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

John A. Patten, Western Michigan University,
Professor and Director, Manufacturing Research Center
Lei Dong, PhD Student, UNC Charlotte, Center for Precision Metrology
Jimmie A. Miller, Chief Engineer, UNC Charlotte, Center for Precision Metrology

Keywords: High Pressure Phase Transformation, Silicon, Electrical and Optical measurements, in-situ, scratching

Abstract
Scratching experiments were carried out on silicon wafers (100) with diamond styli of nominal 2, 5, and 10 µm radius. Relevant parameters varied and investigated were: load (10 to 100 mN) and depth of penetration, material hardness, electrical current effects and measured resistance, and speed effects. Direct electrical heating of the metallic high pressure phase of silicon during scratching experiments with currents ranging from 1 mA to 1 A (for resistance heating the material) resulted in substantial thermal softening of the metallized silicon. In addition to the in-situ measurements, post process analysis included AFM and SEM imaging and analysis. For currents above 100 mA (and loads of 50 mN and higher), substantial electrical resistance heating took place resulting in estimated temperatures of 600°C and a corresponding dramatic drop in hardness to about one-half (6 GPa) the nominal ambient conditions value. Direct heating and subsequent softening of the high pressure metallic phase of silicon is proposed as a possible manufacturing augmentation mechanism to reduce tool wear and increase productivity for silicon processing. Further testing with infrared-optical heating will be discussed and reviewed.

1. Introduction

The scratching tests simulate the cutting, grinding and polishing processes. Previous work (Hirata, 2002) demonstrated the feasibility of measuring electrical resistance changes during scratching. The basic idea of the current research is to generate heat and in-situ thermal the metallic soften high pressure phase. This appears to be a very promising way to machine brittle materials in the ductile regime. It is a well-known fact (G.M.Pharr, 1992) that semiconductor materials have an increased conductivity when undergoing a high pressure phase transformation to a metallic state. If a current passes through the metallized material, there will be heat generated within the material (I^2R). As the current increases from micro-amps to mili-amps and even to 1 amp, the heat generated will increase and thus the temperature will rise. With the heat generated by the electrical current, the silicon can be softened and a lower hardness of the material can be obtained. The lower hardness will subsequently result in less tool wear during machining.

In accordance with the above idea, two experiments have been carried out. They are both contact preloaded scratching tests. Scratching is a close simulation to the real cutting, grinding and polishing processes. Unlike one-dimensional indentation, scratching is two dimensional. The first experiment is called scratching with variable currents and the second is infrared detecting and heating system. In the first experiment, a wide range of currents have been tried during scratching with diamond tips. The depth and width of the scratching groove at different current values have been measured and compared to determine the thermal softening effect. The second experiment uses infrared source to detect the high pressure phase transformation and to potentially generate heat to soften the material, i.e. IR laser.

2. Scratching with variable currents

2.1 Experimental setup

Scratching tests are carried out on a silicon wafer with gold patterns on the surface. The silicon wafer is single crystal n type with orientation of (100). A layer of chromium and gold pad with thickness of 3000 angstroms is vacuum deposited. The gold is used for the electrical connection only. Scratching direction is [011]. The stylus is mounted onto a profilometer. Forward scratching speed is 0.305mm/sec and backward speed is 1.5 mm/sec. Scratching length is about 700 µm. Contact preload ranges from 10mN to 100mN. The electrical resistance is measured across the scratch (gap), during scratching. Diamond tips used are of nominal 2, 5, and 10 µm radius. Figure 1A is the diagram of scratching movement.

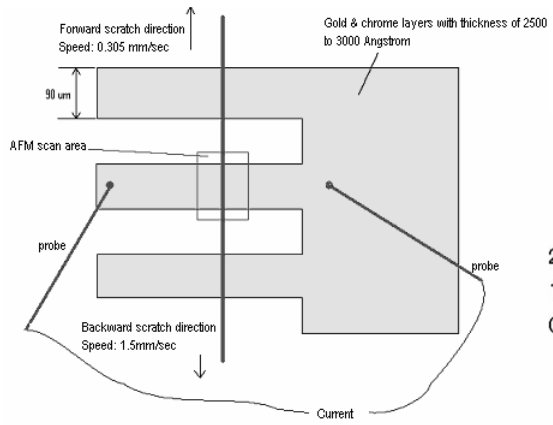

Figure 1A: Diagram of scratching test

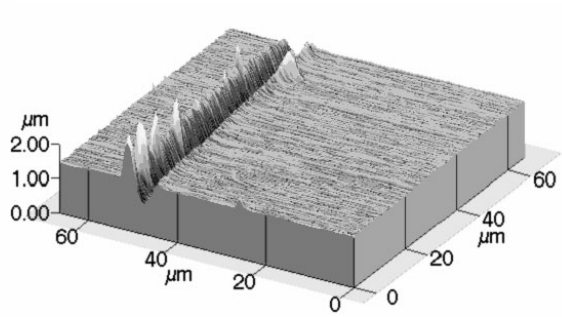

Figure 1B: AFM Image of scratch

2.2 Experiment results

Weights from 10mN to 100mN have been used for the scratching test. We found that a suitable result obtained by using a 5 um radius diamond tip and 5 to 7 gram weight. A smaller tip is very easy to break, while a larger tip needs many scratches to remove the gold layer. Two different circuits have been tried. One of them provides a constant current and the other provides a constant voltage between the two probes.

Test one: constant current circuit

A circuit is designed to provide constant current with the range from 1 micro ampere to 1 ampere. Scratching groove sizes are measured on AFM for different current values. The results, as shown in figure 2, indicate a trend that the higher the current, the larger the groove depth and width. A plausible explanation for this effect is that electrical heat softens the silicon resulting in plastic deformation.

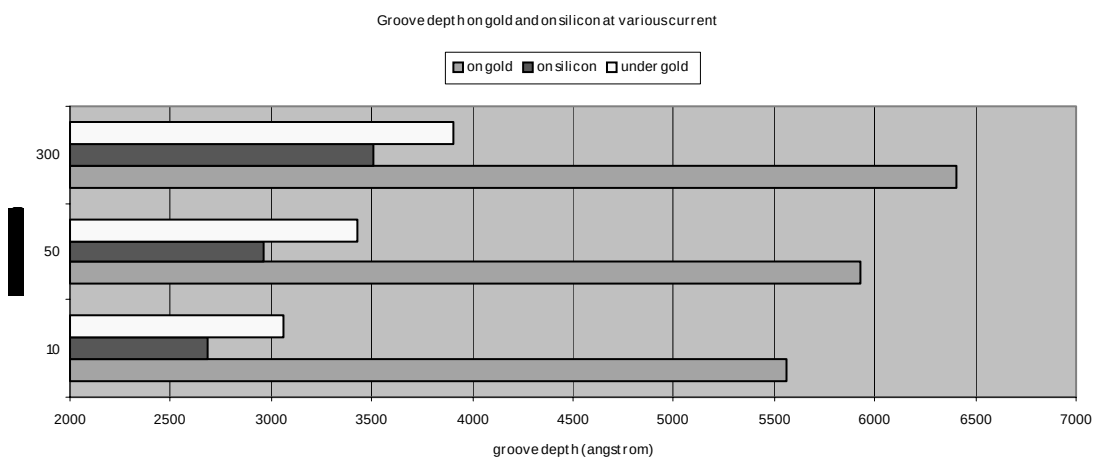

Figure 2: Depth of scratch, on gold and silicon, at various currents (5 gram load with total of 15 scratches)

Test two: V-I circuit (constant voltage)
A constant voltage is applied and a much higher current (approx. 0.65 amps, compared to the max current of 300 mA in the above experiment) is obtained compared to test one, with the result as shown in figure 3. In both test one and two, the probes are set only at the middle gold finger. Therefore current is mainly going through the middle finger. As we can see from figure 3, on the middle finger we have the largest depth and width of the scratch, suggesting that the current produced greater deformation, presumably due to the thermal heating and softening.

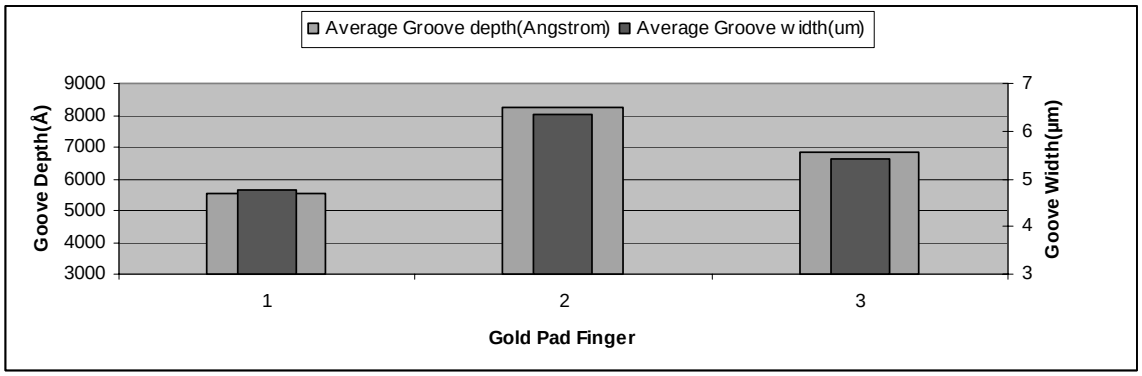

Figure 3: Scratch groove depth and width, with and without current (5 gram load with total of 15 scratches)

3. Infrared detecting and heating system

3.1 Infrared Detecting System setup
A 10 µm diamond tip is attached to the end of a fiber infrared laser (wavelength of 1300 nm, 8mW power), as shown in figure 4. The fiber (2.5mm diameter) with diamond tip is mounted on a profilometer to generate the scratches. As diamond is transparent to infrared light, the laser's infrared light from the fiber end, passes through the diamond tip and into the wafer. With preloaded weight, the diamond tip scratches on pure silicon wafer with crystal orientation (111). Under the wafer there is a 3mm diameter InGaAs detector to measure the transmitted signal. The experimental set up is shown in figure 5.

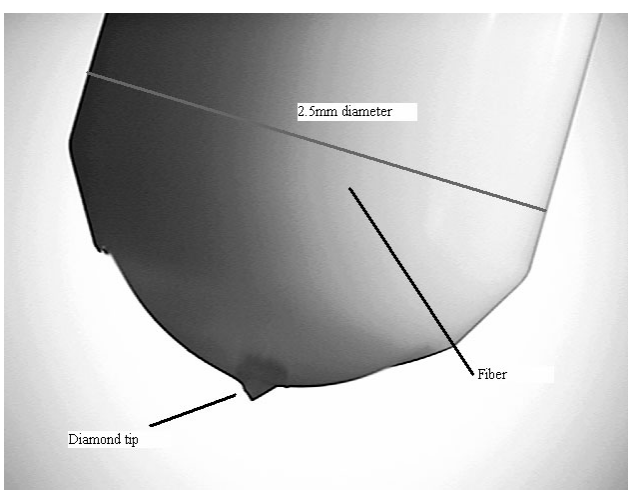

Figure 4 Fiber end with diamond tip (before coating)

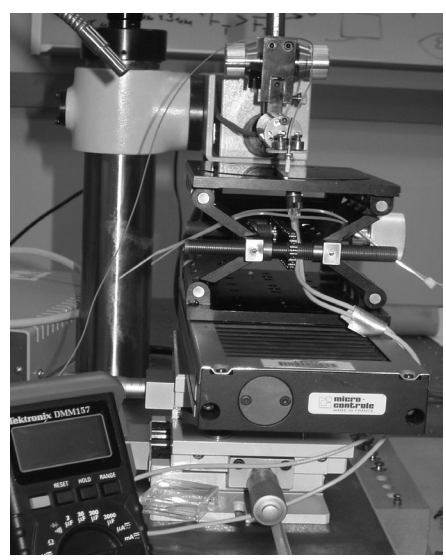

Figure 5: System set up

3.2 Experiment results

A layer (3000 Angstrom) of gold is evenly vacuum deposited on the surface of the fiber end to block the infrared light. When the tip starts scratching the gold on the tip is removed. This insures the infrared light only emits from the very end of the tip. If during scratching a metallic phase is generated, then it will block some of the infrared light and we can detect this change as metals are not transparent in the IR. The scratching length is about 4 mm, which spans across the 3mm diameter detector. Voltage readings with and without preload are compared. Loaded scratching has a lower voltage value, which is proportional to the transmitted IR. Unlike the electrical heating experiment, this test is for a single scratch at a speed of 0.1mm/sec. Figure 6 shows the results with 40mN load. The data clearly indicates a change in the transmitted IR (reduction) due to the applied load, possibly due to the lower transmittance of metallized silicon.

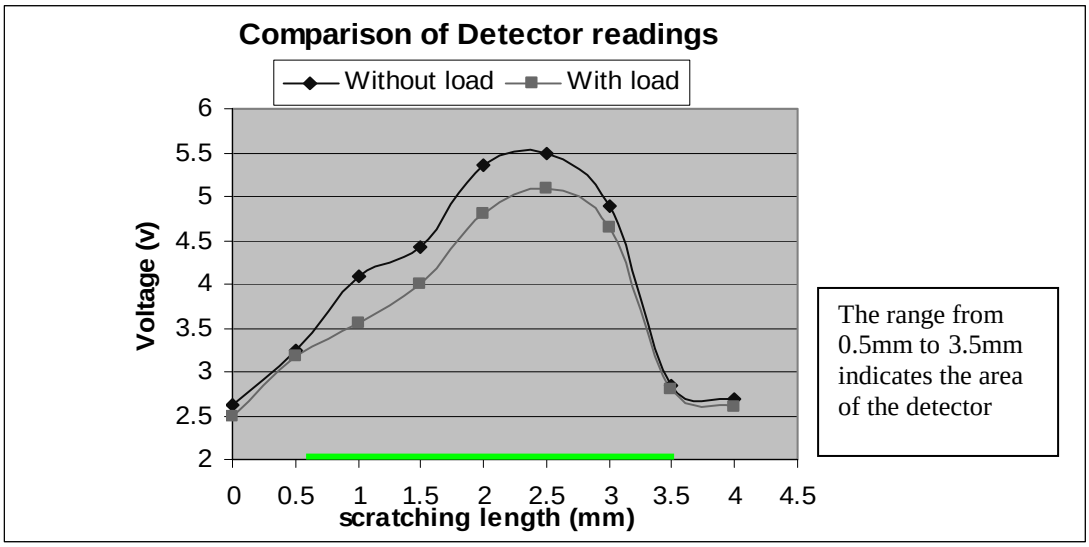

Figure 6: Scratching with and without preload

3.3 Future work

Based upon the success of detecting the high pressure (metallic) phase transformation in-situ via infrared, the next step is to design a laser system to heat the material. If this can be done, it should be a more practical way to carry out ductile-regime machining.

Reference

[1] G.M. Pharr, W.C. Oliver, R.F. Cook, P.D. Kirchner, M.C. Kroll, T.R. Dinger, and D.R. Clarke, Electrical Resistance of Metallic Contacts on Silicon and Germanium during Indentation, Journal of Materials Research 7, pp. 961-972 (1992).

[2] John J. Gilman, Material Science and Engineering, A287 (2000)272-275.

[3] A.B. Mann, Philosophical Magazine A, 2002, Vol.82, No.10, 1921-1929

[4] Tom Juliano, Yury Gogotsi, J.Mater.Res. Vol. 18, No.5, May 2003

[5] Nobukastu Hirata, Daniel Abbott, John A. Patten, Robert J. Hocken, "Measurements of the electrical conductivity of silicon during scratching: An in-situ measure of the high pressure phase transformation", http://www.micro.physics.ncsu.edu, 2002.