Final Report for Period: 07/2006 - 12/2006
Submitted on: 02/23/2007
Principal Investigator: Patten, John A.
Award ID: 0403650
Organization: Western Michigan Univ
Title:
FRG: High Pressure Phase Transformations of Silicon, Germanium and Silicon Nitride

Project Participants

Senior Personnel

Name: Patten, John
Worked for more than 160 Hours: Yes
Contribution to Project:
John is the PI for this project and manages the overall operation and performance of the research program, including budgets, workshops, meetings, and coordination of research. John is involved in several aspects of the research program, including: scratching and machining experiments, modeling/simuation (FEA), material models, and analysis (ScAM, SEM, TEM, AFM, etc.).

Name: Miller, Jimmie
Worked for more than 160 Hours: Yes
Contribution to Project:
Dr. Miller, Chief Precision Engineer at UNCCharlotte, coordinates the work of graduate students at UNCC in cooperation with Dr. Patten (PI). Specifically, Dr. Millers assist with the following project tasks: Scratching and machining tests, purchasing and budgets, analytical instrumentation (AFM, SEM), coordinate machine shop (DTM) and metrology lab usage.

Name: Scattergood, Ronald
Worked for more than 160 Hours: Yes
Contribution to Project:
Ron is a co-PI on the project is primarily responsible for conducting scribing and machining (DTM) experiments at NCSU, and development of process models. Ron also oversees work on TEM and SEM analysis at NCSU.

Name: Nemanich, Robert
Worked for more than 160 Hours: Yes
Contribution to Project:
Bob is a co-PI on the project and is primarily responsible for conducting the Raman and micron Raman experimental analysis program at NCSU.

Name: Pharr, George
Worked for more than 160 Hours: Yes
Contribution to Project:
George is a Co-PI on the project and is primarily responsible for work related to nanoindentation, material models, and process modeling of indentation. George also oversees work on TEM and SEM analysis at UT-K.

Post-doc

Name: Jang, Jai-il
Worked for more than 160 Hours: Yes
Contribution to Project:
Jae-il is working with Prof. Pharr at UT-K. Jae-il's contribution to the project during the first two years includes nanoindentation experiments and analytical evaluation of semiconductors (silicon) and ceramics (silicon nitride and silicon carbide). Jae-il has created the baseline data for all experiments related to the study of the high pressure phase transformation of these materials. Jai-il works closely with Jennifer (NCSU) on the analyses (post process) of the materials being evaluated. His contribution includes the design and coordination (with ORNL-APS) of the in-situ nanoindentation and x-ray diffraction analysis of the high pressure phases of semiconductors and ceramics. This experimental program is quite ambitious as it attempts to combine the analytical evaluation (x-ray) with the experimental program (nanoindentation) using in-situ observation of the high pressure phase of these materials. He is also coordinating SEM and TEM analysis for the entire research group. Jae-il is being supported (salary) from this research grant.

Graduate Student

Name: Dong, Lei

Worked for more than 160 Hours: Yes

Contribution to Project:

Lei Dong is a PhD student. Her work involves detection of the high pressure metallic phase of silicon in-situ during mechanical deformation. She has also assisted with some preliminary electrical and optical detection/heating experiments to measure and manipulate the metallic high pressure phase of silicon. Lei is also invovled in the machining experiments and finite element modeling of the scractching process.

Name: Songqing, Wen

Worked for more than 160 Hours: Yes

Contribution to Project:

Songqing is a second year PhD student working in Prof. Pharr's laboratory at UT-K. Songqing has applied, and been approved to work within the ORNL facilities to conduct these experiments. Songqing is supported (stipend and tuition) directly from this research grant. Songqing is developing methodologies for fabrication of TEM samples using the FIB facilities at ORNL.

Name: Randall, Travis

Worked for more than 160 Hours: Yes

Contribution to Project:

Travis is a graduate student working with Prof. Scattergood at NCSU. He is conducting the scribe-bend testing experimental program and analytical evaluation. These tests are aimed at determining the deformation characteristics of semiconductors and ceramics, such as the ductile and brittle response, and subsequent residual stress analysis. He works closely with his counterparts from NCSU (Jennifer) and UNCC (Lei) to coordinate their testing and evaluation programs. Travis has also initiated the single point diamond turning (SPDT) experimental program using a diamond turning machinine (DTM).

Name: Huening, Jennifier

Worked for more than 160 Hours: Yes

Contribution to Project:

Jennifer graduated in 2004 with a Masters degree working with Prof. Nemanich at NCSU. Jennifer performs two major functions relative to this research program. First she conducts the micro-Raman analyses for all of the experimental testing programs, and secondly she coordinates the web site construction and operation. She works with a web

Name: Ajjarapu, Satya

Worked for more than 160 Hours: Yes

Contribution to Project:

Satya graduated in 2004 with a Masters degree working with Prof. Patten. Satya's work involves machining of ceramics and analysis of the machining experiments, including SEM, AFM, and surface profilometry. He also performs most of the machining simulations (FEA) of the experiments. These FEA experiments help us to understand the details of the material deformation at high pressures. Satya's experiments are partially supported by this grant. His stipend and tuition is supported by other grants and contracts. Satya works closely with collaborators at NCSU (Jennifer) ORNL-HTML (Sam McSpadden) to coordinate work related to sample preparation and post process analysis of machining experiments.

Name: Fesperman, Ronnie

Worked for more than 160 Hours: Yes

Contribution to Project:

Ronnie graduated in 2005 as a Masters student working with Prof. Patten. He works with Satya to perform the machining tests and experimental evaluations. Ronnie's experiments are supported from this project. His stipend and tuition is supported by other grants and contracts. Ronnie works closely with material suppliers (workpiece and tooling) to obtain the appropriate supplies needed to conduct our experiments. This past year Ronnie worked at ORNL to perform some fundamental fracture testing of machined ceramic test specimens. Ronnie has also provided numerous design/build services for fixtures, tool, and insrumentaion, inlcuding the dynamometer used in the machining experiments.

Name: Madithe, Nawaz

Worked for more than 160 Hours: Yes

Contribution to Project:

Nawaz graudated in 2004 as a Masters student working with Prof. Patten. Nawaz has contributed to the design and construction of an induction heating system, in collaboration with an industrial partner, to evaluate heating of the high pressure (metallic) phase in-situ. Nawaz is also working on the design and construction of a test fixture to be used for machining experiments on ceramics (silicon carbide and silicon nitride), coupled with infrared temperature measurements and force measurements. Nawaz works closely with Prof. Matt Davies at UNCC in the design of the experiments and construction of the machining fixture, and measurement systems.

Name: Jacob, Jerry

Worked for more than 160 Hours: Yes

Contribution to Project:

Jerry started working on this project in Jan. 2004. He is primarily respondsible for initiating the machining simulation/numerical analysis of the SiC cutting process. He will work closely with Bis Bhatt to perform the actual machining experiments. Jerry will also follow-up on the work that Satya has done on machining of silicon nitride

Name: Bhattacharya, Biswarup

Worked for more than 160 Hours: Yes

Contribution to Project:

Bis started working on this project in Jan. 2004. He is principally responsible for the SiC ceramic machining experimental program at WMU. Bis will work at ORNL-HTML User Facility this summer (2004) to perform a series of machining experiments on polycrystalline SiC.

Name: Kennedy, Tim

Worked for more than 160 Hours: Yes

Contribution to Project:

Tim will be continuing the work of Travis at NCSU (who graduated in 2004) specifically working on machining and TEM studies.

Name: Mariayyah, Ravishankar

Worked for more than 160 Hours: Yes

Contribution to Project:

Ravi is continuing the work of Satya, Ronnie and Nawaz at UNCC (Satya and Nawaz graduated in 2004 and Ronnie graduated in 2005). Specifically he works on single point diamond machining, AFM and SEM evaluations.

Name: Menon, Deepak

Worked for more than 160 Hours: Yes

Contribution to Project:

Deepak is setting up the Nanocut II device to perform instrumented nanometer level 'cuts' on single crystal and CVD SiC, to study the deformation behavior at the nanoscale. Deepak is also using the micro tribometer to study the ductile behavior, and the ductile to brittle transition, of SiC.

Name: Gilbert, Ben

Worked for more than 160 Hours: Yes

Contribution to Project:

Ben is working on the micro Raman system (visible and UV) to evaluate and characterize the surface/subsurface damage (and phase transformations) which occur due to deformation processes, such as nanoindentation, scratching, and machining.

Name: Kattumenu, Ramesh

Worked for more than 160 Hours: Yes

Contribution to Project:

Ramesh had primary responsibility for performing experiments on the micro tribometer and nanoindenter. This work concentrated on single crystal and CVD SiC materials.

Undergraduate Student

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Name: Mitchell, Tatiana

Worked for more than 160 Hours: Yes

Contribution to Project:

Tatiana is working to implement a polishing system to prepare ceramic samples for testing (scratching and machining).

Years of schooling completed: Sophomore

Home Institution: Same as Research Site

Home Institution if Other:

Home Institution Highest Degree Granted(in fields supported by NSF): Doctoral Degree

Fiscal year(s) REU Participant supported: 2005

REU Funding: REU supplement

Name: Williams, Andre

Worked for more than 160 Hours: Yes

Contribution to Project:

Andre is being trained to operate the nanocut device and the machining simulation software, AdvantEdge. He began working with the HPPT group at WMU in the fall of 2005 and continues to participate and contribute to the research program.

Years of schooling completed: Junior

Home Institution: Same as Research Site

Home Institution if Other:

Home Institution Highest Degree Granted(in fields supported by NSF): Doctoral Degree

Fiscal year(s) REU Participant supported: 2006 2005

REU Funding: REU supplement

Organizational Partners

Oak Ridge National Laboratory

We are working closely with three different groups at ORNL: HTML,
Metals and Ceramics, and APS. HTML (Sam McSpadden, Peter Blau) has contributed to sample preparation (silicon nitride disks)and experimental analysis (acoustic microscope, SEM, TEM and micro Raman). We collaborate by working at HTML with their scientific staff, Lei Dong (UNCC) worked at their facility to use the acoustic microscope, and Bis Bhatt and Ravi M. have worked at their facility to use their newly acquired diamond turning machine (DTM) to single point diamond turn SiC in the ductile regime. Dr. Pharr is also collaborating with Dr. Becker at HTML to investigate single crystal silicon nitride materials, relative to nanoindentation work and study of the high pressure phase transformations. The Metals and Ceramics group supports Dr. Pharr's work by providing access to the Nanoindentation facility, and they support the entire research group by providing addiional sample preparation (polishing of silicon nitride) and post process TEM analysis. The APS facility (Ben Larson, Gene Ice) is working closely with Dr. Pharr and Jae-il (UT-K) to design and build the test apparatus to conduct the in-situ x-ray analysis of the high pressure phase transformation during nanoindentation. APS has a nanoindenter head, which is used the synchrotron radiation source to probe the high pressure material in-situ during nanoindenting. Dr. Pharr is also collaborating with Michael Lance on incorporating a diamond anvil cell (DAC) into a micro Raman system for the purpose of performing in-situ Raman analysis to detect HPPT in ceramic materials. Songqing is working with ORNL on FIB fabrication and TEM analysis.

Third Wave Systems, Inc.

Third Wave Systems, Inc. and UNCC and now WMU have had an ongoing collaboration for about six years. Currently this collaboration includes UNCC and WMU having access to the TWS software AdvantEdge (TWS provides the software license free of charge to UNCC and WMU). Additionally, and more importantly TWS, WMU and UNCC collaborate closely on the design and development and testing of the software for use on machining of ceramics (simulation of the machining process). Troy Marusich and Chris Brand, and others, at TWS work closely with Prof. Patten, Prof. H. Cherukuri, Satya and Lei Dong at UNCC, and Jerry Jacob at WMU to evaluate and validate the use of the software code to study the ductile machining of ceramics (silicon nitride and silicon carbide). Most of this work has been focused on 2-D simulations in the past, however we have extended this work to include 3-D simulations, such as scratching of SiC.

Caterpillar Inc, Technical Center

Caterpillar and UNCC are collaborating on a machining of ceramics

project. Two of our students, Satya and Ronnie, worked closely
with Cat to conduct and evaluate an experimental testing program, this has included fracture testing of machined silicon nitride test samples.

Coors Ceramics, Co

We initiated working with CoorsTec in 2003. Initially they provided polycrystalline SiC for testing purposes. More recently they have provided SiC powder for advanced rotary diamond anvil cell experiments (performed in cooperation with Texas Tech Univ, and CVD coated SiC for machining tests evaluation.

Tohoku University

Dr. Patten spent a sabbatical at Tohoku Univesity from Jan. 2003 to the end of July 2003. During this period, Dr. Patten and his colleague Dr. Gao conducted some preliminary machining experiments on single crystal silicon carbide. Two companies were involved in this initial research, a Japanese tooling (diamond) company (Osaka) and a German material (SiC) company (SiCrystal AG).

University of Idaho

We have access to and use their UV Raman system, for evaluation of SiC. Dr. Bergman is our collaborator at UI.

Mound Laser & Photonics Center, Inc.

Mound has prepared some laser machined samples for our use in single point diamond turning experiments. The results of our preliminary work led to the submission of a STTR proposal (Mound and WMU) in 2006.

Raytheon Company

Raytheon has provided material samples (Poco Graphite CVD coated SiC) and scientific support for the project.

Texas Tech University

Valery Levitas and Dr. Y. Ma at Texas Tech have been conducting rotary diamond anvil cell experiments on SiC. This work is aimed at determining the existence of any high pressure phase transformations in this material. This work has lead to confirmation of the direct amorphization of SiC under high pressure and high shear.

PENN STATE UNIVERSITY

Eric Marsh is conducting some fly cutting experiments, single point diamond turning, of various SiC samples (single crystal and CVD coated).

Cree Research Inc

Cree is providing some finanical support and samples for proprietary research on SiC.

Bullen Ultrasonics

Bullen is providing material samples (single crystal Si electrodes) and financial support for materials and supplies (cutting tools) for the single point diamond turning experiments.

Moore Nanotechnology Systems, LLC

Moore Nanotechnology is working with us in cooperation with a one of our other partners, Bullen Ultrasonics, on process development for machining silicon electrodes.

Edge Technologies LLC

Edge is helping us to develop cutting tools for machining ceramics (Si_3N_4 and SiC), and special tips for scratching and nanoindenting research. Edge also quite often will resharped our used diamond tools free of charge as in-kind support of our project.

Center for Tribology Research

CETR provided the micro tribometer and nanoindenter used in part of this research. The equipment was partially purchased with an MRI grant from NSF.

Poco Graphite

Poco provided CVD SiC samples used in the research program.

RAPT Industries, Inc.
RAPT performed some polishing of samples in prepartation for testing.

Other Collaborators or Contacts

We are/have been collaborating with a number of additional companies, primarily in the area of sample preparation, materials, and special tooling. These include: Solutions Technology, Chardon Tool, Sumitomo Ind., Silicon Sense, Nano-instruments, et al.

We are also collaborating with a number of individuals/organizations associated with our Workshop (High pressure phase transformations of semiconductors and ceramics: http://www.wmich.edu/mfe/hppt/ as part of this NSF Grant.

Researchers and organizations include: S. Hsu and Brian Lawn (NIST), W. Oliver (MTS), J. Gilman (UCLA), Y, Gogotsi and S. Domnich, (Drexel), P. Pirouz (CWRU), E. Marsh (Penn. St.U), I. Marinescu (Univ. Toledo), L. Zhang (Univ. Sydney), Valery Levitas and Y. Ma(Texas Tech), Imin Kao (SUNY-StonyBrook), Jiwang Yan, T. Kuriyakawa, and Wei Gao (Tohoku Univ. Japan), Ning Fang (Utah), Murli Manghani (Hawaii), Tom Dow (NCSU), Jim Cuttino, Matt Davies, Stuart Smith, Bob Hocken and Harish Cherukuri (UNCC), Ben Larson (ORNL-APS), Trevor Page (Newcastle, UK)

Activities and Findings

Research and Education Activities: (See PDF version submitted by PI at the end of the report)
Mission: To characterize the role and influence of high-pressure phase transformations in silicon, germanium, silicon nitride, and silicon carbide during the manufacturing process.

Research Areas:

1. Nanoindentation: Characterize the ductile and brittle behavior as a function of indenter radius, included angle(s) and penetration depth, load an unload rates, unload % and hold times. Two emerging research thrusts involve in-situ detection of the high pressure phases during indentation. One involves using a diamond anvil cell within a Raman system, and the other is based upon using a nanoindenter on a beam line at a synchrotron facility. These projects are being jointly conducted with ORNL (HTML and APS).

Summary: To observe the existence of indentation-induced phase transformation (IIPT), nanoindentation tests were executed on Si, Ge, single crystal SiC (4H amd 6H)and Si3N4, polycrystal Si3N4 (GS44), and polycrystal SiC (6H)samples.

An extensive study was undertaken to explore the effect of indenter angle and tip radius on the phase transformation and cracking behavior of Si, Ge, SiC (single crystals of polytype 6H), Si3N4 (commercial grade polycrystalline), and S.C. SiN. The indenters used were all triangular pyramids with centerline to face angles of (in degrees) 35, 45, 55, 65., 75, and 85. After indentation, evidence for phase transformation and cracking was obtained from nanoindentation load-displacement curves and high resolution scanning electron microscopy (SEM). In addition, for Si, micro-Raman spectroscopy and a limited amount of transmission electron microscopy (TEM) of specimens prepared in cross-section by focused ion beam milling (FIB) were also used to explore the HPPT behavior.

The forward and reverse phase transformations have been carefully studied for a variety of load, rate, and indenter geometries. The HPPT for Si, Ge, SiN and SiC, have all been fairly well charterized by these indentation experiments.

2. Scratching: In-situ optical detection and heating of the metallic high-pressure phase. Optical measurements are made in-situ during scratching of Si and SiC using a IR laser system. The metallic high pressure phase can be detected as a substantial decrease in optical (transmission) signal, i.e. a semiconductor (tansparent to IR laser) to metallic (opague to IR) phase change. Similarly, the opaqueness of the metallic high pressure phase leads to absorption of the IR radiation and subsequent heating and softening (preferentially).

Summary: During this past year we have used the opaqueness of the metallic high pressure phase of Si and SiC to accomplish in-situ laser heating using the IR laser. Significant thermal softening, due to laser heating, has been demonstrated. This work has lead to a patent application and future plans for using this system for laser assisted machining of ceramics at the micro level (proposal submitted to NSF February 2007).

In addition to the IR laser experiments, instrumented scratch testing with a micro tribometer (funded in part by a recent NSF MRI grant) has greatly contributed to our knowledge and understanding of the ductile to brittle transition of CVD coated SiC. The ductile to brittle transition (D-B-T)of SiC, at room temperature, has been established in the range of 50 nm to 850 nm, based upon material (crystallography) and process conditions.

3. Material removal: Describe the brittle and ductile behavior as a function of crystallographic (for single crystal) orientation and machining conditions.

Summary: This aspect of the project deals with the ductile machining of semiconductors (Si and Ge) and ceramics (silicon nitride (Si3N4) and silicon carbide (SiC)) by single point diamond cutting tool and the numerical simulation of the cutting process. While the experimental work determines the threshold for transition behavior, the numerical modeling provides a better understanding of the mechanics of the cutting process. The specimen materials undergo high pressure phase transformations at their hardness values (e.g. 12 to 22GPa for silicon nitride, and 20-35 GPa for SiC)under hydrostatic and shear stress conditions. The cutting tools used are single crystal diamond and polycrystalline diamond. Machining is carried out at both micron and sub-micron levels. Machining is accomplished using a PRECITECH Nanoform 350 diamond turning machine (DTM) and a reconfigured micro tribometer. Depths of cut range from 10 nm to over 40 micrometers (for silicon nitride). SiC (single crystal) has been machined at depths of cut from 10 to 1000 nm, with rake angles of 0 to -45 degrees. All the cuts made were imaged and the surface properties studied for brittle or ductile behavior. Chip/debris studies were also done to characterize the mode of material removal. Force data was collected to determine the type of fracture.

The process of ductile mode machining of silicon nitride and silicon carbide has been modeled numerically to study the effects of various parameters such as rake angles, tool tip radius, cutting speeds and feeds on the process. The simulations are carried out using the commercial special purpose metal cutting FEA software ADVANTEDGE, developed by Third Wave Systems. The main objective of the study was to determine the various temperature and pressure conditions that are conducive to ductile material removal of the workpiece material. In addition, machining forces are also studied so as to obtain a correlation with the experimental values. Since realistic modeling of the process is extremely difficult, deviations from the experimental values are expected, but they are not significant. The numerical results also help to determine the general behavior and the threshold for ductile to brittle transition

Raman Spectroscopy is being used as the workhorse, along with TEM, SEM and AFM, to characterize phase transformations during indentation, scratching, and machining.

Findings: (See PDF version submitted by PI at the end of the report)

1. Indenting: (Nanoindentation)

Indentation-Induced Phase Transformation of Semiconductors and ceramics

Tests performed: One step loading and unloading test.
Cyclic Loading and unloading tests
Partial Unloading (% unload) and hold (time).
Indentations with different indenter geometries.

Silicon subjected to high-pressure indentations undergo a phase transformation. The phase transformations (of silicon) have been found to be depended on many process parameters including: geometry of indenter, peak or max load, and rate of loading/unloading. It appears, i.e. a concensus is emerging, that the phase transformations may be nucleation controlled and thus limited. Once the high pressure phases are nucleated, it appears that the phase tranformation continues rapidly, leading to the observed pop-in and pop-out events during nanoindentation. In particular, larger affected zones, and longer times (active processing time) appear to be conducive to generating the phase transformations (forward and reverse), assuming that sufficient pressures/stresses are established to drive the transformations.

Indentation Behavior of SiC: Phase transformations, i.e. an amophous remnant, have not been identified in ductile indended SiC (amorphization during DAC and machining do occur). It is possible that the detection system, Raman spectroscopy, is not sensitive enough to detect the small volume change. Areas of reseach include: wavelength of Raman laser, and laser heating of measurement zone, i.e. the measurement instrument is affecting the measured material state via thermal heating.

Based on the nanoindentation loading/unloading curves and SEM observations of the hardness impressions, no phase transformation could be identified in SiC or Si3N4 (however DAC and machining did reveal phase transformations to an amorphous or nanocrystalline phase). In addition to ductile or plastic behavior, extensive chipping was observed in the SiC which is promoted by smaller indenter angles and higher loads. In Si3N4 both ductile/plastic deformation and chipping were observed. These would presumably be the major mechanisms of material removal during sharp contact.

For Si and Ge, ductile extruded material was observed around the hardness impressions for indentations made with the 35 degree and 45 degree indenters, but not with higher angles. The extrusions are primary evidence for the phase transformation and suggest the tool geometry plays an important irole n the material removal process. Based on cross-sectional TEM work, it is clear that the phase transformation in Si occurs at most of the indenter angles with the exception of the 85 degree indenter, for which indentation is fully elastic and recoverable. However, material is extruded only at the smaller angles; at larger angles, the transformed metallic phase is constrained to regions near the tip of the hardness impression and does not escape to the surface. When the transformed material extrudes from the side of the indenter, the pop-out in the load-displacement data characteristic of the reverse phase transformation disappears. We have also observed for the first time nanoindentation-induced phase transformations in germanium.

Micro-Raman studies were conducted on Si, Ge, SiN and SiC. Results show that both crystalline (metastable forms) and amorphous phases are formed near the hardness impressions, with the exact nature depending on several variables including indenter angle, peak load, and loading/unloading rate. Maps of the distribution of the phases in the surface were made from the micro-Raman data. The micro-Raman shows the extruded material is amorphous, whereas material near the center of the hardness impression is comprised of the metastable crystalline forms Si III and/or Si XII. Maps like these are providing a clear picture of the distribution of the transformed phases in and around the hardness impressions.

2. Scratching: We have scratched silicon wafers using a diamond stylus. The optical transmission, through the silicon, is measured in-situ during scratching. We found that the optical transmission (IR) dropped significantly during scratching. This change in optical transmission is attributed to the metallization of silicon during contact deformation. These experiments confirm the findings from our previous work using electrical detection and heating conditions. These experiments were extended, with the use of higher power IR laser (400 mW) to accomplish micro-laser assisted scratching of Si and SiC. We demonstrated significant thermal softening, via laser heating, for both single crystal Si and SiC. It is planned to advance this technology, micro laser assisted scratching, to our machining operations (a separate proposal has been submitted to NSF, February 2007).

3. Machining: Results from this project indicate that at low feeds,
small tooltip (cutting edge) radius and at all cutting speeds (very slow to very fast: mm/s to m/s) ductile machining of Silicon Nitride and Silicon Carbide is possible. High negative rake angles also promote ductile behavior. Sharp cutting edge radii, less than the depth of cut, appear critical to achieve the best results with ductile machining. For Si and SiC, this necessitates the use of single crystal diamond cutting tools. For silicon nitride, which is not as brittle as the other silicon based materials, a larger depth of cut can be used to achieve ductile machining. This permits the use of less expensive polycrystalline diamond tools which have a larger cutting edge radius, i.e. not as sharp as single crystal diamond tools.

TEM evaluation was performed on the ducitle chips/debris generated from the machined SiC single crystal material. The ductile chips/debris were found to be amorphous, indicating a phase transformation during machining from single crystal to amorphous. The origin of the phase transformation, which has not been seen in the corresponding indentations nor reported in the literature, was evaluated, and has been confirmed by our Rotary DAC experiments. It is hypothesised that significant shear is needed to drive a high pressure phase transformation in SiC, which would explain why similar behavior (crystalline to amporphous phase transformations) have not been reported for diamond anvil cell (DAC)and indentation experiments. We have conducted rotary DAC, in cooperation with colleagues at Texas Tech and Brookhaven National Lab, to probe for phase transformations in SiC under high shear conditions. Amorphization of SiC has been identified to occur at high pressure and high shear in the rotary DAC (as it has occured during machining), conditions which normally don't occur in DAC or indentation, but which do occur during machining.

Ductile Regime Diamond Turning of Si for HPPT Analysis: See attached file.

4. Micro Raman Studies:

Ultraviolet Raman spectroscopy is employed to analyze the structure of indented 6H-SiC. The unequal shift in the transverse optical (TO) and longitudinal optical (LO) Raman modes to higher wave numbers indicate that regions within the indent are under biaxial compressive stress. The amount of stress can be determined from previous work on 6H-SiC subjected to induced stress in a diamond anvil cel.

Ultraviolet and visible Raman spectroscopy depict the structure of micro and nano machined polycrystalline Ô-Si3N4. In Si3N4, UV light is a surface sensitive probe and visible light can reveal information of the bulk of the material. The broadened UV spectra indicate that the surface of the machined Si3N4 is amorphous for the machining depths of cut analyzed. These spectra are compared to UV spectra of ground Si3N4 which show sharp crystalline peaks. In addition, the sharp peaks in the visible Raman indicate that the bulk of the material is crystalline.

5. Material Models: Efforts were successful to develop more realistic constitutive models based upon nanoindentation experiments. The models include the effects of the phase transformations (except for the volume changes), but they do not model the brittle material behavior. These models have been incorporated into the FEM machining simultions.

Training and Development:

Working in a collaborative environment is one of the biggest rewards associated with this project. We have assembled a unique team of qualified researchers (faculty, staff, students, and industry) that have demonstrated their desire and interest in working in close collaboration across the four participating universities. As this is a multi-disciplinary and inter-disciplinary research group, we benefit tremendously from cross fertilization of ideas, from each others insights and experiences. The innovation and creativity resulting from this collaborative experience has propelled us to pursue and achieve significant results that could not have been possible otherwise.

Our on-line collaboration (web site and e-mail) in addition to our periodic live group meetings, and our highly successful annual HPPT workshop have resulted in a dynamic research environment, which in itself is a unique educational experience.

We are training students in the sciences, engineering disciplines, and technology to work as a team towards a group goal, while maintaining their own discipline identity. The students (and faculty) learn continually from each other through our daily exchanges and communication, augmented with on-site visits.

The workshops held in August bring together most of the key players (national and international groups) working in this research field. We have had representation from five of the world's seven continents! The exchange of ideas and understanding, along with the synergy of the event is a capstone in our research program. In particular, the students participating in this unique forum are exposed to some of the best and brightest, and most productive, researches working in this area. For these students this is a rare opportunity to interact with such as diverse and internationally recognized group of scholars.

Outreach Activities:

Presentations: In addition to our in-house presentations, the group's members regularly participate outside our research program by giving presentations to various groups. This includes participation at other forums within our respective universities, and activities outside our home institutions. Presentations include seminar series at WMU, UNCC and NCSU, associated with the precision engineering programs, and presentations done for or in conjuction with our other collaborators, such as ORNL-HTML, TWS, Tohoku University, Kitami University, etc. Particularly noteworthy is our strong showing at the ASPE annual meetingsand the MRS fall meetings, where most of the PIs presented the results of the research from this project. The research program continues to undertake an international component as participants attend our workshop from Tohoku Univ. in Sendai Japan, and Kitami Institute of Technology in Hokkaido Japan, Newcastle in UK, and Univ. Syndey in Australia.

Web Site: The research programs web site: http://www.micro.physics.ncsu.edu, is used for two purposes.
First it is an easy way for the various participants to communicate their research progress (some of this is maintained in a 'private' or secure area of the web site where a login and password are required). This provides us with an avenue for ready dissemination of our research results and provides an excellent mechanism to review and evaluate every aspect of the research program. The web site is also very useful for communicating our research programs and results to the larger scientific community. The web site is periodically updated, with new results of

the research program, including experiments, presentations, and publications.

Workshop: The workshops provide us the opportunity to focus on efforts for disseminating research results and progress to a small group of researchers working in this area of specialization. Contributions from others outside our group provides a broader and more rounded presentation, and has lead to additional collaborative relationships.

2003 http://www.continuinged.uncc.edu/hppt/workshop.htm
2004 http://www.micro.physics.ncsu.edu/Brochure%202004.PDF
2005 http://pharr.engr.utk.edu/HPPT/
2006 http://www.wmich.edu/mfe/hppt/

The fourth and final workshop for this funded project was held August 14-15, 2006 at Western Michigan University in Kalamazoo Mi.

Journal Publications

John A. Patten (WMU), Ronnie Fesperman, Satya Kumar - UNCC
Sam McSpadden, Jun Qu, Michael Lance - ORNL
Robert Nemanich, Jennifer Huening - NCSU, "High pressure phase transformation of silicon nitride", Applied physics letters, p. 4740, vol. 83, (2003). Published

John Patten, Lei Dong, Jimmie Miller, "Electrical and Optical Detection and Heating of the High Pressure Metallic Phase of Silicon In-situ During Scratching with Diamond and its effect on the material?s hardness
", Proceedings ASPE 2003 Annual Meeting, p. 507, vol. 30, (2003). Published

J.-I. Jang, S. Wen, M.J. Lance, I.M. Anderson, and G.M. Pharr, "Cracking and Phase Transformation in Silicon During Nanoindentation", MRS Proceedings, p. 1, vol. 795, (2004). Published

J.J. Huening, R.J. Nemanich, J.-I. Jang, G.M. Pharr, X. Chen, and L. Bergman, "UV Raman Scattering in 6H SiC Indents", Annual Conference of the American Society for Precision Engineering, p. 415, vol. 30, (2003). Published

J.-I. Jang, S. Wen, M.J. Lance, I.M. Anderson, and G.M. Phar, "Cracking and Phase Transformation in Silicon During Nanoindentation", MRS Symp Proc: Thin film stresses and mech. prop. X, p. U8.15.1-6, vol. 795, (2004). Published

J. Qu, P.J. Blau, A.J. Shih, S.B. McSpadden Jr., G.M. Pharr, and Jae-il Jang, "Scanning Acoustic Microscopy for Non-destructive Evaluation of Subsurface Characteristic", 6th International Conference on Frontiers of Design and Manufacturing, p. 1, vol. , (2004). Published

S. Wen, J. Bentley, J.-I.. Jang, I.M. Anderson, and G.M. Phar, "Characterization of Nanoindentations in Silicon by Cross-sectional TEM", Microx.Microanal. (Annual meeting of the Microscopy Society of America): Suppl. 2, p. 56-57, vol. 10, (2004). Published

Satya K. Ajjarapu, Ronnie R. Fesperman, John A. Patten and Harish P. Cherukuri, "Ductile Regime Machining of Silicon Nitride: Experimental and Numerical Analyses", CIRP, p. 1, vol. , (2004). Published

Satya K. Ajjarapu?x, Ronnie R. Fesperman?x, John A. Patten? and Harish P. Cherukuri, "Experimental and Numerical Investigation of Ductile Regime Machining of Silicon Nitride", Numiform, p. 122, vol. 8, (2004). Published

Satya K. Ajjarapu?x, Ronnie R. Fesperman?x, John A. Patten, Harish P. Cherukuri?x and Chris Brand, "Experimental and Numerical studies of Ductile Regime Machining of Silicon Nitride", TWS User's Conference, p. 8, vol. 2004, (2004). Published

Satya K. Ajjarapu, Ronnie R. Fesperman, John A. Patten and Harish P. Cherukuri, "DUCTILE REGIME MACHINING OF SILICON NITRIDE: A NUMERICAL STUDY USING DRUCKER-PRAGER MATERIAL MODEL", NAMRC, p. 519, vol. 32, (2004). Accepted

S.K. Ajjarapu, J.A. Patten, H. Cherukuri, C. Brand, "Numerical simulations of ductile regime machining of silicon nitride using the Drucker-Prage material model", J. Mechanical Engineering Science, Proc. Instn Mech. Engrs, p. 1, vol. 218, (2004). Published

B. W. Austin, T. Randall and R. O. Scattergood, "Residual Stress Bend
Effect Due to Diamond Tip Scribing of an Al2O3-TiC Composite
Ceramic", Ceramic Transactions, Amer. Ceram. Soc., p. 117, vol. 156, (2004). Published

T. Randall and R.O. Scattergood, "Characterizing Residual Stress in
Scribes on Silicon Using Deflection Measurements", Proceedings of ASPE
18th Annual Meeting, p. 1, vol. 30, (2003). Published

Lei Dong and John Patten, "In-Situ Infrared (IR) Detection and Heating of the High Pressure Phase of Silicon during Scratching Test", MRS 2004 Symposium R, Proceedings, p. 285, vol. 841, (2005). Published

Lei Dong and John Patten, "In-Situ Infrared (IR) Detection and Heating of the High-
Pressure Phase of Silicon during Scratching Test", International Journal of Manufacturing Technology, p. 1, vol. 7, (2005). Published

John Patten, Wei Gao, Kudo Yasuto, "Ductile Regime
Nano-Machining of Single Crystal Silicon Carbide", Journal of Manufacturing Science and Engineering, p. 522-532, vol. 127, (2005). Published

Jae-il Jang , M.J. Lance , Songqing Wen a Ting Y. Tsui , G.M. Pharr, "Indentation-induced phase transformations in silicon: influences of load, rate and indenter angle on the transformation behavior", Acta Materialia, p. 1759, vol. 53, (2005). Published

Jae-il Jang, M. J. Lance, Songqing Wen and G. M. Pharra!, "Evidence for nanoindentation-induced phase transformations in germanium", APPLIED PHYSICS LETTERS, p. 131907, vol. 86, (2005). Published

Songqing Wen, Jae-il Jang, George Pharr, "Cross sectional TEM studies of indentation induced phase transformations in Si: Indenter Angle Effects", MRS Symp. Proc. R (Fundamentals of Nanoindentation and Nanotribology III), p. 279, vol. 841, (2005). Published

Jae-il Jang, Songqing Wen, JJ Huening, RJ Nemanich, George Pharr, "Micro Raman mapping and analysis of indentation induced phase transformation in germanium", MRS Symp. R Proc. (Fundamentals of Nanoindentation and Nanotribology III), p. 291, vol. 841, (2005). Published

J. Walter (Huening), M. Liang, Jae-il Jang, J. Patten, G. Pharr, R. Nemanich, "UV Raman scattering analysis of indented and machined 6H SiC and beta-Si3N4 Surfaces", MRS Symp. T Proc. (Surface Engineering-Fundamentals and Applications), p. 75, vol. 843, (2005). Published

Scattergood R. O., "Ductile Grinding of Brittle Materials
", Ceram. Trans.
Indentation Techniques in Ceramic Materials Characterization, p. 131, vol. 156, (2004). Published

Kennedy T, Randall T. and Scattergood R. O.,, "TEM and Raman
Spectroscopic Analysis of HPPT in Diamond Turned Silicon Single
Crystal", Precision Engineering Center Annual Mtg,, p. 51, vol. xxii, (2005). Published

J.A. Patten, J. Jacob, A Grevstad, B. Bhattacharya, "Numerical simulations comparing SPDT Experiments on Silicon Carbide", TWS International Users' Conference, Detroit Michigan May 4, 2005, p. 7, vol. 2005, (2005). Published

Jerry Jacob and John Patten, "Numerical and Experimental analysis of 3-D ductile scratching of CVD SiC", TWS Internation Users Group Meeting, Chicago Il, p. 1, vol. 1, (2006). Published

Bis Bhatt and John Patten, "Single Point Diamond Turning of CVD coated Silicon Carbide", ASME MSEC Conference, p. 1, vol. 1, (2006). Accepted

Jerry Jacob and John Patten, "Numerical and Experimental evaluation of the ductile regime machining of Silicon Carbide", ASPE Annual Meeting Conference Proceedings, p. 1, vol. 1, (2005). Published

Bis Bhatt and John Patten, "Ductile Regime Machining of Silicon Carbide", ASPE Annual Conference Proceedings, p. 1, vol. 1, (2005). Published

Jerry Jacob and John Patten, "3-D Nano-scratching in silicon and
silicon carbide", TWS International Users Conference, p. 1, vol. 1, (2006). Published

Tim Kennedy, Ron Scattergood, "High Pressure Phase Transformation of Silicon", Precision Engineering Center Annual Report, p. 9, vol. 1, (2006). Published

Biswarup Bhattacharya, John Patten, Jerry Jacob, "Single Point Diamond Turning of CVD coated Silicon Carbide", 2006 ASME International Conference on Manufacturing Science and Engineering, p. 41, vol. 1, (2006). Published

Jerry Jacob, John Patten, "Comparison between numerical simulations and experiments for single point diamond turning of silicon carbide", NAMRC 2007, p. 1, vol. 1, (2007). Accepted

Lei Dong, John Patten, "Real time Infrared (IR) thermal imaging of laser-heated high pressure phase of silicon", ALAC 2007, p. 1, vol. 1, (2007). Submitted

Deepak Ravindra and John Patten, "Determining the Ductile to Brittle Transition (DBT) of a Single-Crystal 4H-SiC Wafer by Performing Nanometric Cutting", SME ISAAT 2007, p. 1, vol. 1, (2007). To be submitted March 9

Andre Willliams, John Patten, "Numerical Simulation of Vibration Assisted Machining", ASPS Spring Topical Meeting, 2007, p. 1, vol. 1, (2007). Submitted

T. Kennedy, R. Scattergood, "TEM and Raman Spectroscopic Analysis of
High Pressure Phase Transformations in Diamond Turned Single Crystal
Silicon", Precision Engineering Center Annual Report, p. 9, vol. 2006, (2007). Accepted

Books or Other One-time Publications

John Patten - WMU
Harish Cherukuri - UNCC
Jiwang Yan - KIT, "Ductile Regime Machining of Semiconductors and Ceramics
(Section/Chapter 6)", (2004). Book, Published
Editor(s): Y. Gogotsi, S. Domnich
Collection: High Pressure Surface Science and Engineering: High Pressure Phase Transformations
Bibliography: ISBN0750308818
Institute of Physics (IOP)
Bristol UK

Randall, J. T., "Characterizing the Ductile response of Brittle
Semiconductor Materials to Dynamic Contact Stresesses", (2004). Thesis, Published
Editor(s): NCSU
Collection: MS Thesis
Bibliography: NCSU MS Thesis

Walter, J. J. H., "Raman Scattering Analysis of Structural
Transformations in Precision Engineered Si, 6H-SiC and b-Si3N4", (2004). Thesis, Published
Editor(s): NCSU

Collection: MS Thesis
Bibliography: NCSU MS Thesis

Satya Ajjarapu, "Experimental Analysis of Ductile Regime Machining of Silicon Nitride", (2004). Thesis, Published
Editor(s): UNCC
Collection: UNCC MS Thesis
Bibliography: MS Thesis

Ronnie Fesperman, "Diamond Turning of Silicon Nitride", (2005). Thesis, Published
Editor(s): UNCC
Collection: UNCC MS Thesis
Bibliography: MS Thesis

Nawaz Maditheti, "Study of induction heating of silicon wafer and machining of polycrystalline silicon carbide", (2004). Thesis, Published
Editor(s): UNCC
Collection: UNCC Creative Design Project
Bibliography: Creative Design Project

Jerry Jacob, "Numerical and experimental analysis of ductile regime machining of silicon carbide", (2006). Thesis, Published
Editor(s): Western Michigan University
Collection: MS Thesis
Bibliography: WMU Library

Bis Bhatt, "Single Point Diamond Turning of Silicon Carbide", (2005). Thesis, Published
Editor(s): Western Michigan University
Collection: MS Thesis
Bibliography: WMU Library

John Patten, Jerry Jacob, Ning Fang, Eric Marsh, "Ductile Machining and High Pressure Phase Transformations of SiC and Si3N4", (2007). Book, Submitted
Editor(s): Jiwang Yan and John Patten
Collection: Semiconductor Machining in the Micro-Nanoscale
Bibliography: NA

Jerry Jacob, "Numerical Simulations on Machining of Silicon Carbide", (2006). Thesis, Published
Editor(s): NA
Collection: MS Thesis
Bibliography: WMU Library

Lei Dong, "In-situ detection and heating of the high pressure metallic phase of silicon during scratching", (2006). Thesis, Published
Editor(s): NA
Collection: PhD Dissertation
Bibliography: UNCC Library

Songqing Wen, "A Kinetic Study of Indentation Pop-out in Silicon",", (2006). Thesis, Published
Editor(s): NA
Collection: PhD Dissertation
Univ. of Tennessee
Bibliography: NA

Web/Internet Site

URL(s):

http://www.micro.physics.ncsu.edu

Description:

This is the official web site for our research project.

Other Specific Products

Product Type:

workshop web sites

Product Description:

Web site for workshops:

2003 http://www.continuinged.uncc.edu/hppt/workshop.htm
2004 http://www.micro.physics.ncsu.edu/Brochure%202004.PDF
2005 http://pharr.engr.utk.edu/HPPT/
2006 http://www.wmich.edu/mfe/hppt//

Sharing Information:

Available via our program web site: http://www.micro.physics.ncsu.edu

Contributions

Contributions within Discipline:

The education and training of students working in a multi-disciplinary research environment produces significant results throughout the careers of those participating in this research program. This effort has similarly contributed to the students' experience during the course of the studies and research.

The workshop activity greatly increased the dissemination of our research results, in a concentrated and focused manner, to insure a broader impact of our research program.

Contributions to Other Disciplines:

High pressure phase transformations, HPPT, are now known to occur in many materials (such as gases, liquids, and solids), and are accessed across many disciplines (engineering: mechanical and manufacturing, materials science: semiconductors and ceramics, physics, geology, chemistry, etc.). The fundamental science and discoveries associated with HPPT in semiconductors and ceramics has initiated a new field of research that was previously unknown. Through the contributions of our FRG (in particular, publications, presentations, and the workshops) we have greatly disseminated the results of our research to the greater scientific community thereby significantly extending the reach of our contributions to other disciplines.

Contributions to Human Resource Development:

The students, undergraduate and graduate, participating on this research project have developed significantly during the four and on-half years. They have developed advanced experimental, numerical and analytical skills, and have advanced their project and teamwork skills (soft skills).

Of particular note is that the project resulted in the graduation of three females, two PhDs and one MS degree!

Contributions to Resources for Research and Education:

Our primary contribution has been the HPPT web site: http://www.micro.physics.ncsu.edu and the annual workshops.

Contributions Beyond Science and Engineering:

A small firm, Third Wave Systems (TWS), is beginning to commercialize the results of our research. TWS is a software company that develops machining simulation products and R&D services for the industrial/manufacturing community. They are including the results of our ceramics machining project into their advanced software products and services.

Categories for which nothing is reported:

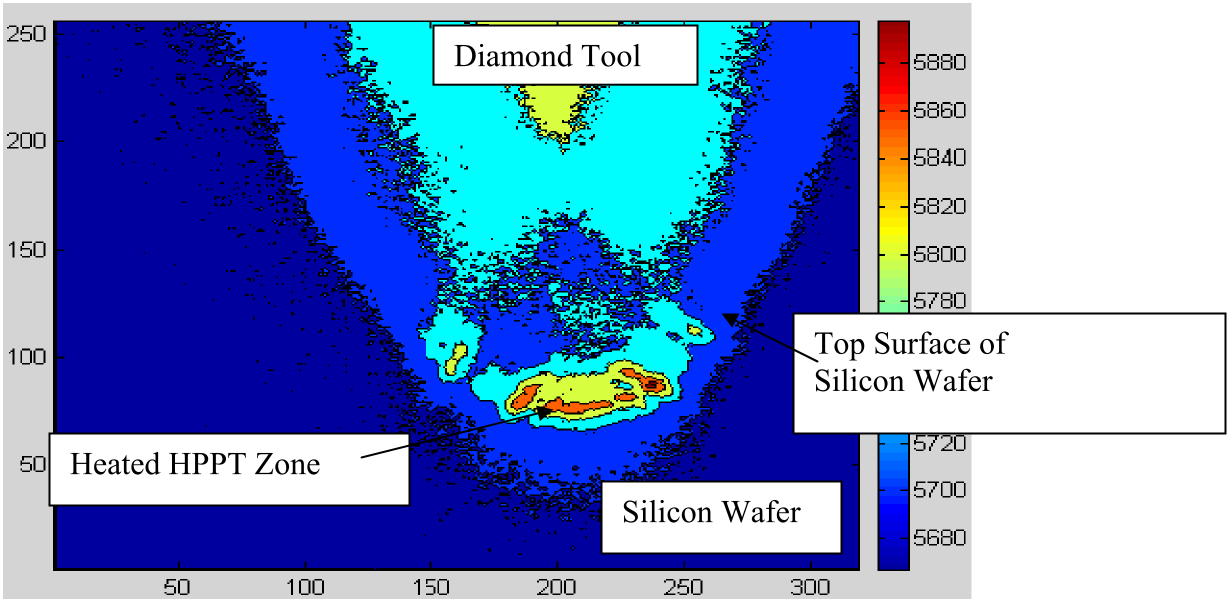


Figure 1Thermal camera image: preferential heating of sub surface high pressure phase transformed region

High Pressure Phase Transformations Workshop 2006

ABOUT THE WORKSHOP

August 14-15 2006

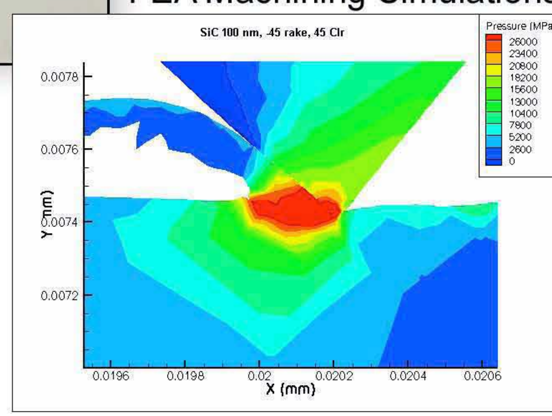
The workshop is a part of a National Science Foundation (NSF) Focused Research Group (FRG) research program. This research initiative includes materials scientists, manufacturing/mechanical engineers, and physicists. The goal of the workshop is to convene a larger audience to explore and discuss the nature of high pressure phase transformations that occur during indentation and machining processes and to elaborate on the research opportunities they provide. The workshop presentations and posters will convey information and theories about the latest research efforts in this field. Visit our technical web site http://www.micro.physics.ncsu.edu

Click here for online registration

SPDT of CVD coated SiC

Picture showing the optical quality of the surface finish of ductile machined CVD SiC

FEA Machining Simulations



WORKSHOP LOCATION

Western Michigan University
College of Engineering
and Applied Sciences
Kalamazoo, Michigan

WORKSHOP GOALS

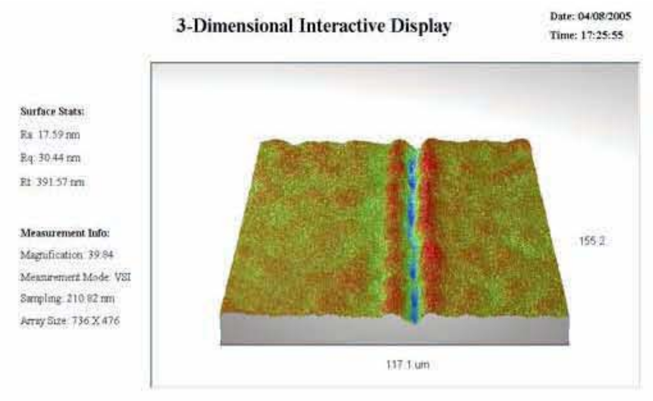
- Knowledge and awareness of recent advances regarding the high pressure phase transformations of semiconductors and ceramics.
- Interact with the broader scientific community working on these materials.
- Discuss technical and manufacturing issues associated with advanced engineering of the materials
- Participate with future program and planning activities

High Pressure Phase Transformations Workshop 2006

Conference attendees are encouraged to make an oral or poster presentation. Abstracts may be submitted to John Patten at john.patten@wmich.edu. The abstract submission deadline is July 17th.

Topics at the meeting include but are not limited to:

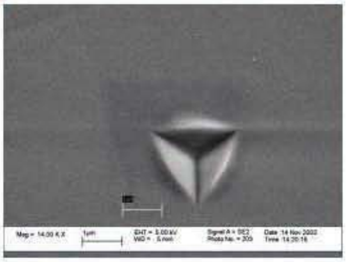
- High pressure phases (HPP) and phase transformations (HPPT) of semiconductors and ceramics: Si, Ge, Si_3N_4, SiC, etc.
- HPP of other materials
- Machining and single point diamond turning
- Nanoindentation
- Polishing and chemo-mechanical polishing
- Ductility of semiconductors and ceramics
- HPPT to metallic phases
- Mechanical, thermal, electrical and optical effects in HPP
- In-situ and post-process analysis of HPPT
- Amorphous and crystalline material dependences of HPPT
- Surface science and engineering
- Friction and wear at the nano-scale



Who should attend?

Engineers and Scientists working on advanced engineering materials and involved in design, testing, and manufacturing. Structural ceramics, microelectronics, and optical components will be highlighted. Knowledge about the high pressure behavior of semiconductors and ceramics will be emphasized.

Nanoindentation



50 mN (no cracking and no chipping)

Workshop Organizers

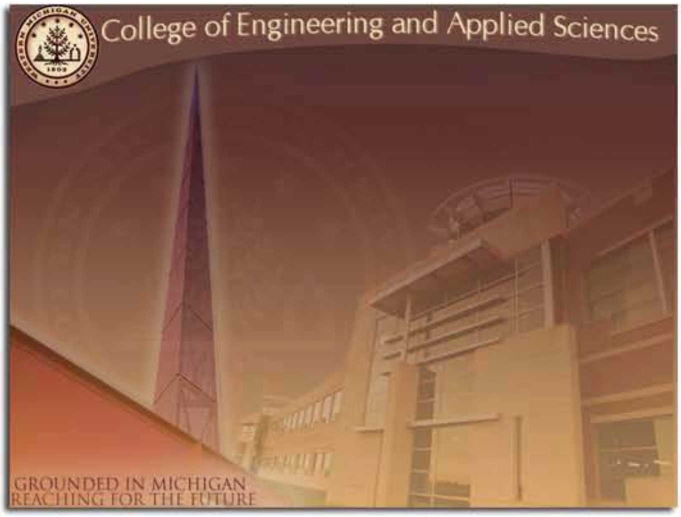
John Patten
John.Patten@wmich.edu
(269) 276-3246

Organizing Comittee

John Patten (WMU)
Ron Scattergood (NCSU)
Bob Nemanich(NCSU)
George Pharr (UT-K)

Topics

- HPPT of Semiconductors and Ceramics
- Machining of Ceramics
- Nanoindentation
- Micro Raman
- Micro Beam x-Ray
- Diamond Anvil Cell
- In-situ detection and characterization techniques
- HPPT modeling and simulation



ACCOMODATIONS

- We have reserved a block of rooms at the Holiday Inn West Kalamazoo for the nights of August 13 and 14, make your reservations early. For reservations, call 269-375-6000 Rate: $79.00 / night + tax

Hotel Accommodations
The Holiday Inn West 2747 South Eleventh St. Kalamazoo, MI 49009 269-375-6000 www.kalamazooholidayinn.com

WORKSHOP FEE

- The workshop will be held at the College of Engineering and Applied Sciences on the Parkview Campus of Western Michigan University. The workshop fee is $75.00 per person which includes morning coffee/ danish, lunch on Monday, and the conference banquet on Monday evening. Please register online.

Click here for online registration

Holiday Inn

The tentative workshop schedule is:

Monday Aug 14	
7:30-8:30A	Registration and Coffee/Danish
8:30-9:00	Welcome
9:00-10:15	Session 1
10:15-10:45	Break/Coffee
10:45-12:00	Session 2
12:00-1:00P	Lunch
1:00-2:15	Session 3
2:15-2:45	Break/Refreshments
2:45-5:00	Session 4
6:30	Conference Dinner

Tuesday Aug 15	
7:30-8:30A	Coffee/Danish
8:30-10:45	Session 5
10:45-11:00	Break/Coffee
11:00-12:00	Summary and Discussion
12:00P	Adjourn

Kalamazoo, Michigan

All sessions take place in room D-115

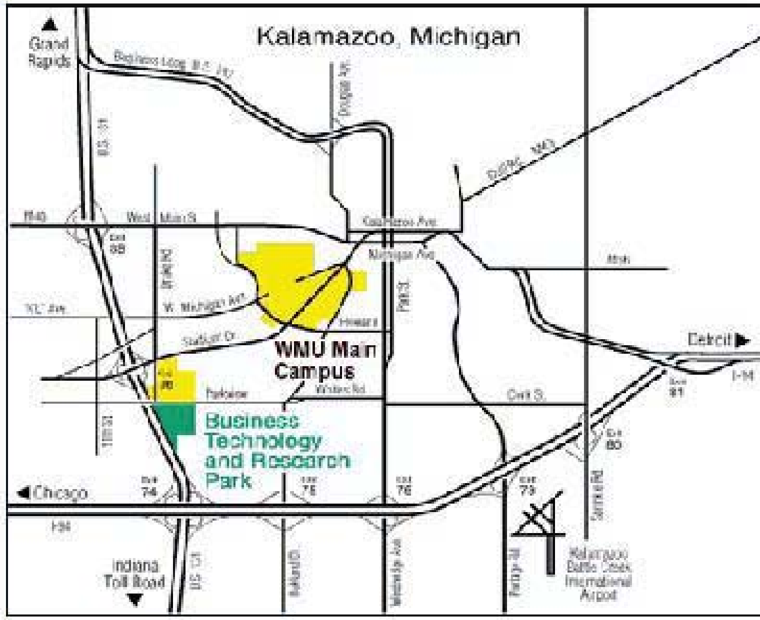
DIRECTIONS - to WMU College of Engineering and Applied Sciences

From I-94
At exit #74, turn north on U.S. 131, go 2.8 miles, then follow the directions listed below for U.S. 131.

From U.S. 131
At exit #36A, turn east onto Stadium Dr. Turn right at first light which is Drake Rd. Continue on Drake Rd. Through the next light (at Parkview Ave.) into the WMU Parkview Campus. You will now be on Campus Drive.

From the Main WMU Campus
From the corner of Stadium Dr. and Howard, go west on Stadium Dr. until you come to Drake Rd. Turn left onto Drake and continue south through the next light (at Parkview Ave.) and into the WMU Parkview Campus. You will now be on Campus Drive.

* Visitor Parking is located on College Circle
* Student and Employee parking located behind the College of Engineering via Engineering Way.



Click here for online registration