ELASTICITY AND FRACTURE OF DLC-COATED GLASS UNDER HERTZIAN CONTACT STRESSES

ELIZABETH CHRISTINE MILLER
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Reviewed and approved* by the following:

Carlo G. Pantano
Distinguished Professor of Materials Science and Engineering
Thesis Supervisor

David J. Green
Professor of Ceramic Science and Engineering
Honors Adviser

* Signatures are on file in the Schreyer Honors College
ABSTRACT

Coatings are applied to substrates for a variety of reasons ranging from improving the strength of the substrate to optical property modification. However, it is often unknown how the coating will affect the mechanical properties of the combined system. The aim of this thesis is to determine the mechanical properties of the coated and uncoated sides of a commercially available diamond-like carbon (DLC) coated soda lime silica float glass. The reduced elastic modulus of the uncoated glass and the effective reduced modulus of the DLC coated glass were determined using the Hertzian direct contact method, which allows for an elastic stress analysis. Controlled-load Hertzian cone cracking was used to measure the maximum load at failure and characteristic strength. The experimental results revealed that some variability existed in the coating and its properties across the surface, which was correlated with variable thickness as confirmed by characterization techniques such as Raman spectroscopy and scanning electron microscopy. However, this variability was not substantial enough to affect the ability of the coating to improve the mechanical properties of the glass. The DLC coated glass effectively exhibited higher strength, higher elastic modulus, and higher mean and median load at cone cracking than the uncoated glass.
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Chapter 1

Introduction

Coatings are applied to glasses for various applications such as improving scratch resistance, thermal control, optical property modification, and enhancing strength. Diamond-like carbon (DLC) coatings are often used as a scratch-resistant barrier on commercially available products such as supermarket scanner windows, compact discs, and digital video discs. These coatings may be applied in various ways depending on the desired application and properties of the film. However, little is known about how a DLC coating on a float glass affects the mechanical properties of the combined coating and glass system. The aim of this thesis is to determine the Young’s modulus, mean and median fracture load and stress, and the characteristic strength of a commercially available DLC coated float glass using Hertzian contact. Through this analysis, the effect of the coating on the substrate and the mechanical properties of the combined system will be determined.

Section 1.1 Literature Review

Coatings are applied to glasses for numerous reasons but are most commonly used to improve the mechanical properties of the glass. The actual strength of a glass is usually less than the theoretical value of strength due to surface flaws. Therefore, a coating can be useful in counteracting the weakening effect of these flaws by acting as a mechanical barrier and improving the properties of the surface. Coatings may be either hard coatings such as a ceramic or compliant coatings such as a polymer depending on the desired properties and applications.
The interaction between the coating and the substrate is essential to understanding how the coating behaves. Tensile stresses at the interface of the coating and the substrate can be minimized through thoughtful coating selection, taking into account thermal expansion mismatch, lattice matching, reactivity, and other physical properties, and by strongly bonding the coating to the substrate. A compliant coating can impart strength to a surface by applying stresses to the substrate which oppose the tensile stresses that occur during normal loading. Stiff coatings reduce the stresses in the substrate but increase tensile stresses in the coating. More load is required to deform the coated substrate than is needed to deform the uncoated substrate. Coatings also give rise to radial shear stresses at the interface of the coating and the substrate. Coatings can protect and improve the properties of the surface of the glass by creating a physical barrier, unlike treatments such as tempering or other methods that treat the glass itself. Also, coating is advantageous in that it is not limited by the geometry of a substrate.

Diamond is an interesting material for coating applications due to its high hardness. Several types of “diamond films” are used for coatings including chemical vapor deposition (CVD) diamond, amorphous diamond, and diamond-like carbon (DLC). Diamond-like carbon films are a combination of sp³ and sp² hybridized carbons with an amorphous network of carbon and hydrogen. The properties of DLC films are strongly dependent on the method of preparation, which affects the sp³/sp² bonding ratio and the hydrogen content. The film may be a mixture of polycrystalline and amorphous regions and vary in surface morphology and composition.

Several methods are used to apply DLC coatings to substrates depending on the desired scale and use. Ion beam deposition uses a beam of carbon or hydrocarbon atoms that are fired toward a substrate. The impact of the ions on the substrate induces sp³ bonding. Ion beam deposition can also be plasma-assisted where carbon ions are produced by plasma sputtering a graphite cathode. Mass-controlled ion beam allows for more controlled deposition of a single ion.
species at a well-defined ion energy. It is believed that the film which is being studied in this thesis was deposited via the plasma-assisted ion beam process.

Sputtering is a common method of deposition for large-scale industrial processes due to the ease of scale up. However, it does not produce the same quality of film as a more controlled process. The most common method of laboratory film deposition is plasma-enhanced chemical vapor deposition (PECVD) with a low bias voltage. The hydrogen content and thus the properties of a film deposited by this method can be easily tailored, depending on the sources of carbon.

Other methods such as cathodic arc and pulsed laser deposition can be used to deposit DLC films, but they are more rare and limited to laboratory application.

A “phase diagram” of carbon-hydrogen compounds and their nomenclature is summarized below in Figure 1-1.

![Figure 1-1. Ternary phase diagram of carbon-hydrogen compounds](image-url)
DLC films are used currently in applications such as grocery store bar code scanner windows, compact discs (CDs), and digital video discs (DVDs) as well as other commercial applications such as scratch-resistant table tops. The main application of DLC films at the present time is to form a scratch-resistant barrier on a given substrate, but it is not known exactly how the coating can be utilized to improve the mechanical properties of the substrate.

In this study, Guardian Industries’ DiamondGuard® glass, a commercial diamond-like carbon coated float glass, will be studied. Float glass as a general class of material is a soda lime silicate glass which is made by “floating” a glass ribbon on top of molten tin, resulting in a flat, smooth piece of glass. Because the glass is in contact with tin on one side and in contact with air on the open surface, the chemical composition of the two sides will vary, with one side exhibiting higher levels of tin oxide in a decreasing profile toward the bulk. The composition of both surfaces can vary from that of the bulk composition. The compositional differences between the surfaces may also affect mechanical properties such as the hardness and elastic modulus of each respective surface. However, this observation is still controversial as other studies have found that the compositional variations do not yield differences in the mechanical properties.

Diamond-like carbon coatings can be used as a mechanical barrier in that they resist scratching and have exceptional mechanical properties. The average modulus of a DLC film is reported to be between 99 and 112 GPa while the Poisson’s ratio is determined to be between 0.36 and 0.46. The hardness of DLC is taken to be 80 GPa whereas diamond is 100 GPa. However, as noted earlier, the elastic modulus, hardness, and other properties can vary depending on method of deposition as well as method of testing. The expected modulus of soda lime silica float glass is ~70.9 GPa with a Poisson’s ratio of 0.22 and a hardness of ~6.3 GPa. It is believed that the application of hard, high modulus coatings (DLC) to a softer substrate (glass) can improve the mechanical properties of the two-material system.
A useful method for testing the effectiveness of coatings is Hertzian contact with a hard spherical indenter. Hertzian contact has been used to study a variety of stiff coatings\textsuperscript{2,3}. In a hard coating, the stress field created by large-diameter indenters is elastic and is therefore easier to analyze than a plastically deformed material\textsuperscript{1}. The radius of contact $a$ between the spherical indenter and the surface of a linear elastic continuum is given by the equation

$$a^3 = \frac{4kPR}{3E_s}$$  \hspace{1cm} (1.1)

with

$$k = \frac{9[(1-\nu_s^2)E_i + (1-\nu_i^2)E_s]}{16E_i}$$  \hspace{1cm} (1.2)

where $P$ is the applied load, $R$ is the radius of the indenter, $E$ is the Young’s modulus, and $\nu$ is Poisson’s ratio. The subscripts S and I refer to the values for the surface and the indenter, respectively. As the applied load increases, the radius of the contact area increases accordingly. Measurements can be made until the body cracks to determine the load at failure or kept in the elastic regime for pure Young’s modulus measurements.

The maximum stress at failure $\sigma_{\text{max}}$, which occurs directly outside the contact area, can be calculated using Hertzian contact data according to the equation

$$\sigma_{\text{max}} = \frac{P(1-2\nu_i)}{2\pi a^2}$$  \hspace{1cm} (1.3)

where all parameters are the same as those defined in Equations (1.1) and (1.2). Surface stresses caused by the indentation are tensile and act as the driving force for ring and cone crack initiation\textsuperscript{10}. Maximum stress values can be used for failure analysis such as Weibull statistics and to determine the characteristic strength of a given material.
When the Hertz equations are applied, it is assumed that the material of interest is homogeneous. In the case of a coated structure, additional modifications need to be made to account for the two materials in the system. For simplicity of analysis in this study, it is assumed that the body is homogenous, as the coating is insignificantly thin in comparison to the contact diameter between the surface and the spherical indenter. Thus the data can be analyzed and an elastic modulus value determined for a given sample. However, the elastic modulus value that is calculated for the coated system using this method is thus an “effective” modulus of the combined coating and substrate system rather than a representation of the actual stress state in the glass substrate or the DLC coating individually. As part of this study, a theoretical calculation will be performed, as demonstrated by Liu et al\textsuperscript{12}, in which the Hertz theory is extended to account for a composite coated body.

**Section 1.2 Research Considerations**

**Section 1.2.1 Manufacturability**

Depending on the desired quality as well as the proposed use, the method by which DLC films are fabricated can vary. As mentioned in Section 1.1, a DLC coating can be applied via an ion beam deposition method, mass-selected ion beam, sputtering, cathodic arc, pulsed laser deposition, plasma enhanced chemical vapor deposition (PECVD), and other techniques. Some processes such as mass-selected ion beam and PECVD are more suited to laboratory experiments due to their complexity and ease of deposition control on a small scale. Sputtering is more desirable for industrial applications, as it is more versatile and easy to scale up for large bodies.

In today’s production methods, a coating is applied to protect the glass before it is transferred to another facility where the DLC is applied. During this transfer period, it is more
likely that the glass will be damaged, decreasing the strength of the substrate and possibly the effectiveness of the coating. If the need for handling was eliminated, it would be more likely that the substrate would be less flawed and that the coating would be well-bonded to the substrate. In addition, less energy and transportation costs would be expended, as the glass does not need to be transferred to another facility or machine.

If a method for the DLC coating application can be designed to be in the manufacturing production line, meaning that it does not need to be transferred to another facility or machine for coating purposes, the manufacturability of the product would be greatly improved. By decreasing the flaws on the surface to be coated as well as increasing the quality of the bonding, a better coating and combined system would result. However, even though additional processing is needed to apply the coating currently, the advantages gained from the DLC coating such as scratch-resistance far outweigh any negative effects that the handling may have on the glass substrate. In line DLC coating application would optimize the effectiveness of the coating.

Section 1.2.2 Economic Issues

Float glass is comparatively inexpensive and easy to produce as far as industrial glass products are concerned. Glass is durable, stable, and has well-studied properties. For most applications, a simple soda-lime composition of float glass is sufficient unless the application is highly specialized. Therefore, starting with a low-priced substrate allows for a variety of coatings to be applied to tailor the application of the glass.

By applying a coating to the glass, it may be possible to create a thinner yet stronger glass for a given application instead of using a thicker glass to increase failure load. By using less glass, the total cost of the raw materials may be decreased so long as the cost of the coating does not offset the decrease in the cost of the substrate. Also, glass requires a somewhat large energy
contribution to produce, so manufacturing a thinner glass will reduce the energy consumption required for production.

Because the DLC coating is carbon-based and does not include any expensive toxic metals, the starting material is reasonably priced when compared to rare earth metals and other costly materials that are sometimes found in coatings. By altering the process by which the coating is applied, simple carbon can take on numerous chemical structures and properties that open up a variety of industrial and commercial applications.

**Section 1.2.3 Health and Safety**

Diamond-like carbon coatings already find use in applications such as supermarket scanner windows, compact discs (CDs), and digital video discs (DVDs) where a scratch resistant coating is desired. Because of the carbon-based nature of the coating, DLC is safe for use in numerous applications with which humans come into contact everyday. Supermarket bar code scanner windows come in very close contact with food that will be consumed by humans, so it must have a safe and non-transferrable coating. CDs and DVDs can be found in virtually every home, so they too must be safe for such close contact with humans. The surfaces are coated with DLC and then an additional lubricant coating is applied on top of the DLC. The DLC acts to reduce scratching and potentially dangerous breakage.

DLC coatings can be applied to glass to prevent scratching of the surface. Scratches can significantly reduce the strength of glass, making it more likely to break and pose a hazard for handlers of the glass. Coatings such as DLC can be applied to glasses so that it will reduce extreme wear and prevent scratching, improving the safety of the glass substrate.
Section 1.2.4 Sustainability

Hertzian contact is a simple method for determining the elastic modulus of a given material. The experimental setup is straightforward and can be used repeatedly once the apparatus is built. Hertzian contact has been used frequently in the past for mechanical testing, so the technique is well-known and well-understood. Very little machining or cleaning is needed to prepare a sample. Because the contact is elastic until the body fails or unless unexpected plastic deformation occurs, Hertzian contact is a non-destructive way to determine the modulus and other properties of a given material. Therefore, once the material has been tested, it still can be used for its intended purpose.

Due to the size of the indentations (< 3mm in most cases), many indents can be made on a given area, meaning that only a small sample is necessary to glean large amounts of information. Therefore, less material is needed for testing purposes, so the tests can be repeated numerous times. Less material for performing the trials allows for a sustainable method of mechanical testing.

Carbon as a coating is a sustainable material, as carbon can be recovered from many sources. Because it is a readily available resource, it can be used sustainably for many years and easily altered based on the desired application of the DLC coating.

Section 1.2.5 Environmental Issues

As far as coatings are concerned, DLC is a more environmentally friendly alternative than coatings such as tin oxide, which contain toxic metals. Carbon is a readily available and sustainable material. Because DLC is a carbon-based film, disposing of a film when it has reached the extent of its usability results in the fairly innocuous product of CO₂. While large
amounts of CO$_2$ are not desirable for the environment, they can be more readily reabsorbed into the atmosphere and the environment or reprocessed more conveniently than heavy metals, which may form toxic compounds when recycled. Carbon disposal is also much more inexpensive than toxic metal removal, which requires special handling.

By applying a coating to a piece of glass and improving its strength, a thinner glass can be used for the same application as a thicker piece of glass. The production of glass uses a large amount of energy. Reducing the amount of glass that needs to be produced may reduce the total energy consumption needed to produce a given product, which is a step in the right direction for greener manufacturing processes.
Chapter 2

Experimental Procedure

Section 2.1 Statement of Work

Diamond- and ceramic-coated glasses are now commercially available and yet little is known about their contact behavior. The aim of this project is to assess the contact damage behavior of a diamond-like carbon coated glass and compare it to the uncoated glass. The project plan consisted of the following steps.

1. Use Hertzian contact to determine the contact area diameter dependence on indentation force for coated and uncoated glass. Determine the elastic modulus of uncoated glass and effective elastic modulus of coated glass.

2. Use Hertzian contact to determine the indentation load for the onset of cone cracking in coated and uncoated glass. Use Weibull statistics to compare data (maximum applied stress and failure probability).

3. Use SEM to characterize the microstructure of the coating and the crack path.

4. Use Raman spectroscopy to determine the chemical composition and thickness of the DLC coating.

5. Compile a thesis and poster presentation reporting the results of the study. Present results in the Department Poster Competition during the Annual Student Awards Convocation in April 2010.
Section 2.2 Elastic Modulus Measurements Using Hertzian Indentation

To determine the Young’s modulus of the DLC-coated and uncoated sides of the glass samples, Hertzian indentation was performed using a tungsten carbide (WC) sphere (E = 600 GPa, \(\nu= 0.23\)) with a nominal radius of 1 mm. The radius was measured using an optical microscope image and ImageJ and was found to be 1.014 mm. From this observation, it was assumed that the tip was spherical. The image of the indenter tip is shown in Figure 2-1.

![Indenter sphere and housing](image)

Figure 2-1. WC indenter ball as seen in an optical microscope

The apparatus for applying load for Hertzian direct contact consisted of a stepping motor (Klinger Scientific Model UE71, Garden City, NY) mounted on a steel block, which was used to apply load incrementally to a lever arm and thus to the sample. The load was measured by a load cell (Sensotec Model 31, Columbus, OH) with a 2.2 kN capacity and displayed on a load readout device (Sensotec GM Model 060-3147-01, Columbus, OH). As the load was increased manually, measurements of the contact diameter were made after every increment of approximately 10 N. Observations of the contact diameter were made throughout the loading and unloading process on an inverted microscope (Epiphot TME, Nikon Kogaku K.K., Tokyo, Japan). All glass samples
were cleaned with isopropanol before testing, and gridlines were marked on the sample in indelible ink to keep track of the locations of the indents. The setup of the microscope is shown below in Figure 2-2.

![Figure 2-2. Microscope apparatus for Hertzian contact modulus measurements](image)

An example of the measured contact diameter and Newton’s rings that surround the contact area is shown in Figure 2-3 on a sample of fused quartz. Observations of contact area are similar on the glass samples.
In order to accommodate the DLC coated samples, which were thicker than other samples that had been tested previously on this apparatus, a new stepping motor support setup was constructed. The well in which the sample sits above the objective lens was made deeper so that the microscope could focus on the top of the sample where contact diameter would be measured. Also, the steel rods, which support the stepping motor, were replaced with newer steel rods to ensure that the setup for applying load was level and stable.

Section 2.3 Cone Cracking Measurements

Cone cracking measurements were made on an Instron 5866 universal testing machine load frame (Instron Corp., Norwood, MA). All samples were cleaned with isopropanol before indentation. The load was applied at a rate of 1N/s starting from zero load. Acoustic emission sensors were used to determine the load at which the sample cone cracked. The trigger was set to register an audible signal between 40 and 50 dB, and when triggered, the testing frame would unload at a rate of 5 N/s to zero load. Indents were placed 3 mm apart center to center so that stress fields from other nearby indents would not interfere.
Figure 2-4. Hertzian contact setup for cone cracking

The setup for cone cracking is shown in Figure 2-4. Cracks were examined using an optical microscope to determine whether the acoustic emission trigger was indeed a cone crack. Images were taken using differential interference contrast (DIC) microscopy and a polarizer to capture the cone cracking below the surface, which is indicated by a change in brightness and hue.

The cone-cracked samples were evaluated using the Wyko NT1100 Optical Profiling System (Veeco Instruments, Inc., Tucson, AZ) to determine the depth of the cracks and the behavior of the coating and glass surface after cracking. Surface roughness measurements were taken to verify that the uncracked surface was smooth before any indentations were made.

The maximum load cone cracking data were analyzed using Weibull statistics. The maximum load was recorded when the acoustic emission signal was triggered, and if the sample had cone cracked at this point, the data was included in the analysis. Any indents that triggered the acoustic emission detector but did not show signs of a cone crack upon examination were not included in the Weibull analysis.
The best form of the Weibull equation is to use a least-squares fit to the data. The relation is linear, and this line is represented by the equation

$$\ln \ln \left( \frac{1}{1 - F} \right) = m \ln(\sigma) - m \ln(\sigma_0)$$

(2.1)

where \( F \) is the failure probability of a sample, \( m \) is the Weibull modulus, \( \sigma \) is the stress at failure of a sample, and \( \sigma_0 \) is the characteristic strength of the material. If data are plotted as \( \ln \ln \left( \frac{1}{1 - F} \right) \) versus \( \ln(\sigma) \), a straight line is obtained, and the Weibull modulus, or the slope of the line, can be determined easily.

The intercept of the equation for the Weibull plot is used to calculate the characteristic strength of the material based on the equation

$$\text{Intercept} = -m \ln \sigma_0.$$  

(2.2)

The intercept and slope were determined by linear regression.

The data that is included in the Weibull analysis consist of indentations that cone cracked during loading. Points that triggered but did not cone crack are not included in the data analysis. Any indents that did not produce an acoustic emission signal before the maximum load for the load cell (1 kN) was reached were included in the calculation of failure probability for all of the samples. However, they were not plotted on the graph for the determination of the Weibull modulus, as these data can only be treated as failing above a given load. As these samples were tested but did not crack, they need to be included in the total number of samples for the failure probability calculation, but because they did not fail, they not assigned a failure probability.

The median failure load and median stress at failure were determined by extracting the load of the middle sample in the Weibull rankings. The mean failure stress was calculated according to

$$\sigma_{\text{avg}} = \sigma_0 \Gamma(1 + \frac{1}{m})$$

(2.3)
where $\sigma_0$ is the characteristic strength, $m$ is the Weibull modulus, and $\Gamma$ is the gamma function. This equation was used to determine the mean failure stress rather than simply taking an arithmetic average to account for the samples that did not fail during the allotted test period. A simple average would not be a true representation of the mean failure load, as the uncracked points would either be omitted, making the average too low, or would skew the data to an artificially high average failure stress and load.

**Section 2.4 Characterization**

To understand the physical properties of the coating, characterization of the DLC coating and its interaction with the float glass substrate were needed. Raman spectroscopy was performed on the DLC coated glass to determine the thickness of the coating as well as the nature of its structure (i.e. $sp^3$ or $sp^2$). A scan was performed on the uncoated float glass to act as a background scan for the DLC coated sample. Scanning Electron Microscopy (SEM) was conducted on the samples to determine the physical properties of the coating including thickness. A FEI Quanta 200 Environmental SEM (FEI Company, Hillsboro, OR) was used to examine the cone-cracked indents from Hertzian direct contact to determine the crack paths and whether any additional information could be determined regarding the properties of the coating and its bonding to the substrate. A piece of DLC coated glass that had been manually scratched with a diamond scribe was examined for comparison.
Chapter 3

Results and Discussion

Section 3.1 Hertzian Contact Elastic Modulus Measurements

For the direct contact measurements, most samples were loaded until failure, but several samples on the DLC coated side were loaded only in the elastic regime in the interest of time. Failure is not necessary to determine the modulus of the material, but it does allow estimation of the maximum load at failure, which was a useful benchmark for the expected behavior in later cone cracking measurements. An example of loading until cracking on the DLC coated side of a sample is seen in Figure 3-1. The slightly brighter halo surrounding the ring crack on the surface is the cone crack, which extends below the glass surface.

Figure 3-1. Cone crack image from direct contact measurement on DLC
The contact radius as measured through the inverted microscope was plotted versus the load that was recorded from the load cell readout. In Equation 1.1, the relationship between $a^3$ and $P$ is linear so that the slope is equal to $\frac{4kR^3}{3E_s}$. By plotting the data in this fashion, the modulus can be determined from the slope of the line. A plot for the DLC coated sample is shown in Figure 3-2. A plot for the uncoated side of the sample is shown in Figure 3-3.

![Graph](image-url)

**Figure 3-2.** Radius of contact cubed versus load for a DLC coated sample
From the direct contact method on the inverted microscope, the reduced moduli of the uncoated side as well as the DLC coated side of the sample were determined according to the slope of the equation plotted in the $a^3$ versus $P$ graphs. This relation is given by the equation

\[
E_{RS} = \frac{3RE_{RI}}{4mE_{RI} - 3R}
\]  

(3.1)

where $R$ is the radius of the indenter sphere (1.014 mm), $E_{RI}$ and $E_{RS}$ are the reduced elastic moduli of the WC indenter and the contact surface, respectively, and $m$ is the slope of the line calculated from plotting the data. The results of the reduced modulus calculations are presented in Table 3-1.
Table 3-1. Reduced modulus values from Hertzian contact

<table>
<thead>
<tr>
<th>Sample</th>
<th>Reduced Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DLC Coated Glass</td>
<td>78.0 ± 10.</td>
</tr>
<tr>
<td>Uncoated Glass</td>
<td>70.6 ± 8.6</td>
</tr>
</tbody>
</table>

The reduced modulus value of the float glass corresponds well to the value of the elastic modulus reported in the literature. However, the reduced modulus of the DLC coated sample is much less than the elastic modulus values reported in the literature review for DLC (99 to 112 GPa). This decreased modulus value indicates that the contact of the indenter with the sample is not simply confined to contact between the indenter and the coating. The indenter strains the sample to a depth where the properties of the glass substrate beneath the coating are expressed in the data measurements. Thus, the modulus measurements are more indicative of the properties of the substrate with slight improvement due to the properties of the high modulus coating. As was mentioned previously, the Hertz equation applies to homogenous samples, so the experimental value is more of an effective modulus of the system than a representation of the coating itself. This distinction of the behavior of the coated system will be further explained in the theoretical model in Section 3.1.1.

Both data sets appear to generate linear plots. However, it was believed that there should be non-linearity in the graph of radius of contact cubed versus load for the coated side because the coating has a higher elastic modulus than the float glass. The data should reflect a decrease in modulus as the load increases and the stress field extends further into the substrate. On the graph of experimental data in Figure 3-2, this non-linearity is not observed, and the graph seems linear throughout, as was seen with Hertzian contact on uncoated glasses previously\textsuperscript{10} and on the experimental data taken on the uncoated side of the float glass. Low load nanoindentation may be needed to observe this hypothesis so that the deformation field is exclusively in the coating.
For one test on the DLC coated glass (Figure 3-4) and for one test on the uncoated glass (Figure 3-5), a second-order polynomial seemed to be a better fit for the data than the linear regression that had been successful for the majority of the data. Both of these trials were stopped before the sample had cracked due to the loading, as the maximum capacity for the direct contact system had been reached. The DLC coated sample cracked on unloading, but the uncoated sample did not. This observation indicates that the linear elastic fit which seemed to apply for most of the samples may not necessarily be accurate when the samples are subjected to high loads. Additional tests are needed to confirm whether this behavior is observed in other samples that experience high loads without ring or cone cracking and to determine why only a few samples exhibit this behavior. Other equations relating the radius of contact to the applied load may be needed to properly describe the loading behavior.

![Graph showing Radius of contact vs. Load](image)

**Figure 3-4.** Hertzian contact on DLC coated glass with polynomial fit
Section 3.1.1 Theoretical Calculation of Young’s Modulus

Because Equations (1.1) and (1.2) assume that the elastic contact is with an isotropic continuum and therefore cannot be applied to a coated substrate, further analysis was needed to determine how the contact area would vary with load in a coated system. To understand how a stiff coating on the surface of the glass should affect the effective modulus of the system, a theoretical analysis of the composite DLC coating-glass structure was performed. A stiff coating on a substrate can be modeled according to an extended Hertzian theory introduced by Liu et al\textsuperscript{12}. By setting parameters for the Young’s modulus and Poisson’s ratio of the coating and the substrate, the effective modulus of the two-material system can be calculated. All theoretical calculations were performed in MATLAB. The physical setup of the model is shown in Figure 3-6.
For the DLC, an elastic modulus of 100 GPa and a Poisson’s ratio of 0.22 were assumed, and for the float glass an elastic modulus of 70 GPa and a Poisson’s ratio of 0.22 were used, which are based on the experimental literature values\textsuperscript{9,11}. The value of Poisson’s ratio was chosen to be the same for both the DLC coating and the glass substrate due to the fact that on the sample, the coating is of trivial thickness when compared to the contact diameter. The coating is on the order of a few $\mu$m whereas the contact diameter is on the order of several hundred $\mu$m. Thus, when the sphere is in contact with the coated surface, it is not in contact with the coating exclusively, as the indenter applies stress to depths that would reach beneath the coating to the substrate. In this way, the indenter feels the influence of the glass substrate underneath.
Therefore, the Poisson’s ratio of the float glass is a better choice for the DLC coating than the Poisson’s ratio of the DLC itself.

For this model, a non-dimensional coating thickness, \( H \), is defined by the equation
\[
H = \frac{h}{a_{0s}}
\]  
(3.2)
for elastic substrates such as glass. The thickness of the coating, \( h \), was assumed to be 2 \( \mu \)m, which originated from a preliminary Raman spectroscopy spectrum of the DLC coated sample (see Section 3.4.1). The contact radius for the substrate and coating, denoted \( a_{0s} \) and \( a_{0c} \), respectively, are calculated using equation
\[
a_{0} = \sqrt[3]{\frac{3WR}{4E^*}}
\]  
(3.3)
where \( E^* \) represents either the reduced modulus for contact between the substrate and the indenter in the case of \( a_{0s} \) or the reduced modulus for contact between the coating and the indenter in the case \( a_{0c} \), assuming that the coating is infinitely thick. \( R \) is the experimental value of the indenter radius (1.014 mm), and \( W \) is the applied load. Load values ranging from 1 to 200 N were used for the calculation. The theoretical contact radius, \( a \), for the coated body for a given load can be calculated according to
\[
\bar{a} = a/a_{0s} = 1 + (\gamma - 1) \left\{ 1 - \exp\left[ -\frac{\pi^{2}}{8}\sqrt{\pi(H/d)} \right] \right\}
\]  
(3.4)
where
\[
d = 1 + (\gamma - 1) \left\{ 1 - \exp\left[ -\frac{\pi^{2}}{8}\sqrt{\pi(2H/(1 + \gamma))} \right] \right\}
\]  
(3.5)
and
\[
\gamma = a_{0c}/a_{0s}
\]  
(3.6)
The variable $\bar{a}$ is a dimensionless quantity normalized by values from the Hertz theory whereas $a$ is the contact radius of interest.

The contact radius cubed was then plotted versus load (Figure 3-7), and the slope of the line was determined in the same way as the Hertzian direct contact experimental data for modulus measurements.

![Figure 3-7. Plot of theoretical radius of contact cubed versus load](image)

The theoretical effective reduced modulus, $E_{RS}$, of the surface was then calculated using Equation (3.1). From this calculation, the effective reduced Young’s modulus of the surface of the DLC coating on the float glass substrate was determined to be 74.6 GPa. The experimental
value that was determined through Hertzian direct contact is 78.0 GPa. The two effective elastic modulus values are in good agreement.

The values that were chosen for the elastic moduli of the DLC and the float glass in this model were based on experimental values found in the literature. Because it is common that a range of values are reported in the literature, the values that were chosen were median values. Especially in the case of the diamond-like carbon, for which a variety of numbers are reported, the selection of slightly different elastic modulus values may have lead to a theoretical value that was more directly in line with the experimental values. For example, a higher quality, more homogenous DLC coating with higher sp$^3$ content will have a higher modulus value than a lesser quality film with higher sp$^2$ content. Careful selection of modulus values given more information about this specific coating may offer a truer representation of the actual coated system. Further experiments on this particular coating are needed to determine which modulus values would give the optimal theoretical calculated effective modulus value.

**Section 3.2 Cone Cracking**

To determine whether the signals that triggered the acoustic emission sensor were indeed due to cone crack formation, the indents were examined in the optical microscope in differential interference contrast (DIC) mode with a polarizer. A typical cone crack is shown in Figure 3-8.
In Figure 3-9, a cone crack on the uncoated side of the sample is shown. Similar characteristics to the other cone cracking images (color variation, concentric ring cracks on the surface) are seen in this image. Color variation in DIC mode is due to changes in depth in the sample and cracking beneath the surface.

Figure 3-8. Example of a cone crack resulting from Hertzian contact on DLC coated glass

Figure 3-9. Example of a cone crack on an uncoated glass sample
In Figure 3-10, an example of cone cracking at a very high load is shown. On this sample, the test was stopped at a load of 809 N, and the cone crack resulted from unloading. Spalling of the coating, mist and hackle from glass failure, and multiple ring cracks are visible. This image shows that at high loads, the coating may separate from the substrate, but it is not clear as to whether the separation is just the coating or whether the coating and some of the underlying glass are removed.

Figure 3-10. DLC cone cracking at high loads

In addition to visual inspection, optical profilometry was used to determine the morphology of the cone cracks. A typical profile scan for the DLC coated glass is shown in Figure 3-11, which cracked at a load of 555 N. The edges of the indent extend upward above the surface of the sample, which indicates surface uplift and pileup as a result of the cone crack. Also, in the bottom of the cone crack, a small indent remains after the load was removed, indicating
plastic deformation. The plastic deformation in the pit of the cone crack was observed on all of the DLC samples that were examined in the profilometer.

![Figure 3-11. Depth profile of typical cone crack on DLC coated glass](image)

A line scan of the uncoated cone-cracking indent is shown in Figure 3-12, which was loaded to 362 N. A similar surface uplift and pileup at the edges of the cone crack is observed in these indents, so the coating did not have an additional effect on the way in which the surface reacted to the cone cracking. However, the residual indent on the bottom of the cone crack is not observed in the uncoated glass cone cracks to the extent that is seen on the DLC cone cracks. Thus, while the surface is displaced downward in cone cracks on both samples, the surface of the DLC coating seems to be plastically deformed whereas the uncoated glass does not.
Section 3.2.1 Residual Indents in DLC Coating

Optical microscopy revealed that some of the samples had triggered the acoustic emission detector, which would indicate that the sample had cracked, but had no visible cone or ring crack. However, while no crack could be seen, a slight indent was present on the surface, which was indicated by changes in hue and brightness on the DIC optical microscope images, as shown in Figure 3-13. This change in brightness is indicative of a change in surface depth, which would indicate that while nothing is visible to the naked eye, an indentation or some other deformation did occur to trigger the acoustic emission detector. This observation is unexpected, as the contact was expected to be completely elastic so that no residual indents should be present after removing the load.
The presence of these shallow indents was confirmed using an optical profilometer. The depth profile shown in Figure 3-14 is of the indent pictured in Figure 3-13. This particular indentation is approximately 77 nm deep, as shown in the upper line scan, and 263 µm wide on the surface of the glass, as shown on the lower line scan. It is believed that these indents resulted from localized plastic deformation of the coating. This plastic deformation was unexpected, as the Hertzian contact method is believed to be a form of contact that creates elastic stress fields. However, the elastic recovery of DLC films has been studied previously, and it was found that recovery was not perfect for films with reduced sp³ content and increased sp² content. Therefore, the imperfect recovery of the coating could be indicative of increased sp² content, but further analysis is required to confirm this hypothesis.
Section 3.3 Weibull Analysis of Cone Cracking Data

From the cone cracking data, characteristic strength, median failure load, and mean and median failure stress could be determined. In the cone-cracking tests for the DLC coated side, several of the cone cracking indents did not trigger the acoustic emission detector by the time the limit of the load cell (1 kN) had been reached. The tests were stopped manually by triggering the acoustic emission sensor physically or by triggering the safety mechanism on the Instron for safety purposes. Some of these tests triggered on unloading, indicating the presence of a crack,
while some of the samples had no crack visible even after unloading. For the purposes of the Weibull plot, the indents that did not trigger the acoustic emission detector by the time the capacity of the load cell was reached were included in the failure probability data analysis. However, they are not shown on the plots themselves and were not used for the fitting of the equation to the data. On the uncoated side of the glass sample, all of the indents triggered the acoustic emission detector, and none of the indents needed to be stopped manually. All of the indents that resulted in cone cracks on the uncoated side are plotted on the graph.

For the calculation of maximum strength $\sigma_{\text{max}}$ on the Weibull plot according to Equation (1.3), a Poisson’s ratio of 0.22 was assumed for the coated substrate, and the Young’s modulus value that was determined via direct Hertzian contact was used. For the float glass, a Poisson’s ratio of 0.22 as well as the modulus that was calculated through Hertzian contact in this experiment was used. The value of 0.22 is the Poisson’s ratio for float glass that is found in the literature\textsuperscript{7}.

As demonstrated in the theoretical model, the same Poisson’s ratio was used for the DLC coated glass as was used for the uncoated float glass because the contact diameter between the WC indenter and the sample surface is on the order of several hundred $\mu$m, which is far larger than the thickness of the coating. The indenter sphere deformed the coating in such a way that its influence would have reached the glass substrate beneath the coating. With contact diameters that are orders of magnitude larger than the coating thickness, the behavior of the sample is essentially as if the coating were nonexistent. Therefore, a Poisson’s ratio of the DLC would produce characteristic strengths that are not representative of the true strength of the coated system.
The Weibull plot for the DLC coated sample is shown in Figure 3-15, and the Weibull plot for the uncoated sample is shown in Figure 3-16. The equation for the linear fit to the data from which the Weibull modulus is obtained is in the upper right hand corner of each plot.

Figure 3-15. Weibull plot of DLC-coated glass cone cracking data
Table 3-2. Weibull moduli for DLC coated and uncoated glass

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weibull Modulus (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DLC Coated Glass</td>
<td>14.1</td>
</tr>
<tr>
<td>Uncoated Glass</td>
<td>15.4</td>
</tr>
</tbody>
</table>
The maximum stress for the DLC coated sample that did not fail is 1.73 GPa at a load of 877 N. In Table 3-3, the characteristic strength, mean and median failure stresses, and the median failure load for both the DLC coated and uncoated glass are calculated.

Table 3-3. Characteristic strength, mean and median failure stress, and median failure load for cone cracking

<table>
<thead>
<tr>
<th>Sample</th>
<th>Characteristic Strength (GPa)</th>
<th>Mean Failure Stress (GPa)</th>
<th>Median Failure Stress (GPa)</th>
<th>Median Failure Load (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DLC Coated Glass</td>
<td>3.35</td>
<td>3.23</td>
<td>1.49</td>
<td>558</td>
</tr>
<tr>
<td>Uncoated Glass</td>
<td>1.33</td>
<td>1.29</td>
<td>1.29</td>
<td>357</td>
</tr>
</tbody>
</table>

The DLC coated glass resulted in a higher characteristic strength, higher mean and median failure stress, and higher median failure load than the uncoated glass for approximately the same data distribution, as demonstrated by the similarity in the Weibull moduli. The mean failure stress of the DLC coated glass is higher than the median failure stress because the median failure stress is calculated from the data points of samples which actually cone cracked while the mean failure stress is computed using the calculated characteristic strength value. From these values, it can be concluded that the coating is an effective method for improving the effective strength of the glass and enhancing its ability to withstand higher loads and stresses before cracking.
Section 3.4 Characterization

Section 3.4.1 Raman Spectroscopy

After examination of the failure loads, elastic modulus data, and observed differences in the behavior of the coating across the surface during mechanical testing, it was hypothesized that structural or physical heterogeneity existed in different areas of the coating. Raman spectroscopy, which has been used previously to examine carbon films, was used to determine the bonding structure and thickness of the DLC film.

In the Raman spectrum in Figure 3-17, the peaks at ~1500 cm\(^{-1}\) and ~2700 cm\(^{-1}\) correspond to peaks for graphite, and peaks at ~1350 cm\(^{-1}\) indicate diamond. These two sets of peaks confirm that there exists a mixture of sp\(^2\) and sp\(^3\) bonded carbon in the film. The broadness of the peaks represents a lack of crystallinity and greater disorder in the bonding, which is to be expected from the more amorphous DLC. The small peaks at ~1100 cm\(^{-1}\) and ~600 cm\(^{-1}\) in the DLC on glass spectrum (due to the glass) were not observed everywhere and varied in intensity, suggesting either small holes in the coating or very thin regions of the coating in which the glass was exposed.
In addition to exploring the bonding distribution in the DLC, Raman spectroscopy was used to estimate the thickness of the coating. Using the area of integration around 1500 cm\(^{-1}\), a depth profile was generated for the DLC as seen in Figure 3-18. From this profile, it can be determined that the coating is 1-2 µm in thickness with measured values ranging from 0.6 µm to 4 µm. These differences of the DLC-glass interface may be more analytical than real. Further analysis is needed to confirm these observed differences in coating thickness.
Section 3.4.2 Scanning Electron Microscopy

To determine if further information could be learned about the coating properties, the cone-cracked Hertzian direct contact samples were examined in an ESEM. From the overview of an indent, as shown in Figure 3-19, it is observed that the failure resulted in multiple ring cracks on the sample surface, which was also seen in the optical microscope. In addition, the coating appears to have chipped off around the edge of the crack during failure.
Upon closer inspection of the edge of the crack, the chipping of the coating was seen more clearly. It is believed that pieces of the coating were sticking up out of the ring crack (Figure 3-20), but they could not be examined in such a way as to glean more information about the properties of the coating. Hackle was found on the chipped edge of the crack, seen in Figure 3-21, which is characteristic of glass fracture.
Figure 3-20. Close up SEM image of ring cracks and chipped coating

Figure 3-21. SEM image of hackle around cone crack edge
For comparison to the micrographs of the cone cracked samples, a piece of DLC coated glass was scratched with a diamond scribe and examined in the ESEM. Light pressure was not sufficient to penetrate the DLC coating to the substrate. However, upon applying strong pressure to the surface, the coating separated from the substrate, seen on the right side of Figure 3-22, revealing the glass underneath the coating. Because the coating removed glass when it separated from the substrate, it can be stated that the coating was well-bonded to the glass.

A well-bonded coating exhibits better properties than a coating that is not as well-adhered to its substrate. This bonding could account for the fact that even though the coating exhibited

Figure 3-22. SEM image of manually scribed DLC coating
variability in thickness, it did not seem to affect the ability of the coating to improve the properties of the glass substrate.

While the information about the coating’s bonding is useful, SEM did not reveal as much about the properties of the structure of the coating as was desired. Additional characterization will be needed to determine the behavior of the coating after cone cracking and the coating’s microstructure.
Chapter 4

Conclusions

Hertzian elastic contact methods were used to calculate the elastic modulus of the coated and uncoated sides of a diamond-like carbon (DLC) coated glass sample. Controlled-load Hertzian contact was used to determine cone cracking behavior and maximum failure loads, which were analyzed using Weibull statistics. From these experiments, it can be determined that the DLC coated glass sample has higher strength and a higher Young’s modulus than the uncoated glass samples. The median failure load and mean and median maximum failure stresses during cone cracking are higher in the DLC coated glass than the uncoated glass, which corroborates that the strength of the coated glass is higher. All measured properties are representative of the effective behavior of the coated glass, which is a combination of the properties of the DLC and the float glass. In addition, the experimental data for the effective Young’s modulus was consistent with the extended Hertz theory suggested by Liu et al. Characterization of the coating suggests variability in coating thickness and structure, but the variability is not substantial enough to severely affect the ability of the coating to improve the mechanical properties of the glass. It can be concluded that the DLC is effective as a mechanical barrier to enhance the properties of the float glass.
Chapter 5

Future Work

Further characterization of the coating is required to determine the extent of the variability in thickness and chemical structure. To determine the mechanical properties such as elastic modulus and stress in the coating for the coating exclusively, nanoindentation, which would not deform the substrate in the way that the large indenter sphere does during Hertzian contact, may be performed. With improved knowledge of the coating itself, an optimized theoretical calculation of the effective elastic modulus of the DLC coated glass can be obtained. Also, additional parameters of the coating and the combined DLC coated glass system such as fracture toughness can be calculated for further mechanical analysis. Studies of the interface between the DLC and glass may also prove useful to understand the coating-substrate interactions and effect of these interactions on property measurement.
Chapter 6

References


ACADEMIC VITA OF
ELIZABETH C. MILLER
ecm5054@psu.edu

Education

The Pennsylvania State University, University Park, PA
Scholar in the Schreyer Honors College
Bachelor of Science in Materials Science and Engineering
Minor in Chemistry
Semester of Graduation: May 2010

Relevant Courses:
Processing of Ceramics, Ceramics Laboratory, Thermal Properties of Materials,
Crystal Chemistry, Materials Characterization, Optical Properties of Materials,
Electrical and Magnetic Properties, Materials Process Kinetics, Thermodynamics of
Materials, Mechanical Properties of Materials, Phase Relations in Materials Systems,
Materials Engineering Methodology & Design, Modern Physics, Process Quality
Engineering, Technical Writing

Work Experience

DAAD Research Internships in Science and Engineering (RISE) Program, Leibniz
Universität Hannover, Hannover, Germany
RISE Intern, May 2009 – August 2009
• Created a process for the preparation of a ceria-yttria-zirconia ternary system from
  raw powders for use in dental ceramics
• Modified laboratory machinery for use with zirconia ceramic materials and
  produced pressed and sintered samples for testing
• Characterized the ceramic samples using SEM, XRD, and EDS to determine if a
  single phase system had been created

International Materials Institute for New Functionality in Glass, Lehigh University,
Bethlehem, PA
REU Summer Researcher, May 2008 – August 2008
• Developed a method for the design and manufacture of Fresnel micro lenses in
  chalcogenide glass thin films
• Used photolithography and wet etching to fabricate lenses and characterized
  samples using optical microscopy, X-ray photoelectron spectroscopy, and other
  techniques
• Wrote an abstract for the National Science Foundation and co-authored a paper
  titled “Chalcogenide glass thin film resists for grayscale lithography” which was
  presented at SPIE Advanced Lithography in February 2009

Charles River Laboratories, Horsham, PA
Intern, June 2007 – August 2007
• Inspected animal tissue specimens from pre-clinical trials for sample integrity
• Organized, inventoried, and maintained tissue and formulation samples in the
  laboratory archives
• Completed training in Standard Operating Procedures and Good Laboratory
  Practices
Honors

- ASM International John M. Haniak Scholarship
- Pittsburgh Chapter of the American Ceramic Society J. Earl Frazier Memorial Scholarship
- C. Phillip Cook, Jr. Memorial Scholarship in Ceramic Science and Engineering
- Sam Zerfoss Memorial Scholarship
- George L. Ellis Scholarship - Two Year Recipient
- Schreyer Ambassador Travel Grant Recipient
- Dean’s List - Spring 2007, Spring 2008, Fall 2008, Spring 2009, Fall 2009
- North Penn Area Scholarship - Two-Year Recipient
- National Merit Scholarship Commended Scholar

Papers


Conference Presentations


Activities

  - Team Leader, First-Year Student Academic Committee: Spring - Fall 2009
- Material Advantage (Student Organization of ASM, TMS, ACerS, and AIST): Spring 2008 - Present
  - Secretary: Fall 2009 - Spring 2010
- Teaching Assistant, MatSE 402 - Materials Process Kinetics: Spring 2010
- Keramos: Spring 2009 - Present
- Penn State Sinfonietta: Spring 2007 - Spring 2010
- Penn State Dance Marathon (THON) Rules and Regulations Committee Member: Fall 2009 - Spring 2010
- Schreyer Honors College Career Development Mentor: Fall 2009 - Present
- Society of Distinguished Alumni Mentoring Program: Spring 2009 - Present
- Mentoring with Honors Program: Spring 2009 - Present
- Fresh START Day of Service Volunteer: Fall 2006
  - Team Leader, Fresh START Day of Service: Fall 2007, 2008, 2009
- AIChE THON Team: Fall 2006 - Spring 2008
  - Chair, Family Relations: Spring 2007 - Spring 2008
- American Institute of Chemical Engineers (AIChE): Fall 2006 - Spring 2008