Determining the Ductile to Brittle Transition (DBT) of a Single-Crystal 4H-SiC Wafer by Performing Nanometric Cutting

Deepak Ravindra¹,a and John Patten¹,b

¹1903 West Michigan Avenue, Kalamazoo, Michigan, 49008-5314 USA
a,deepak.ravindra@wmich.edu, b, john.patten@wmich.edu

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Abstract. Silicon carbide, like other brittle materials, is known for its poor machinability. However, ductile-regime machining is possible under certain conditions. This can be achieved if machining occurs at depths less than the critical depth of cut. Beyond this value, a Ductile-to-Brittle Transition (DBT) occurs and the material behaves in a brittle-fracture manner. The purpose of this research is to determine the DBT for a single-crystal 4H-SiC wafer by performing Nanometric cutting (Nanocuts) experiments. The depth of cut is adjusted over a range of 100nm to 1000nm in order to cover the entire ductile to brittle-regime and the corresponding material removal behavior. The Nanocuts were carried out using the Nanocut II, a second generation prototype experimental machining instrument. The Nanocuts were imaged and measured using an Atomic Force Microscope (AFM) and the height profile from the scanned images was used to determine the DBT.

Introduction

Interest in SiC is growing due to its excellent mechanical properties such as extreme hardness, high wear resistance, high thermal conductivity, high electric field breakdown strength and high maximum current density. Due to its high hardness and brittle characteristics, SiC is a very challenging material to machine especially at the micro/nano level. The most common polytypes of SiC are 2H, 3C, 4H, 6H, and 15R. The numbers refer to the number of layers in the unit cell and the letter designates the crystal structure where C=cubic, H=hexagonal, and R=rhombohedral.

Patten and Gao¹ state that ceramics in general undergo a phase transformation to an amorphous phase after a machining process. This transformation is a result of the High Pressure Phase Transformation (HPPT) that occurs when the high pressure and shear caused by the tool is suddenly released after a machining process. This phase transformation is usually characterized by the amorphous remnant that is present on the workpiece surface and within the chip.¹ There are two types of material removal mechanisms of interest for the current paper, these are the ductile mechanism and the brittle mechanism.² In the ductile mechanism, plastic flow of material in the form of severely sheared machining chips occur, while material removal is achieved by the intersection and propagation of cracks in the brittle fracture mechanism. Due to the presence of these two competing mechanisms, it is important to know the DBT depths (or critical size) associated with these materials before attempting a machining operation. The purpose of this paper is to report on nanometric cutting experiments performed to determine the DBT depth for a single crystal 4H-SiC. A polished single crystal wafer was used in this experiment program, which provides for an excellent reference surface for
determining the DBT and to establish the corresponding critical depth. These wafers are also of high purity with few defects.

**Experimental Method**

The Nanocut II (a second-generation prototype) was used to make the nanometer level cuts. The Nanocut II was designed to perform nanometer depth cuts based on the commanded depth by the operator as executed by the control program. The main components of this equipment are the frame, PZT tube (provides x, y and z displacement), capacitance gage (displacement feedback), force sensors, sample holder, tool holder, and hysteretic positioners. The PZT tube is used to position the sample relative to the tool, to establish the depth of cut. The Z (depth of cut) position is determined via the capacitance gage, and the two orthogonally placed force sensors measure the cutting and thrust forces. There are four dual axis flexures used in the device to decouple the cutting and thrust forces and to support the tool stage positioning wedge actuators. Fig.1 shows a top view of the Nanocut II.

![Fig.1. A top view of the Nanocut II used to perform the Nanocuts.](image)

The series of cuts were performed on rectangular pieces, which were cleaved from a 3” (76.2mm) 4H-SiC wafer. The wafer was cleaved relative to the primary flat of the wafer, which is in {10\bar{1}0} plane with the flat face parallel to the <11\bar{2}0> direction. The rectangular sample size obtained from the wafer was about 12mm by 6mm. Identifying the crystal orientation is important to determine the preferred cutting direction. The SiC sample was mounted on the sample holder with an adhesive. To minimize any external vibration, noise and distortion, the Nanocut II was placed on an air vibration isolation table. A single-point diamond tool with a rake angle of -45 degrees and a clearance angle of 5 degrees was used to perform the cuts. For the results reported here, the diamond tool was oriented such that the Nanocuts were performed parallel to the primary flat.

Two sets of experiments were carried out on different rectangular samples, taken from the same wafer and positioned at the same crystallographic orientation. The first set contained cuts with commanded depths of 100nm and 500nm. The second set contained cuts with commanded depth of 1000nm. The expected DBT was in the range of 100nm to 1000nm, and these experimental cut depths covered this entire region. Three cuts were done for each commanded depth to obtain comparable data. The second set of deeper cuts (1000nm and greater if necessary) would only be carried out if there were no brittle characteristics in the first set of cuts (100nm and 500nm). The cuts were done in array
pattern to help with imaging. Since the Nanocuts in this experiment are fairly small (approximately 20µm in width and 120µm in length), identifying them in the microscope can be challenging. Fig.2(a) shows a schematic representing the pattern created with the Nanocuts on the rectangular SiC samples; the 100nm and 500nm cuts were performed on one sample and the 1000nm cuts were performed on a second sample. Fig.2(b) represents an actual image of three of the cuts (two at 100nm and one at 500nm) obtained from an optical microscope at 40X magnification. The other 100nm and 500nm cuts are outside the field of view at the magnification shown.

The cuts are made from right to left and the cuts are wider than they are long due to the geometry of the tool (10mm nose radius) and maximum stroke of the PZT in the cutting direction (20-30 µm), which establishes the length of cut.

**Results**

Three different programmed depths of cuts were planned to be carried out on the first sample and two on the second sample. Fig.3 shows the thrust force and the cutting force for each corresponding depth of cut. The cutting forces are measured to be more than the thrust forces for all of the three reported depths of cuts. Both the cutting forces and the thrust forces increase as the depth of cut increases. The 50nm depth of cut is not reported in Fig.3 as only one cut at that depth was successful and it could not be identified with the AFM. Each of the other reported depths had three successful/repeatable cuts, for which the force data were averaged.

A typical height profile obtained from the ductile regime is generally “V” or “U” shaped representing an imprint of the diamond tool cutting edge. Fig.4(a) shows a typical height profile, obtained from an AFM, of a cut performed in the ductile region. The depth of this cut was measured to be 245nm and the programmed depth of cut was 500nm. The actual depth of cut was measured to be less than the commanded or programmed depth of
cut; this is a characteristic of the device and process and is consistent with previous results. The “V” shape seen in the height profile of Fig.4(a) is a characteristic of a ductile cut.

Fig. 4(a). An AFM scanned section of a cut along with its height profile. The programmed or commanded depth of this cut was 500nm, at the cross-section indicated the measured depth is 245nm.

Fig. 4(b) shows a height profile of a cut where there is an indication of a DBT. This is evident by the poorly defined ductile “V” shape in the height profile. The depth of cut was measured to be 836nm using an AFM and the programmed depth of cut is 1000nm.

Fig. 4(b). An AFM scanned section of a cut where the brittle characteristic of the material is visible.

Fig. 4(c), confirms that the material removal is in the brittle-regime and there is no clear definition in the height profile of this cut. The measured maximum depth of cut is 952nm and the programmed depth of cut of 1000nm. It can be seen in Fig. 4(c) that there could be more than one peak in the brittle region as the fracture process results in uncontrolled material removal.

Fig. 4(c). An AFM scanned section of a cut where the brittle characteristic of the material is observed.

The edges along the cuts can also be observed to determine the ductile or brittle cutting conditions. The ductile cut has a much more defined and straight edge as seen in Fig.4(a) compared to the jagged edges seen in a brittle cut shown in Fig.4(b & c). A clearer
picture of the uneven edges along a brittle cut is shown in Fig. 5(a). The jagged edges and chipped material along the left edge of the cut are caused due to crack propagation, and uncontrolled material removal in the brittle regime. This brittle material is clearly seen in Fig. 5(b), where the cross section of a brittle cut is analyzed using the Wyko RST interferometric microscope. The actual depth varies from zero at the ends (top and bottom of the cuts, outside the field of view) to a maximum in the middle.

Fig. 5(a) Optical image (20x) of 1000nm cut showing brittle fracture (cutting direction right to left)

Fig. 5(b). White light interferometric microscope image of 1000nm brittle cut

As seen in Fig. 6, the brittle characteristics are clearly shown by the height profile graph from an AFM image. The programmed depth for this particular cut is 1000nm (1µm) but the maximum depth measured in this cut is 1.17µm. This characteristic (programmed depth < actual depth) was also observed by Hung and Fu in their experiment to study the ductile-regime machining of silicon, where the microcracks could extend deeper than the depth of cut below the machined surface.8

Fig. 6. AFM scanned section where the maximum measured depth of cut (1170nm) is more than the commanded/programmed depth of cut (1000nm).

In the case of a cut under extreme brittle conditions of SiC, there is no direct control of resultant material removal; i.e. the command depth does not provide a one-to-one correspondence to the actual depth of cut. It is this stage (beyond the DBT) where crack initiation, propagation and growth occur. These events can lead to catastrophic brittle failure of the material.
Conclusion

Although SiC is well known for its brittle characteristics, it is still possible to plastically deform this material at small scales (nominally less than a micrometer) to achieve ductile machining. In order to machine a semiconductor or ceramic in the ductile-regime, it is crucial to know its DBT depth. The DBT depth was found to be between 820nm-830nm as measured on a single crystal 4H-SiC wafer, cut parallel to the wafers primary flat orientation i.e. the \{10 

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References


