Micro-Raman Investigation of Residual Stresses in SiC/SiC Composites

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MICRO-RAMAN INVESTIGATION OF RESIDUAL STRESSES IN SiC/SiC COMPOSITES

A thesis submitted in partial fulfillment of the requirements for the Departmental Honors Degree

By

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May 2016
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ABSTRACT


Ceramic Matrix Composites (CMCs) are being developed for use in extreme operating conditions. Specifically, there is interest to replace superalloys with Silicon-Carbide/Silicon-Carbide (SiC/SiC) CMCs in the hot section of gas turbine engines because of their lower densities, high temperature performance, and oxidation resistance. Due to the high temperature processing conditions of SiC/SiC CMCs, there are thermal residual stresses inherent to the material. This study focused on using micro-Raman Spectroscopy on as manufactured SiC/SiC CMCs to measure and investigate the residual stresses within a fiber and the matrix material. Following the silicon Raman active mode at 520 cm$^{-1}$ and the SiC Raman active mode around 790 cm$^{-1}$ within the composite, residual stresses within the matrix and the reinforcing fibers could be investigated with a spatial resolution of 1 micron. Understanding the residual stress will enable better understanding of behavior and life performance in application environments.
ACKNOWLEDGMENTS

I would like to thank my technical advisor and mentor Dr. Craig Przybyla of the Air Force Research Laboratory for all of the help and support he has provided me while working at AFRL. I would not be where I am today if it were not for his guidance and advice.

I would also like to thank my advisor, Dr. Maher Amer of Wright State University. Dr. Amer has helped immensely with his knowledge and expertise.

Many people at AFRL have not only helped on this project, but have also helped me to grow as a researcher. I would like to thank Jennifer Pierce, Dr. Travis Whitlow, Dr. Eric Jones, and Larry Zawada for their help.

Lastly, I would like to thank my friends and family for their constant support. Andrew Detwiler, Stephen Hawkins, Brenna Nowacki, Nick Engel, and Nathan Levkulich have been a great source of encouragement. Most importantly, I would like to thank my parents and sisters for always being there and providing me with the resources to be successful.
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I. INTRODUCTION

This chapter provides a background on Ceramic Matrix Composites (CMCs), SiC/SiC CMCs, residual stresses, and Raman Spectroscopy. The first section will describe basic CMCs, their properties, and their reinforcement types. The next section contains information about the applications of SiC/SiC CMCs and how they are manufactured. The third section discusses residual stresses and their effects in CMCs. In the last section, the technique of Raman Spectroscopy is discussed.

1.1 Ceramic Matrix Composites

1.1a Basic Properties of CMCs

A basic Ceramic Matrix Composite (CMC) consists of ceramic reinforcements (in the form of fibers or particulates) surrounded by a ceramic matrix [1]. Typical ceramics are known to be brittle, but by adding a second phase material in the form of particles, fibers, or whiskers, crack growth is interrupted [2]. Continuous fibers within the matrix provide the highest increase in toughness and strength of CMCs when compared to other forms of reinforcement [2]. CMCs are being developed to replace high temperature metal alloys in high-temperature applications or enable system concepts previously inaccessible due to the temperature limitations of current commercially available structural materials. The density of CMCs is significantly less than current high temperature alloys, and CMC exhibit mechanical properties sufficient for a wide range of applications [1, 3].
1.1b Reinforcement Types

Figure 1.1: Reinforcement types for composites include (a) particulate reinforcements, (b) discontinuous whisker reinforcements, and (c) continuous fiber composites [4].

There are multiple types of reinforcements for composites, including particulate reinforcements, discontinuous fiber whisker reinforcements, and continuous fiber composites, including laminate or woven composites [5]. Figure 1.1 shows a particulate reinforced composite, a discontinuous whisker reinforced composite, and a laminate composite [4]. By adding ductile particles to a brittle matrix, such as a ceramic matrix, the toughness of the material is increased because cracks cannot move as easily through the particulates [4]. Particulate reinforced ceramic composites are used in applications such as cutting tools, bridges, and buildings [4]. Discontinuous ceramic whisker reinforcements can either be used to strengthen materials, or act as a “filler” material to help reduce costs of the composite material [5]. Continuous fiber reinforcements, when oriented in a unidirectional manner, provide the maximum strength and stiffness in the fiber direction [4]. In order to make the composite strong in both the longitudinal and transverse direction of the fibers, a laminate can be made [4]. For laminates, several layers of a continuous fiber composite are stacked upon one another with alternating orientations [5]. Figure 1.2 shows an example laminate composite with varying orientations for each layer [4]. In woven materials, the fibers are interlocked with one
another by weaving, braiding, or knitting the fiber tows [5]. This orientation creates an out-of-plane orientation, allowing for the ability to have structural, thermal, or electrical properties in this direction [5]. The ability to change reinforcement type or orientation enables optimization of a material for a particular application.

Figure 1.2: An example laminate composite is shown with various fiber orientations [4].

1.2 Silicon-Carbide/Silicon-Carbide Composites

Silicon-Carbide continuous fiber reinforced Silicon-Carbide matrix CMCs (SiC/SiC CMCs) are able to withstand high temperature environments, have oxidation resistance, and have mechanical properties that make them suitable for use in advanced engine capabilities [2]. Ni-base superalloys are currently the primary material used for hot section components of gas turbine engines, but SiC/SiC CMCs have a combination of properties that make them more promising than their metal counterparts. SiC/SiC CMCs also exhibit oxidation resistance at temperatures much higher than superalloys, making them suitable for use in an oxidizing environment [2]. SiC/SiC CMCs enable many possible improvements to current gas turbine engines, including reduced cooling
requirements, simpler component design, reduced structural weight, and improved fuel efficiency [3]. Components that may be replaced by CMCs include hot gas path components, as imaged in Figure 1.3 [6].

![Image of engine components](image)

**Figure 1.3:** The parts highlighted in this image are currently metallic parts that are candidates to be replaced by CMCs [6].

### 1.2a Processing Techniques for SiC/SiC Composites.

CMCs are produced through various processing techniques, including, but not limited to, chemical vapor infiltration (CVI) and slurry melt infiltration (SMI) [1, 2]. CVI and SMI are the two techniques used to manufacture the material used in this study. Because of the high melting temperatures of the ceramic materials used to make CMCs, processing temperatures up to 1400° C are not uncommon [2, 3]. It is important
to note that finding compatible fiber and matrix materials is vital. Due to chemical and mechanical interactions at the bonding surface, degradation of the fiber reinforcement during processing can occur [2]. Also, to ensure toughness as well and strength in CMCs, sufficient conditions for debonding and stress transfer must be met, respectively, at the bonding interface between the fiber and the matrix [2]. In some cases, a fiber coating may be applied to ensure a proper interface between the fiber and matrix [2].

Figure 1.4: The CVI process in which reactant gases are exposed to a porous preform, depositing a solid matrix on top of the fibers [2].

In CVI, a porous preform of fibers is exposed to reactant gases which penetrate the pores of the preform [2]. The gases react to form a solid matrix on top of the fibers [2]. Using the CVI process, complex shaped objects can be formed, creating components
such as turbine nozzle flaps, rotors, and combustors [2]. A diagram is shown of the CVI process in Figure 1.4 [2].

The SMI process involves infiltration of a prepreg fiber preform is infiltrated with matrix material that is heated to become viscous. When the matrix molten matrix material comes into contact with the fiber preform, the wetting properties of the fiber preform are such that it wicks the molten matrix material into and around the fibers [7]. In some cases, the fiber preform is first infiltrated with a resin [7]. After consolidation, pyrolysis leaves reactant components from the resin in the porosity which react with the molten material being infiltrated, forming a secondary matrix [7]. In SiC/SiC SMI materials, a Si metal is typically infiltrated into a preform with significant excess carbon. The molten Si metal reacts with the excess carbon to form a predominantly SiC matrix [3, 6]. This process results in high densification and only leaves a small percent of porosity [2]. The matrix processing techniques can be seen in Figure 1.5 [7].

![Matrix Processing Diagram](image)

**Figure 1.5: Melt Infiltration Process for SiC/SiC CMCs [7].**

### 1.3 Residual Stresses

Residual stresses are inherent to the material, and do not depend on any external loads [8]. Residual stresses are either tensile or compressive, and all of the residual
stresses within the material come to equilibrium, summing to zero force and moment resultants [8]. Residual stresses occur due to manufacturing processes and can also develop during the life cycle of the component [8]. Residual stresses affect the material, and can be considered as an addition to the loading stresses [8]. Depending on the location and type of residual stress, it can help or hinder the material [8]. Compressive residual stresses prevent crack growth by biasing the average stress in the compressive direction during loading [4]. On the other hand, tensile residual stresses promote crack growth [4]. There are various methods of measuring residual stresses, including Raman Spectroscopy, X-Ray Diffraction, FIB Sectioning with Digital Image Correlation, and many other techniques [8]. However, Raman spectroscopy technique is far superior to all other techniques in respect to its spatial resolution. While some techniques, such as X-Ray Diffraction have a spatial resolution on the order of millimeters, Raman provides a spatial resolution on the order of micrometers [9].

1.3a Residual Stresses in SiC/SiC Composites

In SiC/SiC composites, thermal residual stresses are proportional to the mismatch in the coefficients of thermal expansion (CTEs) between the fiber and matrix materials, and the difference between the high processing temperature and room temperature [2]. If the matrix CTE is higher than the fiber CTE, then there will be inherent axial tensile stresses in the matrix, and axial and radial compressive stresses in the fiber [2]. Conversely, if the matrix CTE is lower than the fiber CTE, then there will be axial compressive stresses in the matrix and axial and radial tensile stresses in the fiber [2]. In order to reduce thermal residual stresses, it is important to minimize the difference in the CTEs [2].
As discussed previously, in SiC/SiC SMI CMCs, molten silicon metal is melt infiltrated into the preform of fibers with excess carbon. Most of the silicon reacts with the excess carbon, forming silicon carbide. However, not all of the silicon reacts, leaving residual pools of silicon. During cooling and solidification of silicon, there is a significant volume expansion [10]. This volume expansion then induces compressive residual stresses in the surrounding material.

1.4 Raman Spectroscopy

Raman spectroscopy is a technique used to measure various fundamental vibration modes of a material [11]. When using Raman spectroscopy, a laser is first focused onto the area of interest for the material. Then, the intensity, in terms of its wavelength, of the scattered radiation is then measured [11]. These readings are interpreted by the dispersion system, and then detected. A simple diagram of a Raman spectrometer is displayed in Figure 1.5 [11].

![Figure 1.6: A simple diagram of a Raman spectrometer [11].](image)

By using a motorized stage, Raman mapping can be done. In this technique, many Raman measurements are completed, and a conglomerate map of these measurements is produced. These maps can be used to measure chemical composition, phase structure, and stresses within a material in a nondestructive manner [9]. Raman spectroscopy has high strain sensitivity, with a high spatial resolution, making it ideal to
measure residual stresses [12]. Results from Raman spectroscopy are typically reported in terms of the wavenumber of the constituent being measured [11]. A shift in the known value for the wavenumber of a material indicates a stress in the material [13]. Raman spectroscopy can be used to analyze the mechanical states of heterogeneous materials, such as multilayer composites [13].
II. MATERIAL INFORMATION, MECHANICAL TESTING, AND
EXPERIMENTAL SETUP

This chapter will give a description of the material tested, the mechanical testing completed on the material, and the experimental setup used for Raman Spectroscopy. The first section describes the material orientation, its fibers, and its processing. The second section gives an overview on mechanical testing previously completed on the material. The last section outlines the experimental setup used to conduct Raman Spectroscopy.

2.1 Material Information

2.1a Material Orientation

The material investigated in this study was supplied by the Air Force Research Laboratory, and was produced by Honeywell Advanced Composites in 2001. It is a SiC/SiC SMI CMC, containing continuous Syrlamic ceramic fibers. The fibers were first woven into a five-harness satin weave preform. Various types of weave patterns are imaged in Figure 2.1, including the five-harness satin weave [7]. For this set of material, two slightly different processing techniques were used, resulting in differing mechanical properties.
2.1b. Sylramic Fibers

In SiC/SiC CMCs, SiC fibers are used. These fibers are produced by curing and heat treating material from polymer precursors. When taken through the heat treatment, which causes impurities to decompose, the polymer precursor converts into ceramic silicon-carbide fibers [14]. The specific type of fiber used for the material in this study is Sylramic fiber. In order to produce a fiber with a higher tensile strength, boron is added into the fiber’s microstructure as a sintering aid, and the fibers are sintered at 1600° C after going through the heat treatment process [3, 14]. By adding the sintering aids, after processing the resulting fiber is highly dense with a higher tensile strength than the non-sintered fibers [14].
2.1c. Sample Processing

For Plate 1, the fiber preforms first went through CVI of boron nitride [3]. This preform was then placed into a CVI SiC reactor [3]. After going through the CVI processes, a SiC particulate is infiltrated into any of the remaining porosity at room temperature [3]. Following this, silicon metal, at 1400° C, is melt infiltrated into the fiber preform [3]. The silicon reacts with excess carbon to form silicon carbide. This process results in a low porosity for the matrix material [3]. The processing for this material is outlined in Figure 2.2.

![Figure 2.2: Processing Steps for Plate 1](image)

For Plate 2, the fiber preform went through a similar process, with one major change. Before any CVI or MI, the preform goes through another thermal treatment, developed by NASA [3, 14, 15]. The heat treatment forces the excess boron, from the sintering aids, to diffuse to the surface and interact with nitrogen in the environment, creating an in-situ boron nitride (iSBN) fiber coating around the fibers [3, 14, 15]. The fiber preform then goes through the same CVI and MI process as the previous plate. The processing steps for Plate 2 are listed in Figure 2.3.

![Figure 2.3: Processing Steps for Plate 2](image)

By diffusing the boron to the surface, the tensile strength of the fiber is retained and the creep resistance and electrical conductivity are improved [3, 15]. Also, the BN fiber coatings prevent the SiC fibers from directly contacting one another, which provides a
more stable fiber in conditions where oxidation can occur [3, 15]. The comparison of the different contact areas of fibers for the two plates can be seen in Figure 2.4 [3].

![Figure 2.4: With the in-situ boron nitride coating, the SiC fibers are separated from one another [3].](image)

### 2.2 Mechanical Testing

Mechanical testing was completed on the samples at Honeywell Advanced Composites testing facility. The properties in Table 2.1 were from tension tests in a room temperature environment [16]. The stress-strain plots for these tests can be found in Appendix I.

<table>
<thead>
<tr>
<th></th>
<th>Peak Stress (MPa)</th>
<th>Peak Strain (%)</th>
<th>Proportional Limit (MPa)</th>
<th>Elastic Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Plate 1</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample 1</td>
<td>250</td>
<td>.2546</td>
<td>150</td>
<td>209</td>
</tr>
<tr>
<td>Sample 2</td>
<td>292</td>
<td>.3234</td>
<td>165</td>
<td>192</td>
</tr>
<tr>
<td><strong>Plate 2</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample 1</td>
<td>447</td>
<td>.5814</td>
<td>180</td>
<td>198</td>
</tr>
<tr>
<td>Sample 2</td>
<td>398</td>
<td>.5384</td>
<td>150</td>
<td>199</td>
</tr>
</tbody>
</table>

Table 2.1: Mechanical Properties of Honeywell Material [16].
It is clear that the difference in the mechanical properties for the two plates is due to the different processing techniques used for each plate and its effect on the composite microstructure. The material for Plate 1 had a degraded ultimate tensile strength, due to boron on the fiber surface aiding silica based glass formation when the composite was processed in an oxygen rich environment [3]. When the glass formed, neighboring fibers were bonded, resulting in the reduced ultimate tensile stress [3]. The boron on the fiber bulk usually occurred at the fiber grain boundaries, resulting in reduced creep resistance, rupture resistance, and thermal conductivity [3]. These issues were alleviated in the Plate 2 material by employing the previously mentioned technique, developed by NASA, of adding mobile boron sintering aids, resulting in improved mechanical properties [3].

2.3 Experimental Setup

A sample was taken from each of the as manufactured plates produced by Honeywell. This sample was then mounted and polished to a 1 micron finish, as pictured in Figures 2.5 and 2.6.
Raman spectra was then collected on the material using a Renishaw 2000 system, as imaged in Figure 2.7, equipped with a 514.5 nm excitation Ar+ laser, a 1800 line/mm grating, and a back depleted CCD for data collection. Laser power at the sample was kept below 2 mW to avoid sample heating. A 200x objective was used to focus on the sample. A motorized stage was used to map the sample with a step size of 2 micrometers, including a fiber and the matrix surrounding the fiber. The signals were detected using a CCD camera. The data collected was then imported into MATLAB®, and the outputs produced were chemical composition and residual stress maps of the area analyzed.
III. RESULTS

This section outlines the data acquisition, the methods used to analyze the data, and the results from the areas of interest. First, specific regions were chosen to collect Raman spectra from. Next, data analyzation was completed using MATLAB®. Lastly, composition maps and residual stress maps were produced. A discussion of these results will also be provided.

3.1 Data Acquisition

For this experiment, specific areas of interest were chosen to analyze data from. An area including both silicon carbide matrix and unreacted silicon was chosen. In Figure 3.1, an area of interest from Plate 2 is shown.
By using WiRE® software produced by Renishaw®, areas of the samples were mapped in a bi-directional sequence with a step size of 2 µm and Raman spectra were collected from each point. The Raman spectra were analyzed to determine peak position, width, and area of both silicon and silicon carbide Raman peaks. Typical Raman spectrum showing both silicon (around 520 cm⁻¹), and silicon carbide peaks (around 790 and 970 cm⁻¹) is shown in Figure 3.2 [17]. The peak position and integrated peak area data were imported into MATLAB®, and used to produce material composition and stress distribution maps.

![Figure 3.2: Typical Silicon and Silicon Carbide peaks [17]](image)

### 3.2 Chemical Compositions and Residual Stress Maps

MATLAB® was used to create contour maps based on the Raman mapping experimental results. Figure 3.3a shows the residual unreacted silicon distribution calculated from the integrated intensity of the silicon Raman band around 520 cm⁻¹ and Figure 3.3b shows
the residual stress measured in the silicon based on the shift in the silicon Raman peak position for Plate 1. Similarly, Figure 3.4 shows the same maps for Plate 2.
Figure 3.3: Plate 1 Unreacted Si Distribution and Compressive Residual Stress Maps
Figure 3.4: Plate 2 Unreacted Si Distribution and Compressive Residual Stress Maps

3.3 Discussion of Results

As previously mentioned, during processing of MI SiC/SiC CMCs, the mismatches in the coefficients of thermal expansions cause residual stresses when the material is cooled. In this case, when the melt infiltrated silicon reacts with excess carbon, an exothermic reaction occurs [19]. This reaction results in residual stresses in both the fibers and the matrix. Within the fiber, residual tension occurs, and the matrix is placed under residual compressive stresses [19]. The in-plane cracking strength, at low temperatures, is increased due to the residual compressive stress within the matrix [19]. However, when the composite reaches high temperatures, near 1400° C, the residual stresses no longer have an effect on the material [19].

<table>
<thead>
<tr>
<th>Material</th>
<th>Coefficient of Thermal Expansion (10^6/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>2.6 [19]</td>
</tr>
<tr>
<td>Silicon Carbide</td>
<td>4 [20]</td>
</tr>
<tr>
<td>Sylramic Fiber</td>
<td>5.4 [14]</td>
</tr>
<tr>
<td>Sylramic-iBN Fiber</td>
<td>5.4 [14]</td>
</tr>
</tbody>
</table>

Table 3.1: Coefficients of Thermal Expansion for Si, SiC and Sylramic Fibers

As seen in Table 3.1, the coefficients of thermal expansion for the Sylramic fibers are higher than both constituents of the matrix material. As a result, the matrix material is in compression, which is confirmed by the Raman data collected. The compressive residual stresses in Plate 1 peak at 0.9 GPa, and the compressive residual stresses in Plate 2 peak at 6 GPa. Also, Plate 1 had higher unreacted silicon content, while Plate 2 had a
lower unreacted silicon content. These compressive stresses are beneficial to the overall mechanical properties of the material, as they hinder crack propagation within the material.

In the woven SiC/SiC composite, the first mode of damage is through cracking perpendicular to the loading direction [2]. The initial cracking propagates from porosity between the fiber tows and travels through the matrix material. The cracks then travel to the transverse tows of the composite and continue to propagate through the transverse fiber bundles, eventually moving into the longitudinal fiber bundles. This ultimately leads to fibers breaking, and failure within the composite [2]. The matrix material in these two plates has a mean stress that is biased toward compression due to residual stresses from manufacturing processes. These compressive stresses inhibit the initial crack growth as the cracks cannot propagate as easily. Also, it is important to note that the MI process leaves a low porosity, which also obstructs cracks from propagating [16].

On the macro scale, Plate 2 had better overall mechanical properties than Plate 1. This can be related back to the behavior on the micro scale through the Raman data. The Raman residual stress maps show a higher compressive stress in the silicon matrix for Plate 2. While the higher compressive residual stresses are not the only reason for the improved mechanical properties in Plate 2, they do play a role in the increased strength of the material. To this end, it can be summarized that different processing techniques for SiC/SiC composites result in significantly different mechanical performance. Plate 1 samples showed a much higher strength and toughness compared to Plate 2 samples. Micro-Raman investigation revealed that this increase in both strength and toughness of the composite is associated with significant increase in compressive residual stresses in
the composite matrix as well as a significant decrease in the amount of unreacted silicon. Our results are in agreement with theory of composite mechanics.
IV. CONCLUSIONS AND FUTURE WORK

Raman Spectroscopy is a technique that is capable of mapping both composition and residual stresses in melt infiltrated SiC/SiC composites. Two plates of material that went through different processing techniques were studied. Plate 1 had higher unreacted silicon content with lower compressive residual stresses in the range of 0.8-0.9 GPa, while Plate 2 exhibited lower unreacted silicon content with higher compressive residual stresses in the range of 5-6 GPa. The differences in mechanical properties can be attributed to the microstructural properties, including the in-situ boron nitride fiber coating, as well as the microstresses within the matrix of the material.

Further study will be completed on these two plates of material. The materials will go through annealing, in which they will be heated and cooled slowly. This should release the residual stresses within the material and strengthen it. The materials will go through 1 hour cycles in a furnace at 1315° C in an argon rich environment. The residual stresses will be measured in similar areas of interest after each hour cycle for both plates.
WORKS CITED


APPENDIX I

Plate 1 Sample 1: Stress-Strain Plot

In Figure A.1, the stress-strain plot is shown for Sample 1 of Plate 1. The peak stress value is at 250 MPa, with a proportional limit of 150 MPa. The elastic modulus of the specimen was measured at 209 GPa.

![Stress-Strain Plot](image)

Figure A.1: Stress-Strain Plot of Plate 1 Sample 1 from a Tension Test of MI Syrlamic Material [17].
Plate 1 Sample 2: Stress-Strain Plot

In Figure A.2, the stress-strain plot is shown for Sample 2 of Plate 1. The peak stress value is at 292 MPa, with a proportional limit of 165 MPa. The elastic modulus of the specimen was measured at 192 GPa.

Figure A.2: Stress-Strain Plot of Plate 1 Sample 2 from a tension test of MI Sylramic Material [17].
Plate 2 Sample 1: Stress-Strain Plot

In Figure A.2, the stress-strain plot is shown for Sample 1 of Plate 2. The peak stress value is at 447 MPa, with a proportional limit of 180 MPa. The elastic modulus of the specimen was measured at 198 GPa.

![Stress-Strain Plot](image)

Figure A.3: Stress-Strain Plot of Plate 2 Sample 1 from a tension test of iSBN MI Sylramic Material [17].
Plate 2 Sample 2: Stress-Strain Plot

In Figure A.4, the stress-strain plot is shown for Sample 2 of Plate 2. The peak stress value is at 398 MPa, with a proportional limit of 150 MPa. The elastic modulus of the specimen was measured at 199 GPa.

Figure A.4: Stress-Strain Plot of Plate 2 Sample 2 from a tension test of iSBN MI Sylramic Material [17].