

The Effects of Fluids on the Deformation and Ductile-to-Brittle Transition of Silicon

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This research evaluates the effect of fluids on the deformation and ductile to brittle transition of silicon. Scratches were made on a polished silicon wafer surface by using a diamond stylus under increasing loads until the brittle transition was detected. The scratches were drawn while the diamond-silicon point of contact was completely submerged in one of the following fluid environments: air, de-ionized water, acidic chemical mechanical polishing (CMP) slurry or an alkaline CMP slurry. The CMP slurry environments produced a suppression of the brittle transition compared to the air environment resulting in an apparent increase in toughness. Under higher loads, the de-ionized water and both CMP slurries resulted in an apparent increase in hardness compared to air.

Keywords: Silicon, ductile machining, brittle transition, CMP, single-point diamond turning

Introduction

The search for a better understanding of brittle material behavior led to the discovery of a “ductile regime” of operation.¹ Precision machining of brittle materials in the ductile regime is well documented.^{1-3, 5-7} Cutting fluids are often used in silicon processing to achieve favorable results. Water is a cooling agent for high speed, high temperature grinding operations and during single-point diamond turning (SPDT). Chemical mechanical polishing (CMP) uses slurries to mechano-chemically effect the material removal rate. The CMP fluid is thought to soften the silicon surface, thereby allowing ductile material removal. Cutting fluids that promote ductile cutting can also negatively impact the machining process by increasing tool wear.⁴ Cutting fluids and their effect on silicon machining is the focus of this research. Silicon scratching experiments in previous research revealed an “apparent increase in hardness” due to the presence of water and polishing slurries when compared to dry air abrading.⁵ The present research more than doubles the number of data points of the original experiments and offers additional insight to the mechanisms involved with the ductile to brittle transition of silicon.

Experimental Procedure

This experimental procedure involves scratching an N-typed, (100), arsenic doped, 0.005 ohms-micrometer, single crystal polished silicon wafer. The scratches were created in four different fluid environments: air, de-ionized water, Klebesol 30H50 chemical slurry and Klebesol 30N50 chemical slurry. *Table 1* shows comparative data. The scratches were drawn by a profilometer at a constant velocity of 0.5 mm/s. A two-micron radius, diamond-tipped, stylus was used to create the scratch. The scratches were drawn in the $\langle 0\bar{1}1 \rangle$ and $\langle 01\bar{1} \rangle$ directions. Finally, the silicon wafer was imaged in an AFM. A minimum trace area of 40 x 40 microns was used to gather sample data. The scratch topography was then analyzed using two and three-dimensional AFM images.

Table 1 Characteristics of fluids

Fluid Description	Chemical Property	pH Level	Particle	Particle size
Air	N/A	N/A	N/A	N/A
De-ionized water	Neutral	6	N/A	N/A
Slurry 30H50	Acidic	2	Colloidal silica	50 nm
Slurry 30N50	Alkaline	10	Colloidal silica	50 nm

Results

Identification of the ductile material behavior was qualitatively characterized by scratch topography. Evidence of ductile regime scratching was characterized as being continuous, rounded and smooth with pile-up on either side of a well-defined groove. *Figure 1* shows a typical ductile scratch. The dependent variable for this research was scratch depth, which was measured by use of AFM imaging. The ductile-brittle transition in silicon under each of the fluid condition was also determined from the AFM images. Under applied loads of up to 20 mN, all of the fluid environments yielded a ductile response. When comparing the scratch profiles of the air, de-ionized water, and both slurry environments at these loads minimal form/shape variation was noticeable. The distinguishing feature in the resulting geometry at these loads is the scratch depth. The scratch depth, at each applied load, is an indication of the material's hardness.

Discussion

Figure 2 charts the scratch depths of each fluid at 20 and 30 mN loads where ductile deformation occurred. Scratching in air resulted in the most penetration. The CMP slurry did not produce the softening expected, based upon commonly accepted practices. The use of the CMP slurry during scratching resulted in less penetration compared to air under equivalent loads. Scratching the silicon in the slurry environments at loads of 20 mN and 30 mN clearly exhibits a material behavioral change. The depth of penetration, while scratching the silicon under these conditions, is up to 40% less than the depth of penetration of the air and water environments. Resistance to scratch penetration is analogous to material hardness. Both slurry environments induced what may be characterized as an apparent increase in hardness of the silicon surface. The depth and degree of this reaction is not known and is viewed simply as a near surface change. In addition to the slurries being resistive to penetration, they also withstood greater loads before the initiation of the ductile to brittle transition. A 50-mN load was needed to induce the brittle transition in the 30H50 slurry compared to a 30-mN load for air. See *figure 3* for a summary of all brittle transition loads. The load induced ductile to brittle transition is analogous to the material's toughness. Toughness is the ability to withstand a load without fracture. Load can be viewed as an indicator of toughness. CMP slurries produced both a hardening and

toughening effect compared to air. Not only did the slurries resist penetration but also they retarded the onset of the ductile to brittle transition and thus retarded cracking.

The present experiments were not refined enough to show the well-documented phenomenon that machining in water promotes brittle fracture when compared to air⁶. The load increment of 10 mN was too large to differentiate the ductile to brittle transition in air and water. The apparent increase in hardness, in a water environment compared to air environment, is supported by the shallower depths of penetration at higher loads. *Figure 4* shows a progression of silicon scratches from a ductile deformation into an increasingly more brittle zone. *Figure 5* shows the geometric changes in a scratch as the silicon goes through the ductile to brittle transition. The scratch groove is no longer smooth and continuous.

Conclusion

Topographical conditions of scratches drawn on n-type, (100), single-crystal, polished, silicon wafers have been analyzed. Scratches were made on the wafer surface in one of four fluid conditions: air, de-ionized water, acidic polishing slurry or alkaline polishing slurry. The scratches were performed under increasing loads until the ductile to brittle transition occurred. This research measured scratch depths from the purely ductile zone until a definite presence of the brittle transition was observed. The use of CMP slurry, and to a lesser extent water, reduced the deformation of the silicon, at higher loads, compared to the air, i.e. an apparent increase in hardness. The CMP slurries also allowed for higher loads before the onset of brittle fracture, i.e. an apparent increase in toughness.

Acknowledgements

This research was funded in part by a National Science Foundation REU grant and the Ronald E. McNair Post Baccalaureate Achievement Program.

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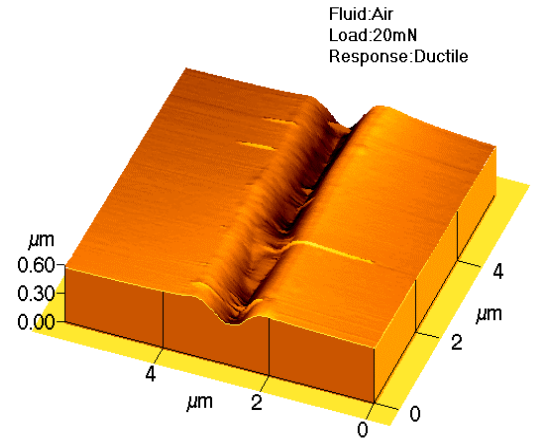
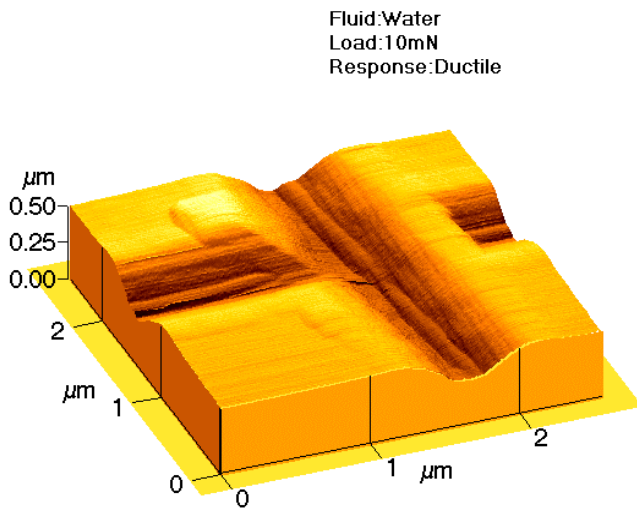


Figure 1 Smooth, rounded, and continuous surface typical of ductile regime scratching

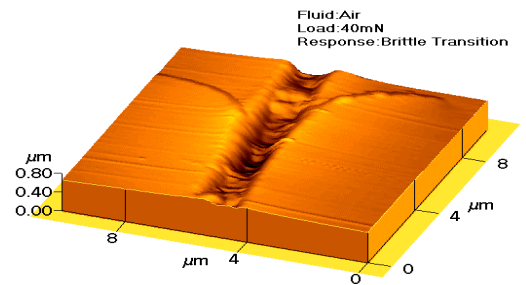
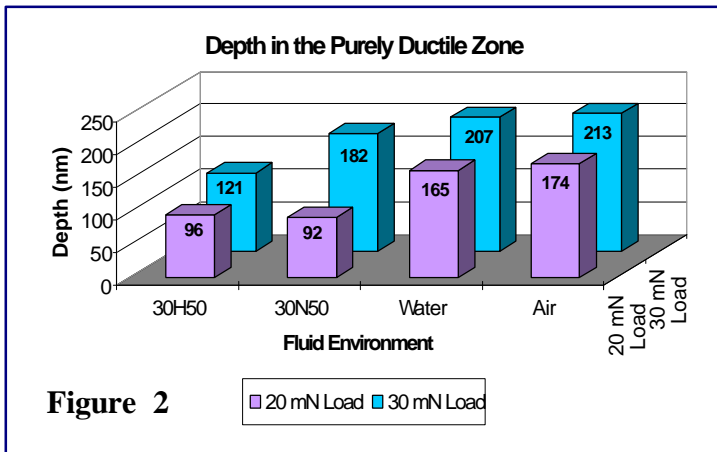
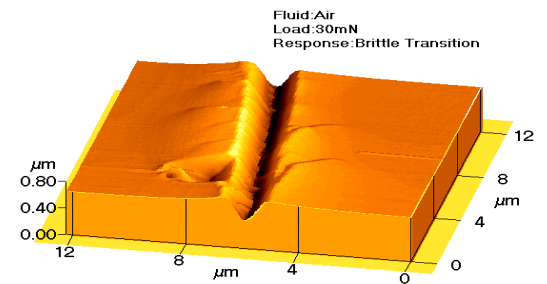


Figure 4 AFM Images showing the change in scratch topography as silicon goes through the ductile to brittle transition.

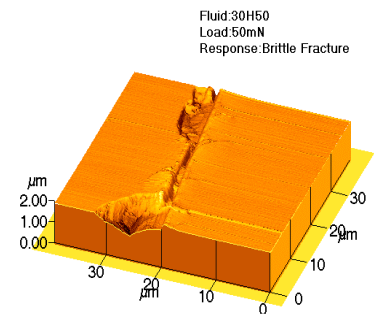
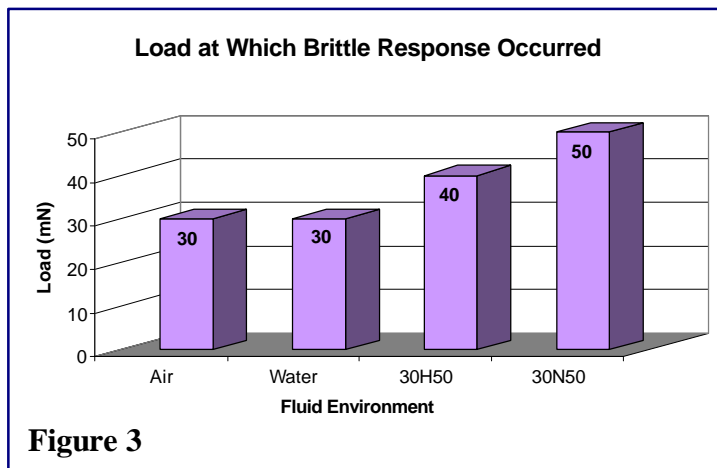


Figure 5 Scratch resulting in brittle fracture