A workshop to promote the use of high energy x-ray diffraction experiments and detailed computational analyses for understanding multiscale phenomena in crystalline materials

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Poster Abstracts

L. BALOGH, LOS ALAMOS NAT. LAB., LBALOGH@LANL.GOV 3

M. BRANDES, OHIO STATE UNIV., MS_BRANDES@MATSCENG.OHIO-STATE.EDU 4

J. BUNN, UNIV. OF TENNESSEE, JBUNN3@UTK.EDU 4

M. CIL, UNIV. OF TENNESSEE, MCIL2@TIGERS.LSU.EDU 5

P. KENESEI, ARGONNE NAT. LAB., APS, KENESEI@APS.ANL.GOV 5

F. KIM, UNIV OF TENNESSEE, FKIM@UTK.EDU 6

M. KOKER, MAX PLANCK INTEL. SYS., GERMANY, KOKER@IS.MPG.DE 7

F. LI, CARNEGIE MELLON UNIV., SFLI@CMU.EDU 8

M. MAJKUT, QUEEN'S UNIV., CANADA, MAJKUTM@ME.QUEENSU.CA 8

C. MARIANETTI, COLUMBIA UNIV., CHRIS.MARIANETTI@COLUMBIA.EDU 9

M. OBSTALECKI, CORNELL UNIV., MARK.OBSTALECKI@GMAIL.COM 9

D. PAGAN, CORNELL UNIV., DCP99@CORNELL.EDU 10

J-S. PARK, CORNELL UNIV., JP118@CORNELL.EDU 11

A. POSHADEL, CORNELL UNIV., ACP42@CORNELL.EDU 12

M. SANGID, PURDUE UNIV., SANGIDM@GMAIL.COM 12

B. SCHUSTER, ARMY RES. LAB., BRIAN.E.SCHUSTER.CIV@MAIL.MIL 13

P. SHADE, AIR FORCE RES. LAB., PAUL.SHADE.CTR@WPAFB.AF.MIL 14

M. SILBERSTEIN, BECKMAN INST. - UIUC, MEREDITH.SILBERSTEIN@GMAIL.COM 15

A. STEBNER, NORTHWESTERN UNIV., A-STEBNER@U.NORTHWESTERN.EDU 16

S-L. WONG, CORNELL UNIV., SW296@CORNELL.EDU 17

In Situ Spatially Resolved Orientation Mapping and Strain Measurements within a Twinned Grain during Loading of Magnesium

L. Balogh, Los Alamos Nat. Lab., lbalogh@lanl.gov

(Co-Authors: D. Brown, B. Clausen, W. Liu, S. Niezgoda & C. Tomé)

The presented research is part of a current BES funded research project at LANL focusing on the multi-scale study of deformation behavior of hexagonal materials. One of the main goals of the project is to model the twin deformation process as it is integral to the deformation of hexagonal metals. Previous in situ neutron diffraction loading measurements have shown that twinning is the major contributor to the plastic deformation up to about 10% total deformation, with a twin volume fraction above 70%. The neutron diffraction and 3DXRD measurements conducted at the 1-ID APS beamline also indicated that the initial strain state in the twins is far from the average of the polycrystal. The 3DXRD technique makes it possible to determine the average stress tensors for the parent and twin crystal separately, but the averaging is over the corresponding crystal volume. The 3D polychromatic differential-aperture X-ray microscope (DAXM) technique performed at the 34-ID APS beamline is able to probe the local orientations and strains inside the parent or twin in a loaded sample with micrometer spatial resolution, providing a more detailed characterization of the twinned grain. This information is paramount in the development and validation phases of the polycrystal deformation modeling effort and for connecting to novel direct numerical simulations of the twinning process using finite elements or FFT methods. The orientation maps recorded in the unstrained state reveal that the initial microstructure of the sample is not twinned. In the plastically deformed state, with the sample being under load, lots of (10.2) type twinning are revealed. The stacking of orientation maps allows a 3D characterization of the microstructure inside the bulk, up to 450 microns deep, which is a unique feature of the DAXM method. In a twinned grain, along a line intersecting the twinned region, one component of the intra-granular strain is measured, using the DAXM technique in monochromatic mode. The results reveal that strain inside the parent grain is different on the two sides of the major twin lamella, but the strain in the twin lamella fills the gap continuously. Further possibilities of applying the DAXM method to the investigation of the plasticity of hexagonal materials are discussed.

Characterization of Deformation and Fracture in Advanced Engineering Alloys

M. Brandes, Ohio State Univ., ms_brandes@matsceng.ohio-state.edu

The deformation and fracture behaviors of advanced alloys are strongly influenced by material structure and chemistry. In many cases, these materials are composites composed of a variety of individual crystallites whose mechanical responses are affected by a number of factors including, but not limited to, their compositions, structures, morphologies, crystallographic orientations, orientations relative to neighbor crystallites, and active deformation mechanisms. In this work, the fundamental mechanical responses of single- and poly-crystalline titanium alloys are investigated at the micro-scale by small scale testing and electron microscopy. These responses are then related to the deformation and fracture phenomena observed in macro-scale polycrystalline materials subjected to a variety of loading conditions. Recent results derived from X-ray based experiments in polycrystalline materials indicates that these techniques may provide a direct bridge between micro- and macro-scale phenomena of crystalline materials.

A New Approach to Measure Lattice Strains under Torsional Shear Using In Situ Neutron Diffraction for Polycrystalline Materials

J. Bunn, Univ. of Tennessee, jbunn3@utk.edu

(Co-Authors: R. Woracek, D. Penumadu, C. Hubbard)

Torsion provides a unique opportunity to study the mechanical behavior of materials subjected to a state of pure shear stress and is of significance as many engineering components fail under the influence of shear, or combination of shear and axial stresses. However, measurement of shear strain is limited to (near) surface measurements with most techniques. The authors introduce an experimental approach by applying the concept of a “strain rosette” for the measurement of shear strain based on lattice spacing changes under in-situ torsional loading, using recent data obtained at ORNL-NRSF2 in conjunction with a custom developed axial-torsional loading-system. Aspects of the technique, such as required gauge volume size ($\leq 1mm^3$) and precise alignment using a Laser Tracker system are addressed. The lattice specific shear moduli for Fe(211), Fe(200) and Fe(110), which were experimentally determined while elastically loading a circular steel rod (d=6mm), are presented and compared to Young’s moduli of the same sample.

Measurement of Strain of Silica Sand Particles Using 3D X-Ray Diffraction

M. Cil, Univ. of Tennessee, mcil2@tigers.lsu.edu

(Co-Authors: K. Alshibli , P. Kenesei & U. Lienert

Extensive research has been conducted to better understand the behavior of granular materials under compressive/shearing loads within the framework of continuum and discrete element theories. However, experimental measurements of stresses and strains of individual particles within the granular assembly remains a challenge in spite of advancement in many technologies. Recent developments in the three-dimensional x-ray diffraction (3DXRD) enable measuring strains within individual sand particles in situ during loading. This paper presents preliminary results. A single column of silica sand particles with average particle size between 0.595 mm and 0.841 mm was prepared inside an acrylic tube with an inner diameter of 1 mm. Multiple 3DXRD scans were acquired at subsequent load increments. For each load increment, strain tensors were evaluated and the load-strain relationship was examined. In spite of some fluctuations on the strain evolution, the analysis yielded a linear decrease of volumetric strain versus load increment. Axial strain values have a range from 10^{-4} to $1.5x10^{-3}$. The research team is planning to continue working on improving the measurements and analysis.

Extending the Spatial Information: High Energy X-Ray Imaging at APS 1-ID

P. Kenesei, Argonne Nat. Lab., APS, kenesei@aps.anl.gov

(Co-Authors): U. Lienert & J. Almer

The high-energy diffraction measurements provide results about the lattice orientation and lattice strain through the reciprocal space, however the spatial information has very limited accuracy, typically it is determined by the detector pixel size, detector distance and the beam size, and sometimes this information is very indirect. To improve the interpretation of the evaluated results we can recover the spatial structure of the sample based on simultaneously made radiography or tomography during the far field in-situ diffraction measurements. Due to the penetration length of the high energy X-ray we can make tomography on heavy metal samples and several millimeter thick samples and can identify internal features, like voids, cracks, various phases. We will present some examples to demonstrate our new capabilities at APD 1-ID.

High Resolution Neutron and X-ray Imaging of Partially Saturated Silica Sand

F. Kim, Univ of Tennessee, fkim@utk.edu

(Co-Authors: D. Penumadu, J. Gregor, N. Kardjilov, I. Manke, and M. Boin)

High resolution neutron (13.7 µm/voxel) and X-ray (11.2 µm/voxel) tomography results of partially saturated granular materials are presented. Partially saturated sand is composed of silica sand (SiO2), water (H2O) and air (O2) phases. The interactions between different phases govern the mechanical behavior of granular material. Neutrons and X-rays provide complementary contrast of the three phases on images. The water phase is well identified with a neutron image, and the silica phase is much more clearly shown with an X-ray image. The dual- modality of neutrons and X-rays make the detailed spatial analysis and identification of the three phases with high confidence by utilizing both contrasts. The neutron energy selective radiography result of a silica sand phase is additionally presented. Different neutron energies provided different amounts of attenuation corresponding to the d-spacing of the silica sand. The result is compared with the simulation. The data presented in this research is of critical importance to many disciplines that involve multi-phase flow visualization and analysis, granular mechanics that considers quantitative 3-D distribution of voids (gas and liquid phases) and solids, geotechnical and petroleum engineering, and contaminant transport through vadose zone.

Line Broadening due to Elastic Mechanical Stresses

M. Koker, Max Planck Intel. Sys., Germany, koker@is.mpg.de

(Co-Authors, U. Welzel & E. Mittemeijer)

When a stress is applied to a polycrystal, the elastically anisotropic grains in a massive body cannot deform freely to comply with an imposed state of mechanical stress; instead they must adapt their mechanical response to their surroundings. Various grain-interaction models exist to describe the stress and strain behavior of individual grains within an aggregate. These models can be employed for calculating mechanical and diffraction (X-ray) elastic constants of polycrystalline specimens from single-crystal elastic data. Experimental investigations have revealed that the Neerfeld-Hill and Eshelby-Kröner models, for massive, bulk polycrystals, and a recently proposed effective grain-interaction model for thin films provided excellent estimates for the (mechanical and) X-ray elastic constants of polycrystals. Further experimental investigations of elastically induced grain interactions are necessary to understand the heterogeneous strain and stress distributions within a specimen. This work provides a report of synchrotron diffraction investigations, as well as a computational investigation, of diffraction-line broadening induced by elastic loading of various polycrystalline specimens. The results indicate that the extent of the diffraction-line broadening calculated employing traditional grain-interaction models is much lower than the extent of the experimentally determined broadening. The variety of environment, and thus the heterogeneity of the stress/strain states experienced by the individual grains exhibiting the same crystallographic orientation in a polycrystal, cannot be accounted for by traditional grain-interaction models, where all grains of the same crystallographic orientation are considered to experience the same stress/strain state. The degree of broadening which can result from the heterogeneity of the environments of the individual crystallites on the basis of a finite element algorithm has been calculated as well. The obtained results have vast implication for diffraction-line broadening analysis and modeling of the elastic behavior of polycrystals.

Forward Modeling Reconstruction Using Adaptive Sampling

F. Li, Carnegie Mellon Univ., sfli@cmu.edu

The Forward Modeling Method (FMM) is an effective, albeit brute force method to reconstruct two-dimensional orientation maps from a rotating sample x-ray experiment, such as 3DXRD and near-field HEDM. By exploiting the inherent parallelism of this method, FMM reconstructions can be performed with most small scale supercomputers (~100-1000s of cores). Nonetheless, the sizable resource required for the orientation reconstruction has proved to be frustrating, and has essentially prohibited any meaningful real-time feedback during data collection. Here, an adaptive sampling extension to the existing FMM is described, which results in a wall-clock speed-up of a factor between 10-100 over brute force FMM. When combined with a Monte Carlo parameter optimization scheme, reconstruction in the time-scale of data acquisition has become readily available. A brief introduction to geometrical analysis of the resulting 3D microstructure will also be introduced.

A Study of Deformation Twinning in Magnesium by In Situ Three-Dimensional X-Ray Diffraction

M. Majkut, Queen's Univ., Canada, majkutm@me.queensu.ca

(Co-Authors: J. Oddershede, H. Abdolvand1, B. Diak & M. Daymond)

Tensile samples were cut from an annealed rolled plate of magnesium alloy AZ31B. In-situ three-dimensional X-ray diffraction was used to map the centre of mass and volume of each grain, as well as the elastic strain and stress in each grain during straining along the plate normal direction to ~1.6 % total strain. The rolling texture results in the majority of grains oriented favorably for {10-12}<-1011> tensile twinning, the dominant twinning mode in Mg. Typically, a Schmid factor approach is used to describe twinning, but non-Schmid factor behavior has been reported. Two mechanisms proposed to explain this behavior are: (a) twin formation is controlled by the ease with which neighboring grains can accommodate the transformation and (b) twin nucleation and/or growth is preferred at specially oriented grain boundaries. This study aims to investigate these local grain neighborhood effects on twin initiation and growth. Results are interpreted on a statistical level as well as locally for a select subset of grains.

Failure Mechanisms of Graphene under Tension

C. Marianetti, Columbia Univ., chris.marianetti@columbia.edu

Recent experiments established pure graphene as the strongest material known to mankind, further invigorating the question of how graphene fails. Using density functional theory, we reveal the mechanisms of mechanical failure of pure graphene under a generic state of tension at zero temperature. One failure mechanism is a novel soft-mode phonon instability of the K1 mode, whereby the graphene sheet undergoes a phase transition and is driven towards isolated hexagonal rings resulting in a reduction of strength. The other is the usual elastic instability corresponding to a maximum in the stress-strain curve. Our results indicate that finite wave vector soft modes can be the key factor in limiting the strength of monolayer materials.

In situ Measurement of Lattice Strain Using Synchrotron Radiation and a Conical Slit System

M. Obstalecki, Cornell Univ., mark.obstalecki@gmail.com

(Co-Authors: W. Tayon, R. Storer, A. Beaudoin, U. Lienert & P. Kenesei)

Large-scale deployment of aluminum-lithium (Al-Li) alloys (e.g. AA2195) could prove advantageous for the aerospace industry due to their combination of high specific stiffness and low density when compared to traditional high strength aluminum alloys (e.g. AA2219). Unfortunately, these alloys exhibit a stronger tendency for intergranular delamination within the highly textured, lamellar microstructure. Prior experimental evidence indicated the development of strain incompatibility between adjacent grains, prompting the in situ investigation of lattice strains for multiple grains within the constrained, interior of a specimen. Synchrotron X-ray diffraction was used to measure changes in lattice spacing in an AA2195 sample during tensile loading. The conical slit system at Argonne National Laboratory's Advanced Photon Source was utilized to isolate a single grain orientation of interest within this highly textured alloy. Trends were realized in all six independent stain components and revealed an elevated level of stress triaxiality in the initial loading steps. At higher loads, the level of triaxiality decreased, approaching the theoretical value for an isotropic material. An increased propensity for void formation and delamination fracture has been linked to elevated triaxiality.

A Methodology to Determine Single Crystal Mechanical Response Using High Energy X-Ray Diffraction

D. Pagan, Cornell Univ., dcp99@cornell.edu

(Co-Author: M. Miller)

An experimental method has been developed to gather in situ, high temperature strain measurements of single crystal semiconductor materials using high energy x-ray diffraction (HEXRD). Current efforts are focused on determining the temperature dependent, single crystal elastic moduli and critical resolved shear stress of Aluminum Nitride (AlN). Recent work has consisted of the creation of a diffraction pattern to strain tensor data reduction method using measurements made during a beam time at the Cornell High Energy Synchrotron Source. A method to interpolate between discrete detector pixel intensity values using finite elements for improved strain resolution has been adapted for use with single crystal data. Artificial diffraction data mimicking experimental conditions is currently used to understand and improve the experimental method.

Determination of Residual Stress Fields in Polycrystalline Alloys Using Depth-Resolved Synchrotron Radiation Strain Pole Figure Measurements and a Multiscale Finite Element Method

J-S. Park, Cornell Univ., jp118@cornell.edu

(Co-Authors: U. Lienert, E. Demir, M. Miller, & P. Dawson)

Residual stress is a self-equilibrating stress field that exists in a component that can substantially alter its mechanical behavior. Quantifying the residual stress in a component is therefore important, especially for the design of machines in which high reliability and performance are sought. In this work, a new experimental technique was developed. It combines the high energy synchrotron x-rays and a set of conical slits to measure lattice strain pole figures (SPFs) for diffraction volumes located inside a component non-destructively. A low-solvus high-refractory (LSHR) Ni-based superalloy component was fabricated using an interference-fit geometry to induce a 3D stress gradient throughout the component. SPFs were measured at various locations within the component. A finite element-based multiscale optimization method that enforces appropriate boundary conditions and equilibrium at the continuum-scale and simultaneously satisfies the SPF measurements was employed to obtain the full stress field that exists in the component. The trends in SPFs measured using the new experimental technique and the full stress field obtained using the new multiscale optimization method compare favorably with the analytic approximation of the stress field that was expected to exist from a thermoelastic analysis of the interference-fit component.

Influence of Spurious Deformation Modes on Deviatoric Lattice Strain Uncertainty for Polychromatic X-Ray Microdiffraction experiments

A. Poshadel, Cornell Univ., acp42@cornell.edu

X-ray microdiffraction is a powerful technique for conducting lattice strain measurements with high spatial resolution. However, there has been limited validation of the technique to date. An experiment was conducted at the Advanced Light Source to assess the uncertainty of deviatoric lattice strains measured using polychromatic x-ray microdiffraction. We demonstrate that the measurement uncertainty is different for each component of the deviatoric strain tensor. Monte Carlo simulations of the experiment are used to explain the differences in uncertainty. The simulations point to the existence of spurious deformation modes that arise erroneously in the strain calculation due to measurement noise and limited pole figure coverage. Methods for reducing measurement uncertainty are proposed.

Microstructural-Based Fatigue Modeling

M. Sangid, Purdue Univ., sangidm@gmail.com

Excessive scatter is observed in the fatigue response of a nickel-based superalloy, which is partly attributed to the variability in the microstructure. There is great interest in linking the microstructure to fatigue properties using a multi-scale approach that focuses on integrating the results of atomic simulations (slip in γ–γ' and dislocation-grain boundary interactions) to the continuum level. Our approach is to model the energy of a persistent slip band (PSB) structure and use its stability with respect to dislocation motion as our failure criterion for fatigue crack initiation. Through this methodology, the fatigue life is predicted based on the energy of the PSB, which inherently accounts for the microstructure of the material. From this framework, we construct simulated microstructures based on the measured distributions of grain size, orientation, neighbor information, and grain boundary character, which allows us to calculate fatigue scatter using a deterministic approach.Excellent agreement is shown between the model predictions and experimental data.

Multiscale Modeling of Aluminum Oxynitride (AlON)

B. Schuster, Army Res. Lab., brian.e.schuster.civ@mail.mil

(Co-Authors: G. Gazonas, J. McCauley, J. Clayton, R. Leavy, D. Casem, B. Rice, S. Batyrev & S. Weingarten)

The US Army Research Laboratory (ARL) has assembled a multidisciplinary team that is investigating the structure-property relationships for a model ceramic system at a range of length scales and applied strain rates. The primary focus has been in characterizing the response of aluminum oxynitride (AlON) which is a cubic spinel ceramic with superior mechanical and optical properties that make it attractive in numerous industrial and defense applications. Quantum mechanical and molecular dynamics simulations have been employed to examine the elastic response and deformation mechanisms at the atomic scale. Nanoindentation and microcompression experiments have been used to examine the properties of single crystal specimens at the micrometer scale. Finite element analysis simulations have examined the response of polycrystalline volumes of AlON using synthetic microstructures generated using Monte Carlo grain growth algorithm for equiaxed polyhedral. The high strain rate response of bulk polycrystalline specimens has been investigated using Kolsky bar compression and dynamic fracture experiments.

Micro-Tensile Testing and 3D-EBSD Characterization of Pure Ni Multi-crystals

P. Shade, Air Force Res. Lab., Paul.Shade.ctr@wpafb.af.mil

(Co-Authors: M. Groeber , M. Uchic, R. Wheeler, Y. Choi b, T. Turner , D. Dimiduk & C. Woodward)

This poster describes the application of a dual beam focused ion beam-scanning electron microscope (DB FIB-SEM) outfitted with an electron backscatter diffraction (EBSD) system to characterize the internal microstructure and local lattice rotations within micro-scale test samples that have been deformed with in-situ SEM tensile testing. The objectives of this study are to develop an experimental methodology that can provide a high-fidelity 3D characterization of the internal grain structure of mechanical test samples that contain a limited number of grains; obtain knowledge of the external boundary conditions and measurement of the resultant stress-strain behavior of the same test samples; and, perform a 3D characterization of the internal lattice rotations that develop after a modest amount of plastic deformation. Such information is needed to assess and guide the further development of modeling and simulation methods that predict the local plastic deformation response of polycrystalline ensembles.

Connecting the Uniaxial Stress-Strain Behavior of Sulfonated Polytetrafluoroethylene (Nafion) to its Microstructural Evolution

M. Silberstein, Beckman Inst. - UIUC, meredith.silberstein@gmail.com

The microstructure of the commercially available ionomer membrane Nafion has been extensively studied for its use both as a polymer electrolyte membrane in low temperature fuel cells and as the main component in ionic polymer metal composite actuators. Both of these applications require a combination of mechanical performance and cation conductivity. This work evaluates the simultaneous evolution of the microstructure and stress with applied mechanical deformation and discusses it in the context of a pre-existing micromechanically based model. Small and wide angle x-ray scattering is conducted real time during monotonic, cyclic, and stress relaxation uniaxial tensile testing of Nafion NRE212. Building on existing literature, the structure of Nafion is taken to be composed of randomly oriented elongated crystallites and randomly oriented cylindrically shaped ionic microdomains, with intermediate amorphous regions. The microstructure is found to be initially transversely isotropic in the membrane plane. Each microstructural feature evolves closely with macroscopic strain and has its own time and strain dependent evolution distinct from that of stress. Yield is found to be governed by the amorphous regime with the crystallites providing extra stiffness and inelastic recovery beyond the yield stress. This work is promising for the development of a coupled mechanical and electrochemical model for Nafion.

Micromechanics of Shape Memory NiTi Martensite

A. Stebner, Northwestern Univ., a-stebner@u.northwestern.edu

(Co-Authors: C. Brinson, S. Padula, R. Noebe, D. Gaydosh, A. Garg, D. Brown, S. Vogel, B. Clausen & T. Sisneros)

As the number of shape memory alloys (SMAs) and applications continues to increase and the significance of texture evolution in shape memory behaviors becomes more evident, the need for models that simulate the relationships between microstructural evolution and macroscopic responses becomes more apparent. The Simplified Multivariant Model is the most recent among several generations of multivariant, micromechanical models that have been formulated and numerically implemented at Northwestern University to address this need in both single crystal and polycrystalline systems. Recently, methodologies have been developed to couple these simulations with diffraction data as a way to concurrently validate micro and macro scale predictions of the model, and also gain further insight into the diffraction data, such as examination of intermediate microstructural features (e.g., habit plane variant configurations). These methodologies will be presented and illustrated through new empirical evidence of NiTi martensite texture evolution during multi-axial loading events. Furthermore, recent empirical observations during large straining of SMAs have unveiled a new mechanism that is not currently included in multivariant models. In addition to elasticity, transformation twinning, and slip, deformation twinning has been shown to be an active and substantial deformation mechanism for shape memory NiTi and UNb. Thus, a new mechanism required of multivariant models is motivated through new diffraction data.

Simulating diffraction patterns from virtual polycrystals under in situ cyclic loading

S-L. Wong, Cornell Univ., sw296@cornell.edu

(Co-Authors: J. Schuren, P. Dawson, M. Miller & J-S. Park)

Despite of nearly a century's worth of research in the area, fatigue failure of a material due to cyclic loading remains a challenging problem. Extending fatigue life in polycrystalline metals depends on developing a fundamental, micromechanical understanding of the microcrack initiation phase. Our approach is to conduct a coordinated suite of finite element simulations on highly-resolved virtual polycrystals (HiResVP) and high energy diffraction microscopy (HEDM) experiments to quantify and interpret the micromechanical response of polycrystals under in situ cyclic loading. The simulations are intended to replicate the physical experiments, which utilize HEDM techniques to interrogate the deformation of individual grains within a polycrystalline sample under in situ cyclic loading. The comparison between the simulations and experiments is made at the detector image. For individual crystals within a polycrystal, the goal is to quantify key features of their diffraction peak profiles and their evolution under cyclic loading.