CHARACTERIZING RESIDUAL STRESS IN SCRIBES ON SILICON USING DEFLECTION MEASUREMENTS

Supplemented With Raman Characterization

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1.0 INTRODUCTION

It has been well documented that single crystal silicon undergoes phase transformation with the generation of high pressure on the surface by micro-indentation and diamond point scribing. Investigations of the transformation region created report phase transformation from diamond cubic (dc) structure to beta-tin phase. The transformation to the metallic beta-tin phase is thought be responsible for anomalous plastic flow behavior without fracture seen in contact loading experiments. 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 unloading depending on experimental conditions. 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. It is hoped that 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.

A scribing apparatus was used to translate a diamond tip across a surface at a constant rate for different loads. With the appropriate conditions, the diamond tip is used to produce scribes on single crystal silicon samples generating brittle-to-ductile transformation of the material beneath the scribe. This anomalous plastic flow has been attributed to a high pressure phase transformation to a metallic β-tin phase.[1] The residual stresses within the scribe traces that result from the elastic-plastic constraint of the displaced material create a bending distortion in a flat-plate geometry wafer sample. The deflection is measured and extracted using optical interferometery techniques. The angle of deflection in the flat plate geometry correlates to the residual stresses created using a bend effect model developed for previous scribing research. Stress approximations will be coupled with Raman, SEM, and AFM characterization to aid in understanding the nature of the material deformation within the scribe zone.

2.0 PROJECT DETAILS

This investigation entails collecting bend data as well as Raman measurements for (100) and (111) crystallographic orientations of silicon, scribing with a Dynatex tip. Scribing along the <100> and <110> will be studied for the (100) orientation. <110> and <112> directions will be studied for the (111) orientation. The deflection angle data will then be correlated to the residual stresses created by scribing using a bend effect model. Indenting was also performed using Vickers and Dynatex tips to study the indentation regions for phase transformation with Raman spectroscopy.

2.1 EXPERIMENTAL SETUP

A Zwick micro hardness tester is fitted with a Dynatex diamond tip (Figure 1a) in a custom holder. A motorized stage is used to create translation at a constant rate of 0.250 mm per second. A holder was designed to hold the cutting edge of the diamond tip at an angle of 3.3 degrees from the sample surface as shown in Figure 1b. The desired load is placed on the loading sleeve and the tip is positioned and contacted to the surface of the sample. The stage is translated with relation to the diamond tip producing the scribe. This setup can achieve repeatable loading below 50 mN and with relatively easy tip alignment in the translation direction.

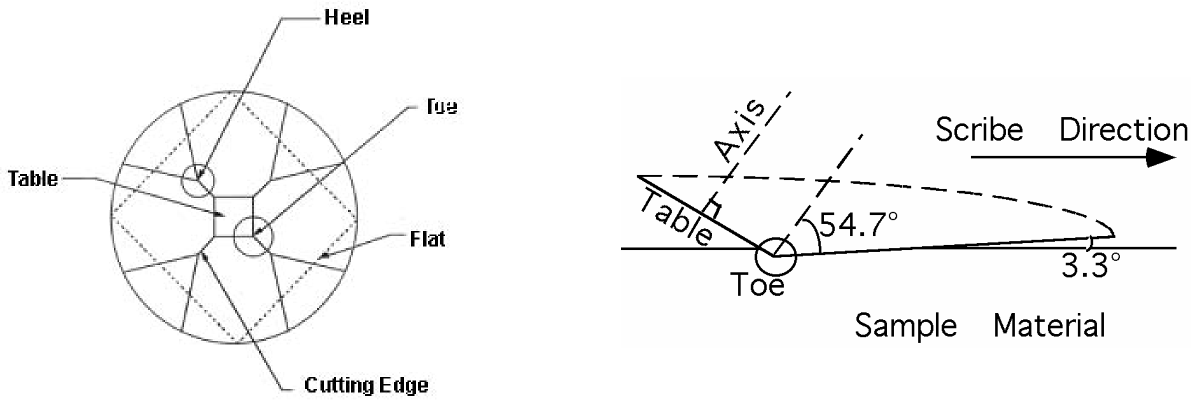

Figure 1. (a) Schematic of Dynatex Tip (b.) Geometry of Tip Relative to Surface [2]

Topographic data is collected for a 1cm x 2cm x 525 μm sample using a ZygoGPI Laser interferometer before and after scribing. Multiple, evenly spaced (50-100 μm) scribes are placed on the surface to generate measurable deflection (the effects of multiple scribes are additive to the bend) and to produce a more statistically significant measurement than that of a single scribe. The resulting profile created is the net bending effect, this is used to approximate the angle of deflection from the initial state.

Characterization of the scribe/indent region was accomplished with Micro-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 3 to 4 μm 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. [1]

2.2 EXPERIMENTAL RESULTS

Scribing with the Dynatex tip across a 2 cm x 1 cm x 525 mm plate of single crystal silicon yields a measurable deflection on the order of tenths of microns. This behavior is seen in of each of the crystallographic direction studied for both orientations if the load applied is enough to produce cutting action in the material.
Figure 3 shows two SEM images distinguishing between the two general responses. The image on the left is scribed on (100) silicon in the [011] direction at 5g load and appears to have no fracture around the scribe and cutting action appears plastic. The plastically displaced material, whether driven by the β-tin metallic phase or not, is pushed by the tip into the surrounding elastic material creating residual stress and deflection in the sample geometry. The image on the right was done at 30g load and shows a large amount of fracture. The action of the diamond indenter may create localized stresses at the tip which exceed the fracture limit, but it is unclear how the transformation region affects this. One of the limitations inherent is that with elastic fracture, removal of the plastically deformed regions may be possible and the bend effect model will not accurately give measure of residual stress for scribes. The bend effect will be reduced relative to a scribe with no fracture.

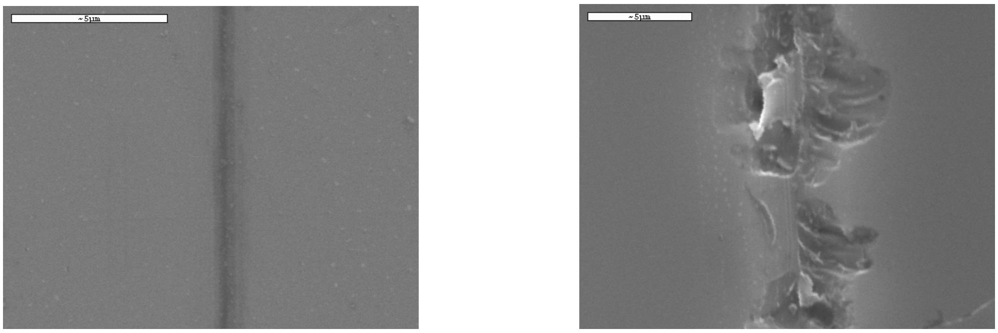

Figure 3. SEM images of scribes (100)[011] at 5g and 30g respectively.

Scribing between 1-20g in the various crystallographic directions with a sharpened tip shows an approximately linear relationship between the normal load applied to the tip and the deflection generated by the residual stresses. Figure 4 shows this trend for (100)[011] and (111)[-1-12], respectively.

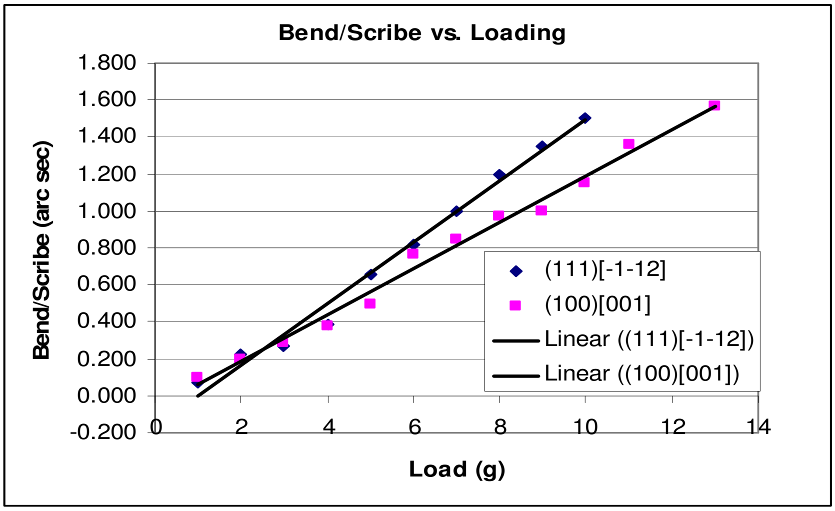

Figure 4. Bend angle generated per scribe versus load applied at tip.

The scribes for the data presented in Figure 4 appear to be fracture free along the scribe traces. These two directions represented the best cutting directions seen for their respective orientations. The linear trend could indicate a correlation between the load applied and the amount of plastic deformation occurring beneath the scribe. Experiments for repeatability of these results are currently underway. Other combinations of crystallographic direction and orientation are to be tested.

Another point of interest to note is the difference between results previously obtained during diamond point turning (DPT) of silicon wafers and those presented here from scribing with the Dynatex tip. Blackley et al. showed that for DPT, the [100] direction on (100) produced more favorable cutting behavior (fracture free, plastically generated) than the [011] direction [3.] For scribing with the Dynatex tip the opposite seems to be true. Figure 5 shows bend angle generated per scribe versus load applied at the tip. Optical microscopy revealed the onset of fracture for [011] to occur at 15 g with the Dynatex tip. The fracture threshold for the [100] direction on the other hand occurred around 7.5 g. The data in Figure 5 reflects the unpredictability of using deflection measurements for the [100] direction above 5 g in the prediction of stresses, while the trend seems to be linear up until the fracture point for the [011] direction. This is attributed to fracture that removes plastically deformed regions, thereby reducing the residual stresses and bend angles for the [100] direction.

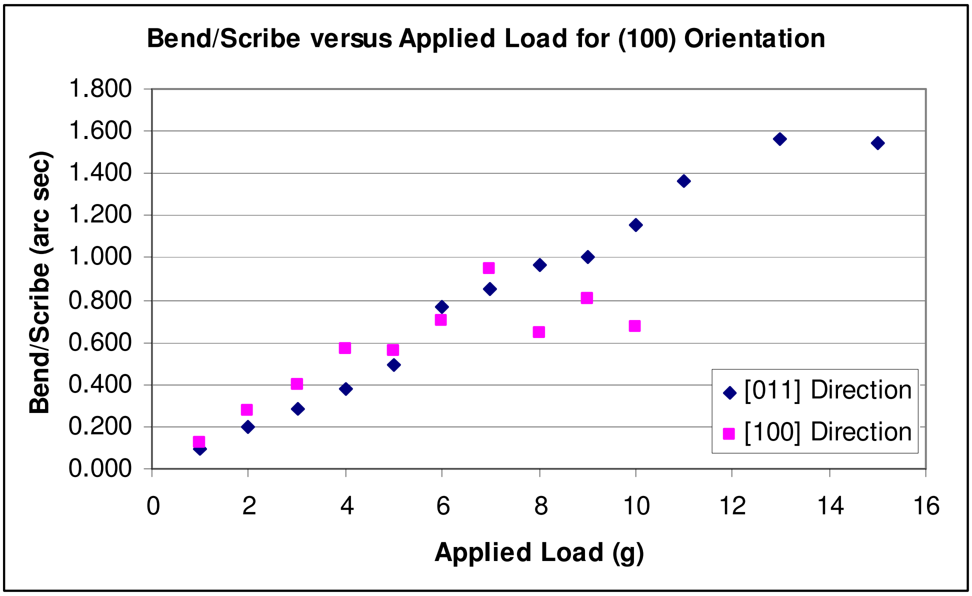

Figure 5. Bend angle generated per scribe versus load applied at tip.

Raman was performed on both Vickers scribes (1-15g) and Dynatex scribes (2-40g) with no apparent sign of peaks corresponding to the phases commonly seen after scratching tests. It is not known to the authors at this point why this has occurred. It could be possible that the phase transformation may not be occurring with the Dynatex tip due to the nature of the geometry tip and that the plastic displacement is by some other mechanism, for example, dislocations. It is also possible that the tip may be removing transformed material as it passes. The detection proficiency of the Raman unit may be responsible as well. If the penetration depth of the laser is much greater than that of the transformation zone, or if the laser spot size is large compared to the scribe width, the Raman spectra for the transformation product may not be detected. Sufficient data is not yet available to draw a conclusion for scribing experiments. Indenting experiments are currently underway to test the Raman unit in indenting conditions already known to generate phase transformation. [1]

3.0 CONCLUSIONS

The bend angle generated due to scribing is on order of arc sec per scribe depending on load applied to the tip. The deflection appears to increase linearly for scribes with minimal fracture, but hasn't shown to be repeatable for specific values of bend angle with similar applied loads. For worn tips bend angle drops dramatically (an order of magnitude) at certain point "threshold." Raman measurements yielded no sign of phase transformation within the scribe regions for both Dynatex and Vickers geometries, contrary to what was anticipated by the authors. This is still being investigated. Work continues with using dead-weight loading to create scribe traces with better experimental procedure that aim to improve current data and repeatability. The bend effect model used to correlate sample deflection to an estimate of residual stress within the scribe trace is being tailored to this investigation. Indenting experiments are currently underway to reproduce the experimental findings of others [1] and gain better insight into the lack of Raman data indicating the expected phase transformations within the scribe region. Under development is a piezo-actuator driven indenting apparatus. This will aid in performing controlled indents and load control during scribing to test the effects of loading/unloading rate of the tip into the surface.

REFERENCES

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