

# NSF-FRG: HPPT Semiconductors and Ceramics, Meeting Minutes of January 10, 2003

## Introductions:

Dr. Jim Miller introduced to group. Jimmy will be added to e-mail list.

## 1. New Items:

A. Use of Acoustic Microscope (AM) at ORNL-HTML to image machined SiN by Patten. Use of polished surfaces would give better results. AM clearly showed subsurface plastic deformation, due to machining, distinctly from ground region. John will send fully machined samples to Sam McSpadden to image. Note: Sam is suppose to provide polished SiN disks for the group to use. John will follow up with Sam.

## 2. Workshop:

A. Everyone should review the brochure and provide Lyndee with feedback for changes and corrections.

B. Everyone should review the workshop program and make suggestions.

i. Suggested additional topics.

ii. Invite or suggest possible speakers.

a. Industrial representatives.

Nanoindentation Warren Oliver (George to follow up)

Machining (Ron and John will identify), Precitech, Pneumo

Cree: Bob can invite someone?

SRC

Semitech

LLNL – Ken Blaedel (John to contact)

Applications

St. Gobain (sp?) (Ron to contact-Subramanian)

Bearing Co. SKF (John to contact)

Grinding Machines-Meister Grinders – Lou Padnos  
(John to Contact)

C. Invited speakers

i. John has invited J.J. Gilman (UCLA)

ii. John will contact Lee Cook at Rodel and Tomizowa (sp) for CMP

## 3. Summary of research since Oct. meeting.

A. Nanoindent: George and Jun-el have nanoindented SiC, both in ductile and brittle mode. They provided some samples for Bob and Jennifer to look at. These were coated with gold (for SEM imaging); they will also provide some uncoated samples.

B. Scratching and Electrical Heating of Si: Lei has been exploring the parameter space (radius of stylus, load, and current) to establish suitable range of variables. Previous work by Nobu (at UNCC) only used a fixed current of 1 mA, and one size tip

radius of 9  $\mu\text{m}$  (where 10 g mass produced most consistent results). It appears that higher loads (>5 g) may be required to completely remove the gold contact material and expose the Si substrate.

C. Scribing and bending: Ron

- i. Scribing silicon, looking at orientation (plane) and direction.
- ii. Pursuing lower loads 10 mN (to avoid fracture).
- iii. Higher loads result in fracture.
- iv. Ron requested some CMP material from John.  
(John provided Ron with low and high pH slurries w/o grit).
- v. Have not done anything with SiN wafers yet (will look at later).
- vi. The SiN disks are too small for this test.
- vii. Ge will be investigated for a comparison test.

D. Micro-Raman of indented Si, SiC and machined SiN: Bob and Jennifer

- i. Results of SiN bars and disks, machined surface and chips presented.
  - a. Si inclusions to be used as a marker, at  $520\text{ cm}^{-1}$
  - b. Need more chip material to evaluate.  
A lot of noise (due to limited material sample)  
John to send Jennifer more chips.  
Larger quantities tend to be from brittle machining  
(10 and 30 micrometer depth of cut)  
Note: used Ge substrate to eliminate Raman signal overlap.
  - c. Bars and disks look alike (Gs-44 HIP material).
- ii. Results of Si indentation and scribes presented.
  - a. Si indents (of unknown history):
  - b. Picking up 2nd peak indicative of strain and/or phase transform
- iii. Prelim results of SiC nanoindents presented.
  - a. Indented area looks identical to native material.  
SiC is transparent to Raman Laser
  - b. Possibly UV laser could sample better,  
i.e. just probe the near surface  
Univ. of Idaho has such a system (Bob to investigate using  
their system)
- iv. Si scribes: Scribes show residual stress and amorphous and possibly new phases.

E. Machining simulations:

- i. John (Satya and Lei) will conduct some additional simulations using TWS to study residual stress to compare to micro Raman and scribe  
Results

F. Infrared of SiN: Lei tested all of the SiN samples with her IR detection system and they were all transparent to the IR, and thus suitable to probe for any high-pressure metallic phases.

#### 4. Research topics-Discussion.

##### A. In-situ micro Raman, no progress to report.

Bob will consider a simpler design (e.g. a small poker) and a suitable diamond to perform tests. The discussion evolved and concluded that a simpler design might be readily implemented to make the experiment easier to conduct. Bob will lead this effort. Generally the group thought this potential experiment important enough not to give up on yet. We will re-evaluate in August, or before, when Bob has something to share with the group. Other alternatives discussed included seeking additional funding, perhaps through a SERG or NER (exploratory research grant), or additional funding for our FRG (to put back some of the original funding that was cut out, or by adding another year with funding to the current project) or collaboration with others (e.g. Gogotsi).

##### B. In-situ x-ray of nanoindent: Collaboration with ORNL and APS (Gene Ice).

Jae-il will pursue this experiment now that he has clearance to work at the lab. George will initiate conversation with ORNL to pursue this experiment.

##### C. SiC samples: Bo to get prices on 6H 2" wafers.

##### D. Single crystal Si<sub>3</sub>N<sub>4</sub>: George to get some samples from Paul Beeker at ORNL.

Note: George will provide some uncoated indented SiN (polycrystal wafers).

E. Polycrystal SiC and S.C. SiC. John has obtained some (3) single crystal SiC wafers (from Lou Padnos of Meister Grinding). One was sent to Eric Marsh at Penn. State to scratch, and the other two John will take to Japan to perform machining tests. Two of the wafers are 6H and one is 4H. One of the 6H wafers is on a sapphire substrate (this one will be scratched by Eric).

John has also ordered some polycrystalline SiC from Coorstec. This is a structural ceramic that we will use for our preliminary machining work (to determine how machinable the material is).

##### F. Fluid Scratching and Cutting:

- i. Ron to make some scribes with the CMP slurries (slurry provided by John).  
John (Lei) can also make scratches with slurry if needed.
- ii. George will coordinate TEM cross sections of scratches made with and without slurries (best to compare scratches made at the same load).
- iii. John (Satya) will pursue cutting tests using NIST exp. Alcohol fluid to study tool wear.

Note: Recent work by Zhang et al. concluded also that the ductility of the high-pressure  $\beta$ -Sn phase of Si was decreased with the use of water [Key Eng. Mat. Vol. 233-236 (2003) pp. 609-614]. This is the same conclusion I (John)

reached previously, see ASPE 2000 p. 604 conf. Proc.

G. Micro Raman HPP (SiC and Si<sub>3</sub>N<sub>4</sub>): Bob, nothing to report (We should conduct a literature survey to see if anything is available).

H. Germanium Wafers: Ron to pursue purchasing, in the meantime he will send some pieces to George to indent.

I: Boron Doping of diamond (type II-B, to make conducting) Bob and George, nothing to report.

J. Vanadium carbide indenters: George, nothing to report.

Note: at this time there is no need to follow up on I and J, they are for future ref. These items (conductive indenters) are a possible alternative to use in the elect. Measurement experiments conducted by Lei.

K. Nanocut of SiC: John (in coordination with Bob Hocken) will pursue having another student (Dan Abbott) perform some nanocuts on SiC.

L. Scratching of SiC: John and Lei will pursue scratching one of the wafers that Bob provided earlier. Also (see above 4 E above) John sent a SiC wafer to Eric Marsh at PSU to scratch.

M. SiC collaboration with Paul Beeker of ORNL: George will pursue, see 4 D above. (Note: George, does this mean that Paul also is a source for SiC or that he simply has an interest in this material?)

N. Crystal structure of SiN wafers: Ron to give Bob a thin film wafer to evaluate the crystal structure (amorphous or polycrystal). Note: John has the balance of the SiN wafers in the clean room at UNCC (contact John Hudak the lab manager if more wafers are required-we purchased 25 wafers so we have lots of them to use).

O. Other materials to consider: SiGe, Ceramics, AlTiC, Sapphire, AlN  
No further discussion at this time .

P. Scratching Stylus Radius: John has found a source (EMI gage) for various diamond stylus tips we have purchased a 10 micrometer tip, and we can also obtain 2 and 5 micrometer tips, to give us a range of stylus sizes. (Note: George also has a variety of nanoindenter tips (shape – included angle) and spherical tips of various sizes to use).

Q. Infrared imaging - temp. measurements: John has a student, Nawaz, working with Matt Davies to pursue these measurements at UNCC. Initially Nawaz will use the SiC samples ordered from Coorstec for the fixture design. John is pursuing obtaining samples of GS-44 SiN of the same geometry to use in addition to SiC. The samples are thin walled cylinders compatible with Matt Davies instrument set up.

R. Infrared sensing and heating: Lei will modify the scratching apparatus to implement the IR detection system for in-situ detection of the metallic high-pressure phase during scratching.

Note: If Q and R are successful, we plan to use a higher output IR laser to perform heating of the sample during scratching to see if we can thermally soften the high pressure metallic phase, and measure the temperature during scratching. This would augment the electrical heating system that Lei is currently using to thermally soften the Si during scratching.

S. Scribing/Bend tests of SiN (wafers) See. 3 C and 4 N above. Ron will dice the SiN wafers to the appropriate size for testing, as noted above, the SiN disks are too small (diameter) to be used. (Note: the potential problem with the SiN wafers is that if the material is amorphous, we won't have a marker to use to determine if a phase transformation if any has occurred, i.e. no crystalline to amorphous transition as we get with crystalline materials).

- i. Pursuing reduced load to achieve ductile response (with blunt edge).
- ii. Use of a range of loads for the knife/chisel edge to produce ductile response.
- iii. Other materials: nothing to report. See 4 O above.
- iv. Looking at scribe direction effects for S.C Si.

#### T. Machining Simulations

- i. Testing of pressure sensitive model complete (by Satya), will write up paper of results.
- ii. 3-D simulations (of scratching) Lei and Satya have performed some initial simulations. Will pursue simulating the scratching of Si.

Note: There is another model available to test, it's referred to as a "damage model", Satya at UNCC is working with TWS to test this model for use with SiN. TWS has performed some simulations and Satya will confirm these results.

#### U. Machining: SiN

- i. Fracture specimens sent to Cat in early Jan.
- ii. Satya and Ronnie Fesperman at UNCC will conduct cutting tests with various rake angles (single crystal and polycrystal diamond tools) and cutting fluids on the GS-44 disks.

V. Induction Heating: Nawaz is completing the assembly of the apparatus and will send it to the induction heating company to have the balance of system installed. A diamond tip was mounted on the end of a glass rod to perform the scratches with. The induction heating company will perform the preliminary testing for us (proof of concept).

5. Web site: Jennifer

- A. Everyone should update his or her personal web pages.
- B. Everyone should provide Jennifer with material to put under the Research (results) page. This is where John hopes to be able to obtain useful information for the annual report due in March (see item 6 below).

Statements, paragraphs, short descriptions with pictures would be useful (this does not have to include results, work in progress is ok too).

6. **Annual report: Due March 31**

We need to beef up the web site so that John can use it to find the information needed for the annual report. Everyone should make sure their information is complete/accurate and please post as much detail about the progress of your research as possible (pictures, short statements, etc. would be very helpful). Please make this a top priority to guarantee a successful annual review and continued funding.

7. Future Meetings: None planned until the Aug. workshop.

8. Other/new business:

Bob suggested that we need to put an effort out towards publications (all agreed). We should all be looking for opportunities to target our efforts (abstracts and papers). One suggestion is that we could all focus on a particular conference to present at.

Possibilities include:

- A. American Ceramic Society
- B. MRS Ceramic symposium
- C. ASPE
- D. Others?