High Pressure Phase Transformation and Ductility in Diamond Turned Single Crystal Silicon

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1.0 Introduction
It has been well documented that single crystal silicon and germanium undergo phase transformation with the generation of high pressure on the surface by contact processes such as micro-indentation, scribing, and single point diamond turning. Investigations of the transformation region created report phase transformation from diamond cubic (dc) structure to the amorphous structure. The transformation to the metallic beta-tin phase is thought be responsible for anomalous plastic flow behavior without fracture seen in contact loading processes. The beta-tin phase is not generally seen at room temperature or pressure but is rather identified by an amorphous phase or R8/BC8 crystal structure seen after material unloading, depending on experimental conditions. [1]

The implication of the ductile behavior is, that using careful machining conditions, single crystal silicon may be ground or diamond turned in the ductile regime. Ductile machining will reduce cracks and dislocations generated by machining on the surface and subsurface regions that can reduce the mechanical integrity and lower the operation lifetime of the components. The mechanisms governing the material removal (a combination of microcracking, dislocation motion, and structure change- HPPT) in ductile-regime turning are not well understood. The work presented here is mainly focused on the appearance of amorphization (structure change) in a ductile-turned region. Secondary focus is placed on the characterization of surface finish and cutting behavior as a function of crystallographic orientation and cutting direction, feed rate, and rake angle.

2.0 Experimental
A Rank Pneumo ASG 2500 Diamond Turning Machine is used to turn various orientations of single crystal silicon wafers at room temperature as a function of feed rate (1-15 um/rev) and tool rake angle (-30° and -45°) with a large radius (3mm) round-nosed tool.

Characterization of the machined surface was accomplished with Macro-Raman spectroscopy done at room temperature using an ISA U-1000 scanning monochromator. Raman excitation was done with the 514.5 nm line of an Argon-ion laser, with a spot size of approximately 1mm in diameter. Raman spectra were taken in the 200-600 cm⁻¹ range which contains the characteristic peaks normally associated with crystalline, amorphous, and various metastable crystalline phases seen in other research. A spectral resolution of ≈ 4 cm⁻¹ was utilized, and the laser power was ≈ 5 mW.

Surface finishes of the ductile-turned regions were measured using a Zygo New View 5600 white light interferometer. MetroPro, the software suite used to analyze the interferometer data, reports a measure of surface roughness by the root mean square (RMS) parameter and peak to valley (PV) values. Measurements were taken for from the center outward in regions within and outside the predicted fracture pattern area (further explained in Results section.)

3.0 Results and Discussion
Optical quality, low RMS surfaces (1-10 nm) were created by single point diamond turning single crystal Si wafers in the ductile regime. Ductile regime material removal was evident by the absence of fracture damage and repeated feed marks in the surface as well as generation of continuous chips. Increasing feed rate seemed to create an increase in surface roughness as shown in Figure 1.
Feed rate seemed to be the limiting factor for generating a damage free surface. At increasing feed rates, certain directions on a wafer face initiated fracture at before others, creating symmetric damage patterns on the surface. The more negative rake angle tool tended to suppress the onset of fracture in the favored direction allowing machining at higher feed rates. The damage patterns for the wafer orientations tested ((100), and (111)) were very similar to those found in previously and were explained qualitatively by an orientation stress model used in that research. [2] These are shown in Figure 2. For surfaces that were ductile turned with similar parameters of tool rake and low feed rate, there appeared to be no difference in surface roughness and profile shape with changing cutting directions.

The RMS (and PV) value of ductile turned surfaces was limited by the effect of the condition of the tool tip. While tips were freshly sharpened, experimental preparation (i.e. part touch off and preliminary facing cuts) perhaps dulled the tips enough to result in non-ideal material removal (deformation under the tool and elastic spring back.) This was evident by higher-than-theorized PV values, and machining profiles that did not fit theoretical shape (but were similar to realistic expectations) as measured by interferometry techniques. Additionally, machining runs on Si were subject to tip wear and damage considerations as evident by the repeated patterns of tip damage features into the part surface. Despite these various problems, surfaces turned within the ductile regime had RMS values in the range of <1 nm to 10 nm increasing with feed rate.

Raman measurements of the ductile turned surfaces for various machining conditions indicated what is thought to be the presence of a near-surface amorphous layer. The presence of this layer is thought to be a remnant of a rapid back transformation from the beta-tin metallic phase. The most
A noticeable trend for each of the parameters tested is the drastic decrease in amorphous intensity coupled with an increase in dc peak intensity when switching feed rates from 1 to 5 μm/rev. While the Raman measurements did not always clearly indicate the existence of this layer for samples machined at 5 μm/rev, closer direct evidence difference in amorphous signal intensity between crystal orientations, cutting direction, or rake angle as seen in Figure 3.

**Figures 3.** Raman spectra for various conditions taken on ductile turned surfaces showing heavy dependence of amorphous/dc intensity ratio on feed rate but little else.

**Figure 4.** TEM image of Silicon chip generated during ductile regime turning and corresponding electron diffraction pattern showing crystalline structure of chip.
TEM results shown in Figure 4 indicated that the chips have the Si dc crystal structure. Si chips displayed a morphology of smaller (1-2 um) clustered chips. Despite the amorphous nature of the near surface machine regions, TEM failed to show any direct evidence for amorphization within the chips. It is thought that intense local heating during machining may cause recrystallization of the amorphized material as the thin chips lack cannot dissipate heat quickly.

4.0 Conclusions
Low RMS surfaces were achieved by ductile regime turning with high negative rake, large radius round nosed tools for various crystallographic orientations of single crystal silicon. Symmetric fracture patterns resulting from high feed rate machining were the limiting factor for ductile regime machining. Surface characteristics such as roughness and profile seemed to show little dependence on cutting direction within the ductile regime. Raman measurements indicated the presence of a near surface amorphized layer for regions where ductile turning occurred. Based on the relative intensity of the amorphous peak, the Raman scans indicated that lower feed rates produce more amorphous phase, ie., a thicker surface layer. This layer thought to be the remnant of a back transformation from the metallic beta-tin phase, which could be responsible for the ductile behavior during machining. TEM analysis of the chips showed them to be crystalline in nature, indicating rapid recrystallization of the chips during machining.

References