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Ductile-brittle transition detection in scratching of single crystal silicon using charged particle emissions

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Abstract

During machining of single-crystal silicon, material removal involves two modes ductile shear-based removal and brittle fracture-based removal. Ductile shear-based chip removal occurs when fracture is suppressed due to local stress conditions along with reduced chances of defect involvement and is desirable for achieving better surface integrity of the machined silicon wafer. In this work, we use charged particle emissions to identify mode of material removal (ductile or brittle) during scratching of a silicon wafer. Scratching tests were performed using a pin-on-disc tribometer setup with a conical diamond tip indenter, in which the wafer was held at an inclined position to achieve a varying-depth tapered scratch. The varying-depth scratch test was performed in such a manner that both ductile-to-brittle and brittle-to-ductile modes occur in a single scratch test. The charged particles emitted during the material deformation were collected using a Faraday plate mounted in the vicinity of the indenter and the intensity of the charged particles were measured using a sensitive femto/picoammeter. The scratch depth was measured using a 3D surface profiler and the mode of fracture was identified by examining crack density per unit length in a scanning electron microscope. These results were then correlated with the emission intensity signals. From the experimental results, a positive current intensity was observed for ductile mode of scratching and highly varying current intensity signal is observed during brittle mode of scratching. The results obtained were consistent over time and exhibited good repeatability. The present work indicates suitability of employing charge emission signals to detect mode of material removal during scratching of silicon. This work can be field-tested by conducting diamond turning experiments of silicon in real-time machining environment further testing the scope of use of charged particle emission to monitor real-time machining process.

Keywords: Ductile and brittle fracture; charged particle emission; silicon

1. Introduction

Single crystal silicon is being used as one of the prominent materials in the field of Infrared (IR) optics, laser optics, X-ray beam deflections, etc. for night vision and thermal imaging applications [1]. In most of these applications, highly finished crack free surfaces in silicon is desirable, which is processed by ultra-precision single point turning and/or abrasive polishing. During diamond turning, silicon is subjected to surface (micro-cracks) and sub-surface damage which depends on the process parameters. Fundamentally, material removal in single crystal silicon (brittle material) occurs due to defects like dislocations movement and vacancy within a grain. These defects initiated by the cutting tool do not penetrate into the newly formed surface in ductile shear-based material removal. While in brittle-fracture based material removal, silicon fractures suddenly without any warning (plastic deformation) and the defects initiated by the cutting tool leads to crack and fractures on the surface [2].

To machine silicon in ductile mode, the contact pressure exerted by the cutting tool on silicon should be about 12 GPa [3]. The diamond cubic structure of silicon will transform to metallic phase in the above contact pressure leading to ductile shear based material removal [4]. Upon pressure unloading,

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metallic phase of silicon converts to amorphous phase. Contact pressure more than 16 GPa will lead to brittle fracture based material removal and below 12 GPa will lead to elastic deformation of the surface, where no phase transformation occurs [3].

If the contact area between the tool and silicon increases, the pressure exerted by the tool on silicon decreases, leading to insufficient pressure for phase transformation from diamond cubic to metallic phase [5]. Thereby, ductile shear-based material removal cannot occur. Contact area increases, when either the depth of cut or width of cut increases. The minimum depth up to which ductile mode of deformation occurs is defined as critical depth of cut (d_c).

Researchers have defined ductile to brittle transition based methods such as: visual observation of cracks on the scratch path [7,8], force measurement [8], Acoustic Emission (AE) measurement [9] during scratching of silicon, etc. M. Yoshino, et al. [6] had performed scratching study on silicon at low cutting speed of 265.9x10^{-6} m/s in vacuum inside a Scanning Electron Microscope (SEM) and the d_c was determined as 600 nm, below which no cracks were observed on the scratch path. Hao Wu, et al. [7] conducted scratching experiments on silicon at speeds of 1 mm/min (0.00002 m/s) with two different indenter shapes namely, truncated conical tip (60° included angle) and conical tip (120° included angle), and the d_c was reported as 318 nm and 52 nm respectively, based on visual interpretation of scratch path under SEM. Arkadeep Kumar, et al. [8] had defined ductile to brittle transition using normal and tangential forces during scratching under dry and wet conditions. Koshimizu, et al. [9] had performed scratching and indentation of silicon to define ductile to brittle transition using acoustic emission signals. The transition region of the scratch (region where cracks start to form before catastrophic brittle fracture starts to occur) is usually ignored and not analysed in the above works. In this work, the ductile or brittle mode of cutting in silicon has been detected by Charged Particle Emissions (CPE), as well as the transition region has also been well captured by the signal. Transition region is important for defining the d_c.

Charged particles will be emitted from a material, when it abrades with another material [10], during material shear, fracture [11], etc. Jagadeesh, et al. [12] has captured charged particle emission current intensity during machining of mild steel, stainless steel and copper in atmosphere and reported that material removal rate is proportional to emission intensity. Nakayama, et al. [10] had performed scratching experiments on conductors (low electrical resistivity), semi-conductors and insulators (high electrical resistivity), reporting that emission intensity increases with resistivity of the material. Molina, et al. [13] had conducted scratching experiments in vacuum on silicon using a diamond indenter and reported the emission of negatively charged particles during brittle fracture. Hisham, et al. [14] reported a voltage drop in signal during scratching of silicon, confirming the transformation of metallic phase (Si-II) from semi-conducting phase (Si-I). Metallic phase has a lower electrical resistivity than semi-conducting phase. None of the prior reported studies have observed the transition from ductile-to-brittle during scratching using charged particle emissions.

Machining and scratching processes are analogous in that machining involves material removal using a single point cutting tool by overlapping a series of scratches. Hence, in this work, varying-depth scratching experiments were conducted in atmosphere to detect the ductile-brittle transition of silicon using charged particle emissions. Charged particle emission current intensity signal was captured, during the scratching test on single crystal silicon using a conical diamond tip indenter. The scratch formed involves all the material deformation zones (ductile, transition and brittle). The objective is to distinguish these various deformation zones in the signal. Initially, the scratch path was analysed to find the length of ductile, transition and brittle regions. The ductile region is identified by no cracks in the scratched path. Transition region is identified by cracks and brittle region by fractures in the scratch path. From the length of each regions, the time taken for each of these regions was computed and correlated to the signal. The depth of the scratch in each of these regions are also measured to define the d_c of the scratching process.

2. Experimental details

Scratching is a material removal process which involves shear and fracture of material, charged particles are likely to be emitted. Fig. 1 shows emission of charged particles (positive and negative ions) from a material, when it is scratched by an indenter moving with a linear velocity v and applying a normal force F_n on the material. These charged particles (with kinetic energy) are captured by the Faraday plate (good electrical conductor), mounted on the arm of the tribometer above the indenter, and measured as emission current intensity with respect to time by the a femto/picoammeter.

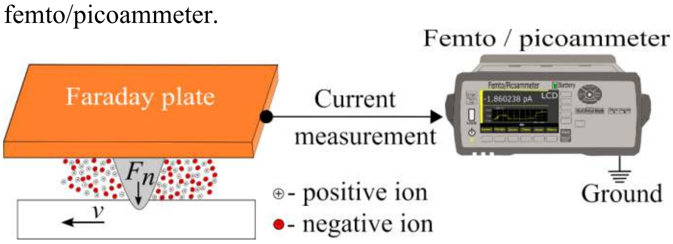

Fig. 1 CPE measurement during scratching.

Rotational scratching experiments were conducted at room temperature and pressure on a one-side polished single crystal p-type silicon wafer (100) of 100 mm diameter and 1 mm thickness using a pin-on-disc tribometer as shown Fig. 2. The silicon wafer was mounted on a steel disc using an adhesive tape. The steel disc was ground initially to reduce its surface roughness and waviness and achieve the taper angle needed. The taper angle (α) was provided by placing the silicon wafer on a thin shim (50 µm) as shown in Fig. 3. A conical diamond tip indenter of 90° conical angle and a shank diameter of 7 mm was used to perform the scratching on the silicon wafer. Scratching was performed at a constant v of 0.071 m/s and a varying-depth tapered scratch was produced. From Fig. 2, it is seen that the scratch was produced in a partial circular arc. The lever arm is lifted manually after one revolution, so that the indenter loses contact with the silicon wafer and does not contact the scratch which was made. The scratch is produced in such a way that the depth is minimum at the start and end portions and maximum in the middle portion. Thus, the transition region occurs twice in a single scratch test: ductile-to-brittle at the start of scratch and brittle-to-ductile at the end of scratch. The surface integrity of the scratch was examined under a SEM and the depth of the scratch was measured using a 3D surface profiler.

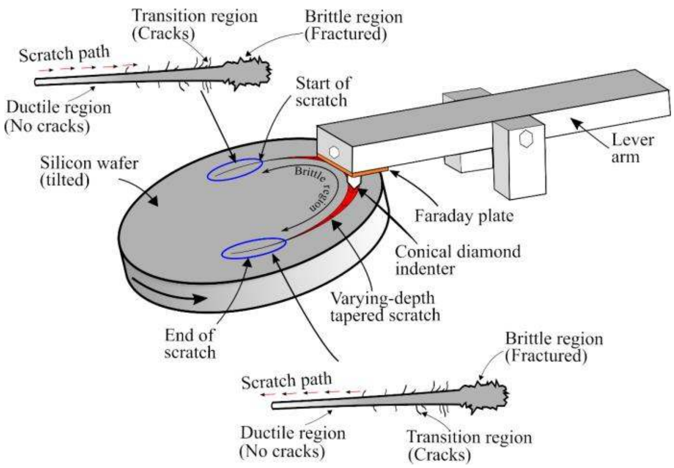

Fig. 2 Schematic of the experimental setup.

Relevant electrical insulations were provided between the Faraday plate, indenter and arm of the tribometer to avoid discharge of charges generated in the scratching zone as follows:

- The shank of the indenter is press fitted on to a nylon holder to electrically insulate it from the Faraday plate.
- The conical portion of the indenter is surrounded by an electrical insulation tape to avoid leakage of charges through the shank.
- Faraday plate is a thin strip of copper (thickness 0.5 mm) and was affixed to the arm of tribometer by an electrically insulating adhesive tape.

The distance between the silicon wafer and Faraday plate was 3.5 ± 0.5 mm (‘*d*’ in Fig. 3). The Faraday plate was large enough to capture all the charged particles emitted from the scratch path. Emission current was measured for every 1 ms (10^{-3} s – millisecond) and the resulting data was transferred to a computer from the femto/picoammeter. Femto/picoammeter is highly sensitive, which was confirmed by making a small indentation on the silicon wafer, which results in a small spike in the signal. The measurement range set in the femto / picoammeter is ± 200 pA (10^{-12}A – pico ampere). The instrument captures current values up to 6 decimal places and the accuracy in the above range is ± 0.5 % of reading + 5 fA offset (fA – 10^{-15}A – femto ampere) [15], which is sufficiently sensitive for our measurement. The next available range for setting in this instrument is ± 2 nA (10^{-9} A – nano ampere), and this results in an accuracy of ± 0.2 % of reading + 300 fA offset.

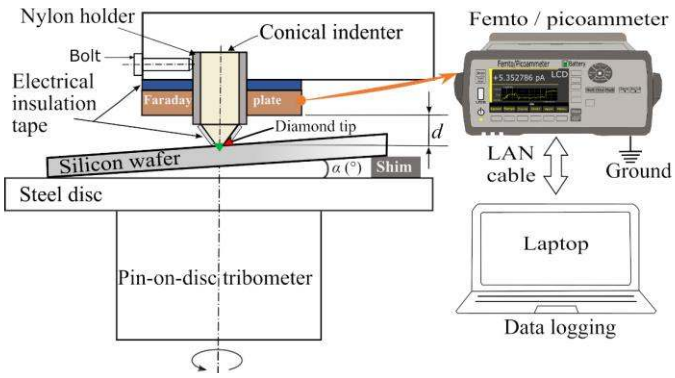

Fig. 3 Schematic of cross-sectional view of the experimental setup.

Sample experiments conducted using both the ranges showed that the signal intensity remained in the pA range for most of the signal time. The femto/picoammeter was powered by an in-built lithium ion battery which reduces the noise in the signal during emission current measurement.

3. Results and Discussion

A typical emission current signal, as a function of time, acquired during the scratching process is shown in Fig. 4. The SEM micrographs along the various regions of the crack path are shown in Fig. 5, Fig. 6, Fig. 7, Fig. 8 and Fig. 9. The relevant regions of ductile, transition and brittle are marked in Fig. 4. The current signal intensity is in the range of a few 100s of pA. The signal intensity before scratching fluctuates between -20 to -50 pA, which indicates the motion of charged particles in the atmosphere. After beginning of scratch, the emission signals strongly change well over and beyond this -20 to -50 pA variation indicating that emissions from the scratching phenomenon clearly exist.

3.1. Ductile region at the start of the scratch (D1)

A sudden spike in signal occurs when the scratching begins, and the signal intensity jumps to +6 pA. As shown in Fig. 4, after the initial spike, the intensity of the signal is in the range of +77 pA to -28 pA. However, it is observed that the signal has a positive current intensity for most of the time and in this region the scratch depth continuously increases. The location of the start of scratch is obtained from SEM. The scratch path was observed continuously till the first crack appears. The location of this first crack as well as some regions without any cracks are also noted. These regions with no cracks on the scratch path are identified as the ductile regions (Fig. 5). The scratch length (calculated based on SEM micrographs) from the start till the location of the first crack is defined as ‘ductile region at the start of the scratch’ (D1). The corresponding time taken is indicated in Fig. 4 and the length of the D1 region is 33.84 mm.

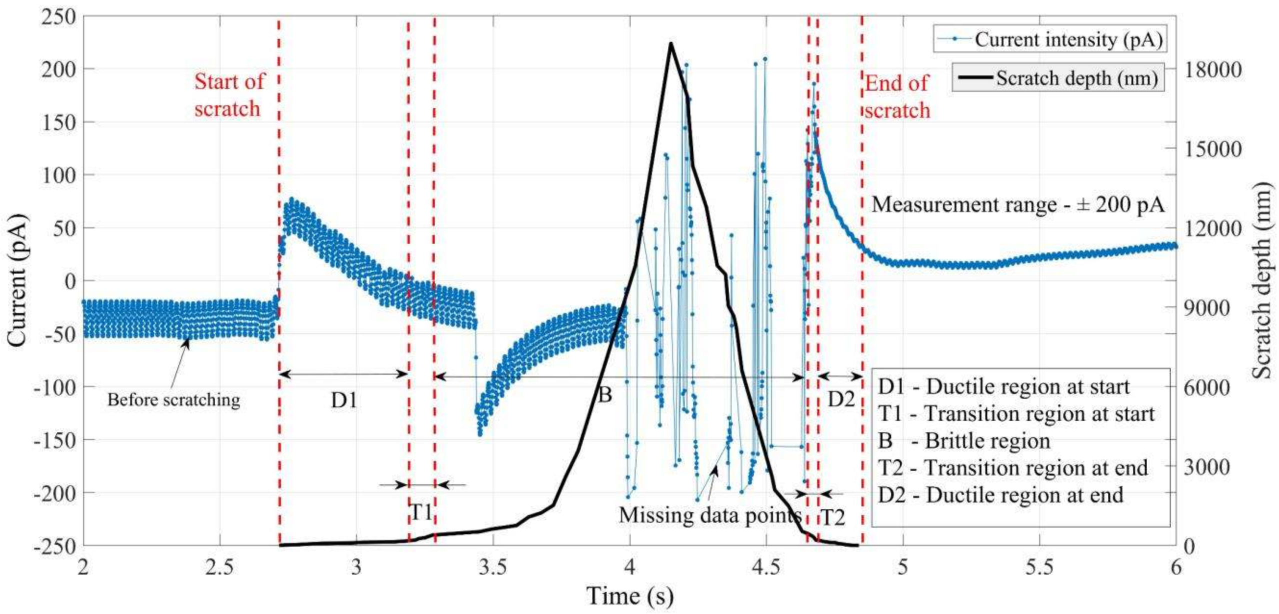

Fig. 4 Emission current intensity signal measured during the scratching process along with scratch depth (nm).

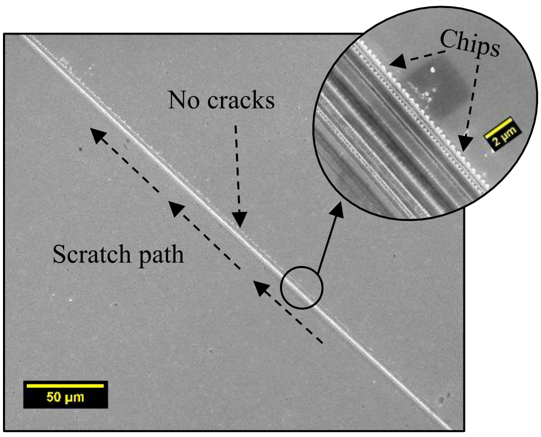

Fig. 5 SEM image of ductile region at the start of the scratch (D1).

3.2. Transition region at the start of the scratch (T1)

Transition region (T1) at the start of the scratch is identified by the presence of surface cracks in the scratch path (Fig. 6). As the scratch depth increases, the crack density (number of cracks per unit length) increases along the scratch path in this region. At the start of transition region, short cracks are observed (Fig. 6a). As the scratch depth increases, the cracks branch out leading to more cracks as well as the crack length increases significantly (Fig. 6b). The scratch length of 6.19 mm (calculated from SEM images) from the first crack till the first fracture is defined as 'transition region at the start of the scratch' (T1). The emission current intensity is negative in this region and is in the range of -28 pA to -40 pA (Fig. 4).

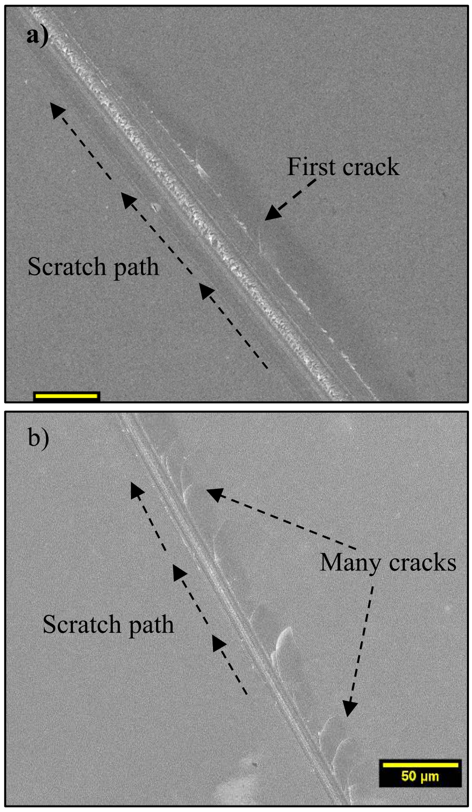

Fig. 6 a) SEM image shows first crack in the transition region at the start of the scratch (T1); b) SEM image shows many cracks in the transition region at the start of the scratch (T1).

3.3. Brittle region (B) of the scratch

Brittle region (B) of the scratch is identified by fractures in the scratch path (Fig. 7). In this region, the crack density increases and fractured regions starts appearing along the scratch path. Fig. 7a shows fractures at the start of brittle region and Fig. 7b shows fractures at the end of brittle region. The intensity of the signal changes from negative to positive at the initial portion of the brittle region and then becomes highly erratic with signal varying in both positive and negative current intensities. Also, there are many missing data points in this region, which indicates that the emission current value is out of the set measurement range (± 200 pA) i.e. signal intensity increased to nA range (Fig. 4). The scratch depth in the brittle region is more than 500 nm for most of the signal time. Therefore, a greater number of charged particles are expected to be emitted leading to high emission current intensity. The scratch length of the 'brittle region' (from the first fracture till the last fracture along the scratch path) is obtained as 97.90 mm from SEM micrographs.

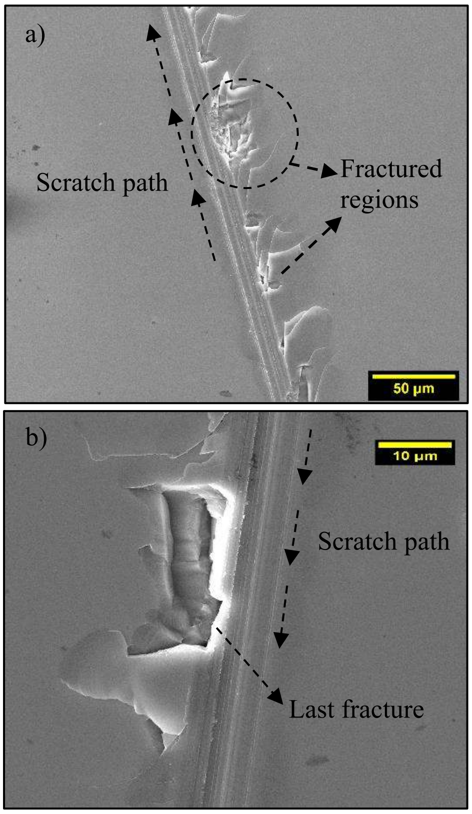

Fig. 7 a) SEM image shows fractured regions in the brittle region of the scratch (B); b) SEM image shows last fracture in the brittle region of the scratch (B).

3.4. Transition region at the end of the scratch (T2)

The depth of scratch reduces and hence the fractures disappear leading to transition region at the end of the scratch. Crack density (number of cracks per unit length) starts reducing along the scratch path in this region. The scratch length of 2.54 mm (calculated from SEM images Fig. 8a and Fig. 8b) from the last fracture of brittle region till the last crack along the scratch path is defined as 'transition region at the end of the scratch' (T2). The corresponding time taken is indicated as 'T2' in Fig. 4. The emission current signal ranges between -23 pA to +180 pA in this region. Also, there are some missing data points, indicating the current intensity is again out of ± 200 pA. At the start, the transition region occurs after the ductile region (smaller scratch depth), while at the end, it occurs after the brittle region (larger scratch depth). As scratch depth increases, current intensity increases (a greater number of charged particles emitted). Therefore, current intensity at the transition region during ductile-to-brittle and brittle-to-ductile are different. This may be due to Faraday plate, being larger than the indenter, capturing ions both from ahead and behind the indenter from already scratched regions. During the start of scratch, already scratched regions are in ductile mode of deformation (smaller current intensity), while at the end of scratch, already scratched regions are in brittle mode (larger current intensity).

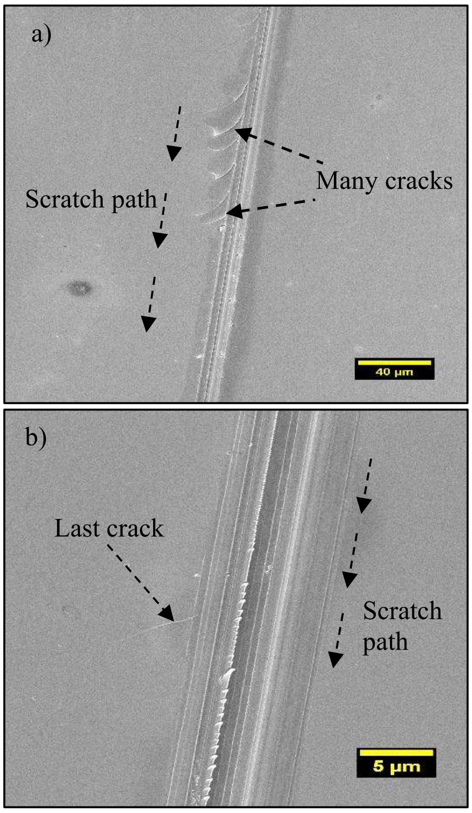

Fig. 8 a) SEM image shows many cracks in the transition region at the end of the scratch (T2); b) SEM image shows last crack in the transition region at the end of the scratch (T2).

3.5. Ductile region at the end of the scratch (D2)

At the end of scratch, region with no cracks is identified as ductile region (D2) (refer Fig. 9). In this region, the depth of the scratch further reduces compared to transition region and the cracks have almost disappeared in the region. The scratch length of 10.88 mm was calculated from the last crack of the transition region till the end of scratch and the time taken is shown as 'D2' in Fig. 4. The scratch depth decreases along the scratch path in this region and it is reflected as a drop in signal from +118 pA to +32 pA. The signal stabilizes between +14 pA to +18 pA as scratching stops. The emission current intensity at region 'D2' and 'D1' at start and end of scratch have similar emission current signature.

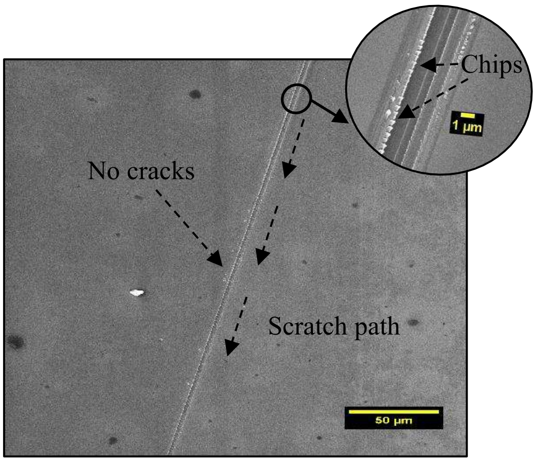

Fig. 9 SEM image of ductile region at the end of the scratch (D2).

The width of the scratch is small in the ductile region as compared to brittle or transition region which is evident from the SEM images (Fig. 5, Fig. 6 and Fig. 7). Ductile region length at the start and end of scratch are not same indicating that the scratch is not symmetric, possibly due to the surface thickness variations of the silicon wafer, as well as the limitation of not having a controlled depth experimental setup. The experiment has been repeated two times using the same process conditions and a similar trend in emission current signal has been consistently observed. In the repeat experiment, crystallographic orientation effects are not the same since the start position of scratch can vary.

3.6. Critical depth of cut (d_c)

The d_c of silicon depends on several factors including tool tip geometry (shape of indenter), tool rake angle, cutting speed, anisotropy of silicon etc. Hence, the region where the first crack appears may not be addressed as the d_c always [2]. In this work, d_c is defined based on steepest slope method [2]. Number of cracks per unit length (crack density) in the transition region at both the start and end of the scratch is calculated and plotted against the average depth of the scratch (from 3D surface profiler) in this region of cracks (Fig. 10). A polynomial trend line is fitted to the data points ($R^2 = 0.75$). Slope of the trend line at each average depth is calculated and the depth having the steepest slope is defined as the d_c. At the steepest slope, number of cracks in the scratch disappears. As per this method, d_c of silicon is 181 nm, when using a conical diamond tip indenter of 45° negative rake angle at a cutting speed of 0.071 m/s. At the start of the scratch, d_c occurs at an intensity of -28 pA i.e. at the start of transition region and at the end of scratch it occurs at an intensity of +120 pA.

The d_c occurs in the transition region where the signal is either decreasing or increasing depend on the transition from ductile to brittle or brittle to ductile. Further signal processing is required to understand how the signal varies at this critical depth region and in general in the transition region.

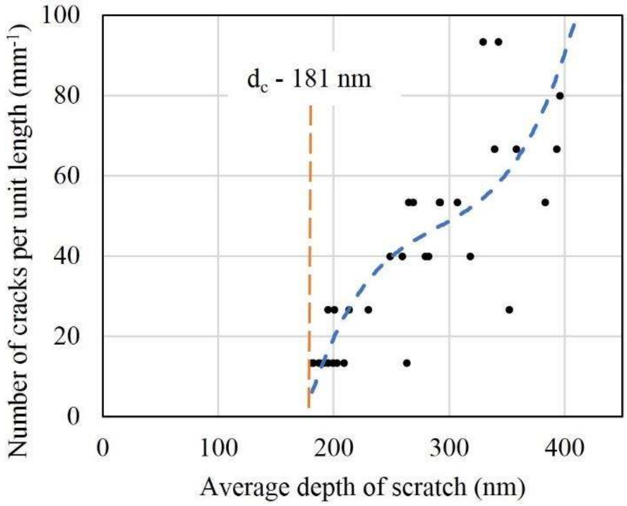

Fig. 10 Number of cracks per unit length vs. average depth of scratch along the scratch path (both start and end of the scratch).

4. Conclusions

The important conclusions from this study are as follows,

- During the varying-depth scratching experiments on single crystal silicon using a conical diamond tip indenter, the emission current signal exhibits distinct signatures at various material deformation regions (ductile, transition and brittle regions of the scratch) with good repeatability.
- Emissions display positive current intensity for ductile shear-based material removal. During brittle fracture-based material removal, the signal intensities are very high and varies significantly in both positive as well as negative current intensity for most of the signal time.
- The signal intensities in the transition region of start and end zones of the scratch are different. Transition occurs from ductile-to-brittle at the start i.e. low current to high current intensity. While at the end, it occurs from brittle-to-ductile i.e. high current to low current intensity. The setup captures ions both ahead and behind the indenter, so it can capture ions from previously scratched regions, which may be the reason for a different current intensity at the start and end.

In future, experiments will be conducted with Faraday plate paced in such a way that it will capture ions only ahead of the indenter. We also intend to carry out signal processing analysis to understand the onset of the various deformation modes of scratched material removal. This work paves the way for a novel in-situ process monitoring method to be developed, and field tested in diamond turn machining of silicon to detect onset of brittle mode of machining enabling corrective action to be taken.

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