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13. SUPPLEMENTARY NOTES Extensive progress was made in the development of the experimental regimen to measure the lattice rotation associated with indentation into single crystals and bicrystals of various face-centered cubic metals of interest to the Air Force.					
14. ABSTRACT The experimental effort concentrated on wedge indentation under quasistatic conditions in single crystals of Ni, Cu, and Al as well as bicrystals of Al. Wedge indenters with included angles of 60°, 90° and 120° were used in the single crystals and an included angle of 90° was used near the grain boundary of the Al bicrystal. The lattice rotations were experimentally determined via EBSD. The experimental results were processed to obtain all non-zero components of the Nye dislocation density tensor. An important advance was to derive the analytical expression for the Lower Bound on the geometrically necessary dislocation density based upon the measured Nye dislocation density tensor associated with the two-dimensional deformation state. Numerical computations using single crystal plasticity characterized the deformation state associated with the wedge indentation in the single crystal and the bicrystal. There was a strong qualitative agreement between the simulations and the experiments, but a theory that explicitly incorporates strain gradients in its formulation was shown to be necessary to achieve a quantitative agreement.					
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Final Performance Report

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Title of Project

Combined Experimental and Computational Study of Plastic Deformation in Crystals and Bicrystals for the Development of Multi-Length Scale Constitutive Models

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Principal Investigator

Jeffrey W. Kysar
Associate Professor
Department of Mechanical Engineering
244 Mudd Building, Mail Code 4703
Columbia University
500 W. 120th Street
New York, NY 10027

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Executive Summary

This document is the final report for grant FA9550-06-1-0214 from the AFOSR entitled "Combined Experimental and Computational Study of Plastic Deformation in Crystals and Bicrystals for the Development of Multi-Length Scale Constitutive Models." The experimental objectives of the work were all achieved, including the ability to unambiguously determine the rigorous lower bound of the density of geometrically necessary dislocations in the specimens, in both single crystals on nickel, copper and aluminum, as well as in bicrystals of aluminum. Computational models of the experiments were able to qualitatively describe the measured fields, but the models employed were not able to quantitatively predict the degree of lattice rotation observed in various specimens without modification of the constitutive parameters. Nevertheless, the experimental regimen developed in this project will serve as a benchmark for the development of constitutive relations. Two Ph.D students were funded by this study, both making important contributions in terms of the advancement of the experimental methods and data analysis. The PI received two significant awards during the course of this project. One is 2006 Presidential Early Career Award for Scientists and Engineers (PECASE) at the White House. The other is the 2006 Department of Energy (DOE) Early Career Scientist and Engineer Award through the DOE Office of Defense Programs.

Objectives of Research

As delineated in the proposal for this project, the objective of this work is to enhance the overall understanding of physics-based constitutive relationships for ductile metals under conditions of high strain and also high strain gradient under quasistatic loading conditions.

The main results of the study are expected to be a definitive set of experimental results which are amenable to direct comparison with detailed numerical simulation for the purpose of parameterization and validation of physics-based multi-scale constitutive models in the high strain gradient and finite strain regime. It is expected that a reasonable set of constitutive parameters will be obtained for one such multi-length scale constitutive model.

This will be accomplished in the following ways:

1. Induce two-dimensional plane strain heterogeneous deformation states in a variety of specially oriented single crystals and bicrystals of various metals.
2. Employ diffraction-based methods to measure lattice rotation which is introduced by loading in single crystals and in bicrystals. All the lattice rotation will occur in the plane of the two-dimensional deformation state.
3. Calculate all non-zero components of the lattice curvature tensor and the Nye dislocation density tensor from lattice rotation measurements.
4. Estimate density of geometrically necessary dislocations on each of the effective in-plane plastic slip systems.
5. Use the results from single crystal experiments to provide feedback for parameterization and validation of constitutive relations which account explicitly for the presence strain gradients and dislocation densities and the flux of dislocation densities on individual slip

system. Numerical implementation of the constitutive relations will be in collaboration with researchers at Lawrence Livermore National Laboratory (LLNL).

6. Experiments and simulations in single crystals will allow parameterization and validation of constitutive relations valid within each grain of a polycrystalline material.
7. Experiments and simulations in judiciously oriented bicrystals will allow incorporation of the grain boundary properties and grain sizes into the constitutive relations.

Status of Effort

The experimental effort concentrated on wedge indentation under quasistatic conditions in single crystals of Ni, Cu, and Al as well as bicrystals of Al. Wedge indenters with included angles of 60°, 90° and 120° were used in the single crystals and an included angle of 90° was used near the grain boundary of the Al bicrystal. The lattice rotations were experimentally determined via EBSD. The experimental results were processed to obtain all non-zero components of the Nye dislocation density tensor. An important advance was to derive the analytical expression for the Lower Bound on the geometrically necessary dislocation density based upon the measured Nye dislocation density tensor associated with the two-dimensional deformation state. Numerical computations using single crystal plasticity characterized the deformation state associated with the wedge indentation in the single crystal and the bicrystal. There was a strong qualitative agreement between the simulations and the experiments, but a theory that explicitly incorporates strain gradients in its formulation was shown to be necessary to achieve a quantitative agreement.

Accomplishments and New Findings

In most metals, the predominant mode of plastic (i.e. irreversible) deformation is through the generation and motion of dislocations in the crystalline lattice. Dislocations are line defects in a crystalline lattice that can be thought of as the line of termination of an extra half-plane of atoms in the material. Further, a dislocation typically exists on a particular plane associated with the crystal lattice, the dislocation can be considered as having a "positive" or a "negative" character depending upon the orientation of the extra half planes of atoms relative to the plane on which the dislocation exists.

If one considers a given volume of a crystal lattice, one expects to find dislocations of both positive and a negative character, but not necessarily in equal numbers; in general either the positive or the negative population will exist in greater number. Therefore, the overall density of dislocations is typically considered to consist of two subsets: the statistically stored dislocation (SSD) density and the geometrically necessary dislocation (GND) density. The SSD density has equal numbers of positive and negative dislocations. The GND density is the overall net density of signed dislocations, so that it characterizes the dislocations that remain after all the SSD dislocations of one sign have been matched up with one of the opposite sign.

Since a dislocation is fundamentally the line of termination of an extra half-plane of atoms, it is expected that a sufficiently high density of dislocations will induce a change of shape in the crystal lattice. It turns out, however, that the dislocations of one sign in the SSD density of dislocations tend to offset the effects of dislocations of the opposite sign so that no long-range effect is induced into the geometry of the crystal lattice by the SSD density. However since all

the dislocations assigned to the GND density are of the same sign, the GND density induces a change in the geometry of the crystal lattice—hence the name geometrical necessary dislocations density.

The experimental method used in this research is to measure the lattice rotation due to plastic deformation of the indented regions. The lattice rotation is then used to calculate the lattice curvature tensor. The lattice curvature tensor is related, in turn, to a second-rank non-symmetric tensor called the Nye dislocation density tensor. There then exists a relationship between the Nye dislocation density tensor and the GND density. However it is not generally possible to unambiguously determine the GND density from the Nye dislocation density tensor because there are typically more dislocation slip systems on which different types of dislocations can exist than there are components of the Nye dislocation density tensor. For the special two-dimensional deformation state introduced in the crystals and bicrystals in this research, there are two non-zero values of the Nye dislocation density tensor but there are three effective in-plane slip systems. Hence the linear system that relates Nye dislocation density tensor to GND densities has three unknowns but only two equations. Therefore the most that can be done is to *estimate* the GND density, as described in detail in the proposal.

A significant advance that has been accomplished as a result of this project is to derive the analytical expression for the *Lower Bound of the GND density* that is consistent with the measured lattice geometry. The lower bound is a fundamental quantity because the actual GND density as well as all possible estimates of the actual GND density can not be smaller than the lower bound.

Also, the analytic expressions for the lower bound on GND density become rigorous solutions for the GND density for those regions of the deformation field where only one or at most two dislocation slip systems are active. This is important because for the case of wedge indentation into a single crystal, analytical and numerical studies of the deformation state indicate that there are several regions within which only one or two effective in-plane slip systems are active. In those regions, then, it is possible to unambiguously determine the GND densities on each of the active slip systems.

Experimentally, the experiments to indent the single crystals have been very fruitful. The form of the lattice rotation field varies only slightly as a function of the included angle of the indenter. Furthermore, there is a strong discontinuity in lattice rotation that exists directly below the indenter tip. The magnitude of the lattice rotation discontinuity appears to have a maximum value that is a function of the material. There will be more emphasis on characterizing the maximum angle of lattice rotation discontinuity in future experiments. It is important to note that the numerical simulations which employ conventional crystal plasticity constitutive stress-strain relationships are able to predict the qualitative form of the lattice rotation field, however they are not able to accurately predict this angle of lattice rotation discontinuity due to wedge indentation by different included angles with the same set of constitutive parameters. This emphasizes the need for numerical simulations that directly account for the GND densities, since the lattice rotation discontinuity is induced by the presence of geometrically necessary dislocations.

The experiments on the bicrystal give direct evidence of the degree of transmission of plastic deformation across a grain boundary. Wedge indentation was performed in one grain of the bicrystal a distance of about 50 micrometers, and parallel, to the grain boundary. EBSD was then used to measure the lattice rotation that occurred in the indented grain as well as the degree of lattice rotation induced in the neighboring grain. Again conventional crystal plasticity numerical simulations are able to qualitatively predict the form of the lattice rotation field on both sides of the grain boundary; however they are not able to predict the magnitude of the lattice rotation in the neighboring grain. In fact, the conventional finite element simulations—which assume no displacement discontinuity across the grain boundary—severely over-predict the magnitude of the lattice rotation in neighboring grain, which indicates the interactions between the grain boundary and the dislocations that accompany plastic deformation are key to the accurate prediction of mechanical response in polycrystalline materials. This again demonstrates the necessity to employ numerical models that directly account for GND densities and strain gradients, as well as the need for accurate models of grain boundaries.

There were extensive interactions between the PI, the supported graduate student and researchers at Lawrence Livermore National Laboratory the development of models of the experiments. That work is still in progress and has not yet yielded any publishable results.

The PI has published three papers in which the plastic deformation around cylindrical voids in single crystals has been predicted analytically and numerically, as well as studied experimentally. The plasticity formulation in these papers assumed conventional crystal plasticity wherein the effects of strain gradients are ignored. In order to extend that work, the PI has written two papers with collaborators in Denmark to investigate the effect of strain gradients on the deformation and stress fields. One paper considered the effect of a single cylindrical void in a strain-gradient hardening material. It was found from the numerical simulations that inclusion of strain gradient terms in the evaluation dramatically changed the stress state as well as the deformation state. In essence, once the strain-gradient terms became dominant, the deformation state reduced to strain localizations that emanated from the void surfaces, rather than having a diffuse, less-heterogeneous deformation state, at least for the strain-gradient plasticity formulation chosen for the study. The other paper that resulted from the collaboration was a study of a porous single crystal that contained an infinite array of cylindrical voids. The study was motivated by the fracture process of void growth and coalescence that occurs in ductile metals. The results showed that a void in a single crystal can have significant interactions with its neighboring voids due to the fact that plastic deformation occurs by single slip within angular sectors surrounding the voids. The strain gradient terms served to significantly modulate the degree of plastic deformation that occurred in the region between neighboring voids.

In addition to the studies to characterize the plastic deformation due to indentation in single crystals and bicrystals, the PI and his research group have been involved with experiments to measure plastic properties of different material types at the nano length scale.

One such experiment developed in the PI's laboratory was to measure the mechanical properties of nanocrystalline thin films of free-standing copper via a bulge test. The films tested were on the order of 200 nm to 600 nm in thickness with an average grain size of 35 nm. The results demonstrated that the stress at which plastic deformation initiates is of the order of many

hundreds of MPa, much higher than analogous materials with a micrometer grain size. The results also showed strong evidence of grain boundary sliding as well as shear localizations that extended across grain boundaries. This line of research has the potential to give information as to the conditions under which there are transitions in deformation mechanisms from dislocation-mediated plasticity within individual grains to grain boundary sliding. Such experiments are important in order to be able to implement such transitions in deformation mechanisms in multiscale computer simulations.

The PI and research group also developed methods to characterize the mechanical deformation of nanoscale metal specimens that have a thickness of 100 nm, a width as small as 250 nm and a length of several micrometers. The specimen is suspended horizontally above a silicon substrate by two anchors at the ends of the specimen. Mechanical loading is applied by a nanoindenter to measure the resulting load and displacement history. Two different material systems have been tested with this method: nanoporous gold specimens and single crystal gold specimens. The nanoporous gold specimens have an open cellular structure with interconnecting voids of the order of 40 nm and interconnecting ligaments also have a length scale of about 40 nm. The elastic stiffness of such a material was shown to be about 10 GPa and the stresses in the ligaments at failure were shown to be of the order of 1.5 GPa. In addition to being of fundamental interest, this type of nanoporous material offers the potential to develop sensors and actuators due to the very high surface area to mass ratio as large as 8 m²/g. The single crystal gold nanoscale specimen experiments provide fundamental insight into the mechanical behavior of metals at that length scale. The results were particularly interesting because the yield stress was shown to be stochastic at the nanometer length scale; values of yield stress of 400 MPa are not uncommon. In addition, the force-displacement results had severe drops in load during the tests conducted at a prescribed displacement rate. Each load drop corresponds to an avalanche of dislocations. It did not prove possible to quantify the precise number of dislocations in each avalanche, nor was it possible to determine *a priori* the position of the resulting shear localization. Future experiments (funded by the National Science Foundation) are designed to allow the position of localization and to determine the number of dislocations in an avalanche. Such experiments will provide a critical baseline against which to compare dislocation dynamics simulations in a multiscale framework.

Another study which was partially funded with this grant was a set of experiments and simulations to determine the mechanical properties of monolayer graphene molecules were determined. The results included the second-order elastic modulus which determines the linear elastic behavior and an estimate of the third-order elastic modulus which determines the non-linear elastic behavior. In addition, the distribution of the breaking force strongly suggested the graphene to be free of defects, so the measured breaking strength of the films represented the intrinsic breaking strength of the underlying carbon covalent bonds. These experiments showed conclusively that graphene is the strongest material that has ever been measured. After appropriate normalization, the breaking strength of pristine (i.e. defect free) graphene was measured to be 130 GPa, which is some 100 times stronger than the best metal alloys. Since graphene consists entirely of carbon covalent bonding, it is not unreasonable to expect that it is indeed the strongest material that could exist. Thus, the measurements are fundamental in the sense that the experiments place an upper bound on the strength of any material.

The PI has also been involved with research into Laser Shock Peening (LSP) of materials. LSP is a technique to introduce a compressive residual stress state on a surface of a material for the enhancement of fatigue lifetime. The material is prepared by putting an ablative layer on the surface to be treated and the region to be treated is immersed in water. A high intensity ($\sim 4 \text{ GW/cm}^2$) laser incident on the ablative material is pulsed ($\sim 50 \text{ ns}$). The resulting interactions between the laser and the ablative material form a high-pressure plasma. The presence of the water serves to constrain the plasma against expansion so that a significant portion of the energy in the plasma is directed into the material. The process introduces plastic deformation into the surface that helps to enhance the fatigue lifetime. The PI and collaborators at Columbia University have been investigating LSP in which the incident laser flux is focused to a diameter of about 12 micrometers. At this length scale, the process has been denoted as micro-Laser Shock Peening, or μLSP . The process has been applied at Columbia to various materials, but the main material of interest to this project is to induce deformation in single crystals of aluminum and copper as well as near grain boundaries in bicrystals. Electron Backscatter Diffraction (EBSD) has been used to measure the crystal lattice rotation associated with the plastic deformation induced by μLSP . This provides a direct means of characterizing the degree of plastic deformation associated with the process that is of value for model validation of stress-strain constitutive relationships at high strain rates.

Personnel Supported

There have been two Ph.D. students supported by the project. One student, Yuki Saito, finished his Ph.D. at the end of August 2007. He had been supported by this project for over one year. A new graduate student, Muin Oztop, has been associated with the project since June 2007. He received a salary for the months of June, July and August of 2007 but was supported by a Teaching Assistantship during the Fall 2007 semester. As a consequence, it was not necessary to pay for his stipend or his tuition from this project during that semester. That freed up money from the project that went toward the purchase of a G-200 MTS nanoindenter, as approved by the program manager. He was then supported again during the Spring 2008 and Fall 2008 semesters until the project finished. During the summer months of 2008, Muin Oztop did research at Lawrence Livermore National Laboratory in collaboration with scientists on the development of constitutive models and experimental methods. In addition, the PI received summer salary from the project.

The Ph.D. thesis for Yuki Saito is publicly available in the library at Columbia University.

Publications

The following publications with acknowledgement to AFOSR for this project have been submitted, published or are under preparation since the project began. All the data, methods and conclusions are published in the various papers.

Lee D., Zhao M., Wei X., Chen X., Jun S. C., Hone, J., Herbert E. G., Oliver W. C. and Kysar J. W., (2006) "Observation of plastic deformation in free-standing single crystal Au nanowires, *Applied Physics Letters*, **89**, Art. No. 111916.

- Paper selected for inclusion in the Virtual Journal of Nanoscale Science and Technology.

Lee D., Wei X., Chen X., Zhao M., Jun S. C., Hone J., Herbert E. G., Oliver W. C., Kysar J. W., (2007) "Microfabrication and mechanical properties of nanoporous gold at the nanoscale", *Scripta Materialia*, **56**, 437-440.

Chen, H., Wang, Y., Kysar, J.W., and Yao, Y.L., (2007). "Study of anisotropic character induced by microscale laser shock peening on a single crystal aluminum." *Journal of Applied Physics*, **101**, 024904.

Lee D., Wei X., Zhao M., Chen X., Jun S. C., Hone, J. and Kysar J. W., (2007) "Plastic Deformation in Nanoscale Gold Single Crystals and Open-Celled Nanoporous Gold", *Modelling and Simulation in Materials Science and Engineering*, **15**, S181-S192.

- Paper featured on cover of journal issue
- Paper featured on advertising brochure for journal.

Kysar J. W., Gan Y. X., Morse T. L., Chen X., Jones, M. E., (2007) "High strain gradient plasticity associated with wedge indentation into face-centered cubic single crystals: Geometrically Necessary Dislocations", *Journal of the Mechanics and Physics of Solids*, **55**, 1554-1573.

Wang, Y., Chen, H., Kysar, J. W., Yao, Y. L. (2007) "Response of Thin Films and Substrate to Micro Laser Shock Peening", *Journal of Manufacturing Science and Engineering*, **129**, 485-496.

Borg U. and Kysar J. W., (2007) "Strain Gradient Crystal Plasticity Analysis of a Single Crystal Containing a Cylindrical Void", *International Journal of Solids and Structures*, **44**, 6382-6397.

Borg U., Niordson, C. F., and Kysar J. W., (2008) "Size Effects on Void Growth in Single Crystals with Distributed Voids", *International Journal of Plasticity*, **24**, 688-701.

C. Lee, X. D. Wei, J. W. Kysar, and J. Hone, (2008) "Measurement of the elastic properties and intrinsic strength of monolayer graphene. *Science*, **321**, 385-388.

Kysar, J. W. (2008) "Direct comparison between experiments and computations at the atomic length scale: a case study of graphene" *Scientific Modeling and Simulation*, **15**, 143-157.

Saito Y. and Kysar J. W. (2007) "Investigation of nickel crystals under wedge indentation with various included angles: Experiments and simulations", under preparation.

Saito Y. and Kysar J. W. (2007) "Deformation associated with wedge indentation into symmetric tilt aluminum bicrystal: Experiments and simulations", under preparation.

Saito Y. and Kysar J. W. (2007) "Analytical Solution of Wedge Indentation into Single Crystals under Plane Strain Conditions", under preparation.

Interactions and Transitions

The PI and other members of his research group presented the following papers at technical meetings, conferences and universities since the beginning of the project.

Invited Presentations

- Kysar, J. W., "Plastic Deformation in Nanoscale Materials", International Union of Theoretical and Applied Mechanics Symposium on Plasticity at the Micron Scale, Copenhagen, Denmark, May 21-25, 2006.
- Kysar, J. W., **Keynote Address:** "Analytical Solutions for Single Crystal Plasticity: Voids, 'Laser Shocks' and Indentations, Plasticity 2006, Halifax, Nova Scotia, July, 2006.
- Kysar, J. W., "Nanoporous Gold Thin Films: Fabrication, Properties, and Applications", Materials Research Science and Engineering Center Seminar, Columbia University, September, 2006.
- Kysar, J. W., "Measurement of Nye Dislocation Density Tensor and Geometrically Necessary Dislocation Density Based Upon Lattice Rotation Measurements", Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, MA, February, 2007.
- Kysar, J. W., "Measurement of Nye Dislocation Density Tensor and Geometrically Necessary Dislocation Density Based Upon Lattice Rotation Measurements", Graduate Aeronautical Laboratory (GALCIT) Colloquium, California Institute of Technology, Pasadena, CA, May, 2007.

Contributed Presentations

- Kysar J. W., Lee D., Chen X., "Mechanical Properties and Failure Mechanisms of Free-Standing Nanoporous Gold", Spring 2006 Materials Research Society Meeting, San Francisco, CA, April, 2006.
- Kysar, J. W., "Measurement of Nye Dislocation Density Tensor and Geometrically Necessary Dislocation Density Based upon Lattice Rotation Measurements, Plasticity 2006, Halifax, Nova Scotia, July, 2006
- Lee, D., Kysar, J. W., Chen, X., and Zhao, M., "Mechanical Properties of Gold Single Crystal Nanowires" 2006 ASME International Mechanical Engineering Congress, Chicago, IL, November, 2006.
- Kysar, J. W., and Lee, D., "Multifunctional Structures Based Upon Nanoporous Metals", 2006 ASME International Mechanical Engineering Congress, Chicago, IL, November, 2006.
- Wei, X., Lee, D., Chen, X. and Kysar, J. K., "Plane-strain bulge test for nanocrystalline copper nanoscale films" Fall 2006 Materials Research Society Meeting, Boston, MA, November, 2006.
- Wei, X., and Lee, D., and Chen, X. and Kysar, J. W., "Plane-strain Bulge Test for Nanocrystalline Copper Thin Films", 2007 Annual Conference for the Society of Experimental Mechanics, Springfield, MA, June 2007.

- Saito, Y. and Kysar, J. W., "Mechanics of Deformation Under Indentations in Single Crystals", 2007 Annual Conference for the Society of Experimental Mechanics, Springfield, MA, June 2007.
- Kysar, J. W., "Experimental Lower Bounds on Geometrically Necessary Dislocation Densities", 2007 McMat Mechanics and Materials Conference, Austin, TX, June 2007.

Popular Press

- PI was interviewed and quoted in article published on-line in *Scientific American* entitled "Fact or Fiction?: An Opera Singer's Piercing Voice Can Shatter Glass", by the author Karen Schrock. Article appeared on-line on August 23, 2007.
- Several dozen articles on the graphene experiments have appeared, including in MIT's Technology Review, the New York Post, Le Monde (Paris), as well as articles in many other newspapers and magazines. In addition, negotiations are underway for the PI to appear in a television program on advances in science.

Consultative and Advisory Functions

- PI served on a review panel for the Department of Energy for the evaluation of a university Predictive Science Academic Alliance Program at California Institute of Technology, from October 22 to 24, 2008.

New Discoveries, Inventions or Patent Disclosures

- Provisional Patent has been filed entitled "Force, pressure, or stiffness measurement or calibration using graphene or other sheet membrane"

Honors and Awards

- PI received the 2006 Presidential Early Career Award for Scientists and Engineers (PECASE) at a White House ceremony on November 1, 2007.
- PI received the 2006 Department of Energy (DOE) Early Career Scientist and Engineer Award through the DOE Office of Defense Programs.