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Ductile Mode Material Removal of Ceramics and Semiconductors

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DUCTILE MODE MATERIAL REMOVAL OF CERAMICS AND SEMICONDUCTORS

by

Deepak Ravindra

A Dissertation
Submitted to the
Faculty of The Graduate College
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Advisor: John Patten, Ph.D.

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Ceramics and semiconductors are hard, strong, inert and lightweight. They also have good optical properties, wide energy bandgap and high maximum current density. This combination of properties makes them ideal candidates for tribological, semiconductor, MEMS and optoelectronic applications respectively. Manufacturing these materials without causing surface and subsurface damage is extremely challenging due to their high hardness, brittle characteristics and poor machinability. However, ductile regime machining of these materials is possible due to the high-pressure phase transformation occurring in the material caused by the high compressive stresses induced by the single point diamond tool tip. In this study, to further augment the ductile response of the machined material, single point scratch tests are coupled with a micro-laser assisted machining (μ-LAM) technique. The high pressure phase is preferentially heated and thermally softened by using concentrated energy sources (i.e. laser beams) to enhance the ductile response of the material. The focus here is to develop an efficient manufacturing technique to improve the surface quality of ceramics and semiconductors to be used as optical devices (mirrors and windows). Machining parameters such as the depth of cut, feed, cutting speed and laser power are optimized in order to make the manufacturing process more time and cost effective. Also, the science behind the thermal
softening effect during the formation of high-pressure phases is experimentally studied by isolating the temperature and pressure effect. Micro-laser assisted scratch tests successfully demonstrate the enhanced thermal softening in silicon (Si), silicon carbide (SiC) and sapphire resulting in greater depths of cuts (when compared to similar applied loads for cuts with no laser), greater ductile-to-brittle transition depths and smaller cutting forces. Imaging and characterization techniques such as optical microscopy, light interferometry, XRD, surface profilometry, OIM, AFM, scanning acoustic microscopy, electron microscopy and Raman spectroscopy are utilized to quantify the ductile mode material removal process. Ductile mode machining is experimented on nine ceramics and semiconductors including Si, SiC (three polytypes: 4H, 6H and 3C), sapphire, quartz, spinel, AlON and AlTiC.
DEDICATION

I dedicate this dissertation to my wonderful family, particularly to my understanding and patient wife, Seena, who has put up with my frequent moodiness over the recent years. Her unconditional love and believe in me has been a great boost of inspiration throughout this journey. Also to my loving parents, without whose love, encouragement, and support, I would have never accomplished what I did. Their priceless advice and hard work will always be treasured and appreciated for the rest of my life.
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Since most of my experimental work was conducted on the universal micro-tribometer by CETR Inc., it is safe to say that I had a ton of technical questions regarding
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CHAPTER 1

INTRODUCTION

A wide range of materials including metals and alloys, ceramics, glasses, semiconductors and composites are manufactured to meet service requirements to a given geometry, accuracy, finish and surface integrity.\(^1\) Metals and alloys in general are easier to machine in the ductile regime because of their high fracture toughness, low hardness, non-directional bonding, low porosity, large strain to fracture and high impact energy. Non-metals, on the other hand, such as ceramics and semiconductors, are characterized by covalent or ionic bonding, limited slip systems for plastic deformation, high hardness and low fracture toughness.\(^2\) It is due to these major differences that ceramics and semiconductors are classified as “difficult to machine” nominally brittle materials.

Ceramics and semiconductors however, have some attractive properties compared to metals and polymers, which make them useful for specific applications such as for high temperature and high power devices. Ceramics are a very broad class of materials with a wide range of properties.\(^3\) One often distinguishes between traditional ceramics such as tableware, pottery, sanitary ware, tiles, bricks and advanced engineered ceramics, where depending on the specific application one or several of the following properties are utilized: high strength at high temperatures, wear resistance, corrosion resistance, low density, low thermal conductivity, low electric conductivity, favorable optical, electrical or magnetic properties, biological compatibility. The focus of this dissertation, however,
is on advanced ceramics and semiconductors with a wide range of mechanical, optical, electrical and chemical properties. In many cases physical properties are important such as electronic, optical and magnetic ceramics/semiconductors. In other cases, better mechanical properties as compared to metals are essential for material selection. Ceramics and semiconductors are also used because of their physical properties that are often designed to resist failure due to mechanical or thermal loading.

The major drawback of most semiconductors and ceramics is their brittleness, i.e. failure without a preceding plastic deformation. In other words, brittleness here means that failure occurs without prior measurable plastic deformation. This is due to the strong atomic bonding of ceramics, which lead to high stresses for the motion of dislocations. In essence, brittle materials feature low ductility and low fracture toughness at room temperature and atmospheric pressure so that fracture will occur once the atomic linkage forces are exceeded. Thus, brittle failure (fracture) initiating from small flaws before plastic deformation is possible. This fact can also be expressed in low resistance against crack extension, which is characterized by the fracture toughness. The absence of local plastic deformation leads to failure at locations of high local stresses, e.g., at crack tips, at contacts between different material or during thermal shock. In metals, these strain-controlled local stresses lead to small plastic strains.

Dramatic advances have occurred in ceramics technology since the mid-1990’s. New and improved ceramics and semiconductors are now available that have much higher strength and toughness than prior ceramics. Major research and development has been accomplished in the past 20-30 years to increase the capability of ceramics and semiconductors for thermal, wear, optical, corrosion and structural applications. These
intrinsic properties and developments make advanced ceramics and semiconductors an exceptional candidate in harsh environments such as high temperature, strong radiation, and corrosive and abrasive media. Some of the application fields utilizing advanced ceramics and semiconductors include automobile, aerospace, petroleum, nuclear, military and biological/medical industries. Manufacturing these materials are extremely challenging due to their high hardness, brittle characteristics and poor machinability. Severe fracture can result when trying to machine ceramics and semiconductors due to its low fracture toughness. However, from past experience it has been proven that ductile regime machining of brittle materials is possible. Some studies indicate that ductility is related to the high-pressure phase transformation (HPPT) that occurs when these materials are subjected to high hydrostatic pressures and shear stresses.

1.1 MATERIALS

Complexity in materials applications is the basis of technological advancement and hence there is a continuous search for new and improved materials. Today’s engineering applications and environment demand materials that are light in weight, strong, tough, corrosion resistant, durable, wear resistant, resistant to various hazards of nature, safe for our health, etc. In this research, several ceramics and semiconductors are studied. The ceramics studied here include silicon carbide (SiC), silicon (Si), quartz (SiO₂), aluminum oxynitride - AlON (AlₓOᵧNₙ), sapphire (Al₂O₃), spinel (MgAl₂O₄) and aluminum titanium carbide (AlTiC). However, three different SiC polytypes (4H, 6H and 3C), Si, quartz and aluminum oxide materials are studied in detail throughout this research.
1.1.1 SINGLE CRYSTAL SILICON

Single crystal Si is the principal material used for solid-state electronics and infrared optical technologies. Si is widely used in the fabrication of semiconductors and micro-electro-mechanical (MEMS) components. On the quantum scale that microprocessors operate on, the presence of grain boundaries would have a significant impact on the functionality of field effect transistors by altering local electrical properties. Therefore, microprocessor fabricators have invested heavily in facilities to produce large single crystals of Si. Over the recent decades, many stable and metastable solid phases of Si have been observed in pure hydrostatic pressure tests and other deviatoric tests such as micro- or nanoindentation.10

1.1.2 SINGLE CRYSTAL SILICON CARBIDE (4H AND 6H)

SiC is commercially available in various forms/phases (polytypes) such as single crystal (flame fusion, Czochralski, hydro-thermal, sublimation, etc.), polycrystalline (sintered and chemically vapor deposited [CVD]) and amorphous (commonly plasma deposited CVD). 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. The structure of the three major SiC polytypes (which are used in this study) is shown in Figure 1.1.

![Figure 1.1: Crystal structure of 3C, 4H and 6H SiC](image-url)
Polished single crystal wafers are becoming more common in the high power, high temperature electronics device industry. In this research, the 4H and 6H single crystal wafers are used as it provides for an excellent reference surface in determining the ductile-to-brittle transition (DBT) and to establish critical machining parameters. These wafers are also of high purity containing few defects, which will help in closely predicting the actual brittleness of the material.\textsuperscript{12} The 4H and 6H single crystal SiC wafers are typically used for:

- Optoelectronic Devices
- High-Power Devices
- High Temperature Devices
- High-Frequency Power Devices
- III-V Nitride Deposition

1.1.3 CHEMICALLY VAPOR DEPOSITED POLYCRYSTALLINE SILICON CARBIDE (3C-\(\beta\))

The fully dense beta cubic polycrystalline SiC CVD coating (\approx 250\,\mu m thick, applied on top of a SiC substrate) is a potential candidate to be used as mirrors for surveillance, high energy lasers (such as airborne lasers), laser radar systems, synchrotron x-ray, VUV telescopes, large astronomical telescopes and weather satellites.\textsuperscript{13} The primary reasons CVD coated SiC is preferred for these applications is that the material possesses high purity (\geq 99.9995\%), homogeneity, density (99.9\% dense), chemical and oxidation resistance, cleanability, polishability and thermal and dimensional stability. Machining SiC is extremely challenging due to its extreme hardness (\approx 27\, GPa) and
brittle characteristics. Besides the low fracture toughness of the material, severe tool wear (even on the single crystal diamond tool) also has to be considered due to its poor surface finish in the as-received form. Previous researchers have successfully been able to precisely grind CVD-SiC (using high precision grinding) but this process is very expensive and the fine abrasive wheels often result in an unstable machine/process.\textsuperscript{14} In order to fulfill the requirements to be qualified as a good candidate for optical mirrors, the surface finish for this material has to be close to mirror finish (Ra < 20nm). This is not easy to achieve from an as received CVD coated work piece that has a fairly rough surface (especially in the case of the POCO Graphite SuperSiC-2 discussed in Chapter 3)\textsuperscript{15} with Ra values exceeding 4.0 µm. Several machining techniques such as lapping, polishing, grinding, laser machining, and diamond turning are being used today in an attempt to micro- or nano machine this brittle material to a high quality, low roughness and damage free surface.

1.1.4 QUARTZ (FUSED SILICA)

The high demand in optical and electronic industry has been consistently pushing the barriers in various nanotechnology areas such as single point diamond turning (SPDT). In fact, it was just in the 1960’s where precision engineering involved a maximum precision of about 10µm.\textsuperscript{16} Mirror finishing of brittle materials such as glass is a fairly new concept for SPDT due to the challenges (such as tooling, machining accuracy, brittle material properties, etc.) that had to be overcome in the precision manufacturing industry.\textsuperscript{17,18} Attempts have been made recently to machine glass in a manner similar to ductile metals by using SPDT.\textsuperscript{19} SPDT has shown positive effects
when performed on materials which are often used as aspherical shapes in electronic and optical products. Such advances are essential in order to economically produce high quality ceramic and glass parts.

Glasses exhibit excellent optical and mechanical properties (good thermal resistance, excellent corrosive resistance, high hardness, etc.) which are needed for advanced technology applications. However, severe challenges arise when attempting to machine these extremely brittle materials to obtain excellent surface finish for optical devices. The combination of relatively high hardness (≈ 9.8GPa) and brittleness (low fracture toughness) plays an important factor in limiting glass materials from being easily machined using just a conventional machining technique (i.e. grinding, polishing, lapping etc) without causing brittle fracture.\(^\text{20}\) SPDT is one of the fabrication techniques that meet the demand of today’s precision engineering requirements, which requires both fast production rate and higher surface quality for optical devices.

Quartz (fused silica) is also experimented with in this research. Quartz has been one of the emerging materials due to its abundance in nature (quartz is known as the most abundant nonmetallic mineral on earth). For this research, a synthesized fused silica (Spectrosil 2000.\(^\text{21}\)) was used. Spectrosil 2000 is an ultra pure synthetic fused silica manufactured by Saint-Gobain Quartz PLC. This material possesses a chemical purity of 99.999% and is manufactured using an environmentally friendly process, which results in a material that is both chlorine-free and bubble-free. The bubble-free material enables a smoother surface finish after machining as it has very few or no impurities.
1.1.5 ALUMINUM OXIDE

Aluminum oxide (generally referred to as alumina) has the same composition as sapphire (Al$_2$O$_3$), which accounts for its high hardness and durability. Alumina ceramic is produced by compacting alumina powder into a shape and firing the powder at high temperature to allow it to densify (sinter) and slowly deposit into a solid, polycrystalline or single crystal part. Alumina is used in several applications such as seal rings, pump parts, chemical laboratory ware, spark plug insulators, heat exchange media and many more. It is used in these applications because of its excellent combination of properties, including high hardness, high wear resistance, good chemical resistance, smooth surface, good dimensional stability, decreased friction and reasonable strength.\(^{22}\) Alumina is presently the lowest cost high-performance ceramic due to the large quantity produced.\(^{23}\) In this study, two forms of aluminum oxide are experimented on: AlON (aluminum oxynitride, Al$_{23}$O$_{27}$N$_5$) and sapphire (Al$_2$O$_3$).

1.2 DIAMOND TOOLING

Diamond, the hardest material known to mankind, is an ideal candidate for cutting tools due to its ultra-high hardness (≈ 100 GPa), high resistance to wear and good thermal conductivity (~ 33.2 W/ (cm K)). This extreme hardness of diamond is necessary when attempting to cut hard and abrasive materials like SiC (micro-hardness ≈ 25-27 GPa)\(^{24}\). The general hardness ratio (tool: workpiece) preferred in industry is about 10:1 although in the case of diamond and SiC the tool/workpiece hardness ratio is only about 4:1, where the minimum acceptable value is suggested to be 5:1.\(^{25}\) This relatively low hardness ratio between the tool and workpiece, results in significant tool wear which will
be discussed in the later sections of this dissertation. The tetrahedrally-bonded structure (diamond cubic structure shown in Figure 1.2) of diamond makes it possible to fabricate tool tips with very sharp cutting edges (20 to 50 nm).

![Figure 1.2: The cubic structure of diamond.](image)

The ability to fabricate single crystal diamond tools with extremely smooth finish (Ra ≈ 3 nm), makes it ideal for SPDT ceramics to improve surface finish with a high degree of accuracy. Synthetic single crystal diamond tools and styluses with approximately the same properties as natural diamond, are used for all experiments discussed in this study. These synthetic diamonds are usually grown at high temperature and pressure from carbon feedstock. The effect of the brittleness and propensity of diamond to chip (resulting in tool breakage) is discussed in the later chapters. This drawback of the diamond chipping limits its application and necessitates larger included angle and nose radii for SPDT hard materials, such as ceramics.

### 1.3 NONDESTRUCTIVE TESTING

Nondestructive testing (NDT) is a wide group of analysis techniques used in science and industry to evaluate the properties of a material, component or system without impairing its future usefulness. Because NDT does not permanently alter the
article being inspected, it is a highly-valuable technique that can save both cost and time in material analyses and research.\textsuperscript{29} NDT methods often rely upon the use of electromagnetic radiation, sound and inherent properties of materials to examine samples. In this study, several NDT techniques are used on the ceramic and semiconductor samples for various analyses such as surface finish (white light interferometer), subsurface damage (optical microscopy and scanning acoustic microscopy), high pressure phase transformation (Raman spectroscopy), chip characteristics (transmission electron microscopy and Raman spectroscopy), crack formation (acoustic emissions and optical microscopy), fracture/brittle behavior (acoustic emissions and optical microscopy) and material crystal orientation (orientation imaging microscopy and x-ray diffraction).

1.4 MANUFACTURING PROCESS: SINGLE POINT DIAMOND TURNING (SPDT)

Traditionally machining brittle materials produces a damaged layer that is typically removed by chemical or mechanical polishing. However, micro/nano-machining of brittle materials is possible if sufficient compressive stress is provided causing a ductile mode material removal process in which material is removed by plastic deformation (chip formation). Suppression of a brittle response when cutting at a nanometric scale helps avoid the generation of surface and subsurface defects, such as cracks.\textsuperscript{30} To operate in the ductile regime, the cutting process and parameters must be thoroughly examined.

A good manufacturing technique should provide both high product quality and cost effectiveness. There are several manufacturing techniques such as turning, milling, polishing, lapping and honing. Traditionally, turning and milling are considered roughing
or semi-finishing operations. Polishing is used to obtain the final surface finish; this operation is carried out to remove micro-cracks and scratches (these damages are usually caused by previous manufacturing operations). Lapping and honing generally are carried out for obtaining form and shape accuracy such as flatness and sphericity.

SPDT was chosen as the preferred material removal method as it offers better accuracy, quicker fabrication time and lower cost when compared to grinding and polishing. The high demand for quality surfaces in optical and electronic industry has been consistently pushing and breaking the barriers in various nanotechnology areas such as SPDT. Such advances are essential in order to economically produce high quality semiconductor, ceramic and glass parts. SPDT is well known for providing mechanical and optical advantages due to the level of precision of the equipment and the diamond cutting tool. SPDT is an ideal material removal candidate in this study as it is capable of:

- machining in the ductile regime: able to control several parameters precisely such as depth of cut, cutting speed and feed, allowing ductile mode machining
- precisely controlling the material removal process in the nanometer range
- cost efficient (tools can be reused after relapping)
- time efficient: the limit of ductile regime machining can be pushed to minimize the number of passes unlike polishing where only very little material can be removed at once, and
- providing good surface finish (Ra < 40 nm can be achieved)
1.5 MACHINING EQUIPMENT

Although several pieces and types of equipment were used throughout the course of this study, the two that were extensively used were the Universal Micro-Tribometer and the Micro-Laser Assisted Machining system.

1.5.1 UNIVERSAL MICRO-TRIBOMETER (UMT)

Almost all the material removal tests discussed in this dissertation were performed on the Universal Micro-Tribometer (UMT) which is produced by the Center for Tribology Research Inc. (CETR). This equipment was developed to perform comprehensive micro-mechanical tests of coatings and materials at the micro/nano-scale. This system facilitates cutting speeds as low as 1 µm/sec at nanometric cutting depths. The Tribometer is a load controlled device where the required thrust force is applied by the user to obtain the desired depth of cut (based on the tool geometry and workpiece material properties). The equipment includes a dual-axis load cell that is capable of monitoring the thrust and cutting forces (obtained as an output parameter from the cutting experiment). The main reason for choosing the UMT is due to its versatility where it facilitates quick setup changes to perform various forms of testing such as scratch tests, indentations and SPDT. These setups and tests will be discussed in detail in the following chapters.

1.5.2 MICRO-LASER ASSISTED MACHINING SYSTEM

Since one of the key focuses of this study is to determine the proof of concept and advantages of micro-laser assisted machining (µ-LAM), a µ-LAM system comprising a
laser source, fiber optic cables, specially designed optics and a custom made diamond tool are used to preferentially heat the workpiece during the material removal process. A total of three continuous wave (cw) infrared (IR) fiber lasers are utilized for this study. The IR beam is ideal in this case as most of the semiconductors studied are almost transparent to these wavelengths at its atmospheric or room condition phase. The main goal here is to preferentially heat only the high pressure phases (since these are the ductile phases where plastic deformation occurs), which is believed to be absorbing the IR beam. The specifications of the laser systems used in this study are summarized in Table 1.

Table 1-1: Laser specifications for the IR fiber lasers used in this study.

<table>
<thead>
<tr>
<th>Laser Device</th>
<th>Wavelength, $\lambda$</th>
<th>Power Range</th>
<th>Beam Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furukawa</td>
<td>1480 nm</td>
<td>0 – 400 mW</td>
<td>10 $\mu$m</td>
</tr>
<tr>
<td>Visotek</td>
<td>970 nm</td>
<td>0 – 10 W</td>
<td>100 $\mu$m</td>
</tr>
<tr>
<td>IPG*</td>
<td>1070 nm</td>
<td>10 – 100W</td>
<td>10 $\mu$m</td>
</tr>
</tbody>
</table>

*Operates with a beam delivery optics (BDO) unit by Laser Mechanisms Inc.

The various setups, testing methods and the actual science behind the $\mu$-LAM process will be discussed in detail in the subsequent chapters.

1.6 MEASUREMENT TECHNIQUES

When it comes to inspecting and measuring micro/nano-scale features, there are limits that are being pushed back by developments in the metrological technology. In nanomachining of brittle materials, it is important to choose the right measurement technique to monitor and evaluate the material removal process. It is also equally important for the measuring instrument to have a high enough magnification and
resolution to image, measure and analyze features at the nano-scale. Several challenges are faced when selecting suitable measurement equipment and techniques including the surface finish of the measured area (i.e., white light interferometers in general require a smooth and reflective surface to obtain an accurate and reliable measurement), feature size and required resolution, working distance, sample optical properties, time of measurement (post processing or in-situ) and sample preparation (e.g., some techniques like electron microscopy require the sample to be small in size and sometimes conductive). Measurement techniques utilized in this study are not limited to optical and electromagnetic techniques, but also contact measurement and sensor techniques such as stylus based profilometry, atomic force microscopy, acoustic emissions and strain gage based load sensors. The measurement techniques used throughout this research program include high resolution optical microscopy (to measure feature sizes down to 200nm), white light interferometry (to measure depth profiles of features and surface finish of substrates at the nano meter level), atomic force microscopy (to measure surface features such as depth of cut and cracks that are less than 100nm), scanning electron microscopy (to obtain a three dimensional view and measure features at a very high magnification and resolution), stylus profilometry (to measure rough surfaces), load sensors/cells (to measure/monitor forces and detect fracture during the material removal process), acoustic emissions sensors (to detect the onset of brittle fracture during the material removal process) and liquid temperature lacquers (to estimate the temperature of the laser assisted material removed zone).
1.7 DISSERTATION STRUCTURE

This dissertation consists of seven different projects or sub-studies that are discussed in individual chapters (Chapters 3 through 9). All seven chapters focus on a common aim, which is to study the ductile mode material removal process in nominally brittle materials with an emphasis on experimental analysis. These studies contribute not only to optimizing and improving the manufacturing machining process of brittle materials, but also in understanding the science behind the ductile mode material deformation and removal process. A major part of this dissertation (Chapters 5, 6, 7 and 9) consists of experimental work performed to study the proof of concept, potential feasibility and advantages, and understand the science behind the micro-laser assisted machining process. Although nine different materials are experimented on and discussed in this dissertation, the primary focus is on single crystal Si and SiC (single crystal 4H and 6H, and polycrystalline 3C). Combinations of various testing, imaging, measurement and characterization methods have been utilized throughout this study. A brief overview of the individual sub-studies (chapters) is given in the following sections.

1.7.1 SUBSURFACE DAMAGE ANALYSIS OF SINGLE POINT DIAMOND TURNED CVD-SILICON CARBIDE AND QUARTZ

This study investigates the subsurface damage of CVD-coated SiC and quartz after a SPDT operation. The samples were investigated under an optical microscope after the machining operation and no signs of surface damage were identified. The surface roughness of all samples continued to improve after SPDT, which further confirms ductile regime machining. However, at the initial stages, no attempts were made to
observe the subsurface conditions of the machined regions. The aim of this study is to make sure that ductile regime machining has not caused any subsurface damage such as micro-cracks. If no subsurface damage is observed, then this would confirm the initial assumption that since no surface damage is seen therefore subsurface damage will not occur for this particular machining process. This would also prove that the term (and theory) “ductile regime machining” is true for both surface and subsurface conditions. A total of three samples were investigated: (1) single point diamond turned CVD coated 3C-SiC, (2) single point diamond turned quartz (fused silica), and (3) laser ablated and single point diamond turned CVD coated 3C-SiC. Subsurface damage analysis is carried out on the machined samples using NDT techniques such as optical microscopy, Raman spectroscopy and scanning acoustic microscopy (SAcM) to demonstrate that the chosen material removal method (SPDT) leaves a damage-free surface and subsurface.

1.7.2 THE SIGNIFICANCE OF CRYSTAL ORIENTATION ON THE DUCTILITY OF POLYCRYSTALLINE CVD-3C-SILICON CARBIDE

This study determines the effect of crystal orientation relative to fracture damage, tool wear and friction in the machining process. The experimental program aims to quantify these observations, and result in a substantial increase in knowledge relative to the effect of the chemically vapor deposited crystal orientation on the ductile and brittle behavior of the material. Single point diamond turning was performed on a CVD coated silicon carbide (SiC) material to improve, i.e., decrease, its surface roughness. The experimental results suggest that the CVD polycrystalline 3C-SiC is much more ductile compared to the single crystal 4H and 6H SiC. The outcome of this project is an
evaluation of the crystal orientation in 3C-SiC and correlating it with the enhanced ductility for this particular polytype. Two non destructive techniques were utilized to study the crystal orientation of the material: (1) Orientation Imaging Microscopy (OIM) and (2) X-ray Diffraction (XRD). The results from XRD were preferred as it yielded in a stronger diffraction signal compared to OIM.

1.7.3 THE EFFECT OF LASER HEATING ON THE MATERIAL DEFORMATION AND REMOVAL OF SINGLE CRYSTAL SILICON

This topic focuses on studying the effect of laser heating during and after the micro-laser assisted material removal process. Besides studying the effect of laser heating, the feasibility of the μ-LAM system during the machining of silicon is also studied. A study is done to compare the results of scratch tests done on single crystal Si, with and without laser heating. The effects of laser heating are studied by verifying and comparing (to the no laser heating condition) the depths of cuts, ductile-to-brittle transition depths, thrust and cutting forces and surface/subsurface characteristics via scratch tests. Optical microscopy images, force data (cutting and thrust) and cross-sectional cutting profiles using a white light interferometer are correlated in this study. The experiments are done using a single crystal diamond cutting tool (1 mm nose radius with a -45 degree rake and 5 degree clearance angle) to evaluate the machining (depth of cut, feed and cutting speed) and laser heating (laser power) parameters. Once the material removal experiments were completed, characterization analyses were carried out using SEM, white light interferometry, TEM, and micro-Raman spectroscopy. The effect of using optimized laser powers on the depth of cut and high pressure phases are also
discussed where it is observed that optimized laser powers completely anneal the high pressure phases leaving behind only the original diamond structure.

1.7.4 THE EFFECT OF LASER HEATING ON THE MATERIAL DEFORMATION AND REMOVAL OF SILICON CARBIDE: 4H, 6H AND 3C

An extensive study is done to (1) study the thermal softening effect on SiC and (2) analyze the feasibility (demonstrate the proof of concept) of the μ-LAM system in the ductile mode machining process. Scratch tests done with and without laser heating at different conditions were compared. In this study, three different polytypes of SiC are studied: single crystal 4H, single crystal 6H and polycrystalline 3C (CVD coated). Since the as-received surface finish of the CVD-SiC is generally poor, tool wear becomes a major concern during the machining of such abrasive surfaces. A tool wear study is performed for this particular polytype in which two surfaces are machined (with and without laser) and the tools and surfaces are analyzed. Optical microscopy images, force data (cutting and thrust) and cross-sectional cutting profiles using a white light interferometer are correlated in this study. The experiments are done using a single crystal diamond cutting tool (1mm nose radius with a -45 degree rake and 5 degree clearance angle) to evaluate the machining (depth of cut, feed and cutting speed) and laser heating (laser power) parameters. Once the material removal experiments were completed, characterization analyses were carried out using SEM, TEM, and Micro Raman spectroscopy.
1.7.5 HIGH-PRESSURE PHASE TRANSFORMATION: PRESSURE AND TEMPERATURE EFFECTS

The results in this chapter address the fundamental (but not fully understood) science that correlates the pressure and temperature effect that takes place during the micro-laser assisted material deformation and removal process. Scratch and cutting tests are performed on Si with different parameters (i.e. laser power, cutting speed and applied load) and micro-Raman analysis is performed on these scratches and cuts to study (1) the high pressure phases formed and (2) the effect of cutting parameters on the high pressure phases. The annealing effect, from different laser powers, of the high pressure phases in the machined area and chips are also studied. To separate the temperature and pressure effect, indentations with different stages of laser heating were performed on Si. The primary goals of the indentation study is to (1) separate the temperature and pressure effect during the material deformation process and (2) identify the phase the laser heating is softening (i.e. atmospheric, high pressure phases or both). The indent depths were measured for each condition and correlated to the stage (e.g., loading, hold, unloading, etc.) heating was introduced. Micro-Raman analysis is also performed on the indents and later correlated with the scratch tests.

1.7.6 ACOUSTIC EMISSION TO DETECT THE ONSET AND POSITION OF CRACKS AND FRACTURE DURING MACHINING

In this study, AE is evaluated as a method for monitoring the onset of brittle fracture with respect to the relative cutting tool position and location of the cracking events. The broader goal of this study is to examine the onset of fracture using AE signal processing during micro/nano-scratch tests and machining of single crystal Si and SiC.
However, this study is not only limited to identifying the onset of fracture but also correlating the AE spectrum to the location and position of fracture, i.e., cracks (e.g., leading edge, trailing edge or beneath the cutting stylus/tool). The onset of brittle fracture (crack initiative and propagation) during the material removal process is studied using scratch tests. All scratch tests are conducted on the Universal Micro-Tribometer (UMT) by CETR that is equipped with an acoustic emission sensor. Three different band pass filters are utilized as the exact signal range of fracture behavior is not fully understood. The filter ranges include 2 kHz - 200 kHz, 100 kHz - 2 MHz and 500 kHz - 5 MHz. The research goal is to firstly evaluate if the AE system is sensitive enough to detect cracks and fracture behavior, and secondly correlating the AE spectrum to the fracture activity along the cut.

1.7.7 DUCTILE-TO-BRITTLE TRANSITION (DBT) OF CERAMICS AND SEMICONDUCTORS

This chapter discusses results from experimental work carried out to determine the DBT depth of several ceramics and semiconductors of interest, such as silicon carbide (SiC), silicon (Si), quartz (SiO₂), AlON (AlₓOᵧNz), sapphire (Al₂O₃), spinel (MgAl₂O₄) and aluminum titanium carbide (AlTiC). Three different polytypes of SiC are evaluated, there are: single crystal 4H, 6H and polycrystalline 3C. The effect of laser heating on the DBT depth is compared for SiC (all three polytypes), Si and sapphire. Scratch tests are carried out using a 1mm nose radius tool (-45 degree rake and 5 degree clearance angles) to determine the DBT depth of the materials. Scratch tests were chosen to be the principle method of investigation in this study as it is a better candidate for evaluating machining
conditions than indenting because the mechanics during scratching are more applicable to the machining process such as SPDT. The scratch tests are performed with increasing loads (thrust force) to produce the ductile, DBT and brittle regimes. Once the scratch tests are completed, microscope images, force data and profilometer analyses are carried out and correlated in order to determine the DBT depth (with the corresponding cutting and thrust forces). Finally, micro-Raman spectroscopy on sapphire is also discussed. A total of five materials have been characterized using μ-Raman (Si, three SiC polytypes and sapphire). Since the μ-Raman results of Si (Chapters 5 and 7) and SiC (Chapter 6) are discussed in previous chapters, only the results for single crystal sapphire are discussed here (since this is the only chapter in which sapphire is discussed).
CHAPTER 2

RESEARCH BACKGROUND

Materials that are hard and brittle, such as semiconductors, ceramics and glasses, are among the most challenging to machine. When attempting to machine ceramics and semiconductors, such as silicon carbide (SiC), silicon (Si), quartz, etc., especially to improve the surface finish, it is important to carry out a “damage free” machining operation. This can often be achieved by ductile mode machining (DMM) or in other words machining a particular hard and brittle material in the ductile regime. Material deformation and removal processes can be considered in terms of fracture dominated mechanisms or localized plastic deformation. A fracture dominant mechanism for ceramics, i.e., brittle fracture, can result in poor surface finish and surface damage, and also compromises the material’s properties and performance. Therefore, in this study, brittle machining is not the preferred mode or focus of the research.

2.1 DUCTILE-REGIME MACHINING

The insight into the origins of ductile regime machining during single point diamond turning (SPDT) of semiconductors and ceramics was provided by the research done by Morris, et al. A detailed study of machining chips (debris) and the resultant surface was analyzed using TEM to evaluate evidence of plastic material deformation. This seminal research concluded that the machining chips were plastically deformed and
were amorphous (not due to oxidation), due to the back transformation of a pressure
induced phase transformation and contained small amounts of micro-crystalline (brittle)
fragments.

According to the research carried out by Bifano et al.\textsuperscript{38}, there are two types of
material removal mechanisms associated with the machining process: ductile (plastic
flow of material in the form of severely sheared machining chips) and brittle (material
removal through crack initiation and propagation). This research also discusses several
physical parameters that influence the ductile to brittle transition (DBT) in the grinding of
brittle materials. The researchers were successful in performing ductile mode grinding on
brittle materials. However, these researchers did not propose or consider a model or
suitable explanation for the origin of this ductile regime. Bifano et al. later proposed a
model defining the ductile to brittle transition of a brittle material based on the materials
brittle fracture properties and characteristics. A critical depth of cut model was
introduced based on the Griffith fracture propagation criteria. The critical depth of cut
\((d_c)\) formula is as follows:

\[
d_c = \frac{(ER)}{H^2} \tag{2.1}
\]

where \(E\) is the elastic modulus, \(H\) is the hardness and \(R\) is the fracture energy. The value
of the fracture energy \((R)\) can be evaluated using the relation:

\[
R \sim \frac{K_c^2}{H} \tag{2.2}
\]

where \(K_c\) is the fracture toughness of the material. The above two equations can be
combined to represent the critical depth \((d_c)\) as a measure of the brittle transition depth of
cut:
The researchers were successful in determining a correlation between the calculated critical depth of cut and the measured depth (grinding infeed rate). The estimated constant of proportionality was approximated to be 0.15 and this is now added into Equation (2.3) to generate a more accurate empirical equation:

\[ d_c \sim 0.15 \left( \frac{E}{H} \right) \left( \frac{K_c}{H} \right)^2 \]  

The constant of proportionality accounts for the difference in properties (i.e. hardness and toughness) of the material’s atmospheric phase condition (atmospheric pressure) versus the properties of the high pressure transformed phase. For example, it is well known that single crystal Si (Si-I) undergoes pressure-induced phase transformations during mechanical loading using diamond anvil cell (DAC) or nanoindentation tests.\(^{39,40,41,42,43}\) The Si-I transforms to the metallic β-Sn (Si-II) phase under a load of up to 12GPa.\(^{44}\) Only the metallic phase in this case contributes to the plastic deformation (ductile material removal) of the material. However, it is important to note that the hardness at both phases (Si-I and Si-II) are different due to the difference in material and micro-structural properties. Upon pressure release, Si-II undergoes a further phase transformation to a mixed-phase of Si-III (bc8, body-centered-cubic structure) and Si-XII (r8, rhombohedral structure) at a low unloading rate while it transforms to the a-Si phase at a fast unloading rate [3, 7, 8].\(^{45,46}\) The estimated constant of proportionality in Equation (2.4) accounts for the differences in material properties that occur in various phases in brittle materials.
2.2 CRITICAL DEPTH OF CUT

A critical depth (d_c) should be determined before any ductile mode machining operation is carried out. Any depth exceeding the critical depth, which is also known as the ductile-to-brittle transition (DBT) depth, will exhibit brittle fracture. The extent of brittle fracture ranges from the onset of fracture (at the DBT zone) to a fully fractured condition, as the depth is increased beyond the DBT depth. Since the equipment used in the current research program (Universal Micro-Tribometer by CETR) is a load controlled (as opposed to depth controlled) machine, thrust force calculations were carried out for corresponding required depths of cuts. The Blake and Scattergood ductile regime machining model (as shown in Figure 2.1) was used to estimate the required thrust force for a desired depth of cut. In this model it is assumed that the undesirable fracture damage (which extends below the final cut surface) will originate at the critical chip thickness (t_c), and will propagate to a depth (y_c). This assumption is consistent with the energy balance theory between the strain energy and surface energy. However, the estimation from this model is usually about 50% off the actual value as it does not take into account the phase transformation and volume change (~20%) due to the high compressive stresses.
In general, the DBT is a function of several variables such as tool geometry (rake and clearance angle, nose and cutting edge radius), feed rate, and depth of cut.

2.3 HIGH-PRESSURE PHASE TRANSFORMATION (HPPT)

Although materials such as Si and SiC are naturally very brittle, micromachining these materials is possible if sufficient compressive stress is generated to cause a ductile mode behavior, in which the material is removed by plastic deformation instead of brittle fracture. This micro-scale phenomenon is also related to the High Pressure Phase Transformation (HPPT) or possibly direct amorphization of the material. Figure 2.2 shows a graphical representation of the highly stressed (hydrostatic and shear) zone that result in ductile regime machining.

Patten and Gao\textsuperscript{48} state that ceramics in general undergo a phase transformation to an amorphous phase after a machining process. This transformation is a result of the 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 both the workpiece surface and within the machined chip. The amorphous remnant is a result of a back transformation from the high pressure phase to the atmospheric pressure phase due to the rapid release of pressure in the wake of the tool.

There are two types of material removal mechanisms: ductile mechanism and the brittle mechanism. 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 depth) associated with these nominally brittle materials before attempting a machining operation.

Figure 2.2: A ductile machining model of brittle materials showing.

Figure 2.2 shows the plastically deformed chip formation and HPPT zone which is depicted by the blue area in the schematic. The schematic illustrates a ductile cutting model highlighting the high compressive stress and plastically deformed material behavior in nominally brittle materials. A -45° rake angle tool is demonstrated in the schematic. A more negative rake is used to generate higher compressive stresses that
induce the phase transformation process. The region above the HPPT zone (in the chip) and behind the tool (i.e., the resulting machined surface) shown in Figure 2.2 would not be under such high pressures, and thus would back transform to the low pressure phases.

2.4 SURFACE CHARACTERISTICS, FINISH AND SUBSURFACE DAMAGE ANALYSIS

The surface characteristics such as surface finish (roughness) are of major concern for all projects (directly or indirectly) in this study. The ultimate goal for all experiments carried out in this research is to improve the surface finish of a given ceramic/semiconductor. Surface characteristics or finish play an important role in this research as all of the materials studied may require mirror-like finish, for example, in order to maximize the optical qualities for high power laser devices (optical mirrors and windows). It is essential to understand the surface behavior, properties, pattern, characteristics and topography in order to optimize an effective machining process. In general, a surface roughness measurement is done to understand the surface topography.\textsuperscript{50} For the ceramic and semiconductor samples used in this study, the surface roughness is measured using a Mitutoyo Surftest (SJ-401) surface profilometer (stylus contact) and a Wyko (Rough Surface Tester – RST) interferometric profilometer before and after machining/material removal operations. Four main surface roughness parameters are generally used for analyses purposes. These parameters include Ra (Roughness Average), Rq (Root Mean Square (RMS) Roughness), Rz (Average Peak-to-Valley of the Profile) and Rt (Maximum Height of the Profile).
The initial goal when trying to improve the surface roughness of an as-received (not previously ground or polished) ceramic is to reduce its peak-to-valley (Rz) values. The effect of Rz are shown in Figures 2.3, 2.4 and 2.5 where the peak-to-valley of a small section of the workpiece is schematically modeled, in other words, the model is exaggerated to help in visualizing. If the average of the peaks are fairly high (e.g., Rz > 15 µm for the CVD coated SiC explained in Chapter 7), then, several machining passes have to be carried out in order to obtain a smooth or reflective surface (where the maximum depth of cut is determined by the DBT depth of the material, and may only be in the micrometer range). The as-received surface of a CVD coated SiC workpiece is used in this model as an example.

Figure 2.3: A model of a tool positioned before cutting through the rough as-received surface.

Figure 2.4: Tool positioned at the beginning of the second pass.
At this point the tool has completed the first pass and is positioned to begin the second pass over slightly smoother peaks of the unpolished surface.

![Image](image.png)

**Figure 2.5**: Tool positioned for the third pass over the fairly flat surface.

_Figures 2.3, 2.4 and 2.5_ compare the surface topography (peaks and valleys on surface) before and after each machining pass. In general, the surface is shown to be improving as long as the material is removed in the ductile regime (plastic deformation). This trend is consistent with the machining experiments performed in the ductile regime throughout this study. The initial roughing pass concentrates more on removing the major peaks on the surface. The depth of cut of a smoother (flatter) surface as seen in _Figure 2.5_ reduces due the greater contact area between the tool and workpiece surface, using the load control.

There are several factors that could affect the surface quality of the workpiece during machining, including tool geometry, tool wear/damage, external vibration (from the tool assembly or spindle), cutting speed, feed, depth of cut, cutting fluids and friction forces. Fritz Locke et al. in Jahanmir’s book on machining of ceramics\(^5\) describe the correlation between the machining parameters and the produced surface finish. It is stated that the surface quality of the workpiece tends to worsen at higher feeds and this finding
is consistent with the results obtained from this research. The tool nose radius plays an important role in the surface finish of the workpiece. In general, a larger tool nose radius and a smaller feed yields in a better surface. This relationship is given by the equation:

\[ H_{\text{max}} \sim \frac{f^2}{8R}, \quad \text{for } f \ll R \]

where \( H_{\text{max}} \) is the predicted theoretical surface roughness, \( f \) is the feed and \( R \) is the tool nose radius. Generally, the predicted theoretical surface roughness values are significantly less than the actual measured values, as the calculation does not take into account the high pressure phase transformation phenomenon, volume changes due to the phase transformation, minimum chip thickness, tool geometry, actual tool contact area, etc. It is also important to realize that surface roughness degrades with machining time due to tool wear development.\(^{52}\)

It is known that during hardness indentation tests, ceramics and semiconductors are subjected to highly localized stresses that not only could initiate crack formation and plastic deformation but could also cause a change in crystal structure and formation of an amorphous phase.\(^{53}\) This similar concept of contact induced pressure also applies while scratching and cutting, as ceramics undergo an extremely high pressure phase transformation during machining\(^{54}\). Due to the high pressure phase phenomenon, it is possible that subsurface damage occurs in these brittle materials without any indication of surface damage. To eliminate the possibilities of subsurface damage, several non destructive testing (NDT) techniques such as optical microscopy (only for optically transparent materials such as Quartz), scanning acoustic microscopy (SAcM) and Raman spectroscopy were used to analyze the material beneath the machined surface. The specific uses of these NDT techniques are discussed in detail in subsequent chapters. The
aim of these analyses is to make sure that ductile regime machining has not caused any subsurface damage such as voids and micro-cracks.

2.5 DIAMOND TOOL HARDNESS AND WEAR

Due to the hardness and abrasiveness of ceramics and semiconductors, tool wear becomes a major concern when attempting to perform SPDT, as diamond is only about four times ‘harder’ than SiC (100 GPa vs. 25-27 GPa). Although 25-27 GPa is the micro-hardness obtained from past researchers, in this study it is shown that the nano-hardness of SiC can range between 55 – 70 GPa (indent depths below 50 nm) depending on the surface characteristics and polytype. This then makes the tool-to-workpiece hardness ratio less than two for a nanometric cutting process. In precision machining, tool wear is usually observed at the micron level (at times even a few hundred nanometers) as even minor tool wear could play a huge role in changing the surface finish of the workpiece. Generally, the surface finish of the as-received diamond tool is extremely smooth (Ra ≈ 3nm) and this makes it an ideal candidate for machining smooth surfaces for optical devices.

It is important to understand that even minor tool wear can result in a poorly machined surface as this wear is replicated onto the workpiece surface at every revolution/feed. In order to be able to plastically deform brittle materials, an extremely sharp cutting edge radius of the single crystal diamond tools are used. Single crystal diamond tools work reasonably well when machining hard and brittle semiconductors as demonstrated by Jasinevicius et al. in their attempt to cut single crystal Si. Usually, a worn or blunt tool increases the tensile stresses, which leads to brittle fracture (due to
rubbing on the flank face). A blunted tool also reduces the compressive stress that is needed to insure the HPPT in achieving ductile regime machining.

In SPDT, three types of wear occur: rake-face wear, clearance-face wear (flank wear) and tool cutting edge radius wear (rounding or flattening of tool tip). The two most critical types of wear; rake and clearance wear were proposed by Hurt and Decker in order to understand the role of wear in surface degradation.\(^\text{58}\) High compressive stresses exist on the rake face of the tool and are at maximum at the cutting edge.\(^\text{59}\) It is observed that any wear on the clearance/flank face can potentially lead to increased surface roughness. This is consistent with the wear pattern from the SPDT experiments done in this research where the measured flank wear is always greater than the rake wear (as observed in Figure 2.6). Figure 2.6 shows an SEM micrograph of a worn tool taken after single point diamond turning an as-received CVD coated SiC surface. Since the actual cutting edge radius in not visible, a projected edge radius is shown, projected based on edges outside the field of view.

![Figure 2.6: Greater flank wear measured after performing SPDT on CVD-SiC.](image-url)
In order to improve the surface of the workpiece at every cutting pass, the cutting edge is re-sharpened by lapping when signs of tool wear are observed. It is essential to control and minimize the wear of the diamond tools in order to improve the performance and quality of micro or nano-machining in terms of the surface finish and the cost to replace or repair the worn tool. The schematic shown in Figure 2.7 helps understand the tool wear phenomenon in a typical ductile regime cutting process.

![Figure 2.7: Schematic showing the modeled rake and flank wear of the tool.](image)

The schematic in Figure 2.7 demonstrates the physics of chip rubbing that causes the rake face wear and contact with the machined surface that contributes to the flank/clearance wear. This is also consistent with the wear pattern from the SPDT experiments done in previous experiments where the measured flank wear is always greater than the rake wear as the contact area between the tool and the machined area is significantly larger than the contact area between the tool and the chip. This is illustrated in Figure 2.7, where the rake wear region is less than the flank wear due to the amount of contact area of the rake and flank face with the chip and machined area, respectively.
During the machining process, cutting tools are loaded with extremely high forces resulting from the deformation process in chip formation and friction between the tool and workpiece. All contact surfaces are usually clean and chemically very active; therefore the cutting process is connected with complex physical-chemical processes. Wear on the tool, which occurs as the consequence of such processes, is reflected as progressive wearing of particles from the tool surface. Tool wear is generally considered to be a result of mechanical (thermo-dynamic wear, mostly abrasion) and chemical (thermo-chemical wear, diffusion) interactions between the tool and workpiece. There are also different types of wear mechanisms acting on the tool surface during the cutting process that contribute to high cutting forces, high surface roughness, poor dimensional tolerances and lack of shape accuracy. Figure 2.8 summarizes all the particularities in tool wear during an abrasive cutting process.

Figure 2.8: Overview of the causes, mechanisms, types and consequences of tool wear.
2.6 CUTTING TOOL GEOMETRY

The geometry of the tool also plays an important role in machined surface quality, cutting forces, pressure and stresses induced, ductile to brittle transition limits, and depths of cuts. The two main geometric considerations evaluated when deciding on the tool geometry in general are the rake angle and cutting edge radius. According to the research done by Blackley and Scattergood\textsuperscript{63}, the critical depth of cut for germanium increased with a more negative rake angle (0\degree, -10\degree and -30\degree rake angles were experimentally evaluated). The results of this research was further complemented by the research work of Shibata et al.\textsuperscript{64} where diamond turning of single crystal Si was attempted with both -20\degree and -40\degree rake angled tools. The results from this research suggested that the -40\degree rake tool produced a perfectly ductile cut at a 100 nm depth of cut. \textit{Figure 2.7} can be used as a reference as it demonstrates the cross-sectional view of the tool geometry and chip formation.

Yan et al. concluded in their research work that tool damage can be classified into two types: micro-chipping and gradual wear. These results suggest that gradual tool wear is caused by ductile mode machining and micro-chipping of the tool is caused by brittle mode machining. The tool is believed to fail in ductile mode cutting when the flank wear ‘land’ becomes rough due to the micro-grooves and step structures. The tool wear obtained from both modes of machining significantly influences the surface finish (surface roughness), cutting forces and chip formation.\textsuperscript{65} This is because a chipped tool will induce unintended features to the machined surface at every repetitive feed, whereas a worn tool will mostly result in rubbing (and not cutting) where insufficient material removal is observed.
In the research of Patten et al. in decreasing the rake angle to $-85^\circ$, the results showed that both edges of the cutting tool can be used (leading or rake face, and trailing or flank face) to perform ductile mode machining.\textsuperscript{66} This work was then extended to vary the tool rake angle by adjusting the center line of the tool’s cutting edge, the effective rake angle was determine to be $-45^\circ$ ($0^\circ$, $-15^\circ$, $-30^\circ$ and $-60^\circ$ were also available).\textsuperscript{48} The relative amount of ductile cutting was found to increase while the brittle fracture decreased as the rake angle decreased from $0^\circ$ to $-45^\circ$. A more brittle behavior was observed in the cutting mechanism as the depth of cut increased from 100nm to 500nm. Also, in the research of Yan et al., a $-60^\circ$ rake angle tool is used to machine single crystal Si and significantly thick amorphous layers (attributed to ductile mode machining) were produced.\textsuperscript{67} The amorphous remnant in machined Si was observed under a TEM and characterized using Raman spectroscopy.

Finally, the cutting edge radius is also known to have an impact on the DBT of the material. This was reported by Fang et al.\textsuperscript{68} where the cutting edge radius is considered sharp as long as the depth of cut is larger than the edge radius. If the depth of cut is less than the cutting edge radius, then an effective rake is created.\textsuperscript{65} The study of this research suggests that an excessive rake can create/generate higher forces which may lead to additional deformation. For all tools used in this study, the assumed cutting edge radius range is between 20-100 nm. The cutting edge radius is not measured in this study but always assumed to be sharp after a relapping/re-sharpening process.

The tool nose radius, one of tool geometry parameters, has not been systematically investigated, probably due to its intuitive effects on part surface finish; the larger the tool nose radius, the finer the surface finish. Shintani and Fujimura reported
that tool life based on flank wear increases with nose radius, however, it reaches a constant for a nose radius greater than 0.4 mm. On the other hand, tool life based on surface finish showed a local maximum at 0.8 mm nose radii. It was suggested that large nose radii result in severe groove wear, and therefore poor surface finish. Other than surface finish aspects, tool nose radius also affects the uncut chip geometry, and thus, the ratio of uncut chip thickness to the edge radius that may affect the plowing forces in a hard turning process. More interestingly, the distance from the cutting edge to the nominal machined surface changes across the cutting edge and is a strong function of the tool nose radius Figure 2.9, where the uncut chip thickness (h) and the distance (g) from the cutting edge to the final machined surface are illustrated. This variable may also affect the DBT and high pressure phase transformation and sub surface damage depths as indicated in a previous study.70

![Figure 2.9: Local view of the tool nose during machining.](image)

2.7 MACHINING FORCES

Two main forces generated while machining ceramics are cutting forces (F_c) and thrust forces (F_t). The orthogonal cutting model suggested by Blake and Scattergood72 is used to help visualize these coexisting forces. The third force, the feed force, is often
neglected as its magnitude is significantly smaller than the thrust and cutting forces. The depth of cut (d) is sometimes taken to be the uncut chip thickness (t).

![Diagram of orthogonal cutting model showing the direction of forces.](image)

Figure 2.10: Orthogonal cutting model showing the direction of forces.

The research done by Blake and Scattergood\textsuperscript{47} helps to establish a relationship between machining forces and further understanding the frictional force behavior during machining. This behavior is represented as:

\[ F_f = \mu \times F_n \]  \hspace{1cm} (2.1)

where the cutting force \(F_c\) is represented by the frictional force \(F_f\) and the thrust force \(F_t\) is represented by the normal force \(F_n\). Generally the coefficient of friction is less than one (1). However it is possible to obtain an apparent coefficient of friction \(\mu_a\) which is greater than one. The apparent coefficient occurs when the cutting forces are larger than the thrust forces at larger depths of cuts.

Patten and Gao state that higher cutting forces are a sign of ductile regime machining, as ductile mode machining requires more energy (and thus higher forces) to remove material by plastic deformation on a per-unit or volume basis (or at the same depth of cut).\textsuperscript{48} The energy required for brittle fracture is proportional to the crack surface...
area whereas the energy required for plastic deformation is proportional to the volume of the material removed.

2.8 CHALLENGES IN DUCTILE-REGIME MACHINING OF CERAMICS AND SEMICONDUCTORS

Semiconductors and ceramics share common characteristics of being hard and nominally brittle, which stems from their covalent and/or ionic chemical bonding and crystal structure. These materials are important in many engineering applications, but are particularly difficult to machine in traditional manufacturing processes due to their extreme hardness and brittleness. Ceramics have many desirable properties including excellent wear resistance, chemical stability, wide energy bandgap, high electric field breakdown strength, high maximum current density and high strength even at elevated temperatures. All of these properties make them ideal candidates for tribological, semiconductor, MEMS and optoelectronic applications. However, in spite of all these positive characteristics, difficulty occurs during machining and material removal which has been a major obstacle that limits the wider application of these materials. The plastic deformation of these nominally brittle materials at room temperature is much less than in metals, which means they are more susceptible to fracture during material removal processes. Surface cracks generated during machining are subsequently removed in lapping and polishing processes, which significantly increases the machining time and cost. Machining mirror-like surface finishes contributes significantly to the total cost of manufacturing and thus eventual part cost. In some cases, grinding alone can account for 60-90% of the final product cost.\footnote{In this context, developing a cost effective method to...}
achieve a flawless surface in ultra fine surface machining of an optical lens or mirror has become a challenge. In many engineering applications, products require a high quality surface finish and close tolerances to function properly. This is often the case for products made of semiconductor or ceramic materials. The real challenge is to produce an ultra precision surface finish in these nominally brittle materials at low machining cost.

Since ceramics have extreme hardness (i.e. the hardness of SiC is approximately 25-80% of the hardness of diamond), machining them with a diamond tool can be an extremely abrasive process. As a result of this abrasive material removal process, there are several limitations in terms of machining parameters that have to be considered to avoid brittle fracture of the material. The primary limitation in ductile mode machining is the critical depth of cut or the DBT depth of the material. Exceeding the DBT depth during the machining process will result in fracture, leaving a poor surface finish. Another important parameter during machining is the feed (cross-feed). In general, lower feed rates result in a better surface finish; however, lower feed rates also result in more tool wear due to the longer track length covered by the tool during machining a surface. Tool wear is crucial when trying to improve the surface finish of a ceramic or semiconductor. Any wear along the cutting edge (radius, rake and flank wear) will directly affect the machined surface finish, possibly causing cracks and fracture. A small chipped area or crack in the tool tip could potentially grow during the machining process, eventually causing the tool to fail. Tool failure can be observed as a change in the cutting forces during the machining process. In general, low cutting forces are desired to minimize the diamond tool wear. The micro-laser assisted machining (µ-LAM) process, which will be discussed in the Chapters 5, 6, 7 and 9, shows positive results in addressing
the challenges, such as low or small DBT depths, high hardness and high brittleness, faced in conventional ductile regime machining of hard brittle materials.\textsuperscript{75}

2.9 DUCTILE-REGIME MICRO-LASER ASSISTED MACHINING

Current limitations for brittle material machining include the high cost of processing and low product reliability. The cost is mainly due to expensive tools that wear out rapidly, long machining time, low production rate and the manufacturing of satisfactory surface roughness, figure and form. The low product reliability is primarily due to the occurrence of surface/subsurface damage, i.e., cracks, and brittle fracture. In order to develop a suitable process, ductile regime machining, considered to be one of the satisfactory precision machining techniques, has been continuously studied over the last two decades.\textsuperscript{76,77,78,79,80,81,82,83,84} Laser assisted micro/nano machining is another important development in this direction.\textsuperscript{85,86}

Past research has demonstrated that ductile regime machining of these materials is possible due to the high pressure phase transformation (HPPT) occurring in the material caused by the high compressive and shear stresses induced by the single point diamond tool tip.\textsuperscript{87} To further augment the ductile response of these materials, traditional scratch/single point diamond cutting tests are coupled with a micro-laser assisted machining (\(\mu\)-LAM) technique.\textsuperscript{88} A schematic of the basic underlining concept of the \(\mu\)-LAM process is shown in Figure 2.1. An IR beam (passed through a transparent diamond tool) is typically used in this process as Si and SiC are almost transparent at this wavelength. This allows the laser beam only to be absorbed by the high-pressure metallic phases resulting in enhanced ductility and reduced hardness during the material removal
This hybrid configuration thermally heats and softens the HPPT material, making it more ductile, and increasing the critical depth of cut (DoC) (larger DBT depth) in ductile regime machining, resulting in a higher material removal rate. Also, since the hardness of the contact area (high pressure phase) is reduced, this significantly contributes to prolonging the tool life. μ-LAM was successfully carried out on single crystal Si, SiC (single and polycrystalline) and single crystal sapphire yielding a greater DBT (for the scratch performed with laser heating).^{89}

![Figure 2.11: A schematic cross-section of the μ-LAM process.](image)

2.10 CRYSTAL ORIENTATION

Crystal orientation dependency during indentations and ductile regime machining has been studied for decades. Crystal orientation in a workpiece (wafer/disk) has a significant impact on the tribological performance of Si and SiC.^{90} Miller’s indices are commonly used to identify different crystallographic orientations including planes and directions. It has been suggested that the divergence of the various hardness values in SiC
might be attributed to improper techniques to initiate cracking around the indenter caused by vibration, excessive load or impurities.\textsuperscript{91} Winchell examined the hardness of natural faces of the basal pinacoid of both black and green SiC and reported a certain decrease in hardness midway between the \((10\overline{1}0)\) and the \((11\overline{2}0)\) orientations.\textsuperscript{92} In Winchell’s study however, the source or chemical composition of the crystals was not given.

In his research, Keyes studied the cleavage planes relative to the indenter tip loading direction.\textsuperscript{93} When struck by a blunt instrument on an \((0001)\) face, SiC can be made to cleave imperfectly along a series of prism faces \((11\overline{2}0)\). Keyes also noted that cleavage rarely occurred parallel to \((11\overline{2}0)\) faces and frequently conchoidal fracture occurred. He attributes this preferred cleavage direction in both prism zones to an ionic component of bonding as discussed by Jagodzinski.\textsuperscript{94}

The hardness of Si and SiC varies with the orientation and for all planes; the hardness anisotropy is dependent on the temperature. A study done by Niihara demonstrates that the active slip system of 6H-SiC is \((10\overline{1}0) <11\overline{2}0>\) near room temperature and \((0001) <11\overline{2}0>\) at high temperatures (> 800\(^\circ\)C).\textsuperscript{95} Fujita et al. and Maeda et al. have also conducted detail studies of dislocations in single crystal SiC.\textsuperscript{96,97} They concluded that plastic deformation occurred by the movement of \((0001) <11\overline{2}0>\) dislocations above 1000\(^\circ\)C for single crystals and about 1600\(^\circ\)C for polycrystalline SiC. Pirouz et al. have also extensively studied the mechanical and optical properties of single crystal SiC as a function of temperature by growing the substrates in a specific planes and directions.\textsuperscript{98} Noticeable differences in properties have been noticed with planes only 3\(^\circ\) to 4\(^\circ\) degrees off each other at elevated temperatures.\textsuperscript{99}
A few authors have also studied the relationship between crystal orientation and ductile regime machining of Si. In one study, both the indentation and ultra-precision machining of the (111) wafers were investigated. No cracks along any direction were found when the indentation load was below 2.5 N. Using diamond tools (0.637 mm nose radius; 0°, -15° and -24° rake angles) for machining at 1-10 µm depths and 1-2 µm/rev feeds, ductile regime cutting of Si was observed only when cutting at the lowest depths and feeds. However, at larger depths, pitting due to brittle cracks was found in the <112> direction, but not in the <110> direction.

The material orientation relative to chip formation has also been studied. A correlation of the cutting orientation, the shear angle, as well as the DBT was observed. Denote the short form used here, (xyz) <abc> as the measurement in an <abc> direction on the (xyz) plane. When a (001) Si wafer was machined at a 1 µm depth, there was least pitting in the (001) <110> locations. However, the opposite was observed when machining at a 100 nm depth. TEM examination showed a significantly thicker amorphous layer below the 100 nm machined depth (versus the fairly brittle machined region at a 1 µm depth) region as a fully ductile mode material removal process was established. However, it is important also to note that significantly more microcracks were seen when machining at a 1 µm depth.

2.11 NANOINDENTATION

The development of depth-sensing indentation techniques at a nanometric scale, commonly known as nanoindentation, has allowed highly localized hardness and modulus measurements to be performed on very small material volumes. Some previous
work has already shown the net advantages of nanoindentation as a unique tool for the characterization of tribological films.\textsuperscript{102} To achieve a nanometric surface finish, Si and SiC must be cut below the threshold load before the material can exhibit fracture behavior. In nanoindentation, a small force is applied to a material and the depth of indentation is measured. This technique is commonly used to study microcracks in brittle material.\textsuperscript{30} However, it is important to note that the load versus displacement curves (from nanoindentations) do not directly provide information about surface cracks, except where pop-in and pop-out events can be attributed to cracks (during loading and unloading respectively), but these events can also be due to HPPT and back transformations. Often times, electron microscopy techniques are used to study these cracks.

Due to the fact that mechanical contact conditions in an indentation test is geometrically akin to that in micro/nano-machining processes such as SPDT,\textsuperscript{103,104} the understanding of indentation-induced deformation, fracture and microstructural changes can contribute to the understanding of the optical, electrical and mechanical properties of Si and SiC, hence eliminating damages during machining.\textsuperscript{105} Over the past decade, indentation tests have been extensively used to study phase transformations in semiconductors (most commonly Si). Past researchers have found that the phase transformation mechanisms during indentation tests strongly depend on the indenter shape/geometry, loading/unloading rate, applied load, temperature and other factors.\textsuperscript{106,107,108,109,110} Most of these factors directly correspond to actual machining parameters, e.g., the indenter geometry can be correlated to the cutting tool geometry and the loading/unloading rate corresponds to the machining speed.\textsuperscript{10}
A detail study had been done by Cook and Pharr, where the initiation sequence of indentation cracks are investigated in glasses and ceramics. Several crack types such as cone, radial, median, half-penny and lateral were observed using an inverted tester that allows simultaneous viewing of the fracture process and measurement of the indenter load and displacement during contact. It is found that for specific cracks formed and the sequence they form in is strongly influenced by the materials Young’s modulus and hardness. Also a different cracking sequence is observed between crystalline materials and glasses.

2.12 SCRATCH TESTING

Understanding the abrasive resistance of ceramics and semiconductors is essential for a better selection of candidate materials and machining parameters. Single-point scratches are one of the widely used test methods, and a single-point diamond tool can make either a translational motion (creating a groove of constant or variable depth) or circular motion in the form of an arc (usually creating a groove with variable depths). Traditionally, abrasion resistance of materials has been quantified by scratch hardness tests, which is defined as the resistance of a body to penetration by a tangentially moving sharp edge. By analyzing the induced forces and the characteristics of the resulting groove/cut, the fundamental material removal process has been studied and correlated with abrasive wear (surface and tool), machining parameters (forces, depth of cut, speed, etc.) and scratch resistance.

Unlike a relatively simple indentation test, where only normal loads are imposed, scratch tests allow for both normal and tangential forces to be applied. This then better
mimics the actual machining process providing fundamental information on material deformation and removal between two contact surfaces prone to wear. Depending on the process conditions, material properties and microstructural features of the ceramic or semiconductor, the material removal and deformation mechanism and incurred damage/wear can be quite different.\textsuperscript{118,119,120,121,122}

2.13 MICRO-RAMAN SPECTROSCOPY

Micro-Raman (µ-Raman) spectroscopy is an effective non-destructive testing (NDT) technique used to characterize the amorphous remnant and high pressure phase transformed region in the material deformation and removal process (of the surface region) of ceramics and semiconductors. The Raman effect is based on the inelastic scattering of a laser beam. Micro-Raman spectroscopy is the only method that allows non-destructive phase analysis to be conducted in seconds with a spatial resolution of 1 µm (the sampling depth is also dependant on the wavelength of the laser beam) on a non-prepared surface or subsurface.\textsuperscript{123} Another advantage of µ-Raman is that the probing depth can be controlled based upon the wavelength of the laser used (i.e. shorter wavelengths result in shallower probing depths). The 244 nm wavelength (doubled Ar ion laser) is usually preferred when probing depths less than 50 nm (for Si and SiC).\textsuperscript{124} Raman analyses have been successfully used to study various phases in the Si-C-O system.\textsuperscript{125}

Micro-Raman was first combined with indentations to study the transformation toughening of ZrO\textsubscript{2} ceramics.\textsuperscript{126} In the mid 90’s, Lucazeau et al. studied the impressions in Si via Raman spectroscopy and reported only the amorphous remnant phase that was
previously known from TEM. Gogotsi et al. then performed an extensive study on numerous ceramics and semiconductors including diamond, soft graphitic carbon and Si. Silicon has been and is still one of the most extensively studied materials in scientific literature, and for which dozens of studies on high-pressure phase transformations have been published. Researchers such as Daibin, Domnich and Gogotsi have also utilized the laser power in the Raman system to perform in-situ annealing of the high pressure phases in Si.

2.14 ACOUSTIC EMISSION

Acoustic Emission (AE) refers to the generation of transient elastic waves produced by a sudden redistribution of stress in a material. When a structure is subjected to an external stimulus (change in pressure, load, or temperature), localized sources trigger the release of energy, in the form of stress waves, which propagate to the surface and are recorded by sensors. With the right equipment and setup, motions on the order of picometers ($10^{-12}$ m) can be identified. Sources of AE vary from natural events like earthquakes and rockbursts to the initiation and growth of cracks, slip and dislocation movements, melting, twinning, and phase transformations in ceramics and semiconductors. In composites, matrix cracking, fiber breakage and debonding contribute to acoustic emissions. AE’s have also been measured and recorded in metals, polymers, wood, and concrete, among other materials. AE’s originate with stress. When a stress is exerted on a material, a strain is induced in the material. Depending on the magnitude of the stress and the properties of the material, an object may return to its original dimensions or be permanently deformed after the stress is removed. These two conditions
are known as elastic and plastic deformation, respectively. Although AE signals are generated in both cases, plastic deformation often generates a more distinct signal with higher intensities.

The most detectible acoustic emissions take place when a loaded material undergoes plastic deformation or when a material is loaded at or near its yield stress. On the microscopic level, as plastic deformation occurs, atomic planes slip past each other through the movement of dislocations. These atomic-scale deformations release energy in the form of elastic waves which “can be thought of as naturally generated ultrasound” traveling through the object. When cracks exist in a material, the stress levels present in front of the crack tip can be several times higher than the surrounding area. Therefore, AE activity will also be observed when the material ahead of the crack tip undergoes plastic deformation (micro-yielding). Although the use of AE has been extensively studied in the plastic deformation during metal cutting, the focus of this project is to utilize AE as a tool to detect the onset of brittle fracture.

Two sources of fatigue cracks also cause AE’s. The first source is emissive particles (e.g. nonmetallic inclusions) at the origin of the crack tip. Since these particles are less ductile than the surrounding material, they tend to break more easily when the material is strained, resulting in an AE signal. The second source is the propagation of the crack tip that occurs through the movement of dislocations and small-scale cleavage produced by triaxial stresses.

The amount of energy released by an acoustic emission and the amplitude of the waveform are related to the magnitude and velocity of the source event. The amplitude of the emission is proportional to the velocity of crack propagation and the amount of
surface area created. Large, discrete crack jumps will produce larger AE signals than cracks that propagate slowly over the same distance.\textsuperscript{139} Detection and conversion of these elastic waves to electrical signals is the basis of AE testing. Analysis of these signals yield valuable information regarding the origin and importance of a discontinuity in a material. As discussed in chapter 8, specialized equipment is necessary to detect the wave energy and decipher which signals are meaningful.\textsuperscript{140} Collecting AE signal at the right frequency is also crucial in developing a sensitive and useful system for a specific application. In the research conducted by Koshimizu et al., a band-pass filter of 100 kHz – 1 MHz is used to detect the ductile-to-brittle transition during scratch testing of Silicon.\textsuperscript{141} However, it is important to note that only severe fracture events were recorded and not the actual onset of fracture itself; that is to say, detecting the onset of fracture was not the goal of the study.
CHAPTER 3

SUBSURFACE DAMAGE ANALYSIS FOR SINGLE POINT DIAMOND TURNED CVD-SILICON CARBIDE AND QUARTZ

3.1 INTRODUCTION

Silicon carbide (SiC) is used in specialized industries 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.\textsuperscript{36} The fully dense cubic (beta) polycrystalline CVD-coated ($\approx 250 \, \mu m$ thick) silicon carbide (manufactured by POCO Graphite) is a potential candidate to be used as mirrors for surveillance, high energy lasers (such as an airborne laser system), laser radar systems, synchrotron x-ray, VUV (vacuum ultraviolet) telescopes, large astronomical telescopes and weather satellites.\textsuperscript{13} The primary reasons CVD coated SiC is preferred for these applications is that the material possesses high purity (>99.9995%), homogeneity, density (99.9% dense), chemical and oxidation resistance, cleanability, polishability and thermal and dimensional stability. Machining SiC is extremely challenging due to its extreme hardness (micro-hardness $\approx 25$-$27$ GPa) and brittle characteristics. Besides the low fracture toughness of the material, severe tool wear of the single crystal diamond tool has to be considered.
Quartz, also known as silicon dioxide (SiO$_2$), is the most abundant nonmetallic mineral on earth. There are several forms of quartz such as quartz crystals, natural fused silica (amorphous form of SiO$_2$) and synthetic fused silica (polycrystalline). For this research experiment, an amorphous fused silica (Spectrosil 2000) is used. Spectrosil 2000 is an ultra pure synthetic fused silica manufactured by Saint-Gobain Quartz PLC. This quartz material has a wide optical range from 180 nm in the deep ultraviolet transmission through to 2000 nm in the infrared (IR). This material possesses a chemical purity of 99.999% and is manufactured using an environmentally friendly process, which results in a material that is both chlorine-free and bubble-free.

The mechanics of material removal in SiC and glass (quartz) can be classified in two categories: brittle fracture and plastic deformation. Good optical quality surfaces can be achieved by removing the material in a ductile manner. The work of past researchers suggests that glasses do not necessarily behave as brittle material (even at room temperature) especially at the nanometric scale. The strength, hardness and fracture toughness of the work piece material are the governing factors that control the extent of brittle fracture. Some studies include detailed observations of a small amount of plastic deformation in brittle materials during a precision machining operation.

Previous researchers have successfully been able to precisely grind CVD-SiC (using high precision grinding) but this process is very expensive and the fine abrasive wheels often result in an unstable machine/process. Single point diamond turning (SPDT) is chosen as the material removal method as SPDT offers better accuracy, quicker fabrication time and lower cost when compared to grinding and polishing. Although SiC and quartz are naturally brittle, micro/nano-machining these materials is
possible if sufficient compressive stress is generated to cause a ductile mode behavior, in which the material is removed by plastic deformation instead of brittle fracture. This micro-scale phenomenon is also related to the High Pressure Phase Transformation (HPPT) or direct amorphization of the material. The plastic deformation or plastic flow of the material, at the atomic to micro scale, occurs in the form of severely sheared machining chips caused by highly localized contact pressure and shear.

The main idea of this topic of study is to investigate the subsurface damage of CVD coated SiC and quartz after a SPDT operation. The samples are investigated under an optical microscope after the machining operation and no signs of surface damage had been observed. The surface roughness of all samples continued to improve after SPDT which further confirms ductile regime machining (cracks that form from brittle machining often degrades the surface). The aim of the current study is to confirm that ductile regime machining has not caused any subsurface damage such as voids and micro-cracks. If no form of surface damage is observed then this would further validate the initial assumption of no subsurface damage. This would then prove that the term/theory “ductile regime machining” is true for both surface and subsurface conditions. A total of three samples are investigated: (1) single point diamond turned 6” diameter CVD-SiC, (2) single point diamond turned quartz, and (3) laser ablated and single point diamond turned CVD-SiC. Subsurface damage analyses are carried out on the machined samples using non-destructive testing (NDT) techniques such as optical microscopy, Raman spectroscopy and scanning acoustic microscopy (SAcM) to show evidence that the chosen material removal method leaves a damage-free surface and subsurface. If the study proves successful (including the verification of no subsurface
damage), the results and machining methods could be used to benefit the manufacturing process associated with precision engineering and machining of ceramics and semiconductors.

3.2 EXPERIMENTAL METHOD

3.2.1 SPDT OF SAMPLES

It is known that during hardness indentation tests, ceramics are subjected to highly localized stresses that not only initiate crack formation and plastic deformation but also cause a change in crystal structure and formation of an amorphous phase upon release of the load (post indentation analysis).53 This similar concept of contact induced pressure also applies while cutting as ceramics undergo an extremely high pressure phase transformation during machining.54 Due to the high pressure phase phenomenon, it is possible that subsurface damage occurs in these brittle materials without any indication/evidence of surface damage. In this study, the amorphous layer is not considered subsurface damage, instead used as evidence to support a ductile mode machining process.

The main goals of machining SiC and quartz in this study are to improve the surface finish, increase the material removal rate (MRR) and minimize tool wear. All this is done using a SPDT operation. SPDT is chosen due to its low operating cost and short machining time. The universal micro-tribometer (UMT) is used for all SPDT operations in this study. A typical SPDT setup on the UMT for quartz (SPDT for other materials utilized a similar setup) is shown in Figure 3.1. The UMT is reconfigured with some modifications to perform SPDT operations.
The machining parameters, such as the depth of cut and feed, varied for each material and are discussed briefly in the respective sections. Details on surface finish and the impact of each machining parameter on the SPDT process have been previously reported\textsuperscript{150} and will not be discussed as it is not the focus of this study.

### 3.2.2 LASER RAMAN SPECTROSCOPY

Laser Raman spectroscopy is a well known non destructive characterization technique often used for semiconductors. Most of the past research and available literature show that Raman spectroscopy has been used to study machined silicon surfaces, spherical, conical, or pyramid diamond tips or indenters\textsuperscript{151,152}. However, in this study, Raman and micro-Raman (\(\mu\)-Raman) spectroscopy proved to be successful in observing the formation of an amorphous region in diamond turned SiC workpieces. A 633 nm wavelength He-Ne laser (for laser Raman) and an UV-325 nm wavelength He-Cd
laser (for \(\mu\)-Raman) with a 2 \(\mu\)m spot size is used to study the subsurface of the machined SiC regions. The main purpose of Raman spectroscopy in this study is to detect the amorphous layer (that further confirms ductile mode machining) beneath the machined surface.

3.2.3 SCANNING ACOUSTIC MICROSCOPY

SAcM is widely used in non-destructive evaluation (NDE) of materials utilizing high-frequency acoustic waves (60 MHz to 2.0 GHz) to reveal surface topography, subsurface features and elastic properties.\(^{153}\) The Kramer Scientific Instruments (KSI) SAM2000 scanning acoustic microscope at the Oak Ridge National Laboratory (ORNL) is used for this study to investigate subsurface cracks and fracture after SPDT, if any. The concept of SAcM is schematically illustrated in Figure 3.2. Acoustic waves are produced by a transducer, pass through the coupling liquid (usually distilled water), and reflect from the focal plane (located at a distance ‘\(z\)’ below the specimen’s surface). The reflected acoustic echoes from individual regions are detected during scanning and are used to assemble images.\(^{18,154}\)

![Figure 3.2: Schematic diagram of scanning acoustic microscopy.\(^{18}\)](image)

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\(^{153}\) The Kramer Scientific Instruments (KSI) SAM2000 scanning acoustic microscope at the Oak Ridge National Laboratory (ORNL) is used for this study to investigate subsurface cracks and fracture after SPDT, if any. The concept of SAcM is schematically illustrated in Figure 3.2. Acoustic waves are produced by a transducer, pass through the coupling liquid (usually distilled water), and reflect from the focal plane (located at a distance ‘\(z\)’ below the specimen’s surface). The reflected acoustic echoes from individual regions are detected during scanning and are used to assemble images.\(^{18,154}\)
3.2.4 OPTICAL MICROSCOPY

Light or optical microscopy is the primary means for scientist and engineers to examine the microstructure of material. Although optical microscopy is most often used to image surface features, in this study it is used to examine subsurface conditions by altering the focal distance (focusing below the surface). However, this technique is only possible for optically transparent materials such as quartz. Optical microscopy utilizes visible light and a system of lenses to magnify images of small samples/features. A standard optical microscope generally consists of an ocular lens (eyepiece), objective turret/revolver (to hold multiple objective lenses), objective lenses (with various magnifications), focus wheel to move the stage (coarse and fine adjustment), frame, light source (a light or a mirror), diaphragm and condenser lens and stage (to hold the sample).

The optical principles of microscopes include image formation, magnification and resolution. Image formation can be illustrated by the behavior of a light path in a compound light microscope as shown in Figure 3.3. A specimen (object) is placed at a position $A$ where it is between one and two focal lengths from an objective lens. Light rays from the object firstly converge at the objective lens and are then focused at position $B$ to form a magnified inverted image. The light rays from the image are further converged by the second lens (projector lens) to form a final magnified image of an object $C$. 
3.3 RESULTS AND DISCUSSION

3.3.1 CVD COATED 6” DIAMETER SiC - SPDT

Ductile mode SPDT is carried out on a 6” diameter disk, improving its surface roughness (Ra) from 1.25 µm (as-received) to approximately 85 nm in six machining passes.\textsuperscript{155} The machining parameters used in the six machining passes are summarized in Table 3.1. The machining parameters are chosen based on the material’s surface finish after every pass and the ductile-to-brittle transition depth (i.e., never exceeding the DBT depth, which is 550 nm for this material). The initial three passes yielded in actual depths of cuts that are larger than the DBT depth of the material. This is a good indication of possibly some brittle mode machining, however, from Figure 2.1 (Chapter 2), it is understood that any micro-cracks that do not extend beyond the surface damage depth ($y_c$) for that depth of cut will not make the surface roughness worse.

Table 3-1: Optimized parameters for improving the surface quality of a CVD-SiC.

<table>
<thead>
<tr>
<th>Pass #</th>
<th>Depth of Cut</th>
<th>Cross-Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.3 µm</td>
<td>30 µm/rev</td>
</tr>
<tr>
<td>2</td>
<td>1.2 µm</td>
<td>30 µm/rev</td>
</tr>
<tr>
<td>3</td>
<td>845 nm</td>
<td>5 µm/rev</td>
</tr>
<tr>
<td>4</td>
<td>255 nm</td>
<td>1 µm/rev</td>
</tr>
<tr>
<td>5</td>
<td>210 nm</td>
<td>1 µm/rev</td>
</tr>
<tr>
<td>6</td>
<td>160 nm</td>
<td>1 µm/rev</td>
</tr>
</tbody>
</table>
The machined surface showed no signs of surface damage such as cracks, pits and voids, therefore subsurface analysis is carried out to confirm the occurrence of a purely “damage-free” ductile mode machining process. Raman spectroscopy and scanning acoustic microscopy are two techniques chosen to detect the formation of the amorphous layer beneath the machined surface and attempt to image subsurface cracks (if any) respectively.

3.3.1.1 LASER RAMAN SPECTROSCOPY

The main purpose of Raman spectroscopy analysis in this study is to detect the amorphous layer beneath the machined surface.

![Laser Raman spectrum of the: (a) unmachined SiC, and (b) machined SiC surface.](image)

In Figure 3.4(a), the spectrum shows the crystalline peaks of the unmachined SiC sample. Comparing these peaks with Figure 3.4(b), it is seen that a combination of the crystalline peaks (sharp peaks) and amorphous peaks (broadband peaks) are formed in the machined surface. The amorphous layer is a good indication of a ductile material removal process and not treated as a form of subsurface damage in this chapter. In general, the thickness of the amorphous layer increases as the depth of cut is increased.
3.3.1.2 SCANNING ACOUSTIC MICROSCOPY (SAcM)

In this study, SAcM is carried out to investigate possible subsurface cracks/voids as results of SPDT operations.

![Figure 3.5: A sequence of images of the machined SiC sample.](image)

*Note: The first image shows the surface and the numbers on the top left of each image represent the scanned depth beneath the machined surface.*

The acoustic microscopy images show no signs of subsurface cracks or damage as deep as 1.5 µm, except normal surface features and feed marks. The surface features seen in the images in Figure 3.5 are pits and voids that existed in the as-received material and is not caused by the SPDT operation. The SAcM analysis is restricted to 1.5 µm below the surface as the images begin to lose resolution and definition beyond this depth of focus.

3.3.2 QUARTZ – SPDT

Ductile mode SPDT is carried out on a 6” diameter disk improving its surface roughness (Ra) from 110 nm (as-received) down to approximately 40 nm in two machining passes. Pass one is carried out at a 650 nm depth of cut and pass two is
carried out at a 360 nm depth of cut (DBT is determined to be approximately 120 nm). A 1 µm/rev feed is used for both passes as the surface is smooth enough (a larger feed would increase the surface roughness). The machining parameters used in the both machining passes are summarized in Table 1.2.

Table 3-2: Optimized parameters for improving the surface roughness of a Spectrosil 2000.

<table>
<thead>
<tr>
<th>Pass #</th>
<th>Depth of Cut</th>
<th>Cross-Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>650 nm</td>
<td>1 µm/rev</td>
</tr>
<tr>
<td>2</td>
<td>360 nm</td>
<td>1 µm/rev</td>
</tr>
</tbody>
</table>

From Figures 3.6(a) and (b), it can be seen that the machined surface showed no signs of surface damage and therefore subsurface analysis is carried out to confirm the occurrence of a purely “damage-free” ductile mode machining process. Optical microscopy and scanning acoustic microscopy are the two techniques chosen to detect and image subsurface cracks, if any are present in the machined surface.

Figure 3.6: Optical microscope images of the machined surfaces: (a) (400x) shows the feed marks of the machined region (left) after Pass 1 and (b) (1000x) shows very light feed marks on the machined region (left side of image) after Pass 2.
3.3.2.1 OPTICAL MICROSCOPY

Since the quartz workpiece used for this study (Spectrosil 2000) is visibly transparent, optical microscopy to a certain extent is utilized to image subsurface conditions. By offsetting the focus and setting the new focal point slightly beneath the machined surface, subsurface damage (if any) can be imaged for an optically transparent sample. Figure 3.7 shows an optical microscope image obtained after focusing below the machined surface (after pass two). No signs of brittle cracks or evidence of brittle machining is visible beneath the machined surface. Using a 400x magnification, a maximum scan depth of approximately 5 μm below the surface is imaged.

![Image](image.jpg)

**Figure 3.7: Image taken after focusing below the machined surface or subsurface.**

The features seen in *Figure 3.7* are regular feedmarks that can be removed via minimal polishing. Based on the images obtained from optical microscopy, no evidence of subsurface damage such as cracks is visible, indicating a ductile regime material removal process.
3.3.2.2 SCANNING ACOUSTIC MICROSCOPY

Figure 3.8: A sequence of SAcM images of the machined quartz sample.
Note: The first image shows the surface and the numbers on the top left of each image represent the scanned depth beneath the machined surface.

The acoustic microscopy images in Figure 3.8 show no signs of subsurface cracks or damage as deep as 1.5 µm, except normal surface features and feed marks. The feed mark features seem most obvious at scanned depths of 250 nm and 500 nm below the surface and tend to fade away at deeper scans. This is consistent and within the range of the total machined depth of approximately 1 µm. Based on the images obtained from SAcM, no evidence of subsurface damage is visible (besides feed marks) indicating a purely ductile regime cutting process.
3.3.3 CVD COATED SIC – LASER ABLATED AND SPDT

The broader aim of this particular set of experiments is to develop a hybrid laser ablation - SPDT machining process for chemically vapor deposited (CVD) SiC. The three main goals of this work are: (1) to improve the final surface quality (in terms of surface roughness and minimum surface and subsurface damage), (2) increase the material removal rate (MRR), and (3) minimize the diamond tool wear. The focus of this work is to study the combination of both methods (laser ablation and SPDT) and identify laser processing parameters (if any) that best facilitates the two step or hybrid process (laser ablation followed by SPDT) in order to make the precision machining process of ceramics more efficient. Both the laser ablation and SPDT processes have been identified as successful material removal methods for SiC\textsuperscript{159,150}.

The POCO Graphite SuperSiC-2 is used for all experiments reported in this paper.\textsuperscript{160} The as-received SiC samples are laser ablated by Mound Laser & Photonics Center (MLPC) using a SuperRAPID picosecond pulsed laser. Picosecond pulsed laser ablation processing is chosen as it is known for having several advantages such as less material damage, reduced thermal load (avoids the unnecessary heating of the surrounding material\textsuperscript{161}, i.e., heat affected zone or HAZ) and increased accuracy due to its short pulse lengths. This shorter pulse length also results in enhanced evaporation of the material hence reducing the melt pool size by preferentially heating and softening the material. Pulsed picosecond laser ablation also reduces the heat-affected zones (HAZ) due to the lower heat load when compared to pulsed laser radiation with nano and microsecond pulse lengths.\textsuperscript{162,163}
The second material removal process (SPDT) is done at the Western Michigan University Nanomanufacturing Laboratory. A single machining pass with a depth of cut of 1 µm and a feed of 1 µm/rev is used. The main focus of this material removal process is to ensure that all machining of the SiC material is done in the ductile regime. The experiments proved successful in determining the preferred combination of laser ablation and SPDT. The combination of parameters for laser ablation and SPDT, that are most effective in improving the surface finish of the material are determined (only the subsurface damage analysis is the primary focus of this section and not the machining results). A subsurface damage analysis, using scanning acoustic microscopy and micro-Raman spectroscopy, is carried out on the machined surfaces to investigate possible subsurface damage. A detailed study of all ablation and machining parameters has been reported in previous literature.

3.3.3.1 SCANNING ACOUSTIC MICROSCOPY

For the laser ablated-SPDT samples, acoustic microscopy imaging is carried out on the control region (as-received surface with no material removal performed), laser ablated surface and single point diamond turned surface to investigate any form of subsurface damage. For this particular analysis, subsurface damage in the form of cracks is imaged. Figure 3.9 shows an example of a subsurface crack imaged approximately 2 µm below the surface of a CVD coated SiC workpiece. Unlike pits, voids and grain boundaries, subsurface cracks have a very well defined and distinct characteristic (wave like form) as identified in Figure 3.9.
Figure 3.9: Subsurface cracks evident approximately 2 µm below the machined surface.

Figure 3.10 compares the surface after the laser ablation process (Figure 3.10(a)) with the surface after SPDT (Figure 3.10(b)). This figure also shows subsurface images from 500 nm below the surface to approximately 2 µm below the surface (in 500 nm increments). For this particular sample (10 mm diameter plateau, the control region (as-received surface) is initially laser ablated with a frequency of 60 kHz (with a single pulse in a burst group). Approximately 50 µm of material is removed in this ablation process from the top of the plateau (surface) in order to reduce the surface roughness (Ra is reduced from 3.75 µm to 3 µm) and rough peaks (peak-to-valley, Rz is reduced from 65 µ to 48 µm). This surface after the laser ablation process is shown in Figure 3.10(a) (labeled “Before SPDT”). After the ablation process, the plateau is single point diamond turned at a depth of cut of 1 µm with a feed of 1 µm/rev. The surface after the SPDT operation is shown in Figure 3.10 (b) (image labeled “After SPDT”). The purpose of the SPDT process is to further improve the surface finish (the Ra is reduced from 3 µm to 1.5 µm) of the laser ablated CVD-SiC. Figures 3.10 c-f represents the subsurface conditions of the single point diamond turned region.
Figure 3.10: A sequence of SAcM images of the laser ablated-SPDT sample.  
*Note: The first two images compare the laser ablated and SPDT surfaces, and the numbers on the top left of each image represents the scanned depth beneath the machined surface.*

From *Figure 3.10* it can be seen that surface cracks generated from the laser ablation process (*Figure 3.10 (a)*) are no longer visible after the SPDT process (*Figure 3.10 (b)*). However, subsurface cracks are clearly visible and become more obvious as it is imaged deeper below the machined surface (*Figures 3.10 (c) through (f)*). These cracks (thermal cracks) are believed to be generated due to the aggressive laser ablation process that removed approximately 50 µm of material from the plateau surface. Thermal cracks in the heat affected zones as a result of aggressive laser ablation have also been observed in past research carried out on ceramics and semiconductors.¹⁶⁵,¹⁶⁶,¹⁶⁷,¹⁶⁸,¹⁶⁹ These cracks are not believed to be generated by the SPDT operation as results from SPDT on the control region did not show any form of subsurface damage (as seen in *Figure 3.5*). These cracks are also not believed to be in the control region (before laser ablation or
SPDT) as Figure 3.11 shows the surface and subsurface images of the control region with no form of subsurface cracks/damage. With this evidence, it is safe to conclude that the surface and subsurface cracks observed in Figures 3.9 and 3.10 (except for Figure 3.10 (b) where thermal cracks are not visible) are caused purely by the laser ablation process and not by the SPDT operation. By comparing both surfaces, it is evident that SPDT improved the surface quality while resulting in no surface cracks.

![Figure 3.11: A sequence of SAcm images of the control region plateau.](image)

*Note: The first image shows the control surface and the numbers on the top left of each image represents the scanned depth beneath the machined surface.*

3.3.3.2 MICRO-RAMAN SPECTROSCOPY

Micro-Raman spectroscopy is used on this sample to detect the amorphous region beneath the single point diamond turned sample. The reason μ-Raman is preferred over regular laser Raman spectroscopy is to minimize the probing depth to below 200 nm. Since only a single pass is carried on this CVD-SiC sample, the resultant amorphous layer thickness (beneath the machined surface) is expected to be less than that of 6” CVD-SiC (where a total of six machining passes were carried out). Using a 325 nm
(ultraviolet) He-Cd laser, a 2-3 μm spatial resolution (0.3 mW power) is obtained. The Raman spectra comparing the unmachined and machined regions are shown in Figure 3.12.

![Figure 3.12: Micro-Raman spectra of the unmachined and machined CVD-SiC.](image)

In Figure 3.12, the spectra show the crystalline peaks of the unmachined SiC sample and the amorphous broadening of the machined surface. It is seen that a combination of the crystalline peaks (sharp peaks) and amorphous peaks (broadband peaks) are formed in the machined surface. It is seen that the 514 cm$^{-1}$ and 765 cm$^{-1}$ crystalline peaks are not obvious in the machined surface and encompassed by the broadband amorphous peaks. Once again, the amorphous remnant beneath the machined surface is a good indication of a ductile material removal process. From past research work, it is estimated that the total amorphous layer thickness is approximately equal to the total machined depth of cut.$^{67}$ Using the same ideology, the estimated thickness of the amorphous layer beneath the CVD-SiC machined surface is approximately 1 μm. However, in order to confirm this, a
cross-sectional TEM analysis is required. This is not intended at this phase of the research as cross-sectional TEM would require destroying the machined sample to get it thin enough for electrons to pass through.

3.4 CONCLUSION

The single point diamond turning experiments were successful in reducing the surface roughness of CVD coated SiC and quartz. The most important consideration when machining in the ductile regime is not to exceed the critical depth/DBT depth of the material, in order to avoid brittle fracture, which leads to a poorer surface finish. Optical microscopy (only for transparent samples like quartz), laser Raman spectroscopy, scanning acoustic microscopy and micro-Raman spectroscopy were successful NDT techniques utilized to investigate subsurface damage in these single point diamond turned ceramics. All subsurface damage techniques showed complementing results, where no signs of brittle material removal was detected. SPDT was successful in improving the surface of SiC and quartz without causing any form of surface and subsurface damage.

3.5 FUTURE WORK

The proposed future work is to perform scanning acoustic microscopy analysis on micro-laser assisted machined samples. The micro-laser assisted machining (µ-LAM) experimental process is discussed in detail in Chapters 5, 6 and 7 for silicon and silicon carbide. µ-LAM is a unique technique where it combines laser heating and SPDT in a single step (i.e., heating and material removal using a diamond tool occurs simultaneously). SAcM will be a useful tool to investigate the possibility of subsurface
thermal cracks that may have occurred during the laser heating process (as demonstrated in the results of this chapter). Although thermal cracks are assumed not to exist in the µ-LAM process, SAcM will certainly be a useful tool to verify this. Even though micro-Raman analysis has been successfully conducted on the micro-laser assisted machined surfaces (see chapters 5, 6 and 7), the Raman spectra does not provide subsurface feature characteristics such as the existence of thermal cracks.
CHAPTER 4

THE SIGNIFICANCE OF CRYSTAL ORIENTATION ON THE DUCTILITY OF POLYCRYSTALLINE CVD-3C-SILICON CARBIDE

4.1 INTRODUCTION

There is a technological need for hard thin coatings and substrates with high elastic modulus, fracture toughness and wear resistance. Silicon carbide (SiC) fulfills such requirements for a variety of applications at high temperatures and for high-wear MEMS and opto-electronics components. The rapid development of SiC for over past two decades has resulted in enormous prospects for its use in microelectronic and optical applications. Mechanical properties of the material play a pivotal role in determining the lifetime of these manufactured devices. The cubic SiC (beta-3C) material, which is the primary focus of this chapter, is chemically inert, can withstand high temperatures, and has high resistance to oxidation. SiC is also a preferred material of choice in hostile environments due to its excellent electronic and thermal properties, including large reverse breakdown voltage, high electron mobility, high saturated electron drift velocity and excellent thermal conductivity relative to silicon (Si). There is a favorable trend towards the use of polycrystalline 3C since it can be deposited on various substrates (as with CVD) and micromachined in a similar fashion to Si.
Potential applications of SiC in various industries have created strong interest in its physical and mechanical properties. Due to the extreme brittleness of the material, there is a great need and desire to understand its fracture behavior. Manufacture of this material is extremely challenging due to its high hardness (25 – 27 GPa), brittle characteristics and poor machinability. Severe fracture can result during the machining of SiC due to its low fracture toughness. However, from past and current research work it has been proven that ductile regime machining of SiC to improve its surface roughness is possible. From these results, it is also evident that CVD 3C (β) SiC is not as brittle as past researchers indicated (ductile to brittle transition of ~ 50nm). This has raised questions (among this group of researchers) that there may be a preferred crystal orientation and fracture toughness relationships in this material that is enabling a fairly large depth of cut (> 1µm), while still providing a ductile response in such a nominally brittle material during a single point diamond turning (SPDT) operation.

Previous researchers have successfully been able to precisely grind CVD-SiC (using high precision grinding) but this process is very expensive and the fine abrasive wheels often result in an unstable grinding process. Single point diamond turning (SPDT) was chosen as the material removal method as it offers increased process flexibility, better accuracy, quicker fabrication time and lower cost when compared to grinding and polishing to achieve nanometric surface roughness. SPDT of SiC disks was proven to be successful in the past and interestingly the material permitted larger depths of cuts than its ductile to brittle transition (DBT) depth (critical depth) that was determined through scratch tests (the claimed DBT of this material was approximately 50 nm). The question was raised to further investigate this material from an atomic point of
view due to the fact that CVD-SiC accommodates larger depths of cuts (i.e. up to 1.3µm) in the ductile regime.

In a total of only six passes, the surface roughness was reduced from 1.25 µm to 85 nm (Ra) and 9.0 µm to 250 nm (Rz). The ability for a nominally brittle material such as SiC to show such ductile response was somewhat unexpected (a ductile response was expected at 50 nm but not at over 1 micrometer). The crystal orientation is related to the fracture response of the material and there could be a preferred crystal orientation, i.e., less brittle response, in this CVD coated material. The ultimate goal (long term) of this study is to account for the observed ductile and brittle response during machining and to possibly identify a preferred machining orientation and direction to obtain the maximum ductile (or conversely minimum brittle fracture) response in this material.

The aim of this particular study is to determine the effect of crystal plane orientation on the SPDT machining characteristics of a CVD coated SiC disk. Both the unmachined and machined regions are analyzed (although no difference in crystal plane and orientation is expected after SPDT). SPDT was performed on a CVD coated SiC disk to improve its surface roughness. A 250 µm thick CVD coated Super-SiC from Poco Graphite is used in this study. The outcome of this project is a precursor to the evaluation of the effects of crystal orientation in determining the resultant surface roughness, tool wear (due to friction) and material removal rate (discussed in chapters 6 and 9). Two non destructive testing (NDT) techniques are used to study the crystal orientation of the material: (1) Orientation Imaging Microscopy (OIM) and (2) X-ray Diffraction (XRD).
4.2 EXPERIMENTAL METHOD

4.2.1 ORIENTATION IMAGING MICROSCOPY

The beam of a Scanning Electron Microscope (SEM) strikes a crystalline material mounted at an incline around 70° causing the electrons to disperse beneath the surface, subsequently diffracting among the crystallographic planes. The diffracted beam produces a pattern composed of intersecting bands, termed electron backscatter patterns, or EBSPs. The patterns can be imaged by placing a suitable film or phosphor screen in close proximity to the sample in the SEM sample chamber. This phenomenon is illustrated in Figure 4.1.

![Figure 4.1: Electron diffraction in a SEM.](image)

This technique allows microstructural phase and crystal orientation information to be determined at very specific points in a sample. OIM is powerful tool for investigating of polycrystalline microstructure including local crystallographic textures, grain boundary analysis, phase identification and relationship between phases, crystallographic analysis of microgram quantities of material and qualitative strain mapping. The spatial resolution varies with the accelerating voltage, beam current and spot size of the SEM along with the atomic number of the sample material. The TSL/EDAX OIM has a
lateral resolution of about 200 nm and capable of sampling depths in the range of 2 – 5 nm. Indexable patterns can be obtained from about 0.05 µm with a field emission source.

4.2.2 X-RAY DIFFRACTION

X-ray diffraction (XRD) is a versatile, NDT technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials. The general concept of XRD is shown in Figure 4.2.

![Figure 4.2: General concept of XRD on a sample.](image)

X-rays primarily interact with electrons in atoms. When x-ray photons collide with electrons, some photons from the incident beam will be deflected away from the original direction of travel, much like billiard balls bouncing off one another. If the wavelength of these scattered x-rays did not change (meaning that x-ray photons did not lose any energy), the process is called elastic scattering (Thompson Scattering) in that only momentum has been transferred in the scattering process. These are the x-rays that are measured in diffraction experiments, as the scattered x-rays carry information about the electron distribution in materials. On the other hand, in the inelastic scattering process
(Compton Scattering), x-rays transfer some of their energy to the electrons and the scattered x-rays will have different wavelength than the incident x-rays. Diffracted waves from different atoms can interfere with each other and the resultant intensity distribution is strongly modulated by this interaction. If the atoms are arranged in a periodic fashion, as in crystals, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms. Measuring the diffraction pattern therefore allows the experimenter to deduce the distribution of atoms in a material.\textsuperscript{180}

The peaks that are obtained from an x-ray diffraction pattern can be directly correlated to the atomic distances. For a given set of lattice planes, with an inter-plane distance of $d$, the condition for a diffraction (peak) to occur is given by Bragg’s law.

$$2d \sin \theta = n \lambda \text{..........................} (4.1)$$

In the equation, $\lambda$ is the wavelength of the x-ray, $\theta$ is the scattering angle and $n$ represents the order of the diffraction peak. It is vital to understand Bragg’s law in order to interpret x-ray diffraction data.

In order to determine the orientation distribution of crystalline grains in a polycrystalline sample, texture measurements or pole figures are plotted in polar coordinates. The plotted pole figures consist of the tilt and rotation angles with respect to a given crystallographic orientation. The center of a pole figure represents the zero angle point where the grain is normal to the surface.
4.3 RESULTS AND DISCUSSION

4.3.1 ORIENTATION IMAGING MICROSCOPY

A total of 5 different areas on the CVD-SiC are imaged: Figure 4.3 shows the scanned locations on the sample. Both machined and unmachined (polished) areas are investigated. Areas 1, 2, 3 and 4 are machined areas (with varying depth of cuts from 250 nm to 1 μm) whereas area 5 is an unmachined (as-received state was a previously polished) area.

Since all 5 scanned areas show repeatable (similar) results, only two areas will be discussed; a machined (Area 1) and an unmachined area (Area 5). All scanned areas are similar in size where the dimensions are 112 x 327 μm.
One of the first steps in this process is to determine the phase of the material (i.e., alpha or beta). From Figures 4.4 and 4.5, it is obvious that the dominant phase in this material is the beta (β) phase. This is known by the scattering signal obtained when a scan is carried out for each of the different phases (alpha and beta scans were carried out in this case). If a significant amount of dark/black area is visible, this means that the signal for that particular phase is weak (as seen for the alpha phase scan in Figures 4.4.
The alpha phase is generally formed at temperatures greater than 1700 °C and has a hexagonal crystal structure whereas the beta phase (zinc blende crystal structure) on the contrary is formed at temperatures below 1700 °C.\textsuperscript{181}

The next step is to try and determine the crystal direction that is parallel to the surface normal. This is done by analyzing the signal at the center of the pole figures for a chosen crystal orientation.\textsuperscript{182} The center of a pole figure represents the crystal plane normal to the sample surface.

![Figure 4.6: OIM pole figures of four crystal orientations of the machined region.](image)

![Figure 4.7: OIM pole figures of four crystal orientations of the unmachined region.](image)
From Figures 4.6 and 4.7, the strongest signal at the center/origin (which represent $90^\circ$ normal to the surface) is in the $<112>$ direction. The signal strength is slightly stronger in scan area 5 compared to area 1 (1.685 vs. 1.439) due to the smoother surface finish in the unmachined (polished) area. The machined area generally contains feed mark features that increase the surface roughness (only when compared to a polished surface and not an as-received surface).

4.3.2 X-RAY DIFFRACTION

In this technique, unlike OIM, the crystal direction in which the analysis is done is stated by the user. The three most probable crystal planes (from OIM and literature review) were investigated; $<001>$, $<111>$ and $<112>$. The unmachined area (Area 5) was investigated as the smoother surface finish (polished region) is expected to yield in a slightly stronger signal.

Figure 4.8: XRD pole figures of the unmachined region for three crystal planes.
Figure 4.8 shows that the strongest signal (3.66) that occurs at the center of the pole figure (which represents the crystal plane normal to the surface) is in the <111> direction. The results from the XRD were chosen for the hypothesis in this research as it yielded in a significantly stronger signal (1.685 vs. 3.66) than OIM. Now that the preferred crystal direction has been identified as <111> using XRD, a detailed literature review on previous research work was conducted to correlate the fracture toughness and the crystal orientation for 3C-SiC. Since the reported micro-hardness for single crystal SiC (4H, 6H and 3C) and polycrystalline SiC (3C) are in the same range (25-30 GPa), the fracture toughness of these materials are correlated to explain the enhanced ductility of the 3C-polycrystalline CVD-SiC.

Kikuchi et al. performed an extensive study to correlate the brittle dynamic fracture in crystalline cubic 3C-SiC using inter-atomic potential. In this study, brittle fracture dynamics for three low-index crack surfaces, i.e., <110>, <111> and <100>, is studied. The results exhibit significant orientation dependence: <110> fracture is cleavage, whereas <111> and <001> fractures are unstable against slip and branching, respectively.

In another study conducted by Pharr et al., the dependence of fracture toughness on crystallographic orientation for single-crystalline cubic β-SiC is investigated. The results here were then compared to the fracture toughness of a polycrystalline β-SiC. X-ray diffraction is performed on the samples to determine the orientation of the crystals. This current dissertation research concluded that the major difference in fracture toughness is among the single crystal SiC and the polycrystalline 3C-SiC (the fracture toughness value of the polycrystalline is almost three times greater than the single
crystalline). There is no significant difference in fracture toughness values among all planes experimented for the single crystal 3C-SiC substrate.

Since SiC can exist in single crystal, polycrystalline and amorphous forms, it is important to compare the relative mechanical properties for specific applications. For this reason, Reddy et al. extensively studied the mechanical properties of 3C-SiC films for MEMS applications. In this study, the mechanical properties of single crystal and polycrystalline 3C-SiC films are studied by means of nanoindentation using a Berkovich diamond tip. The results of the study show that polycrystalline SiC films have better mechanical properties (including fracture toughness) and therefore are very suitable to operate in harsh environments. Table 4.1 summarizes experimental data from all previous research work discussed in this chapter.

Table 4.1: Fracture toughness for various polytypes, crystallinity and sources of SiC.

<table>
<thead>
<tr>
<th>Material</th>
<th>Crystal Direction</th>
<th>Reported Fracture Toughness</th>
<th>Literature Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single crystal 3C-SiC</td>
<td>&lt;110&gt;</td>
<td>1.26 MPa-m^{0.5}</td>
<td>Kikuchi et al.</td>
</tr>
<tr>
<td>Single crystal 3C-SiC</td>
<td>&lt;111&gt;</td>
<td>1.52 MPa-m^{0.5}</td>
<td>Kikuchi et al.</td>
</tr>
<tr>
<td>Single crystal 3C-SiC</td>
<td>&lt;001&gt;</td>
<td>1.30 MPa-m^{0.5}</td>
<td>Kikuchi et al.</td>
</tr>
<tr>
<td>Single crystal β-SiC</td>
<td>&lt;110&gt;</td>
<td>1.44 MPa-m^{0.5}</td>
<td>Pharr et al.</td>
</tr>
<tr>
<td>Single crystal 3C-SiC</td>
<td>Unknown (not provided)</td>
<td>1.59 MPa-m^{0.5}</td>
<td>Jayadeep et al.</td>
</tr>
<tr>
<td>Single crystal 3C-SiC</td>
<td>&lt;100&gt;</td>
<td>1.00 MPa-m^{0.5}</td>
<td>Zenong et al.</td>
</tr>
<tr>
<td>Single crystal 3C-SiC</td>
<td>&lt;001&gt;</td>
<td>1.00 MPa-m^{0.5}</td>
<td>Zenong et al.</td>
</tr>
<tr>
<td>Single crystal 4H-SiC</td>
<td>&lt;1010&gt;</td>
<td>1.21 MPa-m^{0.5}</td>
<td>CREE, USA</td>
</tr>
<tr>
<td>Single crystal 6H-SiC</td>
<td>&lt;0001&gt;</td>
<td>1.25 MPa-m^{0.5}</td>
<td>SI Crystal, Germany</td>
</tr>
<tr>
<td>Polycrystalline CVD-β-SiC</td>
<td>Unknown (not provided)</td>
<td>3.46 MPa-m^{0.5}</td>
<td>Pharr et al.</td>
</tr>
<tr>
<td>Bulk Polycrystalline 3C-SiC</td>
<td>Sintered</td>
<td>4.60 MPa-m^{0.5}</td>
<td>Jayadeep et al.</td>
</tr>
<tr>
<td>Polycrystalline CVD-3C-SiC</td>
<td>&lt;111&gt;</td>
<td>3.00 MPa-m^{0.5}</td>
<td>POCO Graphite</td>
</tr>
</tbody>
</table>

Two main conclusions can be drawn from Table 4.1: (1) polycrystalline 3C-SiC has significantly higher fracture toughness values than any of the single crystal SiC materials, and (2) the <111> plane in the single crystal 3C-SiC has the highest fracture toughness value irrespective of the source of the material (among the known planes).
Therefore the enhanced ductility of the 3C-\textit{SiC} polycrystalline SiC (POCO Super-SiC) seen in this study is believed to be due to these two conclusions drawn.

Finally, once the direction normal to the surface plane is determined, the orientation distribution function (ODF) is obtained to investigate if this specific crystal direction has a particular orientation pattern or if it is randomly orientated.

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure4_9}
\caption{ODF of the <111> crystal direction.}
\end{figure}

\textit{Figure 4.9} shows that there is not a strong enough signal to conclude that this particular crystal orientation is grown in a specific pattern/direction. Looking at the highlighted box (that represents 90° normal to the surface), it is clear that the <111> crystal direction is randomly orientated in this particular material.

\subsection*{4.4 CONCLUSION}

The results from the XRD were chosen for the study in this research as it yielded a significantly stronger signal (1.685 vs. 3.66) than OIM. From the XRD results, it can be concluded that the crystal direction normal to the surface is in the <111> direction. However, the ODF shows that the crystals are randomly orientated in this specific material. The <111> crystal direction presumably has a higher fracture toughness thus
making the CVD coated SiC less brittle (than the single crystal SiC) and therefore
enabling larger depths of cuts while performing ductile regime machining. This
assumption is strongly supported by the research conducted by past researchers where all
supporting data has been summarized in Table 4.1. The two main aspects that contribute
to the enhanced ductile response of the 3C polycrystalline SiC are (1) polycrystalline 3C-
SiC has a significantly higher fracture toughness than any of the single crystal SiC
materials, and (2) the <111> direction (which is the direction of the material in this
research) in the single crystal 3C-SiC has that highest fracture toughness value
irrespective of the source of the material. This is believed to be due to the available grain
boundaries (grain sizes in the Poco 3C-SiC averaged between 20–30 µm) in the
polycrystalline material that impedes crack propagation, hence limiting the extent of
brittle fracture. Since the lattice structure of adjacent grains differs in orientation, it
requires more energy for a crack to change directions and move into the adjacent grain.
The grain boundary is also much more disordered than inside the grain, which also
prevents the cracks from moving in a continuous slip plane (as is the case in single
crystals).

4.5 FUTURE WORK

The proposed future work is to perform single point scratch tests (with similar
parameters to that performed on the 3C polycrystalline SiC discussed in this chapter) to
determine the DBT along different crystal directions on single crystal 3C-SiC. Single
point scratch tests (using a diamond cutting tool) best mimic the SPDT operation and
therefore will be a good mode of examining the crystal orientation dependence of the
ductile response in the material. Micro-laser assisted scratch tests will also be done to compare the in-situ heating effect, DBT and crystal orientation dependence of the material.
CHAPTER 5

THE EFFECT OF LASER HEATING ON THE MATERIAL DEFORMATION AND REMOVAL OF SINGLE CRYSTAL SILICON

5.1 INTRODUCTION

Optical and semiconducting crystals such as silicon (Si) have progressively found more important applications in the field of modern opto-electronics. Single crystal semiconductors are normally considered to be fragile and to exhibit brittle response under conventional machining conditions. Obtaining optics quality surfaces on Si has been an ongoing critical problem in the ultra-precision manufacturing field. Traditionally, Si is polished to a mirror-like surface finish is obtained. However, polishing has several drawbacks such as low production efficiency, poor ability to automatically control the process, low form/shape accuracy for non planar surfaces, edge effect of the workpiece and limited ability to produce aspherical surfaces.\textsuperscript{189}

Precision diamond turning at the micro/nanometric scale of infrared materials such as Si can consistently achieve mirror-like surface finish while maintaining minimal or no subsurface damage if the proper equipment, setup, parameters and diamond tooling is used. This is achieved by machining under conditions of small chip thickness dictated by the ductile regime machining threshold.\textsuperscript{190,191} However, it is possible to remove
material at a higher removal rate by understanding the high pressure phase transformation phenomena and incorporating laser heating in the machining process.

Laser radiation is a powerful tool for micro/nanomachining different materials because it can be focused to micron-sized spot diameters and the thermal load can be controlled by changing the delivered laser power/intensity.\textsuperscript{192} Lasers have been widely utilized in metallic materials since the early 1970’s.\textsuperscript{193} More recently, lasers have been utilized in machining non-metallic materials such as ceramics, plastics and semiconductors for the mechanical and electronic and optical industries.\textsuperscript{194} The ability of lasers to precisely preferentially heat micron and sub-micron features, in otherwise hard to machine materials such as ceramic and semiconductors, has created a rapidly growing interest in understanding the parameters controlling the limits and capabilities of this process.\textsuperscript{195}

It has been demonstrated that ductile regime machining of semiconductors at room temperature, i.e., below the thermal softening temperature at which dislocations become readily mobile, is possible due to the high pressure phase transformation (HPPT) occurring in the material caused by the high compressive and shear stresses induced by a single point diamond tool tip. To further augment the ductile response of nominally brittle materials, traditional single point scratch tests are coupled with a micro-laser assisted machining ($\mu$-LAM) technique. In this study the effects and benefits of laser heating on the material removal of Si is studied by means of single point scratch testing. The effect of laser heating on material’s hardness and the ductile-to-brittle transition (DBT) of the material, along with the optimization of machining parameters such as
applied load, depth of cut, cutting force, cutting speed, laser wavelength and laser power is studied.

The study is done by comparing the results of scratch tests on single crystal silicon, with and without laser heating. The effects of laser heating is studied by verifying the depths of cuts for scratch tests carried out on Si with increasing loads (thrust force), wherein the scratch shows both ductile and brittle response (with a ductile to brittle transition (DBT) region within the scratch). Scratch tests are chosen to be the principal method of investigation in this study as it is a better candidate for evaluating machining conditions than indenting because the mechanics during scratching are more applicable to the machining process, such as single point diamond turning (SPDT). Optical microscopy, load sensors (for force measurements), white light interferometry, electron microscopy, nanoindentation and micro-Raman spectroscopy are utilized in this study.

5.2 EXPERIMENTAL METHOD

All of the material deformation and removal experiments are performed on the Universal Micro-Tribometer (UMT) which is produced by the Center for Tribology Research Inc. (CETR). This equipment is developed to perform comprehensive micromechanical tests of coatings and materials at the micro scale. This system facilitates cutting speeds as low as 1 µm/sec at nanometric cutting depths. The UMT is a load controlled device where the required thrust force ($F_z$) is applied by the user to obtain the desired depth of cut (based on the tool geometry and workpiece material properties). Since the UMT is load controlled, very high vertical resolution is achieved based on the load cell range selected (up to 1 mN force resolution and a minimum depth of 1 nm is
achievable). The UMT is equipped with a dual-axis load cell that is capable of constantly monitoring the thrust and cutting forces, \( F_x \) (obtained as an output parameter from the cutting experiment).

The \( \mu \)-LAM system is incorporated within the UMT to perform in-situ laser assisted cuts (the setup is shown in the following sections). Three different lasers are utilized in this study to experiment on different wavelengths, powers and beam sizes. The laser specifications utilized in this study are shown in Table 5.1.

Table 5.1: Laser devices and specifications.

<table>
<thead>
<tr>
<th>Laser Device</th>
<th>Wavelength (( \lambda ))</th>
<th>Power Range</th>
<th>Beam Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furukawa</td>
<td>1480 nm</td>
<td>0 – 400 mW</td>
<td>10 ( \mu )m</td>
</tr>
<tr>
<td>Visotek</td>
<td>970 nm</td>
<td>0 – 10 W</td>
<td>100 ( \mu )m</td>
</tr>
<tr>
<td>IPG*</td>
<td>1070 nm</td>
<td>10 – 100 W</td>
<td>10 ( \mu )m</td>
</tr>
</tbody>
</table>

Note. *Operates with a beam delivery optics (BDO) unit by Laser Mechanisms Inc.

All lasers used in this study are continuous wave (CW) fiber coupled lasers due to ease of attaching a cutting tool/stylus to a SMA (sub-miniature version A) or FC-PC (fiber-optic connector for physical contact) fiber coupler where it is fairly straightforward to deliver the laser beam to the tool. The lasers utilized are in the near infrared (NIR) region as the atmospheric phase of Si (Si-I) absorbs less than Si-II (at high wavelengths such as 1480 nm) and the high pressure metallic phases of Si (Si-II) absorbs more at lower wavelengths (not comparing to Si-I but only looking at the Si-II trend). Figure 5.1 shows the relative absorption of Si phases at different wavelengths. A combination of this difference in absorption makes it ideal for experimenting on the enhanced ductility of the Si-II phase and reduced brittleness of the Si-I phase.
It is seen from Figure 5.1 that the absorption of Si-I and Si-II continuously decreases as the wavelength increases from 500 nm to 1500 nm. However at higher wavelengths (e.g., 1500 nm) the metallic Si-II phase starts absorbing more whereas the atmospheric Si-I phase is almost transparent. As the wavelength decreases from 1000 nm to 500 nm, Si-I starts absorbing more than Si-II. However, the amount of absorption in Si-II also increases significantly from 1500 nm to 500 nm (10% to 51%). These combinations between the absorption of Si-I and Si-II at low and high wavelengths make it ideal to study the enhanced ductility of the metallic Si-II phase as well as the possible decrease in brittleness in the atmospheric Si-I phase. Since the laser beam is focused at the tool-workpiece contact zone, it is assumed that the primary phase being heated is high-pressure transformed the SI-II phase. The experimental work to study the effect of the laser assisted material removal process on the high pressure Si phases is further discussed in Chapter 7.
5.2.1 LASER BEAM ALIGNMENT

In the µ-LAM experiments, it is crucial that the laser beam is perfectly aligned to the diamond tool cutting edge exactly at the tool-workpiece contact spot to ensure: (1) maximum laser power is delivered to the workpiece (2) the laser heating effect only occurs at the intended spot and (3) the thermal softening effect is maximized. A misalignment in the laser beam causes a poorly focused beam, unintended thermal expansion of the substrate and most importantly a failure to capitalize on the thermal softening effect. Among the lasers used in this study, only the high powered IPG laser requires manual alignment as the beam is converged through a BDO and delivered to the diamond tool. Several equipment/instruments such as an optical microscope (imaging), a thermal evaporator (to deposit an opaque coating) and a power meter (measure laser throughput) is utilized to align the beam.

5.2.2 LOW POWERED LASER

An infrared (IR) diode laser (λ=1480 nm and P_{max}= 400 mW) with a Gaussian profile beam of 10μm in diameter is used in this part of the experiments. The laser beam is guided through a 10 μm fiber optic cable to a ferrule, which is attached to the diamond stylus. The µ-LAM system is configured in such a way that the laser beam passes through the diamond tip (stylus) and impinges on the Si workpiece at the tool-workpiece interface (contact). A typical scratch test setup along with the µ-LAM system is shown in Figure 5.2. All scratches/cuts are performed on a single crystal Si wafer on the {100} plane along the <110> direction.
5.2.2.1 SCRATCHING WITH A STYLUS

A 90° conical single crystal diamond stylus with a spherical end tip radius of 5 μm is used as the scratch tool. The details of the diamond tip attachment are depicted in Figures 5.3(a) and (b). The laser emerges from a 90° conical single crystal diamond tip with 5 μm radius spherical end.

In this set of experiments, two conditions of scratches are performed: with and without laser heating. The scratches are carried out at low cutting speeds (1 μm/sec) in...
order to maximize the thermal softening of the material during the laser heating process. Scratch lengths of 500 µm are produced on the Si wafer. The applied load is increased linearly with time from 20 mN to 90 mN along the 500 µm scratch length. This specific load range is chosen as the material is expected to exhibit both a ductile and brittle characteristics, with a DBT within the range. The scratch test parameters are summarized in Table 5.2.

Table 5-2: Scratch testing parameters using a stylus and low powered laser.

<table>
<thead>
<tr>
<th>Scratch #</th>
<th>Load Range (Fz)</th>
<th>Condition</th>
<th>Cutting Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20 – 70 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>2</td>
<td>20 – 70 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
</tbody>
</table>

*400 mW is the maximum output power, approximately 150 mW is actually delivered to the workpiece material and the remaining power is lost due to scattering and reflections.

The loads, depths of cuts, cutting forces, laser power and scratch topography are measured, imaged and correlated to determine the laser heating effect on the ductile response during the material removal mechanism.

5.2.2.2 CUTTING WITH A SPDT TOOL

A 1.3-1.5 mm nose radius (the nose radius was measured after the tool was manufactured, hence the range) single crystal diamond cutting tool with a -45° rake and 5° clearance angle is used for this set of experiments. An actual cutting tool is used to better mimic the physics and machining parameters of the material removal process during an actual SPDT operation. For this particular cutting tool, no alignment was required as the tool is equipped with a built-in ‘self-aligning’ optics system. The main aim of this set of experiments is to analyze the thermal softening effect of Si using a cutting tool. The scratch test parameters used here are summarized in Table 5.3.
Table 5-3: Scratch testing parameters using a cutting tool and low power laser.

<table>
<thead>
<tr>
<th>Load ($F_z$)</th>
<th>Condition</th>
<th>Cutting Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>10 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>15 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>15 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>20 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>20 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>25 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>25 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>50 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>50 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
</tbody>
</table>

*400 mW is the maximum output power, approximately 255 mW is actually delivered to the workpiece material and the remaining power is lost due to scattering and reflections.

A total of 10 cuts (approximately 300 µm long) are carried out with and without laser heating to measure the differences in depths of cuts, widths of cuts and cutting forces. All cuts are well within the DBT load for this specific tool geometry and material (i.e., the DBT load for Si using this cutting tool is approximately between 170 – 190 mN). Only low loads (shallow depths) are experimented here to control the width of cut, matching it to the laser beam diameter. If the cuts are too wide the beam only covers a small portion along the center of the cut. In a low powered laser, it is important to ensure that the laser beam covers sufficient area of the deepest portion of the cut to maximize on the thermal softening effect. The loads, depths of cuts, cutting forces and laser power are measured, imaged and correlated to determine the thermal softening effect.

5.2.3 HIGH POWERED LASER

A 1 mm nose radius single crystal diamond cutting tool (-45° rake and 5° clearance) is used for this set of experiments. An IR diode laser ($\lambda=1070$ nm and $P_{max}=100$ W) with a Gaussian profile beam of diameter approximately 10 µm is used in this investigation. The laser beam is guided through a custom made fiber optic cable to a
collimator, which is attached to a BDO. The BDO then converges the beam (from 7.8 mm to 10 μm) and delivers it to the diamond cutting tool. A typical scratch test setup along with the high laser powered μ-LAM system is shown in Figure 5.4.

![Figure 5.4: High powered μ-LAM scratch test setup on the UMT.](image)

Using the high power laser setup, significantly more cutting experiments are conducted to correlate the laser heating effect with the load/depth of cut, cutting force, DBT and cutting speed. The availability of higher laser powers allow cuts to be performed at higher cutting speeds without compromising on the thermal softening effect.

5.2.4 TEMPERATURE MEASUREMENT ESTIMATION

To further understand the material behavior at the micro level, it is important to have a reliable temperature estimation of the heated material/phase. Temperature is estimated/measured here using the OMEGALAQ liquid temperature lacquers that come
in a wide range of temperatures from 79 °C to 1038 °C.\textsuperscript{198} Since the size of the contact area/phase transformed region is extremely small, in the order of tens of micrometers, it is extremely difficult to use a probing technique to measure temperature with the required resolution and accuracy. The setup used to estimate the temperature of the laser heated zone is schematically illustrated in Figure 5.5.

![Figure 5.5: Schematic showing temperature measurement scratch test setup.](image)

A micro-laser assisted cut is performed on a polished Si wafer that has a layer of liquid temperature lacquer deposited on the back-side of the wafer. Several other configurations were considered, but this setup is assumed to be the most accurate as it takes into account the optical properties of the Si-I and Si-II (high pressure metallic phase) phases.

5.2.5 MICRO-RAMAN SPECTROSCOPY

A Renishaw inVia Raman microscope is used to detect and characterize the high pressure phases in the machined region and accumulated machined chips. In this particular analysis, the effect of varying laser powers and cutting speeds on the high pressure phase formation is studied. Also, the effect of laser power/heating on the
machined chips is studied by varying the Raman laser power intensity (in-situ heating). Using a 442 nm (visible blue) He-Cd laser, a 1-2 μm spatial resolution is obtained. Figure 5.6 shows the Raman instrument used for these analyses. A visible laser source is used in this experiment to obtain better Raman intensities that would be helpful in differentiating the various high pressure phases.

Figure 5.6: Renishaw inVia Raman microscope and laser sources.

5.2.6 TRANSMISSION ELECTRON MICROSCOPY OF SILICON CHIPS

Transmission electron microscopy (TEM) is used to observe the microstructural changes in the machined chips. A number of previous authors have used TEM to observe the phases of the machined chips and subsurface of the machined layer of Si.\(^{199}\) A 200 kV JEOL JEM-2010-F TEM is used in this analysis. This equipment can provides a 0.17 nm resolution at 200 kV energy. The TEM is also equipped with an EDS detector and selected area diffraction (SAD) capabilities to identify the phases in the machined chips. Since the chips imaged are relatively small (less that 30 μm in length), a special carbon coated TEM grid (lacey carbon film) is used. This enables small substrates to be placed on the grid without falling through the opening (the regular TEM grid has a 30 μm x 30 μm grid opening). Although the existence of several other substances/debris on the grid is
common, EDS is used to confirm the validity of the area being imaged (EDS spectra of the Si chip shown in appendices). An image of the equipment setup is given in the appendices.

5.3 RESULTS AND DISCUSSION

5.3.1 LASER BEAM ALIGNMENT – 100 W IPG LASER WITH LASER MECHANISMS BDO

The first step to aligning the laser beam relative to the tool cutting edge is to perform a preliminary beam positioning. This is done using the guide beam (visible beam), where a distinct pattern (a recognizable pattern is obtained based on previously aligned tools) is obtained using the x, y and z beam focus knobs. The pattern acquired here is a projection of the tool geometry where the cutting edge is along the nose radius and the beam reflection on the rake face. Figure 5.7 shows a projected beam pattern using the guide beam on to a linear stage.

Figure 5.7: Projected beam image using a visible laser beam.
Once the preliminary beam position is acquired, the tool is then coated to make sure that the diamond is opaque to the IR laser beam. Several coating methods such as magnetron, plasma sputtering and thermal evaporation are utilized. In general, the coatings obtained from the thermal evaporation process works best in blocking almost all of the laser power. Either an aluminum (Al) or a silver (Ag) coating approximately 100 nm thick is used. Once the tool is coated, a scratch is performed on Si to expose only the contact region, i.e., the contact region between the tool tip and workpiece. This scratch is generally performed at very low loads (< 50 mN) to ensure that the opening is only large enough for the 10 μm beam to pass through. *Figure 5.8* compares a coated tool before and after the scratch.

![Figure 5.8: Coated tool edge before and after the scratch: (a) fully coated tool before the scratch, (b) coated tool with the contact area exposed after the scratch and (c) high magnification of the exposed region that will be used for alignment.](image)
After the opening has been made via scratching, the guide beam is used to align the spot centered to the opening; since the guide beam and the actual IR beam are assumed to be collinear. A power meter (New Focus Inc. Model-3831) is positioned below the diamond tip to optimize the power throughput once the beam is centered. Power measurements are generally a good indication and guide throughout the alignment process. Therefore, power measurements are taken at all stages, i.e., after the preliminary beam positioning, after the tool has been coated, after the coating has been removed via scratching and finally once the beam is aligned. Figure 5.9 compares the output power at the tool tip at all stages during the alignment process.

![Output vs. Input Power for Beam Alignment Stages](image)

**Figure 5.9: Power throughput at the tool tip after each alignment stage.**

*Note. The remaining laser power is lost due to scattering and internal reflections.*

The lowest power throughput of less than 2% is observed when the tool has been fully coated. However, it is also seen that equally low throughputs are observed after the coating is removed. This means that the preliminary beam position was off-centered from the actual tool tip-workpiece contact area. The highest power throughput of
approximately 23% is obtained once the beam has been fully aligned to the cutting edge, as expected.

There are several methods to cross-check the alignment of the beam by varying the laser power during a scratch test. Figure 5.10 shows a 2 mm scratch at a constant load done with varying laser powers. The power is increased with time to a maximum (70 W) at the middle of the cut. Once the maximum is reached, the power is then reduced with time until it reaches the initial starting point power (30 W). The maximum power here is defined by the power high enough to cause a discoloration along the cut due to excess heating and not by the maximum the laser is capable of (100 W).

Figure 5.10: Bell-curve representing the power correlating with the discolored cut region.

Since the discolored region along the cut correlates to the maximum power on the bell-curve in Figure 5.10, this is a good indication that the beam is well aligned. In contrast, a misaligned beam may not cause sample discoloration at all or discoloration away from the scratch. The second method is to use high enough powers in the middle of a cut that would cause a burn mark. Although burning/melting the material during the
actual cutting process is not desired, this is indeed a good way to check the beam alignment.

![Figure 5.11: Burn marks intentionally created along the cut: (a) beam misaligned (b) beam well aligned along the nose radius.](image)

*Figure 5.11* compares a misaligned beam where the burn mark is not along the cut/scratch and a well aligned beam along the nose radius where the burn mark is centered along the cut/scratch. The relative power intensity with respect to the burn mark along the cut (i.e., making sure the burn mark along the cut correlates to time the highest power is used) is used to determine the beam position relative to the rake and flank face of the tool.

5.3.2 LOW POWERED LASER

5.3.2.1 SCRATCHING WITH A STYLUS

The load range (20-70 mN) performed on these scratches is ideal for this study as it had both; the ductile and brittle regime along the same scratch. *Figure 5.12* shows two scratches that represent the two conditions: without (scratch 1) and with laser heating (scratch 2). The ductile to brittle transition is identified somewhere between the ductile
and brittle regime of the scratch using optical microscopy, white light interferometry and force analysis based on the variations in cutting forces. Figure 5.13 shows a high magnification optical microscope image used to identify brittle fracture along the scratch. It is seen in Figure 5.13 that the scratch performed without laser heating exhibits brittle fracture along the cut much before the scratch performed with laser heating. The indication of brittle fracture for the scratch done with laser heating is not seen in Figure 5.13 as it is outside the field of view.

Figure 5.12: Scratches done with two different conditions: (1) without, and (2) with laser heating.

Figure 5.13: Optical image shows fracture along the scratch performed with no laser.

In this study, there are two different analyses done based on the results obtained from the scratch tests. The first analysis compares the depth of cut and cutting forces ($F_x$) for a constant thrust force ($F_z$) for both cutting conditions: with and without laser heating.
In this analysis, scratches analyzed for both conditions are in the ductile regime. The results summarized in Table 5.4 show that for the same amount of applied thrust force ($F_z = 20\text{mN}$), the scratch performed with laser heating yielded in a greater depth of cut (400 nm vs. 280 nm). It is also evident that the scratch performed with laser heating yielded a slightly lower cutting force for an equal applied thrust force, although the scratch performed with laser heating is significantly deeper. A scratch without laser heating done at higher loads to result in a depth of cut of 400 nm will most definitely result in higher cutting forces due to the hardness of the material.

<table>
<thead>
<tr>
<th>Cutting Condition</th>
<th>Thrust Force ($F_z$)</th>
<th>Cutting Force ($F_x$)</th>
<th>Depth of Cut</th>
<th>Scratch Nature</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Laser</td>
<td>20 mN</td>
<td>6.0 mN</td>
<td>280 nm</td>
<td>Ductile</td>
</tr>
<tr>
<td>With Laser</td>
<td>20 mN</td>
<td>5.5 mN</td>
<td>400 nm</td>
<td>Ductile</td>
</tr>
<tr>
<td>No Laser</td>
<td>35 mN</td>
<td>9.0 mN*</td>
<td>480 nm*</td>
<td>DBT</td>
</tr>
<tr>
<td>With Laser</td>
<td>42 mN</td>
<td>8.0 mN*</td>
<td>710 nm*</td>
<td>DBT</td>
</tr>
</tbody>
</table>

*Just before the DBT occurs.

The second analysis is to study the effect of laser heating on the DBT of the material. To determine this, two-dimensional scratch/groove profiles obtained using a white light interferometer are analyzed. Figure 5.14 shows the cross-section of the two scratches taken at an equal thrust force of approximately 35mN. It can be seen that the scratch performed with laser heating (left) exhibits a perfectly ductile behavior with a depth of 700 nm, whereas the scratch done without laser heating indicates initial fracture behavior at a depth of 480 nm. The brittle behavior is identified by the imperfect pattern or poor definition of the groove edge which is a representation of the stylus imprint on the material. Figure 5.15 illustrates a three-dimensional scratch profile that gives a clearer graphical illustration of the nature of the scratch: i.e., ductile or brittle. Here, it
can be clearly identified that the scratch performed with laser heating still exhibits ductile behavior whereas the scratch performed with no laser heating shows brittle behavior for similar thrust forces. The well defined edges depict the stylus imprint as good indication of ductile response of the material (seen in the scratch performed with laser heating) shown in Figure 5.15.

![Figure 5.14: Cross-section depth profile of the scratches.](image)

![Figure 5.15: 3D scratch profiles of the scratches with and without laser heating.](image)

*Figure 5.14: Cross-section depth profile of the scratches.*

*Figure 5.15: 3D scratch profiles of the scratches with and without laser heating.*

*Figure 5.16* shows the cross-section of the same two scratches taken at an equal thrust force of approximately 42 mN. It is clearly observed that the scratch performed with laser heating indicates initial fracture behavior at a depth of 710 nm. At this load, the scratch performed with no laser heating shows signs of severe fracture. In comparison, the DBT depth of the scratch performed with laser heating is approximately 230 nm greater, ~ 48% greater than the DBT depth of the scratch performed without laser heating.
Figure 5.17 shows the cutting force comparison for the scratch done with and without laser heating for a linearly increasing thrust force/load. Force data analyses are yet another way utilized to confirm the DBT and identify the beginning of fracture behavior in the material. As seen in the cutting force data, instability (large peaks and valleys on a linearly increasing line) in cutting forces ($F_c$) are usually correlated to fracture occurrence along the scratch. Another advantage of force analysis is that fracture occurrence can be observed in-situ via the instability observed in the cutting forces during the scratch test experiments. This is an important tool while attempting ductile mode machining of brittle materials as the force data will indicate the beginning of brittle behavior allowing the machinist to reduce the depth of cut or thrust force in real-time, avoiding fracture or even catastrophic failure in the material.
5.3.2.2 CUTTING WITH A SPDT TOOL

Since the aim of this set of experiments is to determine the laser heating effect using a regular cutting tool, instead of a stylus, a total of ten cuts are performed on a single crystal Si wafer. These cuts are performed at five different loads with (WL ∼ 255 mW)) and without laser (NL) heating conditions. A summary of all scratch parameters and results is given in Table 5.5.

Table 5-5: Summary of cuts performed with a cutting tool using low laser powers.

<table>
<thead>
<tr>
<th>Thrust Force ($F_z$)</th>
<th>Heating Condition</th>
<th>Cutting Force ($F_x$)</th>
<th>Width of Cut</th>
<th>Depth of Cut (DoC)</th>
<th>% Increase in DoC</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 mN ± 0.5%</td>
<td>No Laser</td>
<td>18.8 mN ± 0.8%</td>
<td>32 µm</td>
<td>50 nm</td>
<td>≈20%</td>
</tr>
<tr>
<td>50 mN ± 0.5%</td>
<td>With Laser</td>
<td>18.5 mN ± 0.7%</td>
<td>32 µm</td>
<td>60 nm</td>
<td></td>
</tr>
<tr>
<td>25 mN ± 1.0%</td>
<td>No Laser</td>
<td>6.6 mN ± 3%</td>
<td>19 µm</td>
<td>31 nm</td>
<td>≈13%</td>
</tr>
<tr>
<td>25 mN ± 1.0%</td>
<td>With Laser</td>
<td>6.5 mN ± 5%</td>
<td>19 µm</td>
<td>35 nm</td>
<td></td>
</tr>
<tr>
<td>20 mN ± 1.5%</td>
<td>No Laser</td>
<td>4.7 mN ± 7.3%</td>
<td>17 µm</td>
<td>28 nm</td>
<td>≈14%</td>
</tr>
<tr>
<td>20 mN ± 1.3%</td>
<td>With Laser</td>
<td>4.9 mN ± 8.1%</td>
<td>18 µm</td>
<td>32 nm</td>
<td></td>
</tr>
<tr>
<td>15 mN ± 1.7%</td>
<td>No Laser</td>
<td>2.9 mN ± 7.8%</td>
<td>15 µm</td>
<td>26 nm</td>
<td>≈8%</td>
</tr>
<tr>
<td>15 mN ± 1.7%</td>
<td>With Laser</td>
<td>3.1 mN ± 6.9%</td>
<td>15 µm</td>
<td>28 nm</td>
<td></td>
</tr>
<tr>
<td>10 mN ± 2.9%</td>
<td>No Laser</td>
<td>1.8 mN ± 5.4%</td>
<td>10.5 µm</td>
<td>16 nm</td>
<td>≈25%</td>
</tr>
<tr>
<td>10 mN ± 2.8%</td>
<td>With Laser</td>
<td>2.0 mN ± 3.2%</td>
<td>10.5 µm</td>
<td>20 nm</td>
<td></td>
</tr>
</tbody>
</table>
The depths and widths of cuts are measured using a white light interferometer that provides angstrom resolution in the vertical direction; in this case, for the depth of cut measurement. The cutting forces for the cuts performed with laser heating are approximately the same as the cuts performed with no laser heating, even though all cuts performed with laser heating resulted in greater depths for an equal applied load. This means that the relative cutting forces are lower (when comparing the depth-to-cutting force ratio) for the cuts performed with laser heating; i.e., a cut done with no laser will certainly yield a greater cutting force to match the depth of cut done with laser heating. The laser heating effect is clearly demonstrated in Figure 5.18 where the trend suggests that at equal applied loads, the cuts performed with laser heating yield greater depths of cuts when compared to the cuts with no laser heating.

![Thrust Force vs. Depth of Cut](image)

Figure 5.18: Thrust force versus the depth of cut for both conditions.

Visually, there are no differences observed between the cuts (i.e., the cuts done with laser heating showed no discoloration). Figure 5.19 shows an optical microscope
image comparing a cut done with and without laser heating under a similar applied load.

![Image](image.png)

**Figure 5.19:** A segment of the scratch done at 20 mN.

Finally, an example of the force data comparing both heating conditions (no laser and with laser) for a cut done at 10 mN (applied load) is shown in Figure 5.20.

![Force Data](force_data.png)

**Figure 5.20:** Force data comparing with and without laser heating conditions.

The force data suggest that the cut performed with laser heating yields slightly higher cutting forces due to the greater depth of cut (60 nm vs. 50 nm). The average forces (over the entire 300 μm cut) are reported in Table 5.5.

### 5.3.3 HIGH POWERED LASER

For this set of experiments, several machining parameters such as the applied load (resultant depth of cut), laser power, cutting force and cutting speed are analyzed. The
availability of higher laser powers (up to 100 W) allows for cutting at higher speeds, which further helps in understanding the thermal softening effect at higher material removal rates. It is important to note that since a shorter wavelength laser is used here (1070 nm versus 1480 nm in the previous section) the absorption of both the Si-I and Si-II phase increase (based on Figure 5.1).

5.3.3.1 APPLIED LOAD VERSUS LASER POWER

The first set of experiments performed in this section is to study the correlation between the applied load (thrust force, $F_z$), resulting depth of cut and laser power. A set of cuts consisting of three loads (50 mN, 100 mN and 150 mN) and three heating conditions (no laser, 10 W and 45 W) are applied. All cuts performed were in the ductile regime as brittle mode machining is not intended in this study. A 10 W laser power is chosen as one of the testing parameters as it is the lowest limit in this particular laser unit (IPG power range 10 – 100 W). On the other hand, the 45 W laser power is chosen as it is determined to be the optimized laser power for this particular cutting speed (1 μm/sec). This is determined by preliminary experiments performed to optimize the laser power for a specific cutting speed. The power is determined to be optimized when the maximum depth of cut is achieved for equal applied loads (e.g., comparing 0 W, 10 W, 20 W….etc.). Too low (lack of heating) of a laser power will not result in sufficient thermal softening where as an excess of laser power (excess heating) causes the material to melt or burn. Figure 5.21 shows an example of a burn melting mark on the surface of Si caused by excess laser power/heating.
Figure 5.21: A burn mark on Si as a result of excess laser heating.

The burn mark in Figure 5.21 is made intentionally to assist during the beam alignment process (beam in this particular image is not aligned). The depth of the burn mark shown is in the range of 350 – 400 nm (measurement using a white light interferometer shown in appendices).

Table 5.6 summarizes the scratch parameters and results obtained from this set of experiments where the applied load ($F_z$), depth of cut, width of cut, cutting force ($F_x$) and laser power is compared.

Table 5-6: Scratch test parameters at a constant cutting speed.

<table>
<thead>
<tr>
<th>$F_z$</th>
<th>Heating Condition</th>
<th>Average Depth</th>
<th>Average Width</th>
<th>$F_x$</th>
<th>Power Density/Unit Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 mN</td>
<td>No Laser</td>
<td>45.0 nm</td>
<td>16 μm</td>
<td>14 mN</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>47.5 nm</td>
<td>17 μm</td>
<td>13 mN</td>
<td>7 GW/cm</td>
</tr>
<tr>
<td></td>
<td>45 W</td>
<td>51.0 nm</td>
<td>19 μm</td>
<td>12 mN</td>
<td>33 GW/cm</td>
</tr>
<tr>
<td>100 mN</td>
<td>No Laser</td>
<td>80.5 nm</td>
<td>32 μm</td>
<td>32 mN</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>84.5 nm</td>
<td>33 μm</td>
<td>32 mN</td>
<td>7 GW/cm</td>
</tr>
<tr>
<td></td>
<td>45 W</td>
<td>92.0 nm</td>
<td>35 μm</td>
<td>32 mN</td>
<td>33 GW/cm</td>
</tr>
<tr>
<td>150 mN</td>
<td>No Laser</td>
<td>104.0 nm</td>
<td>36 μm</td>
<td>58 mN</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>123.0 nm</td>
<td>37 μm</td>
<td>58 mN</td>
<td>7 GW/cm</td>
</tr>
<tr>
<td></td>
<td>45 W</td>
<td>131.5 nm</td>
<td>40 μm</td>
<td>57 mN</td>
<td>33 GW/cm</td>
</tr>
</tbody>
</table>

Note. All forces reported are within a 7% error.
All cuts reported in Table 5.6 are carried out at a constant speed of 1 μm/sec. The trend is consistent for all three applied loads where the cuts performed with laser heating yield greater depths than the cuts performed with no laser heating. The cuts performed with the optimized laser power (45 W) yields the greatest depth of cut, which is expected. This effect is also explained by the total power density delivered per unit length. Power density per unit length is used in this study to normalize different cutting speeds that will be discussed in later sections (power density per unit length, per second). The cut done at 45 W also yields a significantly higher power density (33 GW/cm vs. 7 GW/cm at 10W), thus, further contributing to the enhanced ductility of the material. The power density calculation is shown in the appendices. The width of cut is generally a function of the depth of cut as it takes the impression of the tool tip; i.e., deeper cuts result in wider cuts. Finally, the cutting forces are approximately the same for all conditions (for equal loads). This is promising due to the fact that although the cuts with laser heating yielded greater depths, the cutting forces do not increase proportionally. Low cutting forces are often desired during a machining process to minimize tool wear.

The effect of laser heating on the depth of cut for all three applied loads is clearly illustrated in Figure 5.22. From the figure, it is also observed that the laser heated cuts at 150 mN yielded the highest depth differences, i.e., 18% and 26%, 10 W and 45 W respectively. This is believed to be caused by the wider cut (at higher loads) that provides the optimized machined area per Watt of laser heating. The cuts performed at a 150 mN load are approximately 35 – 40 μm wide whereas the laser beam spot side is approximately 10μm focused at the deepest part of the cut, which is at the center of the cut.
5.3.3.2 APPLIED LOAD, LASER POWER AND CUTTING SPEED

The aim here is to study the effect of laser heating at different cutting speeds. The combination of laser powers and cutting speeds is important to understand as higher cutting speeds will eventually be utilized in SDPT operations (future work). The first set of experiments study the effect of minimum laser powers on cutting speeds and depths. For this experiment, the minimum power (10 W) is combined with a constant applied load (150 mN) and three cutting speeds: (1 μm/sec, 2 μm/sec and 5 μm/sec. Figure 5.23 illustrates the effect of these parameters on the depth of cut, which is the basis in analyzing the thermal softening effect.
Figure 5.23: The correlation of depths of cuts and cutting speeds for a low laser power.

*Figure 5.23* clearly illustrates that the cutting speed certainly has an impact on the final depth of cut for an equal applied load (in this case 150 mN) and constant laser power (10 W). An example of a cut performed at a 10 W laser power is shown in *Figure 5.24*. The chip accumulation at the end of the cut is a good indication of a ductile mode machining process.

*Figure 5.24*: Laser assisted cut with chip accumulation.
Although all cuts performed with laser heating yielded greater depths than the cuts with no laser heating, the cut performed at a speed of 1 μm/sec and 10 W yielded the deepest cut, approximately 20 % deeper than with no laser. However, as the cutting speed is increased, the increase in depth of the cuts with laser becomes less significant. This is because of the lack of thermal softening effect that occurs at higher cutting speeds. To study this effect, a different set of experiments are performed where optimized laser powers are determined for each of the three cutting speeds: 1 μm/sec, 2 μm/sec and 5 μm/sec. The experimental data is summarized in Table 5.7.

Table 5-7: Experimental summary comparing parameters at different cutting speeds.

<table>
<thead>
<tr>
<th>Cutting Speed</th>
<th>Laser Power</th>
<th>Applied Load</th>
<th>Cutting Force</th>
<th>Depth of Cut (nm)</th>
<th>A Depth</th>
<th>Width of Cut (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 μm/sec</td>
<td>No Laser</td>
<td>50mN ±2.0%</td>
<td>14mN ±18%</td>
<td>45-50</td>
<td>6-8%</td>
<td>19.3</td>
</tr>
<tr>
<td></td>
<td>45W ±2W</td>
<td>50mN ±4.5%</td>
<td>12mN ±20%</td>
<td>49-53</td>
<td></td>
<td>16.4</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>100mN ±1.8%</td>
<td>32mN ±10%</td>
<td>78-83</td>
<td>11-13%</td>
<td>32.4</td>
</tr>
<tr>
<td></td>
<td>45W ±2W</td>
<td>100mN ±1.8%</td>
<td>32mN ±9%</td>
<td>88-92</td>
<td></td>
<td>34.1</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>150mN ±1.3%</td>
<td>58mN ±4%</td>
<td>115-118</td>
<td>11-14%</td>
<td>37.8</td>
</tr>
<tr>
<td></td>
<td>45W ±2W</td>
<td>150mN ±1.6%</td>
<td>57mN ±5%</td>
<td>128-135</td>
<td></td>
<td>39.8</td>
</tr>
<tr>
<td>2 μm/sec</td>
<td>No Laser</td>
<td>50mN ±5.4%</td>
<td>14mN ±18%</td>
<td>55-60</td>
<td>8-9%</td>
<td>22.2</td>
</tr>
<tr>
<td></td>
<td>50W ±2W</td>
<td>50mN ±18%</td>
<td>11mN ±34%</td>
<td>60-65</td>
<td></td>
<td>21.5</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>100mN ±4.6%</td>
<td>28mN ±10%</td>
<td>80-85</td>
<td>12-13%</td>
<td>25.5</td>
</tr>
<tr>
<td></td>
<td>50W ±2W</td>
<td>100mN ±10%</td>
<td>29mN ±12%</td>
<td>90-95</td>
<td></td>
<td>27.1</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>150mN ±2.7%</td>
<td>48mN ±5.6%</td>
<td>100-108</td>
<td>11-15%</td>
<td>31.6</td>
</tr>
<tr>
<td></td>
<td>50W ±2W</td>
<td>150mN ±6.5%</td>
<td>48mN ±7.0%</td>
<td>115-120</td>
<td></td>
<td>32.9</td>
</tr>
<tr>
<td>5 μm/sec</td>
<td>No Laser</td>
<td>50mN ±7.4%</td>
<td>14mN ±20%</td>
<td>60-65</td>
<td>7-8%</td>
<td>19.7</td>
</tr>
<tr>
<td></td>
<td>55W ±2W</td>
<td>50mN ±23%</td>
<td>12mN ±35%</td>
<td>65-70</td>
<td></td>
<td>20.5</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>100mN ±3.8%</td>
<td>32mN ±7.3%</td>
<td>80-85</td>
<td>23-25%</td>
<td>27.1</td>
</tr>
<tr>
<td></td>
<td>55W ±2W</td>
<td>100mN ±11%</td>
<td>32mN ±17%</td>
<td>110-115</td>
<td></td>
<td>29.6</td>
</tr>
<tr>
<td></td>
<td>No Laser</td>
<td>150mN ±3.7%</td>
<td>58mN ±5.4%</td>
<td>135-140</td>
<td>11-14%</td>
<td>40.3</td>
</tr>
<tr>
<td></td>
<td>55W ±2W</td>
<td>150mN ±8.4%</td>
<td>57mN ±9.9%</td>
<td>150-160</td>
<td></td>
<td>41.9</td>
</tr>
</tbody>
</table>

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The data suggest a consistent trend where the depths for cuts performed with laser are greater than with no laser. However, it is important to note that as the cutting speed is increased, higher laser powers are required to have optimum thermal softening effects at that particular cutting speed. Figure 5.25 illustrates the effect of cutting speeds and optimized laser powers on the depth of cut. The optimized laser power is determined by performing cuts at a constant load and speed at increasing intervals of laser power. The depth of cut is then measured for all cuts and the power that yields the deepest cut is defined as the optimum laser power. It is important to note that excess laser powers will cause excess heating resulting in a material that is too soft causing it to flow back into the cut groove and resulting in a shallower depth of cut.

![Cutting Speed vs. Laser Power Summary](image)

**Figure 5.25:** Effect of optimized laser powers on the depth of cut.

This set of experiments further emphasizes the importance of optimizing the laser power for different cutting speeds in order to maximize on the thermal softening effect. The trend in Figure 5.25 also serves as a benchmark for future work where higher cutting speeds are required (e.g., for SPDT operations).
5.3.3.3 DUCTILE TO BRITTLE TRANSITION

The effect of optimized laser heating on the DBT of Si is determined. A single cut with increasing loads (50 mN to 300 mN) is performed with and without laser heating. Since a 1 µm/sec cutting speed is used, the corresponding optimized laser power (45 W) is implemented for the cut performed with laser heating. Figure 5.26 shows an example of a cut that exhibits both ductile and brittle regimes with a DBT zone and its corresponding depths of cuts.

![Image showing a cut with ductile and brittle regimes and DBT depths](image)

**Figure 5.26**: A cut exhibiting both ductile and brittle regimes.

The DBT of the material is identified at the point where constant brittle activity starts, in the case of Figure 5.26 the DBT depth is approximately 185 nm. Table 5.8 shows the difference in DBT depths between the ‘no laser’ and ‘with laser’ conditions.

<table>
<thead>
<tr>
<th>Cutting Condition</th>
<th>Thrust Force ($F_z$)</th>
<th>Cutting Force ($F_x$)</th>
<th>DBT Depth</th>
<th>Increase in DBT</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Laser</td>
<td>175 mN</td>
<td>105 mN</td>
<td>185 nm</td>
<td></td>
</tr>
<tr>
<td>45 W</td>
<td>200 mN</td>
<td>120 mN</td>
<td>260 nm</td>
<td>41 %</td>
</tr>
</tbody>
</table>

Table 5-8: DBT scratch test parameters.
The experimental data suggest a significant increase (~ 41%) in the DBT depth for the cut performed with optimized laser powers. This increase in ductility is vital in contributing to higher material removal rates during a SPDT operation. Also, the cutting forces did not increase significantly (increased by only 14%) for the cut with laser, even though a much deeper cut was obtained.

5.3.3.4 HARDNESS OF MATERIAL REMOVED REGIONS

After completing all scratch tests, the hardness of each material removed region is compared to the unmachined region via nanoindentations. A NanoIndenter XP (MTS) with a Berkovich indenter (a triangular pyramid with a centerline-to-face angle of 65.3°) is used. An image of the Nanoindenter setup is shown in the appendices. Figure 5.27 shows a load-displacement curve for a cut performed on the Si wafer with no laser heating.

![Figure 5.27: Load-displacement curve for a scratched region.](image)

The indentation depth (displacement) is kept to a maximum of 100 nm for all nano-indents performed in this study. This is to ensure that the indented region is within
the phase transformed region. All regions analyzed here have been previously cut at an applied load of 150 mN that yielded in depths of cuts greater than 100 nm. From literature, it is known that the phase transformed/amorphous layer thickness is approximately equal to the depth of the cut. A displacement discontinuity in the loading and unloading curves, commonly referred to as “pop-in” and “pop-out” respectively is observed in Figure 5.27 (this phenomenon is noticed in all indented regions analyzed). These events are commonly interpreted as an abrupt phase transformation (accompanied by a volume change) from Si-I to Si-II (for “pop-in”) and Si-II to the Si-XII phase (for “pop-out”) on pressure release. However, there is still insufficient evidence to fully understand the physics governing these events.

The hardness of four different regions are obtained; unmachined (pristine) surface, cut performed with no laser heating (at 150 mN), cut performed with low laser power – 10W (at 150 mN) and cut performed at optimized laser power - 45 W (at 150 mN). Figure 5.28 shows the hardness comparison for all regions experimented using a cutting tool and various laser powers. A total of five indents are performed on each region (the averages along with the distribution ranges are provided in Figure 5.28)

![Hardness comparison for different test regions](image_url)

**Figure 5.28: Nanoindentation hardness for various scratched regions.**
The average hardness of all material removed regions (with and without laser heating) are higher (by almost 15%) than the average hardness of the original (pristine) Si surface. The effect observed is similar to that reported for conventional machined metals, where the near-surface layer always becomes harder due to the work-hardening effect.\textsuperscript{203} In metals, the work hardening effect is governed by the dislocation movements within the crystal structure. However, for semiconductors like Si, the increase in hardness in the material removed regions (as observed here) is still not clear and requires further investigation. There is no significant difference in hardness among the material removed regions when comparing the laser assisted cuts to the cut performed with no laser heating.

5.3.4 TEMPERATURE MEASUREMENT

Three power-temperature ranges were obtained from the tests using the liquid temperature lacquers. The threshold of the lacquers can be easily identified once reached, as a significant difference in surface texture is noticed. At times, a burn mark on the coated surface is observed indicating the lacquer has reached its threshold temperature. Since there is no direct method (in-situ) to monitor the change in surface texture of the lacquer coating, the laser power is gradually increased and the coating is periodically imaged under a microscope. \textit{Figure 5.29} shows four examples of temperature lacquers reaching its respective threshold temperatures.
Figure 5.29: The effect of laser heating on temperature indicating lacquers.

From Figure 5.29, it is observed that the effect obtained from the 510 °C lacquer is least defined. This is because the threshold temperature was achieved rapidly at a fairly low laser power (15-17 W). The experimentally measured temperatures for various power ranges are summarized in Table 5.9. It is also important to note that the tool is moving at a very low speed of 1 μm/sec. This means that in an actual cutting test, these temperature measurements are bound to change as the cutting speed is varied, or in other words, higher temperatures are expected for slower speeds at similar laser powers.

Table 5-9: Estimated temperatures for different laser power ranges.

<table>
<thead>
<tr>
<th>Laser Power</th>
<th>Measured Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 – 17 W</td>
<td>≥ 510 °C</td>
</tr>
<tr>
<td>22 – 24 W</td>
<td>≥ 704 °C</td>
</tr>
<tr>
<td>28 – 30 W</td>
<td>≥ 816 °C</td>
</tr>
<tr>
<td>38 – 40 W</td>
<td>≥ 1038 °C</td>
</tr>
</tbody>
</table>
5.3.5 MICRO-RAMAN SPECTROSCOPY

Raman spectroscopy is used in this study to detect and analyze phase transformations for scratched regions and machined chips. The first comparison done using μ-Raman is to study the formation of high pressure phases as a function of cutting speeds. Figure 5.30(a) compares the μ-Raman spectra for the cut done with no laser heating and the cuts done with minimum laser power (10 W) for all three cutting speeds: 1 μm/sec, 2 μm/sec and 5 μm/sec. From the earlier experiments, discussed in previous sections, there is a change in depths noticed when the cutting speed is varied for a constant laser power of 10 W. However, the Raman spectra in Figure 5.30(a) shows that high pressure phases (HPPs) are formed for all conditions, including the cut with no laser heating, i.e., the shallowest cuts. Similar HPPs (Si-I, Si-III, Si-IV and Si-XII) are identified for all cuts. Although not considered a HPP, Si-I is believed to be seen in the spectra for two reasons; (1) the Raman laser could be probing past the HPPs into the crystalline material or (2) the laser heat during μ-LAM recrystallizes the HPPs back to its original diamond phase. The annealing effect of the HPPs is clearly seen in Figure 5.30(b) where all HPPs have been annealed back to the original Si-I phase for the cuts performed with optimized laser powers. This is further discussed in detail in Chapter 7.
The next Raman analysis is performed on the accumulated chips collected at the end of the scratch. Chips from only two cutting conditions are compared here; (1) no laser heating at 150 mN and (2) 10 W laser power at 150 mN. Cuts done at optimized laser powers did not produce sufficient chip accumulation for the Raman analysis as it is believed to have been disintegrated due to the heating effect from high laser powers. This is because once the chips are detached from the workpiece, the surface area of the chips is too small for the heat to dissipate. Figure 5.31 compares the Raman spectra of the unmachined (pristine) surface to the chips obtained from the cuts (with and without laser heating).
The chips from both cutting conditions show amorphous silicon that is believed to have formed due to the back transformation and Si-IV (one of the stable HPPs). The existence of both of these phases is consistent with previous work where a-Si is attributed to the back transformation of the high pressure phases during the ductile mode material removal process and Si-IV is known as the intermediate phase before the final recovery of Si-I. However, the chips from the laser machined cut also exhibit the Si-I phase which is due to the recrystallization/annealing effect caused by the laser radiation (heat). To confirm this phenomenon, the laser source of the Raman microscope is utilized to perform in-situ heating on the chips collected from a cut performed with no laser heating. Figure 5.32 clearly depicts the annealing of the amorphous and Si-IV phases to form Si-I. The Raman power is increased at every stage and at approximately 50% power (3.5 mW), Si-I begins to form. At 100% power (7 mW), the Si-I peak is seen to be dominant.

Figure 5.31: Raman spectra of the unmachined Si surface and machined chips.
Figure 5.32: Annealing of the chips to form the Si-I phase.

Now that the annealing/recrystallization process of the chips is better understood, a phase transformation sequence diagram (for the chips) is constructed (Figure 5.34). SEM micrographs at different locations and magnifications of the plastically deformed chips used for Raman microscopy are shown in Figure 5.33.

Figure 5.33: Micrograph of plastically deformed chips used for Raman analysis. Note. Image magnification going clockwise – 1800x, 3500x, 7000x, and 11000x.
5.3.6 TRANSMISSION ELECTRON MICROSCOPY OF SILICON CHIPS

In order to characterize the microstructure of the machined chips after the cutting process, TEM analysis is carried out. In this analysis only the chips from the cut performed with no laser are studied. The chips obtained from the cut performed with laser heating are very brittle and insufficient in quantity; therefore, none were detected on the grid. Figure 5.35(a) shows a bright-field TEM image of a small part of the machined chip. Figure 5.35(b) displays the electron diffraction pattern from the chip which shows clearly a diffused halo ring. This is indicative that the chip is fully amorphous. This finding is consistent with previous work.

Figure 5.35: TEM analysis of machined chip: (a) bright-field TEM image of a part of a ductile chip and (b) selective area diffraction pattern which indicates that the structure of chip is amorphous.
As a result of the phase transformation, the chips (similar to the machined surface) are expected to be amorphous (in contrast to the starting single crystalline structure). The existence of the amorphous remnant in the chips is a substantial evidence of ductile mode machining. TEM analysis done by past researchers on Si chips from SPDT operations show both the amorphous region and crystalline structure indicating phase transformation (crystalline material transformed into an amorphous structure) due to significant plastic deformation during the material removal process. \(^{206}\)

5.4 CONCLUSION

Micro-laser assisted scratch tests were successful in demonstrating the enhanced thermal softening in single crystal silicon resulting in greater depths of cuts (when compared to similar applied loads for cuts with no laser) and greater ductile to brittle transition depths. Laser heating was successfully demonstrated as evidence by the significant increase in the ductile response of single crystal Si in the \(\{100\}\) plane along the \(<110>\) direction. Laser assisted (heating, thermal softening and reduced brittleness) material removal resulted in greater depths of cuts at less applied thrust forces, smaller cutting forces and a larger critical depth of cut. Force analyses (thrust and cutting), optical microscopy, white light interferometry, electron microscopy and Raman spectroscopy served as useful analyses (measurement and characterization) methods to detect the enhanced ductile response and reduced brittle fracture as a result of preferential material heating. There is a consistent trend, no matter the type of cutting tool used (stylus or cutting tool) or laser (variation in power, beam size and wavelength), showing several advantages for cuts performed with the \(\mu\)-LAM process. Results obtained from
this study are promising to further implement the \( \mu \)-LAM technique in machining operations such as single point diamond turning. Lower cutting forces obtained from the \( \mu \)-LAM process are favorable to minimize tool wear while machining abrasive ceramics/semiconductors such as Si. The results from this study also will benefit the manufacture of brittle materials as laser heating is proven to decrease the brittle response in ceramics and semiconductors (in this case silicon) which can result in high productivity rates (i.e. higher material removal rate).

5.5 FUTURE WORK

Single point diamond turning (SPDT) experiments will be carried out using the \( \mu \)-LAM setup. For the SPDT experiments, several machining parameters such as cutting speed (changes as a function of workpiece radius), depth of cut and feed will be experimented and optimized. The ultimate goal for the SPDT experiments is to improve the surface finish of an as-received Si workpiece. In this particular study, however, the surface finish is not expected to improve as the initial SPDT experiments will be carried out on a polished Si wafer (Ra<10nm). The surface features (feed marks) from the machining experiments will generally cause the surface roughness to increase. However, these experiments will be a good indication on the effects of laser heating in SPDT. The cuts will be compared to conventional SPDT cuts (with no laser heating) with similar machining parameters for various depths of cuts and feeds in the ductile regime. The \( \mu \)-LAM-SPDT parameters such as depth of cut, feed, cutting speed and laser power will then be optimized to achieve the maximum material removal rate while improving the surface finish, and minimizing tool wear. In addition to the machining work, TEM
analysis will be performed on chips collected from the µ-LAM process to be compared with the current TEM analysis (on chips from cuts with no laser heating). Finally scanning acoustic microscopy (SAcM) will be performed on the laser machined samples to studied subsurface damage such as thermal cracks (refer to Chapter 3).
6.1 INTRODUCTION

Semiconductors and ceramics like silicon carbide (SiC) share common characteristics of being nominally hard and brittle, which stems from their covalent chemical bonding and crystal structure. SiC is an important material in many engineering applications, but it is particularly difficult to machine in traditional manufacturing processes due to its extreme hardness and brittleness. SiC has many desirable properties, such as excellent wear resistance, chemical stability, wide energy Bandgap, high electric field breakdown strength, high maximum current density and high strength even at elevated temperatures. All of these properties make it an ideal candidate for tribological, semiconductor, MEMS and optoelectronic applications. In spite of all these beneficial characteristics, the difficulty during machining and material removal has been a major obstacle that limits the wider application of SiC.

The plastic deformation of these nominally brittle materials (in this case SiC) at room temperature is much less than in metals, which means they are more susceptible to fracture during material removal processes. Surface cracks generated during machining are subsequently removed in lapping and polishing processes, which significantly increases the machining time and cost. Machining mirror-like surface finishes contribute
significantly to the total cost of the manufactured part. In some cases, grinding and polishing alone can account for 60-90% of the final product cost.\textsuperscript{74} In this context, developing a cost effective method to achieve a flawless surface in ultra-fine surface machining of an optical lens or mirror has become a challenge. In many engineering applications, products require a high quality surface finish and close tolerances to function properly. This is often the case for products made of semiconductor or ceramic materials. The real challenge is to produce an ultra-precision surface finish in these nominally brittle materials at relatively low machining costs.

Current limitations for brittle material machining include the high cost of processing and low product reliability. The cost is mainly due to the high tool cost, rapid tool wear, long machining time, low production rate and the manufacturing of satisfactory surface figure and form. The low product reliability is primarily due to the occurrence of surface/subsurface damage, i.e., cracks, and brittle fracture. In order to develop a suitable process, ductile regime machining, considered to be one of the satisfactory precision machining techniques, has been continuously studied over the last two decades.\textsuperscript{76,77,78,207,80,81,82,83,84} Laser assisted micro/nano machining is another important development in this direction.\textsuperscript{85,86} Laser assisted machining constitutes an alternative to traditional manufacturing and can be used to machine a variety of materials including metals, ceramics, glass, plastics and composites. The addition of heat during a machining process adds several advantages such as minimize tool wear, prevent tool breakage, reduce chatter, increased material removal rates and less mechanically induced material damage.\textsuperscript{208,209} Early research on workpiece preheating in particular, using a laser source, focused on improving the machinability of stainless steels, titanium alloy
and nickel based superalloys.\textsuperscript{210} However, the low laser absorptivity of metals in the infrared, as compared to ceramics, and additional cost for operating early high-power lasers made the technique economically unattractive.\textsuperscript{211}

In previous research work, it has been demonstrated that ductile regime machining of brittle materials is possible due to the high pressure phase transformation (HPPT) occurring in the material caused by the high compressive and shear stresses induced by the single point diamond tool tip.\textsuperscript{212,176} To further augment the ductile response of these materials, traditional scratch/single point diamond turning tests are coupled with a micro-laser assisted machining (\(\mu\)-LAM) technique.\textsuperscript{88} A schematic of the basic underlining concept of the \(\mu\)-LAM process is shown in Figure 6.1. This hybrid method is implemented to increase the critical depth of cut (DoC) (larger DBT depth) in ductile regime machining, resulting in a higher material removal rate. \(\mu\)-LAM was successfully carried out on single crystal Si yielding a greater DBT depth (for the scratch performed with laser heating).\textsuperscript{89}

![Figure 6.1: A schematic cross-section of the \(\mu\)-LAM process.](image)
The objective of the current study is to determine the effect of laser heating (using the \( \mu \)-LAM process) on the material removal of SiC using single point scratch testing, using a stylus and single point diamond cutting tool. The scratch tests are carried out to examine the effect of thermal softening of the high pressure phases formed under the diamond tip. The effect of laser heating on the ductile-to-brittle transition (DBT) of the material along with the optimization of machining parameters such as applied load, depth of cut, cutting force, cutting speed, laser wavelength and laser power is also studied. The three most commonly used polytypes of SiC are studied in this research; (1) 4H (hexagonal) single crystal SiC, (2) 6H (hexagonal) single crystals SiC and, (3) 3C (cubic) CVD coated polycrystalline SiC.

6.2 EXPERIMENTAL METHOD

All single point diamond scratch tests are performed on the Universal Micro-Tribometer (UMT) that was developed (by CETR, Inc.) to perform comprehensive micro-mechanical tests of coatings and materials at the micro scale. This versatile equipment facilitates cutting speeds as low as 1 \( \mu \)m/sec at nanometric cutting depths. Since the UMT is load controlled, very high vertical resolution is achieved based on the load cell range selected (up to 1 mN force resolution is achievable). The UMT is equipped with a linear stage, high precision air-bearing spindle (obtained separately and incorporated to the UMT), tilt stage and dual-axis load cell that is capable of constantly monitoring the thrust \( F_z \) and cutting forces, \( F_x \) (obtained as an output parameter from the cutting experiment).

The \( \mu \)-LAM system is coupled with the UMT to perform in-situ laser assisted cuts
(the setup is shown in the following sections). Two different lasers are utilized in this study to experiment on different wavelengths and powers. The beam diameter for both laser units is fairly small (~10 μm) as the cuts performed on SiC are fairly narrow (<15 μm). Since the lasers used have a Gaussian beam profile, a smaller beam size delivers maximum power (power density) to the center of the cut (the deepest region of the cut that is most prone to brittle fracture). The laser specifications utilized in this study are shown in Table 6.1.

Table 6-1: Laser devices and specifications.

<table>
<thead>
<tr>
<th>Laser Device</th>
<th>Wavelength (λ)</th>
<th>Power Range</th>
<th>Beam Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furukawa</td>
<td>1480 nm</td>
<td>0 – 400 mW</td>
<td>10 μm</td>
</tr>
<tr>
<td>IPG*</td>
<td>1070 nm</td>
<td>10 – 100 W</td>
<td>10 μm</td>
</tr>
</tbody>
</table>

*Operates with a beam delivery optics (BDO) unit by Laser Mechanisms Inc.

Similar to the work done on single crystal silicon (Si), reported on in chapter 5, all lasers used in this study are continuous wave (CW) fiber coupled lasers due to the relative ease of attaching a cutting tool/stylus to a SMA/FC-PC fiber coupler (i.e., to deliver the laser beam to the tool). All lasers utilized are in the near infrared (NIR) region, between ~1000 nm and 1500 nm wavelengths. Figure 6.2 shows the absorption of the atmospheric SiC phase at different wavelengths for all three polytypes. The absorption of the high pressure SiC phases is still unknown at this point but the research to study these optical properties via diamond anvil cell testing is in progress.
From Figure 6.2, it is observed that the average absorption of 4H-SiC decreases marginally with increasing wavelengths from 500 nm to 1500 nm. In the case of 6H-SiC, the percentage absorption largely depends on the wavelength from 500 nm to 1000 nm. However, after 1000 nm, the absorption of 6H-SiC stays almost constant. Lastly, 3C-SiC has the highest average absorption (≥ 95%) at all wavelengths at its atmospheric condition/phase (cubic structure). From the absorption data, it is expected that the brittle/atmospheric phase of 4H-SiC benefit the least and the 6H-SiC and 3C-SiC benefit significantly. However, this only assumes that the laser irradiation reduces the brittleness of the atmospheric phase of SiC (as the absorption of the high pressure phases are not known yet).

6.2.1 STYLUS COUPLED WITH LOW POWERED LASER

An IR fiber laser (λ = 1480 nm and P\textsubscript{max} = 400 mW) with a Gaussian profile beam of 10 μm in diameter is used in this part of the study. A distribution of the Gaussian beam intensity is shown in Figure 6.3. It is estimated that approximately 42% of the total
power (~ 65 mW out of a total output at the tip of 155 mW) is within a 2 μm diameter center area. This is beneficial in this study as the scratches performed are less than 3 μm in width.

The laser beam is guided through a 10 μm core fiber optic cable to a ceramic ferrule, which is attached to the diamond stylus. The μ-LAM system is configured in such a way that the IR beam passes through the optically transparent diamond tip (stylus) and impinges on the SiC workpiece at the tool-workpiece interface (contact), as illustrated in Figure 6.1. The stylus based scratch test setup is similar to that used for Si and is shown in Figure 5.2 (Chapter 5). All scratch tests using a stylus are performed on a single crystal 4H-SiC wafer on the {1010} plane along the <1010> direction. A 90° conical single crystal diamond stylus (with a spherical end tip radius of 5 μm) is used as the scratch tool. The details of the diamond tip attachment are depicted in Figures 5.3(a) and (b). The laser emerges from a 90° conical single crystal diamond tip with 5 μm radius spherical end.

In this part of the study, two conditions of scratches are performed: with and
without laser heating. The effects of laser heating are studied by verifying the depths of cuts and the nature of the scratches (i.e. ductile, DBT or brittle) for diamond stylus scratch tests carried out on single crystal 4H-SiC with increasing loads (thrust force). 4H-SiC is chosen for the stylus tests to study primarily the thermal softening effect on only the high pressure phases since the atmospheric phase of this polytypes absorbs the least at this wavelength (refer to Figure 6.2). The scratches are carried out at low cutting speeds (1 µm/sec) in order to maximize the thermal softening effect of the material during the in-situ laser heating process (since this is a low powered laser). Scratch lengths of 500 µm are produced on a 4H-SiC wafer specimen. The loads are increased linearly with time from 2 mN to 70 mN along the scratch. The scratch test parameters are summarized in Table 6.2.

### Table 6-2: Scratch testing parameters using a stylus and low powered laser.

<table>
<thead>
<tr>
<th>Scratch #</th>
<th>Load Range (Fz)</th>
<th>Condition</th>
<th>Cutting Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20 – 70 mN</td>
<td>No Laser (0 W)</td>
<td>1 µm/sec</td>
</tr>
<tr>
<td>2</td>
<td>20 – 70 mN</td>
<td>With Laser (350 mW*)</td>
<td>1 µm/sec</td>
</tr>
</tbody>
</table>

*400 mW is the maximum output power, approximately 155 mW is actually delivered to the workpiece material and the remaining power is lost due to scattering and reflections.

The load range is selected such that the scratches show both a ductile and brittle response (with a DBT region within the scratch). The loads, depths of cuts, cutting forces, laser power and scratch topography are measured, imaged and correlated to determine the laser heating effect on the ductile response during the material removal process.

#### 6.2.2 LASER BEAM ALIGNMENT

For the micro-laser assisted machined cuts performed in this study, good beam
alignment is crucial to deliver maximum laser power to the workpiece. The main challenge is to perfectly align a 10 μm IR beam to the cutting edge of the tool (exactly at the tool-workpiece interface). A misalignment in the laser beam causes a poorly focused beam, unintended thermal expansion of the substrate and most importantly a failure to capitalize on the laser heating and thermal softening effect. Since removing the tool each time from the BDO unit during the alignment process is not desired, an in-situ alignment technique is developed. This technique is different from the alignment procedure discussed in Chapter 5.

6.2.3 CUTTING TOOL COUPLED WITH HIGH POWERED LASER

A 1 mm nose radius single crystal diamond cutting tool (−45° rake and 5° clearance), similar to that used for SPDT experiments, is used for this set of experiments. An IR diode laser (λ = 1070 nm and P_{max} = 100 W) with a Gaussian beam profile of diameter approximately 10 μm is used in this investigation. The laser beam is guided through a fiber optic cable to a collimator, which is attached to a BDO. The BDO then focuses the beam (from 7.8 mm to 10 μm) and delivers it through the transparent diamond tool to the cutting edge. A scratch/cutting test setup along with the high laser powered μ-LAM system used for most of the experiments discussed in this chapter is shown in Figure 6.4.
Figure 6.4: High powered µ-LAM scratch test setup on the UMT.

Figure 6.4 illustrates a cutting setup used to perform material removal operations on 3C-SiC (a similar setup is used for the other polytypes). The reason a 10° inclined stage is used here is to compensate for an error during the diamond shaping process that caused the clearance angle to be offset by 10°. Due to the inclination in the stage, the effective rake angle is reduced to -55° (versus an intended -45°). However, since the relative differences are studied in these experiments (comparing conditions with and without laser heating), a -55° effective rake angle did not alter the overall objective along the course of study. Since no form of fluid cooling system can be implemented within the setup due to the lack of accessibility/space, an air cooling system is used. Another interesting artifact observed from *Figure 6.4* is the IR beam radiation at the tool tip. Although the IR beam cannot be seen by the naked eye, the camera lens enables the IR beam to be visible.

In this set of experiments, scratch tests are conducted to correlate the laser heating effect with the load/depth of cut, cutting force, DBT and cutting speed for all three SiC polytypes. The availability of higher laser powers allow cuts to be performed at higher
cutting speeds without compromising on the thermal softening effect. As the eventual goal of this research is to perform micro-laser assisted SPDT operations, the effect of laser heating incorporating the cross-feed parameters is also examined. Since the current setup limits the rotary motion of the workpiece, shaping cuts are performed to mimic the material removal kinetics of a SPDT operation. The load cell is currently inverted and used at the workpiece end (lower/bottom of UMT) to obtain better resolution from a smaller load range sensor (in this case a 5000 mN load cell is used). In a shaping operation, the tool has a reciprocating movement; the cutting taking place on the forward stroke along the whole length of the surface being generated, while the reverse stroke is made with the tool raised above, i.e., to clear the workpiece surface to avoid damage to the tool or the workpiece. The next stroke, cutting motion, is made when the tool has been moved by the feed distance and then lowered down and into the surface to create the next cutting pass, in this case horizontally across the surface (to replicate the cross-feed).

6.2.4 TEMPERATURE MEASUREMENT

Estimating the temperature achieved at the high pressure phase zone due to laser radiation is crucial in selecting machining parameters and understanding the material behavior at the microstructural level. To estimate these temperatures for various laser power ranges, OMEGALAQ liquid temperature lacquers, similar to that discussed in Chapter 5 are used. Since the size of the contact area/phase transformed region is extremely small, on the order of tens of microns, it is extremely difficult to use a probing technique (including thermal cameras) to measure temperature with the required resolution and accuracy. Temperature estimates for the heated zone are obtained for all
three SiC polytypes using a scratch setup similar to that shown in Figure 5.5 (Chapter 5). A micro-laser assisted cut is performed on a SiC workpiece that has a layer of liquid temperature lacquer deposited on the back-side of the wafer. An alternative configuration was initially tried where the thermal paint is simply applied to the surface of the workpiece and exposed to direct laser radiation. However, this technique proved very inaccurate as it did not correlate to the workpiece or high pressure phase properties. As a result, the temperature estimates obtained with the lacquer applied to the back-side of the workpiece is used instead.

6.2.5 MICRO-RAMAN SPECTROSCOPY

Micro-Raman (µ-Raman) spectroscopy is an effective non-destructive technique (NDT) used to characterize the amorphous remnant and high pressure phase transformed region in the machined surface of ceramics and semiconductors. Micro-Raman spectroscopy is one of the few, if not the only method that allows non-destructive phase analysis to be conducted in seconds with a spatial resolution of 1 µm (also depending on the wavelength of the laser beam) on a non-prepared surface or below the surface of the material.

The main goal of the µ-Raman analysis is to detect the occurrence of ductile mode machining, which is typically represented by the amorphous remnant and phase transformed region due to the high pressures at the tool tip-workpiece interface. A Renishaw inVia Raman microscope is used to detect and characterize the high pressure and back transformed phases in the machined region and accumulated machined chips. Using a 325 (Ultraviolet) He-Cd laser, a 1-2 µm spatial resolution is obtained. The UV laser is used here to minimize the probing depth, as the depths of cuts in this study (with
SiC) are not deep (less than 50 nm for most of the cuts). It is well studied that the shorter the Raman laser wavelength, the less the probing depth beneath the surface. This is important to ensure that the Raman laser probes within the range of the subsurface amorphous/high pressure phase layer thickness, which is approximately equal to the depth of cut. Probing too deep will result in only analyzing the bulk material properties and not the intended phased transformed region.

6.3 RESULTS AND DISCUSSION

6.3.1 STYLUS COUPLED WITH LOW POWERED LASER

In this study, there are two different analyses done based on the results obtained from the scratch tests performed on 4H-SiC. The first analysis compares the depth and cutting forces ($F_x$) for a constant thrust force ($F_z$) for both cutting conditions (with and without laser heating) to quantify the laser heating and thermal softening, and the relative hardness as a result of irradiation of the laser beam at a constant cutting speed. For this analysis, only the ductile region of the scratches is analyzed. The results summarized in Table 6.3 show that for the same amount of applied thrust force ($F_z = 30$ mN), the scratch performed with laser heating yielded a greater depth of cut (145 nm vs. 95 nm). It is also evident that cutting forces are equal for both these conditions for an equal applied thrust force (although the scratch performed with laser heating is significantly, ~50%, deeper). A scratch without laser heating done at higher loads to obtain a depth of 145 nm, comparable to the heating laser scratch performed at 30 mN resulting in a 10 mN cutting force, will most definitely result in higher cutting forces due to the hardness of the
The load range (2-70 mN) performed on these scratches is ideal for this study as it has both the ductile and brittle regime along the same scratch. The DBT is identified somewhere in between the ductile and brittle regime of the scratch using optical microscopy, white light interferometry and force analyses (from variations in cutting forces). Figures 6.5(a) and (b) show two scratches that represent the two conditions: without and with laser heating. It can be seen by comparing Figures 6.5(a) and (b) that the onset of brittle fracture occurs much earlier into the scratch, i.e., shallower depth, for the scratch performed with no laser heating (at identical loads). This initial indication of fracture behavior can be related to the force data seen in Figure 6.10 where the instability in cutting forces starts to occur.

![Micrographs showing the onset of brittle fracture along the scratch: (a) magnification at 200x, and (b) magnification at 1000x.](image)
Figure 6.6 shows both scratch conditions in the brittle region, i.e., at higher loads or loads above the critical load (load at the point the DBT occurs). From the image, it can be seen that even in the brittle region, the extent of brittle fracture occurring in the scratch performed with laser heating is less severe than the fracture seen along the scratch performed with no laser heating.

The second analysis done is to study the effects of laser heating on the DBT of the material. To determine this, cross-sectional scratch/groove profiles obtained using a white light interferometric profilometer is analyzed. Figure 6.7 shows the cross-section of the two scratches taken at an equal thrust force of approximately 35 mN. It can be seen that the scratch performed with laser heating (left) exhibits a perfectly ductile behavior whereas the scratch done without laser heating (right) indicates slight fracture (brittle behavior) of the material. The DBT depth identified for the scratch performed without laser heating just before the point of fracture is approximately 105 nm. The brittle behavior is identified by the imperfect pattern of the groove edge which is a representation of the stylus imprint on the material. It is important to note from Figure 6.7, that the scratch performed without laser heating is (apparently) deeper (210 nm vs.
113 nm) as it is difficult to control the depth when the material removal mechanism is brittle (i.e. difficult to predict the actual depth due to fracture of the material). The clear and defined edges that depict the stylus imprint are a good indication of ductile response of the material (as seen in the scratch performed with laser heating).

Figure 6.7: Cross-section of scratches obtained from a white light interferometric profilometer.

Figure 6.8 shows the cross-section of the same two scratches (at a different point) taken at an equal thrust force of approximately 40 mN. The DBT depth identified for the scratch performed with laser heating just before the point of fracture is approximately 240 nm. At this load, the scratch performed with no laser heating shows signs of severe fracture. In comparison, the DBT depth of the scratch performed with laser heating is approximately 135 nm (~ 130%) greater than the DBT depth of the scratch performed without laser heating (105 nm). The difference in DBT depths for both conditions is clearly illustrated in Figure 6.9.
From Table 6.3, it is seen that the cut performed with laser heating yields a slightly higher cutting force at the DBT. This is due to the higher thrust force (40mN vs. 35mN) and larger depth of cut (240 nm vs. 105 nm).

Analyzing the force data after the scratch experiments helps in correlating the onset of brittle fracture along the scratches. Brittle mode material removal is usually seen in the force data (especially cutting forces as it is more sensitive to brittle fracture) and
can be identified by its unstable behavior (higher standard deviation/ higher peaks-valleys in the force plots). Figure 6.10 shows the force data plot obtained from both scratching conditions (with and without laser heating). Monitoring the cutting forces during the material removal process is also an effective in-situ method to detect the onset of brittle regime machining (onset of fracture occurrence).

![Figure 6.10: Plot shows cutting force and thrust force data for both scratches.](image)

6.3.2 LASER BEAM ALIGNMENT

A new in-situ technique is developed to perform beam alignment without having to remove the tool. Since the diamond tool is attached to the BDO via a screw-in mechanism, removing the tool each time (e.g., for coating after performing preliminary beam positioning) is not desired as this will alter the position of the tool during re-assembly (depending on how tight the tool is screwed-in each time). To ensure the tool
position (relative to the beam) does not shift, beam alignment is performed while the tool is fixed on the μ-LAM system. *Figure 6.11* shows the setup used to perform in-situ beam alignment on the UMT.

*Figure 6.11*: In-situ laser beam alignment setup on the UMT.

Once the tool-workpiece contact area has been identified via the microscope, the guide beam (visible beam) is centered to the opening. Recall from Chapter 5 that the opening is created by first coating the diamond tool (with an opaque coating) and then removing the coating only at the contact area via scratching (i.e., a low load scratch is performed on a Si wafer). The microscope used here is a digital camera with a 550x magnification lens attachment. The advantage of using a highly magnified digital camera is that it provides sufficient working distance (between the lens and the tool tip) that enables easy beam positioning and focusing. *Figure 6.12* shows the guide beam focused to the contact area where the cutting edge is well defined and the main reflection of the beam is on the tool rake face of the tool (which is ideal). Once the guide beam is aligned, it is assumed that the IR beam is also aligned, as both beams are collinear.
Power measurements are generally a good indication and helpful guide throughout the alignment process. Therefore, power measurements are taken at all stages, i.e., after the preliminary beam positioning, after the tool has been coated and once the beam is aligned. *Figure 6.13* compares the output power (at the tool tip) at all stages during the in-situ alignment process.

*Figure 6.12: Guide beam aligned to the tool contact area.*

*Figure 6.13: Power throughput at the tool tip after each alignment stage.*

*Note. A newly designed tool (better optical efficiency) is used where approximately 45% power throughput is obtained (versus 23% for the tool discussed in Chapter 5)*

The lowest power throughput of less than 1% is obtained when the tool has been fully coated (throughput almost 0 W) and the highest power throughput of approximately
45% is obtained once the beam has been aligned to the tool cutting edge.

6.3.3 CUTTING TOOL COUPLED WITH HIGH POWERED LASER

Scratch tests are chosen to be the principal method of investigation in this study as it is a better candidate for evaluating machining conditions than indenting because the mechanics during scratching are more applicable to machining processes such as single point diamond turning (SPDT). The effect of laser heating on the ductile response during the material removal process is studied for three SiC polytypes; (1) 4H (hexagonal) single crystal SiC, (2) 6H (hexagonal) single crystals SiC and, (3) 3C (cubic) CVD coated polycrystalline SiC.

6.3.3.1 SINGLE CRYSTAL 4H-SIC

6.3.3.1.1 LOAD, LASER POWER AND DEPTH OF CUT

The first set of cuts done is to evaluate the effect of laser heating on the depth of cut for equal applied loads (thrust force). A range of heating conditions/laser powers from 10 W to 50W are studied and compared to the cut done with no laser heating. Table 6.4 summarizes the scratch parameters and results obtained from this set of experiments where the applied load ($F_z$), depth of cut, width of cut, cutting force ($F_x$) and laser power is compared.
Table 6-4: Scratch test parameters at a constant cutting speed.

<table>
<thead>
<tr>
<th>(F_z)</th>
<th>Heating Condition</th>
<th>Average Depth</th>
<th>Average Width</th>
<th>(F_x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>85 mN</td>
<td>No Laser</td>
<td>20.0 nm</td>
<td>9.5 μm</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>24.5 nm</td>
<td>10.0 μm</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>26.0 nm</td>
<td>10.5 μm</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>28.5 nm</td>
<td>10.5 μm</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>23.0 nm</td>
<td>10.0 μm</td>
<td>29 mN</td>
</tr>
<tr>
<td></td>
<td>50 W</td>
<td>19.0 nm</td>
<td>9.5 μm</td>
<td>26 mN</td>
</tr>
<tr>
<td>100 mN</td>
<td>No Laser*</td>
<td>23.0 nm*</td>
<td>10.5 μm</td>
<td>36 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>29.0 nm</td>
<td>11.0 μm</td>
<td>37 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>31.0 nm</td>
<td>11.5 μm</td>
<td>38 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>35.0 nm</td>
<td>12.0 μm</td>
<td>38 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>27.0 nm</td>
<td>11.0 μm</td>
<td>34 mN</td>
</tr>
<tr>
<td></td>
<td>50 W</td>
<td>24.0 nm</td>
<td>10.5 μm</td>
<td>33 mN</td>
</tr>
</tbody>
</table>

* Partially brittle cut due to excess loads.

All cuts reported in Table 6.4 are carried out at a constant speed of 1 μm/sec. It is seen that the depth of cut increases (for equal applied loads) up until the optimum laser power is reached (in this case 30 W). Beyond this apparent optimum laser power, the depth of cut starts to decrease. This is believed to be due to excess laser heating that causes the material to become too soft, that it then flows back into the groove at the trailing edge of the tool. Although the atmospheric phases of SiC are not known to melt at any pressure (only sublimes beyond 2700 °C)\(^{217}\), it is still possible to get the material soft enough at high temperatures to cause it to flow back into the cut groove. Also, there is the possibility of the high pressure phases melting or flowing similar to metals. However, this is assuming that the high pressure phases in SiC are metallic (like in Si), but this has not yet been confirmed. The cutting forces stay almost constant for both conditions (with and without laser heating) although the cut performed with low and optimized laser powers yielded greater depths. However, lower cutting forces are observed for the cuts done at higher laser powers (40 W and 50 W). Besides the fact that these cuts are not as deep as the cuts with an optimum laser power, the lower cutting
forces are yet another indication that the material is significantly softer. The importance of using an optimum laser power to obtain the greatest depth of cut is once again highlighted in Figure 6.14 where the highest percentage increment in depths is at an optimum laser power (43% at 85 mN and 52% at 100 mN).

Figure 6.14: Histogram showing the effect of laser power on the depth of cut.

6.3.3.1.2 CUTTING SPEED AND OPTIMUM LASER POWER

The next analysis done is to determine the correlation between optimum laser powers, cutting speeds and depths of cuts (illustrated in Figure 6.15). This is important to predict the thermal softening effect and material behavior at higher cutting speeds, for example during an actual SPDT operation. The surface power density delivered per unit length (cut length) in a second is seen to decrease as the cutting speed increases. The power density delivered per unit length (cut length) in a second is calculated based on the laser power, beam diameter and cutting speed.
Figure 6.15: Correlation between cutting speed, laser power and depth of cut.

The trend observed in Figure 6.15 suggests that the thermal softening effect can be maximized even at higher cutting speeds as long as sufficient/optimum laser powers are used. The depth of cut (in the range of 34 – 36 nm) and cutting force remain almost constant for all three speeds suggesting that similar effects can be achieved at much higher cutting speeds (with proportional laser powers). Presumably, based on the preliminary calculations (shown in the appendices) and observed trend (in Figure 6.15), at a maximum power of 100 W, a cutting speed of 1.5 cm/sec could be achieved (with 21 MW/cm surface power density deliver per unit of cut length). However, the thermal softening effectiveness at this cutting speed and laser power is yet to be determined.

6.3.3.1.3 SHAPING CUTS

The final cutting experiments for 4H-SiC are implemented with a cross-feed factor into the material removal mechanics (overlapping feed cuts as seen in Figure 6.17). To replicate an actual SPDT operation, shaping cuts are performed with three different feeds: 1 μm, 2 μm and 5 μm. All shaping cuts are performed at a constant speed of 1 μm/sec. An equally wide region of approximately 30 μm is machined for comparison.
purposes. This means that at a feed of 5 μm, fewer shaping cuts are required than at a feed of 1 μm to achieve similar machined area widths. To help in understanding this, a schematic is constructed in *Figure 6.16* to compare the total machined area widths and widths of individual cuts with respect to small and large feed machining conditions.

![Figure 6.16: Width of cut comparison to feed: (a) small feeds and (b) large feeds.](image)

For the condition with no laser heating, an applied load of 85 mN is chosen as the material starts to exhibit brittle behavior beyond this load. For the cuts performed with laser heating (30 W), an applied load of 120 mN is used for the similar reason. *Figure 6.17* summarizes the results from the shaping cuts for both conditions.

![Figure 6.17: Shaping cuts for three feeds comparing with and without laser heating conditions.](image)
The machined regions for all three feeds carried out with laser heating are significantly deeper than the regions with no laser heating. The enhanced ductility due to the laser heating and thermal softening effect enables the use of higher applied loads without causing brittle fracture (85 mN vs. 120 mN). \textit{Figure 6.18} shows three machined regions using 30 W of laser power. The feedmarks in each region are clearly visible similar to that observed in a turning operation.

\textbf{Figure 6.18: Three machined regions with the corresponding feeds.}

The trend also suggests that when the feed is increased, the depth of cut of the region decreases. This is consistent with the current scientific understanding and it is true for both, ‘no laser’ and ‘with laser’ machining conditions. When the feed increases, the length of the tool path and contact area in every subsequent cut decreases causing a shallower depth of cut. This effect is graphically illustrated in the \textit{Figure 6.16}. The data in \textit{Figure 6.17} shows a very promising trend in significantly increasing the material removal rate during a micro-laser assisted SPDT operation.
6.3.3.1.4 HARDNESS EVALUATION VIA NANOINDENTATION

The following analysis evaluates the hardness of the machined regions via nanoindentations, where the experimental procedure is similar to that discussed in Chapter 5. Figure 6.19 shows the hardness data comparing the unmachined region and the machined regions: with and without laser heating. Only the deepest region, obtained using the smallest feed, is examined for each condition.

![4H-SiC-Hardness Comparison for Machined Regions](image)

**Figure 6.19: Nanoindentation hardness for 4H-SiC machined regions.**

*Figure 6.19* shows that the average hardness (5 indentations per region) of the machined region with no laser heating is the lowest. This is possibly due to the amorphous remnant (amorphous film on the crystalline material) on the machined surface that is generally characterized as a somewhat softer material when compared to the original crystalline material. Since the depths of indents are limited to below 50 nm, the chances of indenting mostly into an amorphous surface layer are high. The hardness of the region machined with laser heating is comparable to the hardness of the unmachined (pristine) surface. This could possibly be due to the annealing effect caused on the amorphous layer. The exact reasoning for this is still unknown and yet to be investigated. The important note to be made from the data in *Figure 6.19* is the
nanoindentation hardness values, 55 GPa to approximately 68 GPa (range includes all conditions tested), that are significantly higher than the previously assumed/reported micro-hardness values (between 25 – 27 GPa). The hardness values observed here are consistent with the hardness values obtained during diamond anvil cell tests that are currently in progress. This can be explained by the indentation size effect phenomenon where the hardness is seen to increase as the contact size decreases.\textsuperscript{220}

6.3.3.1.5 RAMAN SPECTROSCOPY

An ultraviolet 325 nm wavelength μ-Raman laser is used in this analysis. Figure 6.20 show the Raman spectra for both the cuts and the chips accumulated at the end of the cuts (similar to that seen in Figure 6.18). Figure 6.20(a) shows that that there is no change in the Raman spectrum between the machined (with and without laser heating) and the unmachined region. This does not mean that high pressure phases do not exists but could also be an issue of the equipment resolution where the current probing depth is too deep (probes way below the high pressure region). A slight shift in the original 977 cm\textsuperscript{-1} peak is observed in the machined regions. This is believed to be due to the residual stresses that are caused during the material removal process. In Figure 6.20(b), the machined chips accumulated from both machining conditions show amorphous characteristics, which is consistent with past TEM work.\textsuperscript{221} However, the spectra for the chips accumulated from the laser machined cuts show significantly more amorphous characteristics (broadband peaks) and three new peaks (495 cm\textsuperscript{-1}, 755 cm\textsuperscript{-1} and 915 cm\textsuperscript{-1}) possibly due to the thermal effect. From the scratch tests results, it is seen that laser heating contributes to the enhanced ductility (phase transformation) and therefore a
thicker amorphous layer beneath the machined surface is expected. Interestingly, the newly formed 495 cm\(^{-1}\) peak correlates to the Si-IV high pressure phase peak found in silicon. This could be due to the breaking of the Si-C bond at elevated temperatures or a high pressure/back transformed phase, but further investigation is required to confirm this. The formation of the new 915 cm\(^{-1}\) peak could also be related to the possible annealing effect that could be occurring due to the laser irradiation, similar to that seen in Si chips.

Figure 6.20: Micro-Raman spectra for 4H-SiC: (a) machined region with two conditions compared to the unmachined surface, and (b) chips accumulated from the machining process.
6.3.3.2 SINGLE CRYSTAL 6H-SIC

6.3.3.2.1 LOAD, LASER POWER AND DEPTH OF CUT

The first set of experiments are carried out to quantify the correlation between the applied load, laser heating (power), depth of cut, ductile response and cutting force. Table 6.5 summarizes the scratch test parameters and results done at a constant cutting speed of 1 µm/sec.

Table 6-5: Scratch test parameters at a constant cutting speed.

<table>
<thead>
<tr>
<th>$F_z$</th>
<th>Heating Condition</th>
<th>Average Depth</th>
<th>Cut Nature</th>
<th>$F_x$</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 mN</td>
<td>No Laser</td>
<td>15.0 nm</td>
<td>Fully Ductile</td>
<td>36 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>17.5 nm</td>
<td>Fully Ductile</td>
<td>34 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>18.0 nm</td>
<td>Fully Ductile</td>
<td>33 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>14.5 nm</td>
<td>Fully Ductile</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>12.0 nm</td>
<td>Fully Ductile</td>
<td>28 mN</td>
</tr>
<tr>
<td>120 mN</td>
<td>No Laser</td>
<td>17.5 nm</td>
<td>Fully Ductile</td>
<td>48 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>19.5 nm</td>
<td>Fully Ductile</td>
<td>46 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>20.5 nm</td>
<td>Fully Ductile</td>
<td>46 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>16.0 nm</td>
<td>Fully Ductile</td>
<td>46 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>16.0 nm</td>
<td>Fully Ductile</td>
<td>46 mN</td>
</tr>
<tr>
<td>140 mN</td>
<td>No Laser</td>
<td>18.5 nm</td>
<td>10% Brittle</td>
<td>58 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>25.0 nm</td>
<td>Fully Ductile</td>
<td>58 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>26.0 nm</td>
<td>Fully Ductile</td>
<td>58 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>19.0 nm</td>
<td>Fully Ductile</td>
<td>56 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>19.0 nm</td>
<td>Fully Ductile</td>
<td>57 mN</td>
</tr>
<tr>
<td>160 mN</td>
<td>No Laser</td>
<td>23.5 nm</td>
<td>50% Brittle</td>
<td>67 mN</td>
</tr>
<tr>
<td></td>
<td>10 W</td>
<td>27.0 nm</td>
<td>30% Brittle</td>
<td>70 mN</td>
</tr>
<tr>
<td></td>
<td>20 W</td>
<td>28.0 nm</td>
<td>20% Brittle</td>
<td>68 mN</td>
</tr>
<tr>
<td></td>
<td>30 W</td>
<td>21.5 nm</td>
<td>10% Brittle</td>
<td>68 mN</td>
</tr>
<tr>
<td></td>
<td>40 W</td>
<td>21.0 nm</td>
<td>5% Brittle</td>
<td>67 mN</td>
</tr>
</tbody>
</table>

Note. Highlighted rows represent the deepest cut

The trend in Table 6.5 is consistent with that seen in 4H-SiC where the cut done with an optimum laser power is the deepest cut (for an equal applied load). Beyond the optimum laser power (in this case 20 W), the depth of cuts decrease due to excess laser heating. It is also important to note that by increasing the laser power, the extent of brittle
fracture can be reduced. This is clearly seen in cuts performed at an applied load of 160 mN. The optical microscope images in Figure 6.21 clearly show the difference in brittle fracture for the cut performed with no laser (labeled NL) heating and the cut with the optimum laser power (labeled 20 W).

![Image: Figure 6.21: Comparing the brittleness along the center of the cut.](image)

This further confirms the enhanced ductile response in brittle materials due to the preferential heating or thermal softening effect. Although yielding greater depths of cuts, the cutting forces for the cuts performed with laser heating are less compared to the cuts performed with no laser heating. This is yet again an added benefit of the µ-LAM process that will assist in prolonging the life of the cutting tool. i.e., lower cutting forces should translate into reduced tool wear.

### 6.3.3.2.2 CUTTING SPEED AND OPTIMUM LASER POWER

The aim here is to identify the optimum power for higher cutting speeds and to correlate the measured depth of cuts to the thermal softening effect. Figure 6.22
compares the scratch test results for three different cutting speeds (1 µm/sec, 2 µm/sec and 5 µm/sec) with the corresponding optimum laser power.

![Optimum Power vs. Cutting Speed](image)

**Figure 6.22:** Correlation between cutting speed, laser power and depth of cut.

The results provide evidence that maximizing the thermal softening effect at higher cutting speeds is possible with the optimized laser power. In this particular case, two different applied loads are used and the trend for both loads is similar where the cuts with laser heating are deeper than the cuts with no laser heating, for all speeds. The optimized laser power is coincidentally the same for all three cutting speeds; 20 W. The power density delivered per unit length for the 1 µm/sec, 2 µm/sec and 5 µm/sec speeds cuts are fairly high and within the same order of magnitude; 28 GW/cm, 14 GW/cm and 6 GW/cm respectively.

### 6.3.3.2.3 SHAPING CUTS

Once the optimum laser power and ductile threshold applied loads are identified, shaping cuts are performed to factor in the cross-feed parameter to study the effect of
laser heating during machining. An applied load of 100 mN is used for the cuts done with no laser heating whereas a load of 140 mN is used with laser heating as these are the loads defined as the threshold before the material exhibits brittle behavior (refer to Table 6.5). Figure 6.23 summarizes the shaping cuts data for three different feeds; 1 µm, 2 µm and 5 µm).

![Graph showing shaping cuts data](image)

**Figure 6.23**: Shaping cuts for three feeds comparing with and without laser heating conditions.

Results from the shaping cuts demonstrate a significant increase in the depth of cut for the machined region with laser heating. Also, the depth of cut tends to decrease as the feed is increased as has been the case for previous experiments. These findings are consistent with that in 4H-SiC where a dramatic increase (~ 50%) in material removal rate is obtained as a result of thermal softening (preferential heating).

A surface roughness analysis on the machined surfaces is performed using a white light interferometer. Figure 6.24 compares the finish of the machined surfaces with and without laser heating to the unmachined surface.
Figure 6.24: Surface roughness comparison for machined regions: (a) average roughness (Ra), and (b) peak-to-valley (Rz).

Since all shaping cuts are performed on a polished wafer, the feedmark features from the cuts can result in an increase in surface roughness. This is clearly seen in the Ra value of the machined region done with no laser heating at the lowest feed (1 µm). The Ra for the regions performed with laser heating however, did not change much from the unmachined surface. For the peak-to-valley values, in general, the trend suggests that ductile mode machining (with and without laser heating) reduces the peaks and valleys. Once again, the Rz value for the cuts at the lowest feed (no laser heating) is higher due to the feedmarks on the surface. Since the area analyzed for this study is fairly small (~6000 µm²), a more accurate surface finish study will be carried out once an actual SPDT process is performed. Due to the inconsistency between the Ra and Rz trends (in Figure 6.24), it is determined that the machined area is too small to make a conclusion on surface roughness, therefore similar analyses are not carried out for the 4H and 3C-SiC. A detail surface roughness analysis will only be carried out after SPDT has been performed on SiC, providing a larger machined area to analyze.
6.3.3.2.4 HARDNESS EVALUATION VIA NANOINDETATION

The hardness of the machined regions (feed at 1μm) is studied via nanoindentations and is summarized in Figure 6.25. The indent depths are limited to 50 nm to ensure the hardness measured consists of mostly the high pressure phase zone below the machined surfaces. A total of five indentations are performed on each region.

![Figure 6.25: Nanoindentation hardness for 6H-SiC machined regions.](image)

The average hardness of both the material removed regions (with and without laser heating) are higher (by almost 30%) than the average hardness of the unmachined (pristine) 6H-Si surface (not similar to the observation in 4H-SiC). The increase in hardness in the material removed regions is still not clear and requires further investigation. There is no significant difference in hardness among the material removed regions (i.e., comparing the laser assisted cuts to the cut performed with no laser heating). Also, it is important to note that at a low indentation depth (in this case 50 nm), the surface roughness caused by the feed marks (1 μm feed) could also be affecting the hardness results.
6.3.3.2.5 RAMAN SPECTROSCOPY

A 325 nm (ultraviolet) wavelength $\mu$-Raman laser is used in this analysis to probe shallower depths. Figure 6.26 shows the Raman spectra for the cuts and the chips accumulated at the end of the cuts.

![Raman Spectra Image]

**Figure 6.26:** Micro-Raman spectra for 6H-SiC: (a) machined region with two conditions compared to the unmachined surface, and (b) chips accumulated from the machining process.

*Figure 6.26(a)* shows that there is no change in the Raman spectra between the machined (with and without laser heating) and the unmachined region. Similar to the observation for 4H-SiC, this does not imply the non-existence of high pressure phases but could be an issue of the equipment resolution where the current probing depth is far too deep and possibly probing way below the high pressure region. A slight shift in the original 890 cm$^{-1}$ peak is observed in the machined regions. This is believed to be due to the residual stresses that are caused during the material removal process. Another
interesting point to note is the existence of the 521 cm\(^{-1}\) in the spectrum for all regions. Although this is identified as the crystalline peak of silicon, it is possible to obtain impurities (such as Si) during the manufacturing process of the wafer as this peak is observed in the unmachined surface.

In Figure 6.26(b), the machined chips accumulated from both machining conditions show both crystalline and amorphous characteristics, which is consistent with previous work.\(^{221}\) However, the spectrum for the chips accumulated from the laser machined cuts show significantly more amorphous characteristics (broadband peaks) due to the significantly deeper machined region (~ 50% deeper than the region with no laser heating). The other difference seen in the spectrum for the chips accumulated from the laser machined cuts is that the 890 cm\(^{-1}\) peak is not evident anymore (more amorphous broadening seen here) and could be attributed to a possible annealing effect.

6.3.3.3 POLLYCRYSTALLINE CVD COATED 3C-SIC

6.3.3.3.1 LOAD, LASER POWER AND DEPTH OF CUT

For the polycrystalline 3C material, a combination of five applied loads and three laser powers are used to study the effect of laser heating on the material removal of the material. Each cut is approximately 100 µm long and done at a constant cutting speed of 1 µm/sec. Figure 6.27 summarizes the scratch test parameters and results for all experimented conditions.
All cuts reported here are in the ductile regime as studying the effect of laser heating in the brittle regime is not the scope of this research. The histogram in Figure 6.27 shows that the cuts done at 10 W laser power are the deepest for all applied loads. In this case, 10 W is the optimum laser power for a 1 μm/sec cutting speed. All cuts done with laser heating yielded larger depths than cuts with no laser heating, except for the cut done at 100 mN with 30 W of laser power. Due to the narrow area of the cut performed at the lowest load, there may be an excess of the thermal softening effect at high laser powers such as 30 W (i.e., excess laser radiation per unit area). This then causes the material to become too soft and it may flow back into the cut groove after the tool has passed by. The case of excess heating holds true for all cuts done with laser heating above 10 W power. The other important point to note from Figure 6.27 is that the cutting forces for the cuts done at all laser powers are almost the same as the forces for the cuts
without laser heating; although the cuts done with laser heating are in general (except for the 100 mN and 30 W condition) significantly deeper. This is certainly a great advantage in actual machining conditions where the material removal rate can be increased without causing additional stresses to the cutting tool. Figure 6.28 compares the cuts done at equal applied loads (250 mN) but different heating conditions (laser powers). Visually, no differences are noticed among the cuts, as all cuts are completely in the ductile regime (though the depths vary). The pits and voids seen along the cuts are not cause by the material removal process but where already in existence in the CVD material. Also, it is important to note that the cuts do not appear as smooth as the cuts done on 4H and 6H-SiC as this is a polycrystalline material with grain sizes ranging from 20 µm to 30 µm.

6.3.3.3.2 CUTTING SPEED AND OPTIMUM LASER POWER

This part of the study analyzes different laser powers at three different cutting speeds; 1 µm/sec, 2 µm/sec and 5 µm/sec. Although a total of two different applied loads
were experimented (120 mN and 200 mN), only the cuts performed at 200 mN are discussed in this section. *Figure 6.29* summarizes the results from these set of cuts.

![Figure 6.29: Correlation between cutting speed, laser power and depth of cut.](image)

The results demonstrate that maximizing the thermal softening effect at higher cutting speeds with optimized laser powers is important to obtain the largest depth of cut. For the cuts done at 1 µm/sec, the deepest cut is achieved at a 10 W laser power. However, as the cutting speed is increased to 2 and 5 µm/sec, the deepest cut is obtained at a 20 W laser power. Also notice that the effect of excess heating where the material apparently becomes too soft at 30 W becomes less apparent as the cutting speed is increased. This effect is confirmed by the depths obtained at 30 W that continue to increase as the cutting speed is increased. The power density delivered per unit length for the 1 µm/sec, 2 µm/sec and 5 µm/sec speeds cuts are fairly high and within the same order of magnitude; 28 GW/cm, 14 GW/cm and 6 GW/cm respectively.

### 6.3.3.3 SHAPING CUTS

To replicate an actual machining operation such as SPDT, shaping cuts are performed on the material with three different feeds; 1 µm, 2 µm and 5 µm. For this
study, equally applied loads (120 mN) are used for both conditions (with and without laser) to study the effect of laser heating with the incorporated feed parameter. Keeping the applied loads the same will enable direct depth comparisons for all machined regions. 

*Figure 6.30* shows the machined region depth of cut for all three feeds and both heating conditions (no laser and 10 W).

![3C-SiC: Machined Region Comparison](image)

*Figure 6.30: Shaping cuts for three feeds comparing with and without laser heating conditions.*

Results from the shaping cuts demonstrate a significant increase in the depths of cuts (~50%) for the laser assisted machined region for all three feeds. Also, the depth of cut tends to decrease as the feed is increased as has been the case for 4H and 6H-SiC. The average cutting forces for both conditions (no laser and 10 W) are almost equal (within 2%) despite the laser assisted machined regions being significantly deeper. For a CVD material, this is favored as the as-received surface roughness values are very high (Ra > 4 μm and Rz > 18 μm). For such rough and abrasive surfaces, tool wear is a major concern, and micro-laser assisted machining shows solid evidence in addressing/solving this ongoing problem. 

*Figure 6.31* compares the machined region for both conditions. The plastically deformed chips accumulated at the end of the cuts are a good indication of a ductile mode material removal process.
6.3.3.3.4 TOOL WEAR ANALYSIS

A tool wear analysis is carried out on an as-received CVD coated 3C-SiC sample (Super SiC-2 from Poco Graphite Inc.) for both conditions; with laser heating and without laser heating. Approximately a 2 mm² region is machined (shaping cuts) for both conditions with a cutting speed of 5 µm/sec and a 30 W laser power (only applicable for the laser assisted machined region). A 30 W laser power is chosen for this analysis (although 20 W is defined as the optimum for this cutting speed – refer to Figure 6.29) to soften the material beyond its optimum point. A slightly higher laser power is chosen here since the goal of the roughing pass (the first pass on an as-received surface) is not to achieve the deepest cut but to flatten the sharp peaks that often contribute to severe tool wear. An applied load of 300 mN and a feed of 10 µm is used for both conditions. Table 6.6 summarizes the depth of cut and surface roughness parameters after machining both regions.
Table 6-6: Depth of cut and surface finish of machined regions.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Ra</th>
<th>Rq</th>
<th>Rz</th>
<th>Rt</th>
<th>~ Depth of Cut</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Received</td>
<td>5.22 μm</td>
<td>6.24 μm</td>
<td>19.65 μm</td>
<td>39.9 μm</td>
<td>N/A</td>
</tr>
<tr>
<td>With Laser (30W)</td>
<td>1.32 μm</td>
<td>1.58 μm</td>
<td>2.63 μm</td>
<td>15.05 μm</td>
<td>13 – 14 μm</td>
</tr>
<tr>
<td>No Laser</td>
<td>1.69 μm</td>
<td>1.97 μm</td>
<td>1.52 μm</td>
<td>11.49 μm</td>
<td>9 - 10 μm</td>
</tr>
</tbody>
</table>

After the regions are machined, the surface roughness of each plateau is measured using a Mitutoyo surface profilometer. Four main surface roughness parameters are recorded for analysis purposes; Ra (Roughness Average), Rq (Root Mean Square (RMS) Roughness), Rz (Average Peak-to-Valley of the Profile) and Rt (Maximum Height of the Profile). In general, both machined regions significantly improved the surface finish, however, the laser assisted machined region yielded in a greater depth of cut (~ 40% deeper). Extremely deep cuts, as seen in Table 6.6, are normal during the roughing pass as the sharp peaks tend to break off at high cutting forces. Figure 6.32 shows a micrograph comparing the micro-laser assisted machined region to an unmachined region. The reflective region is a good indication of an improved surface as light from the microscope only reflects on smooth enough surfaces.

Figure 6.32: Comparing the laser-assisted machined surface and the as-received surface.
Since two different tools are used to machine both regions (to measure tool wear), both tools are imaged in a SEM after the machining operation. Figure 6.33 compares the cutting tool tip contact region for both tools.

![Figure 6.33: SEM micrograph of tools after machining: (a) tool used for the micro-laser assisted cuts, and (b) tool used for the cuts with no laser heating.]

Despite the aggressive machining parameters, no tool wear is observed on either tool. The features seen on the tools in Figure 6.33 are just a collection of debris from the machining process. The cutting edges for both tools are very well defined with no signs of wear or chipping. To obtain measurable tool wear, an actual SPDT operation with the μ-LAM setup will be carried out in the future to cover significantly more track length.

### 6.3.3.3.5 RAMAN SPECTROSCOPY

A 325 nm (ultraviolet) wavelength μ-Raman laser with a spot size of approximately 2 - 3 μm is used in this analysis. Figure 6.34 shows the Raman spectra for both, the cuts and the chips accumulated at the end of the cuts similar to that seen in Figure 6.31. Figure 6.34(a) shows that there is amorphous SiC in both machined regions. This is defined by the broadband peaks noticed in both machined region spectrums. The
amorphous layer is a good indication of a ductile material removal process. In general, the thickness of the amorphous layer increases as the depth of cut is increased. Although the peaks are almost identical for the unmachined and no laser machined conditions, the spectra for the laser machined surface shows the formation of a few new peaks; 871 cm\(^{-1}\), 960 cm\(^{-1}\), 963 cm\(^{-1}\), representing some unidentified phases. The formation of these new peaks is believed to be induced by the thermal effects such as annealing and recrystallization during the laser assisted machining process. A slight shift in the original 970 cm\(^{-1}\) peak is also observed in the laser machined region. This is believed to be due to the residual stresses (which shift the spectrum to lower wave numbers) that are caused during the material removal process.

Figure 6.34: Micro-Raman spectra for 3C-SiC: (a) machined region with two conditions compared to the unmachined surface, and (b) chips accumulated from the machining process.
In Figure 6.34(b), the machined chips accumulated from both machining conditions show amorphous characteristics, which is consistent with past TEM work on SiC. It is important to note that the spectra for the chips accumulated from the laser machined cuts show a distinct new peak at 547 cm\(^{-1}\) and a broadband peak at 900 cm\(^{-1}\). Once again, the formation of these new peaks is believed to be attributed to the thermal softening effect (possible annealing effect).

6.3.3.4 COMPARING THE 4H, 6H AND 3C POLYTYPES

This section compares the laser heating and thermal softening effect on the extent of material removal, i.e., depth of cut and enhanced ductility/reduced brittleness (DBT depth) of the 4H, 6H and 3C SiC polytypes. Table 6.7 shows the DBT depths for all three polytypes with its corresponding applied loads (F\(_z\)) and cutting forces (F\(_x\)).

<table>
<thead>
<tr>
<th>Polytype</th>
<th>Fz</th>
<th>Condition</th>
<th>DBT Depth</th>
<th>Fx</th>
</tr>
</thead>
<tbody>
<tr>
<td>4H-SiC</td>
<td>85 mN</td>
<td>No Laser</td>
<td>20 nm</td>
<td>31 mN</td>
</tr>
<tr>
<td></td>
<td>120 mN</td>
<td>With Laser (7 W)*</td>
<td>40 nm</td>
<td>46 mN</td>
</tr>
<tr>
<td>6H-SiC</td>
<td>120 mN</td>
<td>No Laser</td>
<td>17.5 nm</td>
<td>48 mN</td>
</tr>
<tr>
<td></td>
<td>140 mN</td>
<td>With Laser (9 W)*</td>
<td>26 nm</td>
<td>58 mN</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>450 mN</td>
<td>No Laser</td>
<td>42.5 nm</td>
<td>230 mN</td>
</tr>
<tr>
<td></td>
<td>600 mN</td>
<td>With Laser (4.5W)*</td>
<td>117.5 nm</td>
<td>330 mN</td>
</tr>
</tbody>
</table>

*Actual throughput power measured at the tool tip

It is seen that all cuts done with laser heating are able to withstand higher applied loads without exhibiting brittle fracture. In this case, this is the key to increasing the DBT depth of the material. The effect of laser heating is evident where greater loads can be applied, significantly increasing the DBT for all three polytypes. This is highlighted in
Figure 6.35 where the DBT depth is compared for all three polytypes and both conditions; with and without laser heating.

![DBT Depth Comparison for SiC](image)

Figure 6.35: DBT depth comparison for SiC.

The most significant difference in DBT depth (~ 176%) is observed in the 3C polycrystalline SiC. This is believed to be due to two factors; (1) the 3C absorbs the most laser radiation at these wavelengths (refer to Figure 6.2) and (2) the polycrystalline cubic structure is the least brittle material among the three polytypes (refer to Chapter 4). The polycrystalline material also has the highest fracture toughness value (about double the toughness of 4H and 6H) further explaining the enhanced ductility even without laser heating. Overall, the experimental results suggest that laser-assisted scratch tests are successful in enhancing the ductile response of all three SiC polytypes.

6.3.4 TEMPERATURE MEASUREMENT

A similar setup and technique as discussed in Chapter 5 is used to estimate the temperature in all three SiC polytypes while accounting for the atmospheric and high pressure phase optical properties. The temperature threshold of the lacquer is recorded
when a change is observed in the surface texture of the applied coating. The
experimentally measured temperature for various power ranges are summarized in *Table 6.8*. It is also important to note that the tool is moving at a very low speed of 1 μm/sec.
This means that these temperatures measured will change, where the temperature will reduce as the cutting speed is increased.

<table>
<thead>
<tr>
<th>Material</th>
<th>Laser Power</th>
<th>Measured Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>4H-SiC</td>
<td>12 – 14 W</td>
<td>≥ 704</td>
</tr>
<tr>
<td></td>
<td>18 – 20 W</td>
<td>≥ 1038</td>
</tr>
<tr>
<td>6H-SiC</td>
<td>13 – 15 W</td>
<td>≥ 704</td>
</tr>
<tr>
<td></td>
<td>20 – 22 W</td>
<td>≥ 1038</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>10 – 12 W</td>
<td>≥ 704</td>
</tr>
<tr>
<td></td>
<td>14 – 16 W</td>
<td>≥ 1038</td>
</tr>
</tbody>
</table>

The temperature estimates relative to the laser powers are approximately the same for both 4H and 6H-SiC. However, the 3C-SiC reaches its threshold temperature with less laser power due to the higher absorption of the atmospheric phase of the material at these wavelengths (refer to *Figure 6.2*). However, the experimentally determined temperatures also takes into account the high pressure phases generated as the temperature measured (with the temperature indicating lacquers) is a result of the laser beam that passes through both the atmospheric and high pressure phase. This trend, where lower powers are required to heat 3C-SiC is consistent with all the laser-assisted scratch tests reported in this chapter.

6.4 CONCLUSION

Micro-laser assisted scratch tests were successful in demonstrating the enhanced laser heating and thermal softening in single crystal and polycrystalline SiC resulting in
greater depths of cuts when compared to similar applied loads for cuts with no laser, and
greater ductile to brittle transition depths. Laser heating was successfully demonstrated as
evidenced by the significant increase in the ductile response of single crystal 4H-SiC,
single crystal 6H-SiC and polycrystalline 3C-SiC. Laser assisted (heating, thermal
softening and reduced brittleness) material removal resulted in greater depths of cuts at
less applied thrust forces, smaller cutting forces and a larger critical depth of cut. Force
analysis (thrust and cutting), optical microscopy and white light interferometry served as
useful techniques to detect the enhanced ductile response and reduced brittle fracture as a
result of preferential material heating (of the high pressure phase transformed material).
Micro-Raman spectroscopy served as an important tool to characterize the amorphous
remnant beneath the machined surfaces (for 3C-SiC) and accumulated plastically
deformed chips. Results obtained from this study are promising to further implement
micro-laser assisted machining (µ-LAM) in operations such as single point diamond
turning. Lower cutting forces obtained from the µ-LAM process are favorable to
minimize tool wear while machining abrasive ceramics/semiconductors such as silicon
carbide. The results from this study also will benefit the manufacture of brittle materials
as laser heating is seen to decrease the brittle response of the atmospheric phase and
enhance the ductility of the high pressure phases in ceramics and semiconductors which
results in higher productivity rates (i.e. higher material removal rate).

6.5 FUTURE WORK

Single point diamond turning (SPDT) experiments will be carried out using the µ-
LAM setup. For the SPDT experiments, several machining parameters such as cutting
speed (changes as a function of workpiece radius), depth of cut and feed, and laser power (assuming the 100 W laser is used) will be experimented and optimized. The ultimate goal for the SPDT experiments is to improve the surface finish of an as-received SiC workpiece (which is demonstrated in the 6H polytype) with minimal or no tool wear (which was not verified herein due to the limited length or track length of cut). The cuts will be compared to conventional SPDT cuts (with no laser heating) with similar machining parameters for various depths of cuts and feeds in the ductile regime. The micro-laser assisted-SPDT parameters such as depth of cut, feed, cutting speed and laser power will then be optimized to achieve the maximum material removal rate while improving the surface finish.

Micro-Raman spectroscopy will be performed on the cuts carried out to identify the DBT and also on the SPDT machined surfaces. For the scratch performed to identify the DBT, three different regions will be analyzed; the ductile region, the transition region and the brittle region. The spectrums of all three regions will be compared and possible new peaks (expected to be generated in the phase transformed region/chips) will be identified as an evidence of ductile mode machining. Besides identifying new peaks or a shift in peaks, broadband peaks or amorphous broadening of the crystalline peaks is also expected in the machined surface (ductile mode material removal).

The second characterization technique that will be utilized in order to confirm the occurrence of ductile mode machining (due to the HPPT) is transmission electron microscopy (TEM). TEM analysis will be carried out on the machined chips (debris) which are plastically deformed during the ductile material removal process. These chips have been collected after each experiment (with different machining parameters) and will
continue to be collected for characterization purposes. The chips are usually visible at the end of the cut/scratch (a bunch of accumulated chips) or stuck to the diamond tool tip. The existence of the amorphous remnant in the chips is a substantial evidence of ductile mode machining. TEM analysis done by past researchers on chips from SPDT operations show both the amorphous region and crystalline structure indicating phase transformation (crystalline material transformed into an amorphous structure) due to significant plastic deformation during the material removal process. Finally, scanning acoustic microscopy (SAcM) will be performed on the laser machined samples to measure and image subsurface damage such as thermal cracks (refer to Chapter 3).

Finally laser heated diamond anvil cell (DAC) and rotary diamond anvil cell (R-DACR; to introduce the shear component) to simulate actual laser heating and cutting conditions will be performed at Argonne National Laboratories. Laser heated DAC tests have previously been conducted on 4H and 6H SiC (but not on 3C). The high-pressure phases will be identified and retained for optical characterization purposes. The goal for optical characterization tests are to get the optical properties of the high pressure phases for all three polytypes, to be able to choose a suitable laser wavelength during the machining process.
CHAPTER 7

HIGH-PRESSURE PHASE TRANSFORMATION: PRESSURE AND TEMPERATURE EFFECTS

7.1 INTRODUCTION

Machining hard and brittle semiconductors like silicon (Si) have been a challenge in the ceramic manufacturing industry for decades. A combination of its high hardness (hardness of Si = 12 GPa) and low fracture toughness (Si = 1.29 MPa m$^{0.5}$), makes the material removal process both time consuming and relatively high in cost. Besides these challenges, the surface quality of the end products becomes extremely important, especially in the microelectronics and opto-electronics and optics industry. In general, to produce near-perfect surface quality of Si wafers, a series of manufacturing processes is required.Usually, the wafer undergoes several processes such as grinding and/or lapping and polishing. Polishing is usually the last step to obtain mirror-like surfaces, but also the most time consuming step due to the low material removal rate (less than 10 nm each pass). Grinding/lapping often introduces significant amount of surface and subsurface damage, thus making the final polishing process extremely long (hours, days, weeks to months for large optical components).

In the mid-1990’s, the insight into the origins of ductile regime during single point diamond turning (SPDT) of ceramics and semiconductors was provided. Although Si, is naturally very brittle, machining under specific conditions generating significant
compressive and shear stresses can cause a ductile mode behavior, in which the material is removed by plastic deformation instead of brittle fracture. The micro-scale phenomenon that contributes to the ductile mode behavior in nominally brittle materials is referred to as the High Pressure Phase Transformation (HPPT). It is found that a transition from covalent to metallic bonding that is caused from high pressures and shear at the contact interface of the machining tool and the workpiece produces a ductile surface on machined brittle materials.

To further enhance the ductile response of ceramics and semiconductors (in this case Si), traditional single point scratch tests are coupled with a micro-laser assisted machining technique. This hybrid method has demonstrated the capability to enhance the ductile response and increase the ductile-to-brittle (DBT) transition depth in silicon (refer to Chapter 5). A schematic of the basic underlining concept of the μ-LAM technique is shown in Figures 2.10 and 6.1. The μ-LAM system is configured in such a way that the IR laser beam passes through the optically transparent diamond tool tip and impinges on the workpiece material at the tool-workpiece interface. The unique characteristic about the μ-LAM system is the ability to preferentially heat only the contact region at a micro-scale. The effect of laser heating on the phase transformation and annealing of the high pressure phases has been briefly discussed in Chapter 5. In this chapter, a specific study is designed to: (1) characterize the thermal softening effect on the high pressure phases (HPPs) via scratch testing using a cutting tool and micro-Raman spectroscopy and, (2) isolate and study the temperature and pressure effect on the ductile response during the micro-laser assisted material indentation process. The evidence of the
HPPTs during the ductile mode material deformation/removal process using the $\mu$-LAM technique is also discussed.

7.2 EXPERIMENTAL METHOD

7.2.1 MICRO-RAMAN CHARACTERIZATION OF CUTS

Scratch tests are chosen to be the principal material deformation/removal method as it is a better candidate for evaluating machining than indenting, because the scratching parameters such as the depth of cut, width of cut, cutting speed and cutting forces are more applicable to the machining process. Three different scratch conditions are applied: no laser heating, low power laser heating (10W) and optimized laser heating (45W). The optimized laser power is determined as the point that yields the greatest depth of cut without the material melting. Excess laser power could cause the material to become too soft (almost liquidus) where it may flow back in to the cut groove at the trailing edge of the tool (discussed in detail in Chapters 5 and 6). An IR diode laser ($\lambda=1070$ nm and $P_{\text{max}}=100$ W) with a Gaussian profile beam of an approximate diameter of 10 $\mu$m is used in this investigation. All cuts are performed on single crystal Si on the \{100\} plane along the $\langle110\rangle$ direction. A 1 mm nose radius single crystal diamond tool with a $-45^\circ$ rake and $5^\circ$ clearance angle is used. A highly negative rake angled tool is used to generate higher compressive stresses that contribute to the phase transformation. Although the higher negative rake angle ($-20$ to $-60^\circ$) tool used in ductile mode machining generates the intended compressive stresses, it also generates a thicker subsurface damage layer.227 In
this case, the subsurface layer referred to is the amorphous layer below the machined region.

As the equipment used is load controlled (UMT by CETR Inc.), a desired load/thrust force is used to achieve the depth of cut. In this study, an applied load of 150 mN is used for all conditions as this load is found to be close to the upper limit before the material exhibits brittle behavior (brittle behavior previously observed between 170 – 180 mN with no laser heating). A typical scratch test setup used in this experiment is shown in Figure 5.4 (Chapter 5). Since the goal of the study is to investigate the role of laser heating in ductile mode machining, the applied load is maintained below the DBT load, which was determined in preliminary experiments. The cutting speed is kept constant at 1 μm/sec for all scratch conditions. A low cutting speed is used in this study to maximize the thermal softening effect. Table 7.1 shows the scratch test parameters and conditions where the depth of cut is profiled using a white light interferometric surface profiling technique.

Table 7-1: Scratch test parameters to study HPPTs.

<table>
<thead>
<tr>
<th>Heating Condition</th>
<th>Applied Load</th>
<th>Cutting Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Laser</td>
<td>150 mN</td>
<td>1 μm/sec</td>
</tr>
<tr>
<td>10 W</td>
<td>150 mN</td>
<td>1 μm/sec</td>
</tr>
<tr>
<td>45 W</td>
<td>150 mN</td>
<td>1 μm/sec</td>
</tr>
</tbody>
</table>

A Renishaw inVia Raman (μ-Raman) microscope is used to characterize ductile mode machining by analyzing different high pressure phases formed under different scratch conditions. Excellent spatial (1μm) and depth resolutions (10s of nanometers, depending on the wavelength of the laser beam) offered by this technique along with the ease of analysis and fast turn-around times are the main reasons for choosing this technique. In this study, a visible (λ = 442nm) laser is employed to analyze the
structure of the material in the scratched regions on the Si workpiece. A 5 second exposure time with 10 accumulations per sample is used for all data points to increase the Raman intensities and minimize the noise for ease of detecting the HPPs (isolating the noise from actual Raman bands). For all Raman spectra reported here, the relative intensities and not the absolute intensities (arbitrary units) are shown, as the absolute values of intensities of spectra vary from scan to scan. This is due to the non-uniformity of the surface quality, which could emit an inconsistent number of photons of a specific wavelength, as well as the unevenness of the sampled surface relative to the incident angle of the laser beam. However, the relative intensity among the peaks within a single spectrum remains true at all times.

Previously, ductile regime machining of semiconductors, demonstrated using a single point diamond tool, is attributed to the high pressure phase transformation (HPPT) occurring in the material caused by the high compressive and shear stresses induced by the tool tip\textsuperscript{228,176}. Single crystal Si is known to undergo a nonmetal to metallic transition at hydrostatic pressures in the range of 10-13 GPa.\textsuperscript{229} The drop in electrical resistance under applied load suggest pressure induced metallization of Si-I.\textsuperscript{230,41} This HPPT occurs when the $\beta$-Sn (Si-II) structure forms from the diamond structure (Si-I). During the tool stress/pressure release, several metastable phases such as Si-III, Si-XII, Si-IV and Si-XIII have been reported to form from Si-II.\textsuperscript{204} There are at least 13 different polymorphs of Si reported in previous studies. There are several factors that affect a particular transition mechanism is Si such as tool/indenter geometry, maximum applied load, rates of loading and unloading, and temperature (addition of heat).
7.2.2  INDENTATION WITH A CUTTING TOOL

The two primary goals of this part of the study is to (1) separate the temperature and pressure effects on the ductile response of the material, and (2) identify the phase the laser irradiation is softening (i.e. atmospheric/Si-I, high pressure phases (HPPs) or both). The impact of the thermal softening is important to know in order to optimize the μ-LAM parameters such as laser power, laser wavelength, depth of cut and cutting speed. In order to be able to separate both the temperature and pressure effects, indentations are chosen as the testing method in this study. However, unlike conventional indentation tests, an actual cutting tool (1mm nose radius, -45 degree rake and 5 degree clearance angle) is used to simulate the actual cutting operation as closely as possible. The indentations are carried out on a universal micro-tribometer (UMT) which is equipped with a dual-axis load cell, allowing the user to perform a load controlled indent (the indent depth is measured later to study the temperature/pressure effect). Figure 5.4 (Chapter 5) shows the indentation test setup where the μ-LAM system is coupled with the UMT.

Since the main goal in this study is to be able to separate the temperature and pressure effects, it is important to be able to plan a controlled test procedure. Since indentations are well known for being precisely controlled at all stages (loading, hold and unloading), this advantage in exploited where laser heating is only introduced when required, i.e., laser heating can be introduced to heat the surface before the tool makes contact or only when the high pressure phases are formed. For the stages that require heating, 10 Watts of a 1070 nm wavelength CW fiber laser is used.

The indentation stages (shown in Figure 7.1) for this study consist of (1) before contact (the tool is positioned approximately 100 μm above the sample surface), (2)
loading (at the rate of 10 mN/sec), (3) hold (where the intended load is held constant for 30 seconds) and (4) unloading (at the rate of 10 mN/sec). A relatively low indentation load (300 mN) is used to reduce the amount of strain contrast and improve the mechanical integrity of the sample. An applied load of 300 mN is still considered a relatively low load since a blunt 1 mm nose radius cutting tool is used (compared to a regular Berkovich indenter that has a tip radius between 100-200 nm). Examination of low load nano-indents also allows the direct examination of the elastic-plastic interface and possible dislocation slip before the onset of cracking.

![Figure 7.1: Various stages for the indentation tests used in this study.](image)

A total of nine indentation conditions as shown in Table 7.2, are performed to precisely control and understand the heating process by introducing the laser irradiation at specific stages of the indentation process: before contact, loading, hold and unloading.
Table 7-2: Heating stages for nine indentation conditions.

<table>
<thead>
<tr>
<th>Condition #</th>
<th>*Before Contact</th>
<th>Loading</th>
<th>Hold</th>
<th>Unloading</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>5</td>
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<td>6</td>
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<td>7</td>
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</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note. *The tool is positioned approximately 100 μm from the surface of the workpiece. If laser heating is required at this stage, a heating time of 30 seconds is implemented. The green cells represent no laser heating conditions whereas the red cells represent the laser heated conditions.

Each condition is established based on a particular criterion that is expected to provide new information when the laser heating is applied at that stage. The reasoning behind executing each indentation condition is summarized in Table 7.3.

Table 7-3: Selection criterion for indentation conditions.

<table>
<thead>
<tr>
<th>##</th>
<th>Reasoning for Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Benchmark for no laser heating condition. No heating at either stage</td>
</tr>
<tr>
<td>2</td>
<td>Pre-heating the indent surface only before loading. The results (measured depth) here will indicate if the laser is heating the Si-I phase. Comparing the depth with #3 will confirm the phase the laser is heating: Si-I phase, HPPs or both.</td>
</tr>
<tr>
<td>3</td>
<td>Laser heating only the HPP zones. Comparing the depth with #2 will indicate if the laser is heating the Si-I phase or the HPPs, or both.</td>
</tr>
<tr>
<td>4</td>
<td>Preheating the Si-I and then continuing to heat the HPP zones. Comparing this with #2 and #3 may indicate if the beam is heating just the Si-I phase or HPPs, or both.</td>
</tr>
<tr>
<td>5</td>
<td>Laser only heats the loading stage where the HPP transformed area is expected to increase as the load increases.</td>
</tr>
<tr>
<td>6</td>
<td>Laser on heats the HP retained zone. In this stage (hold), the HPP zone has already formed from the loading phase. If the depth is different from what is obtained in #5, it may provide some new information.</td>
</tr>
<tr>
<td>7</td>
<td>Material is only heated during unloading (in indentations, crack formation is common only in the unloading phase) zone where the back transformation is believed to occur.</td>
</tr>
<tr>
<td>8</td>
<td>HPPs are always heated but also during the back transformation (unloading). Based on the current understanding, it is expected that the results here to be similar as #3, however with this combination, the heating effectiveness in the unloading phase can be analyzed further. Also, this condition replicated the current μ-LAM heating sequence during scratch tests.</td>
</tr>
<tr>
<td>9</td>
<td>All zones are heated (including the Si-phase). This would serve as a benchmark for the laser heating condition.</td>
</tr>
</tbody>
</table>
The executed indentation matrix is schematically shown in Figure 7.2 where a 100 μm gap is left between each indent. A total of five indents are performed for each condition to obtain repeatable data.

In order to determine the phases the laser is preferentially heating, the final indent depths are measured. Since the applied load is kept constant at 300 mN for all conditions, a difference in the indent depth would give an insight to the phases being formed and heated under respective conditions. When the laser heats the desired phase and softens it, the indent depth is expected to get larger for the same applied load. This is due to one of the advantages of the preferential heating in the μ-LAM system where the material absorbs the laser energy causing localized heating and thus reducing the hardness of the material (at the tool contact zone). All depths are only measured after the entire indentation process is completed (not in-situ measurement) to obtain the final indent depth after the elastic recovery upon unloading. The indent depths are measured using a WYKO white light interferometric microscope that has an angstrom vertical resolution.

![Indentation matrix for nine conditions.](image)
7.3 RESULTS AND DISCUSSION

7.3.1 MICRO-RAMAN CHARACTERIZATION OF CUTS

The scratch test results and parameters used in this analysis are discussed in detail in Chapter 5. Table 7.4 summarizes the conditions and results from the scratch tests. All cuts are carried out at a constant cutting speed of 1 μm/sec.

Table 7-4: Scratch test parameters at a constant cutting speed.

<table>
<thead>
<tr>
<th>Applied Load</th>
<th>Heating Condition</th>
<th>Average Depth</th>
<th>Cutting Force</th>
<th>Power Density/ Unit Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>150 mN</td>
<td>No Laser</td>
<td>104.0 nm</td>
<td>58 mN</td>
<td>N/A</td>
</tr>
<tr>
<td>10 W</td>
<td></td>
<td>123.0 nm</td>
<td>58 mN</td>
<td>7 GW/cm</td>
</tr>
<tr>
<td>45 W</td>
<td></td>
<td>131.5 nm</td>
<td>57 mN</td>
<td>33 GW/cm</td>
</tr>
</tbody>
</table>

Note. All forces reported are within a 7% error.

From the scratch test parameters in Table 7.5, it is clear that the scratches performed in the laser assisted mode result in a greater average depth of cut than without laser. It may also be noted that that the depth of cut increased as the laser power is increased even at the same applied load (150 mN) i.e., the cut performed with the maximum optimized laser power (45W) yields the greatest depth due to the maximum thermal softening effect whereas the cut performed with low laser power (10 W) is deeper than the cut with no laser heating, however, not as deep as the cut done with the maximum optimized power (45 W). Furthermore, the cutting forces remain almost constant for all three conditions although the cuts with laser heating resulted in greater depths of cuts.

To correlate the HPPT and laser heating effect during the material removal process, μ-Raman spectroscopy is performed on the material removed regions. The spectra presented in Figure 7.3 are obtained using a 442 nm laser.
Figure 7.3: Micro-Raman spectra comparing unmachined and machined regions: (a) pristine (unscratched) single crystal Si wafer and scratches performed, (b) without laser, (c) using 10 W laser power, and (d) using optimized laser power (45 W).

Figure 7.3(a) shows the 521 cm\(^{-1}\) Raman peak of the pristine (unscratched) single crystal Si wafer. The spectra shown in Figure 7.3(b, c and d) correspond to those obtained from scratches performed (b) without laser (c) at a laser power of 10 W and (d) 45 W respectively. The spectra in Figures 7.3 (b) and (c) shows additional peaks that are signature for crystalline bulk material indicating that a phase transformation has occurred at the surface due to the material removal process. It is seen that three new phases, Si-III, Si-IV and Si-XII as well as amorphous Si are identified in addition to the Si-I phase. From previous research work, it is known that metallic Si-II is unstable below approximately 4 GPa upon unloading and therefore transforming to other phases such as Si-I, Si-III, Si-IV and Si-XII, depending on the pressure release conditions. The high
pressure phases in Figures 7.3(b) and (c) mostly (based upon the occurrence frequency in a single spectrum) contained Si-III phases which were consistent with previous research work (this phase forms at the lowest pressure upon unloading).[^234]

The µ-Raman spectrum for the scratch performed with optimized laser power (Figure 7.3(d)) shows the 521cm\(^{-1}\) Si-I peak, similar to that seen in the unscratched surface. This is attributed to the recrystallization of the material, during which the high pressure phases have apparently annealed due to the preferential heating of the µ-LAM process. Annealing of the high pressure phases leads to the recovery of the original diamond structure (Si-I) at temperatures above 1000 °C. The experimentally measured temperature of the machined region is determined using temperature indicating lacquers, which shows that the temperature exceeds 1000 °C at 45 W of laser power (refer to Chapter 5). This clearly confirms the recrystallization of the surface layers in the machined region at these temperatures, as has been demonstrated in previous work.[^235]

From Table 7.4, although the power densities per unit length are high for both laser machined conditions (10 W and 45 W), the power density per unit length for the optimum power (45 W) is 4.5 times greater than that obtained using minimum power (10 W). This is also an important factor that could cause the HPPs to anneal. The annealing of the high pressure phases has been previously observed during nanoindentation, after subjecting it to annealing at different temperatures and durations.[^45] It is believed that the HPPs are formed for all cuts and heating conditions as this is the governing phenomena for a ductile mode material removal process.[^236]

The phase transformation events and sequence have been well documented by previous research work. At atmospheric pressure, single crystal Si exists in a cubic
diamond structure (Si-I), which transforms into a metallic β-tin structure (Si-II) under hydrostatic pressures of ~12 GPa (approximately equal to the measure hardness).\textsuperscript{237} Upon unloading or decompression, Si-II transforms into several metastable phases depending on the load release conditions. During slow unloading, as is done in this experiment, the first metastable phase to form is a rhombohedral structure (Si-XII) at approximately 10 – 12 GPa.\textsuperscript{238} Of course, other phases (Si-VIII and Si-IX) have been identified in previous work upon rapid release, but not observed in this study.\textsuperscript{239} On further pressure release, a reversible transition from Si-XII to the body-centered-cubic structure (Si-III) occurs. This creates a mixture of Si-XII and Si-III at ambient pressure. Initial research conducted on heated indentation tests of Si identifies the SI-IV phase as the intermediate phase before the recovery of Si-I.\textsuperscript{240} However, the presence of Si-IV in diced and lapped Si wafers without heating has also been previously reported.\textsuperscript{241} The latter findings could be attributed to the shear component that is not present during an indentation process or also due to the large depth of cuts (> 100 nm). Si-IV is also observed in the Raman spectra (\textit{Figure 7.3}) from both the material removed regions (with and without laser heating), however, the pressures at which this phase forms have not been identified. The structural data and corresponding Raman bands for the Si phases relevant to this study is summarized in \textit{Table 7.5}.

\textbf{Table 7-5: Structural data for crystalline phases of Si with corresponding Raman bands.}

<table>
<thead>
<tr>
<th>Phase Designation</th>
<th>System</th>
<th>Pressure Region</th>
<th>Raman Band</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si – I</td>
<td>Cubic</td>
<td>0 – 12.5 GPa</td>
<td>521</td>
</tr>
<tr>
<td>Si – II</td>
<td>Tetragonal</td>
<td>8.8 – 16 GPa</td>
<td>N/A</td>
</tr>
<tr>
<td>Si – III</td>
<td>Cubic</td>
<td>2.1 – 0 GPa</td>
<td>386, 400, 438</td>
</tr>
<tr>
<td>Si – IV</td>
<td>Hexagonal</td>
<td>Unknown</td>
<td>494</td>
</tr>
<tr>
<td>Si - XII</td>
<td>Trigonal</td>
<td>12 – 2.0 GPa</td>
<td>353</td>
</tr>
</tbody>
</table>

\textit{Note: Si II, IV and XII occur upon unloading (not during loading).}
7.3.2 INDENTATION WITH A CUTTING TOOL

All nine indentation conditions (45 indents in total), are performed on the same Si wafer with a precisely controlled process (keeping all stage and carriage movement velocities constant). An optical microscope image of the actual indentation matrix is shown in Figure 7.4 (indent conditions can be correlated to Figure 7.2).

![Indentation matrix showing all nine conditions.](image)

All indents are measured using a white light interferometer that provides angstrom resolution for depth measurements. A typical cross section depicting the depth of indentation is shown in Figure 7.5. The measured depth in this particular profile is 72 nm.
The defined edges along the indent are a clear indication that the indent performed is within the ductile regime of the material. Indents with cracks usually do not exhibit a perfect replication of the tool tip (as seen in Figure 7.7), indicating that the indent is carried out in the brittle regime. The depth at the deepest region along the indent, in this case at the center of the indent, is recorded. A three-dimensional surface profile showing the extruded material and the deepest region at the center of the indent is illustrated in Figure 7.6.

The data in Figure 7.8 represents the measured depths of indents for all conditions. The average depths are reported in the histogram along with the corresponding range (deviation) of depths for each condition. A total of two indents are
eliminated in the reported average as they are determined to have chips along the edges and therefore causing a significant deviation in the data. An example of a discarded indent measurement is shown in Figure 7.7.

![3-Dimensional Interactive Display](image)

**Figure 7.7**: An example indent that is discarded due to chipping.

It is seen from the cross-sectional profile in Figure 7.7 that the chipped region at the edge of the indent causes an undefined profile that provides a deceivingly large depth that is the depth of the chipped area and not the indent.

![Average Indent Depths](image)

**Figure 7.8**: Average depths of indents for all nine conditions.
The data in Figure 7.8 suggest that the shallowest indents are obtained from conditions 1 and 2 where no heating at any stage and only preheating of the Si-I phase are implemented respectively. Conditions 3 through 7 all yielded indent depths within the standard deviation/total range of each other. This group (conditions 3 through 7) is analyzed together due to a common factor where selective heating is introduced at either one of the high pressure formation/existent stages. Conditions 3 and 4 heat the same stages of HPP formation, except that condition 4 also preheats the Si-I surface. However, no significant difference in the indent depth is noticed between conditions 3 and 4. Within this group, between conditions 3 through 7, the deepest indents are obtained in condition 5 where only the loading stage is heated. This could be attributed to the fact that the high-pressure phase zone is continuously growing in size and thickness during constant loading, therefore the thermal softening effect is maximized during this stage. On the other hand, condition 7 is the least deep among conditions 3 through 7 as only the unloading stage is heated. During the unloading stage, the maximum thickness of the phase transformed region is believed to have occurred (as maximum load is already achieved at the end of the “loading” stage) and therefore the thermal softening effect in this condition is minimal, but still existent. The deepest indents are obtained for condition 8 as all the stages where the high pressure phases are present is being heated. Finally, in condition 9 the indents are not as deep as in condition 8 as the initial surface is also preheated and excess heating is believed to be occurring. When the material is excessively heated, it is believed that the material becomes too soft and flows back into the indent feature once the tool is released. This phenomenon is explained in detail in
Chapters 5 and 6 where similar effects have been observed during laser assisted scratching of Si and SiC.

To study only the temperature effect on the high pressure phases and to determine the phase heated by the laser irradiation, a total of three indentation conditions are investigated in detail (conditions 1, 2 and 8). These conditions isolate the temperature effect from the pressure effect during the HPPT process. A highly magnified microscope image showing indents with these three conditions is illustrated in Figure 7.9. As an example, a single indent has also been isolated to show the extruded material as a result of the indentation process. Condition 1 is carried out with no laser heating at either stage. This condition is a good benchmark for a cut performed at room temperature with no laser heating. Conditions 2 and 3 are carried out to determine the actual phase being heated by the laser during a μ-LAM cutting process (i.e. the atmospheric/Si-I phase or the high pressure phases or both). In, condition 2, the laser is only activated before the tool contacts the surface, heating only the atmospheric/Si-I phase. This condition would replicate a pre-heating process in a scratch/SPDT process. Finally, in condition 3, laser heating is applied during the loading, hold and unloading, heating all the stages that the high pressure phases are generated/exists. This replicates the actual μ-LAM cutting process where the laser is activated the moment the cutting tool touches the workpiece surface. Figure 7.10 compares the average indent depths for these three conditions (1, 2 and 8).
The results show strong evidence that the laser irradiation preferentially heats and greatly affects the high pressure phases. The thermal softening effect is clearly demonstrated in Figure 7.10 where the indents performed while heating the HPPs (condition 8) are approximately 81% deeper than the indents performed with no heating.
(condition 1) or only preheating (condition 2) the Si surface (only heating the Si-I phase). This series of indents successfully isolates the temperature and pressure effects during the material removal process to determine the most benefiting laser heated phase: Si-I or HPPs. The results obtained are consistent with the understanding established during micro-laser assisted scratch tests on single crystal Si discussed in Chapter 5.

A μ-Raman analysis is also carried out on the indents to verify the HPPs formed during the indentation process. Figure 7.11 shows the Raman spectra comparing the pristine surface with the indents performed with no laser heating (condition 1), only preheating the Si surface (condition 2) and with laser heating at all high pressure phase zones (condition 8). Only these three conditions are characterized in this study as no additional information is expected from the Raman spectra obtained in the other conditions.
Figure 7.11: Micro-Raman spectra comparing the pristine surface with the three indent conditions.

The findings of the μ-Raman analysis are consistent with the scratch tests (comparing no laser, 10 W and 45W). Identical spectrums are observed for conditions 1 and 2 which are expected as the high pressure phases are generated in both cases, but not heated. The spectrum in condition 8 shows that the HPPs have been annealed and are transformed back to the original diamond cubic structure (Si-I). This observation is similar to the scratch tests done at the optimized laser power of 45 W. Although only a 10 W laser power is used in the indentation tests, the tool/laser source is stationary and the high pressure phases are heated for a total of 90 seconds (versus a moving tool during a
scratch test). Amorphous Si is also observed in all three indent conditions, but less in condition 8 as most of the a-Si has been annealed. The spectrum obtained from condition 8 shows significantly less amorphous broadening, confirmed by comparing the relative intensities with conditions 1 and 2.

The final analysis carried out is to correlate the calculated hardness (normalized) to each of these indent conditions (1, 2 and 8). The hardness is calculated based on the applied load (300 mN) and the residual indentation area that is measured from the white light interferometric profiles/images (hardness calculations given in appendices). Only the normalized hardness values (relative hardness) are reported to account for errors such as tool geometry, equipment resolution and stability, laser output stability and surface reflectivity during white light interferometry that are assumed constant for all readings. Table 7.6 summarizes the findings for the indentation tests for the three most important conditions (1, 2 and 8).

Table 7-6: Summary of indentation results for conditions 1, 2 and 8.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Average Depth</th>
<th>Phases Detected</th>
<th>Normalized Hardness Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>49.2 nm</td>
<td>a-Si, Si-III, Si-IV, Si-XII</td>
<td>1.00</td>
</tr>
<tr>
<td>2</td>
<td>49.0 nm</td>
<td>a-Si, Si-III, Si-IV, Si-XII</td>
<td>0.96</td>
</tr>
<tr>
<td>8</td>
<td>88.5 nm</td>
<td>a-Si, Si-I</td>
<td>0.53</td>
</tr>
</tbody>
</table>

The normalized hardness values for conditions 1 and 2 are approximately the same further explaining the similarities in the average depths. However, the hardness drops by almost 50% when the laser preferentially heats the HPPs, as seen by the depth increased (> 80%) for condition 8. The apparent reduction in hardness is a significant benefit when machining hard and brittle semiconductors like Si and SiC. The drop in material hardness subsequently contributes to the increase in material removal rate and reduction in tool wear. Hardness values of Si have been previous reported to drop as the
temperature is increased via bulk material heating. These are the first successful experimental results using a micro-laser assisted machining setup, with an actual cutting tool used as the indenter.

With the confirmation that only the high pressure phases were being heated, annealed, and eventually recrystallized during the μ-LAM process, a possible phase transformation sequence diagram for the material removal process is proposed in Figure 7.12. In this particular case, the same sequence can be used to explain both the scratches and indentations as the Raman data suggest that the same HPPs exist for both the scratch tests and the indents.

![Figure 7.12: Illustration of the transformation sequence during the μ-LAM of Si.](image)

7.4 CONCLUSION

Micro-laser assisted scratch tests were successful in demonstrating the enhanced laser heating and thermal softening of the high pressure metallic (Si-II) phase generated while cutting single crystal Si in the {100} plane along the <110> direction, resulting in greater depths of cuts for similar applied loads. Laser assisted (heating, thermal softening and reduced brittleness) material deformation and removal resulted in a greater depths of
cuts at equal applied thrust forces and lower cutting forces (when comparing a depth-to-cutting force ratio). Lower cutting forces (with respect to the relative depths) obtained from the μ-LAM process (cuts with laser heating) are favorable to minimize tool wear while machining abrasive ceramics/semiconductors.

Micro-Raman spectroscopy was successful in showing evidence of HPPTs implying that all material removal was in the ductile regime. The optimized laser power condition (45W) resulted in the deepest cut (for similar applied loads) and leaving behind a “damage-free” or “cured” Si-I diamond structure, similar to that of the original surface. From a manufacturing point of view, this is vital as the material is believed to have returned to its original phase (Si-I) despite undergoing the ductile mode material removal/HPPT process. This is a huge benefit in semiconductor processing as polishing the wafer to remove subsurface damages (surface and sub surface damage, i.e., cracks, and the amorphous and phase transformed layer) consumes majority of the time and cost in producing mirror-finish parts.

It is believed that the HPPT occurs for all three scratch conditions in order for ductile regime material removal, however, laser heating at high laser powers (45 W) completely anneals the HPPTs leaving the material removed surface with a Si-I structure (no high-pressure phases are retained for this condition only). It is important to note that the retaining high-pressure phases and amorphous layer beneath the machined surface often requires additional processing steps such as polishing to be removed. These forms of “unwanted phases” that remain after the material removal process influence the mechanical, optical and electrical functions of Si parts.201 Also, during a machining process, a surface is always exposed to multiple machining passes due to the cross-feed.
This means that in a conventional machining process, each subsequent cut (except for the first cut) is made on an amorphous/HPP layer and not the crystalline layer. This scenario can be viewed very differently in the case of a μ-LAM process where the material is seen to recrystallize back to its original crystalline phase almost instantly. With this recrystallization effect, every subsequent machining cut (during SPDT) can be made on the original crystalline surface while maintaining its mechanical, electrical and optical properties similar to that as the as-received surface (pristine surface).

The results from the indentation tests were successful in isolating the pressure and temperature effects to confirm that the laser is preferentially heating only the high pressure phases (and not the atmospheric, Si-I phase). The results from this study will also benefit the manufacture of brittle materials as laser heating is demonstrated to decrease the brittle response in ceramics and semiconductors (ref. chapters 5 and 6), thereby providing higher productivity rates (i.e. higher material removal rate) by allowing a greater depth of cut without fracturing the material.

7.5 FUTURE WORK

Subsurface characterization analyses are to be carried on micro-laser assisted single point diamond turned surfaces. Once the μ-LAM technique has been successfully implemented for SPDT, the machined regions will be characterized using μ-Raman spectroscopy. These spectra will then be correlated to the results discussed in this chapter. In addition to the μ-Raman work, TEM analysis will be performed on chips collected from the μ-LAM process to be compared with the current TEM analysis (on chips from cuts with no laser heating discussed in Chapter 5) and correlated to the μ-
Raman work done on the machined chips. TEM is also a useful tool to identify phase transitions that are retained in the plastically deformed chips. Finally, the possibility of the formation of an oxide layer due to the laser heating will be investigated. It is well documented that oxide layers form rapidly on Si wafer surfaces above 1000 °C. The formation of an oxide layer (5 – 10 nm thick) on the crystalline surface could also potentially alter the mechanical and optical properties of the material.
CHAPTER 8

ACOUSTIC EMISSION TO DETECT THE ONSET AND POSITION OF CRACKS AND FRACTURE DURING MACHINING

8.1 INTRODUCTION

Research over the last decades has established the effectiveness of acoustic emission (AE)-based sensing methodologies for machine tool condition monitoring, surface conditioning (i.e., detecting the occurrence of fracture) and process analysis.\textsuperscript{244,245} The difference between the AE technique and other non destructive testing (NDT) techniques is that AE detects the activities inside the materials, while other techniques attempt to examine the internal structures of the materials.\textsuperscript{246} The attempts made to address problems of detecting tool wear and surface fracture in single point diamond turning (SPDT) motivated much of this early work. In addition, the sensitivity of the AE signal to the various contact areas and deformation regions in the cutting and chip formation process has led to evaluation of the analysis of AE signals as a basic tool to monitor and analyze the cutting process.\textsuperscript{247,248} In most of the studies conducted by past researchers, the results suggest that the primary source of emission is sliding friction between the tool nose and/or flank and the workpiece.\textsuperscript{249} There are multiple sources of emission during the material removal process, however, correlating these sources to the AE spectrum has always been challenging. Significant challenges exist in incorporating
AE in a micro-dynamic machining process system as there is a large amount of nonlinearity and instability in AE signals.\textsuperscript{250}

Several machining phenomena have been identified as principal AE sources, such as shearing and deformation with a sharp tool, tool wear, chip deformation, sliding friction between the nose and/or flank of the tool and the machined surface, chip segmentation/chip breaking,\textsuperscript{251} sliding friction between the chip and the top face of the tool,\textsuperscript{252} crack formation/initiation, crack growth and tool fracture. During the cutting process, there are several sources of AE signal such as the initial rubbing process (where the tool/stylus is believed to be merely rubbing against the workpiece surface and not removing any material), the start of the cutting process (where material removal begins), ductile mode machining (material removal via plastic deformation) and brittle machining (where crack or chipping initiation occurs). These different material removal processes are identified by a difference in the signal amplitude or the RMS voltage. However, each of these phenomena releases different energy and noise levels, therefore, identifying the right sensor/filter range to study a specific phenomenon is crucial. Also, identifying useful AE signal during the material removal process can be very challenging due to various sources of AE being simultaneously present (other sources include stage/motor movement, vibration, and environment). One of the major problems faced in the application of AE techniques is represented by the analysis and the interpretation of the emitted signals due to the randomness of AE generated processes.\textsuperscript{253} An AE signal is usually described as non-periodic and contains many frequencies that cannot be explicitly described by mathematical relationships.\textsuperscript{254}
AE, defined as the transient elastic energy spontaneously released in materials undergoing deformation, fracture or both, is dependent upon basic deformation mechanisms such as dislocation motion, grain boundary sliding, twinning, and vacancy coalescence.\textsuperscript{255} Acoustic emission can be related to the grain size, dislocation density, and distribution of second phase particles in crystalline materials and is observed during the deformation of these materials.\textsuperscript{256} Since it is also observed during deformation of non-crystalline materials, its generation cannot be attributed solely to the above-mentioned mechanisms and can also be a result of fracture and decohesion of inclusions, realignment and growth of magnetic domains and phase transformations.

Brittle materials such as ceramics and semiconductors have been used in a number of high-performance optical components. For brittle materials, material removal can occur in two modes: ductile and brittle. The ductile mode machining of brittle materials has been an attractive technique for achieving high form accuracy and good surface finish (mirror-like finish) for such components. In the ductile mode, material is removed by plastic deformation without the generation of surface/subsurface cracks, and brittle fracture is the means of removal in the brittle mode (via crack growth and propagation). Where these cracks occur with respect to the cutting tool position (i.e., at the leading edge, trailing edge or beneath the tool) has not been fully studied and understood.\textsuperscript{257} In most cases, the location of the origin during crack initiation/formation has not been a concern until now, especially in ductile regime machining since fracture is often avoided. It is known that there is a ductile-to-brittle transition (DBT) between the two modes. If the depth or force of a cut exceeds a threshold, the mode of material removal will make a transition from ductile to brittle.
The mode of material removal (ductile or brittle) is generally identified by surface quality observation by SEM, AFM or profilometry (stylus or optical) after the machining process.\textsuperscript{258,72} Therefore, the development of an in-situ process monitoring technology is much needed to detect the nature of the material removal mode: ductile or brittle.\textsuperscript{259} In-situ process monitoring technology (compared to force measurements, which are usually implemented in the structural loop, and therefore affect overall stiffness, as with a dynamometer) will also enable the prediction of the machined surface quality. AE can be used for many applications in the machining of brittle materials. However, in this study, AE is proposed as a method for monitoring the onset of brittle fracture with respect to the tool position. The primary purpose of this study is to establish a sensitive enough AE system capable of identifying and detecting the onset of fracture using AE signal processing during micro-scratching tests of single crystal silicon (Si). However, this study is not limited to only identifying the onset of fracture but more importantly also correlating the AE spectrum to the location/position of fracture/cracks (i.e., leading edge, trailing edge or beneath the cutting stylus/tool).

8.2 EXPERIMENTAL METHOD

8.2.1 AE SENSOR POSITIONING

The onset of brittle fracture or cracks during the material removal process is studied using scratch tests as they best resemble the SPDT machining operation. All scratch tests are conducted on the Universal Micro-Tribometer (UMT) by CETR. The current version of the UMT is equipped with an AE sensor coupled with a band pass filter
with a frequency range of 500 KHz - 5 MHz (refer to the appendices for AE spectra obtained using this band-pass filter). However, this sensor is not capable of picking up low frequency fracture activity (100 – 700 kHz), which is required in this study. To solve this problem, two custom band pass filters are utilized to determine the appropriate frequency range based on the observed brittle activity. The two band pass filter ranges are 2 kHz - 200 kHz and 100 kHz - 2 MHz (the filters along with ease of changeability are shown in the appendices). The sensor feedback system is incorporated within the UMT data acquisition system enabling in-situ AE signal monitoring and post-experimental signal processing. Typical scratch test setups with the AE sensor are shown in Figures 8.1(a) and (b). Since the AE sensor is to be positioned as close as possible to the AE source, two different setups are studied where the sensor is either mounted on the cutting tool fixture/holder (sensor will move with the tool during the scratch test) or on the sample stage (sensor will be stationary but closer to the sample/workpiece).

Figure 8.1: Scratch test setup with AE monitoring on the UMT: (a) AE sensor mounted on the tool fixture, and (b) AE sensor mounted on the sample stage.
Two different scratch setups are shown in Figure 8.1. A stylus based scratch setup, with the AE sensor mounted on the tool fixture is shown in Figure 8.1 (a) whereas a scratch test setup with an actual cutting tool (sensor mounted on the sample stage) is shown in Figure 8.2 (b). It is important to note that both (i.e., sensor mounted on tool fixture and sample stage for both the stylus and cutting tool) positions with respect to the sensor mounting are evaluated for both the stylus and cutting tool.

8.2.2 FIVE MICROMETER TIP RADIUS STYLUS

A 60° conical single crystal diamond stylus (with a spherical end tip radius of 5 μm) is used as the scratch tool. Figure 8.2 shows an optical microscope image of the diamond stylus used in these experiments.

The reason a small radii stylus is used here is because sharp tools in general induce inelastic, irreversible deformation and often form three distinctive crack types: radial, median and lateral. These cracks are usually not observed in cuts performed with a cutting tool. The extreme sharpness in a stylus provides well defined crack
patterns that help in correlating with the AE spectra. Several load ranges from 10 mN to 100 mN are experimented on single crystal Si using a single crystal diamond stylus (at a constant cutting speed of 1 μm/sec). Two different band pass filters, 2 kHz - 200 kHz and 100 kHz - 2 MHz, are used in this set of experiments. The scratches along with the cracks are imaged using an optical microscope and correlated to the AE spectra.

8.2.3 ONE MILLIMETER NOSE RADIUS TOOL

A 1 mm nose radius single crystal diamond cutting tool (-45° rake and 5° clearance angle) is used for this set of experiments. The reason an actual cutting tool is used is to generate brittle fracture that would occur in an actual SPDT operation. These fracture characteristics are usually very different from that observed in scratches performed with a sharp stylus. Several loads from 10 mN to 400 mN (at a constant cutting speed of 1 μm/sec) are utilized to intentionally generate brittle behavior. Two different band pass filters, 2 kHz - 200 kHz and 100 kHz - 2 MHz, are used in this set of experiments. Figure 8.3 schematically illustrates the material removal mechanism during a single point scratch test using a single crystal diamond cutting tool.

Figure 8.3: Plastic deformation on a brittle material using a custom cutting tool.
8.3 RESULTS AND DISCUSSION

8.3.1 AE SENSOR POSITIONING

The high-frequency AE sensor is extremely sensitive for detecting material deformation, crack formation and growth, delamination, adhesion and even contamination (usually for polishing). However, mounting the AE sensor at an optimized position is crucial for obtaining the maximum acoustic signal during the material removal process.\textsuperscript{261} Besides positioning the sensor as close as possible to the sample, having a good conductive medium where the AE waves can travel through is also important. The other important criteria when positioning the sensor is to avoid background/unwanted noise. This is usually achieved by mounting the sensor in a stable fixture, where the sensor surface has maximum contact with the mounted fixture surface (the sensor should always be mounted on a smooth surface to maximize the contact area). In this study, the AE sensor position is optimized for obtaining better signals with less background noise during the scratching of Si. \textit{Figure 8.4} compares the AE spectrum obtained when the sensor is mounted on the tool versus the sample stage.
Figure 8.4: AE spectrum at different sensor mounting positions: (a) scratch done with stylus, (b) spectrum obtained when sensor mounted on the tool fixture, and (c) spectrum obtained when sensor mounted on the sample stage.

An increasing load from 10 mN to 80 mN is used for the scratches performed to compare both sensor mounting positions. From Figure 8.4, it is seen that the spectrum obtained with the sensor mounted on the tool (Figure 8.4 (a)) correlates well with the features seen along the scratch edges. This is clearly demonstrated as the spikes in AE signal (peaks in the spectrum) line up with the cracks and fracture behavior seen along the scratch (Figure 8.4 (b)). However, there is a very poor and less obvious correlation observed between the scratch’s brittle characteristics and the spectra obtained with the
sensor mounted on the sample stage (Figure 8.4 (c)). In this case, it is evident that a combination between significant background noise and low levels of AE signal during the fracture events makes it nearly impossible to filter out and determine the important data. The background noise can be attributed to the strain gage movement within the load cell, and floor and building vibrations that affect the lower stage of the equipment more than the upper stage. Also, the absolute RMS signal voltage is significantly lower with the sensor mounted on the sample stage. One of the most significant methods to analyze AE is the measurement of the energy content of the AE signals. The rate with which the energy is transmitted as the AE signal can be directly correlated with the rate of energy generated by the original AE source. A simple way to measure such energy is the evaluation of the RMS voltage of the signals. With these findings, the optimized sensor position is determined to be on the tool fixture. The tests discussed in the following sections utilize this optimized mounting position (sensor mounted on the tool).

8.3.2 FIVE MICROMETER TIP RADIUS STYLUS

Two different band-pass filters are used to correlate the fracture behavior to the AE spectrum. Since very little research has been done previously for this specific purpose (only large scale fracture events and cracks during indentation has been studied), the ideal frequency range is yet to be determined. The first band pass filter used is within a 2 kHz to 200 kHz range. Figure 8.5 shows a scratch done with a linearly increasing load (10 mN to 80 mN) that exhibits both ductile and brittle behavior. The load range is selected specifically to have minimal ductile response and maximum brittle response (but not to the extent of catastrophic failure) to generate cracks along the edges.
Figure 8.5: Scratch with corresponding force data and AE spectrum (2 kHz - 200 kHz).

Figure 8.5 shows a scratch with brittle characteristics correlated to its corresponding AE RMS voltage signal, cutting force ($F_x$) and thrust force ($F_z$). The crack pattern seen along the scratch edges resemble lateral cracks that are usually observed during indentations and micro-scratching. Lateral cracks are horizontal cracks that occur beneath the surface and are symmetric with the load axis. They are produced by excessive tensile stress and often extend to the surface, resulting in a surface feature which may lead to chipping of the surface specimen. The brittle material removal region is identified on the AE spectrum by correlating the RMS voltage spikes with the actual brittle region identified from the optical microscope image. In this case, the lateral
cracks are correlated to the AE spectrum as shown in Figure 8.5. In general, the distance between these cracks can be measured under the optical microscope and therefore this frequency (at which cracks form) can be correlated to the AE spectrum. In this case, the measured distances between the cracks seem to be fairly uniform, where a lateral crack forms every 5-8 µm along the scratch (which also matches the AE signal spike frequency). The pattern recognition and correlation with the AE spectra during a machining process has also been previously studied during the cutting process by negating the influence of other variables. \(^{264}\) In reality, since cracks tend to send out a larger AE signal (producing larger RMS values), the sharp peaks/spikes seen in the AE spectrum here can be correlated to the cracks along the scratch edge.

In general, there are three main types of crack patterns that could occur from scratching brittle materials with a sharp stylus: radial, median and lateral cracks. Each crack is believed to have different patterns/characteristics and each initiates due to different factors such as excessive depth of cut, stress generated from material pile-up, tip geometry and more. \(^{265}\) Besides these three main crack patterns, there are also less common crack patterns such as the umbrella, chevron, branch and fork cracks that also tend to generate during the machining of brittle materials. Some of these crack patterns/configurations are shown in Figure 8.6.
The next scratch is carried out with a linearly increasing load from 20 mN to 100 mN with a 100 kHz - 2 MHz band pass filter. Figure 8.7 shows an optical microscope image of the scratch along with its corresponding cutting forces (Fx) and AE spectrum.
The AE spectrum obtained for this particular band pass filter (100 kHz - 2 MHz) has a very different pattern compared to that seen in Figure 8.5 (2 kHz - 200 kHz). The AE signal in the spectra seen in Figure 8.7 is very sensitive to the cracks or voids formed along the center of the scratch, which are referred to as median cracks. Median cracks are vertical voids that form beneath the surface parallel to the loading direction. Depending on the loading conditions, median cracks may extend upward into the surface, generally in the center of the cut as seen in Figure 8.7. In some conditions, median cracks also join with the surface radial cracks, forming two half-penny cracks (not observed in the cuts reported here). From previous research, it is known that radial cracks generally form at the trailing edge of the tool/stylus due to an excessive depth of cut.\textsuperscript{267}

Although lateral cracks are still present and clearly seen in Figure 8.7 (b) along the scratch, the corresponding signal/filter only picks up the radial-median cracks. A very low signal in the AE spectrum is observed (between cut distance 180 - 200 μm) when severe lateral cracks are formed. This could be attributed to the different levels of noise and energy released by each type of crack that are only picked by specific band-pass filters. In this particular case, the band-pass filter only picks up median cracks as it is generally regarded as the deepest crack among all.\textsuperscript{268} It is important to note that the cutting forces (F\textsubscript{x}) also picks up significant fracture behavior and there is a good correlation between the instabilities seen in the cutting force data and the spikes in the AE spectrum. However, the cutting forces are usually only sensitive to fracture events occurring directly in front or beneath the tool.

It is also important to understand the fundamental mechanics that govern the lateral and median crack formation. The crack formation mechanism has been previously
studied and illustrated. *Figure 8.8* pictorially describes the median and lateral crack systems.

*Figure 8.8: Radial-median and lateral crack systems.*

*Figure 8.8* depicts the crack evolution during complete loading and unloading where the dark region denotes the irreversible deformation zone. Upon loading, at some threshold, a deformation induced flaw is initiated and develops into a median crack. As the applied load is linearly increased, this crack continues to grow. Upon unloading, the median cracks tend to close and lateral cracks start forming at the edge of the plastically deformed zone. These lateral cracks continue to grow and extend to the surface upon further release of the tool. By understanding this process, the formation of the cracks
shown in Figures 8.5 and 8.7 can be better understood and correlated to the material removal process.

8.3.3 ONE MILLIMETER NOSE RADIUS TOOL

A similar experimental method is used for the stylus-based scratch tests. Two different band-pass filters are utilized while performing cuts with an increasing load. The loads are chosen such that the cuts exhibit both ductile and brittle behavior to initiate fracture behavior. The first cut carried out uses the 2 kHz – 200 kHz band-pass filter with a linearly increasing load range of 2 – 30 mN. Figure 8.9 shows the imaged cut along with the AE spectrum and cutting force data. Unlike the scratches obtained with the stylus, the brittle fracture seen in this cut has a very different pattern (more random). Instead of actual cracks, divots/voids are noticed at the center of the cut, which is also the deepest region of the cut.
Figure 8.9: Cut with corresponding force data and AE spectrum (2 kHz - 200 kHz).

The spikes in the AE spectrum correlate reasonably well with the major voids seen along the cut. However, there are also other sources of acoustic activity that is present in this spectrum that could be originating from several other AE events (e.g., chip formation, chip breaking, plastic deformation, rubbing/friction, etc.). To help with isolating the unintended signals, a different band-pass filter is used (100 kHz – 2 MHz).

The second cut is performed with a more conservative load range between 2 – 20 mN. This is to minimize and space out the brittle fracture activity that can then be easily correlated to the AE spectrum. Figure 8.10 shows the imaged cut along with the AE spectrum and cutting force data.
Figure 8.10: Cut with corresponding force data and AE spectrum (100 kHz to 2 MHz).

From Figure 8.10, the brittle fracture events (voids/divots) correlate well with the spikes in the RMS voltage seen in the AE spectrum. It is important to notice that the cutting force ($F_x$) data also complements the AE spectrum. However, the AE signal seems to be very sensitive to brittle fracture events (more sensitive than the cutting forces), which is the intent of this study. The spectrum here only depicts the main fracture events and eliminates other background noises as observed in Figure 8.9. The voids/divots observed in these cuts are believed to be occurring at the leading edge of the tool (in front of the tool) due to the excessive triaxial state of stresses generated. This is confirmed by analyzing the surface profile of the cut using white light interferometry. An
example of the surface profile of a cut with significant amount of brittle fracture is shown in Figure 8.11.

The three-dimensional surface profile confirms that the fracture events occur in front of the tool as no traces of ejected material are observed on the cut surface. It is believed that the material is still ejected to the surface during the fracture occurrence however, the passing/trailing tool removes this ejected material leaving behind only the voids/divots that extend into the machined surface. This phenomenon is schematically illustrated in Figure 8.12. Another interesting observation from Figure 8.10 is that almost all arrows (connection the AE peaks and fracture events on the cut) are not lined up straight, further indicating an offset between the AE signal and the actual fracture event. Since the sensor is mounted on the tool, but the fracture is actually occurring ahead of the tool, this causes the spikes/peaks to appear at an offset (relative to the fracture occurrence) as observed in Figure 8.10. Based on the offset distance and cutting speed, it is estimated that these fracture events occur in the range of 4 - 6 μm ahead of the tool.
Stage 1 shows the tool positioned at the start of the cut on an unmachined surface. When the stresses exceed beyond the allowable threshold, cracks form in front of the tool (leading edge of the tool). Due to excessive triaxial state of stresses, fracture is initiated in front of the tool (stage 2). Material is then ejected, leaving a void or divot behind, due to these fracture occurrences but is later removed by the trailing tool during the cut as illustrated in stage 3. Finally in stage 4, only the void/divot remains with a fairly flat surface containing no or little remains of ejected material (as observed in the profile shown in Figure 8.11). Another scenario that also occurs during a cutting process is fracture at the trailing edge of the tool. This phenomenon is demonstrated in Figure 8.13.
In Figure 8.13, stage 1 shows the tool positioned at the start of the cut on an unmachined surface. When the tensile stresses exceed the allowable threshold, fracture forms behind the tool (at trailing edge of the tool). In general, excessive tensile stresses result in cracks/fracture behind and at the side of the tool. Material is then ejected due to these fracture events and but it is not removed by the tool as the tool has already passed this point at this stage (stage 2). Finally in stage 3, the crack and the ejected material remain with a fairly uneven surface at the end of the cutting process. Lateral cracks that form from median cracks or even pure median cracks due to excess loads would fall into this category (formed in the trailing tensile stress field).

8.4 CONCLUSION

It is important to ensure that sensing methodologies that are applied to process monitoring yield reliable data that can be quantitatively related to the process being investigated or monitored. A successful and sensitive enough AE sensing system has been developed to detect crack initiation and fracture behavior during the material removal of ceramics and semiconductors. The initiation of AE signals correlate well with the crack initiation for all scratch tests on single crystal silicon. Two different band-pass filter ranges have been identified to detect different crack patterns. For a stylus based scratch test, the 2 kHz - 200 kHz band-pass filter is suitable to detect lateral cracks along the edges of the scratch. The deeper median cracks (that often extend to the surface) however are detected using the 100 kHz - 2 MHz band-pass filter. The spikes/peaks in the AE spectra correlate well with the cracks along the scratches. Lateral cracks usually occur at specific and uniform intervals/distances making them easily correlated to the AE
signal spike pattern. For the cut carried out with the 1 mm nose radius cutting tool, the 100 kHz - 2 MHz band-pass filter provided useful data correlating to the voids/divots observed along the center of the cut. The occurrence of fracture activity in front of the tool (at the leading edge) causes these voids or divots and is confirmed from the white light interferometric surface profile scan. This fracture occurring at the leading edge is further confirmed by the offset seen in the AE spikes relative to the actual fracture position along the cut. The cutting force also complemented the AE spectra indicating critical fracture along the cuts, however, AE proved to be a much more sensitive technique to detect crack initiation and fracture behavior during the cutting process. It is also determined that in order to monitor more than one type of crack or fracture event, at least two sensors with different band-pass filters need to be utilized, as different cracks are detected in different frequency ranges.

8.5 FUTURE WORK

The eventual goal of this study is to develop an effective control scheme to attain ductile-regime machining using AE signal feedback. First, a more fundamental study of the origins of the cracks and correlation to AE is required. This is to make sure that the AE system is sensitive enough to pick up all forms of fracture behavior and crack patterns consistently. Also, the AE system will be tested to see if cracks that occur below the machined surface (cracks that do not extend to the surface) can be detected. This will be done using an optically transparent workpiece such as quartz, where microscopy techniques can be used to image below the surface. The next step is to implement AE sensing to monitor brittle fracture events by varying different machining parameters, such
as depth of cut, feed, and laser heating. Once reliable AE signal conditioning has been established, in-situ signal monitoring will be used to push the windows in ductile mode machining to increase the material removal rates.
CHAPTER 9

DUCTILE-TO-BRITTLE TRANSITION OF CERAMICS AND SEMICONDUCTORS

9.1 INTRODUCTION

Ceramics and semiconductors are naturally brittle materials at room temperature and are known for their poor machinability. However, ductile-regime machining is possible under certain conditions (due to the high pressure phase transformation phenomenon). 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. One of the initial steps before carrying out any form of ductile mode machining on brittle materials is to define a DBT or critical depth. Once the DBT is defined (analytically and experimentally verified) for a particular material, all machining parameters such as the depth of cut and feed can be altered in order not to exceed the DBT depth. Exceeding the DBT/critical depth during a machining process will result in material fracture which then generates poor surface finish and at times catastrophic failure of the manufactured part.

For brittle materials like ceramics and semiconductors, material removal can occur in two modes: ductile and brittle. In the ductile mode, material is removed by plastic deformation without the formation of any surface and subsurface cracks. In the brittle mode, fracture dominates the material removal mechanism, usually resulting in
significant surface and subsurface cracks. It is well known that there is usually a ductile-to-brittle transition between these two competing modes for nominally brittle materials. If the depth or applied force of a cut exceeds the threshold value, the mode of material removal will make a transition from ductile to brittle. To achieve high form accuracy and good surface finish for optical and electronic components, usually only ductile mode material removal of brittle materials is desired for the finishing processes.

The transition from ductile to brittle mode during the material removal process of nominally brittle materials (semiconductors and ceramics) is described in terms of the energy balance between strain (volume) energy and surface energy. Relatively little energy is required to initiate and extend a crack to fracture brittle materials where atoms are separated and a new surface along the failure path is exposed. However, significantly more energy is required to plastically deform brittle materials without generating cracks. In brittle mode material removal, cracks are generated, and these cracks play an important role in understanding ductile mode machining. Although many ceramics and semiconductors are stronger than metals, they are not used in many impact prone applications. This is because metals can absorb a large amount of energy before failure, whereas ceramics and semiconductors usually cannot (exceptions are purposeful toughening mechanisms such as the tempering of glass and transformation toughened zirconia). The parameter describing the ability of a material to absorb energy is toughness. In general, ceramics and semiconductors have very low toughness.

Previously, three zones of brittle fracture have been identified during ductile-regime cutting experiments: (1) during chip formation, (2) trailing tensile stress field, and (3) lateral crack formation after tool pass. Brittle fracture can occur in front of the tool
(chip formation zone), below the tool or behind the tool (trailing tensile field). The critical depth can be associated with each of the fracture modes; however, it is the mode that first arises that is the limiting case. From the results and discussion in Chapter 8, it is seen that for fracture that occurs in front of the tool, the final machined surface is fairly smooth as the tool passes the fracture zones and removes all ejected material. However, tensile fracture events that occur at the trailing edge of the tool such as radial, medial or lateral cracks are characterized as catastrophic since they often extend beyond the tool path and machined surface.

In general, using a more negative rake angle tool, to generate higher compressive stresses, helps to prevent fracture at the leading edge of the tool and can assist by rotating the direction and location of the trailing tensile stress field (generally towards the surface). However, there is still a possibility of cutting too deep with a highly negative rake angled tool and causing fracture ahead of the tool (as demonstrated in Chapter 8). This could be attributed to the total high-pressure phase transformed zone that is not large enough to encompass the chip formation zone; therefore deformation of the brittle/atmospheric phase is attempted, thus resulting in fracture. Therefore, fracture in front of the tool could occur due to excessive shear or tensile stresses.

This chapter discusses results from experiments carried out to determine the DBT depth of several ceramics and semiconductors of interest, such as silicon carbide (SiC), silicon (Si), quartz (SiO₂), aluminum oxynitride (AlON), sapphire (Al₂O₃), spinel (MgAl₂O₄) and aluminum titanium carbide (AlTiC). Since SiC is of main interest in this research, three different polytypes are evaluated: single crystal 4H, single crystal 6H and polycrystalline 3C. Single point scratch tests are carried out to determine the DBT depth
of the materials. Scratch tests are chosen to be the principle method of investigation in this study as it is a better candidate for evaluating machining conditions than indenting. This is because the mechanics during scratching are more applicable to the machining process, such as in single point diamond turning (SPDT). The scratch tests are performed within an applied load range (thrust force) that is expected to exhibit the ductile, DBT and brittle regions. Once the scratch tests are completed, optical microscopy and interferometric profilometry analyses are carried out and correlated in order to determine the DBT depth (with its corresponding acoustic emission spectra, and cutting and thrust forces). The results from the depth analysis will be used as guidelines during the selection of SPDT parameters in the future. Finally, micro-Raman spectroscopy on sapphire is also discussed. A total of five materials have been characterized using μ-Raman (Si, three SiC polytypes and Sapphire). Since the μ-Raman results of Si (Chapters 5 and 7) and SiC (Chapter 6) are discussed in previous chapters, only the results for single crystal sapphire are discussed here (since this is the only chapter in which sapphire is discussed).

9.2 EXPERIMENTAL METHOD

The scratch tests are performed on a Universal Micro-Tribometer (UMT) which is produced by the Center for Tribology Research Inc. (CETR). This equipment is developed to perform comprehensive micro-mechanical tests of coatings and materials at the micro scale with a nanometric resolution. This system facilitates cutting speeds as low as 1 μm/sec at nanometric cutting depths. Unlike commercial lathes, the UMT is a load controlled device (versus depth controlled) where the required thrust force/load ($F_z$) is applied by the user to obtain the desired depth of cut (based on the tool geometry and
workpiece material properties). The unit is equipped with a dual-axis load cell that is capable of constantly monitoring the thrust and the cutting forces \( (F_x) \), which are obtained as an output parameter from the cutting experiment. A typical scratch test setup on the UMT is shown in *Figure 8.1* (Chapter 8). A similar setup is used for all DBT experiments except for the tests that utilize laser heating (refer to setup pictures from Chapters 5 and 6). A 1 mm nose radius single crystal diamond cutting tool \((-45^\circ \text{ rake and } 5^\circ \text{ clearance})\), similar to that used in SPDT experiments, is used for this set of experiments. The reason an actual cutting tool is used for these tests is to establish DBT depths that can be used during actual SPDT operations. A stylus generates a more aggressive cut but the depth and loads are not applicable during SPDT. Also, using similar tool geometries generates brittle fracture similar to that in an actual SPDT operation, further enabling the prediction of fracture behavior in these materials during machining.

The scratch test parameters are summarized in *Table 9.1* for all nine materials experimented on. The applied load range is chosen based on some preliminary results where the material is seen to exhibit both ductile and brittle characteristics (with a DBT zone). The DBT depths of a total of five materials of interest are also determined with laser heating. The DBT depths obtained with laser heating are then compared to the DBT depths at room temperature to study the ductile response of the material due to thermal softening. Thermal softening of the material via laser heating has been proven to be beneficial in the material removal process by enhancing the ductility of brittle materials (refer to Chapters 5, 6 and 7). In order to preferentially heat the material, traditional scratch tests are coupled with a micro-laser assisted machining (\( \mu \)-LAM) technique (refer
to Chapters 5 and 6). All cuts reported here are performed at a constant cutting speed of 1 μm/sec.

Table 9-1: Scratch test parameters for experimented materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Applied Load Range</th>
<th>Heating Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>4H-SiC</td>
<td>70 mN – 200 mN</td>
<td>With &amp; Without Laser</td>
</tr>
<tr>
<td>6H-SiC</td>
<td>70 mN – 200 mN</td>
<td>With &amp; Without Laser</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>200 mN – 700 mN</td>
<td>With &amp; Without Laser</td>
</tr>
<tr>
<td>Silicon</td>
<td>50 mN – 300 mN</td>
<td>With &amp; Without Laser</td>
</tr>
<tr>
<td>Sapphire</td>
<td>400 mN – 800 mN</td>
<td>With &amp; Without Laser</td>
</tr>
<tr>
<td>Quartz</td>
<td>50 mN – 300 mN</td>
<td>Room Temperature</td>
</tr>
<tr>
<td>Spinel</td>
<td>50 mN – 300 mN</td>
<td>Room Temperature</td>
</tr>
<tr>
<td>AlON</td>
<td>150 mN – 400 mN</td>
<td>Room Temperature</td>
</tr>
<tr>
<td>AlTiC</td>
<td>200 mN – 600 mN</td>
<td>Room Temperature</td>
</tr>
</tbody>
</table>

The scratch tests are then analyzed using optical microscopy (to detect the onset of brittle behavior), white light interferometry (for depth profile measurements) and force analyses (cutting forces are very sensitive to brittle activity). The final part of the DBT analysis is to be able to relate the DBT depths and forces to a few crucial material mechanical properties that are believed to influence the ductile response during material removal. This study also attempts to correlate the parameter(s) (material property/properties), if any, that is/are most sensitive to the DBT of the ceramic/semiconductor.

9.3 RESULTS AND DISCUSSION

9.3.1 FORCE DATA

Unstable forces (cutting and thrust) are usually a good indication of brittle fracture in the material removal process. The cutting forces are extremely sensitive to fracture and brittle activities, as they generally occur along the cutting direction. This is
clearly illustrated in Figure 9.1, where the effect of brittle fracture along the cut is clearly seen in the cutting force data. During the material removal process using a single point diamond tool, cutting forces can vary due to numerous reasons, such as surface roughness, debris from machining, lack of lubrication/slurry, uneven tool wear, external vibration, etc. In general, the lower the cutting forces, the better the overall performance of the machining process (lower cutting forces are desired). Lower cutting forces are also desired as they significantly extend the tool life during machining.

![Figure 9.1: Force data correlated to ductile and brittle modes.](image)

In Figure 9.1, the force data of a scratch test performed on polycrystalline cubic-spinel is shown. A linearly increasing load from 10 mN to 350 mN is used to exhibit both ductile and brittle modes of material removal. It is clearly observed that the cutting forces are very sensitive to brittle fracture and result in instability when fracture occurs. The initial instability observed (F_x = 30 mN) in the cutting forces is generally the point at which the material starts to exhibit brittle behavior. Beyond this point, the standard deviation in cutting forces increase significantly due to the uncontrolled material removal.
process. Monitoring the cutting forces during the cutting process is also an effective in-situ monitoring method to avoid brittle mode machining.

9.3.2 OPTICAL MICROSCOPY

Analyzing the cuts using optical microscopy is an extremely time efficient and straightforward method of detecting brittle behavior on the machined surface. By using the appropriate magnification, optical microscopy is used to analyze the material removed surfaces. A brittle mode material removal process can be distinctly identified, as it leaves a poor surface finish due to the cracks and voids/divots formed during cutting. Figure 9.2 shows a series of cuts performed on polycrystalline Spinel at different loads (from 100 mN to 300 mN). The optical microscope images of cuts performed on other materials in this chapter are given in the appendices. Besides performing a single cut with a linearly increasing load (as shown in Figure 9.1), performing multiple cuts at different loads (with a constant increment) is also an effective way to determine the DBT depth of the material.
In *Figure 9.2*, it is clearly seen that the first two cuts (100 mN and 150 mN) are perfectly ductile; however, the cut done at 200 mN starts to exhibit brittle behavior. In general, the brittle behavior is noticed along the center of the cut, as this is the deepest region of the cut (due to the 1 mm tool nose radius). Beyond an applied load of 200 mN, the cuts tend to be mostly brittle. In fact, the cut done at 300 mN has severely fractured the surface, leaving a poorly defined cut edge and very deep cracks. In brittle mode machining, once a crack gets to the edge of a cut (outside the compressive zone), it could encounter a tensile field and propagate beyond the scratch edges.
9.3.3 WHITE LIGHT INTERFEROMETRY

In this study, white light interferometry is used to measure the depth of cut and analyze the cut profile for all experimented materials. In some materials, such as AlTiC, the as-received surface appears to be rough (but is not actually rough) due to its polycrystalline nature. The presence of grains makes it challenging to differentiate between a ductile and a partially brittle cut. This scenario is demonstrated in Figure 9.3, where two different cuts on AlTiC are shown (two different applied loads).

![Figure 9.3](image.png)

**Figure 9.3 Cuts performed on AlTiC: (a) a fully ductile cut, and (b) a partially brittle cut.**

Visually, from the microscope images, it is difficult to differentiate between a fully ductile cut (in this case *Figure 9.3 (a)*) and a partially brittle cut (in this case *Figure 9.3 (b)*). The poor uniformity and grain texture on the surface often tends to mislead the experimenter. Particularly, in cases like this, white light interferometry proves to be a huge advantage. The depth profiling obtained from white light scans clearly distinguishes the two material removal modes; ductile and brittle. *Figures 9.4 and 9.5 show an example*
for both cases; a fully ductile cut is shown in Figure 9.4 and a partially brittle cut is shown in Figure 9.5.

**Figure 9.4**: Surface profile of a fully ductile cut.

*Figure 9.4* shows the well-defined tool imprint represented by the cross-sectional profile for the cut done on AlON. There are no signs of material fracture observed in the groove of a perfectly ductile cut.

**Figure 9.5**: Surface profile above indicating the DBT.

*Figure 9.5* indicates the DBT of the Sapphire sample where the well-defined tool imprint is not evident at the tip of the groove. The DBT depth measurement taken in this particular scan is 430 nm.
The first region imaged and analyzed is the ductile region. Figure 9.4 shows a typical 2-D cross-section of a perfectly ductile region along with a three dimensional (3-D) scratch profile. Figure 9.5 shows the cross-section at the DBT zone (the point at which fracture is first identified) in Sapphire. The defined feature of the tool imprint seen in Figure 9.4 is no longer visible in Figure 9.5. In this study, 2-D cross-sectional profiling of the cuts is analyzed at every point along the cut to obtain depths of cuts at various points. It is important to perform a three-dimensional analysis along the cut (in this case white light interferometry was chosen as the method of analysis) as the onset of fracture could occur beneath the material removed surface (subsurface) before appearing on the surface (as seen in the optical microscope images).

9.3.4 ACOUSTIC EMISSION

As discussed in Chapter 8, acoustic emission (AE) is a great tool for detecting fracture behavior during a material removal process. In fact, AE sensors are more sensitive to the onset of fracture events than load cells. In brittle regime machining, the AE spectrum correlates well with the cutting force data, as seen in Figure 9.6.
Figure 9.6 correlates the AE spectrum and the cutting forces for both material removal modes: ductile and brittle. The two different material removal modes are also identified in the optical microscope image and cross-sections of the depth profile. The brittle region in the optical microscope image is identified by the cracks formed along the scratch edge. The poorly defined groove edges seen in the cross-sectional profile of the brittle region along the scratch are a clear indication of brittle mode machining.
9.3.5 DBT DEPTS AND MATERIAL PROPERTIES

9.3.5.1 DBT AT ROOM TEMPERATURE

The DBT zone is identified by the onset of brittle fracture. At this point the material removal process is no longer fully ductile (partial ductility is possible) but neither is it fully brittle; and is hence termed the DBT zone. In order to improve the surface finish in ceramics/semiconductors, it is important to keep the entire material removal process in the ductile zone. After correlating the force data, optical microscope images and white light profilometry scans, the DBT parameters are obtained and summarized in Table 9.2. In this section, only the cuts done at room temperature (no laser heating) are discussed.

Table 9-2: DBT parameters for ceramics and semiconductors of interest.

<table>
<thead>
<tr>
<th>Material</th>
<th>DBT Load*</th>
<th>DBT Cutting Force*</th>
<th>Avg. DBT Depth*</th>
</tr>
</thead>
<tbody>
<tr>
<td>4H-SiC</td>
<td>85 mN</td>
<td>31 mN</td>
<td>20.0 nm</td>
</tr>
<tr>
<td>6H-SiC</td>
<td>120 mN</td>
<td>48 mN</td>
<td>17.5 nm</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>450 mN</td>
<td>230 mN</td>
<td>42.5 nm</td>
</tr>
<tr>
<td>Silicon</td>
<td>175 mN</td>
<td>105 mN</td>
<td>185.0 nm</td>
</tr>
<tr>
<td>Sapphire</td>
<td>650 mN</td>
<td>430 mN</td>
<td>122.5 nm</td>
</tr>
<tr>
<td>Quartz</td>
<td>200 mN</td>
<td>127 mN</td>
<td>87.5 nm</td>
</tr>
<tr>
<td>Spinel</td>
<td>200 mN</td>
<td>133 mN</td>
<td>205.0 nm</td>
</tr>
<tr>
<td>AlON</td>
<td>320 mN</td>
<td>186 mN</td>
<td>187.5 nm</td>
</tr>
<tr>
<td>AlTiC</td>
<td>400 mN</td>
<td>265 mN</td>
<td>425.0 nm</td>
</tr>
</tbody>
</table>

Note: *Measurement obtained just before the DBT (first point of fracture occurrence).

The DBT depths provided in Table 9.2 are averages of five measurements taken along the cut region. The light interferometric measurement parameters such as objective lens magnification, scan speed, scan area and light intensity, are kept constant for all measurements. Based on the literature review and material data sheets (provided by the manufacturers), material properties/characteristics that are believed to be influential in the DBT are shown in Table 9.3.
Table 9-3: Material properties for ceramics and semiconductors experimented.

<table>
<thead>
<tr>
<th>Material</th>
<th>Form</th>
<th>Hardness, (GPa)</th>
<th>Fracture Toughness (MPa.m^0.5)</th>
<th>Young’s Modulus, (GPa)</th>
<th>Density, (g/cc)</th>
<th>Poisson’s ratio, (ν)</th>
<th>Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC-4H(^{275})</td>
<td>Single Crystal</td>
<td>26</td>
<td>1.21</td>
<td>330</td>
<td>3.22</td>
<td>0.14</td>
<td>N/A</td>
</tr>
<tr>
<td>SiC-6H</td>
<td>Single Crystal</td>
<td>26</td>
<td>1.21</td>
<td>330</td>
<td>3.22</td>
<td>0.14</td>
<td>N/A</td>
</tr>
<tr>
<td>SiC-3C</td>
<td>Polycrystalline</td>
<td>27</td>
<td>3</td>
<td>466</td>
<td>3.17</td>
<td>0.17</td>
<td>20-30 µm</td>
</tr>
<tr>
<td>Si(^{276})</td>
<td>Single Crystal</td>
<td>12</td>
<td>0.91</td>
<td>150</td>
<td>2.33</td>
<td>0.17</td>
<td>N/A</td>
</tr>
<tr>
<td>Quartz(^{277})</td>
<td>Amorphous</td>
<td>9</td>
<td>0.92</td>
<td>73</td>
<td>2.21</td>
<td>0.16</td>
<td>N/A</td>
</tr>
<tr>
<td>Spinel (Cubic)</td>
<td>Polycrystalline</td>
<td>12.1</td>
<td>1.72</td>
<td>277</td>
<td>3.58</td>
<td>0.26</td>
<td>100-200 µm</td>
</tr>
<tr>
<td>Sapphire(^{278})</td>
<td>Single Crystal</td>
<td>15</td>
<td>2.5</td>
<td>344</td>
<td>3.98</td>
<td>0.27</td>
<td>N/A</td>
</tr>
<tr>
<td>AlO(_{N}) (Cubic)</td>
<td>Polycrystalline</td>
<td>13.8</td>
<td>2</td>
<td>315</td>
<td>3.67</td>
<td>0.24</td>
<td>150-250 µm</td>
</tr>
<tr>
<td>AlTiC</td>
<td>Polycrystalline</td>
<td>19</td>
<td>4.3</td>
<td>390</td>
<td>4.26</td>
<td>0.25</td>
<td>&lt;1µm</td>
</tr>
</tbody>
</table>

The wide range of material hardness (9 GPa to 27 GPa) experimented here includes various forms, such as single crystals, polycrystals and amorphous materials. To maintain consistency among the materials, all hardness values reported here are micro-hardness values (not nano-hardness). This is an important factor, as the nano-hardness of some materials (such as 4H and 6H-SiC) can be almost three times the hardness values reported in Table 9.3 (refer to Chapter 6). In order to determine if any one of the mechanical properties greatly influence the DBT depth, each property is initially compared with the DBT depth. However, there is no direct correlation observed between both mechanical property (Young’s modulus (E), fracture toughness (K\(_{IC}\)), hardness (H) and Poisson’s ratio (ν)) and the DBT depth of the materials. These individual correlations are given in the appendices. This is reasonable, as there are several other parameters such as the form of material (single crystal, polycrystalline or amorphous), grain size (if polycrystalline), mode of fracture (during the DBT), cutting direction with respect to the crystal/grain orientation, etc. that have to be studied in detail in order to correlate any particular material property to the DBT depth. However, this is not the intent of the
The current study. There have been past studies reporting a huge variation in crack behavior and fracture toughness values even in the same wafer, when cutting along different directions.\textsuperscript{111}

Since the hardness and fracture toughness values are the two main properties of interest during the machining process, these properties are analyzed together. In general, to achieve high material removal rates and low tool wear, a material with low hardness and high fracture toughness is desired (only from a machining point of view and not for a specific application). Therefore, the DBT depths of the materials are compared to a hardness-to-fracture toughness ratio (known as the brittleness index). The brittleness index is often closely related to the critical load to initiate a median-radial crack.\textsuperscript{279}

However, the hardness values used here are calculated based on the Griffith’s fracture propagation criterion (recall Equation 2.4 in Chapter 2). Calculated hardness values are used instead of the literature obtained hardness values, as the calculated hardness does incorporate the shear component that is not present in hardness indentation tests (i.e., literature hardness values are based on indentation tests and not scratch tests). It is important to incorporate the shear component to better mimic the actual cutting operation as previous studies have reported a reduction in phase transformation pressure by a factor of 3-4 by introducing the plastic shear component.\textsuperscript{280} Table 9.4 summarizes the calculated hardness values for the studied materials (sample hardness calculation given in the appendices).
Table 9-4: Hardness and calculated toughness for all materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Form</th>
<th>Calculated Hardness, (GPa)</th>
<th>Fracture Toughness (MPa.m^{0.5})</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC-4H</td>
<td>Single Crystal</td>
<td>28.9</td>
<td>1.21</td>
</tr>
<tr>
<td>SiC-6H</td>
<td>Single Crystal</td>
<td>30.2</td>
<td>1.21</td>
</tr>
<tr>
<td>SiC-3C</td>
<td>Polycrystalline</td>
<td>46.2</td>
<td>3.00</td>
</tr>
<tr>
<td>Si</td>
<td>Single Crystal</td>
<td>7.9</td>
<td>0.91</td>
</tr>
<tr>
<td>Quartz</td>
<td>Amorphous</td>
<td>10.3</td>
<td>1.15</td>
</tr>
<tr>
<td>Spinel</td>
<td>Polycrystalline</td>
<td>15.9</td>
<td>1.72</td>
</tr>
<tr>
<td>Sapphire</td>
<td>Single Crystal</td>
<td>21.6</td>
<td>1.89</td>
</tr>
<tr>
<td>AION</td>
<td>Polycrystalline</td>
<td>18.9</td>
<td>2.00</td>
</tr>
<tr>
<td>AlTiC</td>
<td>Polycrystalline</td>
<td>25.7</td>
<td>4.30</td>
</tr>
</tbody>
</table>

In this comparison (in Figure 9.7), there is a trend observed where the higher the brittleness index is, the lower the resulting DBT depth (which is expected). To get a more accurate sense of the relationship between the brittleness index and the DBT depths, it is important to analyze the fracture toughness of the materials based on specific crack mechanisms (modes) that initiated the DBT. This however, is not the intent of this study.

![DBT Depth & Britteness Index](image)

**Figure 9.7: DBT depth versus brittleness index.**

An interesting observation derived from the data is that the top four materials (excluding Si) with the greatest DBT depth (sapphire, AION, spinel and AlTiC) all have a fair bit of aluminum (Al) in their composition. The ductility in Si could be attributed to the metallic phase (Si-II) formed during the high pressure phase transformation that
enhances its ductility (Si behaves like metal under high pressure conditions).

Finally, Figure 9.8 correlates the forces (cutting and thrust) at the DBT with the depths for all nine materials. In this case, an apparent coefficient of friction (COF), which is the ratio between the cutting and the thrust forces, is used.

![DBT Depth & Apparent COF](image)

**Figure 9.8: DBT depth versus the apparent COF.**

The overall trend here suggests that the apparent COF is higher for materials that exhibit a greater DBT depth. This is simply because as the applied load is increased (while still in the ductile regime), the resultant depth of cut also increases. This then causes the cutting force to increase, as the cutting forces are directly proportional to the depth of cut (i.e., larger depths yield in greater cutting forces).

9.3.5.2 DBT WITH LASER HEATING

The aim in this section is to compare the DBT depths with and without laser heating for the five selected materials of interest (as shown in Figure 9.9). Table 9.5 compares the DBT parameters with and without laser heating.
Table 9-5: DBT parameters for materials experimented with and without laser.

<table>
<thead>
<tr>
<th>Material</th>
<th>Room Temperature</th>
<th>Laser Heating</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>DBT Load*</td>
<td>DBT Cutting Force*</td>
</tr>
<tr>
<td>4H-SiC</td>
<td>85 mN</td>
<td>31 mN</td>
</tr>
<tr>
<td>6H-SiC</td>
<td>120 mN</td>
<td>48 mN</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>450 mN</td>
<td>230 mN</td>
</tr>
<tr>
<td>Silicon</td>
<td>175 mN</td>
<td>105 mN</td>
</tr>
<tr>
<td>Sapphire</td>
<td>650 mN</td>
<td>430 mN</td>
</tr>
</tbody>
</table>

It is seen that all cuts done with laser heating are able to withstand higher applied loads without exhibiting brittle fracture. This is the key to increasing the DBT depth of the material. The effect of laser heating is evident where greater applied loads can be applied, hence significantly increasing the DBT for all five materials. This is highlighted in Figure 9.9 where the DBT depth is compared for all five materials and both conditions: with and without laser heating.

![Figure 9.9: DBT depth comparison with and without laser.](image)

The most significant increase in DBT depth (~ 176%) is observed in the 3C polycrystalline SiC. This is believed to be due to two factors: (1) the 3C absorbs more than 95% laser radiation at these wavelengths (refer to Figure 6.2), and (2) the
polycrystalline cubic SiC structure has the highest fracture toughness value (among these five materials), which could be a reason for the reduced brittleness in the atmospheric phase observed during laser heating. Overall, the experimental results suggest that laser-assisted scratch tests are successful in enhancing the ductile response in all five materials.

9.3.5.3 MICRO-RAMAN SPECTROSCOPY OF SAPPHIRE

In this section, only the μ-Raman analysis carried out on sapphire is discussed, as μ-Raman analysis on Si and SiC have already been discussed in Chapters 5 and 6 respectively. Using a 442 nm (visible blue) He-Cd laser, a 1-2 μm spatial resolution is obtained for this analysis. Raman spectroscopy is used in this study to detect and analyze phase transformations for scratched regions and machined chips. *Figure 9.10* compares the Raman spectra for the cuts (with and without laser) and the machined chips collected for both conditions.
Figure 9.10: Micro-Raman spectra for single crystal sapphire: (a) machined region with two conditions compared to the unmachined surface, and (b) chips accumulated from the machining process.

Figure 9.10(a) shows that there is no change in the Raman spectrum between the machined (with and without laser heating) and the unmachined region. This does not mean that high pressure phases do not exist; it could also be an issue of the equipment resolution where the current probing depth of the visible laser (\(\lambda = 442\) nm) is too deep (probes below the high pressure region). Using a shorter Raman wavelength (e.g., 325 nm) may provide some insight to the high pressure phase transformation in sapphire. In Figure 9.10(b), the machined chips accumulated from both machining conditions show significant amorphous characteristics, which is consistent with past TEM work. Since no crystallinity is observed in these chips, it is believed that the chips are fully amorphous.
due to the back transformation process upon pressure release of the tool (as has been observed previously in other ceramics and semiconductors).\textsuperscript{281}

9.4 CONCLUSION

The DBT depths, along with other related parameters such as applied loads and cutting forces for nine different ceramics and semiconductors of interest have been determined and reported here. It is determined that no one particular mechanical property (hardness, fracture toughness, Young’s modulus and Poisson’s ratio) has a significant influence on the DBT depth for any of the materials experimented. These findings are reasonable as there are several other parameters such as the form of material (single crystal, polycrystalline or amorphous), grain size (if polycrystalline), mode of fracture (during the DBT), cutting direction with respect to the crystal/grain orientation, etc. that have to be studied in detail in order to correlate any particular material property to the DBT depth. However, there is a trend observed when the DBT depths are compared to the brittleness index where the higher the brittleness index, the lower the resulting DBT depth. It is necessary to have a consistent comparison between the hardness and fracture toughness for all materials. This is hard to achieve when analyzing the DBT depths due to several reasons. For example, the hardness obtained should only be for the atmospheric phase and not the high pressure phases for all materials. However, it is almost impossible to obtain a hardness value (from indenting or scratching) without forming a high pressure phase. Also, the fracture toughness values compared should be for the same crack system in all materials. This is hard to achieve due to the different forms and crystal structures of each material. Previous studies have reported a significant difference in cracking
mechanisms (modes) in comparing two materials: Si and SiC. However, correlating the material properties to the ductile response of each material was not the focus of this study.

Laser heating was successfully demonstrated as evidenced by the significant increase in the ductile response of five selected materials of interest (three SiC polytypes, Si and sapphire). Laser assisted (heating, thermal softening and reduced brittleness) material removal resulted in smaller cutting forces (when comparing the relative depth-to-cutting force ratio) and a larger critical depth of cut (DBT depth). Finally, Micro-Raman spectroscopy served as an important tool to characterize the amorphous remnant in the accumulated plastically deformed chips of sapphire (μ-Raman of Si and SiC are discussed in chapter 5 and 6 respectively). The results of this study will be extremely beneficial as guidelines in machining parameter selection and material selection for all SPDT experiments involving either of these experimented ceramics and semiconductors.

9.5 FUTURE WORK

A closer analysis on selected materials (Si and SiC) will be carried out. Single crystal substrates will be used to identify the fracture dependency along different cutting directions. An attempt will be made to identify different crack modes and correlate them to the corresponding fracture toughness value. Also, the DBT experiments will be repeated with and without laser heating with the use of cutting fluids, and the results will then be compared to the results reported in this chapter. Finally, the effect of laser heating on the remaining materials (quartz, AlON, AITiC and spinel) may be studied. This will
further widen the usability of the micro-laser assisted machining technique to a wider range of materials.
CHAPTER 10

RESEARCH CONCLUSION

10.1 CONCLUDING REMARKS

It is vital to fully understand the mechanism of ductility in brittle materials to determine the necessary and sufficient conditions to maintain ductile response during a machining process that could provide a dramatic improvement in the manufacture of brittle materials. Improved machinability contributing to better physical aspects (mechanical, optical and electrical), improved surface finish, reduced subsurface damage, high form accuracy, increased material removal rates and minimal tool wear will be particularly important in the semiconductor industry where a high quality of machined surfaces are most demanded. As a matter of fact, ductile regime machining has already been a key advancement in the development and improvement of ceramic and semiconductor manufacturing over the past decade. Understanding the mechanics behind ductile regime machining is necessary in avoiding any form of fracture that may potentially degrade the material’s surface. It is also important to understand the fracture toughness (fracture tolerance) in ceramics and semiconductors when attempting to machine them. Ceramics and semiconductors are extremely brittle materials, but can be machined at the nano/micro scales. The two most dominant properties that seem to affect the ductile response of the material are hardness (HPPT) and fracture toughness. A study is conducted (discussed in Chapter 9) to obtain the DBT depths of nine different ceramics
and semiconductors with a wide range of material properties such as hardness (9 GPa to 27 GPa), fracture toughness (0.91 MPa.m$^{0.5}$ to 4.3 MPa.m$^{0.5}$), Young’s modulus (73 GPa to 466 GPa), forms (single crystal, polycrystalline and amorphous) and Poisson’s ratio (0.14 to 0.27). It is determined that no one particular mechanical property (hardness, fracture toughness, Young’s modulus or Poisson’s ratio) has a significant influence on the DBT depth for either of the materials experimented. These findings are reasonable as there are several other parameters such as the form of material (single crystal, polycrystalline or amorphous), grain size (if polycrystalline), mode of fracture (during the DBT), cutting direction with respect to the crystal/grain orientation, etc. that have to be studied in detail in order to correlate any particular material property to the DBT depth. However, there is a trend observed when the DBT depths are compared to the brittleness index where the higher the brittleness index, the lower the resulting DBT depth. The results of this research further confirm that brittle materials can be machined in the ductile-regime and will be extremely beneficial as guidelines in machining parameter selection and material selection for future single point diamond turning (SPDT) experiments involving these ceramics and semiconductors.

In preliminary work done in this study (discussed in chapter 3), ductile mode SPDT had been carried out on (1) single point diamond turned 6” diameter CVD-SiC, (2) single point diamond turned quartz, and (3) laser ablated and single point diamond turned CVD-SiC. However, previously, only the surface finish/conditions have been analyzed. In this study, however, the subsurface conditions of these machined samples are investigated (discussed in Chapter 3). Optical microscopy (only for transparent samples like quartz), laser Raman spectroscopy, scanning acoustic microscopy and micro-Raman
spectroscopy were successful NDT techniques utilized to investigate subsurface damage in single point diamond turned ceramics and semiconductors. All subsurface damage techniques showed complementing results where no signs of brittle material removal was detected. The subsurface damage analysis study concludes that SPDT was successful in improving the surface of SiC and Quartz without causing any form of surface and subsurface damage.

The major content of this research focuses on increasing the material removal rate in the ductile regime by further enhancing the ductility in brittle materials. In past research, it has been demonstrated that ductile regime machining of semiconductors and some ceramics is possible due to the high pressure phase transformation (HPPT) occurring in the material caused by the high compressive and shear stresses induced by a single point diamond tool tip. To further augment the ductile response of nominally brittle materials, traditional single point scratch tests are coupled with a micro-laser assisted machining (µ-LAM) technique. In this study the effects and benefits of laser heating and subsequent material softening on the material removal of Si, SiC and sapphire (Chapter 9) are studied by means of single point scratch testing. The effect of laser heating on the ductile-to-brittle transition (DBT) of the material along with the optimization of machining parameters, such as applied load, depth of cut, cutting force, cutting speed, laser wavelength and laser power, is also studied. The effect of laser heating on the ductile mode material removal process of four different semiconductors has been studied in detail (Chapters 5, 6 and 7): single crystal Si, single crystal 4H-SiC, single crystal 6H-SiC and polycrystalline CVD coated cubic SiC.
Micro-laser assisted scratch tests were successful in demonstrating the enhanced thermal softening in Si and SiC (all three polytypes) resulting in greater depths of cuts (when compared to similar applied loads for cuts with no laser) and greater ductile-to-brittle transition depths. Laser assisted (heating, thermal softening and reduced brittleness) material removal resulted in greater depths of cuts at less applied thrust forces, smaller cutting forces and a larger critical depth of cut. Force analyses (thrust and cutting), optical microscopy, white light interferometry, electron microscopy and Raman spectroscopy served as useful analyses (measurement and characterization) methods to detect the enhanced ductile response and reduced brittle fracture as a result of preferential material heating. Results obtained from this study are promising to further implement the $\mu$-LAM technique in machining operations such as SPDT. Lower cutting forces obtained from the $\mu$-LAM process are favorable to minimize tool wear while machining abrasive ceramics/semiconductors such as SiC. No tool wear was detected in these tests due to small machined areas (short track length covered by the tool), however, more testing will be performed in the future to machine a larger area for tool wear analysis. The results from this study also will benefit the manufacture of brittle materials as laser heating is proven to decrease the brittle response in ceramics and semiconductors, which can result in high productivity rates (i.e. higher material removal rates). The effect of laser heating was also studied on single crystal Sapphire where the DBT depth was successfully increased as a result of thermal softening (Chapter 9).

To further understand the thermal softening effect on the ductile response in semiconductors (in this case Si), $\mu$-Raman spectroscopy and indentation tests are implemented (Chapter 7). Micro-Raman spectroscopy was successful in showing
evidence of the formation of the metallic phase (Si-II), based upon the back transformed phases observed such as Si-III, Si-IV, Si-XII and amorphous Si, implying that all material removal was in the ductile regime. The formation of the metallic phase is necessary for a ductile mode machining process as the material can be easily removed without damaging the underlying hard semiconductor (Si-I). It is also determined that by using optimized laser power conditions when machining Si, the resultant cut is the deepest cut (for similar applied loads) and leaves behind a “damage-free” or “cured” Si-I diamond structure, similar to that of the original surface. In other words, no high pressure phases are retained when optimum laser powers are used during the material removal process. From a manufacturing point of view, this is beneficial, as the material is believed to have returned (annealed) to its original phase (Si-I) despite undergoing the ductile mode material removal/HPPT process. This is a significant advantage in semiconductor processing as polishing the wafer to remove subsurface damages (amorphous and phase transition layer) consumes the majority of the time and cost in producing mirror-finish parts. The results from the indentation tests were successful in isolating the pressure and temperature effects to confirm that the laser is preferentially heating only the high pressure phases (and not the atmospheric, Si-I phase). The results from this study will also benefit the manufacture of brittle materials as laser heating and thermal softening is demonstrated to decrease the brittle response in ceramics and semiconductors, thereby providing higher productivity rates (i.e., higher material removal rates) by allowing a greater depth of cut without fracturing the material.

From the three polytypes of SiC studied, the polycrystalline 3C-SiC is found to be the least brittle (greatest DBT depth). The reason for this is also studied in this research.
Two different techniques, x-ray diffraction and orientation imaging microscopy, are implemented to study the effect of crystal orientation on the ductile response of 3C-SiC. It is found from this study that the two main factors that contribute to the enhanced ductile response of the 3C polycrystalline SiC are: (1) polycrystalline 3C-SiC has a significantly higher fracture toughness than any of the single crystal SiC materials, and (2) the <111> direction (which is the direction of the material in this research) in 3C-SiC has that highest fracture toughness value irrespective of the source of the material. This is believed to be due to the available grain boundaries (grain sizes in the Poco Graphite 3C-SiC averaged between 20–30 µm) in the polycrystalline material that impedes dislocation movement, hence limiting fracture/crack growth. Since the lattice structure of adjacent grains differs in orientation, it requires more energy for a dislocation to change directions and move into the adjacent grain. The grain boundary is also much more disordered than inside the grain, which enhances the toughness and increases the resistance to crack propagation.

Finally, a successful and sensitive enough AE system has been developed to detect crack initiation and fracture behavior during the material removal of ceramics and semiconductors (Chapter 8). In this study, two different band-pass filter ranges (2 kHz - 200 kHz and 100 kHz - 2 MHz) have been identified to detect different crack patterns such as lateral, median, radial and voids caused by fracture at the leading edge of the tool. The spikes/peaks in the AE spectra correlate well with the cracks along the scratches, enabling the detection of fracture behavior during cutting. The cutting forces (instabilities in forces observed during severe fracture) also complemented the AE spectra indicating critical fracture along the cuts, however, AE proved to be a much more sensitive
technique to detect crack initiation and fracture behavior during the cutting process. It is also determined that in order to monitor more than one type of crack/fracture event, at least two sensors with different band-pass filters need to be utilized, as different cracks are detected in different frequency ranges.

In general, there are several machining parameters such as depth of cut, applied load, cutting speed, feed and laser power, that can be adjusted in order to increase the material removal rate (MRR). However, in applications such as precision machining, there are often tradeoffs when increasing the MRR. For example, an increase in feed can reduce the total machining time (increase material removal rate) and also reduce tool wear; however, a higher feed rate also results in higher surface roughness. On the other hand, the trade-off at lower feeds is that the measured tool wear is much more than at higher feeds due to the longer track length covered by the tool at lower feeds (i.e., the MRR is less but the resultant surface finish is better compared to higher feeds). The key to a successful and productive ductile mode machining brittle materials is understanding the material removal mechanics and optimizing the processing parameters. The findings in this research will lead to development of improved machining technologies by utilizing the ductile mode material removal mechanism and reduced brittle mode material removal in the manufacture of ceramics and semiconductors.

10.2 FUTURE WORK SUMMARY

Single point diamond turning (SPDT) experiments on Si and SiC will be carried out on a commercial diamond turning machine (DTM) using the µ-LAM setup. For the SPDT experiments, several machining parameters such as cutting speed (changes as a
function of workpiece radius), depth of cut and feed, and laser power (assuming the 100 W laser is used) will be experimented and optimized. The ultimate goal for the SPDT experiments is to improve the surface finish of an as-received SiC workpiece with minimal or no tool wear (which was not verified herein due to the limited length or track length of cut). The micro-laser assisted-SPDT parameters such as depth of cut, feed, cutting speed and laser power will then be optimized to achieve the maximum material removal rate while improving the surface finish.

Micro-Raman spectroscopy will be performed on the cuts carried out to identify the DBT and also on the SPDT machined surfaces. For the scratch performed to identify the DBT, three different regions will be analyzed: the ductile region, the transition region and the brittle region. The spectrums of all three regions will be compared and possible new peaks (expected to be generated in the phase transformed region/chips) will be identified as evidence of ductile mode machining. Besides identifying new peaks or a shift in peaks, broadband peaks or amorphous broadening of the crystalline peaks is also expected in the machined surface (ductile mode material removal).

The second characterization technique that will be utilized in order to confirm the occurrence of ductile mode machining (due to the HPPT) is transmission electron microscopy (TEM). TEM analysis will be carried out on the machined chips (debris) which are plastically deformed during the ductile material removal process. These chips have been collected after each experiment (with different machining parameters) and will continue to be collected for characterization purposes. The accumulated chips are usually visible at the end of the cut/scratch or stuck to the diamond tool tip. The existence of the amorphous remnant in the chips is substantial evidence of ductile mode machining. TEM
analysis done by past researchers on chips from SPDT operations show both the amorphous region and crystalline structure indicating phase transformation (crystalline material transformed into an amorphous structure) due to significant plastic deformation during the material removal process. Finally, scanning acoustic microscopy (SAcM) will be performed on the laser machined samples to measure and image subsurface damage such as thermal cracks (refer to Chapter 3).

Laser heated diamond anvil cell (DAC) and rotary diamond anvil cell (R-DACR, to introduce the shear component) to simulate actual laser heating and cutting conditions will be performed at Argonne National Laboratories. Laser heated DAC tests have previously been conducted on 4H and 6H SiC (but not on 3C). The high-pressure phases will be identified and retained for optical characterization purposes. The goal for optical characterization tests is to get the optical properties of the high-pressure phases for all three polytypes, to be able to choose a suitable laser wavelength during the machining process.
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APPENDIX A
SUPPLEMENTARY INFORMATION – CHAPTER 5
Figure A1: 200 kV JEOL JEM-2010-F TEM
Figure A2: EDS of machined Si chips obtained with no laser heating
Power Density Calculation:

In this study, a surface power density analogy is used:

\[
\text{Power Density (PD)} = \frac{[\text{Laser Output Power (W)}]}{[\text{Beam Area (cm}^2)]}
\]

For a 10 μm beam diameter, the beam area is:

\[
\text{Beam Area} = \pi r^2 = \pi (0.001)^2 \text{ cm} = 3.142 \times 10^{-6} \text{ cm}^2
\]

Sample calculation:

Laser Power Output = 9 W
Cutting Speed = 2 μm/sec = 0.0002 cm/sec

\[
\text{PD} = \frac{[9 \text{ W}]}{[3.142 \times 10^{-6} \text{ cm}^2]} = 2.86 \text{ MW/cm}^2
\]

Now incorporating the speed parameter to obtain surface power density per unit length (per second)

\[
\text{PD/Length of Cut Exposed in a Second} = \frac{2.86 \text{ MW/cm}^2}{0.0002 \text{ cm}} = \textbf{14 GW/cm}
\]
Figure A3: NanoIndenter XP (MTS) with Berkovich tip
Figure A4: Image showing melted Silicon (due to excess laser heating) with surface profile
APPENDIX B
SUPPLEMENTARY INFORMATION – CHAPTER 7
Hardness Calculation from Indentations:

The hardness of the material is estimated/calculated based on the residual indentation area measure using a white light interferometer.

\[
\text{Hardness (H)} = \frac{\text{Maximum Applied Load (P}_{\text{max}})}{\text{Residual Indentation Area (A}_{r})}
\]

Residual Indentation Area (A_r):

Assuming an ellipse based on the microscope images

\[
A_r = \pi a b
\]

Sample hardness calculation:

P\text{}_{\text{max}}: \ 300 \text{ mN} = 0.3 \text{ N}

Measured Indent Dimensions: a = 1.7 \mu m \ & b = 33 \mu m

\[
A_r = \pi (1.7 \times 10^{-6}) (33 \times 10^{-6}) = 1.762 \text{ m}^2
\]

Hardness (H) = (0.3 \text{ N}) / (1.762 \text{ m}^2) = 1.70 \text{ GPa}
APPENDIX C

SUPPLEMENTARY INFORMATION – CHAPTER 8
Figure C1: AE amplifier with two band-pass filters

*Note: Red arrows show the contact points between the filter and amplifier*
Figure C2: Force data (cutting and thrust) plotted along with the AE RMS amplitude for a brittle cut performed on Si.

*Note: At this point, no AE spectrum is shown for ductile-DBT regions as the AE sensor is not sensitive enough to show any noticeable changes in the spectrum (only sensitive when fracture occurs).*
APPENDIX D

SUPPLEMENTARY INFORMATION – CHAPTER 9
Figure D1: Cuts performed on AlON with varying loads
Figure D2: Cuts performed on Quartz with varying loads
From the charts in Figure 9.6, there is no specific trend or pattern observed that indicate the dominance of any one material property on the DBT depth. The only observation that can be made here is that the three materials that have the highest hardness values (4H, 6H and 3C-SiC) yield in the lowest DBT depths (from Figure D3 (c)).
Critical Depth of Cut Relationship:
In order to evaluate the influence of material properties on the ductile-to-brittle transition rate, a broad range of ceramics were chosen for grinding. By comparing the ductile response to the material properties during grinding, a critical depth of cut model had been established. The critical depth model originates from a formula describing the critical depth of fracture during indentation of hard materials (proposed by Lawn, Jensen and Aurora in 1976). Based on the Griffith fracture propagation criterion, the critical depth of penetration \(d_c\) is represented as:

\[
d_c = \frac{E R}{H^2}
\]

where \(E\) is the elastic modulus, \(R\) is the fracture energy and \(H\) is the material hardness. In general, for materials that exhibit a plastic zone near the crack tip, the value of \(R\) can be evaluated using Griffith’s classical crack propagation analysis. In order to define fracture energy at small scales, it can be represented by a dimensionless analogous measure of energy to propagate cracks:

\[
R \sim \frac{K_c^2}{H}
\]

In 1986, Marshall and Lawn defines the quantity \(K_c^2 / H\) as the effective measure of brittleness. Combining this quantity to the critical depth model proposed in 1976 yields in a ductile-to-brittle transition depth measure:

\[
d_c \sim \left(\frac{E}{H}\right) \cdot \left(\frac{K_c}{H}\right)^2
\]

This relationship has shown a remarkable degree of consistency in predicting critical indentation depths and grinding in-feeds (depths). This relationship was extensively verified by grinding experiments by Scattergood and Bifano in 1988 and a dimensionless constant of 0.15 was introduced to compensate for tool geometry errors.
\[ d_c \sim 0.15 \left( \frac{E}{H} \right) \left( \frac{K_c}{H} \right)^2 \]

However if the right hardness (hardness of the brittle phase) and fracture toughness (the exact mode of fracture as obtained during the experiments) values are used, this dimensionless coefficient is not required. It is important to note that this relationship is not suitable for materials that exhibit a significant variation in fracture toughness with varying indentation depths.

A sample hardness calculation for 3C-CVD-SiC used in Chapter 9 is shown.

\[ d_c \sim \left( \frac{E}{H} \right) \left( \frac{K_c}{H} \right)^2 \]

E = 466 GPa (from literature)

\[ K_c = 3.00 \text{ MPa.m}^{0.5} \text{ (from literature)} \]

H = Unknown

\[ d_c = 42.5 \text{ nm (obtained from experiments)} \]

Substituting the values:

\[ (42.5 \times 10^{-9}) \sim \{ (466 \times 10^9) / (27 \times 10^9) \} \cdot \{ (K_c) / (27 \times 10^9) \}^2 \]

\[ H = 46.2 \text{ GPa} \]

It is important to note that the critical depth of cut \((d_c)\) of 3C-SiC obtained from a scratch test (discussed in Chapters 6 and 9) is significantly smaller than the maximum machining cutting depth reported in Chapters 3 and 4 (42.5 nm versus 1.3 \(\mu\)m). This phenomenon can be explained by the schematic in Figure D4.
Fracture damage generally initiates at the critical depth of cut ($d_c$) and continues upwards along the tool nose to the top of the uncut shoulder. It is important to note that fracture damage can occur in ductile-regime machining, however, the requirement being that it does not propagate below the finished surface. These fracture damages will be removed by every subsequent cut in a machining process, allowing a final machined depth (which is a function of feed) that is significantly greater than the critical depth of cut ($d_c$). In conclusion, as long as the fracture damage does not replicate below the finished surface, a fracture-free surface is obtained and ductile-regime machining conditions are achieved.
APPENDIX E

PUBLICATIONS AND STUDY RELEVANCE
Publications and Study Relevance:

This section lists my publications and the relevance of each paper to specific parts of this study/dissertation.

   - Reports the indentation experiments performed to isolate the temperature and pressure effect that occurs simultaneously during the μ-LAM process. The paper also talks about the preferential heating process and the phase that benefits most from the laser irradiation process (relates to Chapters 5 and 7).

   - This paper discusses the single point cutting tests (scratch tests) performed on Si and 4H-SiC to optimize the μ-LAM parameters. The results demonstrate the enhanced ductile response in these nominally brittle materials from the thermal softening effect (relates to Chapters 5, 6 and 7).

   - Reports the machining process and optimized machining parameters between the hybrid laser ablation-SPDT processes discussed in Chapter 3. This paper also reports the results obtained from the subsurface damage analysis (using the scanning acoustic microscope).

   - Discusses the laser heating effect on single crystal Silicon (comparing no laser power, minimum laser power and optimized laser power conditions). The high-pressure phases and the annealing effect (from the optimized laser power) are also reported along with relevant micro-Raman spectra (discussed in Chapters 5 and 7).
   - Reports the SPDT process and optimized machining parameters in machining Quartz (relevant to Chapter 3). This paper also reports the results obtained from the subsurface damage analysis (using the scanning acoustic microscope).

   - This book chapter provides detail information on the μ-LAM process along with its benefits and success with Si and SiC (relates to Chapters 5, 6, 7 and 9).

   - This book chapter provides detail information on the origins and progress/development in ductile mode machining (DMM). The science behind DMM along with supporting experimental results is discussed. (relates to all chapters in the dissertation).

   - This paper reports the enhanced ductility (reduced brittleness) of single crystal Si and SiC as a result of thermal softening (relates to Chapters 5, 6, 7 and 9). A detail study comparing applied loads, resulting depth of cuts, cutting forces and laser powers are reported.

   - This paper reports the enhanced ductility (reduced brittleness) of single crystal Si as a result of thermal softening (relates to Chapters 6 and 9). A detail study comparing applied loads, resulting depth of cuts, cutting forces and laser powers are reported.

    - Reports the SPDT process and optimized machining parameters in machining Quartz (relevant to Chapter 3). This paper also reports the results obtained from the subsurface damage analysis (using the scanning acoustic microscope).
   - This paper reports the enhanced ductility (reduced brittleness) of single crystal Si as a result of thermal softening (relates to Chapters 5, 7 and 9). A detail study comparing applied loads, resulting depth of cuts, cutting forces and laser powers are reported.

   - The paper presents all SPDT operations and subsurface damage analysis performed on Quartz and CVD coated SiC. The content of this paper correlates to the work discussed in Chapters 3 and 4.

   - This paper reports the enhanced ductility (reduced brittleness) of single crystal 4H-SiC as a result of thermal softening (relates to Chapters 6 and 9). A detail study comparing applied loads, resulting depth of cuts, cutting forces and laser powers are reported.

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   - This thesis provides detail information on the origins and progress/development in ductile mode machining (DMM). The science behind DMM along with supporting experimental results on Quartz and CVD-SIC is discussed. (relates to all chapters in the dissertation).

- This paper reports a detail study performed on 4H-SiC to indentify the ductile-to-brittle transition of the material using the Nanocut II device. AFM and white light profilometry was used to study the depth profile and cut surface. (relates to Chapters 6, 8 and 9).