

Final Report for Deformation and Failure Mechanisms of Shape Memory Alloys

DOE Award Number: DE-SC0003996 monitored by Dr. John Vetrano
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Project Title: Deformation and Failure Mechanisms of Shape Memory Alloys
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I. Brief Introduction

The goal of this research was to understand the fundamental mechanics that drive the deformation and failure of shape memory alloys (SMAs). SMAs are difficult materials to characterize because of the complex phase transformations that give rise to their unique properties, including shape memory and superelasticity. These phase transformations occur across multiple length scales (one example being the martensite-austenite twinning that underlies macroscopic strain localization) and result in a large hysteresis. In order to optimize the use of this hysteretic behavior in energy storage and damping applications, we must first have a quantitative understanding of this transformation behavior. Prior results on shape memory alloys have been largely qualitative (i.e., mapping phase transformations through cracked oxide coatings or surface morphology). The PI developed and utilized new approaches to provide a quantitative, full-field characterization of phase transformation, conducting a comprehensive suite of experiments across multiple length scales and tying these results to theoretical and computational analysis. The research funded by this award utilized new combinations of scanning electron microscopy, diffraction, digital image correlation, and custom testing equipment and procedures to study phase transformation processes at a wide range of length scales, with a focus at small length scales with spatial resolution on the order of 1 nanometer. These experiments probe the basic connections between length scales during phase transformation. In addition to the insights gained on the fundamental mechanisms driving transformations in shape memory alloys, the unique experimental methodologies developed under this award are applicable to a wide range of solid-to-solid phase transformations and other strain localization mechanisms.

II. Listing of People Funded During Reported Period (4/15/2010-4/15/2015)

The following graduate and undergraduate students were funded under this award. Information on their individual accomplishments during the reported period is provided in Section III.

- *Michael Kimiecik (GSRA):* Graduate Student Research Assistant (GSRA) fully funded by DoE from 2010-2015. Mike focused on the small-scale characterization of SMAs, using specialized scanning electron microscopy techniques to map transformation at the microscale. Mike will graduate in August 2015.
- *Kyubum Kim (GSRA):* Kim was fully funded by DoE from 2010-2013 (on startup funds for his first year). Kim performed and analyzed thermo-mechanical cycling experiments using simultaneous IR/DIC imaging of SMAs resulting in papers to Exp Mech (Best Paper of the Year) and Smart Mat Struct. Kyubum graduated in August 2013.

- Adam Kammers (GSRA): Funded for a semester appointment to assist in development of experimental setups for SEM-DIC. Work funded by NSF and DoE resulted in journal publications listed below.
- Benjamin Reedlunn (GSRA): Funded for a 8 month appointment to examine phase transformation in hierarchical structures (cables) resulting in JPMS publication. Work funded by GM and DoE resulted in journal publications listed below.
- Riddhiman Bhattacharya (Masters): Funded for an 8-month appointment (September 2010-April 2011) to develop experimental setups for micro-DIC.
- Emily Nelson (UG): Undergraduate student researcher worked in the Daly labs on this project from 2010-2011. Work with a graduate student (Ben Reedlunn) resulted in JPMS publication.
- Adam Joyce (UG): Undergraduate student researcher who was hired into the Daly labs from 2012-2013. Worked with graduate student Michael Kimiecik to examine aging effects on microstructural behavior of SMAs.
- Jeremy Liu (UG): Undergraduate student researcher with stipend for Summer 2010 to work on an experimental apparatus for improved stereo DIC imaging. Worked in the Daly labs on this project from May 2010-December 2010 (summer stipend funded by DoE; Fall as independent research project for course credit).

III. Accomplishments for Reported Period and Schedule Status

Overview Bullet List

- Scientific Findings:

Determination that phase transformation (note: for alloys not satisfying the cofactor conditions) exhibits a strong transformation memory with cycling that persists across length scales. Determination that at the microscale, in contrast to current theories used in modeling, transformation is highly heterogeneous sub-grain, grains can contain numerous variants of martensite, similarly oriented grains do not transform similarly, and sub-grain detwinning happens prior to the end of the macroscopic stress plateau.

 - Strong strain memory at the macroscale and mesoscale under hard cycling;
 - Mesoscale strain memory becomes stronger with more unfavorable transformation textures;
 - Comprehensive study of SMAs in structural modes (funded by DoE and GM);
 - Strong strain memory under cycling persists at the sub-grain level;
 - Transformation is heterogeneous at the sub-grain level;
 - The transformation front is diffuse and exhibits a cross-hatched structure;
 - Transformation does not progress contiguously, but irregularly;
 - A single most favorable variant does not nucleate and subsume the entire grain;
 - Similarly oriented grains do not transform similarly, in contrast to a commonly held assumption in mean field theories;
 - Sub-grain detwinning occurs prior to the end of the macroscopic stress plateau;
 - Sub-grain detwinning is related to the accumulation of sub-grain damage/residual strain;
 - Mapping of sub-grain detwinning based on microscale, highly accurate strain fields from in-SEM testing with active habit plane variants determined from compatibility considerations;
 - Experimental quantification, to a high degree of accuracy, twins active in specific grains
- Products:
 - Best Paper of the Year, *IJSS*, 2014
 - Best Paper of the Year, *Experimental Mechanics*, 2011
 - 10 Journal Publications (6 fully funded by DoE; 4 partially funded)
 - +2 Journal Publications (fully funded) additionally in progress;
 - 5 graduate students funded (2 fully, 3 partially)

- 3 undergraduate researchers funded for 8 month – 1+ year internships
- 20 (directly related) invited presentations
- Development of SEM-DIC methodology, including self-functionalized nanoparticle assembly, distortion corrections, associated code and equipment (partially funded by DoE, also funded by NSF).
- 6 multi-session symposia (directly related to this research) organized
- Numerous collaborations started/fostered

Detailed Information on Accomplishments Based from Original Proposal

The PI and her research group have made numerous significant findings in this research. The research is roughly broken into four parts:

- (1) the thermo-mechanical behavior of bulk SMAs,
- (2) fatigue behavior and pattern memory,
- (3) microscopic mechanisms of transformation; and
- (4) size effects on transformation.

The experimental investigations of bulk thermo-mechanical behavior are complete. These tests yielded interesting and novel results, built on the 2011 discovery (funded by this grant) of a strong strain memory in the martensitic phase that exists in the wake of the transformation front upon loading and persists from cycle to cycle. This discovery has important implications for the long-term fatigue behavior of these alloys [Kim and Daly, *Experimental Mechanics*, 2011], and this paper was recently recognized by the journal with the 2013 Hetenyi Award for best paper of 2011. Kim completed his analysis on cycling and texture effects this year, and the results were published in *Smart Materials and Structures* in Summer 2013. Kim successfully defended his doctoral work in August 2013.

In addition to examining bulk SMA behavior, we spent significant effort during this award to develop a new methodology capable of investigating the *in-situ* development of full-field strains on the microstructural length scale. This allowed us to quantify deformation inside of grains and across grain boundaries during phase transformation for the first time, and is particularly useful for quantifying the effect of microstructure on phase transformation. This methodology was difficult to achieve and included the development of methods for nanoparticle self-assembly (for small-scale deformation tracking), complex spatial and temporal deformation codes to correct for distortions inherent in SEM micrographs, and analysis to extract useful relative deformations. In 2013, we refined this approach and utilized it to examine the effect of polycrystalline constraints on phase transformation in SMAs at the microstructural level. An example of the progression of transformation is shown in Figure 1, where ~600,000 strain values are extracted from a ~400 micron square field of view and binned in order to quantitatively examine the progression of transformation.

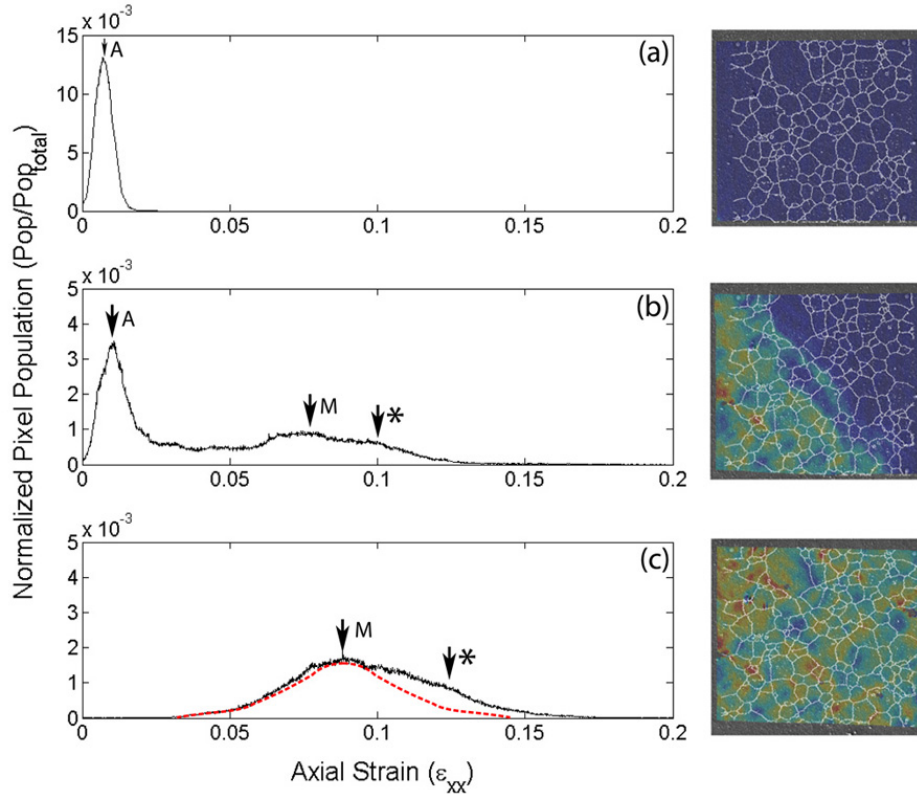


Figure 1: Population distribution of pixels binned by their measured strains. From top to bottom, a microscale region progresses from (a) untransformed to (b) half-transformed to (c) ‘fully’ transformed (note significant transformation heterogeneity). White lines are grain boundaries. Arrows highlight peaks corresponding to elastically deformed austenite (A), transformed martensite (M), and plasticity (*). To highlight the deviation from a Gaussian distribution that is caused by the additional plastic contribution (*), a symmetric distribution centered around the representative martensite strain is outlined in (c) in red. Please see pdf for color.

We were recently able to experimentally quantify, to a high degree of accuracy, twins that are active in specific grains for the first time. We were also able to observe martensitic twin formation in grains under low macroscopic load levels, which is an unexpected and, to the best of our knowledge, previously unobserved phenomenon. An example of twins appearing during low macroscopically applied load is shown in Figure 2a in a ~ 50 micron square field of view. The thin white lines are grain boundaries, and the color bar on the right indicates axial strain. In Figure 2b, the quantified twin (type II) traces are denoted in white and exhibit a nearly perfectly overlay with the high strain (transformation) traces shown in Figure 2a.

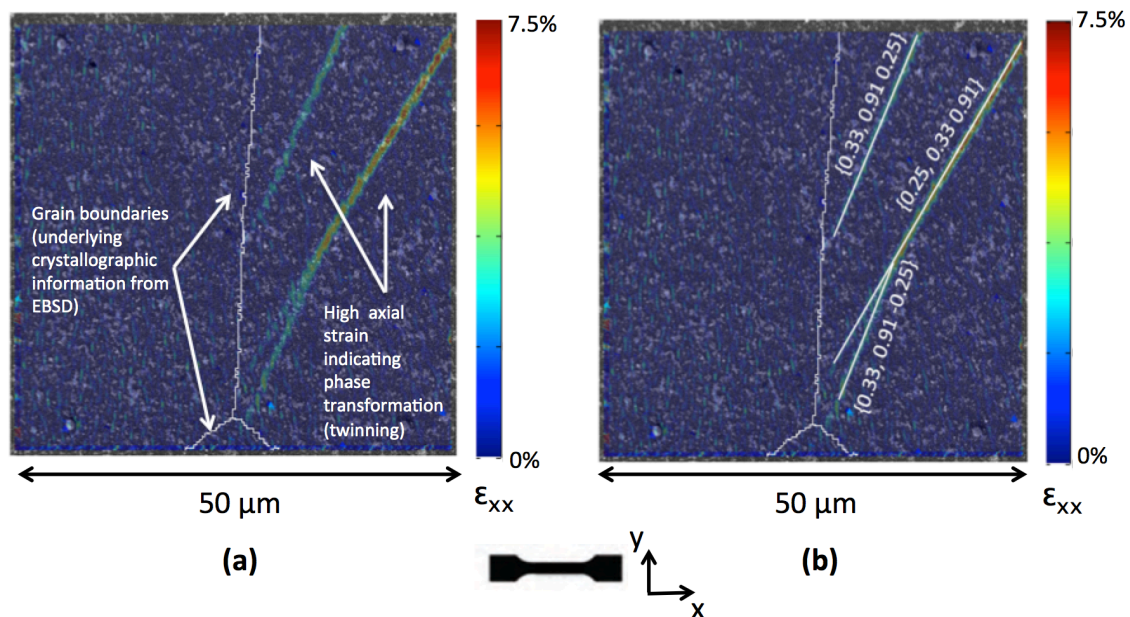


Figure 2: Type II twins quantitatively identified in an SMA under low load by tracking self-assembled nanoparticles on the surface of the specimen and using specialized microscopy techniques. The identification of twins at these low loads has, to our knowledge, not been observed before, and we are further investigating this phenomenon. Figure 2(a) shows the axial strain colormap indicating phase transformation (twins). Figure 2(b) shows the calculated identification traces in white overlaid with the experimentally observed strain traces, with a near perfect overlay. Please see pdf for color.

Given the large amount of new information and insights obtained from the small-scale testing, we focused on the microscale characterization of phase transformation for the last two years of the grant. To further investigate the existence of transformation memory at the microstructural length scale, we first determined a two-step aging treatment that maintained superelastic properties while increasing the average grain size to nominally 5 microns. Phase transformation at the sub-grain level and its relationship with respect to microstructure was investigated under displacement-controlled cyclic loading, resulting in several new findings. First, transformation was determined to be quite heterogeneous even on a sub-grain level, where several martensitic variants could be active inside a single grain. This is in contrast to a modeling assumption that a single most favorable variant will nucleate and subsume the entire grain. Secondly, the transformation was found to progress in a non-contiguous fashion through the propagation of a diffuse front at the microstructural length scale, which is the first time that this has been observed at this length scale and with this resolution. Third, similarly oriented grains were found to not transform similarly (either in terms of mean strain or strain heterogeneity), in contrast to a commonly held assumption in mean field theory. Fourth, sub-grain detwinning was found, a bit surprisingly, to occur prior to the end of the macroscopic stress-strain plateau. Because of the high accuracy of our measurements, we were able to quantitatively match local strain measurements to the specific correspondence variants and habit plane variants that were active in that area, and determine the amount of sub-grain detwinning present and its spatial sub-grain distribution. A journal paper detailing this mathematical analysis is currently being written, as is a publication detailing the relationship between sub-grain detwinning and the accumulation of damage. Graduate student M. Kimiecik will have five or six journal papers resulting from this work, which constituted his doctoral thesis and was fully funded by this award; three are currently published (*Acta Materialia*, *Advanced Materials and Processes*, *Materials Letters*) and two are currently being written on the completed analysis to be submitted this Summer.

(Kimiecik graduates in August 2015). One additional publication is likely on the effect of crystallographic texture on grain level transformation, as a joint publication with a new student in the Daly group who will be continuing this work. An example of grain-by-grain data demonstrating that similar grains do not transform similarly is shown in Figure 3 (Kimiecik et al., *Acta Mat*, 2015).

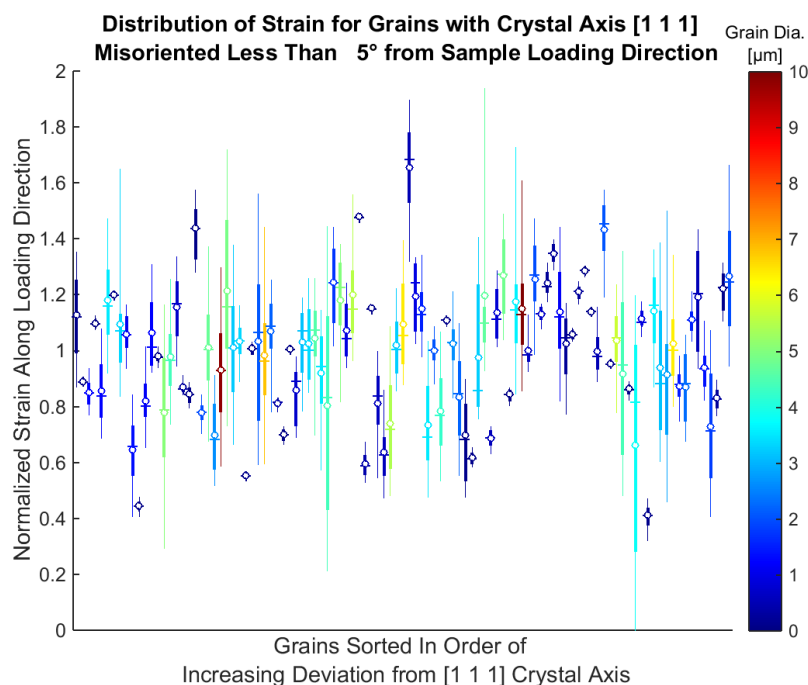


Figure 3: Individual grain strain distributions for grains oriented along different low-index crystallographic directions. To facilitate comparison between the different orientations, all strain values were normalized to the predicted transformation strain of twinned martensite, which is dependent on the crystal orientation. Approximately 5000 grains were sampled over a nominally $100\mu\text{m} \times 100\mu\text{m}$ imaging area; this shows only those misoriented less than 5 degrees from the sample loading direction to illustrate the point that similarly oriented grains do not transform similarly.

The following are specifics on the accomplished research tasks and responsible student:

- **SEM-DIC Spatial and Drift Distortion Corrections:** Complete. GSRA Adam Kammers published a paper on this methodology in *Exp. Mech.* in 2013 (under combined DoE and NSF funding).
- **Small-Scale Tracking Patterns for SEM-DIC Deformation Tracking:** Complete. We have found that nanoparticles and e-beam lithography can produce good tracking patterns when done correctly (nanoparticles in particular are very sensitive to a range of parameters). GSRA Adam Kammers published a paper on patterning methods in *J. Meas. Sci. Tech.* in 2011 and in *Exp. Mech.* in 2013 (under DoE and NSF funding).
- **In-SEM Experiments at the Microstructural Length Scale:** Complete. Experiments and analysis are complete. The student's doctoral thesis (and final journal papers) are being written, to be completed by GSRA Mike Kimiecik's graduation date of August 2015. Mike successfully quantified specific twin formation inside micron-sized grains in Nickel-Titanium through their

strain traces for the first time, developed a nanoparticle patterning method that allows us to examine the smallest (10 micron) fields of view to date, and has done some very nice work mapping specific habit plane variants (transformation modes) to sub-grain strains in order to examine the effects of detwinning its relationship to damage. One paper was just accepted to *Acta Materialia* and another is currently nearing completion (likely to be split into a two part series).

- **Low-Cycle Fatigue of SMAs** Complete. Kyubum Kim (GSRA) finished the tension and low-cycle experiments on bulk thin sheets, specifically examining the effect of cycling and of texture on the local thermo-mechanical characteristics of phase transformation. This was published in *Exp Mechanics* in 2011 and *Smart Mat. Struct.* in 2013.
- **SMAs in Structural Modes:** Complete. Ben Reedlunn (GSRA partially funded by DoE, graduated 2011) and Emily (undergrad funded by DoE who worked in the Daly labs from 2010-2011). Their work resulted in a comprehensive 30-page paper on SMAs in structural modes (bending, tension, compression) that was published in *JMPS*. Two other papers from Reedlunn's work were submitted on SMA cable deformation; both have been published in *IJSS*, both reached the top 10 most downloaded papers in the journal, and one won "Best Paper of 2013" for *IJSS*.
- **Analysis of Constitutive and Finite Element Models:** Complete. M. Kimiecik developed and validated a model to analyze active correspondence and habit plane variants given experimentally-obtained sub-grain transformation strains. This is an entirely new way of examining phase transformation and has directly investigated several outstanding hypotheses in the community (such as the commonly held assumption in mean field theory that similarly oriented grains transform similarly). This is being currently written into the doctoral thesis to be completed by August 2015 and submitted for journal publication.

IV. Products

Journal Papers

A "*" indicates partially funded by DoE, i.e. there were additional funding sources. A. Kammers received NSF funding and B. Reedlunn received GM funds during this period.

This grant resulted in two "Best Paper of the Year" journal awards – in *Experimental Mechanics* (2011) and *IJSS* (2013).

1. M. Kimiecik, J.W. Jones, S. Daly. The Relationship between Detwinning and Damage in the Shape Memory Alloy Nickel Titanium. *To be submitted (preprint available)*, 2015.
2. M. Kimiecik, J.W. Jones, S. Daly. Grain Orientation Dependence of Martensitic Phase Transformation in Polycrystalline Shape Memory Alloys. *Acta Materialia, In Press*, 2015.
3. M. Kimiecik, J.W. Jones, S. Daly. Quantitative Analysis of Phase Transformation in Ni-Ti Shape Memory Alloys. *Advanced Materials & Processes April 2013. (magazine publication)*
4. K. Kim, S. Daly. The Effect of Texture on Stress-Induced Martensite Formation in Nickel-Titanium. *Smart Mater. Struct.*, 22(7): 075012, 2013.
5. M. Kimiecik, J.W. Jones, S. Daly. A New Methodology for Tracking Phase Transformation in SMAs at the Microstructural Length Scale. *Materials Letters*, 95:25-29, 2013.
6. *A. Kammers, S. Daly. Digital Image Correlation Under Scanning Electron Microscopy: Methodology and Validation. *Experimental Mechanics*, 53: 1743-1761, 2013.
7. *B. Reedlunn, C. Churchill, E. Nelson, J. Shaw, S. Daly. Tension, Compression, and Bending of Superelastic Shape Memory Alloy Tubes. *JMPS, Accepted, 2013.*
8. *B. Reedlunn, S. Daly, J.A. Shaw. Superelastic Shape Memory Alloy Cables: Part I – Isothermal Tension Experiments. *IJSS*, 50(20-21): 3009-3026, 2013. ***(IJSS Best Paper Award for 2013).***
9. *B. Reedlunn, S. Daly, J.A. Shaw. Superelastic Shape Memory Alloy Cables: Part II – Isothermal Subcomponent Responses. *IJSS*, 50(20-21): 3027-3044, 2013.

10. K. Kim, S. Daly. Martensite Strain Memory in the Shape Memory Alloy NiTi under Mechanical Cycling. *Invited publication: Experimental Mechanics* 51(4): 641-652, 2011. **(Experimental Mechanics Best Paper Award for 2011.)**

Networks/Collaborations Fostered

- Prof. Michael Sangid, technical collaboration and upcoming DoE collaboration, resulting from complementary abilities developed from this award.
- Research collaboration with Prof. Dick James (Minnesota) and Prof. Marc DeGraef (CMU) regarding the microstructural aspects of SMAs, including student visits between institutions and joint experiments.
- Prof. Mike Sutton, USC. Technical collaboration resulting from developments funded by this work.
- Research discussions with Prof. Tony Rollett (CMU) to compare SEM-DIC microstructural deformation results with simulation.
- Ongoing discussion with Prof. Kaushik Dayal (CMU) regarding phase field models of transformation and the accuracy of various predictive models.
- Prof. Daly and students attended numerous conferences (SMST, SEM, SES, TMS, ASME) and DoE program reviews, resulting in student development, the development of existing collaborations, and the creation of new contacts.

Instruments/Equipment

- Development of SEM-DIC methodology, including associated software and hardware (funded by both NSF and DoE).

Invited Talks Directly Related to This Project

1. UC Riverside, May 2015.
2. HRL Laboratories, March 2015.
3. UC Santa Barbara, March 2015.
4. Caltech, January 2015.
5. Los Alamos National Laboratory, Materials Science, October 2014.
6. Society of Experimental Mechanics, June 2014. **(Journal of Strain Analysis Young Investigator Lecture)**
7. Harvard University, April 2014.
8. Cornell University, April 2014. **(Lindseth Lecture)**.
9. Medtronic, September 2013.
10. Columbia University, September 2013.
11. Banff Centre, Mathematics and Mechanics in the Search for New Materials, July 2013.
12. Society of Engineering Science (SES) Conference, Symposium on Mechanics of Phase Transforming and Multifunctional Materials – **Keynote Speaker**, July 2013.
13. Stanford University, June 2013.
14. Case Western Reserve University, April 2013.
15. Brown University, February 2013.
16. Saturday Morning Physics, University of Michigan Physics Department, February 2013.
Available online at <http://lecb.physics.lsa.umich.edu/CWIS/SPT--BrowseResources.php?ParentId=702>
17. University of Houston, December 2012.
18. Carnegie Mellon University, October 2012.
19. University of Wisconsin – Madison. September 2011.
20. Ohio State University, May 2011.

Symposia Organized Directly Related to This Project

- Symposium Organizer for 2014 Society of Experimental Mechanics Conference (SEM), Fracture and Fatigue Chair (6 sessions)
- Symposium Organizer for 2014 Society of Engineering Science (SES) Conference, Microscale and Microstructural Effects on Mechanical Behavior
- Symposium Organizer for 2014 Society of Engineering Science (SES) Conference, Symposium on the Mechanics of Phase Transforming and Multifunctional Materials
- Symposium Organizer for Symposium Organizer for 2013 Society of Experimental Mechanics Conference (SEM), Fracture and Fatigue Chair (6 sessions)
- Symposium Organizer for 2012 Society of Engineering Science (SES) Conference
- Symposium Organizer for 2011 Society of Engineering Science (SES) Conference

V. Updated List of Other Support (Current and Pending) and Indication of Any Overlap

Support: Current

Project/Proposal Title: Deformation, Damage, and Failure Mechanisms in Ceramic Matrix Composites (co-PI; PI –J.Halloran) [UM# N018187-352635]
Source of Support: University of Michigan (GE-Aviation)
Total Award Amount: \$300,000/year; my share \$100,000/year
Total Award Period: 5/14/2010-current (renewed yearly)
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.5 Sumr: 1.0

Support: Current

Project/Proposal Title: Fracture under Ultrasonic Fatigue (PI; PI – S. Daly) [UM# N012240-399481]
Source of Support: University of Michigan (General Motors)
Total Award Amount: \$50,000
Total Award Period: 1/01/2011- (gift)
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.0

Support: Current

*** Overlap; undergraduate working on this project used nanoparticle and mapping techniques for NiTi developed under DoE grant.**

Project/Proposal Title: Fracture of SMA Microwires (PI; PI – S. Daly) [UM# N013417-347689]
Source of Support: University of Michigan (General Motors)
Total Award Amount: \$50,000
Total Award Period: 1/01/2011- (gift)
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.0

Support: Current

Project/Proposal Title: Integration of Advanced Analysis and Materials Research: Capturing Process Sensitive Materials Information in Compact, Computationally Efficient Models (co-PI; PI – J.Allison) [UM# F030551-070158]
Source of Support: University of Michigan (ONR)
Total Award Amount: \$3,350,745; my share \$356,056
Total Award Period: 9/01/2011-1/1/2016
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.5

Support: Current

Project/Proposal Title: CAREER: Understanding Micromechanisms of Fatigue in Shape Memory Alloys (PI; PI – S. Daly [UM# F032822-072523])
Source of Support: University of Michigan (NSF)
Total Award Amount: \$400,000; my share \$400,000
Total Award Period: 1/01/2013-12/31/2017
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.0

Support: Current

Project/Proposal Title: YIP: A New Approach Towards Characterizing Microstructural Influence on Material Behavior Under Very High Cycle Fatigue (PI; PI – S. Daly [UM# F031559-071175])
Source of Support: University of Michigan (NSF)
Total Award Amount: \$358,018; my share \$358,018
Total Award Period: 9/01/2012-8/31/2015
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.0

Support: Current

Project/Proposal Title: Software Center for Predictive Theory and Modeling (co-PI; PI – J. Allison [UM# F032231-071880])
Source of Support: University of Michigan (NSF)
Total Award Amount: \$11,000,000; my share \$386,414
Total Award Period: 9/01/2012-8/31/2017
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months/Yr Committed to the Project: Cal: 0 Acad: 0.0 Sumr: 0.5

Support: Pending

Project/Proposal Title: Developing Three-Dimensional, Large Data Experimental and Statistical Approaches to Characterize the Effects of Subsurface Microstructure (PI; PI – S. Daly)
Source of Support: University of Michigan (NSF)
Total Award Amount: \$326,364; my share \$326,364
Total Award Period: 9/1/2015-8/31/2018
Location of Project: Univ of Michigan, Ann Arbor, MI
Person-Months Per Year Committed to the Project: Cal: 0 Acad: 0 Sumr: 0.5

Support: Pending

Project/Proposal Title: Deformation Mechanisms at Grain Boundaries in Polycrystals (co-PI; PI – M.Sangid)
Source of Support: Purdue/University of Michigan (DOE-BES)
Total Award Amount: \$660,000; my share \$384,000
Total Award Period: 7/1/2015-6/30/2018
Location of Project: Purdue, West Lafayette IN and Univ of Michigan, Ann Arbor, MI
Person-Months Per Year Committed to the Project: Cal: 0 Acad: 0 Sumr: 1.0

Support: **Pending**

Project/Proposal Title: Characterizing the Microstructure-Dependence of Crack Initiation and Growth in Additive-Manufacture Titanium Alloy Components (co-PI; PI – K. Dayal)

Source of Support: CMU/University of Michigan (NSF)

Total Award Amount: \$317,892; my share \$167,892

Total Award Period: 9/1/2015-8/31/2018

Location of Project: Carnegie Mellon, Pittsburgh PA and Univ of Michigan, Ann Arbor, MI

Person-Months Per Year Committed to the Project: Cal: 0 Acad: 0 Sumr: 0

VI. Cost Status

Not applicable, as this is a final report.