Final Technical Report for DOE Grant DE-SC0002001: *Quantifying Damage Accumulation During Ductile Plastic Deformation Using Synchrotron Radiation*

PIs: R. M. Suter and A. D. Rollett, Carnegie Mellon University

Under this grant, we have developed and demonstrated the ability of near-field High Energy Diffraction Microscopy (nf-HEDM) to map crystal orientation fields over three dimensions in deformed polycrystalline materials. Experimental work was performed at the Advanced Photon Source (APS) at beamline 1-ID. Applications of this new capability to ductile deformation of copper and zirconium samples were demonstrated as was the comparison of the experimental observations to computational plasticity models using a fast Fourier transform based algorithm that is able to handle the large experimental data sets. No such spatially resolved, direct comparison between measured and computed microstructure evolutions had previously been possible. The impact of this work is reflected in numerous publications and presentations as well as in the investments by DOE and DOD laboratories of millions of dollars in applying the technique, developing sophisticated new hardware that allows the technique to be applied to a wide variety of materials and materials problems, and in the use of the technique by other researchers. In essence, the grant facilitated the development of a new form of three dimensional microscopy and its application to technologically critical states of polycrystalline materials that are used throughout the U.S. and world economies. On-going collaborative work is further optimizing experimental and computational facilities at the APS and is pursuing expanded facilities.

This project has had substantial impact through encouraging the materials science and engineering community to adopt 3D orientation mapping into their research programs. It is now understood that the tools exist to validate micromechanical models at the scale of individual grains. People such as Michael Sangid (Purdue), Armand Beaudoin (UIUC), Ashley Spear (U. Utah), Tom Biehler (Michigan State), for example, are now actively using the technique and there are plans to make 3D microscopy available to non-expert users. Discussions are underway with APS management about adding a new, high throughput beamline that would service demands of, for example, additive manufacturing and stress-corrosion cracking research, where large parameter spaces (and, therefore, many samples) need to be explored.

Supported personnel:

- Graduate students

  1. **S. F. Li**: Developed Ice Nine reconstruction software, beginning with a C++ translation of Suter's original Fortran code, that has now been installed on multiple parallel computing systems including: i) the CMU BLOCH cluster, ii) XSEDE Ranger and Stampede, iii) NERSC systems, iv) LANL systems, v) AFRL systems, iv) APS Orthros, vii) Argonne ALCF Mira. Ice Nine includes a variety of accelerator algorithms and exhibits greatly improved robustness. The code is available on GitHub and a more convenient, single site version is being developed. Li led the first generation tensile deformation measurement and developed a library of analysis and visualization tools for probing the 3D data sets that are generated by

nf-HEDM. After completion of his PhD, Li took a position at LLNL and is now working for a start-up company developing medical diagnostic codes and systems.

2. **J. Lind**: Developed robust image analysis code for reducing raw diffraction images to the form used by Ice Nine. The algorithm uses a standard tool from the image processing community that is able to segment near-by and overlapping patterns. This code has enabled the reconstruction of both deformed microstructures and the resolution of small twin grains within larger parent grains. Lind also developed a second generation loading system that is able to perform tensile deformation of millimeter cross-section metallic samples and used this system to study deformation twinning and slip in hexagonal zirconium. After completion of his degree, Lind to a post-doctoral position at LLNL, where he remains currently.
3. **R. Pokharel**: Applied Lind's tensile apparatus to studies of copper samples. She helped develop parallel FFT based plasticity codes and used that capability to model her observations. She developed a variety of statistical and local comparison metrics. She also made extensive use of full field micromechanical simulation to compare with the experimental results. After completion of her degree, she took a postdoctoral position at LANL where she currently works and continues to use nf-HEDM on a variety of materials systems and processing problems.
4. **X. Tan**: Assisted with data collection at the APS, applied the developed reconstruction methodologies to a fatigue fractured nickel superalloy sample thus demonstrating the range of applications possible. He developed registration algorithms and codes for combined absorption tomography and nf-HEDM imaging. Currently employed by Google.
5. **S. Maddali-Vivekanand**: Assisted with data collection at the APS.

- Undergraduate student summer projects

1. Anthony Pacella
2. Graham Spicer
3. George Bargoud
4. Harris Tucker
5. Sean MacGahan
6. Edward Kahn
7. Johanne Rokholt
8. Samikshya Subedi

Publications citing full support from this grant

1. S.F. Li, J. Lind, C.M. Hefferan, R. Pokharel, U. Lienert, A.D. Rollett, R.M. Suter, "Three Dimensional Plastic Response in Polycrystalline Copper Via Near-Field High Energy X-ray Diffraction Microscopy," J. Appl. Cryst., **45**, 1098-1108 (2012). doi:10.1107/S0021889812039519
2. R. Pokharel, S.F. Li, J. Lind, C.M. Hefferan, U. Lienert, R.A. Lebensohn, R.M. Suter, A.D. Rollett, "Quantifying damage accumulation using state-of-the-art FFT method," Materials Science Forum **702-703**, 515-518 (2012).

3. S.F. Li and R.M. Suter, "Adaptive Reconstruction Method for Three-Dimensional Orientation Imaging," J. Appl. Cryst., **46**, 512-524 (2013). DOI: 10.1107/S0021889813005268
4. J. Lind, S.F. Li, R. Pokharel, U. Lienert, A.D. Rollett, R.M. Suter, "Tensile twin nucleation events coupled to neighboring slip observed in three dimensions," Acta Materialia, **76**, 213-220 (2014). DOI: 10.1016/j.actamat.2014.05.021
5. R. Pokharel, J. Lind, A.K. Kanjarla, R.A. Lebensohn, S.F. Li, P. Kenesei, R.M. Suter, and A.D. Rollett, "Polycrystal plasticity: comparison between grain scale observations of deformation and simulations," Annual Reviews of Condensed Matter Physics, **5**, 317346 (2014). DOI: 10.1146/annurev-conmatphys-031113-133846
6. R. Pokharel, J. Lind, S.F. Li, P. Kenesei, R.A. Lebensohn, R.M. Suter, A.D. Rollett, "In-situ observation of bulk 3D grain evolution during plastic deformation in polycrystalline Cu," Int. J. of Plasticity, **67**, 217-2343 (2015). DOI: 10.1016/j.ijplas.2014.10.013

PhD Theses

1. S. F. Li, "*Imaging of Orientation and Geometry in Microstructures: Development and Applications of High Energy X-ray Diffraction Microscopy*," Carnegie Mellon University, May 3, 2011.
2. R. Pokarel, *Spatially resolved in-situ study of plastic deformation in polycrystalline Cu using high-energy X-rays and full-field simulations*, Carnegie Mellon University, April 30, 2013.
3. J. Lind, *In-situ High-Energy Diffraction Microscopy Study of Zirconium Under Uniaxial Tensile Deformation*, Carnegie Mellon University, July 29, 2013.
4. X. Tan, *A New Method for Fracture Surface Studies: Combined HEDM Orientation Mapping and Absorption Tomography Applied to a Nickel Superalloy*, Carnegie Mellon University, February 24, 23014.

Publications using results of grant supported technique developments (partial)

1. P.A. Shade, B. Blank, J.C. Schuren, T.J. Turner, P. Kenesei, K. Goetz, R.M. Suter, J.V. Bernier, S.F. Li, J. Lind, U. Lienert, J. Almer, Rev. Sci. Instr., "A rotational and axial motion system load frame insert for in situ high energy x-ray studies," Rev. Sci. Instr., in press.
2. "Orientation gradients in relation to grain boundaries at varying strain level and spatial resolution", S. Subedi, R. Pokharel and A.D. Rollett, Materials Science & Engineering A, 638 348?356 (2015), http://dx.doi.org/10.1016/j.msea.2015.04.051i.
3. J. Schuren, J. Lind, S.F. Li, J. Bernier, P. Shade, T.J. Turner, P. Kenesei, J. Almer, B. Blank, R.M. Suter, "Changing the paradigm for materials engineering design by integrating high energy x-ray data with modeling," Current Opinion in Condensed Matter and Materials Science, in press. doi:10.1016/j.cossms.2014.11.003
4. P.A. Shade, J.C. Schuren, J.V. Bernier, S.F. Li, B. Blank, J. Lind, P. Kenesei, U. Lienert, R.M. Suter, T.J. Turner, D.M. Dimiduk, J. Almer, "Changing the Paradigm for Engineering Design by Merging High Energy X-ray Data with Materials Modeling,"

Microsc. and Microanalysis, **20**, 1444-1445 (2014).
DOI: http://dx.doi.org/10.1017/S1431927614008952

5. A. Cerrone, C. Stein, R. Pokharel, C. Hefferan, J. Lind, H. Tucker, R.M. Suter, A.D. Rollett, A. Ingraffea, "Implementation and verification of a microstructure-based capability for modeling microcrack nucleation in LSHR at room temperature," Modeling and Sim. in Mat. Sci. and Engr., **23**, 035006 (2015). doi:10.1088/0965-0393/23/3/035006
6. C.A. Stein, A. Cerrone, T. Ozturk, S. Lee, P. Kenesei, H. Tucker, R. Pokharel, J. Lind, C.M. Hefferan, R.M. Suter, A.R. Ingraffea, A.D. Rollett, "Fatigue crack initiation, slip localization and twin boundaries in a nickel-based superalloy", Current Opinion in Solid State and Materials Science, 2014. DOI: 10.1016/j.cossms.2014.06.001
7. A.D. Spear, S.F. Li, J.F. Lind, R.M. Suter, A.R. Ingraffea, "Three dimensional characterization of microstructurally small fatigue-crack evolution using quantitative fractography combined with post-mortem X-ray tomography and high-energy X-ray diffraction microscopy", Acta Materialia, **76**, 413-424 (2014). DOI: 10.1016/j.actamat.2014.05.021
8. L. Wang, J. Lind, H. Phukan, P. Kenesei, J-S. Park, R.M. Suter, A.J. Beaudoin, T.R. Bieler, "Mechanical twinning and detwinning in pure Ti during loading and unloading-An in situ high-energy X-ray diffraction microscopy study," Scripta Materialia, **92**, 35-38 (2014). DOI: 10.1016/j.scriptamat.2014.08.008

Invited presentations by the PIs citing support from this grant

- R. M. Suter
    1. TMS 2010, Seattle, WA: "X-Ray Diffraction Microscopy Studies of Microstructure Responses"
    2. Japan Iron and Steel Institute 2010, Sapporo, Japan: "High Energy X-ray Diffraction Microscopy (HEDM): Microstructure Responses in Polycrystals"
    3. SPring-8 seminar, Hyogo, Japan 2010: "High Energy X-ray Diffraction Microscopy (HEDM): Microstructure Responses in Polycrystals"
    4. University of Kyoto seminar, 2010: "High Energy X-ray Diffraction Microscopy (HEDM): Microstructure Responses in Polycrystals"
    5. Plasticity Conference, San Juan, PR 2012: "Plastic Response in Copper and Zirconium Observed by High Energy X-Ray Diffraction Microscopy Orientation Mapping at APS 1-ID"
    6. TMS-3D, Seven Springs, PA 2012: "High Energy X-Ray Diffraction Microscopy Microstructure Mapping at APS 1-ID"
    7. LANL workshop: Damage Evolution in Structural Materials at the Mesoscale: Models and Experiments, Los Alamos, NM 2012: "High Energy X-Ray Diffraction Microscopy Microstructure Mapping at APS 1-ID"
    8. Denver X-ray Conference, 2012: "High Energy X-ray Diffraction Microscopy (HEDM): Microstructure Responses in Polycrystals"
    9. International Workshop on Computational Mechanics of Materials, Baltimore, MD 2012: "Tracking Microstructural Responses to Tensile Deformation Inside of Copper and Zirconium Using High Energy X-ray Diffraction Microscopy"

10. MS&T, Columbus, OH 2012: "Near-field High Energy X-ray Diffraction Microscopy in Deformed Materials"
11. MRS Fall meeting, Boston 2012: "High Energy X-Ray Diffraction Microscopy (HEDM): Direct Observation of 3D Materials Responses"
12. TMS, San Antonio, TX 2013: "Combined High Energy X-ray Diffraction Microscopy and Tomography Measurements On and Around Fracture Surfaces," "High Energy X-ray Diffraction Microscopy Combined with Tomography: Creating a Direct Link Between Mesoscale Computational Models and Experimental Measurements," and "High Energy X-Ray Diffraction Microscopy: Tracking of Internal Polycrystal Responses to Tensile Deformation"
13. CHESS Seminar, Ithaca, NY 2013: "High Energy X-ray Diffraction Microscopy: Methods, State of the Art, Example Applications"
14. APS Operations Meeting, April 2013: "High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses"
15. Boston University Physics Department seminar, 2013: "High Energy X-Ray Diffraction Microscopy (HEDM): An Inside View of Materials Responses"
16. APS Materials by Design Workshop, 2013: "Potential for using high energy x-ray diffraction microscopy in studies of synthesis"
17. CMU 3D Microstructures summer school, Pittsburgh, PA 2013: "Multi-modal tracking of microstructure responses: nf-, ff-HEDM and High Energy Tomography"
18. Pacific Rim International Conference on Materials, Waikoloa, HW 2013, "High Energy X-ray Diffraction Microscopy: Recent Results and Developments at the APS"
19. Neutron/X-ray Summer School lecture, Argonne, IL 2013: "High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses"
20. ARO Workshop on High Strain Rate Deformation, Johns Hopkins, MD 2013: "High Energy X-ray Diffraction Microscopy: Non-destructive Microstructural Responses in 3D"
21. APS Upgrade Workshop, Argonne, IL 2013: "Scientific Opportunities for High Energy X-ray Diffraction with an MBA Lattice"
22. Plasticity, Bahamas 2014: "In-Situ, Spatially Resolved Observation of Twinning in Zirconium using High Energy X-ray Diffraction Microscopy and Current Developments in Measurement Technique"
23. American Physical Society, March Meeting 2014: "Mesoscale Science with High Energy X-ray Diffraction Microscopy at the Advanced Photon Source"
24. Mesoscale Science Frontiers workshop, Santa Fe, NM 2014: "Emergent Complexity in Polycrystals: Measuring 3D Mesoscale Responses with High Energy X-rays"
25. Neutron/X-ray Summer School lecture, Argonne, IL 2014: "High Energy Diffraction Microscopy at Sector 1: An Inside View of Materials' Responses"
26. TMS-3D, Annecy, France 2014: "Status Report on HEDM at the APS: Measurements and Analysis in 3D"
27. ESRF Workshop on Deformation Mapping, Grenoble, France 2014, "In-situ observation of twinning in Zr under tensile extension"
28. Society for Engineering Sciences, Purdue Univ. 2024: "High Energy Diffraction

Microscopy Studies of Fatigue and Fracture in Superalloys and Characterization of Lamellar Ti-6Al-4V"

29. Johns Hopkins University, Materials Science seminar, Baltimore, MD October 2014: "Mesoscale Science with High Energy Diffraction Microscopy: Observing Heterogeneous Responses in 3D In Polycrystals"
30. TMS 2015, Orlando, FL, March 2015 "HEDM: Spatially Resolved Measurements of Lattice Orientation and Strain in Polycrystals"
31. University of Glasgow, School of Engineering, Scotland, July 2015 "3D tracking of microstructure responses: nf-, ff-HEDM and High Energy Tomography"

- A. D. Rollett (2013 -)

1. Invited lecture at Plasticity-2013, Nassau, Bahamas
2. TMS Annual Meeting, San Antonio, March 2013, Invited
3. Invited Seminar at the Ris Laboratory, Roskilde, Denmark, March 2013
4. Invited Seminar at the Lawrence Livermore Laboratory, Livermore CA, 18th April 2013
5. Invited Seminar at the Deakin University, Geelong, Australia, May 2013
6. Pacific Rim Conference on Materials (PRICM), Hawaii, August 2013
7. Invited lecture at a Workshop at the Natl. Science Foundation, Arlington VA, September 2013
8. Invited Seminar for the Materials Science & Engineering Department at Michigan State University, Lansing MI, November 2013
9. Invited lecture at the Fall MRS Meeting, Boston MA, December 2013
10. Seminar at the Iowa State University, Ames IA, January 2014
11. Seminar at Cornell University, Ithaca NY, January 2014
12. Seminar at Pratt & Whitney, New Haven CT, Feb. 6th 2014
13. TMS Annual Meeting, San Antonio, February 2014
14. Keynote Lecture at the Mach Conference, Annapolis, MD, April 2014
15. Seminar at Northeastern University, Boston MD, April 17th 2014
16. Mesoscale Science Frontiers Conference, Santa Fe, NM, May 2014
17. Seminar at the Army Research Laboratory, Aberdeen MD, May 2014
18. Invited Presentation at the Brookhaven National Laboratory Users Meeting, BNL, May 2014
19. Invited Lecture at ONERA, Paris, France, June 2014
20. Plenary Lecture at the IMRC XXIII, Cancun, Mexico, August 20th, 2014
21. ICOTOM-17, Dresden, Germany, August 2014
22. Keynote lecture at the 10th International Conference on Fatigue Damage of Structural Materials, Hyannis, September 2014
23. Invited Lecture at the Symposium in Honor of Prof. Anthony Ingraffea, Cornell University, Ithaca, September 2014
24. Multiscale Materials Modeling, Berkeley, October 2014
25. ICIP 2014, Paris, October 2014
26. MS&T, Pittsburgh, October 2014
27. Invited Lecture at the University of Tennessee, Knoxville, TN, November 2014
28. Invited Lecture at the Fall MRS Meeting, Boston, December, 2014

29. Seminar at Imperial College, London, March 2015
30. APS, SRI Satellite Meeting, July 13-14, 2015
31. Keynote Lecture at the USNCCM, July 2015
32. Microscopy & Microanalysis meeting, Portland, OR, July 6th, 2015