THE CRYSTALLOGRAPHIC DIRECTION INFLUENCE ON THE BRITTLE TO DUCTILE TRANSITION IN DIAMOND TURNING OF SILICON CRYSTAL

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INTRODUCTION

The feasibility of cutting brittle materials under conditions that lead to a ductile regime has driven many efforts to the comprehension of the material removal mechanism involved. Consequently, the suppression of the brittle response is a desirable aspect in the machining of semiconductors crystals.

The plastic behavior of silicon during diamond turning has been attributed to a pressure induced structural transformation from diamond cubic structure to a metallic characteristic [1], which had demonstrated that diamond-cubic silicon transforms to a denser metallic $\beta$-tin structure at room temperature. In silicon, the critical pressure observed to cause the transformation to a $\beta$-tin metallic phase is in the range of 11.3-12.5 GPa and the pressure to cause transformation into the metastable amorphous semiconductor phase, upon unloading, is about 7.5-9.0 GPa [2]. However, the nominal value of Vickers microhardness is different according to the crystallographic direction of the crystal. In (100) monocrystalline silicon, the Vickers microhardness in the [100] and [110] directions are 12.6 GPa and 9.3 GPa, respectively [2].

In the present paper, the dependence of brittle-to-ductile transition on the crystallographic direction in diamond turning of silicon single crystal is investigated. Microindentation and face cutting experiments were conducted in semiconductor single crystals of silicon (001)-oriented in two directions: [100] and [110]. In addition, the cutting conditions were tested in order to study the effect of crystallographic direction on brittle-to-ductile transition when using large feedrates and submicrometer depth of cut.

EXPERIMENTAL DETAILS

Facing cuts were performed on a single crystal Si polished sample. The specimens were in the form of squares (10 x 10 mm) cut from silicon wafers (100) 1-10 Ω.cm type p ($B = 10^{15}$-$10^{16}$ atoms/cm³) of 55 mm diameter and 500 µm thick. A round nose diamond tool with nose radius of 0.766 millimeters, −5 degree rake angle and a 12 degree clearance angle from Contour Fine Tooling® (UK) was used. A Rank-Pneumo™ (Keene, NH, US) ASG 2500 diamond turning machine was used in the tests. A cutting fluid used was a synthetic water soluble oil with the purpose of cooling. The surface roughness of the diamond turned surfaces was examined by a non-contact type surface measurement system, i.e. Wyko NT 1100 (Veeco Metrology Group). The magnification objectives used in the Wyko NT 1100 was 20X, while the field of view was 232µm×305µm respectively. The Vertical-shifting interferometry (VSI) mode was chosen in the experiment. The surface roughness profiles were then plotted by Vision software from Veeco Instruments, Inc. Figure 1 describes the experimental cutting conditions. The crossfeed was inwards.

![Figure 1. Schematic diagram of the cutting conditions applied in the machining tests.](image-url)
Two cutting conditions were chosen: typical feedrate and depth of cut and large feedrate and sub micrometer depth of cut both aiming at achieving ductile material removal. Consequently, at submicrometer depths of cut using sharp diamond cutting edges, microcracking would be hindered to propagate and higher feedrates could be applied without brittle response.

Cyclic Microindentation tests were carried out using a VMHT MET Leica (Leica Mikrosysteme, Gmbh; A-1170, Vienna, Austria) micro indentation machine using a Vickers pyramidal indenter, applying a 150 mN load with dwell time of 15 seconds in cycles of 1, 5, 10 and 15 times and indentation velocity of 60 µm/sec. Microhardness anisotropy measurements were carried out using angular the range of at least 90°, at intervals of 45°. The micro-Raman spectroscopy study was performed with a conventional T64000 Jobin Yvon spectrometer. The 514.5 nm line of an argon ion laser, focused by an optical microscope on a region of about 1 µm² of the tool surface was used to excite the Raman spectra. The laser power was kept low at about 0.6 mW, in order to avoid heating effects. The laser beam was focused in the sample with 100x lens objective.

RESULTS AND DISCUSSION

Figure 2A) and 2B) show micro Raman scattering of the machined surface in both directions, [001] and [110], respectively. Comparing Figure 2A) and 2B), there is an intensity reduction of the crystalline silicon (c- Si) Raman peak at 521.6 cm⁻¹ for direction [001] and [110], respectively and the presence of a broad band centered at about 470 cm⁻¹, can be noticed in both cases, which can be attributed to the optical band of amorphous Si. It is interesting to observe that the increase of intensity can be attributed to the machining in brittle mode with the increase in feedrate in the soft direction, i.e., [110]. In addition, Figures 2A) and 2B) show that crystal peaks shift to the right from the characteristic cubic diamond structure centered in 521.6 cm⁻¹, with the increase in feedrate. This shift is indicative that the machined surface is under compressive residual stress. The value of this stress can be estimated by the formula proposed by Weinstein & Piermarini [3]:

$$\sigma = \sigma_0 + 0.52P \text{ (Kbar)}$$  \hspace{1cm} (1)

where $\sigma$ is the real position of the characteristic crystalline peak, $\sigma_0$ is the characteristic peak of cubic diamond structure of silicon (521.6 cm⁻¹) and $P$ is the stress within the machined surface in Kbar. The values of the residual stresses estimated by means of this formula are shown in Table 1.

The values obtained show very interesting information: the residual stress increase with the feedrate and no surface stress relief can be detected even with the largest feedrate (10.0 µm/rev) in the [001] direction (Fig. 2A) spectrum [c]). The residual stress in direction [110] is always larger than that in [001] direction. This result corroborates with the fact that the capacity of sustain compressive strain should be larger in the softer direction.

Silicon undergoes phase transformation when the pressure reaches the value within the range of 11.3-12.5 GPa along with the fact that in the presence of shear stress, the diamond structure has been found to become unstable at lower pressures (7-9 GPa) [4]. Based upon this it is possible to assert that it is more difficult to reach the necessary pressure to bring about the phase transformation.
transformation in the softer direction, and consequently, to generate ductile response.

**Table 1.** Estimation of residual stresses on the surface after machining in surface direction [110] and [100] according to peak shift (a-e).

<table>
<thead>
<tr>
<th>Cut. cond. (spectra)</th>
<th>Peak Pos. (1/cm)</th>
<th>Res. stress [110] (MPa)</th>
<th>Peak Pos. (1/cm)</th>
<th>Res. stress [100] (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a)</td>
<td>522.84</td>
<td>238.46</td>
<td>522.34</td>
<td>142.31</td>
</tr>
<tr>
<td>b)</td>
<td>522.88</td>
<td>246.15</td>
<td>522.24</td>
<td>142.31</td>
</tr>
<tr>
<td>c)</td>
<td>523.44</td>
<td>353.85</td>
<td>522.24</td>
<td>123.08</td>
</tr>
<tr>
<td>d)</td>
<td>523.33</td>
<td>332.69</td>
<td>522.34</td>
<td>142.31</td>
</tr>
<tr>
<td>e)</td>
<td>523.19</td>
<td>305.77</td>
<td>523.14</td>
<td>296.15</td>
</tr>
</tbody>
</table>

At one hand, since [110] direction presents lower hardness, and machining is a dynamic material deformation process; because of this it is more likely that in this direction, both, ductile and brittle modes, take place simultaneously. On the other hand, [001] direction hardness value is within the range of pressure required to silicon undergoes phase transformation and the machining regime to occur totally ductile as it is shown. This is exemplified by Figure 3. Figures 3 a) and 3 c) show that for the [001] direction the uncut shoulder presents ductile regime for both depths of cut, i.e., 5 µm and 0.5 µm. Figures 3 b) and 3 d) show that for the [110] direction the uncut shoulder depicts brittleness for the same depths of cut. In the harder direction, ductile mode was achieved under two opposite cutting conditions, i.e., $f = 2.5 \mu m/rev$ $dc = 5 \mu m$ (Fig. 3a), and $f = 10 \mu m/rev$ $dc = 0.5 \mu m$ (Fig. 3b). In the softer direction, a damage free surface finished was only achieved with small feed rate ($f = 2.5 \mu m/rev$ $dc = 5 \mu m$ – Fig.3b). Furthermore, in Fig.3 d), the machined surface presents pits within the cutting groove. The brittle response shown in this direction may be attributed to the fact that the larger difference between transition pressure and hardness can be considered the reason why the microcracks were formed below the cut surface line and on the uncut shoulder as well.

Figure 4 a), spectrum a), shows the Raman spectrum of the machined surface in the [001] direction before microindentation. Figures 4a) spectrum b) up to e) show the sequence of Raman spectra of the cyclically indented machined surface, 1x, 5x, 10x and 15x indentation cycles on the [100] direction. The intensity of the Raman peaks at 521 1/cm due to the Si-I formed is increased after the 5th cycle (spectrum c).

**FIGURE 3.** Images made by Optical profiler of the uncut shoulder from the machined silicon (100) surface, a) [110] direction and, b) [100] direction. Feedrate used was 2.5 µm/rev and the nominal depth of cut was kept constant at 5 µm; c) [110] direction and, d) [100] direction.
Feedrate used was 10 µm/rev and the nominal depth of cut was kept constant at 0.5 µm.

In the [100] direction the onset of the formation of new silicon phases were probed after 10 cycles, showing the onset of Si-XII phase formation at 353 cm<sup>-1</sup>, with the decrease in intensity on the characteristic crystalline peak at 521 cm<sup>-1</sup>. When the 15<sup>th</sup> cycle is done (Fig. 4e) the multiple phases present a slight increase in intensity Raman peaks shift. In the Raman spectrum can be identified the following peaks: at 165, 182, 353, 398 cm<sup>-1</sup> from Si-XII; 386, 440, 490 cm<sup>-1</sup>, from Si-III; and at 495 cm<sup>-1</sup> from Si-IV [1]. Figure 4 b) shows a sequence of non-indented, 1, 5, 10 and 15 indentation cycles of machined Si in the [110] direction. From the first up to the tenth indentation cycle (spectra b and d) the only effect is the generation of an amorphous phase along with the crystalline phase (a-Si +c-Si), denounced by the broad band at 470 cm<sup>-1</sup>, showing the peak at 521 cm<sup>-1</sup>, due to the Si-I, in the diamond cubic structure after the 10<sup>th</sup> step. The formation of a multiple phase's state with several different structural phases is only produced after the 15<sup>th</sup> cyclic indentation, as displayed in spectrum e).

4. FINAL CONSIDERATIONS

Summarizing, we have performed a study on the effect of the crystallographic direction on the generation of brittle and ductile mode material removal in silicon crystal. Residual stresses were estimated by means of the results showed that the [110] direction, which is the softer direction, presented the largest values of residual stresses with maximum 350 MPA. The machined surface presented pits in the [110] direction when submicrometer depth of cut and large feed rates were used. The results for [100] presented the opposite response when compared with the [110] direction in both cases. The difference in the brittle and ductile behavior, for different crystallographic directions, was attributed to the difference between microhardness and transition pressure value to induce phase transformation during machining. The harder direction [100] presents microhardness value approximately the same as the transition pressure value of silicon, which undergoes ductile material removal when phase transformation takes place. Since the softer direction [110] has a lower hardness, the difference between the HV and transition pressure value will be larger and then the phase transformation will be more difficult to take place. Consequently, the onset of brittle mode will be more favorable to take place.

FIGURE 4. a) Raman spectra of (001) Si surface successively indented with 150 mN load in the direction [110] a) machined surface, b) Raman spectra of (100) Si successively indented with 150 mN load in the [100] direction.

REFERENCES