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Attention: Dr. John Vetrano

Submitted by:

Principal Investigator: Professor William D. Nix
Department of Materials Science and Engineering
Stanford University, Stanford California 94305

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Preface

The three-year grant for which this final report is required extends from 2011 to 2015, including a one-year, no-cost extension. But this is just the latest in a long series of grants from the Division of Materials Sciences of DOE and its predecessor offices and agencies. These include contracts or grants from: the Metallurgy Branch of the U.S. Atomic Energy Commission (from the late 1960s to the mid-1970s), the Materials Science Program of the U.S. Energy Research and Development Administration (from the mid- to late- 1970s), and the Division of Materials Science of the Office of Basic Energy Sciences of the U.S. Department of Energy (from the early 1980s to the present time). Taken all together, these offices have provided nearly continuous support for our research for nearly 50 years. As we have said on many occasions, this research support has been the best we have ever had, by far. As we look back on the nearly five decades of support from the Division of Materials Sciences and the predecessor offices, we find that the continuity of support that we have enjoyed has allowed us to be most productive and terms of papers published, doctoral students graduated and influence on the field of materials science. This report will, of course, cover the three-year period of the present grant, in summary form, but will also make reference to the output that resulted from support of previous grants from the Division of Materials Sciences and its predecessor offices.

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I. Introduction

This report begins with a brief account of the accomplishments of the current grant in section II. There we summarize the recent work and call attention to the publications that came from the work and the students who completed their doctoral dissertations during the just completed grant period. In section III of the report we look back to previous grants and highlight the major accomplishments that came from that previous support.

Section IV of the report is a list of all of the publications that have resulted from DOE grant support over the past 10 years. This is followed, in section V, by a listing of the six people who completed their Ph.D. degrees with DOE grant support in that same 10 year period. The abstracts of the dissertations of those six people are reprinted in section VI. This work is, of course, documented in more detail in the listed published papers. The last section of the report, section VII, is a biographical sketch of Prof. Nix. The accomplishments and honors listed there are due, in part, to the steady research support from DOE that he has enjoyed for nearly 50 years.

II. Accomplishments of the current grant

We have been engaged in a program of research on the mechanical properties of materials with micrometer and nanometer scale dimensions and microstructures. This has included work on the mechanical properties of crystalline materials in small volumes, as well as research on the emerging problem of mechanical properties of lithiated silicon nanostructures for lithium-ion batteries.

Research on size effects on strength and plasticity

The research on small-scale plasticity has focused on understanding the dislocation processes responsible for the well known “smaller is stronger” effect on strength, that we, and others, discovered a few years ago, using the micropillar compression technique that we also introduced. Our work has included micropillar compression experiments, as well as computational modeling, using a dislocation dynamics code based on the ParaDis code developed at the Lawrence Livermore National Laboratory. The overall aim of this work has been to determine the role that dislocations play in controlling strength at small scales. The modeling work was done in collaboration with Professor Wei Cai of the Division of Computational and Applied Mechanics in the Department of Mechanical Engineering at Stanford.

Our experimental work on size effects using the micropillar compression technique was completed when Seok-Woo Lee finished his Ph.D. degree in 2011. In part of his work he resolved the problem of why size effects on strength, as indicated by the exponents relating yield strength to pillar diameter, are typically much smaller for BCC metals compared to FCC metals. He found a very simple reason for this difference: BCC metals, which typically have much higher intrinsic strengths because of lattice friction, have yield strengths that are much less affected by the pillar diameter. Indeed, for BCC metals with very small friction stresses, like V, the size effects are very similar to those for FCC metals, as expected. According to this view, the exponent describing the size effect should be close to 1.0 unless other size independent contributions to the strength are present, whereupon the exponent is smaller and can approach 0 for materials that are intrinsically very strong. This finding was documented in the following paper:

S-W. Lee and W.D. Nix, "Size dependence of the yield strength of fcc and bcc micropillars with diameters of a few micrometers," *Philosophical Magazine*, **92**, 1238-1260 (2012).

Notwithstanding this contribution and the contributions of several other authors who have reached the same conclusion, one still finds too many authors attaching too much significance to the irrational exponents that are found experimentally.

Our modeling work using the ParaDis DD code, modified for micropillar plasticity, has focused on understanding size effects on strength without invoking artificial dislocation sources or pinning points, as was done in some of the initial DD modeling of size effects by others. In particular, we have attempted to include only "natural" dislocation sources in our modeling and only pinning points that are formed naturally by dislocation reactions. In the case of BCC metals it had been shown, through MD modeling, that natural cross-slip processes occur where dislocation segments terminate at free surfaces and lead naturally to dislocation sources. We developed a DD model of this mechanism and used it to study size dependent plasticity in BCC metals. The result was a paper published in 2013 showing how the mechanical properties of BCC micropillars could be explained in terms of the competition between dislocation multiplication and depletion:

I. Ryu, W.D. Nix and W.Cai, "Plasticity of BCC micro-pillars controlled by competition between dislocation multiplication and depletion," *Acta Materialia*, **61**, 3233-3241 (2013).

We have recently extended this modeling work to include FCC metals where classical nucleation of dislocations at surfaces was included. The simulation results show stochastic behaviors, in agreement with experimental observations, and reveal that dislocation nucleation at the free surface is the dominant mechanism of plastic flow in small pillars with diameters less than 200 nm, while the operation of truncated dislocation sources is the governing mechanism in large pillars with diameters exceeding 1 micron. In between, both mechanisms come into play in a stochastic way. This is described in the following paper:

I. Ryu, W. Cai, W.D. Nix, and H. Gao, "Stochastic behaviors in plastic deformation of face-centered-cubic micropillars by surface nucleation and truncated source operation" *Acta Materialia*, **95**, 176-183 (2015).

Research of lithiated Si nanostructures

Our study of the mechanical properties of lithiated silicon nanostructures has involved a collaboration with Professor Yi Cui of our department and his research group. The work has involved creating silicon nanostructures, subjecting them to various kinds of electrochemical lithiation and delithiation, studying the shape changes and fracture processes that may accompany these processes and modeling these events using both analytical and finite element methods. The overall aim of the work has been to develop a fundamental understanding of the decrepitation (fracture) processes that currently limit the use of silicon as electrodes in lithium ion batteries.

An early effort to understand decrepitation of silicon during electrochemical lithiation and delithiation involved the application of the theory of electrochemical shock to the fracture of silicon nanowires, analogous to the theory of thermal shock of glasses and ceramics. Using that theoretical approach, we were able to rationalize the existence of a critical size below which decrepitation (fracture) should not occur, in agreement with experiment. This is described in the following paper:

> I. Ryu, J.W. Choi, Y. Cui, and W. D. Nix, "Size-dependent fracture of Si nanowire battery anodes," *Journal of the Mechanics and Physics of Solids,* **59**, 1717-1730 (2011).

Subsequent in-situ TEM studies revealed that the lithiation of crystalline Si does not involve the diffusion of Li into the Si lattice, as envisioned by the theory of electrochemical shock, but instead involves a crystalline to amorphous phase transformation, wherein a lithiated amorphous layer develops at the surface and grows inward and consumes the crystalline Si core. This leads to a completely different picture of how fracture occurs during lithiation of crystalline Si. In brief, the continued lithiation and swelling of the Si core pushes and stretches the existing amorphous shell and that can cause fracture to occur at the surface. The critical pillar diameter below which fracture does not occur is about 300nm. In collaboration with the Yi group, we have modeled and studied this failure process in the following papers:

> S-W. Lee, M.T. McDowell, L.A. Berla, W.D. Nix and Y. Cui, "Fracture of silicon nanopillars during electrochemical lithium insertion," *Proceedings of the National Academy of Sciences of the United States of America*, **109**, 4080-4085 (2012).

> M.T. McDowell, I. Ryu, S-W. Lee, C.M. Wang, W.D. Nix and Y. Cui, "Studying the kinetics of crystalline silicon nanoparticle lithiation with in situ transmission electron microscopy," *Advanced Materials*, **24**, 6034 (2012).

> I. Ryu, S-W. Lee, H. Gao, Y.Cui and W.D. Nix, "Microscopic model for fracture of crystalline Si nanopillars during lithiation," *Journal of Power Sources*, **255**, 274-282 (2014).

These studies revealed that fracture and decrepitation of Si anodes is closely associated with the crystalline to amorphous transformation. We, and others, concluded that some of these fracture problems could be avoided by using amorphous Si as the anode material, so that the stress generating phase transformation could be avoided. Subsequent studies did show that amorphous Si is much more fracture resistant than crystalline Si. In an effort to determine the critical size for fracture of amorphous Si we created micropillars of amorphous Si and observed that even pillars as large as 2.3 microns in diameter do not fracture on lithiation and delithiation, indicating a critical size at least an order of magnitude larger than for crystalline Si. This latter work was reported in the following paper:

L.A. Berla, S-W. Lee, I. Ryu, Y.Cui and W.D. Nix, "Robustness of amorphous silicon during the initial lithiation/delithiation cycle," *Journal of Power Sources*, **258**, 253-259 (2014).

In the course of these studies it became clear that knowledge of the mechanical properties of lithiated Si was limited and that an effort should be made to acquire this data. We responded by developing a technique for studying the mechanical properties of lithiated Si using nanoindentation under the protection of mineral oil. That work was completed in 2014 and the paper describing that work and those results is:

L.A. Berla, S-W. Lee, Y.Cui and W.D. Nix, "Mechanical behavior of electrochemically lithiated silicon," *Journal of Power Sources*, **273**, 41-51 (2015).

We reported a significant creep effect in lithiated amorphous Si that deserves further study.

III. Some accomplishments resulting from previous grants

Our work in materials science at Stanford now spans more than 50 years. Throughout that period we have been engaged in teaching materials science, mainly at the graduate level, and working with doctoral students on the mechanical behavior of materials in both bulk and thin film form. The Division of Materials Sciences of DOE provided the primary support for this research for most of that time. The last doctoral student defended his dissertation in 2014. He was the 79^{th} Ph.D. student to have studied in our group. Some people take note of the relative large number of our students who have become professors. More than 40% of our former students either hold or have held professorships around the world, including professorships at Harvard, Caltech, Cornell, Johns Hopkins, University of Connecticut, Ohio State University, University of Tennessee, KAIST, University of California (Davis), University of California (Irvine), University of California (Riverside), and others. Our work with these students has led to quite a number of awards from materials-related societies, as described in the biographical sketch in section VII of this report. It shows that Prof. Nix is a member of both the National Academy of Engineering and the National Academy of Sciences and a Fellow of the American Academy of Arts and Sciences. These honors and other awards were made possible by the DOE support that he has enjoyed.

From the mid-1960's to the mid-1980's most of our work was on mechanical properties of bulk materials for high temperature applications. That work included work on the mechanisms of diffusional creep, superplastiticy, dispersion strengthened metals and intergranular fracture. But by the mid-1980's it became clear to us that a number of mechanical behavior problems were arising in microelectronics and other thin film technologies that needed attention. But methods for studying mechanical behavior at the sub-micrometer scale were not broadly available at that time and very little attention had been given to understanding mechanical properties of materials at that scale. So we turned our attention to problems of mechanical behavior of thin films and other small-scale structures. That line of research resulted in our most highly cited works.

In 1984 we became aware of the development of the first commercially successful nanoindenter by Nix's former student, Warren Oliver, and his colleague John Pethica (now Sir John Pethica).

We realized that this instrument could be very useful in studying the mechanical properties of thin film materials of interest in microelectronics. We acquired the first such instrument from their company (Nano Instruments Inc.) and began using it to study the mechanical properties of thin films. The nanoindenter is an instrument that allows a very sharp diamond indenter to be pushed into the surface of a solid while measuring both the required force and resulting displacement with high resolution. With Mary Doerner we developed the first broadly applicable method for analyzing the indentation experiments and extracting fundamental mechanical properties such as hardness and elastic modulus. The essence of this paper involves the use of basic results from the theory of contact mechanics to determine the elastic moduli of the contacting materials from the measured stiffness. Here we refer to:

M.F. Doerner and W.D. Nix, "A Method for Interpreting the Data from Depth-Sensing Indentation Instruments", *J. Materials Research*, **1**, 601 (1986).

An improved analysis method, based partly on the Doerner-Nix paper, was published six years later by Warren Oliver and George Pharr, two of Nix's former students. Their paper, in turn, has become THE most highly cited paper in the field of materials science.

We developed a number of other techniques for studying the mechanical properties of thin films on substrates, including substrate curvature methods and microbeam deflection methods using the nanoindenter. These methods and the results that come from them were described in the Mehl Lecture given to the Metallurgical Society (TMS) in 1988. We subsequently published that work in a review paper on mechanical properties of thin films, which became a most highly cited paper:

W.D. Nix, "Mechanical Properties of Thin Films", *Metall. Trans.*, **20A**, 2217 (1989).

Our indentation experiments with self-similar pyramidal indenters indicated that at the sub-micrometer scale hardness is not independent of depth as it is at the bulk scale. An effect characterized by "smaller is stronger" was observed. With Huajian Gao we developed a theory of this effect that has now been widely accepted. The basic idea is that indenting a crystal with a sharp pyramidal indenter imposes gradients of plastic strain on the indented material that require the existence of geometrically necessary dislocations that cause the hardness to be greater than it would be without these extra dislocations. The dimensions of the problem are such that a higher density of such geometrically necessary dislocations is expected at smaller depths of indentation and this explains the indentation size effect.

The discovery of the indentation size effect and the theory we developed has also influenced the field of continuum mechanics. Ordinary continuum plasticity has no internal length scales, so that the stresses needed for plastic flow in a large volume are the same as those required in a small volume. But such classical continuum theories are powerless to describe what happens at a small scale where size effects are observed. The equations we developed for describing the indentation size effect have been generalized by Huajian Gao and Yonggang Huang and used to describe what happens in other kinds of deformation where strong gradients of plastic strain are created, such as at crack tips or in severe bending or torsion. They call this Mechanism-based Strain Gradient Plasticity wherein the microscopic mechanism responsible for the size effect is included as one of the essential elements of the theory. Through this development we now have, for the first time, a continuum theory of plasticity that includes microscopic mechanisms as one of the founding principles. Here we refer to:

W.D. Nix and H. Gao, "Indentation Size Effects in Crystalline Materials: A Law for Strain Gradient Plasticity," *J. Mechanics and Physics of Solids*, **46**, 411-425 (1998).

The "smaller is stronger" effect can arise from gradients of plastic strain that are created in indentation with pyramidal indenters and with bending and torsion. But such gradients are not expected to be created in uniaxial deformation which is approximately homogeneous. So it was of interest to mechanically test materials at a small scale without the gradients of plastic strain that are associated with ordinary indentation. About 10 years ago we found a way to conduct such experiments by combining the capabilities of the newly available focused ion beam machining methods with the nanoindenter using a flat ended punch as an indenter. Basically cylindrical specimens ranging in diameter from about 100 nm to many micrometers are created using focused ion beam machining and the micro-pillars are then tested in uniaxial compression using the nanoindenter fitted with a flat ended punch. The result is an ordinary stress-strain response, much like that used for bulk materials. The surprising finding was that there is a very strong size effect, "smaller is stronger" in spite of the absence of strong gradients thought to be responsible for this effect in other deformation experiments. This finding started a new wave of research on size effects that led to a world-wide effort to understand these effects using the theory of dislocations. The consensus now is that the size effect we observed arises because yielding in these small crystals is dislocation-source controlled. That is, because of the close proximity of free surfaces, dislocations readily escape from the crystal and have to be continually replaced by the creation of new ones at sources. Operating these sources controls the strength. This is unlike bulk materials where the dislocations have little chance to escape from the deforming material and where the interaction of dislocations controls deformation and strength. In addition to uncovering new mechanisms that control strength of small-scale materials, the micropillar compression technique that we developed is now widely used to assess the mechanical properties of a wide variety of materials where only small volumes of materials are available or are of interest. The papers that initiated this line of research are:

M.D. Uchic, D.M. Dimiduk, J.N. Florando and W.D. Nix, "Sample dimensions influence strength and crystal plasticity," *Science*, **305**, 986-9 (2004).

Julia R. Greer, Warren C. Oliver and William D. Nix, "Size Dependence of Mechanical Properties of Gold at the Micron Scale in the Absence of Strain Gradients," *Acta Materialia*, **53**, 1821-1830 (2005).

While nanoindentation was a well-established method for determining mechanical properties of homogeneous materials by the mid-1990's, the problem of determining the properties of thin films on substrates had not been fully addressed. The problem is that the substrate can have a strong effect on the indentation response even if the indenter makes only contact with the film, especially if the properties of the substrate differ from those of the film (the typical case). With Ranjana Saha we developed robust ways to take substrate effects into account in one of our more highly cited papers. Here we refer to:

Ranjana Saha and William D. Nix, "Effects of the substrate on the determination of thin film mechanical properties by nanoindentation," *Acta Materialia*, **50**, 23-38 (2002).

More recently our attention has been focused on modeling strength and plasticity in nanopillars and exploring the mechanical properties of lithiated Si, as described in section II. The papers are attracting much attention and are quickly becoming some of our most highly cited works.

IV. Some publications relating to current and recent grants (past 10 years)

The following is a list of the papers coming from our DOE grant that have been published during the past 10 years.

1. J.N. Florando and W.D. Nix, "A microbeam bending method for studying stress–strain relations for metal thin films on silicon substrates," *J. Mech. Phys and Solids*, **53**, 619-638 (2005).

2. Julia R. Greer, Warren C. Oliver and William D. Nix, "Size Dependence of Mechanical Properties of Gold at the Micron Scale in the Absence of Strain Gradients," *Acta Materialia*, **53**, 1821-1830 (2005).

3. Julia R. Greer and William D. Nix, "Size Dependence of Mechanical Properties of Gold at the Sub-Micron Scale," *Applied Physics A: Materials Science and Processing*, **80**, 1625-1629 (2005).

4. Dae-han Choi and W.D. Nix, "Anelastic behavior of Copper thin films on Silicon substrates: Damping associated with dislocations," *Acta Materialia*, **54**, 679-687 (2006).

5. E.T. Lilleodden and W.D. Nix, "Microstructural length-scale effects in the nanoindentation behavior of thin gold films," *Acta Materialia*, **54**, no.6, p.1583-1593 (2006).

6. S.M. Han, R. Saha and W.D. Nix, "Determining hardness of thin films in elastically mismatched film-on-substrate systems using nanoindentation," *Acta Materialia*, **54**, no.6, p.1571-1581 (2006).

7. D.H. Choi and W.D. Nix, "Anelastic behavior of copper thin films on silicon substrates: Damping associated with dislocations," *Acta Materialia*, **54**, no.3, p.679-687 (2006).

8. J.R. Greer and W.D. Nix, "Nanoscale gold pillars strengthened through dislocation starvation," *Physical Review B*, **73**, no.24, p.245410-1-6 (2006).

9. William D. Nix, Julia R. Greer, Gang Feng and Erica T. Lilleodden, "Deformation at the Nanometer and Micrometer Length Scales: Effects of Strain Gradients and Dislocation Starvation," *Thin Solid Films*, **515**, 3152-3157 (2007).

10. F. Zhang, R. Saha, Y. Huang, W.D. Nix, K.C. Hwang, S. Qu and M. Li, “Indentation of a hard film on a soft substrate: Strain gradient hardening effects,” *International Journal of Plasticity*, **23**, 25–43 (2007).

11. A.S. Budiman, S.M. Han, J.R. Greer, N. Tamura, J.R. Patel, W.D. Nix, “A search for evidence of strain gradient hardening in Au submicron pillars under uniaxial compression using synchrotron X-ray microdiffraction,” *Acta Materialia*, **56**, 602–608 (2008).

12. Jia Zhu, Hailin Peng, A.F. Marshall, D.M. Barnett, W.D. Nix and Yi Cui, “Formation of chiral branched nanowires by the Eshelby Twist,” *Nature Nanotechnology*, **3**, No. 8, 477-481 (2008).

13. G. Feng, A.S. Budiman, W.D. Nix, N. Tamura and J.R. Patel, “Indentation size effects in single crystal copper as revealed by synchrotron x-ray microdiffraction,” *J. Applied Physics*, **104**, 043501 (2008).

14. W.D. Nix, “Yielding and Strain Hardening in Metallic Thin Films on Substrates: an Edge Dislocation Climb Model,” *Mathematics and Mechanics of Solids*, **14**, 207 (2009).

15. William D. Nix, “Exploiting new opportunities in materials research by remembering and applying old lessons,” *MRS Bulletin*, **34**, 82-91 (2009).

16. Seok-Woo Lee, Seung Min Han, William D. Nix, “Uniaxial compression of fcc Au nanopillars on an MgO substrate: The effects of prestraining and annealing,” *Acta Materialia*, **57**, 4404–4415 (2009).

17. Seung Min Han, Mark A. Phillips, William D. Nix, “Study of strain softening behavior of Al–Al3Sc multilayers using microcompression testing,” *Acta Materialia*, **57**, 4473–4490 (2009).

18. C.R. Weinberger, S. Aubry, S-W Lee, W.D. Nix and W. Cai, “Modeling dislocations in a free standing thin film,” *Modeling and Simulation in Materials Science and Engineering*, **17**, No.7, 075007 (2009).

19. S-W. Lee and W.D. Nix, "Geometrical analysis of 3D dislocation dynamics simulations of FCC micro-pillar plasticity," *Materials Science and Engineering A-Structural Materials Properties Microstructure and Processing,* **527**, 1903-1910 (2010).

20. M.A. Hopcroft, W.D. Nix and T.W. Kenny, “What is Young’s modulus of silicon,” *Journal of Electromechanical Systems*, **19**, 229-238 (2010).

21. L.A. Berla, A. Allen, S.M. Han and W.D. Nix, “A Physically Based Model for Indenter Tip Shape Calibration for Nanoindentation,” *Journal of Materials Research*, **24**, 735-745 (2010).

22. A.S. Budiman, C. S. Hau-Riege, W. C. Baek, C. Lor, A. Huang, H. S. Kim, G. Neubauer, J. Pak, P. R. Besser, and W. D. Nix, "Electromigration-Induced Plastic Deformation in Cu Interconnects: Effects on Current Density Exponent, n, and Implications for EM Reliability Assessment," *Journal of Electronic Materials,* **39,** 2483-2488 (2010).

23. S.M. Han, T. Bozorg-Grayeli, J.R. Groves, and W.D. Nix, "Size effects on strength and plasticity of vanadium nanopillars," *Scripta Materialia,* **63**, 1153-1156 (2010).

24. W.D. Nix and S.W. Lee, "Micro-pillar plasticity controlled by dislocation nucleation at surfaces," *Philosophical Magazine,* **91**, 1084-1096 (2011).

25. W.D. Nix, "Beyond the Lab Develop and Follow Your Own Passion," *MRS Bulletin,* **36**, 14-15 (2011).

26. S-W. Lee, S. Aubry, W.D. Nix, and W. Cai, "Dislocation junctions and jogs in a free-standing FCC thin film," *Modelling and Simulation in Materials Science and Engineering,* **19**, 025002 (2011).

27. S.M. Han, E.P. Guyer, and W.D. Nix, "Extracting thin film hardness of extremely compliant films on stiff substrates," *Thin Solid Films,* **519**, 3221-3224 (2011).

28. Y. Yao, M.T. McDowell, I. Ryu, H. Wu, N.A. Liu, L.B. Hu, W.D. Nix, and Y. Cui, "Interconnected Silicon Hollow Nanospheres for Lithium-Ion Battery Anodes with Long Cycle Life," *Nano Letters,* **11**, 2949-2954 (2011).

29. S-W. Lee, D. Mordehai, E. Rabkin, and W.D. Nix, "Effects of focused-ion-beam irradiation and prestraining on the mechanical properties of FCC Au microparticles on a sapphire substrate," *Journal of Materials Research,* **26**, 1653-1661 (2011).

30. D. Mordehai, S-W. Lee, B. Backes, D.J. Srolovitz, W.D. Nix, and E. Rabkin, "Size effect in compression of single-crystal gold microparticles," *Acta Materialia,* **59**, 5202-5215 (2011).

31. I. Ryu, J.W. Choi, Y. Cui, and W. D. Nix, "Size-dependent fracture of Si nanowire battery anodes," *Journal of the Mechanics and Physics of Solids,* **59**, 1717-1730 (2011).

32. M.T. McDowell, S-W. Lee, I. Ryu, H. Wu, W.D. Nix, J.W. Choi, and Y. Cui, "Novel Size and Surface Oxide Effects in Silicon Nanowires as Lithium Battery Anodes," *Nano Letters,* **11**, 4018-4025 (2011).

33. S-W. Lee and W.D. Nix, "Size dependence of the yield strength of fcc and bcc micropillars with diameters of a few micrometers," *Philosophical Magazine*, **92**, 1238-1260 (2012).

34. S-W. Lee, M.T. McDowell, L.A. Berla, W.D. Nix and Y. Cui, "Fracture of silicon nanopillars during electrochemical lithium insertion," *Proceedings of the National Academy of Sciences of the United States of America*, **109**, 4080-4085 (2012).

35. M.T. McDowell, I. Ryu, S-W. Lee, C.M. Wang, W.D. Nix and Y. Cui, "Studying the kinetics of crystalline silicon nanoparticle lithiation with in situ transmission electron microscopy," *Advanced Materials*, **24**, 6034 (2012).

36. S-W. Lee, L.A. Berla, M.T. McDowell, W.D. Nix and Y. Cui, "Reaction front evolution during electrochemical lithiation of crystalline silicon nanopillars," *Israel Journal of Chemistry*, **52**, 1118-1123 (2012).

37. S.M. Han, G. Feng, J.Y. Jung, H.J. Jung, J.R. Groves, W.D. Nix and Y. Cui, "Critical-temperature/Peierls-stress dependent size effects in body centered cubic nanopillars," *Applied Physics Letters*, **102**, 041910 (2013).

38. M.T. McDowell, S-W. Lee, J.T. Harris, B.A. Korgel, W.D. Nix and Y.Cui, "In-situ TEM of two-phase lithiation in amorphous silicon nanospheres," *Nano Letters*, **13**, 758-764 (2013).

39. I. Ryu, W.D. Nix and W.Cai, "Plasticity of BCC micro-pillars controlled by competition between dislocation multiplication and depletion," *Acta Materialia*, **61**, 3233-3241 (2013).

40. M.T. McDowell, S-W. Lee, W.D. Nix and Y. Cui, "Understanding the Lithiation of Silicon and Other Alloying Anodes for Lithium Ion Batteries," *Advanced Materials*, **25**, 4966-4984 (2013).

41. W. Cai, R.B. Sills, D.M. Barnett, and W.D. Nix, "Modeling a distribution of point defects as misfitting inclusions in stressed solids," *Journal of the Mechanics and Physics of Solids*, **66**, 154-171 (2014).

42. I. Ryu, S-W. Lee, H. Gao, Y.Cui and W.D. Nix, "Microscopic model for fracture of crystalline Si nanopillars during lithiation," *Journal of Power Sources*, **255**, 274-282 (2014).

43. L.A. Berla, S-W. Lee, I. Ryu, Y.Cui and W.D. Nix, "Robustness of amorphous silicon during the initial lithiation/delithiation cycle," *Journal of Power Sources*, **258**, 253-259 (2014).

44. M. Sebastiani, C. Eberl, E. Bemporad, A.M. Korsunsky, W.D. Nix and F. Carasseti, "Focussed ion beam four-slot milling for Poisson's ratio and residual stress evaluation at the micron scale," *Surface and Coatings Technology*, **251**, 151-161 (2014).

45. L.A. Berla, S-W. Lee, Y.Cui and W.D. Nix, "Mechanical behavior of electrochemically lithiated silicon," *Journal of Power Sources*, **273**, 41-51 (2015).

46. S-W. Lee, H.W, Lee, I. Ryu, W.D. Nix, H. Gao and Y.Cui, "Kinetics and fracture resistance of lithiated silicon nanostructure pairs controlled by their mechanical interaction," *Nature Communications*, **6**, 7533 (2015).

47. S-W. Lee, I. Ryu, W.D. Nix, and Y.Cui, “Fracture of crystalline germanium during electrochemical lithium insertion,” *Extreme Mechanics Letters*, **2**, 15-19 (2015).

48. I. Ryu, W. Cai, W.D. Nix, and H. Gao, “Stochastic behaviors in plastic deformation of face-centered-cubic micropillars by surface nucleation and truncated source operation” *Acta Materialia*, **95**, 176-183 (2015).

49. K.C. Bennett, L.A. Berla, W.D. Nix, and R.J. Borja, “Instrumented nanoindentation and 3D mechanistic modeling of a shale at multiple scales” *Acta Geotechnica*, **10**, 1-14 (2015).

V. Recent doctoral students relating to the current and previous grants (past 10 years)

The training of Ph.D. students has always been an important feature of this research program. Below is a list of the Ph.D. students who have been supported by recent DOE grants, including the one just concluded. Not shown are the names of 29 Ph.D. students who were supported by previous (DOE, ERDA and AEC) grants. This list is shown here to give an indication of the contribution that is made through the training of Ph.D. students. The last three on this list, Seok-Woo Lee, Ill Ryu and Lucas Berla, all received their degrees during the current grant period.

We note that four of the six doctoral students of the last decade currently hold faculty appointments, following the trend of many former students supported under the DOE grant. Currently, four former doctoral students have grants in the BES Mechanical Behavior and Radiation Effects program (Pharr, Mills, Hemker and Greer).

Doctoral students of the past decade:

30. Julia Rosolovsky Greer (Russia/USA) 2005
“Size Effects in Mechanical Properties at the Sub-Micron Scale”

Title: Professor
Organization: California Institute of Technology
Location: Pasadena, CA

31. Seung Min J. Han (Korea/Canada) 2006
“Methodologies in Determining Mechanical Properties of Thin Films using Nanoindentation”

Title: Associate Professor
Organization: Korea Advanced Institute of Science and Technology
Location: Daejeon, Korea

32. Arief S. Budiman (Indonesia) 2007
"Probing Plasticity at Small Scales: From Electromigration in Interconnects to Dislocation Hardening Processes in Crystals"

Title:	Assistant Professor
Organization:	Engineering Product Development
Location:	Singapore University of Technology and Design, Singapore

33. Seok-Woo Lee (Korea) 2011
"The plasticity of metals at the sub-micron scale and dislocation dynamics in a thin film"

Title:	Pratt & Whitney Assistant Professor
Organization:	Department of Materials Science and Engineering
Location:	University of Connecticut, Storrs, CT

34. **Ill Ryu** (Korea) 2013
"Size Dependent Fracture and Plasticity in Nanorods"

Title:	Post-Doctoral Scholar
Organization:	School of Engineering
Location:	Brown University, Providence, RI

35. **Lucas Alexander Berla** (USA) 2014
"Understanding the Deformation Behavior of Lithiated Silicon and Related Advances in Nanoindentation"

Title:	Research Engineer
Organization:	Exponent Inc.
Location:	Menlo Park, CA

VI. Abstracts of recent Ph.D. dissertations relating to the current and previous grants (past 10 years)

One way to provide a more detailed, yet still brief, account of the accomplishments of the past decade is to reprint the abstracts of the Ph.D. dissertations of the six students who completed their work in that time period. The abstracts below describe all of the main contributions made during that time period. These contributions are described in even more detail in the dissertations and published papers themselves.

Julia Rosolovsky Greer (2005)

"Size Effects in Mechanical Properties at the Sub-Micron Scale"

While "super-sizing" seems to be the driving force of our food industry, the direction of materials research has been quite the opposite: the dimensions of many technological devices are becoming ever smaller each year. This continuous reduction in size drives a great demand for understanding the mechanical behaviour, and in particular, the strength of materials at the sub-micron scale. In bulk form, the yield stress and strength of the material remain nearly constant regardless of the sample size because the sample dimensions are large compared to the length scale characterizing the material's microstructure. However, when the geometries of critical dimensions on a device approach the size of material's microstructure, the size effects prevail, and the bulk properties can no longer be used to predict mechanical behaviour.

Pure metals, as well as some alloys, have been found to exhibit strong size effects at the sub-micron scale: smaller samples consistently yield at higher stress levels. In many earlier experimental studies, the size effects in indentation, torsion and bending were understood in terms of plastic strain gradients that create geometrically-necessary dislocations leading to hardening. Even without the presence of strong strain gradients, the strengths of thin films on substrates are typically found to scale inversely with film thickness. The higher strengths observed in thin metal films relative to their bulk counterparts are usually attributed to the confinement of dislocations within the film by the substrate and in some cases, the passivation, as well. While these studies of thin films constitute important size effects on plasticity, they all arise from the constraining effects of surrounding layers. Another set of size effects has been observed for crystals that are initially dislocation-free. Classic experiments in the 1950's showed that initially pristine metal whiskers yielded at nearly theoretical strengths. In addition, in the earliest stages of nanoindentation, the crystal volume being probed is extremely small and can be dislocation-free. Therefore, in the initial stages of deformation very large indentation stresses are needed to nucleate new dislocations, which leads to a size effect for plastic flow. Finally, several molecular dynamics simulations also agree with the tenet that smaller is stronger. However, in spite of much progress on size effects for plasticity there is still no unified theory for plastic deformation at the sub-micron scale.

In the work presented here, the attention is focused on size effects that arise in unconstrained geometries, in the absence of strong strain gradients, and with non-zero initial dislocation densities characteristic of well-annealed crystals. In this work, gold nanopillars ranging in diameter between 200 nm and several microns were fabricated using Focused Ion beam (FIB) machining with Ga+ ions and microlithography followed by electroplating. These small pillars were found to plastically deform in uniaxial compression at stresses as high as 800 MPa, a value ~50 times higher than for bulk gold. We believe that these high strengths are controlled by the process of hardening by dislocation starvation, unique to very small crystals. In this mechanism, the mobile dislocations have a higher probability of annihilating at a nearby free surface than being pinned by other dislocations. When the starvation conditions are met, plasticity is accommodated by the nucleation and motion of new dislocations rather than by motion and interaction of existing dislocations, as is the case for bulk crystals.

Seung Min J. Han (2006)
"Methodologies in Determining Mechanical Properties of Thin Films using Nanoindentation"

Thin films are critical components of microelectronic and MEMS devices, and evaluating their mechanical properties is of current interest. As the dimensions of the devices become smaller and smaller, however, understanding the mechanical properties of materials at sub-micron length scales becomes more challenging. The conventional methods for evaluating strengths of materials in bulk form cannot be applied, and new methodologies are required for accurately evaluating mechanical properties of thin films. In this work, development of methodologies using the nanoindenter was pursued in three parts: I) creation of a new method for extracting thin film hardness, 2) use of combinatorial methods for determining compositions with desired mechanical properties, and 3) use of microcompression testing of sub-micron sized pillars to understand plasticity in Al-Sc multilayers.

One of the established methods for evaluating mechanical properties in small-scale volumes is nanoindentation, a depth sensing indentation technique wherein the load and contact stiffiess are measured continuously throughout the depth of indentation with sub-nanometer displacement resolution. With its ability to accurately probe mechanical properties in very small volumes, the nanoindenter is therefore suitable for probing desired properties of thin films and other small scale structures. However, the existing nanoindentation hardness model by Oliver & Pharr is unable to accurately determine the hardness of thin films on substrates with an elastic mismatch. Thus, a new method of analysis for extracting thin film hardness from film/substrate systems, that eliminates the effect of elastic mismatch of the underlying substrate, surface roughness, and also pile-up/sink-in, is needed. Such a method was developed in the first part of this study.

The feasibility of using the nanoindentation hardness, utilizing the new method of analysis, together with combinatorial methods to efficiently scan through mechanical properties of Ti-AI metallic alloys was examined in the second part of this study. The combinatorial approach provides an efficient method that can be used to determine alloy compositions that might merit further exploration and development as bulk materials.

Finally, the mechanical properties of Al-$A1_3Sc$ multilayers with bilayer periods ranging from 6-100 nm using both nanoindentation and microcompression were examined. A previous study of these multilayers by Phillips (2003) revealed extremely high strengths for these fine bilayer thickness samples as measured by nanoindentation hardness. In this study, we prepared submicron sized pillars using the focused ion beam (FIB) and performed compression testing with the flat tip of the nanoindenter. The measured yield strengths show the trend of increasing strength with decreasing bilayer period, and agree with the nanoindentation hardness results using the suitable Tabor correction factor. The deformation of the Al-$A1_3Sc$ pillars at large strains showed strain softening that causes inhomogeneous deformation. A new model was developed to account for the inhomogeneous geometry to calculate the stress and strain in this regime of strain softening.

Arief S. Budiman (2007)

"Probing Plasticity at Small Scales: From Electromigration in Interconnects to Dislocation Hardening Processes in Crystals"

Investigations into the role of plasticity in the mechanical behavior of materials have great importance to the field of materials science, especially in today's nano-age where sub-micron and nanoscale devices are built near the size of their microstmctural features. The creation of such small components requires a thorough understanding of the mechanical properties of materials at these small length scales. A synchrotron white-beam diffraction technique utilizing focused X-ray beam into the submicron resolution has proved to be a unique, powerful tool for the study of plasticity due to its sensitivity to local lattice rotation. This capability becomes crucial when the plastic mechanisms of crystalline materials at submicron and nanoscales are increasingly known to deviate from their bulk (classical) mechanisms leading to their unexpected mechanical behaviors. Understanding and controlling plasticity and the mechanical properties of materials on this scale could thus lead to new and more robust nanomechanical structures and devices.

This Scanning X-Ray Submicron Diffraction (ILSXRD) technique developed in the Beamline 7.3.3 at the Advanced Light Source (ALS), Berkeley Lab has been used to study the microstructural evolution at granular level of Cu polycrystalline lines during electromigration. An unexpected mode of plastic deformation was observed in damascene Cu interconnect test structures during an in situ electromigration (EM) experiment and before the onset of visible microstructural damages (void, hillock formation). The deformation geometry and the extent of plasticity observed in this study lead us to conclude that this mode of deformation could have direct bearing on the final failure stages of electromigration.

When crystalline materials are mechanically deformed in small volumes, higher stresses are needed for plastic flow. This has been called the "Smaller is Stronger" phenomenon and has been widely observed. Various size-dependent strengthening mechanisms have been proposed to account for such effects, often involving strain gradients and geometrically necessary dislocations (GNDs). Since the GND density is directly related to local lattice rotation, the USXRD technique provides the key tool to probe the plastic behavior of the materials at small scales. Here we report on the search for strain gradients and geometrically necessary dislocations as a possible source of strength for two cases of deformation of materials at small scales: nanoindented single crystal copper and uniaxially compressed single crystal submicron gold pillars. These observations suggest that plasticity in one case is indeed controlled by the GNDs (strain gradient hardening), whereas in the other, plasticity is not controlled by strain gradients or sub-structure hardening, but rather by dislocation source starvation, wherein smaller volumes are stronger because fewer sources of dislocations are available (dislocation starvation hardening).

Seok-Woo Lee (2011)
"The plasticity of metals at the sub-micron scale and dislocation dynamics in a thin film"

Nanotechnology has played a significant role in the development of useful engineering devices and in the synthesis of new classes of materials. For the reliable design of devices and for structural applications of materials with micro- or nano-sized features, nanotechnology has always called for an understanding of the mechanical properties of materials at small length scales. Thus, it becomes important to develop new experimental techniques to allow reliable mechanical testing at small scales. At the same time, the development of computational techniques is necessary to interpret the experimentally observed phenomena. Currently, microcompression testing of micropillars, which are fabricated by focused-ion beam (FIB) milling, is one of the most popular experimental methods for measuring the mechanical properties at the micrometer scale. Also, dislocation dynamics codes have been extensively developed to study the local evolution of dislocation structures. Therefore, we conducted both experimental and theoretical studies that shed new light on the factors that control the strength and plasticity of crystalline materials at the sub-micrometer scale.

In the experimental work, we produced gold nanopillars by focused-ion beam milling, and conducted microcompression tests to obtain the stress-strain curves. Firstly, the size effects on the strength of gold nanopillars were studied, and "Smaller is Stronger" was observed. Secondly, we tried to change the dislocation densities to control the strength of gold nanopillars by prestraining and annealing. The results showed that prestraining dramatically reduces the flow strength of nanopillars while annealing restores the strength to the pristine levels. Transmission electron microscopy (TEM) revealed that the high dislocation density ($\sim10^{15}$ m^{-2}) of prestrained nanopillars significantly decreased after heavy plastic deformation. In order to interpret this TEM observation, potential dislocation source structures were geometrically analyzed. We found that the insertion of jogged dislocations before relaxation or enabling cross-slip during plastic flow are prerequisites for the formation of potentially strong natural pinning points and single arm dislocation sources. At the sub-micron scale, these conditions are most likely absent, and we argue that mobile dislocation starvation would occur naturally in the course of plastic flow.

Two more outstanding issues have also been studied in this dissertation. The first involves the effects of FIB milling on the mechanical properties. Since micropillars are made by FIB milling, the damage layer at the free surface is always formed and would be expected to affect the mechanical properties at a sub-micron scale. Thus, pristine gold microparticles were produced by a solid-state dewetting technique, and the effects of FIB milling on both pristine and prestrained microparticles were examined via microcompression testing. These experiments revealed that FIB milling significantly reduces the strength of pristine microparticles, but does not alter that of prestrained microparticles. Thus, we confirmed that if there are pre-existing mobile-dislocations present in the crystal, FIB milling does not affect the mechanical properties. The second issue is the scaling law commonly used to describe the strength of micropillars as a function of sample size. For the scaling law, the power-law approximation has been widely used without understanding fundamental physics in it. Thus, we tried to analyze the power-law approximation in a quantitative manner with the well-known single arm source model. Material parameters, such as the friction stress, the anisotropic shear modulus, the magnitude of Burgers vector and the dislocation density, were explored to understand their effects on the scaling behavior. Considering these effects allows one to rationalize the observed material-dependent power-law exponents quantitatively.

In another part of the dissertation, a computational study of dislocation dynamics in a free-standing thin film is described. We improved the ParaDiS (Parallel Dislocation Simulator) code, which was originally developed at the Lawrence Livermore National Laboratory, to deal with the free surface of a free-standing thin film. The spectral method was implemented to calculate the image stress field in a thin film. The faster convergence in the image stress calculation were obtained by employing Yoffe's image stress, which removes the singularity of the traction at the intersecting point between a threading dislocation and free surface. Using this newly developed code, we studied the stability of dislocation junctions and jogs, which are the potential dislocation sources, in a free standing thin film of a face-centered-cubic metal and discussed the creation of a dislocation source in a thin film.

In summary, we have performed both microcompression tests and dislocation dynamics simulations to understand the dislocation mechanisms at the sub-micron scale and the related mechanical properties of metals. We believe that these experimental and computational studies have contributed to the enhancement of our fundamental knowledge of the plasticity of metals at the sub-micron scale.

Ill Ryu (2013)
"Size Dependent Fracture and Plasticity in Nanorods"

As the technology of micro-scale devices evolves to smaller dimensions, the size dependence of fracture and plasticity at small scales attracts more and more attention. This is driven by the knowledge that many mechanical properties at the sub-micron scale differ from those at the macroscopic scale. For the reliable design of MEMS or NEMS devices, obtaining an understanding of the size-dependent mechanical properties of materials is necessary at small length scales.

In the first part of the dissertation, we explore size-dependent fracture of Si nano-pillar (NP) lithium ion battery anodes. Silicon (Si) nanostructures are attractive candidates for Li-ion battery electrodes because they provide both large specific charging capacity and less constraint on the volume changes that occur during Li insertion and extraction. Initially, lithiation induced swelling was modeled by considering the diffusion of lithium atoms through the Si host. However, recent experiments have shown that crystalline Si anodes expand highly anisotropically through the motion of a sharp phase boundary between the crystalline Si core and the lithiated amorphous Si shell. This phenomenon cannot be explained by a lithiation mechanism governed purely by diffusion. Here, we present a phenomenological model for the anisotropic phase boundary motion, which can be understood by the fact that lithiation in crystalline Si is controlled by the reaction kinetics occurring in the narrow regime near the phase boundary between the lithiated Si alloy and crystalline Si. In addition, we develop a microscopic model to describe the size-dependent fracture of crystalline Si NPs during lithiation. We derive a traction-separation law based on the plastic growth of voids, and this law is, in turn, used in a cohesive zone finite element model to describe fracture. The model allows for both the initiation of cracking and crack growth. The initial size and spacing of the nanovoids, assumed to be responsible for the fracture are chosen to conform to recent experiments which have shown the critical diameter of Si NPs to be ~300-400nm. The anisotropy of the expansion is taken into account, and this leads naturally to the observed anisotropy of fracture. It may be possible to use our computed fracture toughness to describe the failure of lithiated Si for other loading conditions and geometries, such as the failure of other Si nanostructures during Li-ion battery cycling.

In another part of the dissertation, size dependent plasticity in BCC metals is explored using dislocation dynamics (DD) simulations. We formulate a three-dimensional, DD model of dislocation plasticity in BCC micro-pillars and use it to study size effects and the effects of initial dislocation density and strain rate on strength. The model is based on the molecular dynamics (MD) simulations of Weinberger and Cai who discovered a surface-controlled cross-slip process leading to dislocation multiplication without the presence of artificial pinning points. We find a "smaller is stronger" size effect that can be explained by the competition between the multiplication rate and depletion rate from the surface of the mobile dislocations. Although DD simulations still require higher strain rates than those found in experiments, our DD simulations predict flow stress dependences on pillar size, initial dislocation density and strain rate that are largely consistent with experiments. An analytical model is constructed to rationalize the behavior of the DD model at high strain rates.

In summary, we have researched the size-dependence of fracture of Si anodes in a Li-ion battery at the sub-micron scale. Moreover, micro-pillar plasticity is analyzed using dislocation dynamics simulation to understand how dislocation behavior relates to mechanical response in BCC metals. We believe that our mechanical modeling has contributed to the enhancement of our fundamental understanding of the size-dependence of fracture and plasticity at the sub-micron scale.

Lucas Alexander Berla (2014)
"Understanding the Deformation Behavior of Lithiated Silicon and Related Advances in Nanoindentation"

In recent years, consumer electronic devices have become increasingly complex and powerful. Accompanying this trend has been an inevitable rise in power consumption of such devices. For rechargeable electronics, rapid energy consumption has required improvement in energy storage capacity of secondary batteries. A widely explored approach to enhancing capacity of lithium ion batteries is to replace the conventional graphitic anode with silicon, which offers much higher capacity for lithium incorporation. However, because silicon, upon lithium insertion, expands in volume significantly, large stresses develop in silicon anodes during lithiation and delithiation. Through various mechanical processes, these stresses lead to anode failure, which in turn gives rise to undesirable capacity fade of silicon anodes during electrochemical cycling. The aim of the herein-described research is to further the understanding of the deformation and fracture behavior of lithiated silicon, which is essential in the pursuit to overcome hurdles associated with using silicon anodes in lithium ion batteries.

In the first part of this work, silicon micropillar lithiation/delithiation studies were employed to assess the robustness of amorphous silicon, relative to crystalline silicon, to lithiation- and delithiation-induced fracture. Even the largest pillars showed no lithiation-induced interior or exterior cohesive fracture. Delithiation of fully lithiated pillars produced internal cohesive fracture initiated by delamination of the pillar/substrate interface at the base of the pillar sidewall. Finite element modeling, indicating concentrated triaxial tensile stresses that move inward and upward with progression of delithiation, provided explanation for the observed fracture evolution. The research findings demonstrate that amorphous silicon is quite robust to fracture during lithiation; the critical size for fracture of amorphous silicon particles upon lithiation is determined to exceed ~2.3 µm. Furthermore, the results reinforce the influence of stress concentrators on crack formation during electrochemical cycling of silicon.

During the pillar fracture research, it became evident that there was a need to better understand the time-independent and time-dependent deformation behavior of lithiated silicon. The second part of this work is focused on subsequent nanoindentation-based lithiated silicon deformation behavior studies. Prior to indenting lithiated silicon films, advances to general nanoindentation techniques were proposed. A new physically based function for nanoindentation indenter tip shape calibration was developed. The function, which accounts for the rounded shape at the indenter tip as well as the pyramidal shape away from the tip, fits calibration data well and returns physically meaningful calibration constants. The function can effectively replace the conventionally used empirical tip shape calibration function.

Next, modifications to the Agilent Technologies Nanoindenter XP stage were implemented in order to allow continuous stiffness measurement indentation of samples immersed in fluids while maintaining high-precision lateral placement of indents. These improvements made possible nanoindentation studies of blistered lithiated silicon films immersed in paraffin oil to slow sample oxidation. Lithium-silicon alloy films of various compositions were probed. Young's modulus and the hardness were found to decrease as lithium content increased. Indentation creep testing was executed on unlithiated, amorphous silicon and heavily lithiated silicon, and the results indicate that lithiated silicon creeps readily compared to unlithiated silicon. In all cases, the viscoplastic flow behavior is consistent with power law creep with a large stress exponent (>20). Interpreting the measured large stress exponents with a model for thermally activated, shear-driven local atomic rearrangement, the activation volume for the transformation is found to be comparable to the volume of a molecular unit of $Li_{15}Si_4$. This suggests that creep of lithiated silicon is controlled not by diffusional flow of individual atoms but rather by concerted rearrangement of small atomic clusters.

VII. Biographical Sketch of Professor William D. Nix

Professor Nix obtained his B.S. degree in Metallurgical Engineering from San Jose State College, and his M.S. and Ph.D. degrees in Metallurgical Engineering and Materials Science, respectively, from Stanford University. He joined the faculty at Stanford in 1963 and was appointed Professor in 1972. He was named the Lee Otterson Professor of Engineering at Stanford University in 1989 and served as Chairman of the Department of Materials Science and Engineering from 1991 to 1996. He became Professor Emeritus in 2003. In 2001 he was awarded an Honorary Doctor of Engineering Degree by the Colorado School of Mines and in 2007 an honorary degree of Doctor of Engineering by the University of Illinois. He received an honorary degree of Doctor of Science from Northwestern University in 2012.

In 1964 Professor Nix received the Western Electric Fund Award for Excellence in Engineering Instruction, and in 1970, the Bradley Stoughton Teaching Award of ASM. He received the 1979 Champion Herbert Mathewson Award and in 1988 was the Institute of Metals Lecturer and recipient of the Robert Franklin Mehl Award of the Metallurgical Society (TMS). In 1995 he received the Educator Award from TMS. He was selected by ASM International to give the 1989 Edward DeMille Campbell Memorial Lecture and in 1998 received the ASM Gold Medal. He gave the Alpha Sigma Mu Lecture to ASM in 2000 and received the Albert Easton White Distinguished Teacher Award in 2002 and the Albert Sauveur Achievement Award in 2003, both from ASM. He also received a Distinguished Alumnus Award from San Jose State University in 1980. In 1993 he received the Acta Metallurgica Gold Medal and in 2001 he received the Nadai Medal from the American Society of Mechanical Engineers. He was elected Fellow of the American Society for Metals in 1978, Fellow of the Metallurgical Society of AIME in 1988 and Fellow of the Materials Research Society in 2011. He received the von Hippel Award from the Materials Research Society in 2007 and in 2011 was awarded the Heyn Medal of the German Society of Materials Science. In 1987 he was elected to the National Academy of Engineering and in 2002 was elected as a Fellow of the American Academy of Arts and Sciences. Prof. Nix was elected to the National Academy of Sciences in 2003.

In 1966 he participated in Ford Foundation's "Residence in Engineering Practice " program as Assistant to the Director of Technology at the Stellite Division of Union Carbide Corporation. From 1968 to 1970 Professor Nix was Director of Stanford's Center for Materials Research. Professor Nix is engaged in research on the mechanical properties of solids. He is principally concerned with the relation between structure and mechanical properties of materials in both thin film and bulk form and is also engaged in research on the mechanical properties of materials for lithium-ion batteries. He is co-author of 450 publications in these and related fields and he has trained 79 Ph.D. students in these subjects in his years at Stanford. Professor Nix teaches courses on dislocation theory and mechanical properties of materials. He is co-author of "The Principles of Engineering Materials", published in 1973 by Prentice-Hall, Incorporated.