Medium-Term (5 year) Plan for the Development of High Energy Diffraction Microscopy (HEDM)

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1) Science

Enormous growth has taken place in the field of multiscale material modeling over the past 10-15 years. The ever-increasing capacity of computing facilities has encouraged theoreticians to model behaviors on increasingly smaller size scales: engineering component, polycrystalline aggregate, single crystal, subgrain/disclocation structure, individual dislocations, particle and atom. The hope and promise of this research is the creation of a truly predictive link between material structure and thermo-mechanical properties and performance measures. However, there is a serious need for fundamental *multiscale* experiments – ones that produce material response data on all relevant size scales. A few are coming on the scene but currently these experiments are difficult to construct and only address one issue at a time. The station proposed here would address this need by enabling experiments that characterize microstructural and micromechanical states within a structural material simultaneously in *real time* during *in-situ* thermomechanical loading. A few examples of the science and technology that are driving the need for this station are described below.

- The fundamental source of fatigue failures and fatigue crack initiation continues to be a dominant unanswered scientific question that costs industries like transportation billions of dollars a year in fatigue failures and over-designed structures. In components like jet engines, fatigue cracks initiate on the grain scale yet design allowables are defined based on macroscopic properties and largely empirical analyses. Maintaining conservative designs results in huge factors of safety. Current generations of polycrystal plasticity models can bridge between these scales but engine makers are very reluctant to employ micromechanical analyses due to a general lack of experimental corroboration. Measuring the mechanical response grain by grain within a polycrystal will not only answer important scientific questions but may enable designers to base allowables on micromechanical properties. This will produce safer structures and will enable smaller factors of safety, which will lower costs.
- Grain boundaries play a dominant role in processes such as the intergrain transmission of inelastic strain. We know that the crystallographic description of the region between crystals is complex and, due to its three-dimensional nature, largely uncharacterized. Materials science has proposed that many macroscopic properties have a strong dependence on grain boundaries yet our experimental understanding comes mostly from two- or even one-dimensional image-based data. The research area of simulation-based grain boundary engineering is currently producing answers to questions that might be rather ill-posed geometrically. Non-destructive three-dimensional structural characterization of polycrystalline aggregates under load, would shed light on these problems immediately.
- All unit cells in a single crystal within an annealed polycrystalline aggregate are, by definition, oriented identically. Due to the incredibly complex nature of the intergrain mechanical environment, however, orientation heterogeneities rapidly develop during thermo-mechanical loading. Misorientations are accommodated by nucleation and propagation of dislocations and the development of dislocation structures. Currently, dislocation structure experiments are being interpreted with extremely simple approximations of micromechanical state. More importantly, we have only begun to understand the fully

> three-dimensional nature of dislocation structures and misorientations associated with grain subdivision. Dislocation models would advance significantly with real micromechanical data. However, measuring the response of thermo-mechanical boundary conditions acting on a grain within an aggregate and dislocation structure formation is currently only a dream. Experimentally linking the subgrain, three-dimensional stress state to the dislocation structure it is inducing would answer a plethora of fundamental questions related to dislocation dynamics and is basically a once-per-career type accomplishment.

Most materials science experiments – even those from the current generation of "*3-D Matsci*" - are fundamentally forensic. We happen upon a fatigue failure or the creation of a dislocation structure and must try to reconstruct what happened based upon what we find at the scene. The only way that we keep pace with simulations and contribute to their further development is by creating **dynamic** experiments that monitor important processes in real time **during** thermo-mechanical excursions and produce **quantitative** data – not just images. In this manner we will enable the experiment and model to work together to uncover understanding of physical phenomena that neither could address on its own. Substantial gains – both scientific and technological – stand to be made. While we have picked around the edges of this work in the past, these data do not currently exist anywhere in the world. The results that come out of this station will be completely unique. We plan to let the materials community drive the research with their science. They will come to the station knowing what they want to measure, just unsure how. Our plan is to lower the barriers to real data so that users leave the station with results, not merely intensities from a detector. As user sophistication increases new experiments never thought possible will be proposed.

2) Status at 1-ID

Near-field grain mapping: A detector with high spatial resolution is positioned close behind the sample and the grain boundary topology is reconstructed. A dedicated setup has been implemented in the B-hutch. The distance to the source is too small for efficient micro-focusing and space constraints in the B-hutch prevent a dedicated setup that combines near- and far-field measurements.

Far-field medium q-resolution: A large area detector is positioned far enough from the sample that it is able to resolve strains but close enough to capture several complete diffraction rings. The strain tensors, orientation matrices, and volumes of many grains are obtained simultaneously. Experiments need to be performed in the C-hutch. The complete setup (beam conditioning, sample stage, sample environment, and detector) needs to be build up for each experiment.

Far-field high q-resolution: A compact load frame is mounted on the C-hutch 3-circle diffractometer. A conventional far-field detector can be moved in and out of the beam and a MAR CCD, which provides high q-resolution, is mounted on a vertical translation stage about 4 m behind the sample. The evolving dislocation structure within individual grains can be observed during deformation. Again, the complete setup (except for the vertical detector stage) needs to be build up for each experiment.

Software: First steps towards fast and automated data acquisition have been taken but further progress is hampered by the non-existence of setups during maintenance periods. Data evaluation software is being developed by partners as described under (6). Recent progress using parallel computing indicates ample potential. However, a work flow needs to be established and the software needs to be accessible to general users.

3) Added value of Mid-term Upgrade

- Dedicated instruments and software development will dramatically improve the efficiency of operation, data quality and lower the threshold to new users.

- Dramatically reduced data acquisition times will provide access to shorter time scale processing and increased sample throughput.
3D near-field mappings take typically several hours now but could be accelerated by an order of magnitude or even more. Likewise, acquiring center-of-mass grain positions by far-field ‘box scans’ will become a routine tool enabling intelligent experiment control rather than following recipes blindly.

- Ability to probe statistically relevant numbers of grains and combine real and reciprocal space information will enable conclusive comparison to modeling.
The combination of near- and far-field setups and ‘box scans’ will provide strain and position (grain neighborhood) information. The SIMULTANEOUS measurement of orientation, x,y,z position and lattice strain is expected to have tremendous impact within the materials and mechanics community. Comparison to models beyond the ‘mean-field’ approximation will become possible.

- Improved spatial resolution down to 1 μm will allow access to smaller grained samples and increased sensitivity to grain topology changes.
Spatial resolution will improve due to: (i) a larger distance to the source and optimized optics, (ii) detector upgrades, (iii) use of diffraction contrast tomography.

- Improved reciprocal space resolution will allow a detailed study of evolving dislocation structures during deformation.
The reciprocal space resolution sets a lower limit to the detectable dislocation density. It is speculated that the ERL would enable coherent diffraction from sub-grains (i.e. complete reciprocal space information is obtained). Then the spatial arrangement and dynamics of individual dislocations may be obtainable.

4) Expected user communities

Contributors to the broad area of 3-D materials science and materials design. These activities engage individuals from physics, materials science, mechanical engineering, civil engineering, aerospace engineering. A core community is using the present HEDM facilities at 1-ID, obtaining excellent GUP ratings.

5) Enabling technology and infrastructure

5.1) <u>White beam station dedicated to HEDM</u>

One of the centerpieces of this proposal is the construction of a hutch dedicated to HEDM which would accommodate dedicated instruments. While experiments are monochromatic, a white beam hutch is required as some micro-focusing optics require direct beam to operate efficiently. Downstream location within the experimental hall would provide a sufficiently large source distance for efficient focusing. The energy range will be about 40-90 keV. Downstream optics requirements include a bent Laue-Laue monochromator, high resolution monochromator, focusing refractive lenses, all delivering ‘standard tomography clean beam’. Short period, small gap undulators and optional reduced horizontal emittance mode for point focusing are requested. At least 50% of the annual beamtime should be allocated to HEDM users.

The hutch will accommodate dedicated and optimized instruments for combined near- and far-field diffraction and high resolution reciprocal space mapping. This includes two large area detectors (a-Si), a medium resolution, and a high resolution detector. The near-field instrument will also provide basic tomography capability (first standard tomography, later also diffraction contrast tomography, and topo-tomography).

5.2) Software and computing infrastructure

We request 0.5 FTE of a beamline oriented scientific programmer.

The main tasks are automated data acquisition, online data evaluation for intelligent experiment control, and workflow from data acquisition through evaluation packages provided by partners (see (6)) to physical parameters (e.g. strain tensors, crystallographic grain orientations, ODF, grain map, …). The use of a computer cluster will be required.

6) Partnerships

Matthew Miller, Professor of Mechanical and Aerospace Engineering, Cornell University: Developed strain evaluation software in collaboration with J. Bernier (LLNL) and collaborated on design and control of compact load frame. He spent part of his sabbatical at 1-ID and contributed one Ph.D. student. Supported by ONR for spatially resolved stress measurements in multiphase materials.

Robert Suter, Professor, Physics department, Carnegie Mellon University: Develops near-field microscopy reconstruction software. Contributed four Ph.D. students and airbearing precision rotation table. New NSF support for fundamental studies of grain growth in 3D.

Wolfgang Pantleon, Senior Scientist, Risø National Laboratory, Denmark: Develops reciprocal space mapping analysis software and contributes to data acquisition automation software. Provides multi-grain indexing software in collaboration with ESRF and contributed two Ph.D. students.

7) Industry and technology transfer

Contacts exists between present HEDM users and the following potential partners: GE, Rolls Royce**,** Naval Research Lab, Air Force Research Lab, Alcoa Technical Center.

8) Estimated budget

The construction of a white beam hutch was estimated to be $440K for the 1-ID phase II upgrade. The cost of downstream components cannot be estimated as it is strongly dependent on the complete beamline layout which is beyond the scope of this proposal.

Detectors: 2 large area a-Si detectors 2 x $160K, high q-resolution detector $200K.

0.5 FTE scientific software engineer: $100K per year.

Optics, ancillary equipment, and instrument upgrades: $500K.

Total cost over 5 years: $1960K.