticity model for a given system. Augmenting the GOE by including the intensity information to create the grain-scale ODF is under development, but the evolution of the GOE from every grain in real time provides unprecedented opportunities for the crystal scale modeling community.

4.2. Integrating spatially resolved data and electron microscopy

There are many useful measures of microstructure that, in practice, do not make use of a spatial component (e.g. dislocation densities, precipitate densities, grain sizes, textures, etc.). Like these quantities, the GOE has merit in its own right, but a full mechanistic understanding of microscale deformation also requires information about the microstructure which cannot be fully represented with non-spatial orientation information. The GOE can, however, be supplemented with other spatial techniques in a rather straightforward manner. Firstly, segmented grains from near-field HEDM maps and 3D EBSD serial sectioning can be, with some effort, correlated with far-field HEDM data [7, 6, 8, 9, 10, 11, 12]. These datasets provide spatial resolved orientation maps which can be of great importance for understanding orientation gradients and local features of interest in the material. Because it is non-destructive, the near-field mapping in particular has the potential to be taken both before and after the experiment, and at increments during an experiment should pausing the experiment be appropriate. Having the spatial map of the same diffraction volume not only provides context to the GOEs by identifying the full neighborhood of each grain, but also provides a road map for subsequent characterization techniques such as electron microscopy.

The microstructure characterizations mentioned thus far only provide information about the orientation, which is insufficient to understanding other important aspects of deformation, such as dislocation structures. Imaging of these structures often requires the use of scanning / transmission electron microscopy, which can be particularly difficult to pair with bulk studies due to the small nature of the sample extracted. Therefore, determining regions of interest and
understanding extraction geometries provided by the GOE histories and spatial map pairings can bolster these complementary techniques by providing context to a powerful, but small-scale sampling tool.

5. Summary

The grain orientation envelope (GOE) has been presented as a measure of the microstructure that can be extracted from far-field HEDM data during continuous loading experiments. An example of the GOE was shown for one grain in Ti-7Al, capturing the non-spatially resolved evolution of orientations up to a strain of 0.03. This measure of microstructure is extracted in addition to the average elastic strain tensor and centroid of a grain. Since a GOE is extracted for every grain in the diffraction volume, the GOE provides a means to discover regions of interest and identify regions for subsequent high-spatial resolution characterizations. In the future, incorporation of the intensity of the signal may be used to populate an orientation distribution function (ODF), rather than simply the envelope. The method presented shows promise for new micromechanical studies to directly link local stress states to microstructural evolution.

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