Summary of NSF-FRG Meeting, 10-18-2002

I. Information

A. Our next meeting is scheduled for **January 10, 2003**, at UNC Charlotte. We will concentrate on the program plan and specific research issues (no presentations planned).

B. If other groups (UNCC and NCSU) want or need access to ORNL-SHaRE facilities (**TEM**), they can coordinate with George to possibly add students to his proposal.

C. The **workshop** is scheduled for Aug. 20-23, 2003 at UNC Charlotte (half day on wed. and fri.).

1. John will put together a plan/program and distribute to the group.
2. We can review this at the January meeting.
3. If you have anybody you would like to invite as a speaker please suggest a name and topic (Gogotsi, Gilman, …).
4. John will invite the NSF program manager (L. Madsen).

D. John will be leaving for Japan in late Jan. and returning in late July.

1. Dr. Jim Miller at UNC Charlotte will be working with Lei Dong during this time.
2. The group can schedule another meeting sometime between Jan. and July if they feel this is necessary.

II. Decisions

A. We will hold off on work on Germanium for the time being.
B. George will pursue the in-situ x-ray work with ORNL.
C. John will pursue scratching with fluids, but George will hold off on nanoindentations with fluid (we will reassess at the January meeting). John and George will work together to obtain TEM cross sections of scratches made with fluids to evaluate the depth of the transformed layer. Current thinking is that the fluids inhibit the high pressure phase transformation (chemo-electro-mechano effect) and this should result in a reduced amorphous layer after scratching.

III. Follow-up

A. Last Meeting

1. Bob to check with Cree about availability of SiC wafers
   a. scrap or samples
   b. purchase new wafers
2. Bob to investigate micro-Raman (literature) of high pressure phases of semiconductors and ceramics
3. Ron to pursue source of Ge wafers.
4. George and Bob will pursue (boron) doping of diamond for electrical conductivity tests (see III.B.9)

B. New Items

1. Everyone should supply Jennifer with materials to be included on the web site (papers, pictures, links, etc.) Please note whether this is to be password protected or not.

2. Bob and George will discuss/evaluate and make recommendations about the micro-Raman during nanoindentation (in-situ). Bob to lead this effort, i.e. coordinate with George.

   A. Explore collaboration with others; possibly Y. Gogotsi, ORNL-HTML, others?

   B. Possibly write a separate proposal for this work, or discuss with program manager about extending our current project a fourth year (with additional budget) to include this work.

   C. Note (JP): It might be useful to do this with Si3N4, as there is already much effort on Si (Gogotsi, et al.) and NSF would be more interested.

3. George and Jae-il will add SiC to their nanoindentation literature survey.

4. SiC indent, scratch

   A. George will perform some preliminary indents on the SiC (6H polytype) that Jennifer provided.

   B. John will attempt to scratch some of the same SiC (it may be too small to work with).

   C. Note: John could attempt using the nanocuts device to cut SiC.

5. George will discuss possibility of SiC collaboration with Paul Bekker at ORNL (also NIST?).

6. Bob will look at crystal structure of Si3N4 wafers (x-ray and Raman). Bob thinks they are probably amorphous (the tech. at UNCC who had the wafers made thinks they are probably polycrystalline as they were grown on a Si substrate).

7. Germanium

   a. Ron will check on source for Ge wafers (Eagle Pricher?)
   Univ. Wafers another source.

   b. Bob could grow 1000 Angstrom thick thin film single crystal (if necessary)

   c. Could look at other alloys, such as SiGe

8. Other Materials of potential interest (no decisions were made about pursuing these materials)

   a. Al(O2)TiC:

i. Ron has some material and gave John a sample (it appears the material behaves in a ductile fashion, and exhibits a ductile-brittle transition).

ii. 3M and Seagate are both interested in this material as indicated in a recent visit by John (potential industrial collaborator)

b. Sapphire: George has samples (they cost about $75-100 each).

c. AlN: Bob has some material.

9. George has source of vanadium carbide for indenters for electrical conductivity tests (see III.A.4) .

10. Jennifer will add password secure area to web site.

11. George gave Bob some indented Si to look at.

12. George has single crystal Si3N4, and he will perform some indents. George will follow up with sources of this material (ORNL-NIST, and German organization).

13. Indenter geometry.

A. George has a range of spherical indenters available: 1, 3, 10, 30, and 100 micrometers, for indentation experiments.

B. George also has indenters with various included angles (sharp and blunt)

C. John will pursue purchasing scratch tips of 2, 5, and 10 micrometer radius.

14. John to send Jennifer previous work on Raman of silicon nitride (identify peaks). Jennifer and Bob to investigate silicon nitride spectrum.

15. Jennifer will look at the silicon nitride samples (bars and chips) that John sent previously

IV: Discussion

A. High pressure phases of Si3N4 (George)

1. George presented evidence of cubic silicon nitride formed at high temp. and press. Apparently these phases are metastable and persist at room conditions

2. John indicated that these high temp. and press. could exist during machining (based upon the simulation results). The back transformed material appears amorphous (from TEM and Raman); no cubic phase noticed.

3. The formation path and reaction rates are different in 1 and 2 above, which probably accounts for the differences.

4. High Temperature Experiments:

a. High temperature in-situ Raman could help identify these high pressure phases. (We have not talked about high temperature indentations.)

b. Lei may be able to generate high temperatures with the IR or electrical heating system.

B. Raman of Silicon Nitride (Jennifer)

1. The micro Raman spot size is 2 micrometer; this should be adequate for seeing indentations.
2. Note: John and Michael Lance at ORNL-HTML used the 729 peak in their previous silicon nitride work (see the web site paper on Phase Transformations ...). We also saw peaks at 862, 926, 937, and 1045.

C. Scribing (5 g, 50 mN) (Ron)

1. Ron will correlate the scribing - bending results to existing models and indentation work.
2. Ron can use the bending results/analysis to determine the ductile-brittle transition (in silicon)
3. Future Work.
    a. Repeat for silicon nitride (identify ductile-brittle transition).
        i. wafers
        ii. disks
    b. Reduce load to determine ductile response with blunt edge.
    c. Extend work to lower and higher loads for knife/chisel edge.
    d. Consider extending to other materials: Sapphire, TiC, Al(O2)TiC, etc.
    e. Evaluate scribe direction (<110>, etc.) for single crystal (Si).

D. Electrical Heating during Scratching (Lei)

1. Initial results (see paper on web site) show electrical resistance drops (significantly) during scratching (similar to Georges results for indentation).
2. Current range is: 1 micro amp to 1 amp (load range 10 mN to 100 mN)
    a. At the high currents the gold contacts melt.
        i. Bob mentioned that gold melts at 370 °C on silicon.
        ii. Gold is deposited on chrome (don't know this combined effect)
3. Lei will be imaging scratches with AFM and SEM.
    a. Lei will correlate depth of plastic deformation with current and load.
4. Speed of scratch can be controlled from micrometers/sec to 3 mm/sec.

E. IR detection (Lei)

1. The IR equipment has been assembled.
    a. We get a good strong signal through silicon.
    b. Lei will look at silicon nitride next.
    c. We attempted to image the IR beam with an IR camera, but were unsuccessful (resolution not sufficient)
        i. Lei try again using a microscope objective.
2. Lei will be working on integrating the IR system with the scratching apparatus.

3. IR Heating: Lei will pursue IR heating if the above experiment is successful.

F. Simulations Machining of Silicon Nitride (Satya)

1. Simulation results show high pressures, of the order of the materials' hardness, are obtained in the deformed region.
2. For slow speeds, the temperatures are not very high, yet plastic deformation results in both the simulations and experiments.
3. Satya and Lei will experiment with the 3D simulation of scratching (waiting on code from the vendor).
4. The force data for the simulations and experiments compare reasonably well.
5. For the GS 44 the ductile – brittle transition appears to occur over the range of 1 to 5 micrometers, base upon machining experiments.

G. Other Related Work (John)

1. Induction Heating System:
    a. Design is complete
    b. Construction of device started.
2. Working with Matt Davies on temperature measurements during scratching and machining
3. John submitted a separate proposal to NSF-NER for building/adapting an IR detection system to an indenter.
4. John submitted a proposal to NSF to develop a high pressure phase transformation FEM for machining applications.