DETERMINATION OF MECHANICAL PROPERTIES BY INDENTATION IN NANOSCALE METALLIC WIRES USING ATOMIC FORCE MICROSCOPY

Evan Malina – URECA 2009 Proposal

Background and Justification

Nanotechnology is a rapidly emerging set of technologies based upon nano-scale atomic devices and material phenomena [1]. Nanomaterials, such as nanoscale wires, are created with diameters as small as tens of nanometers, which is only a few hundred atoms across. Over the past five years, the effect of sample size on mechanical properties in metals has been an active area of research [2-5]. This all resulted from the guarantee that scaling laws established on metals were no longer valid on the nanoscale and, therefore there are new mechanisms and unknown properties that have yet to be discovered in this class of materials.

Nanomaterials are useful because of the extraordinary properties they possess. They exhibit ultra-high strength and hardness, and can be strongly ductile at low temperatures [2]. These properties could lead to many important technological advances, but as the materials have become smaller they have become increasingly difficult to work with and study. Present knowledge of material properties at these nanoscale sample sizes are based solely upon numerical methods and theory without any experimental confirmation. Literature on atomistic simulations of metal nanowires [6-10] with a diameter below 50 nm predicts a combination of ultra-high strength and enhanced ductility simultaneously. However, research attempts made to address this question experimentally have remained elusive for nanowires below 200 nm in diameter.

With recent research advances and acquisition of new instruments, the University of Vermont is in a fairly unique position to explore the effects of size on material properties at the nanoscale. The first hurdle in exploring these nanomaterials was to create them; however this was accomplished by Burchman [11] during his honors thesis at UVM in 2007-08. These wires were created using the synthesis of single crystal Ni nanowires by electrochemical deposition and these wires have diameters varying between 20 and 200 nm(Figure 1a). Atomistic simulations of crystal plasticity by nanoindentation of Ni nanowires have also recently been performed by Dupont and Sansoz [12]. With the new instruments available, these results could be experimentally confirmed; a world first. The possibilities available through the development of new nanomaterials are unprecedented and significant. This research is a step to further understand these materials and to explore their incredible possibilities.

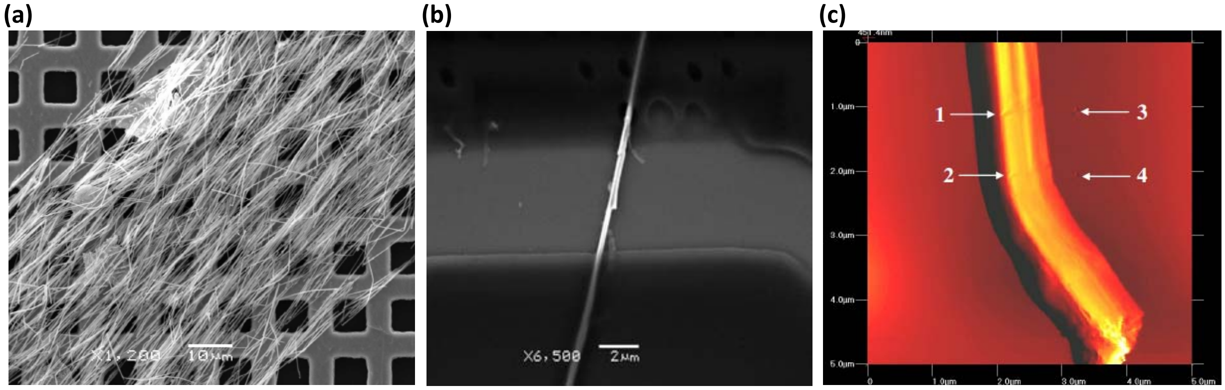

Figure 1. Nanoscale Ni wires grown by electrodeposition in the Sansoz group [11]. (a) A bundle of 200-nm-diameter Ni nanowires on a Cu grid for scanning electron microscopy (SEM) characterization. (b) A nanowire clamped by electron beam lithography for atomic force microscope (AFM) characterization. (c) Four nanoindentations on a Ni nanowire performed by a diamond-tipped AFM cantilever.

Hypothesis

- The hardness of metallic nanowires can be determined using nanoscale indentation techniques
- Nanoscale indentation can be performed by using an atomic force microscope with custom probes

Objectives

This URECA award will enable me to achieve the following objectives in this research:

- To measure the hardness of a Ni nanowire by using a diamond tipped atomic force microscope (AFM) cantilever.
- To experimentally determine the relationship between wire diameter and hardness in single-crystal Ni nanowires of diameter 20 nm to 200 nm.

Procedure and Methods

Background on atomic force microscopy. AFM technique traditionally is used to create three dimensional images of surface areas on the nanoscale. These topographic images are created by moving a sample back and forth under a diamond probe made of a sharp pyramidal tip at the end of a Si cantilever beam. The sample is moved very precisely by a piezoelectric cylinder that expands and contract given different voltages, thus scanning a programmed area (Figure 2a). As the piezoelectric cylinder moves the sample, a Si cantilever deflects as it encounters ridges. A laser beam is pointed at the top of a cantilever that reflects into a photo-sensor, which reads the deflection precisely. The force applied by the cantilever can also be calculated simply from the deflection because its mechanical response is known to be elastic.

For nanoscale imaging, an AFM has a tapping mode where the probe hits the surface of a sample at an intermittent, low-force as not to damage the specimen. After creating a 3-D image of the area, the probe can be programmed to perform nanoindentations at specific locations on the sample with larger forces. Material properties can then be determined by measuring the different indentation areas with the corresponding, known force applied.

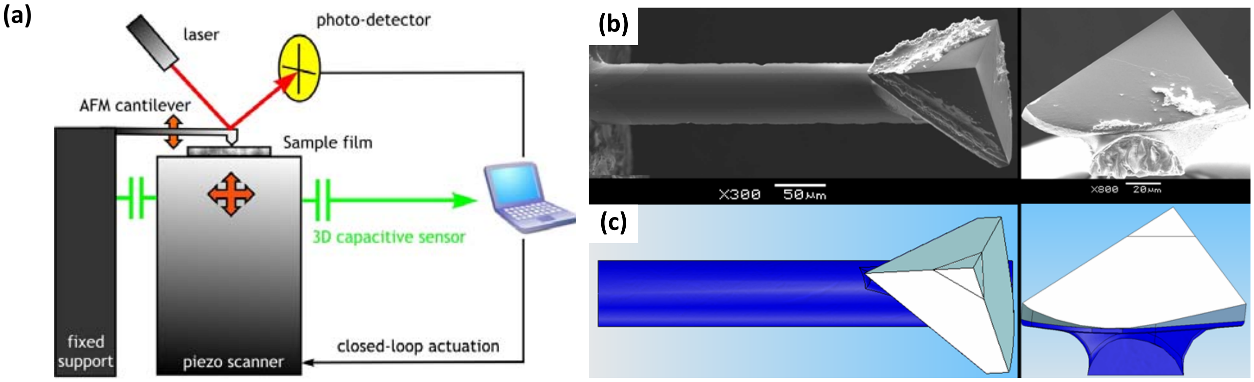

Figure 2: (a) Principle of atomic force microscope cantilever for nanomechanical analysis of materials. AFM cantilever images by (b) scanning electron microscopy, and (c) SolidWorks© 3D modeling.

Stage 1 – AFM-enabled Nanoindentation Testing. It is difficult to indent a wire since it is hard to guide the probe into the nanowire. Figure 1b shows a nanowire clamped on both sides by deposition of a poly(methyl methacrylate) (PMMA) film using electron beam lithography onto a flat Si wafer. This nanowire is approximately 200 nm, but the new nanowires that are being created are on the scale of 20 to 200 nanometers.

Since nanowires are currently being created, and the process to clamp the nanowires to silicon is already being implemented in the advisor's laboratory, **my research will primarily focus on the implementation of the indentation technique for nanowires**. This will be accomplished by first imaging the area with the AFM. A new

AFM probe with a three-sided, diamond, pyramidal tip and sapphire cantilever (Figure 2b) will be purchased (see budget below) and custom made by Micro Star Technologies. With the 3-D image, the probe can be programmed to indent the nanowire at a set velocity and position with the piezoelectric cylinder. The force can be calibrated by first applying the force next to the wire on the Si wafer as seen in Figure 1c [11]. The polished silicon wafers are purchased, and their properties are known, making for a simple calculation to find the force through the size of the indentation. The size of the indentations left by the tip will be measured by the tapping-mode on an AFM at the relevant wire section. The indentation area will be similar to that of a triangle; and with the given scale on the image – the area will be calculated by image analysis software. A Matlab® program will be developed to further analyze the data given the position and velocity of the sample for the force, and the indentation areas of both the silicon wafer and the nanowire. This program will be adapted and enhanced from the preliminary work by Burchman [11].

Stage 2 – Modeling and Test Analysis. The deflection of the cantilever and probe by the nanowire will be modeled using ABAQUS® a Finite Element Analysis (FEA) program. The probe will be modeled from measurements with the SEM using SolidWorks (See for example Figure 2c). This modeling will help refine the calibration of indentation tests and improve the accuracy of the experimental results. The cantilever elasticity (i.e., force constant) will be determined during this iteration as well.

Stage 3 – Measuring Size and Crystal Orientation Effects. AFM-enabled indentations will be performed on [111]-oriented Ni nanowires whose diameter will vary from 20 nm up to 200 nm. *A priori*, the crystal orientation of the wires with respect to the probe will be unknown except for the wire axis (i.e., [111]). Therefore, a series of tests will be performed on multiple nanowires of same diameter in order to determine the effect of crystal orientation on hardness. This data will be compared directly to the results of atomistic simulations currently performed by a graduate student in the Sansoz Group [12].

Interpretation of Results

After measuring the hardness for a series of Ni nanowires, a relationship between diameter and hardness will be established and compared to atomistic simulations performed by others. It is believed that hardness will approach an asymptote as a function of penetration depth. If the hardness of the nanowire does indeed follow this curve – these results will be very valuable to advances in nanotechnology. The hardness along the asymptote will be the defined hardness for that given diameter and these values will be the hardness' plotted on the curve. In diameters of centimeter or greater scale, the hardness increases as diameter increases, but none of this has been determined on the nanoscale. As stated earlier, the behavior of nanoscale wires has yet to be experimentally confirmed, and these findings would help the nanotechnology industry tremendously by allowing them to maximize the strength and produce the longest lasting nanoscale product, it may even allow for the exploration of new nanomaterial uses.

References

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BUDGET PROPOSAL

Item	Cost
One diamond-tipped AFM probe (Micro Star Technology)	$1000
One 1-year license for Finite Element Analysis Software ABAQUS	$2000
TOTAL	**$3000**

BUDGET JUSTIFICATION

The budget includes the fabrication of a diamond-tipped AFM cantilever by Micro Star Technologies in Texas. The probe will be valuable because its geometry must be controlled precisely. The cantilever must be oriented at a 12-degree angle down with respect to the horizontal in order to apply the force perfectly normal to the sample and the diamond tip geometry must be a cube corner, as used in past studies.

The budget also includes a one-year research license from ABAQUS for the use of their finite element analysis package. This FEA is necessary to calibrate the force and penetrations measured by the AFM cantilever.

All other equipments, software (SolidWorks) and high-resolution microscopes including SEM and AFM are already available in the advisor’s laboratory and the School of Engineering.

EVAN MALINA

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Education:

University of Vermont Expected Graduation: May 2010
Candidate for Bachelor of Science in Mechanical Engineering Burlington, VT

- Minors in Mathematics and Business Administration
- **Cumulative GPA: 3.89**
- Member of Tau Beta Pi Engineering Honor Society
- Vice-Chair of ASME at UVM
- Dean's List (Each Semester)
- STudent Academic Recognition (STAR) 2007 (Top 5%)

Essex High School September 2002 – June 2006
National Honor Society Essex Junction, VT

Work Experience:

UVM Physical Plant May 2008 – present
Engineering Intern Burlington, VT

- Acquired knowledge in HVAC and energy management

Dudley H. Davis Student Center August 2007 – May 2008
Production Assistant Burlington, VT

- Developed time management with working and studying

Four Seasons Garden Center Summer 2007
Perennials Assistant Williston, VT

- Customer Relations and maintenance and sales of perennials

Community Service:

First Congregational Church of Essex

Weeklong volunteer projects as member of the church high school youth group:

Hurricane Katrina Relief in New Orleans, LA	April 2006
Back Bay Mission in Biloxi, MS	April 2004
Heifer Project International in Rutland, MA	June 2003

Skills & Relevant Courses;

- Excellent interpersonal skills, exceptional math ability, knowledge in the computer programs: Matlab, SolidWorks, and COSMOSWorks.
- Statics, Mechanics of Materials, Dynamics, and Materials Science.

BIOGRAPHICAL SKETCH - Frederic Sansoz

Degree	**Field**	**Institution**
Ph.D., 2000	Materials Science and Engineering	Ecole des Mines de Paris
M.S., 1996	Materials Science and Engineering	ENSMA, Poitiers, France
Engineer, 1996	Mechanical and Aerospace Engng.	ENSMA, Poitiers, France

Appointments:

2004-present	Assistant Professor, School of Engineering, University of Vermont
2002-2003	Postdoctoral Fellow, Dept. of Mechanical Engineering, Johns Hopkins University
2001-2002	Postdoctoral Fellow, Dept. of Mechanical Engineering, Univ. of Rhode Island
2000-2001	Overseas Research Fellow, SNECMA/French Ministry of Foreign Affairs

Honors and Awards

2008	National Science Foundation (NSF) Faculty CAREER award
2005-07	Three times nominee for Best Poster Award, Materials Research Society Meetings
2005	2nd Prize, "Science as Art" competition, Materials Research Society Spring Meeting

Selected Publications (over a total of 38 publications)

1. V. Dupont and F. Sansoz, "Quasicontinuum Study of Incipient Plasticity under Nanoscale Contact in Nanocrystalline Aluminum", *Acta Materialia*, 56, 6013-6026 (2008).
2. F. Sansoz, H. Huang and D.H. Warner, “An Atomistic Perspective on Twinning Phenomena in Nano-enhanced FCC Metals”, *JOM*, 9, 79-84 (2008).
3. V. Dupont and F. Sansoz, “Multiscale Modeling of Contact-induced Plasticity in Nanocrystalline Metals”, in *Trends in Computational Nanomechanics: Transcending Time and Space*, Ed. T. Dumitrica, Springer, (2009).
4. V. Dupont and F. Sansoz, “Molecular Dynamics Study of Crystal Plasticity during Nanoindentation in Ni Nanowires”, in *Focus Issue on Indentation Methods in Advanced Materials Research*, Journal of Materials Research, in press.
5. F. Bedoui, F. Sansoz and S. Murthy, "Incidence of Nanoscale Heterogeneity on the Nanoindentation of a Semicrystalline Polymer: Experiments and Modeling", Acta Materialia, 56, 10, 2296-2306 (2008).
6. K.A. Afanasyev and F. Sansoz, "Strengthening in Gold Nanopillars with Nanoscale Twins", Nano Letters, 7(7), 2056-2062 (2007).
7. F. Sansoz and V. Dupont, "Atomic Mechanism of Shear Localization during Indentation of a Nanostructured Metal", Materials Science and Engineering C, 27, 1509-1513 (2007).
8. F. Sansoz and V. Dupont, "Grain Growth Behavior at Absolute Zero during Nanocrystalline Metal Indentation", Applied Physics Letters, 89, 111901 (2006).
9. D. Warner, F. Sansoz, and J.F. Molinari, "An Atomistic-Based Continuum Investigation of Plastic Deformation in Nanocrystalline Copper", Int. J. Plasticity, 22, 754-774 (2006).
10. F. Sansoz and J.F. Molinari, "Mechanical Behavior of Sigma Tilt Grain Boundaries in Nanoscale Cu and Al: a Quasicontinuum Study", Acta Materialia, 53, 7, 1931-1944 (2005).

Membership to Scientific and Professional Societies: Materials Research Society (MRS); The Minerals, Metals and Materials Society (TMS); American Society of Mechanical Engineers (ASME)

Reviews for Funding Agencies: ACS-Pretroleum Research Fund, Air Force Office Scientific Research, National Science Foundation, and Vermont EPSCoR

Reviews for Refereed Journals: ACS Nano, Acta Materialia, ASME Journal of Tribology, Computational Materials Science, Experimental Mechanics, Journal of Alloys and Compounds, Journal of Applied Physics, Journal of the Electrochemical Society, Journal of Materials Research, Journal of Materials Science, JOM, Materials Science and Engineering A, Multiscale Modeling in Materials and Structures, Physical Review B, Surface and Coatings Technology, Tribology Transactions