MATERIAL HEALTH MONITORING OF SiC/SiC LAMINATED CERAMIC MATRIX COMPOSITES WITH ACOUSTIC EMISSION AND ELECTRICAL RESISTANCE

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MATERIAL HEALTH MONITORING OF SiC/SiC LAMINATED CERAMIC MATRIX COMPOSITES WITH ACOUSTIC EMISSION AND ELECTRICAL RESISTANCE

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Thesis

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ABSTRACT

Ceramic matrix composites (CMC) composed of Hi-Nicalon Type S™ fibers, a boron-nitride (BN) interphase, and pre-impregnated (pre-preg) melt-infiltrated silicon / silicon-carbide (SiC) matrix have been studied at room-temperature consisting of unidirectional and cross-ply laminates. Quasi-static, hysteretic and uniaxial tensile tests were done in conjunction with a variety of temporary, laboratory-based material health-monitoring techniques such as electrical resistance (ER) and acoustic emission (AE). The mechanical stress-strain relationship paired with electrical and acoustic measurements were analyzed to expand upon current composite knowledge to develop a more fundamental understanding of the failure of brittle matrix laminates, their constituents, and interactions. In addition, a simple but effective method was developed to allow visual confirmation of post-test crack spacing via microscopy. To enhance fidelity of acquired data, some specimens were heat-treated (i.e. annealing) in order to alter the residual stress state. Differences in location, acoustic frequency, and magnitude of matrix cracking for different lay-ups have been quantified for unidirectional and [0/90] type architectures. Empirical results shows complex hysteretic mechanical and electrical behavior due to fiber debonding and frictional sliding of which no general model exists to capture the essence of this CMC system. The results of this work may be used in material research and development, stress analysis and design verification, manufacturing quality control, and in-situ system and component monitoring.
I would like to thank the GE team for their support in this research: Dave Johnson, Doug Carper, Rob Fecke, and Greg Wilson. Kent Smith also was helpful with the microscopy. Emmanuel Maillet, Chris Baker, and Matthew Appleby, and Craig Smith for general CMC education, as well as being available for open conversations and ideas. I also want to thank Emmanuel for his excellent advice on graduate school and research, and also for developing the MATLAB tools that made much of this work possible.

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CHAPTER I

INTRODUCTION

Materials that are strong, light, tough and corrosion resistant at high temperatures are very desirable for engineering applications, but few materials have all of these characteristics. Metals have toughness that ceramics lack, and ceramics can operate at high temperatures (creep resistant), where metals cannot. Nickel-based superalloys with thermal and environmental ceramic coatings are the current-state-of-the-art high-temperature, load bearing material system that can operate above the metal substrate’s melting temperature (~1100°C). At that temperature, a combined cycle gas turbine will operate at 60% fuel efficiency [1][2][3]. However, due to demands for reduced fuel consumption, lighter and hotter engines are required, especially in aviation. Fortunately, a material that is 33% lighter and can withstand up to ~1315°C [4][5] exists that is expected to reduce fleet fuel consumption by up to 3% if fully implemented[4]. Silicon Carbide ceramic composites are a promising, modern engineering material that have proven to outperform many competing materials for specific applications, namely aviation and land-based turbine engines. However, significantly more research is needed to validate the strength and durability of ceramic composites to safely design products with them. Thus, the focus of this work will be understanding the non-linear stress-strain mechanical properties of a specific type of CMCs, namely GE’s Hipercomp®.

The customizability and performance of composites is undoubtedly superior to that of metals, but invariably increases complexity in testing and designing with such material systems. Discerning where damage begins and how it propagates up to the point of ultimate failure is of the upmost importance. Understanding the material properties for a wide variety of composite systems has been of extreme
interest since the inception of engineering composites. Macroscopic, component level, homogenized mathematical models do not capture the micromechanical activity present in CMCs due to an oversimplification of the actual material studied. These assumptions may be reasonable with a two-phase, fiber/polymer matrix system, but fails to capture the details of the more complicated three phase composite system (fiber/interphase/matrix) composites studied here. On the other hand, modelling at an atomistic scale is computationally infeasible at this point in time. A multi-scale, physics-based model is most likely the best approach for predictive CMC design[6]. However, these methods are very complex and so only simple micromechanical models will be used to investigate the materials studied here.

With any modelling approach, experimental validation is critical. Structural/material health monitoring or non-destructive evaluation in a laboratory (as opposed to a component in service) can offer insights that traditional testing (e.g. tensile test) cannot. Figure 1 exemplifies the complexities of composites, where each stage has challenges in and of itself.

![Figure 1 – Size scales to consider when evaluating mechanical properties of materials and structures [6]](image)

This work focuses on using material health monitoring techniques adapted for temporary laboratory use to determine holistic understanding of damage inception and progression of the material system. Monitoring electrical resistance and acoustic emission at various stress states provide information that can
“fill in the gaps” of the scale level for understanding composites. Once the physical mechanisms are identified, prognostic models can then be developed for an arbitrary system to be used by designers to develop safe, functional components.
Ceramic matrix composites (CMC) are modern, engineered, hybrid material structures that combine the refractory properties and stiffness of a ceramic with the strength and toughness of a composite. CMCs are typically a laminated unidirectional, woven textile, braided, or chopped whisker ceramic fiber that is inundated with a ceramic matrix[7]. CMCs are being designed to replace current generation high performance metals for demanding environments, such as single-crystal nickel-based superalloys, due to their ability to endure higher operating temperatures (up to 1400°C) where creep, oxidation and corrosion are the dominant failure mechanisms (Figure 2) [3][5].

Figure 2: Temperature operating range (SX=single crystal Ni superalloy, DS = directionally solidified superalloy, TBC = thermal barrier coating, CMC=Ceramic matrix composite) [5]

There are a variety of CMC material families, such as oxide and non-oxide. Oxide/Oxide (fiber/matrix) composites are commonly alumina (Al₂O₃), mullite (2Al₂O₃·SiO₂), or zirconia (ZrO₂) fiber with
an alumina matrix. They are currently being researched as an alternative to non-oxide CMCs due to their resistance to corrosion, lower cost, and thermal stability, but currently cannot compete with the superior mechanical properties of non-oxide CMCs[8]. Non-oxide CMCs are currently the most promising material for high-temperature, high stress applications with properties of low porosity, low density, lower atomic diffusion, high thermal conductivity, low thermal expansion, high relative toughness, and high matrix cracking stress[9], [10]. Silicon carbide (SiC), specifically SiC fibers with SiC matrix (SiC/SiC), has been established as one of the leading materials to meet these requirements [10].

Realistic, long-term applications of current CMC technologies are land and aviation turbine components(Figure 3) and nuclear reactors[11] [9].

Figure 3-Sectioned view of a 107 MW GE 7FA industrial gas turbine with potential applications for CMCs highlighted [9]

Non-Oxide CMC Processing

The constitutive properties (e.g. mechanical and electrical) of fiber and matrix components of CMCs are highly dependent on the processing methods used. A brief overview will be given of the manufacturing methods for CMCs.
Carbides (e.g. SiC), nitriles (e.g. BN), are relatively modern materials that are commonly used in non-oxide CMC’s[12]. Silicon carbide is desirable due to its stiffness and strength at room temperature, as well as excellent refractory properties[12]. Silicon carbide can be synthesized by mixing powders at high temperature and then compacting, which can be ground into powders and then used in a sintering process or melting at a high temperature.

SiC fibers are manufactured through a pyrolysis process of heating polymer fibers containing silicon up to 1400°C in a N₂ vacuum environment achieving fiber diameters of 8-15µm[12]. These fibers are then cured and heat treated by sintering or pyrolysis. An alternative processing technique is through chemical vapor deposition (CVD), but this process typically produces a larger SiC fiber diameter of about 50 µm, which limits the flexibility of the fiber and ultimately architectures[10].

Once the fibers have been produced, the composite can be constructed from unidirectional, woven, or even braided fibers. Among many other methods to process CMCs, the materials studied in this work were processed by liquid silicon infiltration or melt infiltration (MI). In the MI process, a chemical vapor infiltration (CVI) layer of carbon (C) is deposited. Then, the porous region of the material absorbs liquid silicon or silicon based alloy by capillary action. During absorption, a chemical reaction between the liquid silicon (Si) and carbon create silicon carbide (SiC) forms the matrix of the CMC.

However, there are difficulties with MI type processes. The process temperature is high (1500°C), which can be costly as well as cause issues with fibers thermally reacting with the liquid. Also, the liquid silicon is corrosive with regards to the fiber and thus requires a protective coating, typically of Boron Nitride (BN), which has also been found to serve as a mechanical and chemical diffusional barrier. Another issue with MI is premature pore closure when the liquid is absorbed, leading to undesirable porosity (MI is still much lower porosity than all other processing methods). Finally, in MI processes, large volumes of the CMC can be exclusively SiC and/or Si matrix, which reduces the high temperature performance and creep properties due to low fiber fraction and large areas of pure silicon-which has a relatively low melting temperature. Despite these drawbacks, the MI process offers very low porosity and high thermal conductivity[11].
Fiber coatings have been shown to reduce chemical attack during processing as well as provide a weak mechanical interface between the fiber and matrix that is low modulus, thus relatively flexible, and is capable of deflecting or blunting propagating cracks destined for the load bearing fibers\[9\]. As mentioned, Boron Nitride (BN) is a common coating, which is applied using a low pressure CVD. A visual comparison of a SiC/SiC composite can be seen in Figure 4.

![Figure 4](image)

**Figure 4** -a) Slurry cast MI Hi-Nicalon-S™/BN/SiC [13],b) CVI SiC/SiC Hi-Nicalon-S™, no interphase [14]

**Mechanical Properties of CMCs**

The mechanical properties of CMCs can vary greatly but can be controlled depending on the processing method used and constituent chemistry. It is of great importance for the material to have high tensile strength and to retain those properties at high temperatures. These materials must primarily exhibit high strength, thermal stability, creep resistance and oxidation resistance. Second to those is the desire to have fibers with small diameters, smooth surfaces, high thermal conductivity, low cost, and a pure material lacking impurities(like carbon)[10].
Figure 5 – Monotonic and unload/reload stress-strain plots exemplifying hysteretic behavior of an 8 ply woven CMC [15]. Tau is the interfacial shear stress that transfers load at a matrix crack between the fiber and the matrix.

The challenges in understanding material properties becomes apparent when the linear-elastic region is exceeded with CMCs. A simple monotonic tensile test can yield complex data that is not easily understood without advanced material health-monitoring tools. An even more confounding test is performing hysteresis loops (unload/reload), where many different properties of the material can be extracted with some effort (Figure 5).

As seen in Figure 6, the mechanical response of a monotonic load has been broken down into 4 regimes[9]. Regime I is the linear-elastic where no damage has occurred and the constitutive linear-elastic relationship of solid materials, \( \sigma = E \varepsilon \) (stress = stiffness * strain) holds. Regime II consists of non-linear stress-strain response due to matrix cracking and fibers debonding from the matrix, bridging the matrix cracks. The proportional limit is exceeded here and toughness is achieved where brittle fracture otherwise would have occurred for monolithic ceramics resulting in catastrophic failure. Fibers also continue to
debond which afford the material large strains. Regime III and IV are often indistinguishable, where transverse ply and longitudinal ply matrix cracking saturation may be achieved, fibers begin or continue to fail, until composite failure.

Figure 6 – Typical stress-strain curve for HiPerComp™ 8-ply cross-ply laminated CMC system [9]. Proportional limit stress is a mechanical indication of yielding.

CMC toughness can be roughly compared to the ductility of materials capable of yielding, where stress is redistributed within the specimen as damage occurs[16]. Analogously, individual fiber or matrix failure redistributes the load in a composite and will eventually “gracefully” fail as opposed to a catastrophic failure without any sign of damage beforehand. To make CMCs practical, toughness has been designed into them by using a composite system with fibers that are stronger than the matrix. While the matrix is cracking, the fibers bridge the crack and continue carrying the load. Interestingly, all the CMC constituents are linear-elastic to failure (as are all monolithic, brittle materials), but, when a CMC is properly designed and manufactured, the composite exhibits highly non-linear mechanical properties with permanent deformations, making the damage mechanics nebulous, even under simple monotonic tensile loads [16].
One may expect that if a CMC material does not exceed a yielding point (a 0.005% strain proportional limit offset is used for ceramics), then no damage has occurred. However, acoustic emission is almost always recorded below the proportional limit, indicating damage is occurring and, as previously mentioned, material health-monitoring tools are necessary to detect this. And as with any mechanical component in real-world applications, unexpected loads due to foreign object damage, fatigue, and unknown stress-concentrations need to be accounted for with safety factors and composite toughness.

Fiber properties

SiC fiber properties are customizable to perform optimally in specific applications. For general use, continuous length polycrystalline fibers have been found to perform well under a variety of thermal and environmental conditions but are less capable in high thermal and oxidation environments than single crystal fibers. Fibers with small diameters (<15µm) allow for complex braids and weaves that would otherwise be impossible with a thick, stiff fiber. In addition to flexibility, the small diameter polycrystalline fibers cost less to produce than single-crystal fibers.

Two types of fibers are typically used in CMCs are carbon and SiC. Carbon is a low-cost fiber that has a wide range of thermal and mechanical properties. Unfortunately, carbon fibers are anisotropic with a large, positive coefficient of thermal expansion radially and small and sometimes negative axially, which can lead to early cracking or debonding due to manufacturing [11]. Worse yet, carbon fibers oxidize at relatively very low temperatures (≈450°C), which degrade and are subsequently useless for load bearing at high temperatures. A leading alternative of carbon fibers is SiC, which is mechanically and environmentally superior, but is substantially more expensive. Common SiC fibers and properties can be found in Table 1.

<table>
<thead>
<tr>
<th>Property</th>
<th>iBN-Sylramic Fiber</th>
<th>CVI-BN</th>
<th>CVI-SiC</th>
<th>MI-SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s Modulus, GPa</td>
<td>RT</td>
<td>1204 °C</td>
<td>RT</td>
<td>1204 °C</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.17</td>
<td>0.17</td>
<td>0.22</td>
<td>0.22</td>
</tr>
<tr>
<td>Density, g/cm³</td>
<td>3.2</td>
<td>---</td>
<td>1.4</td>
<td>---</td>
</tr>
<tr>
<td>Coefficient of thermal expansion, 10⁻⁶K</td>
<td>4.6</td>
<td>8.0</td>
<td>5.2</td>
<td>10</td>
</tr>
<tr>
<td>Thermal conductivity, W/m-K</td>
<td>43</td>
<td>21</td>
<td>3.1</td>
<td>1</td>
</tr>
</tbody>
</table>

CVI is chemical vapor infiltration, MI is melt infiltration, and RT is room temperature.
Current generation of available SiC fibers (e.g. Hi-Nicalon-Type S™) exhibit excellent thermal properties due to being processed at high temperatures (1600-2000°C), also making it compatible with the high processing temperatures of the MI manufacturing process [11]. Other issues with SiC fibers are oxygen induced porosity, crystal size, and processing impurities. Fibers produced at lower temperatures form small crystals and have poor creep resistance which can be combated by heat treatment grain augmentation[10]. Porosity can be detrimental to the fiber by reducing density, thermal conductivity and tensile strength. Larger grains perform better at higher temperatures but reduce thermal conductivity and dramatically increase production cost[10]. It has also been found that increased grain sizes produce rougher surfaces which may introduce new fiber failure modes due to the limited fiber slip during fiber/matrix debonding.

Matrix properties

Unlike many other composites, the CMC matrix is typically a high modulus material which is capable of carrying large loads. SiC matrix properties can vary, but commonly used values are presented in Table 1 . The hard, brittle nature of SiC makes it an ideal candidate for high temperature applications, but without a reinforcing fiber, would perform very poorly due to its low fracture toughness (2.5-3 MPa m^1/2)[18]. SiC has an extremely high melting point, but starts to oxidize around 1000°C and the maximum usage temperature is about 1500°C due to oxidation or reduction[12].

Interphase properties

A traditional fiber/matrix composite with optimal constituents is often a resilient, robust material given proper application. However, in extreme environments, where high temperatures and stresses are typical and possibly erratic, as well as a variety of somewhat unpredictable reactive chemical interaction, highly engineered materials are required for not only proper functionality, but for survival in the most severe conditions. To be a viable high-performance material, CMCs require more than a traditional composite design, and the fiber/matrix with a weak interphase can protect the composite (namely fibers) from premature failure. Various interphase materials have been tested, such as hex-BN, and pyrocarbon (PyC). BN will oxidize into borosilicate glass (B₂O₃) in typical operating conditions which is more oxidation resistant as compared to carbon [19], [20], making BN a superior interphase material with respect to
carbon. Carbon suffers from large scale oxidation at medium temperatures which evacuates the carbon in a
gaseous form of CO or CO$_2$ leaving voids or the fiber unprotected.

BN is a soft material and is not meant to carry load, but to deflect and/or blunt propagating cracks
from penetrating the load bearing fibers preventing premature composite failure[11]. BN is a very
compliant material (~20 GPa) with low shear strength which serves as a load transfer medium and crack
energy absorption that may otherwise damage the fibers. Typical interphase thicknesses are 1µm, as
compared to a common HNS fiber diameter of 15µm[20]. An important characteristic of a composite is its
ability to transfer load from the matrix to the fibers, which is determined by the interface bond of the
fiber/matrix interaction. In this case where an interphase exists, by the characteristics of the interphase
between the matrix/interphase and fiber/interphase[21].

CMC Damage

In essence, material damage of composites is a loss of stiffness (Figure 6, Figure 7), where energy is
irreversibly dissipated through internal cracks. On a global/macro scale, this can be generalized as energy
transfer in a thermodynamic continuum. Considering composite damage at this scale, extensive
experimentation and empirical fits to continuum damage models are necessary [22]. A more fundamental
approach is to consider the physical micromechanisms (e.g. fiber debond, frictional sliding, thermal stress,
etc.) of fiber and matrix interaction. Micromechanics attempts to actually capture the microscopic physical activity of the material before, during and after damage.

![Load vs. Displacement Graph](image)

**Figure 7** – Macroscopic damage visible from a stress-strain curve does not capture physical damage phenomenon [23]

The plethora of failure criteria for isotropic metals can put into perspective the difficulty in categorizing composite damage mechanisms and the criteria to follow. For example, the envelope for the common Tresca yield criteria for ductile materials is based on the maximum shear stress of the material[24]. Comparing this to composite structures, dozens of failure criteria may need to be applied for a single component based on whatever damage is expected. Inferring from stress-strain from Figure 7, some form of damage is occurring but no more information is discernible. As mentioned, to properly interrogate the material as the damage is occurring requires structural health monitoring or non-destructive evaluation, and even then there is room for different interpretation. Newer, state-of-the-art methods exist for determining damage types, such as in-situ CT scanning (Figure 8) of a loaded composite, allowing for visual confirmation of the damage types[25], but is very difficult and costly.
Figure 8 - In-situ composite damage detection CT monitoring of a dogbone CMC specimen [25]

Understanding the complexities of damage will lead to a better understanding of the material and the mathematics behind modeling. The dominant damage types of composites are [23][26]:

- 0° ply matrix cracking (crack plane parallel to fiber and load)
  - Dominant damage type that is the source of the non-linear stress-strain relationship.
  - Crack densities in the 0° plies are typically higher than transverse plies due to the higher stresses achieved.

- 90° ply matrix Cracking (crack plane perpendicular to fiber and parallel to load)
  - Often the first damage that occurs is transverse (90° ply) matrix cracking. The crack may or may not penetrate the 0° ply.

- Fiber debonding
  - When 0° ply matrix cracks form, the matrix must slide with respect to the fiber, and this large strains are achieved without fiber breakage. Dependent on 0° ply matrix cracking

- Interphase damage
The interphase with likely degrade and wear with load, especially if fatigue tests are performed. Interphase damage likely contributes to the permanent strain of a cycled material. Environmental degradation, or oxidation is also expected.

- **Fiber Breakage**
  - If material reaches matrix cracking saturation then globally, all the load is carried by the fibers. If matrix cracking saturation is not achieved, then matrix cracking will occur at the same time as fiber failure

- **Composite failure**
  - If the matrix is fully saturated, then composite strength is driven by fiber strength. If the fiber cannot debond from the matrix, low strains may result and little toughness is to be expected

**CMC Modeling**

Various scale levels can be considered when modelling (Figure 9). Practical engineering modeling cannot exist at to atomistic scale currently, so the smallest scale considered when concerning composite components is the micro-scale, where the physics of the interaction of fiber/matrix/interphase is considered. At the mesoscale, a single lamina composed of many fibers is considered. And finally, at the macroscale, an entire component is within scope.

![Figure 9 - Various scales to consider composites](image)

[27]
State-of-the-art multi-scale models begin at the micro-scale, and by informing the next scale larger, can provide fidelity as well as efficiency[27]. However, many different methods of modelling are available, where each have their strengths and weaknesses. Of the various modelling methods, two main categories are evident, top-down or implicit, and bottom-up or explicit models [28][23].

Top-Down Modeling

Top down method is inherently a macroscopic perspective of the material system, and so is a simpler approach that basically approximates the constitutive properties of the material based on empirical data. It is often employed to model extremely complex material systems (e.g. composites). This method is good for a simple, prototype model or single scenario, but is not robust and the predictive capability disappears when used in general material models. One issue with top-down models is that, while simpler to model, is very costly to develop a statistically significant database of experimental results. This method is favorable as material systems increase in complexity.

Bottom-up Modeling

A more fundamental approach is bottom-up modelling. Derived from first-principles, this approach is preferred in many cases, however, depending of the scale considered (micro,meso,macro), computation can become prohibitive. Micromechanics is a popular method for studying composites at a constitutive level for elastic properties as well as damage[22]. Micromechanics of Damage Models (MDM) considers discrete damage types without the need for adding “fitting parameters” by capturing the physics of the composite elements and their interactions[22]. When considering composites at such a fine scale, understanding fiber/interphase/matrix interaction is the goal.

Classical lamination theory provides a simple analytical solution to displacements (which can be solved for stress and strain) given constitutive properties and dimensions of the thin laminated plates[29]. One weakness with modeling orthotropic composites with plates is the assumption of plane stress, where the out-of-plane stress is assumed zero. For thin plates, this assumption is typically valid. With isotropic plates, this is somewhat trivial, but as the complexity increases by introducing anisotropy and stacking plates into a single laminated plate, the resultant stresses are not always obvious. While higher order
theories exist, linear classical laminate plate theory will be used in this work to determine the elastic properties due to its simplicity.

CMCs are tough due to the failure strain of the fiber being much larger than that of the matrix, which allows the matrix to crack and the fiber to continue carrying the load to high strains. Fracture toughness is a desirable characteristic in engineering materials, but is challenging to achieve when the constituents are brittle, leading to modelling difficulties. Also, CMC matrices can carry a large portion of the load and can experience large stresses and crack, as opposed to PMCs where fibers carry the vast majority of the load and has little matrix cracking. The foundation for many micromechanical CMC models are based on the fracture energy-based theories of Aveston, Cooper, and Kelley (ACK) [30]. If the failure strain of the fiber is less than that of the matrix, a single failure will occur with little toughness. However, if the converse is true and matrix cracking occurs (and doesn’t damage the fibers), then the material will achieve some level of toughness-a non-linear increase in strain proportional to stress. Matrix cracking or composite failure is predicted by the differences in energy changes between the fiber and the matrix. It assumes the system of fiber and matrix only are held together by frictional forces. Other have expanded and generalized (Budiansky et al) ACK to crack propagation and steady-state matrix cracking. The scale at which these models are built are only considering a single fiber, and surrounding matrix (Figure 10).
Table 2 – Variables defining material properties and model parameters subsequently used

<table>
<thead>
<tr>
<th>Variable</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>c, f, m, l</td>
<td>Subscript denoting, composite(c), fiber(f), matrix(m) and interphase(l)</td>
</tr>
<tr>
<td>E</td>
<td>Elastic modulus</td>
</tr>
<tr>
<td>v</td>
<td>Poisson ratio</td>
</tr>
<tr>
<td>τ</td>
<td>Interfacial shear stress (sliding resistance)</td>
</tr>
<tr>
<td>V</td>
<td>Fiber-volume fraction</td>
</tr>
<tr>
<td>r</td>
<td>Fiber radius</td>
</tr>
<tr>
<td>s</td>
<td>½ Crack spacing</td>
</tr>
<tr>
<td>x'</td>
<td>Fiber matrix load transfer length or debond length</td>
</tr>
<tr>
<td>σT</td>
<td>Thermal stress</td>
</tr>
<tr>
<td>x'T</td>
<td>Slip due to thermal stress</td>
</tr>
<tr>
<td>u</td>
<td>Crack opening displacement</td>
</tr>
<tr>
<td>y</td>
<td>Counter slip on unload/reload</td>
</tr>
</tbody>
</table>

Figure 10 - Micromechanical model for fiber-matrix interaction. \( x' \) = transfer length, \( 2s \) = crack spacing, \( \tau \)=interfacial shear stress, \( u \) = crack opening [31]

The undamaged composite will have a ½ crack spacing of \( s = \infty \). As cracking occurs, \( s \) will converge on the final crack spacing (inverse of crack density), as determined via microscopy (Figure 12). The applied load is partially carried by the matrix, but when cracks form, will be distributed via a shear stress (\( \tau \)) onto the fibers. When sufficiently far from the crack plane, the matrix will realize the far field stress of the
composite. This assumption shows that the stresses are transferred linearly between the fiber and matrix (Figure 11).

As the crack spacing decreases, the stress in the matrix is reduced until below the matrix strength, which is when matrix crack saturation has occurred. Any additional strain the composite experiences will be due to fibers slipping within the matrix. ACK also claims that during a load/unload test, the modulus will change as a function of crack spacing between \( E_c \) and \( E_f \). The energy dissipated during a single fracture is trivial and is evident by the near linear stress-strain data exhibiting no toughness. The more interesting case is multiple fracture, where energy dissipation of the composite can be large. The material can achieve a large strain to failure and have similar properties of classic engineering metals such as pseudo-yielding.

ACK assumes initially assumes unbonded fiber-matrix interaction, where the fibers may move freely through the matrix without resistance provided by the matrix carrying some load[32]. In later work[30], a perfect bond between matrix and fiber is assumed and calculations for debond length are shown. In this theory, radial and tangential stresses are neglected in the matrix. These stresses may be significant when multiple cracking occurs and the mechanical bond, which is limited by the shear strength of the matrix, is broken and a friction fiber/matrix bond is controlling debond length.

More recent attempts of modeling the mechanical behavior of brittle-matrix ceramics have been done, and most build on the work of Aveston et al. Among many existing models, the Pryce and Smith (PS) unidirectional composite model is simple, yet captures the essence of mechanical properties and damage based on crack spacing contributing to stiffness reduction and residual strain, and will be the focus of most of the modelling work here. As previously mentioned, the toughness seen in CMCs is due mostly by the matrix cracking and shedding their load to the fibers. As more matrix cracking occurs, more toughness is achieved and the load is shifting in and out of the matrix. The PS model cleverly captures the physics of matrix cracking and frictional debond in a general form without the need for model “fitting” the models to experimental data. A number of important assumptions allow the model to be predictable, but simple:

- Constant, unknown interfacial shear stress \( \tau \) (stress profile plots linear)
- Even crack spacing with no crack interaction
- Model is valid only below crack saturation ($x' < s$)
- Fiber/matrix slippage is perfectly reversible (zero debond stress)
- Crack opening displacement is negligible to the overall displacement compared to the fiber strain in the debond region

Figure 11 - PS model showing a large crack spacing and a saturated crack spacing. Model is invalid for $x' > s$ [33]

The large strains that CMCs are capable of are a function of the matrix cracks and fiber extension within these cracks. An interesting point to make is that the crack opening (Figure 10, $u$) is negligible, and this is shown by the fiber stress coming to a point in the profile plots (Figure 11, Figure 12, Figure 13). Also, there is no entry debond stress, which is apparent from the “continuous” fiber stress profile where no stress step exists at the bounds of $x'$. When the PS model reaches matrix crack saturation ($x' > s$), the composite modulus will be the modulus of the fiber, or $E_c = E_f V_f$. The model then becomes invalid because it is impossible to have a load transfer length greater than the crack spacing. Another important assumption is that $\tau$ does not change on cycling, which in reality it most likely degrades on each cycle due to a reduction of surface roughness and changes on the properties of the interphase[33]. This would lead to an increase in hysteresis loop width. Some of these shortcomings are addressed in other modeling attempts [34][35] but add unnecessary complexity for this introductory analysis.
Figure 12 – Experimental Data presented by PS used in the model. s is the ½ crack spacing, and x’ is the load transfer length from the matrix to the fiber [33]

Clearly, at large stresses, the cracking rate slows and the material approaches crack saturation (about 190 MPa in Figure 12) where the load transfer length is equal to ½ the crack spacing. The transfer length (sometimes referred to as the debond length, x’) increases with peak load.

Figure 13 - Loading stress profile of fiber peak stress (solid), fiber unload/reload stress (dashed), x’ = thermal induced transfer length, y = counter slip distance on either unloading or reloading, s = ½ crack spacing, x’ = transfer length of stress from fiber to matrix. Fiber contraction due to the difference in radial stress [33]
CMC fatigue or hysteresis tests are more complex than monotonic testing due to fiber/matrix slippage (compare Figure 11 to Figure 13). To casually conceptualize the stress in the matrix and fiber on a damaged composite with unloading and reloading may be counterintuitive. At peak stress, the exposed fiber in the crack plane experiences the largest fiber stress in the composite, but on unload, the peak fiber stresses move away from the crack plane due to frictional forces. This is shown schematically in Figure 13. Upon inspection of unload stress-strain data and stress-ER (Figure 5, Figure 22), this complex relationship is helpful to understanding less idealized models not considered in this work.

\[ \sigma_f(x) = \frac{\sigma_c}{V_f} + \frac{2\tau x}{r} \]

Equation 1 – \( \sigma_c \) = applied stress, \( V_f \) = fiber volume fraction , \( r \) = fiber radius , \( \tau \) = interfacial shear stress , \( x \) = distance from crack plane. The stress in the fiber is maximum mid crack plane, and reduces as the following function.

\[ \sigma_f^T = \frac{E_f E_m V_m}{E_c} \Delta T (\alpha_f - \alpha_m) \]
\[ \sigma_m^T = \frac{E_f E_m V_f}{E_c} \Delta T (\alpha_m - \alpha_f) \]

Equation 2 – Residual Fiber and matrix Thermal Stress as a function of volume fraction, moduli, change in temperature, and coefficient of thermal expansion. \( \Delta T \) = stress-free temp – final temp = \( T_0 - T_f \),

\[ \sigma_f = \sigma_E \frac{E_f}{E_c} + \sigma_f^T \]

Equation 3 – Far-field fiber stress including fiber thermal stress

\[ \sigma_m = \sigma_m \frac{E_m}{E_c} + \sigma_m^T \]

Equation 4 – Far-field stress of the matrix including matrix thermal stress

\[ E_c = V_f E_f + V_m E_m \]

Equation 5 – Rule of mixtures for unidirectional composite modulus, \( E_c \)

\[ \chi = \frac{r}{2\tau} \left[ \sigma_m \frac{E_m V_m}{V_f E_f} - \sigma_f^T \right] \]

Equation 6 – Fiber to matrix load transfer length, linear function of unknown \( \tau \)

\[ \varepsilon(s, \sigma) = \frac{\sigma}{E_c} + \frac{r}{8\tau E_f} \left[ \sigma^2 + \sigma_{peak}^2 \left( \frac{E_m V_m}{E_f V_f} \right)^2 - 4\sigma_{peak}^2 \frac{E_m V_m}{E_c V_f} + 2(\sigma_f^T)^2 \right] \]

Equation 7 –PS model strain on loading as a function of ½ crack spacing \( s \), applied stress, \( \sigma \), applied peak stress, \( \sigma_c \)
\[
\varepsilon(s, \sigma) = \frac{\sigma}{E_c} + \frac{r}{8s\varepsilon_f} \left[ \left( \frac{E_mV_m}{E_cV_f} \right)^2 (\sigma_{\text{peak}}^2 - \sigma^2 + 2\sigma\sigma_{\text{peak}}) - 4\sigma_{\text{peak}}\sigma_f^T \frac{E_mV_m}{E_cV_f} + 2(\sigma_f^T)^2 \right]
\]

Equation 8 – PS model strain on unloading

Equation 7 and Equation 8 allow for linear-elastic region (first term) as well as the non-linear cracking (second term) to be modeled in one equation. Some observations of this model show: an un-cracked specimen has an infinite crack spacing, the smaller the crack spacing the larger the strain becomes proportional to the stress, and once \( s' \) is equal to \( \frac{1}{2} \) crack spacing, \( s=s' \), then matrix crack saturation has occurred and the effective composite modulus becomes \( E/V_i \). Also notice that the loop width of the stress-strain curve grows with cycling, which is due to the increased slip length. This model is very sensitive to \( \tau \) and \( \sigma_f^T \) and counter each other. However, the shear stress \( \tau \), crack spacing \( 2s \), and residual stress \( \sigma_f^T \) are assumed to be known. While the PS model is simple due to the assumption of evenly spaced cracks and no interaction between them, it is robust at predicting unload and reload stress, which can be non-trivial in CMCs.

Figure 14 – Example of CMC hysteresis model with the validity criteria shown and cracking scheme described by a weibull distribution to fit the reduction of crack spacing. The invalid region is the minimum stress where the transfer length is greater than the \( \frac{1}{2} \) crack spacing.

The unidirectional model of PS gives reasonable results, however, unidirectional composites are not particularly useful in application. A more useful system is a cross-ply laminate, which is what this work focuses on. In order to model damage of cross-ply layups, simultaneous and possibly somewhat independent damage occurs in the \( 0^\circ \) and \( 90^\circ \) plies, and coupling the damage progression is a difficult task. Constituent properties, architecture, and manufacturing quality can vary the damage modes, but in
general, for balanced, symmetric cross-plies, a particular damage sequence occurs. It has even been reported that multiple proportional limits are visible with cross-plies, the first when 90° ply reaches crack saturation and the final when 0° plies saturate[36].

A more appropriate model for the materials in this work would be a cross-ply rather than the unidirectional model that was used. Many analytical cross-ply CMC models exist, but the Pryce and Smith version was considered[37], however it lacked many details necessary to accurately describe the material of this work. Some useful attributes that were included are: independent, simultaneous variable crack densities in 0° and 90° plies, ply load interaction via shear-lag. Major shortcomings are: Cracks are per ply and do not interact with other plies, thermal stresses are neglected, and does not include unload/reload properties. This model was developed in the late 20th century before widespread computation resources were available, so more modern modelling methods (finite element, generalized method of cells) are currently available and being pursued.

Hybrid Modeling

The finite element (FE) approach offers a robust method for approximate solutions of stress and strain for composites, but still lacks the fidelity to supplant other methods. It should be considered a complement to testing and a first attempt test-bed for constitutive information of composites. FE is poor at predicting fiber debonding, but good at structural, macroscopic scale. The micro and meso scale should be modeled via the analytical method[17]. While FE is a powerful tool, the power only lies in the ability of the solutions to be accurate, which is often debatable due to the solutions being numerical (often linear) approximations, and thus must be carefully analyzed for validity. FE can be used as a micromechanical method as proposed by Sun et al [38], but is computationally costly. A good approach to modeling is to use analytical solutions to validate FE solutions at various size scales. To aggregate the constituent properties, Table 1 shows the constitutive relationships to experimentally determined data. These results can be used to determine homogenized lamina properties. This method could also be done based on laboratory experimental results, but the finite element method is usually a reasonable alternative.
Table 3 - Experimental method to determine the 5 independent material constants for a transversely isotropic lamina [39]. Also requires $G_{23} = E_2 / (2(1+v_{23}))$

<table>
<thead>
<tr>
<th>Applied</th>
<th>Measured</th>
<th>Calculated</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma_{11}$</td>
<td>$\varepsilon_{11}$, $\varepsilon_{22} = \varepsilon_{33}$</td>
<td>$E_{11} = \sigma_{11} / \varepsilon_{11}$, $\nu_{12} = -\varepsilon_{22} / \varepsilon_{11} = \nu_{13}$</td>
</tr>
<tr>
<td>$\sigma_{22}$</td>
<td>$\varepsilon_{11}$, $\varepsilon_{22}$, $\varepsilon_{33}$</td>
<td>$E_{22} = \sigma_{22} / \varepsilon_{22}$, $\nu_{21} = -\varepsilon_{11} / \varepsilon_{22}$, $\nu_{23} = -\varepsilon_{33} / \varepsilon_{22}$</td>
</tr>
<tr>
<td>$\sigma_{12}$</td>
<td>$\varepsilon_{12}$</td>
<td>$G_{12} = \sigma_{12} / (2\varepsilon_{12})$</td>
</tr>
</tbody>
</table>

An alternative to FE micromechanics is a closed form approach called generalized method of cells (GMC). By discretizing the material into a repeatable element, constitutive equations for arbitrary number of materials that make up the composite can be determined and analytically solved[40]. This method was utilized in this work due to its robustness and concise software package available.

Continuum damage models (CDM) and dissipated energy density (DED) methods are built on the assumption that damage is a form of energy dissipation and can be modeled via a thermodynamic continua and that the system is much larger than one individual crack. Variables are used to represent damage types and the relationships are derived based on thermodynamic laws. Typical parameters are matrix cracking, interface (fiber-matrix) debonding and sliding, and fiber breakage. The stiffness variation in hysteresis tests shows the degradation of stiffness as a function of maximum stress achieved, as well as a function of cyclical stress, which a continuum model may be sufficient at describing. The toughness visually seen in a stress-strain plot is dissipating energy (Figure 7), but the continuum approach does not elucidate where, thus is somewhat of a nebulous approach to understanding CMCs.

Hybrid multi-scale models can include many of the previously mentioned methods for composite modelling (analytical, fracture mechanics, FE, GMC, etc). To properly define a constitutive relationship of a complex material, such as a CMC, multi-scale models may be required for a holistic solution. This would include accurate representation of reality at the micromechanical, mesomechanical, and macro-mechanical level. One such hybrid model is described by Benardycyk and Arnold [41][42]. This method event includes stochastic fiber breakage. Xia et al has shown that a multi-scale model in concept is a sound approach[6]. FE models do not produce reliable results when modelling the micromechanical effects of a
fiber/interphase/matrix interaction due to a prohibitive number of elements required, making the model inconceivable to solve at this time in computing. However, analytical methods that consider fracture energies of constituents are powerful at the micromechanical level, and can be coupled with finite elements at a macroscopic level that uses the micro results and homogenizes the properties to a macro structure. Another alternative to macroscopic model is with continuum damage models that provide the framework for developing constitutive properties for materials on a macro scale.

Material Health Monitoring

Traditional engineering materials, such as metals, and even plastics, have been relatively simple to design with and have predictable properties: yield-strength, ultimate strength, isotropic stiffness, predictable damage. Ductile materials are less sensitive to impact damage, and if damage has occurred, it is visible to the human eye (cracks, deformation, etc.)[43]. As design requirement become harder to achieve, innovative materials must be developed to overcome the limitations of traditional materials. Composites offer many improved material characteristics, but lack the niceties of classic materials, such a damage detection, simplicity, and cost. This has lead materials engineering into a new era of a data driven paradigm to which customers want instant feedback of the health status of the material in question. A myriad of testing techniques are available, each with positive and negative qualities[43]. Some are more suited for material prototype characterization in a laboratory, while others are actively used in quality inspection for mass-produced items, or in-situ material health monitoring.
Figure 15-An holistic structural health monitoring system in which data is acquired, models are developed and damage is predictable [44]

When studying material damage, an organized approach is necessary to ask the right questions to get the right answers. Is there damage, where is it, what kind is it, how severe, and how long will the structure last are the important questions to answer[45]. Figure 15 shows a general flow of how structural health monitoring can be used to improve a component or structure, from research and development to application. Sound failure models and structural models are necessary for a reliable and safe monitoring system. When using structural health monitoring in a live application, diagnostics help determine the state of the structure, where as in a laboratory, the monitoring techniques and data collected are used to prognose and validate models in development. A similar tool for material interrogation is non-destructive evaluation (NDE). With NDE, the inspection equipment is portable and inspects the material in intervals when not in service. As opposed to health monitoring, which is a permanent, continuous, in-situ, monitoring system [44]. To be clear, many techniques used in this work are referenced as health monitoring where they may in fact be NDE methods due to the nature of the test being destructive.
For this work, two health monitoring methods are used: acoustic emission, and electrical resistance. Acoustic emission is a sensitive, powerful tool that can sense the inception and subsequent of damage in a material. While acoustic emission has been in use for decades, modern computing has made modal acoustic emission possible due to the memory, processing and software requirements. Typically tests can generate many gigabytes of data and often complex digital signal processing methods are employed to glean information. Thus, using acoustic emission outside of a laboratory would be challenging.

Electrical resistance is a relatively new structural health monitoring method that has promise to be used in laboratory as well as in a production environment as a quality metric. It is a low cost, simple measurement that, depending on the material, is very sensitive to damage type and severity.

Acoustic Emission

As a material is stressed, the strain energy stored in the material will increase, and as long as the material remains linear-elastic, the energy remains reversible. The first acoustic event is important because the material is irreversibly dissipating energy through sounds waves (acoustic emission). Most are inaudible, so high-fidelity sensors are necessary to capture the often microscopic events. Monitoring
acoustic events gives a deeper understanding of the damage progression such as, location, magnitude, and frequency.

Figure 16-Traditional AE event captured with a narrow-band transducer with typical parameters

Traditional acoustic emission (or Resonant Sensor Parameter, RSP) methods include narrow-band resonant frequency transducers that parameterize the event with important characteristics such as oscillation counts, amplitude, energy, etc. (Figure 16). While this requires less memory and processing, one major drawback of RSP is that one wave mode is assumed, which is a typically a poor assumption. RSP also makes some analysis nearly impossible, such as fast-fourier transform (FFT) and source location determination due to poor fidelity. In reality, an acoustic event contains a wide range of frequencies from superposed waves, thus modal acoustic emission method is necessary. With modal AE, wide-band transducers (50-2000 kHz) capture and digitize acoustic events which are available for post-processing. From the waveforms (Figure 17), two distinct wave types are apparent: the extensional, or dilatational wave, and the flexural, or shear wave. The dispersion curve (Figure 18) shows the wave velocities as a function of frequency. This work only considers thin-walled specimens, or in engineering terms, a plate with a thickness less than the wavelength. In plates, the dominant wave modes are extensional and flexural, and most other wave types simply do not propagate. This is reasonable.
because the fundamental, or lowest order wave, contains most of the energy compared to the higher order waves.

Figure 17 - Modal Acoustic event (MAE) wide wideband frequency sensors suposed with a traditional acoustic event (TAE) [46]

Dispersion is an important concept when considering wave transmission. As seen from Figure 17, both wave types travel at different velocities based on the wave type, and frequency. From plate theory, the equations for the wave velocity can be derived. Equation 9 shows that wave velocity is only a function of modulus and density (for lower order forms), which is important because an in-situ instantaneous elastic modulus can be determine based one the speed of sound. Figure 18 extensional plot shows higher order effects at high frequencies. For flexural waves, dispersion is significant [47]. This highlights why modal AE waveforms can look complex and require advanced signal processing techniques to digest. An important note is that the stress wave in plates is assume to have free edges, which is violated here due to the presences of 3 transducers, an extensometer and ER clips. It is unknown if the mass of the sensors are significant to affect the wave propagation.
Figure 18-Typical Dispersion curve for CMC. Assumes isotropic plate theory, $E=200$ GPa, $\nu=0.15$ [15]

All equations are from [47] and only first order. $E = \text{elastic modulus}$, $\rho = \text{density}$, $h = \text{plate thickness}$, $\omega = 2\pi f$, $f = \text{frequency}$, $\nu = \text{poisson ratio}$

\[ C_e = \frac{E}{\sqrt{\rho(1 - \nu^2)}} \]  

Equation 9 – Extensional stress wave velocity

\[ D = \frac{Eh^3}{12(1 - \nu^2)} \]  
\[ C_f = \left( \frac{D}{\rho h} \right)^{1/4} \sqrt{\omega} \]  

Equation 10 – Flexural stress wave velocity, $D = \text{plate bending stiffness}$

A typical AE setup is shown in Figure 18. Three sensors are placed along the length of a dogbone specimen. When any of the three sensors detects a perturbation of a specified amplitude, all three sensors “lock-out” and record a specified number of samples from the same event. Once the recording is complete the system is rearmed and “listens” for new events. Properly setting the trigger gain and signal gain is possible through trial and error using pencil lead breaks on the surface and is critical to capturing useful, unsaturated waveforms. The choice of sampling rate is important when using modal AE. If the sampling
rate is too slow, (less than 2x the maximum expected waveform frequency), aliasing will occur and the digitized waveform will not be accurate [48].

Figure 19 – A single AE event arrives at sensor 2 first, then sensor 3 and 1. Due to dispersion, the higher velocity extensional wave reaches the other sensors before the flexural waves and is much smaller amplitude than the flexural wave [46]

Once the data are recorded, several useful calculations can be made. The Energy of a waveform gives a general sense of the magnitude of the event, but may have errors due to varying coupling sensor methods and saturated waveforms.

\[ \text{Energy} = \int V^2 \, dt \approx \frac{1}{\text{sample rate}} \sum_{n=0}^{N-1} V_n^2 \]

Equation 11 – Energy, \( N \) = total samples

The Frequency centroid is a simple metric to simplify the frequency content of the waveform

\[ \text{Frequency Centroid} = \frac{\sum_{n=0}^{N-1} f_n \cdot x_n}{\sum_{n=0}^{N-1} x_n} \]

Equation 12 – Frequency Centroid (FC) of sample \( n \), \( f \) = frequency, and \( x \) = magnitude of fast-fourier transform

Location is very dependent on the speed of sound in the material and the accuracy of the waveform onset, or time-of-arrival (TOA). The TOA of each sensor can be calculated by several methods. The threshold technique is one of the easiest methods, where the first voltage to exceed a threshold
voltage will be the TOA. More advanced techniques have been developed and are preferred due to their ability to avoid false triggers due to erroneous noise before the actual event\[49\], but require calibration. Once the TOAs are calculated, location is determined. From Figure 19, once all the TOAs are calculated, the 1-dimensional location along the length of the specimen is calculated by Equation 13.

\[
\text{location} = C_e \left( \frac{t_{oa_2} - t_{oa_1}}{2} \right)
\]

Equation 13 – time-of-arrival calculation calculated from the extensional speed of sound and the arrival times of the wave on the top and bottom sensor.

Since the extensional wave velocity \(C_e\), is a function of the material modulus \(E\), an in-situ modulus measurement is possible based on an accurate location determination. From work by Morscher [15], a correlation is made of the material modulus and speed of sound, as shown in Figure 20.

![Figure 20](image)

Figure 20 – Experiments showing the relationship of in-situ material modulus and extensional wave velocity [15]

Acoustic Emission has also been correlated with crack density of mini-composites [50] as well as macro-composites [51]. Plotting cumulative acoustic emission and crack density (acquired via microscopy)
against stress, Morscher has shown that cumulative AE energy is a good approximation of crack density (Figure 20) when the final crack density is known.

Figure 21 - crack density is well-correlated to cumulative acoustic emission in a woven CMC [50]

Recent work has been published to link failure mechanism to AE frequency data for carbon-fiber reinforced polymers (CFRP) using peak-frequency and pattern recognition [52]. Results are not definitive, but it is loosely shown that fiber failure types are a higher frequency and matrix cracking and delamination are lower.

Electrical Resistance

Electrical Resistance may be the most promising in-situ material health monitoring technique due to its low cost and simplicity to measure, yet little work has been done to make it useful and available to industry. Possible uses are to enhance material research and development, stress analysis and design verification, manufacturing quality control, and in-situ system and component monitoring. Current work includes unidirectional and cross-ply composites and resistance change due to various damage mechanisms in play. Mechanical models are being developed with the current understanding of damage progression for unload-reload tests in mind [53]–[55]. Empirical results shows complex electrical behavior that may require a physics based mechanical model for frictional fiber debonding post matrix crack and crack closure for subsequent loading cycles. A large body of experimental results for electrical
measurements exists for woven CMCs done, largely by Morscher et al. Resistance health-monitoring is also ideal for high-temperature applications due to the material itself being the sensor, as opposed to ancillary sensors.

Despite the complexity of electrical transmissions in materials, (especially laminated composites), several models have been developed to predict loading history and possible damage mechanisms.

Morscher et al have developed a model that predicts ER as a function of applied load[55]. Modelling ER enhances the understanding of damage progression in CMCs by providing an additional avenue to validate micromechanical models based on physical, complex damage phenomenon. As Morscher describes, the damage properties of CMCs are driven by in-plane transverse matrix cracks, and so, having a sensing technique that is in the same plane will yield the most accurately predicted damage state. The final transverse matrix crack density was determined via post-test microscopy, and intermediate crack densities were assumed to be a function of the cumulative acoustic energy (Figure 21). It is hypothesized that the large change in resistance at high stresses (where little AE is recorded) is due to the loss of electrical contact radially between the fiber and the matrix.

Figure 22 – Time and Stress plots of Electrical Resistance and Cumulative crack density [55]

The model consists of a parallel and series circuit representing the fiber, and matrix properties as well as a variable resistor that represents the electrical contact resistance and decoupling between the fiber and matrix.
Figure 23 - Schematic of electrical model for unidirectional composite, \( N_c = \) number of cracks, \( R_r = \) radial contact resistance, \( R_f = \) crack bridging fiber resistance, \( R_f^x = \) fiber resistance imbedded in matrix, \( R_{Si}^x = \) Silicon resistance [55]

A few important aspects that are neglected in the model may or may not account for the recorded error. Uniform crack spacing (no crack interaction) monotonic loading, and no fiber breakage assumptions should be incorporated in future versions of an ER model.
CHAPTER III

EXPERIMENTAL PROCEDURE

Ceramic matrix composites (CMC) composed of Hi-Nicalon Type S™ fibers, a boron-nitride (BN) interphase, and pre-impregnated (pre-preg) melt-infiltrated silicon / silicon-carbide (SiC) matrix have been studied at room-temperature (Figure 24). Test specimens were acquired from GE Aviation (Cincinnati, Ohio) that were either a balanced and symmetric cross-ply or unidirectional laminated architecture. The cross-ply has surface plies aligned with the loading direction, (0/90)2s, or oriented transverse to the loading direction, (90/0)2s. Unidirectional specimens are 8 plies with fibers in the same direction, or a 08 architecture. Specimens were machined to a dogbone shape and have an overall length of 152.4 or 203.2 mm. During the final phase of manufacturing the laminates, molten silicon is infiltrated into the pre-impregnated lamina tapes which forms a SiC and Silicon mixed matrix. As the laminate cools, complex residual thermal loading occurs, resulting in a residual tensile fiber load and a matrix in compression. To study this result in greater detail, the laminate was thermally exposed. Consequently, hereafter the laminate will be denoted as being either in an as-produced (ap) or annealed (an) state.
Figure 24 – Actual CMC test specimen at the top. Vertical white lines are silver paint to enhance electrical contact. Micrographs of the architectures studied. (90/0)2s, (0/90)2s, and 0_8. When longitudinal or 0° plies are referred to, it is the plies that fibers are in the loading direction. When transverse or 90° are referred to, it is the plies that the fibers are perpendicular to the loading direction.

Tensile Test

Quasi-static, room-temperature, uniaxial, monotonic and hysteretic, interrupted and to-failure tensile tests were done in conjunction with temporary laboratory material health-monitoring techniques electrical resistance (ER) and acoustic emission (AE). The mechanical relationships of stress and strain were paired with electrical and acoustic measurements and analyzed to develop a more fundamental understanding of the failure of brittle matrix laminates, their constituents, and interactions.

Tensile tests were performed on two machines. Initially, tests were conducted on an MTS hydraulic machine with a 10kN load cell using MTS Station Manager Software for control and data acquisition. Most tests used a MTS Criterion electro-mechanical machine with a 30 kN load cell using MTS TestWorks 4 software for program control and data acquisition. A 25.4mm clip-on MTS extensometer was used with a maximum displacement of 2% strain. Some issues arose with the extensometer slipping and is evident
when large positive or negative jumps in strain are visible from a stress-strain plot. These erroneous strain measurement jumps most likely are the cause of the large variances in the residual strains measured with unload-reload loops of some tests. If an individual strain discontinuity was exceptionally large, the strain errors were manually removed. Various loading schemes were used, but the preferred procedure was determined to be a displacement controlled tensile test at 0.1-0.5 mm/min crosshead displacement with either monotonic loading or unload-reload loops performed at predefined strain bounds.

![Image of tensile rig with AE transducers and ER clips](image)

**Figure 25** – The University of Akron tensile rig with AE transducers and ER clips. The uniaxial loading direction is vertical.

As mentioned, the strain measurements, $\varepsilon$ in units %, are taken directly from the extensometer, and stress, $\sigma$ in units MPa, is found by dividing the applied load by the cross-sectional area of the specimen. From the stress-strain data, the elastic stiffness, or elastic modulus of the laminated plate can be calculated from the constitutive equation:

$$E_\varepsilon = \frac{\sigma}{\varepsilon}$$

Equation 14 - Elastic Modulus of the composite laminated plate based on experimental measurements

stress, strain

Since the composite is a balanced, symmetric, the global properties can be assumed to be isotropic, where there are two independent material properties, elastic modulus $E$ and Poisson ratio $\nu$ [56]. However, the lamina that make up the composite should be considered to be a non-isotropic material because the properties vary depending on the orientation of the material. The most appropriate assumption of material properties would be that of transverse isotropy, where the fiber, matrix, and
interphase are homogenized into a lamina, or single layer of the composite and can be represented by five independent material constants $E_1, E_2=E_3, G_{12}=G_{13}, v_{12}=v_{13}, v_{23}$. Calculating the lamina properties of more than two materials is a non-trivial matter with many methods available. Due to the ease and robustness, a finite element micromechanical method is used where the constituent properties and associated volume fractions are known. Refer to Table 3 for equations.

To calculate the instantaneous modulus, or tangent modulus, Equation 15 is a simple method, or a more robust method of a linear-regression of multiple data points can be used. The tangent modulus captures the reduction in stiffness of the material which is an indication of damage. Once the tangent modulus has been calculated, the inverse tangent modulus can be used for various calculations as shown by [57], [58].

$$E_{\text{tan}}^{-1} = \frac{\epsilon_f - \epsilon_0}{\sigma_f - \sigma_0} = \frac{1}{E_{\text{tan}}}$$

Equation 15 – Inverse Tangent Modulus. The slope of two points on a stress strain curve. Subscript f and 0 refer to final and initial

Acoustic Emission

Modal acoustic emission was recorded during tensile tests using the Fracture Wave Detector system from Digital Wave Corporation. Three wide-band (50-2000 kHz) piezoelectric sensors are used to capture acoustic activity, indicating a stress wave that is a result of crack formation or growth. The first sensor is placed in the middle of the specimen and the others are located approximately 30mm above and below the first. During the loading, parameters such as time, strain, load, and resistance are recorded in addition to the digital waveforms of the three sensors. A unique feature of the system is the ability of each sensor to independently detect events. When an event is detected, all three sensors begin recording at the same time and digitize each event separately and simultaneously. This allows for modal analysis where advanced signal processing techniques are used to disseminate information from the waveform, as well as location determination. For this work, 3 sensors are used, at 10 MHz acquisition rate, and 1024 samples/sensor/event. This results in a time window of 102.4μs per event and a capability of acquiring about 3255 events/second[59].
\[
\frac{\text{sensors} \times \text{samples}}{\text{acquisition rate}} = \frac{3 \times 1024}{10 \times 10^6} \approx 614\mu s \propto 3255 \text{ events/second}
\]

Equation 16 - AE calculations for the capture rate for the number of events

These acquisition settings, as well as voltage gain settings, are determined based on experience from past tests, as well as preliminary tests using pencil lead breaks. The acquisition settings can then be optimized depending on user priorities, such as event location tolerance or wave fidelity to minimize saturation and noise. Also, according to the sampling theorem[48], the sample rate must be at least twice the expected maximum frequencies to prevent aliasing which results in lost data when digitizing the waveform. The system is generic and can accept up to 32 sensors, 4096 samples per event, and 20 MHz sample rate. Simple arithmetic shows that, with this maximum configuration, 152 events/second can be recorded.

Signal gain settings, trigger gain settings, and frequency filters can be adjusted depending on the material tested and type of damage expected. The settings used for this work minimized wave saturation but amplified the signal enough to properly determine the time-of-arrival, TOA. The TOA is then determined by an algorithm called first threshold, where the arrival time is when the waveform exceeds a user-specified voltage. More complex algorithms were explored, but are sensitive and require careful calibration[49]. While the first threshold method was sufficient for this work, future work should explore more advanced methods to improve location accuracy and reduce sorting errors.

Vacuum grease is used to enhance acoustic transmission from the specimen to the sensors. Generic spring-loaded clips are used to attach the sensors to the specimen. There is some variability in the wave transmission based on surface roughness, clamping force, and amount of grease applied. The acoustic emission analysis neglects these differences due to the inability to identify the exact transmission coefficient.

Once a test is complete, digital signal processing techniques are used to investigate the ensuing damage such as signal energy, frequency and energy attenuation, and location based on the speed of sound and arrival times at each sensor. The AE system uses a commercial software package called WaveExplorer
for acquisition and analysis, but lacks the ability to augment user-specified analysis tools. So, custom post-processing software was developed in MATLAB[60] to handle the large amounts of data generated, and to support current and future ad-hoc analysis tools. The net time savings on the software development versus processing, sorting, analyzing, and visualizing data were substantial with the in-house codes.

Once the test is complete, post-processing data filters are applied to eliminate any erroneous or valid but error prone acoustic events to simplify the analysis. Filters were used to ensure that AE events occurred in the gage section (defined by the section between the 25.4 mm extensometer) with sufficient energy to determine the time-of-arrival, TOA (Recall Figure 19): a pre-trigger energy ratio < 0.5 V^2/μs and total event energy > 0.5 V^2/μs. These values were determined by trial and error. The pre-trigger energy ratio is calculated based on a ratio of the energy of the pre-trigger and the energy of the event. This eliminates any noise that may have erroneously triggered the AE system. The event energy is used to ensure the wave energy is large enough to accurately determine location. By filtering all events that arrived at the middle sensor first gave us our final AE dataset that represent the damage in the gage section of the specimen.

**Electrical Resistance**

Electrical resistance was captured with an Agilent 34420A micro Ohmmeter and sampled at 1-5 samples/second. The system could not acquire data reliably greater than 5 samples/second. A 4-wire resistance measurement system was used to improve the accuracy of the resistance by applying a DC current through the specimen from the outer two wires while the inner two wires measure the voltage difference. Two wire resistance measurements are not as accurate due to the resistance of the wire contributing to the readings. Electrical contact from the specimen to the copper lead clips is enhanced by silver paint to reduce variation in data from the clip-material contact and to distribute the electrical contact around the entire specimen. Resistance measurements without silver paint (and sometimes with) are often noisy and subject to large, volatile fluctuations. Glass-reinforced polymer tabs were used to electrically insulate the specimen from the machine and to prevent the specimen slipping in the grips when loaded.
When determining the electrical properties of a material, resistance is typically measured with an Ohmmeter or multimeter. However, independent of the material, resistance will increase with length and decrease with cross-sectional area. Thus, to only consider the material's ability to resist the flow of electrons independent of the components' shape or size is a measurement of resistivity. Electrical resistivity, or inversely electrical conductivity, is a material property and can be represented by the following equations [61]:

\[ R = \frac{1}{G} \]

Equation 17 - Electrical resistance is acquired with an Ohmmeter. Resistance and can be converted to conductance depending on the analysis. \( R \) = Resistance, \( G \) = conductance.

\[ \rho = \frac{1}{\kappa} = \frac{RA}{L} = \frac{A}{GL} \]

Equation 18 – Comparing the electrical properties of material of different shapes and sizes should be done with resistivity. \( \rho \) = resistivity, \( \kappa \) = conductivity, \( R \) = measured resistance, \( G \) = conductance, \( L \) = length, \( A \) = cross-sectional area.

\[ \% \Delta R = \frac{R_f - R_0}{R_0} \]

Equation 19 - Another metric to compare the electrical changes in the material is the percent change in resistance. A similar equation can be used with conductance.

Microscopy

In CMCs, matrix cracking allows an otherwise brittle material to be tough and reach high failure strains. Knowing the nature of these cracks and relation to stress is paramount to understanding CMC damage. Thus, optical microscopy is used to visually verify the location and quantity of the cracks, which is used to investigate the post-tensile test damage state. Typically, specimens are mounted in an epoxy so that the viewing angle is orthogonal to the loading direction. A Mitutoyo binocular microscope with an Edmund Optics 1.25 megapixel digital camera was used to record the micrographs. Lighting through the lenses was necessary for visibility, making this microscope the only option for optical microscopy. In-situ microscopy was attempted during a tensile test in an attempt to observe cracking as it occurs, but insufficient lighting limited visibility and no results were acceptable.
The material, in its as-produced state, is expected to have a thermal residual tensile fiber stress which causes post-test crack closure. Crack spacing measurements are recorded from the micrographs of polished specimens, but most of the cracks that exist in the specimen are invisible due to crack closure and results in erroneous crack spacing measurements. Plasma etching is possible to improve crack visibility to some degree, but ablated surface silicon can have the false appearance of cracks and the process is costly and time-consuming. Other methods have been attempted by other authors,[62], such as crack-penetrating dye which was reported to have little success, or physically bending the specimen, which did prove useful. Based on the latter method, a simple mechanical method was developed to make cracks visible. After a to-failure or interrupted tensile test, specimens were mounted in epoxy, edge-polished, and cut in ~2mm thick sections. The cut segments were then flexed in a fixture to induce a surface stress so that the invisible cracks became visible. The crack viewing mechanism uses a simple four-point bend to apply a small load to the specimen to overcome the residual tensile fiber stress. A four-point bend induces a constant stress on the surface of the beam between the inner pins, which provides sufficient length (5-10mm) to determine representative crack spacing values. These improved crack spacing estimates can be used to improve mechanical and electrical models of the composites.

Once the specimens are mounted and polished, digital images were recorded in a grid pattern and then post-processed with photo-editing software to stich the individual images into a single image. Two free software packages proved extremely valuable to automating the onerous task of stitching the images were Fiji [63] and Gimp (see the appendix for explanation and instruction). Once images are stitched and analyzed, the crack spacing’s were counted from the images. The number of cracks counted in a given length is crack density, or inversely, half the distance between each crack is the crack spacing. Depending on the analysis, either may be used, but this work will typically use crack spacing and report the resultant distribution of the crack spacing.

\[2s = \frac{1}{d}\]

Equation 20 - Crack spacing,2s, and d, Crack Density, as prescribed by Pryce and Smith[33].
Figure 26 – The process of cutting, mounting, polishing, cutting and viewing to make closed cracks visible by inducing stress due to bending.

When bending the specimen, estimates of the stress on the surface of the flexed cut section were made to determine if new cracks were forming or existing cracks were propagating. A Mitutoyo analog depth micrometer was used to measure the deflection ($\delta_{\text{bend}}$) of the cut section in four-point bend. With the experimentally determined linear elastic composite modulus, a maximum stress was estimated based on the deflections. From Equation 21, the stress necessary to make cracks visible was well below the peak stress.

$$\sigma_{\text{max}} = \frac{3(L - 2a)Eh\delta}{4a^2(3L - 4a)}$$

Equation 21 – Four-Point Bend Stress Equation [64]

Figure 27 – Four point bend diagram. $\delta_{\text{bend}}$ is the measured deflection of the beam and is used to approximate stress [64].

---

1. post-fracture cut and polish
2. Cut from epoxy mount and stressed in fixture
3. Micrographs recorded and stitched
The result flexing the specimen and subsequent changes in visible cracks is shown in Figure 28.

Without using the bend fixture, crack spacing measurements were very inaccurate. When flexed, many, if not most of the cracks became visible, leading to much better estimates of actual crack spacings. Crack closure was most evident in the unidirectional specimens. Very few cracks were visible in an unstressed state, when stressed in the bend fixture, many cracks became visible. Cross-ply specimens typically had more visible cracks unstressed, but the ratio of visible to invisible cracks was much larger as compared to the unidirectional.

Unidirectional specimens were especially difficult to polish due to matrix spallation and limited matrix visibility for crack viewing. By angling the specimen at about a 15° offset when mounting to polish, the now angled fibers held the matrix in place and matrix cracks were very visible. To visualize the difference in the mount angle, compare the no offset angle in Figure 24, and 15° offset in Figure 28. For all specimens tested in this work, the crack spacing measurements and subsequent micrographs employ the bend fixture that has just been described.
CHAPTER IV

RESULTS AND DISCUSSION

A summary of all the tests done is shown in Table 4. The main focus was on the architecture (i.e., (90/0)2s, (0/90)2s, 0_8) and the annealed state (i.e., ‘ap’, or ‘an’) of the specimens. However, as previously mentioned, the material processing can affect the properties, so there may be some variability when comparing specimens from different manufacturing panels.

Table 4 - Summary of 13 test specimens with physical properties

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</tr>
</thead>
<tbody>
<tr>
<td>PS-3281-T5</td>
<td>PS-3281</td>
<td>(90/0)2s</td>
<td>ap</td>
<td>to failure</td>
<td>0.5 mm/min</td>
<td>12.93</td>
<td>10.16</td>
<td>1.86</td>
<td>203</td>
<td>3367</td>
<td>255</td>
<td>276</td>
<td>224</td>
<td></td>
</tr>
<tr>
<td>3471A-T01</td>
<td>3471A</td>
<td>(90/0)2s</td>
<td>ap</td>
<td>to failure</td>
<td>0.25 mm/min</td>
<td>9.63</td>
<td>10.16</td>
<td>1.88</td>
<td>152</td>
<td>3308</td>
<td>264</td>
<td>262</td>
<td>254</td>
<td></td>
</tr>
<tr>
<td>PS-3281-T8</td>
<td>PS-3281</td>
<td>(90/0)2s</td>
<td>an</td>
<td>to failure</td>
<td>0.1 mm/min</td>
<td>13.03</td>
<td>10.14</td>
<td>1.89</td>
<td>203</td>
<td>3346</td>
<td>231</td>
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<td>174</td>
<td></td>
</tr>
<tr>
<td>PS-3281-T10</td>
<td>PS-3281</td>
<td>(90/0)2s</td>
<td>ap</td>
<td>to failure</td>
<td>0.5 mm/min</td>
<td>12.91</td>
<td>10.14</td>
<td>1.88</td>
<td>203</td>
<td>3333</td>
<td>259</td>
<td>226</td>
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<tr>
<td>3471A-T02</td>
<td>3471A</td>
<td>(90/0)2s</td>
<td>an</td>
<td>0.15, 0.2, interrupted</td>
<td>0.35 mm/min</td>
<td>9.60</td>
<td>10.16</td>
<td>1.88</td>
<td>152</td>
<td>3285</td>
<td>260</td>
<td>206</td>
<td>132</td>
<td></td>
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<tr>
<td>P8-3284-T2</td>
<td>P8-3284</td>
<td>(90/0)2s</td>
<td>ap</td>
<td>to failure</td>
<td>4 kN/min</td>
<td>13.00</td>
<td>10.16</td>
<td>1.88</td>
<td>203</td>
<td>3340</td>
<td>296</td>
<td>242</td>
<td>216</td>
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<tr>
<td>P8-3284-T4</td>
<td>P8-3284</td>
<td>(90/0)2s</td>
<td>ap</td>
<td>to failure</td>
<td>0.5 mm/min</td>
<td>13.01</td>
<td>10.16</td>
<td>1.86</td>
<td>203</td>
<td>3388</td>
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<td>P8-3284-T3</td>
<td>P8-3284</td>
<td>(90/0)2s</td>
<td>an</td>
<td>to failure</td>
<td>0.25 mm/min</td>
<td>12.94</td>
<td>10.14</td>
<td>1.85</td>
<td>203</td>
<td>3395</td>
<td>257</td>
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<td>P8-3284-T6</td>
<td>P8-3284</td>
<td>(90/0)2s</td>
<td>an</td>
<td>0.1, 0.2, 0.3, interrupted</td>
<td>0.5 mm/min</td>
<td>12.92</td>
<td>10.17</td>
<td>1.87</td>
<td>203</td>
<td>3343</td>
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<td>P8-3284</td>
<td>(90/0)2s</td>
<td>an</td>
<td>0.15, 0.2, interrupted</td>
<td>0.35 mm/min</td>
<td>12.91</td>
<td>10.16</td>
<td>1.88</td>
<td>203</td>
<td>3331</td>
<td>284</td>
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<td>P8-3284-T10</td>
<td>P8-3284</td>
<td>(90/0)2s</td>
<td>an</td>
<td>0.3, interrupted</td>
<td>0.9 mm/min</td>
<td>12.91</td>
<td>10.16</td>
<td>1.88</td>
<td>203</td>
<td>3313</td>
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<tr>
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<td>1882</td>
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<td>ap</td>
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<td>7.75</td>
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<td>1.51</td>
<td>152</td>
<td>3138</td>
<td>361</td>
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<tr>
<td>1882-G2-0005-O-T04</td>
<td>1882</td>
<td>0_8</td>
<td>an</td>
<td>0.2, 0.4, to failure</td>
<td>0.35 mm/min</td>
<td>7.79</td>
<td>10.17</td>
<td>1.53</td>
<td>152</td>
<td>3285</td>
<td>365</td>
<td>449</td>
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</tbody>
</table>

Different testing schemes were used for two main reasons: to determine AE, ER, and crack spacing at intermediate stress (as opposed to ultimate failure stress), and investigate the changes in the material during load cycling. Examples of these tests and the data collected are shown in Figure 29. The top row shows the data acquired as a function of time. Some immediate observations can be made about the data shown there that will be discussed in detail. The bottom row shows a typical stress-strain plot with AE and ER. It is evident that the stress-strain relationship is complex and highly non-linear. CMCs are designed to exhibit toughness, which is shown by the large failure strains achieved. When unloaded and reloaded, complex frictional sliding occurs between the matrix, interphase, and fibers and produces non-linear stress-
strain and ER-strain “loops” seen in the bottom plots. Also, the AE events, for the most part, only occur on loading near the largest stress the material has experienced (peak stress). The flat line of AE events indicate no events are occurring. Finally, the changes in resistance vary depending on load cycling and peak stress.

Figure 29 – Time series plots of stress, cumulative AE events, and %ΔR. Three different test schemes are shown: monotonic loading, interrupted unload-reload, unload-reload to failure. Loading scheme is described in Table 4 under “strain (%) hysteresis loops”. See Appendix 3 for all time-series plots

Mechanical Properties

Using the load and displacement results from tensile tests, stress can be calculated from the applied load and strain can be calculated from the displacement of the extensometer. From these stress and strain values, three important values are calculated: the elastic modulus (E), ultimate stress (S_ult), and proportional limit stress (S_pl) (Figure 30).

The elastic modulus, or stiffness of the material, can be calculated by dividing the stress by the strain from values in the linear-elastic region, typically below strains of 0.05-0.1%. The cross-ply composites will have comparable moduli with one another, but smaller as compared to the unidirectional specimen due to the presence of softer 90° transverse plies which the unidirectional architectures lack. The proportion limit-determined with a 0.005% strain offset-is the first indication of damage through non-linear behavior in the stress strain data. The proportion limit can be difficult to predict but is easily found with
empirical data. In the absence of acoustic emission data, the proportion limit is the best approximation of initial damage in the composite. Finally, the ultimate strength ($S_{ult}$) is the stress at which the composite breaks. With any composite, the fibers are the main load carrying material, and so, any material with more fibers in the loading direction, as a unidirectional has more than a cross-ply, it is expected that the unidirectional composite will fail at a higher stress, which is indeed what has been observed.

The results of annealing are shown by a reduction in ultimate stress, but most evident by a reduction of the proportional stress for like architectures. During the process, it is expected that the material is held at a high temperature, where the residual thermal stresses due to coefficient of thermal expansion (CTE) mismatch of the fibers, matrix, and free silicon are relieved. The annealing process, as with metals, probably changes the microstructure of the constituents, relieving the residual thermal stress, but not changing the material modulus.

![Figure 30 – Cross-ply and unidirectional mechanical properties Summary. E = elastic modulus, $S_{pl}$ = proportional limit stress with 0.005% offset strain, $S_{ult}$ = ultimate stress. Error bars are standard deviation.](image)

The source of the variability of the mechanical properties is unknown, but is suspected to be from one or all of the following: materials processing, post-processing heat treatment (annealing), constituent fracture inconsistency, measurement error during testing, or an insignificant sample size. With regards to the processing of the material, the annealing process is unknown. The annealing process reduces the average ultimate strength of the unidirectional by about 5% and the cross-plies by about 10%, and respectively, the proportional limit by about 20% and 30% (Figure 30). This is expected due to the matrix
being at a higher tensile stress (due to the lack of a thermal compressive preload) at a relatively lower applied composite stress.

Test results for stress-strain are shown by architecture in Figure 31, Figure 32, and Figure 33. Large failure strains indicating toughness are evident from these plots. Also note the higher stresses but lower strains typical of the as-produced specimens, and conversely for the annealed specimens. Material hysteresis is measured by unloading and reloading the composite. The non-linear stress/strain relationship is very complex and not well understood, but some hypothesis’ are presented in the literature review that attempt to describe this phenomenon (recall Pryce-Smith model from the literature review).

Figure 31 – Stress-Strain plot of unidirectional laminates with unload/reload loops. See appendix for tabulated data
Figure 32 – Stress-strain plot of (90/0)2s cross-ply laminates with monotonic and unload/reload loops. See appendix for tabulated data.

Figure 33 – Stress-strain plot of (0/90)2s cross-ply laminates with monotonic and unload/reload loops. See appendix for tabulated data.
Figure 34 – Comparison of cross-ply, as-produced composite with high and low failure strain and associated AE and ER produced. High failure strain produces many more AE events and has very large changes in ER. Note the difference in scales between the plots and axes

As-produced specimens typically failed prematurely without experiencing toughening. These specimens had a low failure strain (less than 0.2%), few acoustic events (less than 300) and little change in electrical resistance (less than 25% change). With so few data, little can be said about the damage progression of those materials. The two extremes of failure strain are shown in Figure 34. The left plot, P5-3281-T5 did show toughness and had the largest number of AE events and %ER change out of all the specimens tested. A more typical as-produced specimen is shown on the right. These brittle specimens were of little use for this work other than knowing the elastic modulus and ultimate strength. As compared to the as-produced specimens, all the annealed specimens were tough, and very active acoustically and electrically. Because of this, they were invaluable in providing the AE and ER measurements necessary for this work.

Microscopy

Knowing the stress dependence of cracks is critical to understanding the mechanical properties of CMCs. If no cracks form, then the material remains linear-elastic (at room temperature) and the load-bearing fibers are protected from environmental attack by the SiC matrix. Some factors that contribute to crack formation are fatigue, unexpected mechanical and thermal loads, and pre-existing manufacturing defects. Thermal and environmental degradation are neglected in this work, as all tests were at room-temperature, so all cracking is attributable to only the applied quasi-static load.
Crack visibility is initially poor in polished sections of post-test specimens due to the residual fiber stress closing the matrix cracks. A simple, fast method was developed to overcome crack closure to improve crack spacing measurements by using the bend fixture described in the Experimental Procedure chapter. Figure 28 shows a unidirectional specimen where very few cracks were visible while unstressed, and the vast majority of the existing cracks become visible when stressed. Similar results were found for the cross-ply architecture, but not as many invisible cracks were found as compared with the unidirectional composites.

The mechanical properties of CMCs, especially when considering cyclic loading, are a function of cracking in the 0° plies (refer to the modelling section of the literature review for more information), and so, the 90° crack spacing is not as important for room temperature testing. In this work, only 0° ply crack spacing measurements are reported, but future work should document attempt the transverse crack spacings.

Other more ductile materials, such as metals, typically have very predictable (little variability) strengths, as opposed to brittle materials (i.e. ceramics) which the breaking strengths are much more unpredictable (large variability in strengths). It has shown that the brittle matrices of CMCs have a volume dependent distribution of strengths that follow a statistical Weibull distribution[65], and that fiber strengths also follow a Weibull distribution[16]. Understanding the limitations of the values of crack spacings and the nature of various statistical distributions, most of the common distributions (normal, exponential, etc) can be eliminated due to the inability to exhibit non-symmetric distributions. Knowing that the crack spacing values are typically skewed right, from 0 to positive values, and the peak tends to a positive non-zero value, it is hypothesized that crack spacing (as well as crack density) measurements will fit a 2-parameter (peak and skew) Weibull distribution better than other statistical distributions. As the material is stressed, and cracks form, the crack spacing values will decrease and tend toward a minimum crack spacing, i.e., crack saturation. The few, large crack spacing’s will be outliers against the majority of the values that exist around the mean crack spacing at saturation. Histograms shown in Figure 35 support
this observation. Coupling these crack distributions with cumulative AE events offers realistic cracking
information for general mechanical models with cracking to accurate simulate stress and/or strain.

Unidirectional Microscopy

The simplest architecture studied in this work is the 8-ply unidirectional composite. Initial
attempts of measuring crack spacings in unidirectional specimens were unsuccessful due to large matrix
regions spalling during polishing. It was found that if the specimen was angled slightly (~15°) before
mounting and polishing, the matrix regions remained and matrix cracks perpendicular to the loading
direction were easily seen.

The aggregated ply crack spacing distributions with the fit Weibull distribution are shown next to
the micrographs in Figure 35. The as-produced specimen micrograph shows non-uniform crack spacing
along the length, which is evident from the distribution as the outliers. This observation will be discussed in
more detail in the AE location section. The annealed specimen shown little variability in the crack spacing
values.
Cross-Ply (90/0)2s Microscopy

Unidirectional composites are useful when studying lamiante mechanics because of the simple architecture, however have little practical use due to their poor multi-axial strength. Cross-ply composites have improved multi-axial strength which allows them to be used in real applications. The cross-ply architecture has ½ of the plies oriented 90° to the main loading direction, which gives it out-of-plane strength. Since plies are oriented in two directions, the 90° plies have a lower stiffness as compared to the 0° plies in the loading direction, which is why cross-plies have a lower modulus than unirectional composites. These results are used in the modelling section to approximate pre-damage ply stresses. Crack spacing results for the (90/0)2s cross-ply (Figure 36) indicate that, for specimens that achieved a peak strain greater than %0.02, the surface plies (which are 90° orientation) have the least amount of cracking.
and middle plies have the most. For a (90/0)2s architecture, these results are reasonable, because of the stiff, inner ply experiences a higher stress than the 90° surface plies.

Figure 36 – Micrographs of (90/0)2s cross-ply with average crack spacing per 0° ply over 10mm for annealed specimens of the same panel ply crack spacing and on the far right, aggregated 0° ply crack spacing’s.
The average crack spacing measurements done at the ply level shows a general trend for the composite, but only showing averages does not represent the statistical nature of cracking. To visualize a more holistic perspective on crack spacing statistics, a process similar to that of the unidirectional composites (recall Figure 35) was completed for the cross-ply composites. Crack spacing measurements for the inner longitudinal plies are shown in Figure 36 with a best-fit Weibull distribution curve on the far right. While the values shown are expected to be sound estimates, poor crack visibility is common when analyzing the micrographs and may be a source of error in crack spacing measurements. Cracks are only visible when a well-polished matrix layer is visible. If the fibers are near one another from ply to ply, then cracks are often propagating through the interphase and hidden in the micrographs. Thus, recalling the average ply crack spacing’s from Figure 36, there is some expected but unknown error with the outer 0° ply crack spacing’s due to cracks being hidden by the 90° fibers. Again, as compared to the unidirectional measurements, failure strain was a reasonable predictor for the longitudinal ply crack density. The as-produced specimens were a much lower strain and less cracking, but also, the variation in crack spacing is much higher, indicating the localized banding that was shown with the as-produced unidirectional specimen. In addition to the localized cracking, the as-produced specimens typically had a few, large through-thickness cracks that stand out. This large crack can be seen in the micrograph of specimen P8-3284-T4 in Figure 36.

Cross-Ply (0/90)2s Microscopy

The crack spacing measurements for the (0/90)2s architecture are shown with the middle 90° values for reference. Unlike the (90/0)2s, the middle two plies of the (0/90)2s architecture have a 90° orientation, and the surface plies are now a 0° orientation. The linear-elastic properties of the balanced (e.g., equal number of 0° and 90° plies) (0/90)2s and the (90/0)2s should be the same, however, the onset and progression of damage may not. Results from AE (shown in AE results) indicate that the ply orientation sequence does indeed exhibit unique damage patterns.
Only the 0° crack spacing measurements are used in the crack spacing distributions. These results, when compared to the other cross-ply and the unidirectional specimens, follow a similar trend of increased cracking in the 0° plies as a function of peak strain.

Figure 37 - Micrographs of (0/90)2s cross-ply with average crack spacing per 0° ply over 10mm for annealed specimens of the same panel ply crack spacing and aggregated 0° ply crack spacing's.
Although the architecture is the same, it is important to note that two unique manufactured panels (P5-3281, and 3471A) were used in the (0/90)2s results, and may have unknown differences in the results relative to one another due to differences in the manufacturing conditions.

Does the crack spacing change relative to the length, width, or thickness of the specimen? This is an important question that was investigated. Measurements were taken through thickness, though the length of about 15mm, and through the width of multiple specimens. The most interesting result shows non-uniform crack spacing anomalies through the length that should be investigated in more detail.

Through-thickness cracking in a unidirectional composite is much simpler than a cross-ply composite. When a unidirectional composite cracks, the crack most likely initiates in a single ply and will either grow through more plies or will be contained in a single ply. By inspection of the micrographs (Figure 35), some cracks appear to be isolated, or independent from one another, but most appear to be on the same line and span through-thickness of the entire composite which would indicated that the cracks are not independent of one another. Considering annealed cross-plies, a similar phenomenon is apparent, where many cracks appear to be independent but some appear to exist through-thickness. Where the difference occurs is when as-produced specimens crack. Large, through-thickness cracks are common in as-produced specimens, which seem to interact with many smaller isolated crack systems. The 0° plies have smaller crack spacing, but it is observed that all the 0° ply cracks contribute to a single 90° ply crack which most likely has a crack opening proportional to the number of interacting 0° ply cracks (Figure 38). Cracks in annealed specimens seem to interact less, and are more uniform than as-produced specimens, which has variable crack spacing within plies and along the length.
Figure 38 - P5-3281_t8 (0/90)2s specimen showing dependent 0 and 90 ply cracking. It is expected that the crack opening displacements of the 0 plies is proportional to the opening of the 90 ply.

The excess silicon veins in the composites are a result of the manufacturing method and are not desirable in the finished composite. This silicon causes a reduction in creep resistance due to silicon having a much lower melting temperature than SiC[10]. By comparing micrographs of as-produced and annealed specimens, the proximity of matrix cracks and the silicon veins is very different. Cracking in as-produced specimens rarely occurs near the silicon, whereas cracks in the annealed specimens appear to prefer cracking in the silicon regions. One explanation of this may be due to the thermal mismatch of the constituents. While the exact values are not known, the SiC matrix and the SiC fibers are expected to have different CTEs and upon cooling after manufacturing will result in a thermal residual stress. However, the mismatch is not expected to be large enough to account for the large thermal stresses found in these composites, but, the free silicon may. The localized pockets of free silicon will expand when cooled from a liquid to a solid and result in large tensile loads in the fibers and compressive loads in the matrix. If this is true, then the as-produced specimens will more likely not have matrix cracks near the free silicon, which is observed. Another result will be variable matrix stresses along the length of the composite, which may explain the localized cracking regions observed.
In summary, annealed specimens appear to have more evenly spaced cracks, while the as-produced specimens have very localized damage sites. The observations of ply dependent/independent cracks and through-thickness cracks, and the silicon cracking phenomenon may together contribute to the crack banding observed for all as-produced specimens. The progression of localized cracking implies two cases: crack spacing starts very locally and unevenly spaced but then cracks form between the damage sites and the crack spacing’s are evenly distributed, or the residual stress causes very localized cracks to form in the areas of the laminate that are under lesser residual stresses and the cracking stays localized to failure.

An investigation of through width crack spacing was done to determine if laminate edge effects or surface defects were the cause of smaller crack spacings found internally. The results varied for the (90/0)2s and the (0/90)2s but no statistically significant differences in crack spacings were found. It is still possible that through width crack density may vary. One explanation may be due to Poisson mismatch of 0° and 90° plies causing shear stresses on the edges when a laminate is under load. No evidence of this is found. Another possible reason for more surface cracks is simply due to the brittle nature of ceramics and the sensitivity to surface flaws. However, none of these hypothesis were definitively proven correct.
Figure 40 - Through-width crack spacing. *shows hypothesized trend of crack spacing which has been found to not be true.

Figure 41 - Through-width crack spacing does not significantly change. Each cut was about 2.5mm deep.
Figure 42 - crack densities of 0° plies compared with published results by D. Dunn[62]

Figure 42 gives a summary of the mean crack spacing measurements for the 0° plies. The trend is vertically asymptotic around the proportional limit strain and exponentially decays as a function of peak strain to some unknown final saturated crack spacing. Note that, if crack density were to be plotted, a linear trend would be found with identical fit to the exponential decay of crack spacing.

Typically, crack spacing is dependent on stress, as was shown by the PS model presented in the literature review. This is a simple matter when considering unidirectional composites, as each ply has the same modulus in the loading direction and experiences equivalent stress, as well as strain. Cross-plies, however, typically have different moduli for each ply orientation and because of that, will have different stresses. Assuming cracks only occur perpendicular to the loading direction (no delamination), and based on laminated plate theory, each ply will have the same strain, and stresses will vary based on the ply modulus. When comparing results from the same laminate panel and architecture, stress was a sound predictor for crack spacing, but due to the limited number of samples, is not shown here. However, thermal stresses are unknown due to the complexities of the microstructure of the composite due to processing, and only rough estimates are available based on experimental results. Also, not only do the 0° and 90° plies have different moduli in the loading direction, they are fundamentally different structures.
due to the varying fiber orientation, thus have totally different fracture toughness's, strength's, and damage types. These differences are why modeling and predicting CMC properties are so challenging. Without better understanding of these composites, peak strain proved to be a better predictor of the damage quantity.

Acoustic Emission

CMCs are an ideal application for acoustic emission. Brittle damage is often audible (as opposed to ductile yielding) to the human ear. The acoustic emission recorded during these tests indicate that some type of damage is occurring, and various analysis methods can be used to decipher the wave velocity through the material, event location, relative energy, and the frequency. The total number of events that occurred in the gage section-the area in-between the extensometer-is a function of failure strain. Specimens that failed at strains above 0.3% typically had about 1000-2000 events. These gage events represented only a fraction (20-30%) of the total number of events that were captured, and the rest are either noise or cracking or grip slipping noise from outside of the gage area.

Figure 43 – Cross-ply and Unidirectional beginning acoustic emission activity. Loud is greater than 10x the 1st event energy.

The effects of annealing reduces the absolute stress of the 1st AE, 1st loud AE and the 5th loud AE all by about 25% for all architectures. Similar results are found for the proportional stress and ultimate stress.
Any composite is composed of multiple constituent materials, where each may have very different properties, and thermal CTE mismatch is common. When a change in temperature occurs, such as cooling from manufacturing, these laminates exhibit a residual tensile stress in the fiber and a matrix in a compressive state. Knowing exactly the compressive stress in the matrix is difficult, but estimates of 7-28 MPa[62] are likely low when considering the differences in 1st AE. The difference in as-produced to annealed specimens is about 100 MPa for unidirectional, and 75 MPa for cross-ply by comparing the 1st AE in Figure 43 (Table 5 shows exact calculations). Assuming the initial flaw to cause a matrix crack is the same for the annealed as the as-produced specimen, the 1st AE for an annealed specimen and an as-produced specimen should be the difference of the residual matrix compressive stress. Based on the 1st AE and first loud AE, these results show estimates of the absolute minimum of the residual stress.

Table 5 – Difference in residual stress of the annealed and as-produced specimens. Results from the 1st AE and the 1st loud AE are similar. * indicates standard deviation

<table>
<thead>
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<th>Architecture</th>
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<th>1st Loud AE</th>
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</thead>
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<tr>
<td></td>
<td>as-produced (MPa)</td>
<td>annealed (MPa)</td>
</tr>
<tr>
<td>Uni</td>
<td>374 ± 0*</td>
<td>268 ± 0*</td>
</tr>
<tr>
<td>90/0</td>
<td>219 ± 8*</td>
<td>145 ± 20*</td>
</tr>
<tr>
<td>0/90</td>
<td>202 ± 13*</td>
<td>133 ± 10*</td>
</tr>
</tbody>
</table>

The residual stress state of a unidirectional composite is much simpler as compared to a laminate with various lamina orientations, such as a cross-ply. The unidirectional composite only experiences residual stress when a fiber and matrix CTE mismatch exists. However, with cross-ply layups, the fiber residual stress is caused not only by the fiber/matrix CTE mismatch but is superposed by the mesoscale laminate residual stress caused by the transverse isotropy characteristic of the individual lamina.
Acoustic Emission Energy

Figure 44 - Unidirectional specimens with cumulative acoustic energy. Discontinuities represent unload loops where no AE occurred.

Figure 45 – Cross-ply (90/0)2s cumulative acoustic energy

Figure 46 - Cross-ply (0/90)2s cumulative acoustic energy

Acoustic energy is a simple, useful metric to understanding what types of waves are created by specific damage types. In general, higher frequency waves are often extensional/symmetric waves, which
produce less transverse displacement, and in turn is measured by the sensor as less energy. Conversely, lower frequency waves are flexural / asymmetric causing larger transverse displacements and larger energy (Figure 54). Cumulative acoustic energy shows the rate at which acoustic energy is dissipated in the material, which is assumed to be directly correlated with the amount of damage the material withstands. Although the value of cumulative energy doesn’t have an explicit meaning, due to sensor coupling variances, it is informative when comparing specimens. Figure 44 shows the cumulative energy of an annealed and as-produced unidirectional composite. Clearly, the annealed specimen has more cumulative energy, which is due to having more events, which is expected to be due to more matrix cracks occurring. Cumulative event counts (as opposed to cumulative energy) can also be used to track AE, but is often comparable to the cumulative energy method (Figure 29, Figure 47).

When a CMC reaches large strains, matrix cracking may slow or even stop, indicating crack saturation. Beyond this point, while the strain continues to increase, no matrix cracking occurs because the crack spacing is small enough that the matrix segments cannot reach a high enough stress to crack. Specimens in this work that had a high failure strain developed many cracks, yet, AE activity did not slow, so it is assumed that they are below crack saturation. However, if the cumulative events are stratified into low and high energy by the median energy, we can differentiate what events are high or low energy at a particular stress, as shown in Figure 47. From the right plot, we can see that, near the failure stress, the vast majority of the AE events are low energy because of the small slope of the large energy curve. Assuming that the energy is indicative of a specific damage type, (i.e., fiber breaks, matrix cracking), this plot would provide a stress (or strain) dependent relationship of the type and rate of occurrence of specific damage types.
Figure 47 - Unidirectional annealed specimen with stratified low (blue) and high (red) energy, separated by the median energy value. The left plot is cumulative acoustic energy and the right plot is cumulative events. Top plots is a function of strain, and the bottom plots stress. Notice the plot on the right, the high energy events abate at high stresses and the AE is dominated by low energy events.

When the final crack spacing is determined via microscopy, the intermediate crack spacing can be interpolated based on the cumulative energy plots. A simple calculation of total AE events divided by the gage length (25.4mm) will yield a reasonable crack spacing. Actual final crack spacing per ply for the 8-ply specimen 1882-02-0005-0-TD-4 was about 0.2 mm/crack from Figure 42, but based on AE, is about 0.15 mm/crack/ply, which is a reasonable approximation.

\[ 2s_{AE} = \frac{25.4 \text{mm}}{1394 \text{ events}} \times 8 \text{ plys} \approx 0.15 \text{ mm/crack/ply} \]

Equation 22 - Crack spacing based on AE events from 1882-02-0005-0-TD-4

Approximating cross-ply crack spacing based on AE is not as simple as the method shown for unidirectional laminates due to the difference in the number of cracks in the transverse and longitudinal
plies. No attempt was made to differentiate the AE from transverse and longitudinal plies, so the assumption of all the cross-plies is the AE is generated by the 0 plies only. In reality, both the 0° and 90° plies have different stress that cracking begins, the rate at which the cracking occurs, and the saturation stress, and are most likely not independent of one another. To understand and predict those relationships would be a challenging task, and, as previously explained, is not attempted in this work.

The total damage in the composite appears to be related to the peak strain. The cumulative AE plots show that specimens that fail at high strains have large cumulative AE energies and the opposite is true for the specimens that accumulate little AE energy failed prematurely at low strains.

Figure 48 – (a) Stress-AE Energy. Annealed specimens typically have peak AE activity before failure, as-produced specimens do not reach peak AE. (b) (0/90)2s have a much larger population of high energy events, possible indicating that surface 0° plies are creating many high energy flexural waves.

When comparing AE energy to stress, a few features stand out, highlighted in Figure 48:

a) (0/90)2s specimens have a bimodal distribution of event energy, where the total events are dominated by the lower energy, but have a higher proportion of high energy events than the (90/0)2s architecture. This may be described by the surface 0° plies cracking more than surface 90° creating high energy flexural waves.
b) Peak AE stress, the stress at which the largest number of events occur, for as-produced specimens is typically near or at the composite peak stress. As opposed to annealed specimens of any architecture, the peak AE stress occurs below the composite peak stress.

c) The energy of (0/90)2s laminates commonly have low energy initial AE, indicating that internal 90° plies cracks form first, which agrees with the prevailing theory of cross-ply laminates[66].

Acoustic Emission Location

Determining location from AE must be done with care. The elastic wave velocity is around 10,000 m/s using Equation 9 and Equation 13, and so miscalculating the TOA by just 1μs will give an error of 5mm. The accuracy of the location of the AE event will depend heavily of the algorithm used. For this work, due to ease of application, little calibration required, and issues with recorded waveform anomalies specific to the Digital Wave system used, the first threshold method was used to calculate the TOA. TOA calculations can also be done manually by visually observing each AE wave and handpicking the time when the wave appears to rise. This method is the best in terms of accuracy, but is very tedious. To validate results, 250 TOA hand measurements were taken, where the time of the first peak of the first and second sensor was recorded and then compared to the threshold algorithm. The mean error in TOA determination based on the threshold algorithm was 0.1μs ±0.09 standard deviation, so relative location accuracy for this study is assumed to be about ±0.5mm. Location error can also be attributed to reductions in the speed of sound in the material, which is a function of the instantaneous, or tangent modulus degrading in the material as damage occurs. Absolute location, as opposed to relative location, is less reliable due to the sensor diameter (~10mm), sensor location accuracy, and measured feature error, such as failure location. Understanding the limitations of AE is helpful when analyzing results.
Figure 49 - As-produced unidirectional composite (specimen 1882-02-0005-0-TD1) micrograph showing the localized cracking evident from the AE location banding. Relative location is ~±2mm while absolute error location is larger. The red line indicates failure location. Location 0 is the middle sensor position. Bubble size is proportional to the event energy.

Figure 50 –Annealed unidirectional composite (specimen 1882-02-0005-0-TD-4) micrograph showing even crack spacing at failure. Red line indicates failure location. Location 0 is middle sensor position. Bubble size is proportional to the event energy.
Comparing micrographs to AE location data is insightful but not totally conclusive due to damage and AE anomalies that are not well understood. Figure 49 shows strong localized banding of the AE recorded and is confirmed via the micrograph. A small section shows the large gaps between local cracking, which is consistent along the entire length of the specimen within the gage length. It is unknown why this occurs, but a hypothesis is that it is a function of the local residual stress state varying of the laminate due to heterogeneous silicon distribution throughout the specimen resulting in thermal strain mismatch. Another factor could be due to the brittle nature of the matrix and the distribution of flaws from manufacturing related to the varying consistencies of the constituents. The crack location results are comparable to the annealed specimen (Figure 50), where localized damage is evident at low strains. The damage sites quickly become indiscernible in the annealed specimens at higher stresses, whereas the as-produced specimens stay somewhat localized to failure. However, it is unclear that the local damage hotspots are due to difference in microstructure and thermal stresses of the as-produced specimen, or due to the as-produced specimens having a low failure strain.
Figure 51 – Annealed cross-ply (90/0)2s location versus stress. Bubble size is proportional to energy. Black vertical line indicates the proportion stress limit or PL.

With AE data, the proportion limit is the only method to detect when cracking occurs. In Figure 51 and Figure 52, the black vertical line indicates the 0.005% strain offset proportional limit (PL) stress where the knee is visible in the stress-strain plots. The difference in stress from the PL and uniform cracking is approximately 20 MPa. The stress at which uniform cracking occurs is most likely a function of the proportion limit, but varies, just like the shape of the knee in the stress-strain plot.
Figure 52 – Annealed and as-produced cross-ply (0/90)2s location versus stress. Bubble size is proportional to energy. Black vertical line indicates the proportion stress limit or PL.

While not as evident as the unidirectional specimens (Figure 49, Figure 50), the banding, or localized damage areas are also present with cross-ply composites. For many of the cross-ply specimens, especially P8-3284-T3 and P8-3284-T10, for nearly 20 MPa of loading beyond the 1st AE stress, the cracking is localized within 5mm. Specimen P8-3284-T9 initially has two local damage sites at around -14mm and 16mm. It is unclear if the local damage sites are a function of architecture. Most of the specimens had discernible local damage at low stresses, but once the specimen experienced high strains, post-test microscopy typically had no evident active crack locations except for the as-produced specimens. One such as-produced specimen, P5-3281-T5 did reach high failure strains, and had uniform crack locations at those high stresses/strains despite its as-produced state.

An interesting observation is shown by comparing the log energy histograms found in Figure 51 and Figure 52. As shown before, the (0/90)2s architecture has a bimodal log energy distribution, indicating that a much higher percentage of AE energy is high energy. An explanation of this is that the surface 0°
plies proportionally more than the 90° surface plies of a (90/0)2s, and in doing so, create higher energy waves from cracks on the surface of the specimens.

Acoustic Emission Frequency

As compared to energy, a more complicated analysis technique is wave frequency. Each wave exhibits a large spectrum of frequencies that can be determined by utilizing the fast-Fourier transform (FFT), accessed via MATLAB. The essence of a waveform is the superposition of any number of waves, each of which may have a different frequency and amplitude. Once the waveform is captured, the FFT algorithm determines each of the constituent waves and their associated frequencies.

To visualize an AE event, Figure 53 shows a time series plot of the volts recorded by the sensors indicating perturbation of the specimen’s surface as a result of crack formation or propagation. The plots on the right display the amplitudes of the frequency spectrum that make up an actual AE events, where the peaks show the density of frequencies. The frequency centroid, FC, conveniently simplifies the complicated frequency spectrum to a single value to enable a comprehensible comparison of many events. Care must be taken when using FC because it does mask the information imbedded in a wave.
Figure 53—A single AE event waveform with Fast-Fourier Transform (FFT) and frequency centroid (FC). Top images show an acoustic event dominated by low frequency and the bottom images show an event with both low and high frequencies.

The analysis here only scratches the surface of the potential for future work. A deeper analysis could consist of taking short-time Fourier transforms to look at small time segments of the AE event waveform. When this is done, the attenuation and shifts of frequency as well as wave amplitude are evident as time elapses. An implication of this shows that most of the information gleaned from the waveform can be obtained from a relatively few samples immediately after the TOA, approximately $2^8$ samples for the settings used in this work. In other words, most of the recorded wave shown in Figure 53 are attenuated reflections of the event and is not necessary to record.

Figure 54 shows a high energy event is almost always low frequency, however, low energy events—which make up the vast majority of AE—have a wide range of frequencies (Figure 54). I.E., we can confidently say that a high energy event has a low frequency, and less confidently that a high frequency event has low energy, but not the converse. When the same comparison is made with frequency (Figure
that was previously done with energy (Figure 48), it is inconclusive when frequency can be used as a predictor of damage type. The bimodal distribution is evident with energy but not frequency. This may imply that most of the cracking is occurring in the 0° plies, which, for the (0/90)2s, are on the surface, which provides evidence for the hypothesis that surface cracks are more energetic (due to flexural waves) than internal waves.

Figure 54 - Energy and Frequency are loosely correlated, but not causal. The (0/90)2s architecture has many more high energy events compared to the (90/0)2s. The (0/90)2s architecture also has a large concentration of high energy events.

Figure 55 - AE Frequency versus stress for annealed specimens. Similar to energy of the (0/90)2s architecture, two peaks are evident in the histogram of frequency, again indicating a large amount of event activity in the surface 0° plies.
While not as definitive as energy, frequency centroid is marginally indicative of ply level cracking as shown in Figure 55. Recall the energy histograms of Figure 48, Figure 51, Figure 52, where upon comparing between architectures, the differences are evident:

a) The (0/90)2s composite has two distinct energy groups for AE events, visualized as a bimodal histogram. The frequency centroid also shows a similar distribution of frequencies: around 400 KHz, and around 600 kHz for (0/90)2s, and only around 500 kHz for the (90/0)2s architecture. The lack of clarity of this may be due to the method which frequency is represented, frequency centroid. Better methods of displaying AE frequency may show obvious trends that are not evident now.

b) The (90/0)2s architecture typically has low frequency events at low stress. Literature has shown that the first cracks that form in a cross-ply composite are the 90° plies[66]. This may be evident by the initial lower frequencies measured in the (90/0)2s from cracking in the surface 90° plies and higher frequencies in (0/90)2s from cracking in the internal 90° plies.

The observations presented here should provide some direction for future work to be done to find definitive relationships of the AE parameters discussed based on thermal stresses and architecture of CMC laminates.

Electrical Resistance Overview

Monitoring damage in materials requires not only sensing when damage occurs, which is possible with AE, but also evaluating the integrity of the material before, during, and after damage. Monitoring electrical resistance (ER, ohms), or similarly resistivity (ohm*mm) is useful in this regard. The CMCs used in this work are very electrically conductive relative to other CMCs, mostly due to the silicon used in manufacturing. As the material is stressed, cracks form and grow in transverse and longitudinal plies, changing the materials ability to conduct electricity. Electrical resistivity is an intrinsic material property that is independent of volume. Assuming that the manufacturing variability from panel to panel, or within the same panel has negligible differences in local and global constituent volume fractions, orientation, and
integrity, the measurement error must be due to the poor contact from the copper clips to the silver plaint directly on the specimen surface. Initial measurements of resistivity before any load is applied is summarized in Figure 56.

![Graph](image)

**Figure 56** – Composite resistivity before and after annealing. Error is likely from varying contact resistances of measurements.

Qualitatively equivalent to resistivity, or resistance, the percent change in resistance is useful to compare multiple specimens because all data is initially normalized to zero. Monitoring the percent change in resistance (%ΔΩ) gives an indication that physical change is occurring within the material, and understanding what is occurring to cause the change is the goal of this work.
Figure 57 – Typical unload-reload test, percent change in resistance, and normalized cumulative acoustic energy. ER relationship to damage is evident but confounding.

The complex electrical changes that occur in a damaging material are not entirely evident for a monotonic tensile test, therefore, unload-reload tests were done in an attempt to understand the electrical changes that occur in this material. Unload-reload tests (Figure 57) reveal that no audible damage is occurring below peak stress, however, the non-linear changes in ER indicate some, largely unknown, microstructural activity. Understanding the physical phenomenon of ER change in CMCs is a relatively nascent field of study, thus, many of the findings in this work are preliminary.

During linear elastic loading, measured changes in ER are due to Poisson contraction of the cross-sectional area. These effects are negligible as compared to the increase in resistance experienced when damage occurs. The irreversible changes in ER are evident in Figure 57, where upon unloading, a permanent resistance exists (at 60, 140, and 230 seconds). Another observation of possible significance is, upon unloading and reloading, the change in resistance increases proportionally more than peak stress. This may be an indication that the resistance is also a matter of reversible activity within the composite, such as crack closure and radial contact resistance between the fiber and the matrix.
Figure 58 - Unidirectional Percent change in electrical resistance change versus stress and strain

Figure 59 - Cross-ply (90/0)2s Percent change in electrical resistance change vs stress

Figure 60 - Cross-ply (0/90)2s percent change in electrical resistance change vs stress. Note-P5-3281-T10 had no appreciable change in resistance

The change in resistance becomes very insensitive to stress on unloading and reloading at around 100 MPa to about 190 MPa (Figure 59). It is hypothesized that, as observed by the approximate crack
opening stresses applied in the bend fixture, the initial changes in resistance are due to matrix crack closure on unloading and opening on reloading. Upon reloading, there is a lag of change in resistance as compared to the unloading, which creates a visible loop seen in Figure 58 and Figure 59. This is expected to be due to the frictional sliding of the fibers which varies depending on the direction of loading. This is only possible with a positive residual fiber tensile stress that will close the cracks on unloading, which is expected in these composites due to the matrix properties of expanding when cooled. A physical phenomenon exists in some models for brittle composites that may be of importance when contemplating ER is radial fiber/matrix debond and crack opening/closure. The stress at which the ER crosses on unload and reload may give some indication for the value of the debond stress, which is unknown.

Peak Strain and Peak Resistance

![Figure 61 – Percent change in resistance at failure as a power function of failure strain. Conductance is inversely proportional to resistance](image)

Similar to cracking, the increase in resistance ($\Omega$), appears to be predictable by the material failure strain, as shown in Figure 61.
Electrical Resistance and Inverse Tangent Modulus

A comparison of the mechanical and electrical properties of the composite is shown in Figure 62. Three unload-reload loops were performed, each at progressively higher peak stresses until the specimen failed. New cracking is shown in the time series plots on the left as the large upward slopes of the inverse tangent modulus and the resistance, while the unload-reload trends of inverse tangent modulus and resistance appear very similar.

![Graphs showing tangent modulus and resistance](image)

Figure 62 - Relationship of Percent change in conductivity and the tangent modulus of specimen P8-3284-T6, (90/0)2s, annealed.

A more tangible concept is that of the tangent modulus. The tangent modulus is equivalent to the linear-elastic modulus before damage occurs. Beyond that, the tangent modulus is measurement of the instantaneous stiffness of the material. For metals, after yielding, a monotonic but decreasing tangent modulus is indicative of strain hardening, and when unloaded, the metal typically will resume the linear-elastic modulus. This is not so with CMCs. Damage in CMCs typically initially occurs in the matrix which is not catastrophic to the integrity of the specimen. Upon unloading, the undamaged fibers close the matrix cracks and many CMCs will actually stiffen. It should be clear that the tangent modulus gives an indication of the state of the material, similarly to the changes in the electrical resistance. The similarities are not likely coincidental. The cross-over of the loops of the resistance and the inverse tangent modulus (similarly conductance and tangent modulus) are at very similar stresses, and are expected to be related to residual
thermal stress of the fibers contributing to crack closure when unloading. Little more is known about this relationship, but these properties should be inherent to any meaningful and predictive CMC model.

When calculating the tangent modulus, depending on the level of noise in the stress-strain data, the resolution may vary. The tangent modulus was calculated by making a sequential linear fit of the stress strain data (similar to Equation 15), which required some smoothing to reduce the noise in the raw stress-strain data. The mechanical damage relationships empirically determined ([57], [58]) may be applicable to the electrical properties of the composite as well and should be investigated in future work.

Electrical Resistance and Crack Density

Understanding the relationships of the material properties like tangent modulus and resistivity with and supplemental acoustic is paramount to this work. A simple, linear relationship of crack density (inverse of crack spacing) and conductance (inverse of resistance) is shown in Figure 63. One hypothesis that describes this relationship requires that the electrical current flowing through the specimen is flowing through the matrix. When new cracks form, the current must re-route to a path of higher resistance. When unloaded, some of these cracks will not close due to permanent slippage of the fiber in the matrix, which is also apparent when residual strain is present upon unloading (recall stress-strain plots Figure 31, Figure 32, Figure 33).

![Figure 63 - Crack density is linearly correlated to peak conductivity](image)

Figure 63 - Crack density is linearly correlated to peak conductivity
This empirical relationship links all the material health monitoring methods shown: crack spacing, AE events, and ER measurements and all related to strain and stress. Better inspection methods, more tests, and better fundamental understanding through models will enhance this work and enable unprecedented uses of CMCs.
CHAPTER V

CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

The main focus of this work is on the experimental methods to interrogate laminated composite materials in-situ at room temperature with the material health monitoring methods of acoustic emission and electrical resistance. Post-test microscopy confirmed the existence of damage in the form of transverse matrix cracks and were quantified for the various architectures tested.

Mechanical test results and conclusions

Unidirectional and cross-ply composites were tested in a uniaxial quasi-static tensile test with unload-reload tests to investigate the hysteretic effects of fiber-matrix sliding and its contribution to desirable CMC properties, such as toughness. These experiments may be used to validate the stress and strain prediction of CMC models. The non-linear stress-strain properties of CMCs are attributable to transverse matrix cracks allowing the fiber to debond from the matrix and experience frictional, load-dependent sliding to achieve high strains that an otherwise monolithic material could not.

An annealing process was used to reduce the residual thermal stress of the matrix in the composite which allow for higher failure strains to be reached. Without high failure strains, the composites fail with very matrix cracking leading to few acoustic events and very little change in electrical resistance. With little data from these health monitoring methods, very little can be done to investigate the damage of the materials. The annealing process reduces the ultimate strength of the unidirectional and cross-ply composites by about 5% and 10% respectively, and the proportional limit by about 20% and 30%. The modulus of the material is unchanged. Only a single as-produced specimen achieved a high failure strain,
but it is unclear why. Understanding and predicting the failure strain of as-produced and annealed composites would be of great value and should be investigated further.

Cracking in these composites is statistically variable by nature, and appeared to be well-represented by a two-parameter Weibull distribution. The variability of crack spacing has been found to decrease when the specimens are annealed, and the mean crack spacing decreases as a function of increased peak strain.

Transverse silicon veins are hypothesized to cause local compressive stress regions along the length of the composite and contributing to the non-uniform crack regions visible in micrographs of as-produced specimens.

Acoustic Emission Results and Conclusions

The 1st AE recorded is the best indication of when damage initiated in a CMC. The difference in the 1st AE stress of the as-produced and annealed unidirectional composite indicates a minimum residual stress for the as-produced specimens is around 100 MPa because a positive residual stress in the annealed specimens is still expected.

The annealed unidirectional composite was dominated by high energy events at low stresses and low energy events at high stresses, however it is unclear if the energy level is indicative of specific damage types.

The AE from the cross-ply composites exhibit several interesting unique characteristics. The (0/90)2s composites showed a bimodal energy distribution, which indicates that the surface 0° plies crack more than the surface 90° of the (90/0)2s architecture. Also, the initial energy of AE events was relatively lower energy than that of the (0/90)2s indicating that the internal 90° plies crack first.

The location of an AE event can be calculated based on the speed of sound of the material and the difference in time-of-arrival of the stress wave at sensors above and below the crack source. Plotting the location of all AE events for the as-produced unidirectional composite showed non-uniform crack spacing,
or localized crack banding. The annealed specimen showed low stress localized cracking but became uniform after the proportion limit. This phenomenon of damage localization is not well understood and should be more rigorously investigated.

Modern signal processing techniques provide methods for calculating useful wave properties, such as frequency. A simplification of frequency is frequency centroid, where the entire frequency spectrum is represented by a single frequency. This enables thousands of AE events to be compared together and patterns recognized. Similar to energy, the (0/90)2s architecture has a bimodal frequency centroid distribution around 400 kHz and 600 kHz, and may be explained by the same deductions that were made of energy. While frequency centroid is a convenient simplification, it may be an oversimplification and alternative metrics should be considered.

Electrical Resistance Results and Conclusions

Using the change in electrical resistance as measurement of material integrity is a relatively new tool that has the potential to not only provide information about the stress history of a composite by through in-situ health monitoring, but also enhances composite damage understanding which may lead to better physical predictive models of composites. Understanding both mechanical and electrical constitutive laws of materials will validate or invalidate current models, and give direction to future modelling attempts necessary to make CMCs a viable alternative to current state-of-the-art high temperature materials. When the composites in this work are loaded and unloaded, hysteresis is measured through residual strain, which is also present in the electrical properties as residual resistance. The resistance of a composite with existing cracks also changes uniquely as compared to when new cracks form. This indicates that there unknown physical changes on a micro scale pertaining to the fiber and matrix interaction within the composite.

The peak strain experienced for these composites correlates well to the maximum percent change in resistance via a power law relationship (or linearly to conductance). A correlation of electrical conductance to the average 0° ply crack density has also been shown, where an increase in crack density correlates to a linear decrease to conductance.
The mechanical and electrical constitutive relationships have been shown to be similar. The tangent modulus has been found to be a good indicator of a loss of stiffness in the material indicating cracking. Thus, the inverse tangent modulus (mechanical relationship) will increase as new cracks form and the resistance (electrical relationship) will follow a similar trend. Even during unload and reload, the mechanical and electrical properties show a nearly identical response.

Suggestions for future work

Suggestions for future work should focus on:

- SPLCF (sustained peak low cycle fatigue) testing to simulate application environments.
- Robust algorithm development for determining acoustic time of arrival for location determination
- CMC mechanical and electrical physics based modelling
- Supplement ER and AE health monitoring with Digital Image Correlation
- Validate frequency assumptions to link AE to damage type by simulating stress wave creation and propagation in CMCs
- Continue DC electrical resistance studies but also consider capacitance measurements via AC current.
REFERENCES


[71] G. N. Morscher, “Personal Correspondence on 4/16/2014.”.


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<td>0.1, 0.2, 0.3</td>
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<td>7.8</td>
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<td>1.53</td>
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Time Series and Stress-Strain Plots of All Tests

Unidirectional, $0^\circ$

Crossply, (0/90)2s
Crossply, (90/0)2s
The focus of this research considers only continuous fiber lamina/plies with cross-ply and unidirectional architectures, where the bulk of the work consists of experimental results used to quantify damage in materials. In order to show an application of some of the experimental results presented in the preceding chapters, an elementary attempt of modelling a CMC with damage is shown. The manufacturing process of this material is unknown, so many properties and processing conditions are estimated, such as volume fractions, moduli, Poisson’s ratio, temperature change and the coefficient of thermal expansion (CTE). Measured composite modulus, specimen dimensions, and digital micrograph analysis provides enough information to roughly estimate composite level properties like modulus and ply dimensions, as well as micro-scale information like constituent volume fractions. Based on the volume fractions of all the constituents and the isotropic properties based on literature (fiber[68], matrix[69], silicon[69], interphase[17]), macro scale properties are calculated based on multiple homogenization steps.

The micro scale of a single fiber to the meso scale of the CMC laminate are considered, where the process of modelling with properties summarized in Table 6 will be as follows:

1. Micro-scale (single fiber)
   a. Identify fundamental constitutive elements and their elastic properties
      i. SiC Fiber, BN interphase, SiC matrix, silicon matrix
   b. Determine the fundamental constituent volume fractions via micrograph image analysis
   c. Calculate homogenized properties of matrix consisting of silicon and silicon carbide
d. Develop a representative unit cell of single fiber, interphase and matrix and homogenized
to determine the effective properties assuming a transversely isotropic lamina

2. Meso-Scale (laminated plate)

a. Determine the mechanical properties of a laminated plate based on number of plies,
dimensions, and lamina orientation using classical laminated plate theory

b. Replicate the effects of cracking damage through a non-linear stress-strain relationship of
a laminated CMC plate based on a unidirectional crack dependent model by Pryce and
Smith

Table 6 – Fundamental constituents of this composite system. Free Silicon CTE is valid assuming ΔT = -1400

<table>
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<tr>
<th>Homogenized material</th>
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<th>Volume fraction</th>
<th>Linear Modulus</th>
<th>Poisson Ratio</th>
<th>Shear Modulus</th>
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<td>$v_{xy}$</td>
<td>$G_{xy}$, GPa</td>
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<td>Isotropic Matrix</td>
<td>matrix = Si + Si</td>
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<td>0.18*</td>
<td>154*</td>
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<td>Lamina = fiber+interphase+matrix</td>
<td>1.00</td>
<td>344*</td>
<td>185*</td>
<td>0.18*</td>
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<td>Balanced, Symmetric</td>
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<td>265***</td>
<td>0.13***</td>
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*Method of cells, ** Rule of Mixtures, *** Classical Laminated Plate Theory

Silicon has complex thermal properties that make modelling challenging. As silicon solidifies from
a liquid to a solid, large volume expansions occur, but once solid, has a non-linear, positive CTE [70]. In
order to approximate a linear CTE for silicon, a weighted average value for its solid phase was used as well
as the volume expansion during solidification [70]. The volume expansion during solidification is far greater
(estimates are 10% increase in volume [71]) than the more typical contraction when solid and cooling,
which is why silicon has such a large, negative (expands when cooled) CTE.

Micro-Scale Linear-Elastic Mechanics
Volume fractions were determined by using a micrograph representing a single lamina, or ply.

Figure 64 shows a micrograph of the cross-section of the composite and the associated histogram of the shade of each pixel (from 0-255), and based on the four regions, the volume ratios can be determined.

**Effective Silicon and Silicon Carbide homogenization properties**

In order to model this CMC based on currently available models, certain constituents must be homogenized. Consider a three phase system of a fiber, interphase and a matrix. It is clear that the micrograph shown in Figure 64 has large silicon regions as well as a SiC matrix, but these unique constituents must be homogenized to efficiently model by using a process called the method of cells [27]. When this aggregating method is properly used, two separate materials are consolidated and represented as one, effective material. Figure 65 (step 1) represents a unit cell of silicon represented as dark cells and SiC as the light cells. The homogenized matrix has the properties shown in Table 6.

**Effective Lamina Properties**
Using the constitutive properties of the fiber and interphase and the homogenized matrix consisting of SiC and Si regions, the fiber/interphase/matrix lamina properties can be approximated by using the method of cells again (step 2, Figure 65).

**Step 1-homogenize matrix**

**Step 2-homogenize fiber, interphase, matrix**

![Diagram](image)

Figure 65 - Representative unit cell for the matrix and the continuous fiber lamina to homogenize properties by the method of cells[27]

The homogenized lamina properties are summarized in Table 6. Due to the continuous fiber and interphase layer, the material properties are no longer isotropic, but transversely isotropic, which has five independent material constants, $E_0$, $E_90$, $v_{12}$, $G_{12}$, $v_{23}$. [39], [56]. It is important to model this lamina (which has a soft interphase) as transversely isotropic because the directional properties are significantly different.

The consequences of this are evident when studying cross-ply composites, which are significantly softer than a comparable unidirectional composites due to the transverse properties.

**Meso-Scale Linear-Elastic Mechanics**

The linear-elastic properties of a laminated composite plate were studied by applying classical laminate plate theory[29], [56], [72]. With the assumed properties of a lamina given in Table 6, estimates can be made of the ply stress given the direction of the ply, which is either 0° or 90° in this work. If the
thermal properties are neglected of this composite, the analysis is relatively simple. However, possibly the most perplexing challenge in understanding this composite system is the final thermal stresses caused by the mismatch of constituent material thermal properties, and since the damage of the material is very dependent on the thermal stresses, it requires attention.

If it were possible to model every atom, then a perfect bottom-up model could be developed for this composite and the need for assumptions would not exist. However, the current state-of-the-art technology cannot manage this level of detail [6], so in order to make the modeling feasible, many assumptions are made. The results of Table 6 are possible only from assumptions such as homogenizing the constituents in a transversely isotropic lamina. Being aware and understanding these assumptions is critical to using the various modelling methods, such as method of cells, classical laminated plate theory or models that consider cracking like the Pryce and Smith damage model.

Meso-Scale Effective Linear-Elastic Laminate Properties

Classical-laminated plate theory for continuous fiber-reinforced plates is a simple, fast method to determine the linear-elastic properties of a plate assuming plane stress. To show the differences in thermal loading and applied loading, a 0.05% strain was applied in the ‘x’ direction, or the longitudinal direction as well as a thermal loading from 1400 C° to 0 C° due to the lamina CTE mismatch (α₁ and α₂) and cooling during manufacturing. A trivial modeling case on the meso-scale is the unidirectional composite in Figure 66. The entire composite has the same properties of a single lamina due to the 0° orientation of the plies being the same. Figure 66 shows that each ply has the same stress and strain, and matches the linear-elastic properties shown in the stress-strain plot in Figure 31. Although there is no lamina induced thermal
stress, on the micro-scale, there is still a fiber/matrix CTE mismatch causing the matrix to have a residual compressive stress and the fiber in tension (which is not shown in Figure 66).

Figure 66 – Unidirectional 8 ply composite stress and strain due to thermal residual stresses and applied loading

The ply level stress in the balanced, symmetric cross-ply is much more complicated. Figure 67 shows the estimated stresses of a (90/0)2s composite tested in this work. Not only is there fiber/matrix CTE mismatch, but a lamina CTE mismatch. To visualize the lamina thermal stresses, Figure 67 shows the stresses in the loading direction ($\sigma_x$) due to thermal and applied load. The 90° plies are in compression while the 0° plies are in tension due to the mismatch of the homogenized lamina properties $\alpha_1$ and $\alpha_2$. Once a load is applied, the compressive thermal stresses are overcome in the 90° plies and cracking occurs. To better understand ply level damage, future work should investigate the varying cross-ply stresses as compared to unidirectional plies and compare the differences in the first few acoustic events.
Most theories of how damage occurs in composites, especially for CMCs, is for unidirectional layups[30], [34], [73], [74]. The toughness and complex hysteretic behavior found in CMCs is due to cracking and frictional debonding in the longitudinal 0° plies, as the transverse 90° plies exhibit very little, if any toughness. These unidirectional damage models can be used to predict cross-ply mechanics, but additional assumptions to adapt the model may compound the error. Analytical cross-ply damage models exist, but, again, the assumptions limit the applications to only simple laminated plates[74]. As described in the literature review, many novel damage modeling methods to account for frictional sliding have been proposed, especially late in the 20th century but do not provide a holistic framework for large scale engineering designs with CMCs. However, given the progression of engineering and computing of the last decade, multi-scale modeling appears to be viable for analysis and design. Multi-scale modeling [6], [41] captures the micromechanics of the fiber and matrix and uses that information to create a high-fidelity model.
simulation of an entire structure with realistic computation times [75] to assist in designing state-of-the-art, safe, complex CMC components.

The linear-elastic properties of a laminated plate can be approximated via classical laminated plate theory. However, once cracking begins, these properties are no longer valid and a more complex damage model must be used to explain the toughening and dissipated energy of the CMC. As explained in the literature review, a unidirectional damage model developed by Pryce and Smith[33] was used based on empirical crack spacing measurements. This model captures the essence of damage in a unidirectional CMC but lacks many of the details necessary for a robust bottom-up predictive model. The primary goal of the damage modelling is to provide a simple introduction to CMC damage micromechanics.

Effective PS Damage Model Properties

In order to apply the Pryce and Smith damage model, a homogenization of the interphase and matrix is necessary. Assuming the interphase and matrix stay perfectly bonded, and the fiber debonds from the interphase[76], we can combine the interphase and matrix via rule of mixtures. Note that the volume fractions used here are relative to only the interphase and the matrix, hence V_i+V_m=1. Example calculations for results in Table 6 are shown in Equation 23 and Equation 24.

\[
E_0 = E_I V_i + E_m V_m = 10 \times 0.07 + 364 \times 0.93 = 339 \text{ GPa}
\]

Equation 23 - Rule-of-Mixtures of interphase and matrix for PS model [56]

\[
\alpha_0 = \frac{E_I \alpha_I V_i + E_m \alpha_m V_m}{E_I V_i + E_m V_m} = \frac{10 \times 5.2e-6 \times 6 \times 0.07 + 364 \times 3.5e-6 \times 6 \times 0.93}{10 \times 0.07 + 364 \times 0.93} = 3.50 \times 10^{-6}/^\circ\text{C}
\]

Equation 24- Equation calculating the CTE based in rule-of-mixtures [56]

To simplify the annotation used in the PS damage modelling, the fiber is denoted with the subscript “f”, and the homogenized SiC and silicon are assumed to be an aggregate matrix material, denoted with subscript “m”.

Based on the calculations made for the moduli and CTE in Table 6, the stress of the fiber and the matrix can be estimated.
Recalling Equation 2, and using the experimentally determined composite modulus $E_c=365$ GPa, we can estimate the stress in the matrix at the 1st AE of an as-produced specimen as:

$$\sigma_m^{T} = \frac{E_f E_m V_f}{E_c} \Delta T (\alpha_m - \alpha_f) = 420 \times 339 \times 0.25 \times 1400 \times \frac{(3.5 - 5.1)}{365} \times 10^3 = -218 \text{ MPa}$$

Equation 25 – Residual thermal matrix stress with no applied loading [34]

$$\sigma_m(\sigma) = \sigma \frac{E_m}{E_c} + \sigma_m^{T} = 374 \frac{339}{365} - 218 = 130 \text{ MPa}$$

Equation 26: matrix stress at 1st AE due to applied loading and residual thermal stress of the annealed unidirectional composite [34]

Figure 68 - PS Unidirectional damage model compared to actual test results. Interfacial shear stress, $\tau$, is the only unknown parameter and is best-fit to the model. The matrix CTE of $\alpha_m=4.38 \times 10^{-6}$ was used because it provided a better fit compared to the calculated value of $3.5 \times 10^{-6}$ from Table 6.

The results of the PS model are shown in Figure 68. All the properties for the PS model were taken from Table 6, except the matrix CTE. The calculated value from the table, $\alpha_m=3.5 \times 10^{-6}$ (C°) gave unreasonably large residual stresses when simulated. The value of $\alpha_m=4.38 \times 10^{-6}$ (C°) was found by trial and error to give a much better fit to the residual strain of the actual test results. Recalling Equation 25, the calculated residual matrix stress of -218 MPa for an annealed specimen is abnormally large compared to other calculations[62], especially considering that the annealing process is expected to reduce the residual matrix stress. It is concluded that the value of $\alpha_m=3.5$ must be too small and is likely erroneous due to the method of linear homogenization of SiC and Si as well as assumptions leading to error in the PS model. The interfacial shear stress, $\tau=15$ MPa is an estimate for annealed specimens, but is low compared to other work where $\tau=50$ MPa were found with as-produced SiC/SiC specimens [62]. If $\tau$ is increased, than the simulated loop size would decrease and fit the experimental results better. But without any published work
with results of annealed specimens, the accuracy of this value is unknown. The empirical crack spacing value of $2s = 0.218$ cracks/mm from Figure 42 is expected to be a reasonable estimate. Another source of error for this example is the fiber/matrix load transfer length ($x'$) becomes larger than $\frac{1}{2}s$, which invalidates the PS model (middle plot of Figure 68). Clearly many of the estimates used in this work are not exact, and the model does not capture many of the statistical qualities of these material. The unload-reload portion of the stress-strain plot are modeled as quadratic functions (Equation 7, Equation 8), which by inspection of the actual test results, is not sufficient to capture to micromechanical activity present in this material.

Better models currently exists to model CMC cracking and debonding, but are much more complicated than the PS model demonstrated here and require a significantly larger investment of time and intellect.

Modelling results and conclusions

In order to model a composite, micrograph image analysis tools were used to provide accurate constituent volume fractions. Several homogenization methods, such as rule-of-mixtures and method-of-cells, were necessary to apply this composite to existing elastic and damage models. The residual stress state in a cross-ply CMC is much more complicated compared to a unidirectional architecture due to the transversely isotropic nature and various orientations of the plies.

A simple unidirectional damage mechanics model of CMCs was presented and applied based on empirical data of acoustic emission and crack spacing. The error between experimental results and the model are most likely due to incorrect initial estimates of material properties, namely silicon CTE, and generalizing assumptions of the PS damage model. While this model is not a robust solution to predicting damage in CMCs, it is instructional to understanding the micromechanics of CMC damage.

Understanding residual thermal stress, matrix cracking and closure in longitudinal and transverse plies, fiber debonding and sliding, and interphase wear are non-trivial physical phenomenon that require fundamental understanding when modelling SiC/SiC composites. CMC modelling efforts should focus on not only mechanical but electrical properties to properly capture the physics of CMCs. Predictive
mechanical and electrical models of CMCs is of extreme importance to the application of these materials. Understanding the relationships of stress, strain, electrical, fatigue, and thermal responses of CMCs would allow for application of actual components to be designed and safely used.