Capturing the Response of Polycrystalline Materials at the Mesoscale: Measurements, Modeling and Data Mining

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The development of next-generation engineering materials with transformational properties will rely heavily on the development of predictive models that can link microstructure and its evolution under thermo-mechanical processing with resultant properties/performance. These linkages – and the ability to synthesize "materials on demand" – have long encompassed a significant portion of research within the materials science community. However, without access to experimental data at the relevant length scales, validated models capable of predicting complex phenomena, such as creep, have remained elusive. Both the development of new material systems as well as the adoption of advanced computational methods in engineering design remains dependent on large-scale empirical testing efforts, which are as costly as they are time consuming. Emerging experimental techniques such as High Energy X-ray Diffraction Microscopy (HEDM) address a critical need with respect to validation experiments for multiscale materials models. HEDM measurements provide access to the microstructure and micromechanical state, both inter- and intra- granular, *in situ* for large polycrystalline aggregates. We present results from HEDM measurements as well as complementary finite element simulations of a Titanium specimen subject to uniaxial deformation in situ. The evolution of intragranular misorientations and intergranular stresses during continuous deformation are compared. The evolution of intergranular stresses under creep deformation is also presented. We conclude with a discussion on the burgeoning need for application of data mining techniques to these incredibly rich datasets, both in terms of advancing our understanding of emergent properties/responses at the mesoscale as well as in the context of informing the development/validation of multiscale modeling frameworks.

Interaction between toughness anisotropy and loading conditions of a drawn wire

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Large drawing deformations of a steel wire induce a strong hardening and an important crystalline and morphological anisotropy (see for instance [1]). This strongly influences the material behavior during subsequent loadings. Usually mechanical properties are studied by tension or torsion of drawn wires. The maximum tensile stress increases with the amount of prestrain, and it results mainly from the internal stresses after wiring (see for instance [1]). It is also shown that specific damage mechanisms develop in torsion after drawing, being linked to the microstructure morphological texture [2].

The present work deals with the understanding of the fracture mechanisms in a thin wire during complex loading paths (tension-torsion-bending). A dedicated experiment has been worked out to twist simultaneously two drawn wires. Finite element calculations allow the various stress components to be calculated within the wire sections. They account for the contact and friction stresses between the two wires, for the internal stresses, and for the wire mechanical behavior after large prestains. The longitudinal stress is in tension from the opposite side of the contact point to the center of each wire. The same is observed for hoop stress. The shear stress is rather high on a large part of the section, except close to the center where it is nearly zero.

The stress state is then related to the different fracture surfaces.

- The surface can be inclined with respect to the wire axis, the cracks following the elongated microstructure, or it can be flat and parallel to the wire section with a dimple character. These observations are explained by an interaction between the complex reloading stress state and the strong toughness anisotropy due to the morphological texture [3]. The shear stress at the wire surface allows the crack propagation along the axis. Then the tensile hoop stress leads to the crack propagation toward the center.
- On another hand, when the internal damage resulting from the wire drawing is large enough, the longitudinal stresses give rise to a ductile fracture of a weakened section, traverse to the wire axis.

These first results and calculations have to be completed accounting for the evolution of the internal stresses. This will lead to quantitative predictions of the rupture mechanisms as a function of the drawing amount and of the mechanical behavior of the wires.

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Virtual X-Ray and Electron Diffraction Characterization of Surfaces and Interfaces in Alumina

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Structural characterization of metal-oxide surfaces and interfaces poses a unique challenge for atomistic simulation. For example, in the case of alumina (Al_2O_3), typical atomistic simulation characterization techniques, such as common neighbor analysis and the centrosymmetry parameter are incapable of differentiating the subtle structural differences between stable and metastable alumina phases and thus would be similarly unsuccessful in characterizing interfaces in this material system. To address this challenge, a computational technique capable of calculating experimentally comparable x-ray diffraction line profiles and selected area electron diffraction patterns is developed. These virtual x-ray and electron diffraction techniques are employed to study alumina surfaces and both homogenous and heterogeneous alumina interfaces. Virtual selected area electron diffraction patterns and x-ray line profiles show unique features which differentiate between stable and metastable alumina phases, in agreement with experiment. Virtual diffraction characterization of the homogenous and heterogeneous alumina interfaces are used to study the atomic rearrangements that occur at the interface, with the virtual diffraction methods allowing for a direct validation via experimental data.

Theoretical and experimental X-ray spectroscopy of graphene-based supercapacitor electrodes under realistic operating conditions

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Electric double-layer capacitors, or supercapacitors, store charge via polarization at the electrode-electrolyte interface. Most models of interfacial charging focus almost exclusively on the electrolyte, including the transport, proximity, and arrangement of ions approaching the electrode surface. The electrode is considered fixed, with charge accumulation or depletion as the only response to polarization of the interface. We combine direct ab initio simulation of X-ray absorption spectra under applied voltage bias with corresponding in operando measurements to demonstrate that graphene-based supercapacitor electrodes in fact undergo unanticipated bias-induced electronic structural changes. The data strongly suggest that the origins of these changes arise from phenomena at two different length scales: first, local chemical changes at the nanoscale; and second, broader morphological changes at the mesoscale. Possible scenarios consistent with the observations will be discussed, along with implications for developing more accurate multiscale models of electrochemical operation.

A hierarchical study of grain boundary energy distribution using high-energy x-ray diffraction microscopy

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Studies of grain boundary energy distribution (GBED) have historically been done by measurement of dihedral angles for triple junctions or surface grooving experiments. The number of triple junctions measured typically limits the resulting statistics. Recent advances in 3D x-ray techniques, such as near-field HEDM (nf-HEDM), led to possibilities of probing mm^3 volumes at micron resolution non-destructively. A typical measurement captures ~1000 grains and roughly 10^7 sample points along the grain boundaries per volume. When combined with recently published semi-analytical grain boundary energy function, GBEDs can be examined hierarchically for different classes of boundaries. Here, a class of boundaries may define as the set of all low-CSL boundaries, the set of boundaries near triple junctions, or the set of all random high angle boundaries. The result demonstrates the vastly differing nature of boundaries under different local geometrical and topological constrains.

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3D Digital Reconstruction and Numerical Modeling of Microstructurally Small Fatigue Cracks in an Aluminum Alloy from Synchrotron-Based Measurements

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Advancing the states of materials design and high-fidelity structural prognosis will require a synergistic coupling between multi-scale experimental characterization and numerical modeling approaches. This talk describes recent efforts to couple synchrotron-based measurements of microstructurally small fatigue cracks with multi-scale modeling (mesoscale to macroscale) using three-dimensional finite-element simulations. Ex-situ techniques are employed to characterize three-dimensional fatigue-crack propagation within the microstructure of an aluminum alloy. The experimental characterization involves X-ray tomography along with near-field high-energy X-ray diffraction microscopy (HEDM), which provides a three-dimensional map of the grain morphologies and orientations adjacent to fatigue-crack surfaces. The experimental data is then used to digitally reconstruct and model the measured polycrystalline volume and fatigue-crack morphologies as a way to reproduce the observed crack evolution and thereby compute response fields in the neighborhood of observed crack fronts. Cracks are represented explicitly through the underlying geometry of the finite-element mesh. A concurrent multi-scale modeling technique is employed, whereby a model of the measured polycrystalline volume is embedded within a model of the global fatigue specimen. The mesoscale and macroscale regions are modeled using crystal plasticity and von Mises plasticity, respectively. Such simulation results can be used to inform and validate models that predict three-dimensional crack evolution at the microstructural length scale.

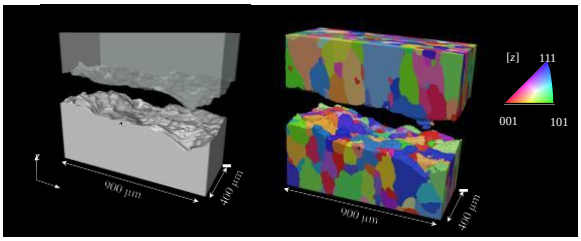

Figure 1. Reconstructions from X-ray tomography (left) and near-field HEDM (right).

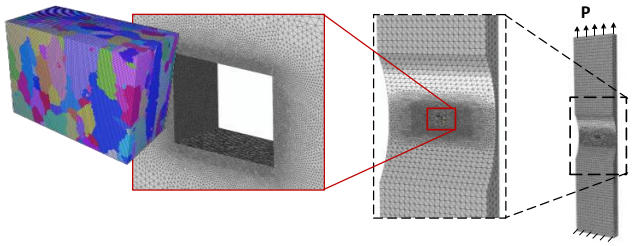

Figure 2. Concurrent multi-scalefinite element model with polycrystalline region from experiment.

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Three-dimensional imaging and numerical reconstruction of graphite/epoxy composite microstructure based on ultra-high resolution X-ray computed tomography

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Three-dimensional simulation of constituent-scale deformation, damage, and failure (i.e. virtual testing) in fiber reinforced polymers (FRPs) requires a detailed and accurate numerical representation of the underlying microstructure. Recent advances in high-resolution X-ray computed tomography (CT) have enabled 3D imaging of FRP constituents at a submicron resolution; however, converting X-ray CT-derived data into useful numerical models remains a significant challenge. Specifically, the relatively low contrast between individual constituents (i.e. fibers and resin), small fiber diameter (~ 5-7 μm), and dense packing (fiber volume fraction ~ 55-67%) in most FRPs render the available off-the-shelf, image-segmentation-based conversion codes inadequate. The work described here seeks to address the aforementioned challenges through a combined experimental and numerical framework aimed at high-resolution imaging, visualization, and numerical reconstruction of FRP microstructures at the fiber length scale. Three-dimensional imaging of an FRP microstructure is performed using a 3D X-ray microscope. A small "matchstick-type" sample of a unidirectional AS4/3501-6 graphite/epoxy composite is imaged with a voxel size of approximately 460 nm. Collection of the X-ray attenuated data and their conversion into a stack of two dimensional digital images is performed using commercially available software. A template-matching based detection algorithm is implemented to "detect" the centroids of individual fibers in each two-dimensional image. Next, a multi-fiber tracker which uses a Kalman filter and a Kuhn-Munkres assignment algorithm is implemented to track the individual fibers throughout the stack of images. The resulting coordinates of fiber centroids in three-dimensional space from the detection-tracking algorithms are then used to generate a solid-model reconstruction of the imaged volume. Finally, statistical analysis is performed to quantify the spatial variation in the microstructure.

Identification of a crystalline constitutive law using multimodal full-field measurements in grain scale

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The crystalline plasticity constitutive models are remarkable for their mechanical behavior description towards crystalline materials. However, their parameters are generally identified with the data at macroscopic scale without considering the effect of grain scale, which is the origin of the heterogeneities. [1-4]

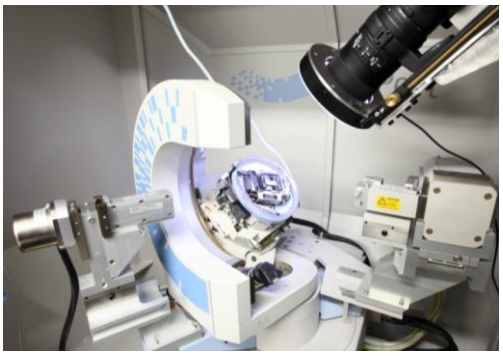

Figure 1: Assembly of the set up for the in-situ full-field measurements

For this reason, a robust procedure to identify this type of model using the grain scale information is sought. Two aspects are developed respectively: measuring the total ε^t and elastic ε^e strain fields and establishing a crystalline aggregate model to simulate as well as identify the parameters of the behaviour model.

In this study, a sample in Al-alloy with around twenty-five grains is subjected to a simple tensile loading. During this test, the total strain field is observed by digital image correlation technique while the elastic one is measured by X-ray diffraction simultaneously. The surface fields' information will then be dedicated to identify the parameters of a crystalline constitutive model, using finite element simulation [5].

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