INVESTIGATION OF THE FRACTURE BEHAVIOR
OF cBN-GLASS COMPOSITES

A Thesis in
Materials Science and Engineering

by

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ABSTRACT

Samples of cBN-borosilicate composites were synthesized and mechanically failed to create fracture surfaces for characterization of the crack propagation. The cBN crystals were also oxidized to determine differences in fracture toughness and crack propagation due to modification of the cBN-glass interface.

The diametral compression test, using a single-edge V-notch, was employed to measure the fracture toughness and create the fracture surfaces for study. This test is attractive because it can be used for brittle materials without loading or gripping difficulties, and the notch is relatively easy to machine into the samples. The fracture toughness for borosilicate glass was found to be 0.56MPa√m. Toughness increased with increasing volume fraction up to 40vol% to a value of 1.41MPa√m for a 40vol% cBN composite, an overall increase of 152%. The addition of oxidized cBN increased the fracture toughness to 1.28MPa√m for a 40vol% oxidized cBN composite, an overall increase of 129% from the borosilicate glass.

SEM images were taken of the fracture surfaces to characterize the crack path through these composites. Two types of direct interactions between the crack and embedded particles were identified based on matched image pairs of the fracture surfaces: transgranular particle failure and interfacial failure.

Digital image analysis and SEM images were used to quantify the areal density of cBN on the fracture surfaces to determine the frequency of crack particle interaction with the cBN crystals. It was found that for the cBN-borosilicate glass composite, where the CTE_{cBN} > CTE_{glass}, the crack preferentially propagates through the glass matrix due to local compressive stresses surrounding the cBN crystal.
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Chapter 1

INTRODUCTION

cBN and diamond are the hardest known materials and are widely used for grinding applications. Both materials belong to a category in grinding applications called ‘super abrasives’ due to their physical and chemical properties. cBN was first synthesized in 1959 under the General Electric trade name, Borazon. cBN and diamond are similar in that they share the same crystallographic structure (cubic and hexagonal close-packed) and have high values of thermal conductivity. There are a few key differences between the two materials that distinguish their use in grinding applications.

Diamond oxidizes above 600°C and backconverts to graphite above 750°C. This imposes critical limitations on the use of diamond in grinding applications. Upon heating, diamond reacts readily with carbide-forming elements such as iron, cobalt, and nickel. cBN is more resistant both to oxidation and to phase transformation into hBN. cBN is chemically inert with iron-, cobalt-, and nickel-base alloys compared to diamond, and because of this, cBN fills an important gap in grinding applications.

Grinding wheels consist of two primary components: the abrasive material (i.e., cBN, diamond, or silicon carbide) and the bonding adhesive (vitreous, resin, metal, and
electroplated bonds). The porosity of the grinding wheel is also an important component and serves to introduce coolant into the wheel and remove abrasives that have dulled.

Glass bonded cBN grinding wheels are generally used in applications such as cutting tool sharpening and production grinding, especially in the automobile industry. The brittle bonding phase gives the grinding wheel good self-sharpening properties, making conditioning of the wheel simpler, but makes the wheel unsuitable for high-impact and heavy pressure operations.\(^1\)

The difference in hardness of glass and cBN also presents an engineering challenge when designing a wheel. The interfacial bond between cBN and glass becomes important to the life of a grinding wheel. If the bond between cBN and glass is too strong, new cBN crystals will not be exposed. If the bond between cBN and glass is too weak, the efficiency of the grinding wheel will be diminished. Residual stress effects due to thermal expansion mismatch between particulates and matrix have been shown to affect the bonding between particulate-matrix and this also applies to glass bonded cBN grinding wheels.

The investigation of cBN-glass composite fracture behavior will involve studying fracture surfaces created under controlled conditions. The composites will be mechanically failed using the diametral compression test with a single edge V-notch. The samples will be fabricated with varying volume fractions and fracture toughness
values for the composites will be reported. The cBN crystal surfaces will be modified to grow oxide, and thereby, to influence the cBN-glass interface in the formed composite.

The fracture surfaces will be examined using scanning electron microscopy in order to gather information about the fracture behavior. By examining and comparing both fracture surfaces of a specimen, evidence of the types of crack particle interaction that occurred during fracture can be determined. The frequency of crack interaction with the particles in this composite will be quantitatively determined. By using digital image analysis software, the areal density of the particles on those fracture surfaces can be used to determine how often the crack interacts with the particles. Based on these findings, thermal expansion mismatch and elastic mismatch between cBN and borosilicate glass will be considered to determine any correlations to this and fracture behavior.

This thesis is presented in five chapters. Chapter 2 presents background and theory relevant to this study including a review of diametral compression theory and testing using a single-edge V-notch, toughening mechanisms in glass ceramic composites, and the use of digital image analysis and SEM images to collect quantitative information. Chapter 3 presents the experimental procedure which includes processing of the samples, their mechanical testing, and the analytical techniques and software packages used to investigate the fracture behavior of these composites. Chapter 4 presents the results and discussion which includes the observations made on the fracture surfaces as well as the procedure developed to quantify cBN content on the fracture
surfaces using the image processing software. Finally, Chapter 5 summarizes the outcomes of this thesis and offers suggestions for future work.
Chapter 2

LITERATURE REVIEW

2.1 Introduction

The literature review is divided into three sections. The first section will discuss the theory and background behind the diametral compression test followed by the use of diametral compression test using a single-edge V-notch to measure fracture toughness. The second section reviews theories related to toughening mechanisms in particulate composites and will discuss studies pertaining to glass ceramic composites with oxide and non-oxide inclusions and the toughening mechanisms identified. A brief review of work performed on cBN-glass systems will also be discussed and this section will end fracture surface roughness measurements used to identify toughening mechanisms. The third section of this literature review will discuss studies that have used digital image analysis software and SEM images to gather quantitative information.

2.2 The Diametral Compression Test

2.2.1 Diametral Compression Theory

The diametral compression test was first introduced by Carniero and Barcellos\textsuperscript{2} in 1953. They applied the test to measure the tensile strength of concrete. It is an attractive
test because it allows the tensile testing of brittle materials without loading and gripping difficulties, and the samples are easy to produce.

The diametral compression test involves the loading of a cylindrical sample along its diameter such that tensile stresses are created perpendicular to the load, as shown in Figure 1.

The sample can fail in one of three ways: tension, compression, or crushing near the points of loading. For diametral compression testing, tension is the desired choice of failure for a sample. Because ceramics are much stronger in compression than in tension, failure due to compressive stresses can be ignored. Crushing of the sample and plastic deformation near the contact points is a concern. But, contact stresses have been shown to be minimized by using bearing strips to distribute the load over a portion of the surface of the sample. These bearing strips act as cushions and are placed between the sample contact points and mechanical tester before testing. For small specimens, items such as cardboard, aluminum foil or other materials that will conform to the sample shape upon application of the load from will suffice.
Figure 1: Diagram of the tensile stresses, $\sigma_{\text{tens}}$, created by load $P$ in the diametral compression test.

Figure 2: Diametral compression test under distributed load.
Hondros developed an exact theoretical stress analysis for the case of a pressure, $P$, applied over two diametrically opposite arcs of angular $2\alpha$. Referring to Figure 2, Hondros showed that the stresses along the $y$-axis can be described by the following equations:

\[
\sigma_x = \frac{2P}{\pi} \left[ \frac{(1 - r^2/R^2) \sin 2\alpha}{1 - 2 (r^2/R^2) \cos 2\alpha + r^4/R^4} - \tan^{-1} \left( \frac{1 + r^2/R^2}{1 - r^2/R^2} \right) \tan \alpha \right]
\]

\[
\sigma_y = \frac{-2P}{\pi} \left[ \frac{(1 - r^2/R^2) \sin 2\alpha}{1 - 2 (r^2/R^2) \cos 2\alpha + r^4/R^4} + \tan^{-1} \left( \frac{1 + r^2/R^2}{1 - r^2/R^2} \right) \tan \alpha \right]
\]

where $\sigma_x$ is the horizontal stress acting along the $y$-axis, $\sigma_y$ is the vertical stress acting along the $x$-axis, $P$ is the applied load, $2\alpha$ is the angle of the disk center to the edges of the load distribution, $r$ is the radial position, and $R$ is the total radius. The largest horizontal tensile stress is found at the center of the disk (where $r = 0$), and is of magnitude

\[
\sigma_{tens} = \frac{2P}{\pi} \left( \frac{\sin 2\alpha}{\alpha} \right)
\]
2.2.2 Diametral Compression Test using a single-edge V-notch for Toughness Testing

Fracture toughness can be determined by introducing an artificial crack into a specimen, which is then loaded to failure. It is important to ensure that the tip of the artificial crack is sharp enough to simulate natural cracks that form in the material. A few different methods have been developed and analyzed for fracture toughness testing by diametral compression. The main differences in these fracture toughness studies are the pre-cracks introduced in the samples. These include a through-notch\textsuperscript{7-10}, a chevron-notch\textsuperscript{11-14}, a Vickers\textsuperscript{15} or Knoop\textsuperscript{16} indentation, a radially edge-cracked disc\textsuperscript{17}, and a single edge notch.\textsuperscript{18-20} Some notching techniques can be challenging and sample dimensions can be a factor. Internal notches and chevron notches require precise machining, and chevron through-notches cannot easily be applied to thin samples.

Fracture toughness via diametral compression with a single edge notch was first introduced by Szendi-Horvath.\textsuperscript{18} His important findings were that sample size and notch depth had no effect on the measured toughness. He also found that the crack was found to be initiated in the center of the disc at the bottom of the groove. After initiation, the crack progressed through the thickness while spreading in the direction of the rim until it was arrested by the compressive field.

For a semi-infinite plate with an edge-crack subjected to tension, the toughness is given by
where \( \sigma_f \) is the failure stress and \( c \) is the notch or crack depth. Combining Eq. 3 and 4 gives,

\[
K_{IC} = \sigma_f Y \sqrt{c} = 1.12 \sigma_f \sqrt{c}
\]

The toughness for any elastic, brittle material tested in diametral compression with a single-edge notch should follow Eq. 5 and is easily calculated from the peak load, diameter, thickness, initial crack length, and load distribution angle, all of which is measureable. The load distribution angle is gathered by measuring the size of the indentation left on the bearing strips.

Damani et al.\textsuperscript{21} found that “the notch width must be of the order of the size of the relevant microstructural or machining-induced defects (e.g. large pores and weak grain boundaries)” to produce accurate toughness measurements. Thus, for the measurement of fracture toughness testing, it is important to have a sharp crack front.

A single-edge notch is relatively simple to machine and is applicable to small or thick samples. A “V-notch” can be machined into the bottom of the single-edge notch enhancing the stress concentrations at the bottom of the “V” to create a sharp crack front. The “V-notch” has been shown to produce a very sharp notch that produces extreme stress concentrations at the bottom of the tip, effectively creating a sharp crack front.
There are two popular ways to produce a V-notch into a sample. A few studies have machined the V-notch by using a grinding wheel with a V-shaped edge (most wheels are round and flat).\textsuperscript{22,23} Another method is to sharpen an existing notch using a razor blade sprinkled with diamond paste.\textsuperscript{24-26}

Clobes and Green\textsuperscript{26} validated the use of a single-edge V-notch for diametral compression test to measure the fracture toughness of porous, fine-grained alumina samples. It was found that the fracture toughness data for their V-notched specimens correlated well with previously published data from other authors that used other notching methods such as a Chevron notch and indentation method, to measure fracture toughness of porous alumina. They further concluded that this test can be used for toughness testing of other brittle systems. The fracture toughness measurements for the experiments presented in this thesis were gathered from samples tested via diametral compression test with a single-edge v-notch.
2.3 Toughening in Glass Ceramic Composites

2.3.1 Toughening Mechanisms in Particulate Composite Systems

The primary toughening mechanisms of particulate composites have been attributed to crack bowing and crack deflection, as illustrated in Figure 3, and crack bridging by ductile particulates. Secondary mechanisms attributed to toughening have been the residual stresses due to thermal expansion mismatch between the particulates and matrix and the grain size.

Crack bowing occurs when the crack bypasses the obstacle by propagating around either side of it. In 1970, Lange\textsuperscript{27} analyzed the energy associated with crack tip...
interaction with second phase particles at the crack front. From microstructural observation, he found that the crack would increase its length by bowing between obstacles. The increased crack length due to crack bowing contributed significantly to the fracture energy. A value for “line-energy” per unit crack length, $T$, was estimated by dividing the elastic strain energy associated with a semicircular crack, by its length. Fractographic evidence for crack bowing has been observed and summarized by Green.\textsuperscript{30,31} In a later study, Lange proposed that the toughening is strongly related to the particle spacing and provided direct evidence in glass/Al\textsubscript{2}O\textsubscript{3} composite.\textsuperscript{28} The importance of obstacle spacing on the toughening contribution was also studied by Evans in 1972.\textsuperscript{29} He found that from stress calculations, the strength of the composites is dependent on the ratio of the obstacle dimension.

In 1983, Faber and Evans,\textsuperscript{32,33} proposed a crack deflection model to predict the toughness of ceramics reinforced with particulates. The principle behind crack deflection is that the crack prefers to travel along the regions of lowest fracture energy. In composites with inclusions, the cracks constantly twist and tilt around the inclusions to follow the path of lowest energy. Since the crack’s geometry is not a straight line, effective toughening will occur because of the increased distance the crack travels. They further found that by evaluating the strain energy release rate, $G$, of a deflected crack, the toughening increment is dependent on the morphology and the volume fraction of the particles. They also found the major contribution to toughness increment was from the twist portion of the crack, not the initial tilt. Microstructural evidence of crack deflection has been observed in many particulate composites\textsuperscript{34-37} and whisker composites.\textsuperscript{38-41}
Crack bridging occurs when the obstacle is left as a bridging ligament behind the crack tip. There are two types of crack bridging. In one case, the bridges are being pulled out of the matrix and have been observed especially in monolithic ceramics\textsuperscript{42} and fiber and whisker reinforced composites.\textsuperscript{43-48} In the other, the bridges are ductile and fail by a plastic deformation process.\textsuperscript{49} These ligaments will make it more difficult to open the crack and leads to an increase in toughness.

The Lange model for crack bowing ignores the effect of a particle stress field in a composite. Since then, the stress field that exists when elastic or thermal mismatch exists between the particle and the matrix has been shown to affect and contribute to toughening.\textsuperscript{50-57,65} This thermal residual stress field in a particulate-reinforced composite is induced by the mismatch in the coefficients of thermal expansion of the matrix and the particulate when the composite is cooled from the processing to room temperature.

Elastic mismatch has also been found to influence the local crack velocity on approaching an inclusion.\textsuperscript{50,51} It was found that the presence of elastic mismatch between inclusion and matrix alters the path of the crack front to avoid the particles. Toughness improvement only results when weak interfacial bonding exits. It has also been shown that for a composite with no elastic or thermal mismatch, no stress concentrations exist and a crack propagating is undisturbed on approaching the inclusion.\textsuperscript{50} In fact, a large increase in toughness occurs because the low elastic and residual stress levels promote effective crack-particle interaction within the composite.\textsuperscript{58}
It has been shown in various studies that introducing inclusions or particulates to glass systems increases their crack resistance thereby increasing fracture toughness. Crack deflection seems to be the dominant toughening mechanisms for glass composites with particulate inclusions\(^{60,62,63}\), but other mechanisms such thermal\(^{65}\) and elastic\(^{52}\) mismatch have also been identified.

### 2.3.2 Nonoxide Particle-Glass Composites

Wadsworth et al.\(^{60}\) studied the effects of adding SiC of various morphologies, whiskers, platelets, and particles, into cordierite glass. For all morphologies, fracture toughness was improved with increasing volume content of SiC. 30vol% of SiC whiskers showed the most improvement. For particles, fracture toughness improved by 37% but only up to 20vol% SiC addition.

By examining SEM images, crack deflection in these composites was readily observed. Crack bridging by particles (1\(\mu\)m) in diameter was not expected because debonding of the interface would allow the crack to circumvent the particle. Contribution of load transfer was minor and the low aspect ratio of particles would not be expected to be sufficient to allow buildup of significant contribution to fracture toughness. The authors explain that since the maximum fracture toughness increment reached 20vol% SiC, crack deflection was the only mechanism.
Chen et al.\textsuperscript{62} studied the effects of adding AlN powders in cordierite glass. The composites were hot pressed and the relative density decreased with increasing AlN. The relative density sharply decreases when content exceeds 30vol\% AlN. The researchers attributed this to constrained sintering and explained that above 30vol\% AlN, unsintered AlN would have formed a high viscous and rigid network resulting in a lower relative density.

Flexural strength data was collected using three point bend samples. The flexural strength of the composites increases from 0 to 40vol\% AlN. The increase in strength was attributed to load transfer from the cordierite matrix to the AlN particles due to good interfacial bonding between the two.

Fracture toughness was measured using micro-indentation method using a Vickers hardness tester. Fracture toughness increases up to 40vol\% AlN by 139\% from 1.27\text{MPa}\sqrt{\text{m}} for pure cordierite glass to 3.05\text{MPa}\sqrt{\text{m}} indicating good toughening effect on AlN particles. It was concluded that the thermal mismatch between the particles leads the AlN particles to introduce residual stresses in the composites, thereby increasing its strength.
2.3.3 Oxide-Particle Glass Composites

Boccaccini et al.\textsuperscript{63} studied the toughening effect of alumina platelets in a borosilicate glass. Composites ranging from 0 to 30 vol\% Al\textsubscript{2}O\textsubscript{3} were fabricated via hot pressing. Single edge-notch beam was used to gather fracture toughness data. Fracture toughness was found to increase up to 250\% for 30 vol\% Al\textsubscript{2}O\textsubscript{3} composites, from 0.77 MPa√m unreinforced glass to 1.92 MPa√m.

The authors stated that the large elastic mismatch (E\textsubscript{D} / E\textsubscript{M} ~7, where E\textsubscript{D} and E\textsubscript{M} are Young’s modulus for the alumina and glass respectively) would indicate a load transfer mechanism in effect as well. The authors also observed indentation induced microcracks on the surface of polished specimens and found crack deflection occurring in the matrix.

Todd and Boccaccini\textsuperscript{64} used fluorescence spectroscopy to measure the thermal residual stresses in these composites. The increment in toughness caused by the compressive residual stresses in the glass indicates a significant toughening effect from this source.

2.3.4 cBN-glass Composites

Limited studies have been done on cBN and glass systems examining the strength or fracture toughness of the composites. One study examined the effect of varying the glass composition with cBN to see composition which yielded the highest flexural
strength. Yang and Kim\textsuperscript{65} varied the composition of glasses containing Al\textsubscript{2}O\textsubscript{3}, B\textsubscript{2}O\textsubscript{3}, Na\textsubscript{2}O, and CaO using orthogonal array testing. Orthogonal array testing is a systematic, statistical way of providing representative coverage of all variable pair combinations, in this case by varying the composition of Al\textsubscript{2}O\textsubscript{3}, B\textsubscript{2}O\textsubscript{3}, Na\textsubscript{2}O, and CaO. The desired compositions were mixed, melted, and crushed until the powders were fine. These powders were then mixed with cBN and the mixture was dry pressed in a rectangular shape and sintered for 30 minutes at temperatures from 800 to 1000\textdegree{}C. The bars were tested via three point bending and flexural strength data was gathered. The glass composition yielding the highest strength was 51\% SiO\textsubscript{2}, 15\% Al\textsubscript{2}O\textsubscript{3}, 26\% B\textsubscript{2}O\textsubscript{3}, 3\% Na\textsubscript{2}O, and 5\% CaO. No explanation was offered as to why this composition of glass yielded the highest strength.

To the best of the author’s knowledge, there have been no studies that have been done to measure the fracture toughness of cBN-glass systems. There have been a few studies that have studied the strength of a vitrified bond cBN grinding wheel.\textsuperscript{66-68} The processing\textsuperscript{69-72}, applications\textsuperscript{73-75}, effect of coolant or lubricant\textsuperscript{76-79}, and trueing parameters\textsuperscript{80-82} (trueing is the removal of dull grains to sharpen a wheel) on vitrified bonded cBN grinding wheels has also been studied.
2.3.5 Fracture Surface Roughness and Fracture Toughness

A few studies have measured the roughness of fracture surfaces to identify toughening mechanisms. Some studies have also been done to relate the incremental increase in toughness, due to volume content of a composite, to the measured roughness.

A correlation between the fracture surface roughness and toughness due to toughening mechanisms was done by Chou and Green\textsuperscript{83} for SiC platelet-alumina composites. SiC-alumina composites containing 30vol\% SiC were hot pressed and the fracture surfaces were produced from a previous study.\textsuperscript{84} They determined the operative toughening mechanism to be crack deflection as opposed to crack bowing.\textsuperscript{84} They hypothesized that crack deflection would produce a rougher surface compared to crack bowing because a crack deflecting forced to deflect around a particle will produce a rougher surface. They were able to validate this by comparing the roughness of a 30vol\% SiC composite and a SiC-free alumina sample.

A scanning laser microscope measured the roughness of a SiC-free alumina sample and a 30vol\% SiC composite fracture surface. They found the average roughness of the alumina sample to be 3.8μm, which is close to the grain size measurement, i.e., 4.2μm, and indicates the validity of the experiment and data. The roughness of the
composites was 10.0µm. Since the roughness of the composites was ‘clearly greater’ than that of alumina and microstructural evidence indicated crack deflection, this provided more concrete evidence that crack deflection is the major toughening mechanism in this composite.

The variation of fracture roughness parameters with platelet content was not determined but an attempt was made by Boccaccini and Winkler. They attempted to relate the variation of fracture roughness with increasing volume fraction of alumina in a borosilicate glass matrix. Samples containing 0 to 30vol% Al₂O₃ were hot pressed and fracture surfaces were obtained by single-edge beam notch technique. The authors showed evidence in a previous study that crack deflection occurs when examining the fracture surfaces of these same composites. Fracture surface roughness was measured by using an optical profilometric technique. Figure 4 shows typical results of the roughness measurements for samples with 0 and 30vol% Al₂O₃ composites. Relative fracture toughness data and relative roughness values were plotted against each other and an increasing linear relationship was found as shown in Figure 5. The relative values are obtained by dividing the measured data by the value corresponding to the unreinforced glass matrix. The authors found a linear relationship and described by the following dimensionless equation:

$$K_{IC,r} = \frac{R_{a,r}}{18.0} + 0.85$$
where $K_{IC,r}$ is the relative fracture toughness of the composites and found by dividing the measured $K_{IC}$ by the $K_{IC}$ of the unreinforced composite. $Ra_r$ is the relative roughness of the fracture surfaces. The researchers concluded that this method can be used to predict the fracture toughness of a dispersion reinforced glass matrix composite from fracture roughness measurements and the toughness of an unreinforced matrix.

Figure 4: Fracture surface roughness measurements of a) borosilicate glass; and b) 30vol% Al$_2$O$_3$ borosilicate composite.
Digital Image Software for Analyzing Microscopic Images

Several studies have been reported in the last decade that uses digital image analysis and SEM images to gather quantitative information. Scientific fields that use digital image analysis have varied from biology, to size morphological features in cancerous tissue cells\textsuperscript{87}; to polymer science, to determine the amount of pores in a PVDF membrane\textsuperscript{88}; and ceramic science, to determine the grain size of powders sintered at various temperatures to correlate grain size to mechanical properties.\textsuperscript{89,90} Digital image analysis has also been used in geology\textsuperscript{91-93}, metallurgy\textsuperscript{94}, and other materials.\textsuperscript{95-98}

Figure 5: Relative fracture toughness of Al\textsubscript{2}O\textsubscript{3}-borosilicate glass composites as a function of relative roughness value showing a linear relationship.
These studies share fundamental procedures with each other in order to gather quantitative information. The procedure can be grouped into three steps: image treatment of the original image (such as removing noise from image acquisition); segmentation and/or thresholding of the image to distinguish features to be quantified; and digital image analysis software to quantify the isolated features in the image. The image treatment, segmentation, and thresholding steps can be reordered depending on the application.

The objective of image treatment is to enhance the quality of the original image by reducing noise, adjusting contrast, and adjusting non uniform lighting from acquisition so that the features to be quantified in the image are easily discernable. An example of the necessity of image treatment is provided by Sun. Sun et al. calculated the porosity, pore diameter distribution, pore area distribution, and pore shape distribution of membrane surfaces. Figure 6 is a picture of a collected SEM image before and after image treatment. The darker circular regions were identified as pores while the lighter dominant regions were identified as the membrane. It was necessary to process the original images first because the collected SEM image had low contrast and background noise that would have had strong effects when measuring the pores. For their image treatment process, Sun enhanced the contrast of the image and applied a median filter to remove background noise to further distinguish the pores on the membrane.
Segmentation is the process of dividing an image in order to distinguish desired objects. Segmentation of nontrivial images (such as fracture surfaces) can be one of the most difficult tasks in image processing. Segmentation accuracy determines the eventual success or failure of the digital image analysis. Many of the image analysis software available today have tools that can carry out segmentation either automatically (by detecting defined, uniform objects such as circles, triangles, and squares), manually (by tracing and isolating the desired objects by the user), or semi-manually (by using tracing tools that can automatically detect edges of desired objects).

Thresholding is the process of converting a grey scale image to a binary image and most basic digital imaging software has this feature. During the thresholding process, individual pixels in an image are marked as “object” pixels if their value is
within a user defined threshold range, and as “background” pixels otherwise. A binary image is then created by coloring each pixel black or white depending on the pixel’s label. Figure 7 is an example of an image before and after a threshold range has been applied. In this study, Masselin et al. measured the porosity, pore density, and mean pore radius of polymer ultrafiltration membranes. For this particular experiment, the pores are easily discernible especially after image treatment. The authors were able to apply a uniform threshold range for all their images.

Figure 7: Images of pores in a polymer membrane. The left picture is the image after SEM acquisition. The right picture is after a threshold range was applied. The black spots indicate the pores while the white background represents the membrane.

Often times because of the quality of the images from acquisition, it may not be appropriate to apply the same threshold range for all images and the user must manually adjust the threshold range to suit that image.

Sun et al. encountered this problem. It was difficult to apply a uniform threshold range to all the pictures because of the variation in contrast for the images. The membrane region in the SEM images is ‘unsmooth’ and some areas share intensity levels
similar to the pores. If a uniform threshold range is applied to all the images, then some regions of the membrane may be regarded as a pore. The authors used a slide control, as shown in Figure 8, to dynamically adjust the threshold until a satisfactory binary image for the region was achieved, and this was done for each image.

![Figure 8: Screenshot showing the adjustable threshold slide. This particular threshold range was able to distinguish all the pores in the membrane, indicated by the red spots in the image.](image)

The quantification methods in digital analysis software available today make use of a binary image. The software will count the number of “object” pixels against the “background” pixels and sum them up. If the “object” pixels are continuous and isolated shapes in the image, the software can use it to calculate parameters such as diameter, area, and density of the shapes in that image.101-103
To the best of the author’s knowledge, there have been limited studies that quantify inclusions or particulates on a fracture surface. The inherent challenge is because of the quality of SEM images for fracture surfaces typically varies in contrast, noise, and uniformity from one SEM image to the next. Since fracture surfaces with inclusions are usually rough, this makes image treatment, automatic segmentation, and thresholding difficult to apply to a sample large enough to represent quantitative values for the fracture surfaces. If a satisfactory binary image can been achieved, quantification becomes relatively simple. A procedure to determine the areal density of cBN crystals on fracture surfaces using digital image analysis and SEM images will be presented and discussed in Sec 4.5.3.1.
Chapter 3

EXPERIMENTAL PROCEDURE

3.1 Processing of Samples

The composites in this research were composed of two materials: cubic Boron Nitride, or cBN (cBN 400, Diamond Innovations, Worthington, OH) in the form of particulate single crystals; and borosilicate glass in the form of frit powder (Pyrex 7740, Corning, NY) and glass plates (Schott Borofloat, Louisville, KY). The physical properties of cBN and borosilicate are listed in Table 1 and were taken from the manufacturer’s specifications.

Table 1: Physical properties of cBN and Pyrex glass.

<table>
<thead>
<tr>
<th></th>
<th>cBN</th>
<th>Pyrex</th>
<th>Borofloat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>3.48</td>
<td>2.23</td>
<td>2.23</td>
</tr>
<tr>
<td>CTE, 10⁻⁶ · °C⁻¹</td>
<td>4.60</td>
<td>3.25</td>
<td>3.25</td>
</tr>
<tr>
<td>Young’s Modulus, GPa</td>
<td>662</td>
<td>64</td>
<td>64</td>
</tr>
<tr>
<td>Knoop Hardness, (kg/mm²)</td>
<td>45</td>
<td>418</td>
<td>418</td>
</tr>
</tbody>
</table>

In an attempt to study the effect of surface modified cBN crystals, some cBN crystals were heated treated at high temperature in a pure oxygen environment. 10g of cBN were placed in an alumina boat (Alumina AD998, Coorstek, Golden, CO). The boat
was placed on a fused silicate plate and placed in the center of a tube furnace. Oxygen flow was introduced in the tube. The heat profile is as follows: 5ºC/min to 1000ºC for 120 minutes. At the end of the hold time, the furnace was turned off and the powders were allowed to cool to room temperature.

The composites used in this research were composed of 0, 5, 10, 20, 30, 40, and 50vol% cBN and the remainder borosilicate glass. Batches for each volume fraction composite were made separately. Using the density for cBN and borosilicate and the simple rule of mixtures, the theoretical density for each volume fraction composite was calculated. From there, the required amount of cBN and borosilicate was known for each volume composite.

The required amounts of cBN and borosilicate powders for a particular volume fraction composite were placed in an agate mortar and stirred for a few seconds. About 3-5wt% of organic binder (Acryloid, Rohm & Haas, Philadelphia, PA), and acetone were then poured into the mortar. The organic binder, soluble in acetone, needed to be properly evaporated and mixed into the powders. Acetone, typically 15.00mL, was added into the batch to achieve a slurry-like consistency. An example of the typical batch amounts for a desired volume fraction is given in Table 2.
The mixture was gently stirred until the acetone was completely evaporated. Care was taken to not overspread the mixture on the sides of the mortar because this would lead the powders to dry up and result in minor inconsistencies in actual volume percent glass in the composites. If this occurred, more acetone was added and a scraper was used to remix the powders into the mixture. It usually took the mixture 15-25 minutes to completely dry. The powders were then carefully poured into a sealable plastic bottle and the bottle was shaken for 1 minute to thoroughly mix the powders.

The powders were then dry pressed into cylindrical pellet samples. A 1” steel die set was used to form the powders into pellets. The type of steel die used was a single action die consisting of three parts: a die, fixed during pressing, a bottom punch, fixed during pressing, and a top punch that moves during pressing.

1.0g of the powders were weighed and carefully poured into the die. The die was lightly tamped on the surface to level the powders and the top punch was inserted and a

<table>
<thead>
<tr>
<th>Volume Fraction Desired</th>
<th>20 %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Batch Amount</td>
<td>7.00 g</td>
</tr>
<tr>
<td>Mass of cBN</td>
<td>0.97 g</td>
</tr>
<tr>
<td>Mass of Pyrex</td>
<td>6.03 g</td>
</tr>
<tr>
<td>Mass of binder</td>
<td>0.21 g</td>
</tr>
<tr>
<td>Volume of acetone</td>
<td>15.00 mL</td>
</tr>
</tbody>
</table>

Table 2: Sample batch composition for a 20vol% cBN composite.
light pressure was applied to pack the powders. The die set was placed in a hydraulic dry press (Carver Model 3812, Wabash, IN). The pressing sequence for each sample is as follows: 4 metric tons for 60 seconds, relieve pressure, apply 4 metric tons for 30 seconds, and relieve pressure. The pellet was then ejected from the die and placed aside.

Thermolysis of the organic binder in the samples was the next step. The samples were placed on top of a refractory brick and set toward the center of a furnace in an atmosphere environment. The burnout heat schedule of the sample was as follows: 3.0ºC/min to 500ºC, 3 hours hold; and 3.0ºC/min to room temperature. The samples were then inspected to see if it survived thermolysis. There was usually no apparent indication of deformation, distortion, or the formation of cracks on the pellet surfaces. After burnout, the mass of the samples was measured again. The bulk density of the green body was then obtained by simply calculating it from the average thickness, diameter, and mass for each sample.

The samples were then placed on a refractory brick and set in a furnace in an atmosphere environment (i.e. no gases introduced into the furnace) for densification. The heat schedule was as follows: 3.0ºC/min to firing temperature, 1 hour hold; and 3.0ºC/min to room temperature. The maximum firing temperatures ranged from 720ºC to 850ºC.
After densification, the densities of the samples were measured by the Archimedes Method using the ASTM D3800-99, “Standard Test Method for Density of High-Modulus Fibers.” A digital scale balance (Mettler Toledo Model AE200, Beaumont Leys, UK) and a setup used to measure density by Archimedes method (suspension wire with sample pan, bridge, and container) was used for this technique. Deionized water at room temperature was used as the immersion media for these calculations.

The sample’s dry mass, $M_d$, was first measured. The sample was then immersed in the deionized water and its wet mass, $M_w$, was measured. The density of deionized water, $\rho_{\text{water}}$, at room temperature is 1.00 g/cm$^3$. The density of the sample, $\rho$, can now be calculated using the following equation:

$$
\rho = \frac{M_D}{M_D - M_W} \rho_{\text{water}}
$$

3.2 Preparing Samples for Mechanical Testing and Characterization

Samples of bulk Pyrex glass plates and 0-50vol% cBN composites were prepared for mechanical testing. A core drill (Model # 150655) Delta, Rockwell Company, Pittsburgh, PA) was used to machine cylindrical samples (diameter = 0.5”) out of the
Some samples composed of 5 and 20vol% cBN were polished for optical microscopy.

3.2.1 Preparing Samples for Mechanical Testing

The preparation of samples for diametral compression test involved grinding the samples to a uniform thickness, notching these samples through the center, and then V-notching to produce a sharper crack. The theory behind v-notching was discussed in Sec 2.2.1.

The grinding machine is equipped with a powerful magnetic base. A smooth, flat steel plate was used to anchor the samples. The steel plate was large enough to accommodate up to 30 samples (6 samples of the same volume fraction in 5 rows) and 2 microscope slides used to align the samples, as shown in Figure 9.

Figure 9: Typical arrangement of samples on a steel plate. The microscope slides were used to align the samples.
The steel plate was heated on a hot plate to about 200-250ºC. An adhesive (Crystalbond 509, Aremco Products Inc., Ossining, NY) was melted on the entire surface of the steel plate. The samples were placed onto the layer of the adhesive and pushed to alignment by the microscope slides, which were also on the layer of adhesive. The steel plate was carefully removed from the hot plate and allowed to cool. The microscope slides were left on the steel plate to make minor alignment adjustments of the samples as the adhesive was cooling and re-solidifying. The samples would remain on the steel plate through the grinding, notching, and V-notching process.

Figure 10 is a schematic of what the samples go through as they are surface ground and notched.

![Figure 10](image)
Figure 10: Stages of samples through the finishing process.

The steel plate was placed on a surface grinding machine ("Challenger", Boyer Schultz Hydraulic Surface Grinder, Broadview, IL) equipped with a water-based oil system. The steel plate and samples were aligned such that a row of samples containing the same volume of cBN was placed parallel to the direction of the grinding wheel. A 5"
diameter, ½” thick diamond-coated wheel (Diamond Devices Inc., Auburn, CA) was used to do the surface grinding of the samples. The grinding wheel was lowered and the samples were ground row by row. This process was completed until the samples were of uniform height. The surfaces of all samples were completely darkened with a permanent marker because it made it visually easy to see when the samples were surface ground to a uniform height.

Once the samples were of uniform height, single edge notches had to be introduced on the surface. The steel plate was mounted on a precision slicing and dicing machine (“Micro-Matic”, Micromech Mfg. Corp., Rahway, NJ) equipped with a water-based oil system and the samples were also aligned such that each row containing the same volume composition of cBN was parallel to the wafering blade. A 4” diameter, 0.012” thick diamond-coated wafering blade (Part # 801-137, Leco Corp., St. Joseph, MI) was used for the notching. The verniers on the slicing and dicing machine were used to locate the center for each row of samples and needed to be aligned properly and accurately. The diameter of the blade thickness was taken into account when locating the center.

The machine was turned on, and the wafering blade was lowered. As soon as the blade made contact with the sample, the verniers were calibrated to 0 and were notched in one direction. Once the composites were notched at that vertical level, the wafering blade was lowered at 0.005 inches (or 0.127mm) and notched in the reverse direction. This two step process was repeated until the desired notch depth was achieved. Care was
taken to minimize torque on the machine motor by making sure the feed rate was slow and the wafering blade was lowered slowly.

Finally, the notch was sharpened with a hand-held razor blade sprinkled with diamond paste. A 0.009” blade (“American Line”, American Safety Razor Co., Staunton, VA) was coated with a 2 micron diamond paste (Leco Corp., St. Joseph, MI) and pushed back and forth for each row. The new V-notch was observed under a stereoscope. If it looked satisfactory, i.e. the new V-notch appeared fairly “sharp,” then the next row was V-notched. Figure 11 compares a notch before and after it has been sharpened. After all the rows were v-notched, the steel plate was washed under tap water for one minute to remove excess diamond paste in the notches.

![Figure 11: Photograph of a sample before and after V-notching.](image)

The samples were removed from the steel plate by placing the steel plate back on the hot plate to allow the adhesive to soften and free the samples. These samples were removed by row and allowed to cool for a minute before placing them in a beaker filled
with acetone. The beaker was then placed in a sonic water bath (model 1510, Branson Ultrasonics Corp., Danbury, CT). Two cleaning steps were employed, each one incorporating a fresh beaker of acetone. Each step consisted of 3 minutes in the sonic bath for the samples.

After cleaning, the samples were placed on a refractory brick and heated to the annealing temperature of the borosilicate glass to eliminate the residual stresses introduced by the machining of the samples. The firing profile was as follows: 3°C/min to 560°C, hold for 1 hour; 1°C/min to 450°C, no hold, 3°C/min to room temperature.

### 3.2.2 Preparing Samples for Optical Microscopy

Samples for optical microscopy analysis went through a 4 step grinding and polishing process. Samples were mounted on a circular steel cylinder fixture (1.5” diameter, 3” height) that was large enough to accommodate 5 samples. The cylinder fixture fits inside a steel circular sleeve which has a thick base, as shown in Figure 12. This allows the samples to remain flat while they are being polished.
The samples were surface ground to a uniform height following the same procedure as described in Sec. 3.2.1.

A 2 speed tabletop polishing machine (Buehler Two Speed Ecomet 5 Polisher, Irvine, CA) was used to polish the samples. The polishing machine is set up such that platens can be removed and interchanged. These platens have removable cloths (8” diameter cloth, Buehler Consumables, MICROCLOTH, Irvine, CA) adhered to it and each cloth/platen is used for one specific diamond suspension sizes (Buhler Metadi Polycrystalline Diamond Suspension, Irvine, CA) to avoid cross contamination when polishing at successively lower sizes. Samples were successively polished using diamond suspensions at 15 μm, 3μm, and 1μm for 5-10minutes each.
3.3 Mechanical Testing

A mechanical testing machine (Model 5866, Instron, Canton, MA) fitted with a 10kg load cell was used to test the composites. The ASTM D3967-05 (2005), “Standard Test Method for Splitting Tensile Strength of Intact Rock Core Specimens,” was closely followed.

Figure 13 is a schematic of how the sample is loaded. The bottom rigid bearing surface was a circular loading stage (5” diameter) of 4340 alloy steel. The top rigid bearing surface was a cylindrical compression fixture (1”diameter, 5”thickness) made of 4340 alloy steel. 4340 alloy steel has a Rockwell hardness of 100 HRC. The diameter and Rockwell hardness of these bearing surfaces easily met the ASTM standard of having the surfaces “at least as great as the specimen thickness” and “not less than 58 HRC”.

Bearing strips were used as stress cushions and acted to absorb the line contact stress and distribute it over a portion of the sample surface as discussed in Sec 2.2.2. Two strips of index cards were stacked and taped on the loading stage and the compression fixture.

Sample dimensions also conformed to ASTM standards. The standard calls for a circular disk with a thickness-to-diameter ratio (L/D) between 0.2 and 0.75. Samples in this study had a thickness of 3.75 to 4.05mm and a diameter of 11.20 to 11.60mm. The ratios in this study ranged from 0.32 to 0.35 satisfying the standard.
Figure 14 is a schematic of the testing apparatus. A computer program (Bluehill 2, Instron, Norwood, MA) was used to control the mechanical tester and collect data. The crosshead speed of the mechanical tester was set at 0.03 mm/min. It was programmed to stop the test automatically when a drop in load occurred. This drop in load signified the sample cracking and was usually accompanied by an audible sound. The sample was usually held in place by the bearing strips along with the horizontal compresses stresses near the load points. A schematic of a load/deflection curve found for this test is shown in Figure 15. The samples usually failed in 2 to 7 minutes. The ASTM standard calls for the sample to fail in 1 to 10 minutes as past research has found that the rates of loading in this range are reasonably free from rapid loading effects.56

The maximum load, the dimensions of the sample, the notch depth, and the width of the bearing strip impressions were taken and used to determine the fracture stress and fracture toughness, as discussed in Chapter 4.
Figure 13: Schematic of the sample/bearing surface portion of the splitting tensile test.
Figure 14: Schematic of the mechanical testing apparatus.
3.4 Microstructural Characterization

The cBN crystals, borosilicate powders, and composites were examined using a scanning electron microscope, or SEM (Hitachi S300N, Hitachi High Technologies, Inc., Frederick, MD) with a tungsten filament, equipped with a secondary electron detector, or SE, an energy dispersive x-ray spectrometer, or EDS (Model OPH045-1038, Prism PGT digital spectrometer, Princeton Gamma-Tech, Princeton, NJ) and a backscattered electron detector, or BSE (Model EPTSERMA, Robinson Detectors, Sydney, Australia). The samples were gold coated in vacuum, using an in-house gold sputtering machine, for 1 minute prior to characterization in the SEM.
An optical microscope (Model BX60M, Olympus Optical Co., LTD., Tokyo, Japan) equipped with a digital camera was used to photograph macroscopic features, such as the notches in the sample. A computer program (Images Plus 2.0, Motic, British Colombia, Canada) capable of measuring features in images was used in conjunction with the digital camera to measure the notch depths of the fracture surfaces. The notch depth can be easily discerned and was measured at three different locations to get an average. The average of all measurements was taken to be the notch depth of the sample.

X-ray diffraction patterns were measured using an x-ray diffractometer, or XRD (Scintag Pad V, Scintag, Inc., Cupertino, CA) equipped with a germanium solid state detector. The scanning angles extended from 5 to 70°, at 0.2°/min step rate. XRD patterns to be collected included standard and heat treated cBN crystals, polished and fracture surfaces of cBN composites, manually crushed cBN composite powders, and borosilicate powders. XRD patterns were analyzed using an XRD pattern, processing, identification, and quantification program (Jade 8.5, MDI, Livermore, CA).

X-ray photoelectron spectroscopy, or XPS (Kartos Axis Ultra System) with a monochromatized Al Kα source (1446.6eV), was used to analyze the chemical makeup of standard and heat treated cBN crystals and boron oxide powder, or B₂O₃ (Sigma Aldrich, St. Louis, MO). The B₂O₃ was used as a standard to determine the atomic concentration of B₂O₃ in the heat treated cBN crystals.
3.5 Analysis of Fracture Surface Images

SEM pictures, with BSE and SE detectors, of surface ground and fracture surfaces of the composites were taken. A method to quantify the amount of crystals on a fracture surface was developed and the use of two image processing software programs was used.

ImageJ (Wayne Rasband, National Institute of Health, Bethesda, MD) is open source software and has a useful feature that can measure and analyze particles in an image. This is done by defining a threshold value for an image. The software will count the number of pixels in that threshold range and sum up their area. Also if the pixels in a given threshold range are adjacent neighbors in all directions, the software will count that range of pixels as a particle, assign it a numerical value, and list its individual size.

Photoshop CS2 (Adobe, San Jose, CA) has many powerful capabilities including the abilities to semi-manually trace features with ease and accuracy. There are a few features in its software package that use image segmentation techniques as discussed in Sec 2.4, such as the magic eraser tool, magnetic lasso tool, and extract feature to isolate regions of interest.

The development of a procedure to determine areal density of cBN on a fracture surfaces was required and will be discussed in Sec 4.5.3.1.
Chapter 4

RESULTS AND DISCUSSION

4.1 Characteristics of cBN Crystals

Physical characteristics of the cBN crystals were first examined using the SEM. The chemical composition and microstructure for standard and heat treated cBN were investigated using XPS and XRD analyses.

Figures 16 and 17 show SEM pictures of cBN crystals as received. These particles vary in shape and size and appear to be blocky. Using the scale markers on the SEM images, they range from 100 to 200μm in size. Crystal faces are shown to either have smooth surfaces or sharp facets and steps. There is evidence of particle failure that may have occurred during their processing. These cBN crystals were used for the composites as received.

Figures 18 and 19 show SEM pictures of the cBN crystals after they have been heated treated in an O₂ atmosphere for two hours. The crystal faces no longer appear to be smooth and the growth of a surface phase is clearly seen to have formed. Sharp facets observed in the as received cBN crystals do not appear to be as well-defined.
XPS was performed on standard and heat treated cBN crystals (and boron oxide for reference). The standard cBN crystal and boron oxide powder data was used as a standard to measure the boron oxide content of cBN crystals treated in oxygen. Table 3 shows the atomic concentration of the amount of boron detected in the form of boron oxide and boron nitride for the boron oxide and cBN crystals (as reference) and the heat treated cBN crystals. It was found that about 45% of the boron detected on the surface of the heat treated crystals was in the form of B$_2$O$_3$. The heat treatment was sufficient to modify the surface of the crystals but not enough to completely oxidize the surfaces. Referring back Figures 18 and 19, the ‘liquid-like’ substance on the crystal faces is probably boron oxide glass formation due to the heat treatment.

XRD was performed on the standard and heat treated cBN crystals as seen in Figure 20 and both are shown to have two sharp peaks at 43º and 51º and the phase identified by the XRD identification program was cubic-BN as expected. Heat treated cBN also has boron oxide in its diffraction pattern, as labeled in the figure. Based on the results of the XPS and XRD data, the heated treated cBN crystals will be referred to as oxidized cBN.

Table 3: Atomic surface composition of standard cBN, B$_2$O$_3$, and heat treated cBN.
Figure 16: SEM image of standard cBN crystals.
Figure 17: SEM images of individual standard cBN crystals showing various physical features.
Figure 18: SEM image of heat treated cBN crystals in an $o_2$ atmosphere for 120 minutes.
Figure 19: SEM images of individual standard cBN crystals showing various physical features.
Figure 20: XRD data of standard and heat treated cBN crystals.
4.2 Physical Properties of the Composites

The sintering temperature for the composites was established on the basis of density using relative density data and microstructural analysis. Composites containing 5 to 50vol% cBN were made and fired to 700, 750, 775, 800, and 850°C. The heating schedule was discussed in Sec 3.1.

The relative densities of all composites were measured using the Archimedes method as discussed in Sec 3.1. Figure 21 plots the relative density of the composites versus the firing temperature. The relative densities of all composites fired at 700°C only reached an average relative density of 84% (not shown), and were thus discarded. Higher relative densities, all greater than 92%, were reached for composites fired above 750°C.

Composites containing 10 to 30vol% cBN exhibited the highest relative density when fired at 750°C. Composites at 40vol% achieved the highest relative density at 800°C and at 50vol% the highest relative density occurred at 850°C.

XRD data was gathered for each composite with the highest relative density to determine any microstructure differences. The results of the XRD patterns for the densest samples are shown in Figure 22. It is shown that cristobalite is present in all composites. However, it can be seen that the amount of cristobalite increases with increasing sintering temperature. The 20vol%, fired at 750°C, shows the least amount of cristobalite formation.
XRD data was gathered for the other composites fired at 750°C as well as cBN-free borosilicate sample to determine any cristobalite peak differences among them, as shown in Figure 23. Since cristobalite peaks were present in all of the composites it was decided to quantitatively determine the amount of cristobalite in the composites sintered at 750°C. This can be done with the XRD analysis software and uses the Rietveld method. The theory behind the Rietveld method is beyond the scope of this thesis and will not be discussed.

In order to determine the amount of cristobalite in the composites, the composites needed to be crushed and a known amount of a quartz standard was added and mixed into the crushed powders. This was done for one sample from each 0 to 40vol% cBN composite. It was found that for all composites, the amount of cristobalite found was about 7 ± 3%. This is a semi-quantitative estimate because the grain size of the cBN is much larger than the grain size of the silica standard and because of the hardness of cBN; it was difficult to crush the cBN crystals to the size of the silica powders. Because the amount of cristobalite does not vary for composites ranging from 0 to 40vol% sintered at 750°C, it can be concluded that the cBN does not enhance devitrification of the glass.

It was decided to fire all standard and oxidized composites at 750°C based on the relative density and the microstructural analysis. All composites fired at 750°C showed high relative density up to 30vol% cBN and the microstructural analysis showed the amount of cristobalite to be consistent from 0 to 40vol% composites.
Figure 21: Relative density of standard cBN composites as a function of volume fraction for different firing temperatures.
Figure 22: XRD data for composites exhibiting highest relative density for each firing temperature.
Figure 23: XRD data of borosilicate glass and standard cBN composites fired at 750°C.
4.3 Microstructural Properties of Composites

Figure 24 shows the pellet-shaped samples after they have been surface ground and notched. These samples are aligned in columns based on volume fraction of cBN, from 40, 30, 20, and 10vol%, left to right. Figure 25 shows polished surfaces of 5 and 20vol% cBN composites. The cBN crystals appear to be well dispersed in the glass matrix.

Figure 24: Photograph of composites aligned on a steel plate after machining. Each column consists of the same volume fraction composite, from 40 to 10vol% starting at the left.
Figure 25: Optical images of polished surfaces of 5 and 20vol% cBN composites. The interfaces of cBN-glass appear to be dense.
4.4 Mechanical Testing of Composites

The diametral compression test using a single edge V-notch was used to fracture the samples, as discussed in Sec 2.2.2. The setup of the test was described in Sec 3.3.

4.4.1 Fracture Toughness Measurements of Standard cBN Composites

Fracture toughness values for cBN-free borosilicate glass and glass plate were gathered for comparison. The cBN-free borosilicate glass samples were prepared as described in Sec 3.1 and the glass plate samples were prepared as described in Sec 3.2.

The peak load measured by the mechanical tester was used to determine the value of the fracture stress, $\sigma_f$, in the following equation:

$$\sigma_f = \frac{2P_y}{\pi dt} \left( \frac{\sin 2\alpha - \alpha}{\alpha} \right)$$

where $P_y$ is the maximum load, $d$ is the diameter of the sample, and $t$ is the thickness of the sample. The length of the impression left on the bearing strips after testing is the ‘$\alpha$’, or the half angle that makes up the cushioned arc length. It was simple to measure the length, usually 1 to 2 mm, and the bearing strips were replaced after each sample tested.
The fracture toughness was calculated using the following general equation:

$$K_{IC} = \sigma_f Y \sqrt{c} = 1.12\sigma_f \sqrt{c}$$

where $T$ is the toughness, equivalent to the critical value of the stress intensity factor $K_{IC}$. The $K$ subscripts, I and C, denote “mode I” and “critical”, respectively. The variable $Y$ is the geometric factor and $c$ is the crack length. The fracture strength is found from the sample dimensions and peak load as in Eq 4.1. The notch depth is assumed to be the initial crack size, $c$.

The notch depth or initial crack size, $c$, was determined by measuring the length of the notch under an optical microscope. It was easy to discern the depth created by notching via the wafering blade and the additional depth of the ‘pre-crack’ created by v-notch with the razor blade. Figure 26 is a picture of a fracture surface illustrating depths of the notch and V-notch and the fracture surface. Three measurements from each half were made and the average was taken to be the initial crack size.
The average fracture toughness values for the glass plates, standard and oxidized cBN composites are plotted in Figure 27. The fracture toughness of borosilicate glass was measured by testing glass plates and by testing samples that prepared by method discussed in Sec 3.1. The fracture toughness of the glass plates was 0.71MPa√m and had a standard deviation of 0.014. The fracture toughness of cBN-free borosilicate glass is 0.56MPa√m. The fracture toughness values for the glass plates agree well with literature, but the fracture toughness values for the cBN-free borosilicate glass is much lower. This could be a result of processing conditions such as the cristobalite found in its microstructure.

The introduction of 10vol% of cBN into the glass yields a fracture toughness of 1.11MPa√m, an increase of 98% from the cBN-free borosilicate glass. The fracture toughness increases further with the addition of cBN crystals up to 1.39MPa√m for
30vol% cBN composite. Fracture toughness stops noticeably increasing at 40vol% cBN composite, yielding an average fracture toughness of 1.41MPa√m. The low relative density for the 40vol% cBN composite, at 94%, can explain why the toughness increase is minimal. A similar effect was found by Chaim and Talanker\textsuperscript{108} where they mechanically tested SiC-cordierite glass composites containing 0-30vol% SiC. The higher volume fraction SiC composite yielded a lower relative density and the authors explained that the increased volume fraction along with the decreased relative density may counteract the reinforcing effect of the SiC platelets and may thus result in a zero net reinforcement, or toughness increases, of the composite.

The fracture toughness of 50vol% cBN composite drops significantly to 0.86MPa√m (results not shown in graph) and can also be attributed to its low relative density (90.5%).

The overall fracture toughness increase from the cBN-free borosilicate glass to the 40vol% cBN composite is 152%. The increase in toughness for cBN-borosilicate glass composites is comparable to other nonoxide-glass composites. For SiC-cordierite glass composites\textsuperscript{60}, fracture toughness was improved with increasing volume fraction of SiC whiskers and particles. 30vol% of SiC whiskers-cordierite glass composites showed the best improvement from SiC-free cordierite glass with a fracture toughness increase of 74%. Fracture toughness for SiC particle-cordierite glass improved by 37% but only up to 20vol% SiC addition.
Chen et al.\textsuperscript{62} studied the effects of adding AlN powders in cordierite glass. Fracture toughness was improved with increasing AlN powder content. 40vol% AlN-cordierite glass composites showed the best improvement from AlN-free cordierite glass with a fracture toughness increase of 139% indicating good toughening effect on AlN particles. It was said that the thermal mismatch between the particles leads the AlN particles to introduce residual stresses in the composites, thereby increasing its strength.

### 4.4.2 Fracture Toughness Measurements of Oxidized cBN Composites

Composites made with oxidized cBN and borosilicate glass were prepared using the procedure outlined in Sec 3.2. Since a noticeable decrease in density and fracture toughness were observed in composites containing 50vol% of standard cBN and borosilicate sample, composites with oxidized cBN were only made from 10 to 40Vol %. The composites were mechanically tested using the exact procedures described in Sec 3.3. The introduction of 10vol% of oxidized cBN into the glass yields a fracture toughness of 1.12MPa$\sqrt{m}$, an increase of 98% from the cBN-free borosilicate glass. Fracture toughness increases to 40vol% oxidized cBN composites to 1.28MPa$\sqrt{m}$.

The overall trend shows for the fracture toughness of oxidized cBN composites shows lower fracture toughness than standard cBN composites. However, it should be noted that the standard deviations between the standard and oxidized cBN composites are
statistically insignificant. Overall fracture toughness increase from the cBN-free borosilicate glass to the 40vol% oxidized cBN composite is 129%. The boron oxide on the surface of the crystals may have melted into the frit powder during the sintering of the samples. Thus, modifying the surface of cBN does little to affect the fracture toughness of the cBN-glass composite based on the fracture toughness measured for standard and oxidized cBN-borosilicate glass composites.

Fracture toughness effects for oxide-glass composites have also been investigated. Boccaccini et al.\textsuperscript{63} studied the toughening effect of Al\textsubscript{2}O\textsubscript{3} platelet-borosilicate glass composites. Fracture toughness increased from 0.77MPa\(\sqrt{m}\) for borosilicate glass to 1.92MPa\(\sqrt{m}\) for 30vol% Al\textsubscript{2}O\textsubscript{3} composites, a total increase of 250% in fracture toughness.
Figure 27: Fracture toughness for glass plates and the standard and oxidized cBN-glass composites as a function of volume fraction of cBN.
4.5 Microstructural Evaluation of cBN Composites

This section is subdivided into three parts. The first subsection will discuss general features of the fracture surfaces of cBN-glass composites. The second subsection will discuss the qualitative analysis of the fracture surfaces and the third subsection will discuss the quantitative analysis of the fracture surfaces.

4.5.1 General Features of Fracture Surfaces

Low magnification pictures of the fracture surfaces of 0 to 50vol% cBN composites and compared with each other as seen in Figure 28. As the volume fraction increases, so does the ‘roughness’ or ‘tortuosity’ of the fracture surfaces. Past studies\textsuperscript{60,63} have shown this to be a clear indication of toughening due to crack deflection.

Figure 29 shows typical individual cBN crystals embedded on the fracture surface for 10 to 30vol% cBN composites. The cBN crystals exposed on the fracture surface vary, from smooth faces to rough surfaces, just like the individual cBN crystals shown in Figure 17.

Figure 30 shows cBN crystals on the fracture surface of a 40vol% cBN composite. At this volume loading, the cBN crystals are in closer vicinity to each other
compared to composites with 10 to 30vol% cBN. At 50vol% it is easy to see the effect of a low relative density, at ~91%, has on the measured fracture toughness. Figure 31 shows crystals on a 50vol% fracture surface. The cBN crystals are much closer together and the glass looks heavily microcracked.

Fracture surfaces of 0 to 40vol% oxidized cBN composites were also examined, as seen in Figure 32. Roughness also increases but there is not much difference that can be seen between these fracture surfaces and the fracture surfaces of the standard cBN composites.
Figure 28: Fracture surfaces of standard cBN composites.
Figure 29: Typical cBN crystals exposed on fracture surfaces for 10, 20, & 30vol% cBN composites.
Figure 30: cBN crystals exposed on a fracture surface for a 40vol% cBN composite.
Figure 31: cBN crystals exposed on a fracture surface for a 50vol% cBN composite.
Figure 32: Fracture surfaces of oxidized cBN composites.
4.5.2 Qualitative Analysis of the Fracture Surfaces

General features of the fracture surfaces have been identified. However, the features identified do not describe how the crack propagates through the composite. Figure 33 illustrates a model of three cases of possible crack particle interactions, showing a crack either propagating through the matrix, through the particle, or the interface between cBN and glass. If opposing fracture surfaces of a composite are carefully examined, definitive information can be gathered about these possible crack particle interactions.

![Diagram of crack particle interactions](image)

Figure 33: Model of three crack-particle interaction possibilities. A crack approaching a cBN-glass interface can go through the glass matrix, through the particle, or through the interface of the cBN-glass.

In order to compare both fracture surfaces, it was decided to image the bottom of both fracture surfaces of a sample simultaneously. The fracture surfaces were carefully aligned and taped together so that the paired locations of the surfaces can be viewed.
SEM images were taken near the edges of where the halves met to produce ‘matched image pairs’ for the fracture surfaces.

Figures 34, 35, and 36 show matched image pairs of fracture surfaces for 10, 20, and 40vol% cBN composites. Mirror locations from both fracture halves were compared and two identifiable crack-particle interactions were found for these composites. In many instances, a cBN crystal found on one fracture piece can also be found on the mirror location of the other fracture piece. This implies that as the crack approached the crystal, it propagated through the crystal and the crystal underwent transgranular particle failure, as seen in Figure 37.

If a crystal is found on one side of a fracture surface but its mirror location on the other fracture surface has an impression, this implies that as the crack approached the cBN-glass interface, the crack propagated through that interface and thus interfacial failure occurred, as seen in Figure 38.

Four image pairs for each volume fraction standard composite were examined in order to determine the occurrence of transgranular particle failure and interfacial failure. The frequency of transgranular particle failure was similar for 10 to 30vol% cBN composites and usually occurred 85% of the time. The frequency of transgranular particle failure for 40vol% cBN occurred 50% of the time. This could be attributed to the lower relative density of this composite and the heavily microcracked interface between cBN-glass as shown in Fig 30.
Qualitatively, it has been shown that interfacial failure and transgranular particle failure are present in 10 to 40vol% composites. The occurrence of each failure type has also been determined. While, this does give information about a crack propagating through the cBN-glass composite, it does not indicate whether the crack prefers to propagate through the glass more than to interact with the cBN.

Figure 34: Matched image pairs of a fracture surface of a 10vol% cBN composite with examples of interfacial and transgranular particle failure labeled at mirror locations.
Figure 35: Matched image pairs of a fracture surface of a 20vol% cBN composite with examples of interfacial and transgranular particle failure labeled at mirror locations.
Figure 36: Matched image pairs of a fracture surface of a 40vol% cBN composite with examples of interfacial and transgranular particle failure labeled at mirror locations.
Figure 37: Transgranular Particle Failure of cBN in a cBN-glass Composite.
Figure 38: Interfacial Failure of cBN in a cBN-glass Composite.
4.5.3 Quantitative Analysis of the Fracture Surfaces

4.5.3.1 Validating a Procedure for Quantifying Fracture Surfaces

Figure 39 shows an example of two extreme cases of crack propagation in a matrix. While it has been shown that a crack propagating through the composite interacts with cBN, by transgranular particle failure or interfacial failure, the frequency to which this occurs has not been established. In order to determine the frequency of crack-particle interaction in a cBN-glass composite, the areal density of cBN on a fracture surface needs to be quantified.

Figure 39: Examples of 2 cases of a crack propagating through a cBN-glass composite. The crack can preferentially propagate through the glass matrix, or when it approaches a cBN-glass interface it can either propagate through the cBN or through the interface.
Digital imaging software has been used to quantify regions of interest as discussed in Sec 2.4. These concepts were applied to the analysis of fracture surfaces. This section will first present the procedure developed, then discuss its validity, and end with a discussion of its development and an explanation of the steps that were chosen to execute the image treatment process.

An SEM image of a surface ground composite was first used. By exposing a random plane of a composite, one would expect to find an area fraction of cBN crystals equivalent to the volume fraction of particles in the composites. The back scattered electron, BSE, detector was used to collect images using the SEM. One of the advantages of using the BSE detector is that it provides high contrast when imaging relatively flat surfaces.

Segmentation on images has been done on well defined objects, such as circular pores in a membrane, etc.\textsuperscript{92-95} Because of the irregularity in shapes for these cBN crystals and the roughness of the fracture surface, it would be difficult to use procedures developed to automatically identify well defined and uniform shapes. Therefore segmentation cannot be done automatically to isolate the cBN crystals. There is technology available that facilitates isolation via semi-manual tracing techniques. Many of the tools featured in Photoshop use image segmentation techniques that have the abilities to semi-manually trace features with ease and accuracy. Photoshop’s “extract tool” was chosen as the ideal tool to use.
With the “extract tool”, objects are highlighted according to the user but using the ‘Smart Highlighting’ feature, edges can highlight as you trace the foreground object, thus making this semi manual tracing.

To test it, the “extract tool” was applied to a surface ground image of a 40vol% cBN composite. Figure 40 are sequential screenshots of how the crystals are segmented from the original image. The top picture is the original image, the middle picture shows the images segmented, and the bottom picture is the final image of the crystals extracted out of the picture, represented by the ‘white’ pixels, and leaving only the glass matrix in the image.

Figure 41 is the next sequential steps for the image treatment process. The glass matrix left in the image is converted to black by adjusting the brightness to 0 and the contrast to 1. The edges in the image are smoothed out by applying a median filter with a setting of 2.0. An appropriate threshold range must be set to account for all cBN crystals and for these images it is set from 65 to 255.

The image is then saved and opened in ImageJ. In Image J, no further image processing needs to be done. ImageJ has a feature called ‘Measure Particles’ and is used for the quantification step. ImageJ will count the number of “object” pixels. For our images, the “object” pixels represent all the cBN crystals and the “background” pixels, the glass matrix. The areal density of cBN crystals is calculated by taking the sum of the “object” pixels and dividing it by sum of the “object” and “background” pixels.
For the 40vol% cBN surface ground image that has undergone the above procedure, the areal density is found to be 39.9%, as seen in Figure 42. This compares favorably with the bulk concentration of the cBN in this 40vol% cBN composite. Results (not presented) for the areal density of cBN crystals in surface ground images of 10-30vol% cBN composites were also close to their respective bulk composition. The expected area densities of cBN crystals were similar to their respective bulk composition and thus validate the procedure developed.

The largest source of variability occurs when segmenting the cBN crystals because the SEM image must have the cBN crystals clearly distinguishable so that the user can segment them. There were some automatic tools in Photoshop such as “magic lasso” or “eraser” that can isolate objects quicker based on pixels that are similar in intensity and adjacent to one another. Even though the surface ground images were acquired with a BSE detector which gave high contrast between the cBN and glass, these automatic tools were still found to be inapplicable since some of the pixels that represented cBN also represented glass. Since these automatic tools were inapplicable to BSE images of surface ground images, it was unlikely to be applicable to SEM images of fracture surfaces that have a lower range of contrast between the cBN and glass.

The “extract tool” was chosen in Photoshop because it allows the user to magnify the image and re-adjust the segmentation of crystals if an error occurs by the user. Because segmentation is performed semi-manually by the user, this tool can also be applicable to cBN crystals on a fracture surface.
Once the crystals are segmented, the steps that follow minimize user dependence and variability. It was found that a few of the pixels that represented the glass matrix were ‘white,’ due to SEM acquisition. As defined earlier, the ‘white’ pixels represent cBN crystals and in order for the quantification to accidentally count ‘white’ pixels that represent glass, it was decided to convert the remainder of the image to ‘black’ so there was no question of the ‘black’ pixels representing glass and the ‘white’ pixels representing cBN.

Because cBN crystals on a fracture surface are easily distinguishable, as shown in Sec 4.5.1 and 4.5.2, it is expected that the procedure described in this section should be applicable for determining the areal density of cBN crystals on fracture surfaces.
Figure 40: Photoshop screenshot of the cBN crystals extracted from the image of a surface ground 40vol% cBN fracture surface.
Figure 41: Photoshop screenshots of the image treatment procedure. From top to bottom, the image is first converted to black and white, then a median filter is applied, and finally an appropriate threshold range is applied.
Figure 42: ImageJ screenshot showing the areal density of a 40vol% cBN composite surface ground image.
4.5.3.2 Quantifying the Areal Density of cBN in Standard and Oxidized Composites

The procedure developed in the previous section was used to determine the areal density of cBN on fracture surfaces for standard and oxidized composites. Five samples from each volume fraction were image treated, taking random fractured halves from each sample. For each fracture surface, two SEM images were quantified to represent a total of 30% of the total fracture surface for that sample. Figures 43, 44, 45, and 46 are examples of images before and after treatment for 10, 20, 30, and 40vol% cBN composites, respectively. The areal density of cBN on the fracture surfaces are labeled for each of these pictures.

Figure 47 is a summary of the areal density of cBN crystals exposed on the fracture surfaces for standard and oxidized composites. It is found that the areal density of cBN on the fracture surfaces is much lower than the value expected for a random crack propagation through the composite (i.e. an areal density equivalent to the projected volume fraction). Error bars have been added to the data to account for the amount of interfacial and transgranular particle failure found in Sec 4.5.2. The “random” data in the graph represents the areal density of cBN crystals on a random surface in the composite.

The areal density of cBN crystals exposed on the fracture surface for both the standard and oxidized composites is far less than the theoretical maximum indicating that
a crack propagating through this system avoids the cBN crystals and prefers to propagate through the matrix, as illustrated by the model in Figure 48. The crack’s preferred mode of propagation is represented by the solid line while the dashed line represents the theoretical maximum, i.e. the areal density of that plane of cBN crystals if quantified.

The thermal expansion of the borosilicate glass (CTE = 3.25x10^{-6}/ºC) is lower than the cBN crystals (CTE = 4.60x10^{-6}/ºC) creating a radial tension and hoop compression field around the particles upon cooling from the sintering temperature. Wei and Betcher\textsuperscript{60} found that for a composite where the CTE\textsubscript{inclusion} > CTE\textsubscript{matrix}, such as the cBN-borosilicate system, a crack will be expected to propagate in the direction parallel to the axis of the local compressive stress and perpendicular to the axis of the local tensile stress in the matrix surrounding the particle. As the crack moves around the particle it can continue to propagate through the matrix or it can be attracted toward the particle interface. Figure 49 applies their model to the cBN-glass system. The compressive force surrounding the cBN causes the crack to deflect the cBN-glass interface and propagate through the glass.

The findings of Wei and Betcher correlate well with the cBN-glass composite. The interpreted results of the areal density of cBN crystals in standard and oxidized composites show the crack preferring to propagate through the glass matrix and must be due to the residual stresses surrounding the cBN crystals.
Elastic mismatch has also been shown to influence crack propagation for a composite where the $\text{CTE}_{\text{inclusion}} > \text{CTE}_{\text{matrix}}$. It was found that the presence of elastic mismatch between the inclusion and matrix alters the path of the crack front to avoid the particles, provided that there is a strong interfacial bond between the inclusion and the matrix.\textsuperscript{50,51} The low areal density of the cBN on the fracture surfaces seems to further support the effect of elastic mismatch.
Figure 43: A 10vol% cBN composite fracture surface before and after image processing, showing an areal density of 2.5% cBN crystals exposed on this fracture surface.
Figure 44: A 20vol% cBN composite fracture surface before and after image processing, showing an areal density of 4.3% cBN crystals exposed on this fracture surface.
Figure 45: A 30vol% cBN composite fracture surface before and after image processing, showing an areal density of 9.1% cBN crystals exposed on this fracture surface.
Figure 46: A 40vol% cBN composite fracture surface before and after image processing, showing an areal density of 14.0% cBN crystals exposed on this fracture surface.
Figure 47: Summary of the areal density of cBN crystals on the fracture surfaces for standard and oxidized composites as a function of volume fraction of cBN. Error bars account for the occurrence of transgranular particle failure and interfacial failure.
Figure 48: Model showing the crack’s preferred mode of propagation through a cBN glass composite. The solid line shows the preferred mode of propagation through the composite, sometimes interacting with cBN via transgranular particle failure or interfacial failure. The dashed line represents the theoretical maximum of the areal density of cBN crystals found on a random plane.
Figure 49: Deflection of crack by particle ($\text{CTE}_{\text{cBN}} > \text{CTE}_{\text{glass}}$) and associated matrix stresses. Crack moving in plane of particle will be deflected by compressive hoop stress axis.
Chapter 5

CONCLUSIONS

Composites of cBN-borosilicate glass, ranging from 10 to 50vol% standard cBN and 10 to 40vol% oxidized cBN, were made. The cBN-glass composites were mechanically tested using the diametral compression test with a single-edge V-notch. The fracture toughness was then calculated using the following general equation:

\[ K_{IC} = \sigma_f Y \sqrt{c} = 1.12 \sigma_f \sqrt{c} \]

The fracture toughness for a cBN-free borosilicate glass is 0.56MPa√m. The toughness increased up to a 40vol% cBN composite to 1.41MPa√m, which was an overall increase of 152% from the borosilicate glass sample. The addition of oxidized cBN increased the fracture toughness to 1.28MPa√m for a 40vol% oxidized cBN composite, an overall increase of 129% from the borosilicate glass. While the overall trend of the fracture toughness increase for oxidized cBN composites were lower than standard cBN composites, the difference between standard and oxidized composites was statistically insignificant.
SEM images were used to identify general features of the fracture surfaces and qualitatively identify two types of crack particle interaction, transgranular particle failure and interfacial failure, based on matched image pairs of the fracture surfaces. Considering only the frequency of transgranular particle failure on the fracture surfaces for 10 to 30vol% cBN composites was found to be 85% of all particle-crack interactions. The frequency of transgranular particle failure for 40vol% cBN composites was found to be considerably less due to lower relative density of these composites. Very little interfacial failure was observed.

A procedure was developed using digital image analysis software and SEM images to determine the areal density of cBN crystals on the fracture surfaces of standard and oxidized composites. This was used to determine how often a crack propagating through these composites interacted with the cBN particulate phase. It was found that for the cBN-borosilicate glass composite, where the CTE\textsubscript{cBN} > CTE\textsubscript{glass}, the crack preferentially propagates through the glass matrix, more than 85-90% of the total fracture surface, and avoids the particles. This is attributed to the residual stress field surrounding the particles due to the thermal expansion mismatch.

The quantitative analysis used to measure the areal density of cBN on the fracture surface can be used to explore other systems where there is an equal or opposite coefficient of thermal expansion from this system. In a previous study it has been found that when there is little to no elastic or thermal mismatch, a crack propagating through the composite is undisturbed on approaching the inclusion, and crack-particle interaction will
increase.\textsuperscript{58} If the areal density of inclusions on the fracture surfaces, whose $\text{CTE}_{\text{glass}} = \text{CTE}_{\text{cBN}}$, is measured and an increase in crack-particle interaction is found, it can provide more quantitative information and further provide evidence to existing theories on the effect of thermal and elastic mismatch in a composite.
Bibliography


