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HYBRID MANUFACTURING PROCESS OF SIC_f/SIC COMPOSITE USING
PRECERAMIC POLYMER

by

Robert Raymond Meinders

A THESIS

Presented to the Faculty of the Graduate School of the
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In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE

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Approved by

Dr. K. Chandrashekhara, Advisor
Dr. Edward Kinzel
Dr. V. A. Samaranayake

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ABSTRACT

Continuous fiber-reinforced silicon carbide (SiC_f/SiC) ceramic composites have been increasingly used due to their high temperature strength and graceful failure mechanisms. A disadvantage is the high cost and lengthy production processes that are required to develop these materials. Polymer infiltration and pyrolysis (PIP) is one of the most attractive fabrication processes for composites due to shape flexibility, mass production and relatively low cost; however, the quality of material obtained by this method has been considered insufficient due to the microstructure defects of the material obtained. This study investigated a hybrid of multiple polymer manufacturing processes to maximize quality of the SiC_f/SiC composites while minimizing manufacturing time. The composites were laid up using a prepreg process then pressed and injected with a preceramic polymer at high pressures. The composite was then cured and pyrolyzed. This study investigated the effect of injection pressures and examines the effects on the composite physical properties and microstructure. The microstructure was evaluated by void density measurements and scanning electron microscopy. The results showed that the pressurized resin injection produces better parts than vacuum assisted reinjection.

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TABLE OF CONTENTS

	Page
ABSTRACT	iii
ACKNOWLEDGMENTS	iv
LIST OF ILLUSTRATIONS	vii
LIST OF TABLES	viii
1. INTRODUCTION	1
1.1. CERAMIC COMPOSITES	1
1.1.1. Material Introduction.....	1
1.1.2. Manufacturing	2
1.2. STUDY OVERVIEW	3
2. MATERIALS	4
2.1. SELECTION.....	4
2.2. POLYMER PRECURSOR.....	4
2.3. MICRO PARTICLE FILLER.....	5
2.4. FIBER REINFORCEMENT.....	5
3. MANUFACTURING.....	7
3.1. OUTLINE	7
3.2. MOLD DEVELOPMENT	7
3.3. SLURRY.....	9
3.4. SAMPLE LAYOUT	9
3.4.1. Vacuum Infused Laminate.	9
3.4.2. Injection Infused Laminate.....	9
3.5. LOW TEMPERATURE PYROLYSIS CURE CYCLE.....	10
3.6. REINJECTION.....	11
4. POROSITY MEASUREMENT.....	13
5. MICROSTRUCTURE ANALYSIS.....	14
6. RESULTS AND DISCUSSION	15
6.1. POROSITY	15
6.2. MICROSCOPY.....	16

7. CONCLUSIONS 20

8. FUTURE WORK 21

BIBLIOGRAPHY 22

VITA 24

LIST OF ILLUSTRATIONS

Figure	Page
3.1. Top Mold CAD (dimensions in inches).....	8
3.2. Pressure Injection Mold (top plate, spacer and O-rings on left).....	8
3.3. High Pressure Resin Injection Machine.....	10
3.4. Low Temperature Pyrolysis Cure Cycle.....	11
3.5. Part Reinjection Setup.....	12
6.1. Image of Vacuum Matrix Showing Incomplete Fiber Wet Out	17
6.2. Image of Void in Vacuum Matrix.....	17
6.3. Image of Uneven Vacuum Matrix with Void	18
6.4. Image of Pressure Injection Matrix Showing Full Fiber Wet Out.....	18
6.5. Image of Pressure Injection Matrix.....	19

LIST OF TABLES

Table	Page
2.1. Properties of SMP-10 as obtained from Starfire Systems data sheet.....	5
2.2. Properties of β -SiC particles obtained from US Research Nanomaterials, Inc.	6
2.3. Properties of Hi-Nicalon as obtained from COI Ceramics, Inc. data sheet	6
6.1. Porosity Measurements	15

1. INTRODUCTION

1.1. CERAMIC COMPOSITES

1.1.1. Material Introduction. Ceramic composites are important for advancements in the fields of high temperature applications. Ceramics are capable of withstanding higher temperatures than metal materials. One of the drawbacks to ceramics is the low damage tolerance exhibited by the materials. It is possible to improve the damage tolerance by developing ceramic fiber reinforced ceramic composites [1, 2]. Continuous fiber reinforcement increase the toughness of the ceramic materials by providing fiber-matrix interfaces that slow crack propagation in the matrix by introducing complex failure mechanisms such as matrix cracking, delamination, fiber pullout and fiber breakage, [3-6]. This improves the fracture toughness and decreases sensitivity to flaws [4].

Silicon carbide fiber reinforced silicon carbide ceramic composites (SiC_f/SiC) are used in this research for multiple reasons. SiC demonstrates excellent corrosion resistance and strength at elevated temperatures and has been cited for nuclear applications for its high damage and radiation tolerance [1, 2, 5, 7, 8]. The material retains strength and chemical inertness at high temperatures [9-11] making it ideal for use in engines and reactor components [7, 12]. Monolithic SiC is a brittle material and is considered unreliable as a structural design component due to unpredictable strength in the presence of flaws [13]. The continuous fiber reinforcement decreases material sensitivity to flaws, increases fracture toughness, and offer substantial improvements in damage tolerance over monolithic ceramic materials.

1.1.2. Manufacturing. Several processes exist to manufacture continuous fiber-reinforced ceramic composites (CFCCs). Currently, chemical vapor infiltration (CVI), and nano-infiltration and transient eutectic (NITE) processes have produced nuclear grade materials commercially, but both processes are cost prohibitive and require additives [14]. Polymer infiltration and pyrolysis (PIP) process is one of the most promising methods to produce low cost CFCCs due to the low-temperatures required for processing and the simple processing equipment. CFCCs manufactured using the PIP method can be fabricated, processed and shaped using relatively low processing temperatures and pressures resulting in reduced production costs [15-18]. The production of SiCf/SiC materials through PIP processes has suffered from long production times required to fully densify composites; however, through the application of pressure injection it should be possible to decrease the time required to reinfiltate composites.

CFCCs manufactured using PIP process have some common flaws. First, CFCCs fabricated using the PIP process have residual porosity of 10 %-15 %. The high level of porosity decreases the strength, the thermal conductivity and hermeticity of composites [19]. With poor hermeticity, oxygen can easily migrate into the material and degrade the strength of the interphase and fibers causing a compromise on the overall strength of the material [20]. Secondly, a non-stoichiometric matrix derived from preceramic precursors have shown poor performance due to excess carbon reacting with oxygen at elevated temperatures lowering the high temperature performance of the material produced.

With breakthroughs in polymer precursors combined with refined manufacturing processing, pyrolysis and heat treatment, it has become possible to produce a dense stoichiometric SiC matrix that is highly crystalline with a β -SiC microstructure. These

developments have brought new possibilities to produce high quality SiC_f/SiC composite material.

1.2. STUDY OVERVIEW

The objective of this study is to investigate the use of high pressure injection to manufacture CFCCs using a preceramic polymer matrix. In this study, samples were manufactured using both a vacuum infusion PIP and an experimental high pressure injection technique that was developed. A comparison of the resulting matrix of the two manufacturing methods was made.

2. MATERIALS

2.1. SELECTION

The materials for the project were selected to produce high quality parts that would be capable of withstanding high temperatures. SMP-10, an allylhydridopolycarbosilane, has been selected as the polymer precursor for its high ceramic yield with near stoichiometric derived SiC, relatively low volumetric shrinkage, and ease of use. Beta phase SiC powder was used as a filler to limit the effect of the volumetric shrinkage of the matrix for the initial pyrolysis. The main focus of this study is to show the improvements of pressure injection manufacturing over vacuum polymer infiltration and pyrolysis (PIP) manufacturing.

2.2. POLYMER PRECURSOR

The allylhydridopolycarbosilane designated SMP-10 is a polymer precursor acquired from Starfire Systems. SMP-10 is an ultra-high purity precursor that yields a near stoichiometric Si:C ratio upon pyrolysis completion. At room temperature the resin is a clear or amber-colored, viscous liquid and has properties as listed in Table 2.1. Starfire Systems data sheet for SMP-10 indicates the polymer undergoes a low temperature green cure between 180 °C and 400 °C. Amorphous SiC is formed at 850-1200 °C with a high ceramic yield of 72 %-78 % and relatively low volumetric shrinkage of ~35%. Nano-crystalline β -SiC is then formed at 1250-1700 °C. The SiC formed is stable up to 1800 °C in air and has a 1:1 silicon to carbon atomic ratio.

Table 2.1. Properties of SMP-10 as obtained from Starfire Systems data sheet

Property	SMP-10
Density (g/cm ³)	0.998
Appearance	Clear, Amber Liquid
Viscosity (cps at 25 °C)	40 to 100
Compatible Solvents	Hexanes, Tetrahydrofuran, Toluene
Flash Point (°C)	89
Surface Tension (N/m ²)	3

2.3. MICRO PARTICLE FILLER

The β -SiC particles used in this research project were acquired from US Research Nanomaterials, Inc. The particles are quoted as 99+% pure. The powder was used to create a slurry for the prepreg process. β -SiC particles were added to the polymer to increase the initial yield of SiC and to reduce the amount of matrix shrinkage during pyrolysis. Table 2.2 shows the properties provided by US Research Nanomaterials, Inc.

2.4. FIBER REINFORCEMENT

The SiC fiber selected for this study is Hi-Nicalon ceramic fiber acquired from COI Ceramics, Inc. The Hi-Nicalon fiber is a multi-filament silicon carbide-type fiber manufactured by Nippon Carbon Co., Ltd of Japan. The fiber is near oxygen free and is homogeneously composed of ultra-fine beta-SiC crystallites and carbon. The high purity of these fibers contributes to the high resistance to oxidation and thermal stability. These fibers are produced for the primary use in high temperature ceramic composites as

Table 2.2. Properties of β -SiC particles obtained from US Research Nanomaterials, Inc.

Property	β-SiC particles
Density (g/cm ³)	3.216
Size (μ m)	1-40
Decomposition Temperature ($^{\circ}$ C)	2700
Compressibility Coefficient	0.21×10^{-6}

reinforcement. Hi-Nicalon ceramic fibers are available as multi-filament tows, woven cloth and as chopped fibers. For this investigation, a plain weave woven cloth has been used. Table 2.3 shows the properties provided by COI Ceramics, Inc.

Table 2.3. Properties of Hi-Nicalon as obtained from COI Ceramics, Inc. data sheet

Property	Hi-Nicalon
Fiber Denier	1800
Density (g/cm ³)	2.74
Composition [wt. % (Si:C:O)]	62:37:0.5
Filament Diameter (μ m)	14
Tensile Strength (GPa)	2.8
Tensile Modulus (GPa)	270

3. MANUFACTURING

3.1. OUTLINE

For this study, two manufacturing methods were examined. A traditional vacuum infiltration manufacturing process and the new high pressure injection process were used to generate the samples examined in the study. Both processes used a hand layout process where materials were laid into a mold. Both samples were subjected to a green body cure and final pyrolysis before being examined and compared.

3.2. MOLD DEVELOPMENT

A high pressure mold was designed and fabricated for this experiment. The mold was designed to be capable of manufacturing a 27.9 cm diameter (11 in. dia.) by 0.635 cm (0.25 in) composite. The mold was required to withstand 10.34 MPa (1500 psi) of hydraulic pressure with a factor of safety of 4. The mold was built in SolidWorks 2013 (Figure 3.1) to model the stresses and analyze the safety of the mold design.

The mold was designed to have three parts: a top plate, a spacer and a bottom plate. Twenty grade 8, $\frac{3}{4}$ "-16 bolts were used to provide the required clamping force to hold the mold together. The top plate was given a resin groove to allow for an even vacuum and to allow the resin to uniformly impregnate parts from the center of the mold outwards.

The parts of the mold were manufactured from 1045 steel. The initial design used a liquid silicon gasket to seal the mold. Pressure testing indicated the presence of a leak and the mold was then retrofitted with a silicon O-ring to seal the mold (Figure 3.2).

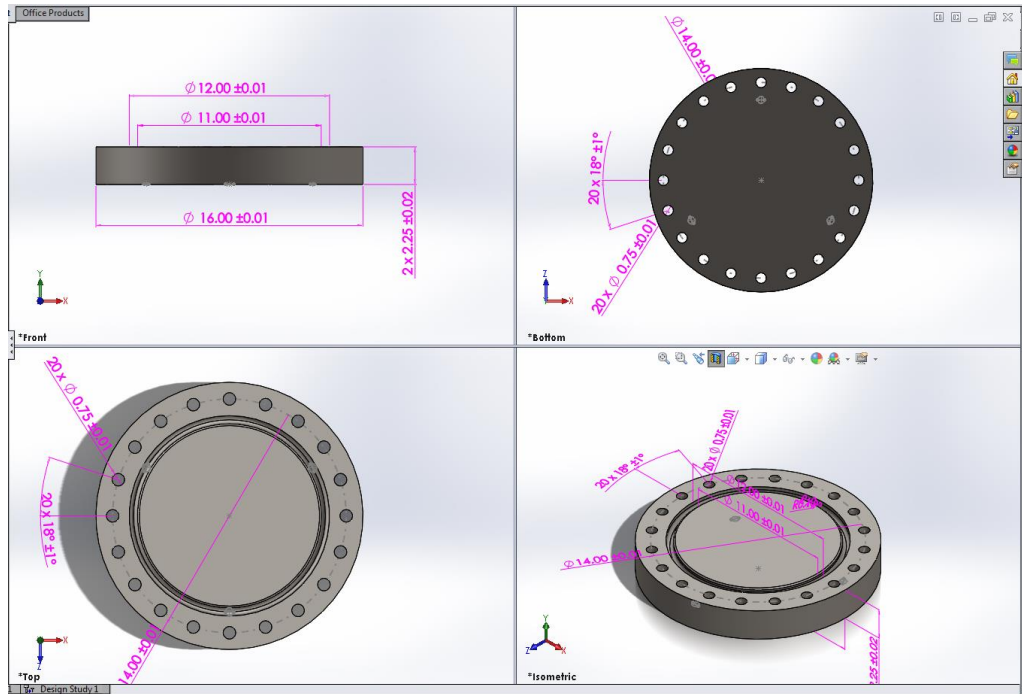


Figure 3.1. Top Mold CAD (dimensions in inches)



Figure 3.2. Pressure Injection Mold (top plate, spacer and O-rings on left)

3.3. SLURRY

The slurry production used β -SiC powder and SMP-10 to generate a paste to apply to the fiber mats. The two components were weighed and mixed manually. The slurry for the initial impregnation of the fiber mat was SMP-10 loaded with β -SiC at 44.5% by weight. The viscosity of the slurry was lowered by cutting with a solvent, methyl ethyl ketone. This was done to aid infiltration and to achieve fiber saturation. The slurry was then applied using a wiper blade to load the fiber mats.

3.4. SAMPLE LAYOUT

3.4.1. Vacuum Infused Laminate. The fiber mat was cut to the shape of the mold. The slurry was combined with the Hi-Nicalon fiber mat in the die to produce a fiber volume fraction of 40 %. After sufficient impregnation the solvent was evaporated from the fiber mat at ambient conditions. The next layer was then placed and impregnated with slurry until a 5 layer laminate was formed. The mold was then closed and placed under 1.38 MPa (200 psi) of pressure and heated to 180 °C for 6 hours with a post cure at 200 °C for 1 hour.

3.4.2. Injection Infused Laminate. Fiber mats were cut to fit within the mold (Figure 3.2). Filler and a breathable peel ply were placed into the mold cavity to lay the part upon. The mats were then loaded with slurry and layered to create a 4 ply laminate. The laminate was then topped with another peel ply and filler followed by the top of the mold. The mold was then sealed and vacuumed. After 1 hour, resin was injected into the mold using a high pressure injector (Figure 3.3) to achieve an injection pressure of 3.447 MPa (500 psi). The pressure was maintained; the mold was sealed and then placed into an oven to bake at 150 °C for 8 hours.

3.5. LOW TEMPERATURE PYROLYSIS CURE CYCLE

The composites were subjected to a low temperature pyrolysis to produce amorphous SiC. The parts were placed in a furnace under argon atmosphere to prevent oxidization of the SiC and to produce a near stoichiometric ratio of Si:C. The samples were heated to 200 °C at a rate of 10 °C per minute. Temperature was held for one hour to allow a full green body cure of the parts. The samples were then heated to 450 °C at a rate of 5 °C per minute and held for 30 minutes. Finally, the samples were heated to 800 °C at a rate of 0.1 °C per minute and held for 1 hour to achieve full pyrolysis (Figure 3.4).



Figure 3.3. High Pressure Resin Injection Machine

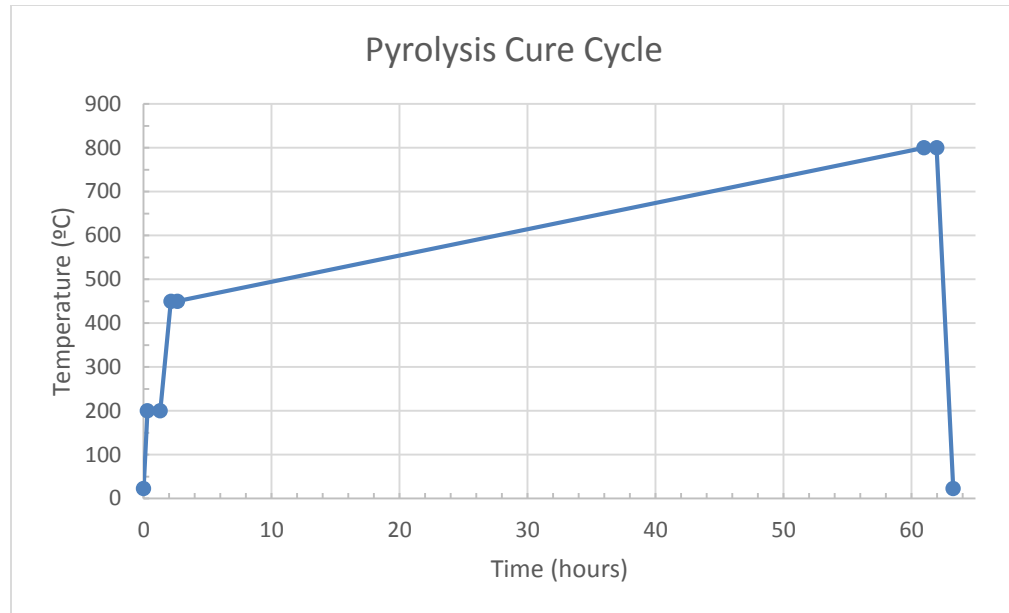


Figure 3.4. Low Temperature Pyrolysis Cure Cycle

3.6. REINJECTION

The composites are subjected to additional injections after pyrolysis. Reinjection forces resin into any existing voids within the composites and fills the voids with polymer.

During reinjection the sample was sealed within the mold and vacuum was pulled overnight. Resin was then pushed into the mold and pressurized (Figure 3.5). A series of five pressure bumps at one minute intervals was applied to improve the infiltration of the part and to ensure a constant fluid pressure in the mold. Pressure was then held for two hours.

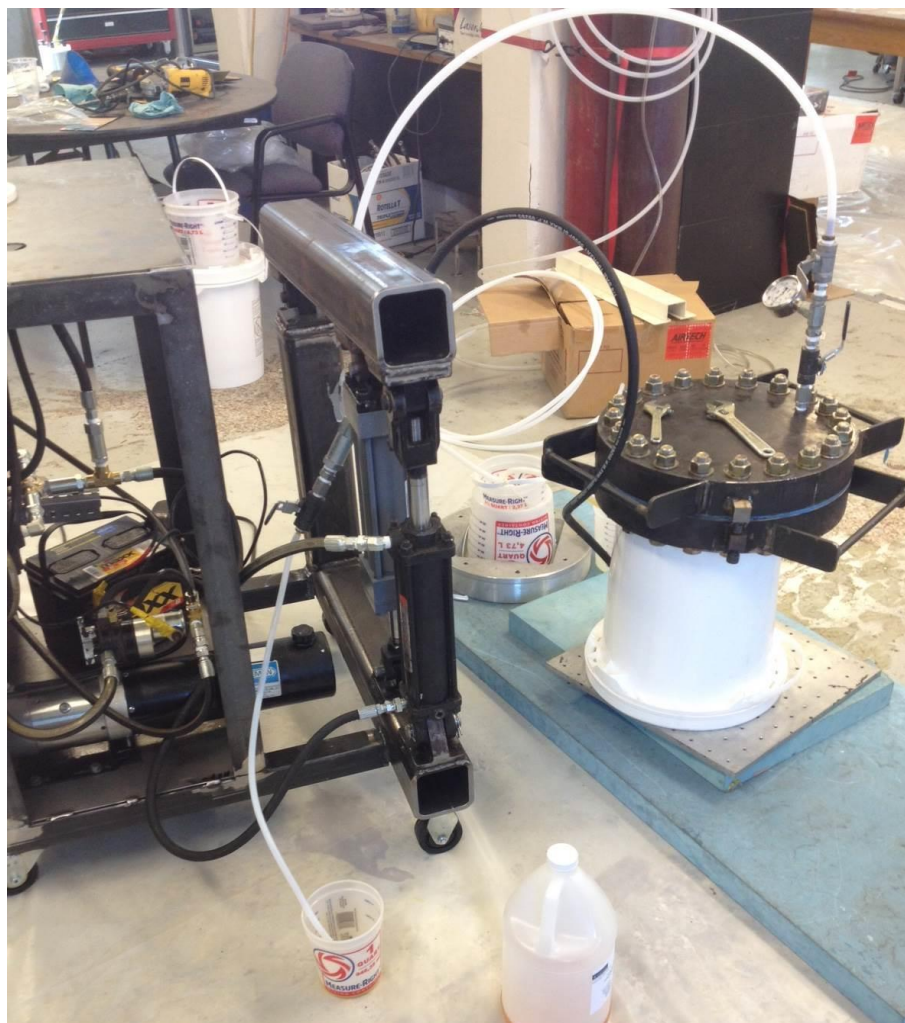


Figure 3.5. Part Reinjection Setup

After two hours, the pressure was relieved and the mold was drained of resin. The sample was then heated to 200 °C for one hour using a ramp rate of 5 °C per minute. After cooling it was subjected to a second pyrolysis cycle following the previously outlined heating schedule.

4. POROSITY MEASUREMENT

The porosity and density of the composites were evaluated using a test based on ASTM standard C20-00 (Reapproved 2010), “Standard Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water.” Specimen panels were dried by heating to 110 °C for 1 hour and then weighed to the nearest 0.001 g. The specimens were next submerged in water, heated to 100 °C. After two hours, they were allowed to cool in the water to room temperature. After 12 hours, the submerged weight of the samples was measured to the nearest 0.001 g. The samples were then removed from the water, loose water was gently removed using a saturated towel and the saturated weight was taken.

5. MICROSTRUCTURE ANALYSIS

The microstructure of ceramic composites greatly impacts the material properties. The presence of voids and different matrix densities has a negative impact. Voids in the material can form from the inclusion of air during the initial lay-up, the inability of polymer to infiltrate the composites and from trapped volatiles from the pyrolysis cycle.

To analyze the quality of the manufacturing processes, scanning electron microscopy analysis was performed using a Hitachi S-4700 Field Emission scanning electron microscope. Samples were cut using a precision saw. Samples were mounted to stages and given an Au-Pd coating using a Hummer VI Sputter Coater. The samples were then viewed and imaged using the scanning electron microscope. Images were captured at 15 kV accelerating voltage and at various magnifications.

6. RESULTS AND DISCUSSION

6.1. POROSITY

Analysis of the composites after the first pyrolysis produced some promising results for the high pressure injection samples. The results are shown in Table 6.1.

Table 6.1. Porosity Measurements

Property	Vacuum Infused	High Pressure Infused
Volume (cm ³)	0.2153	0.1020
Open Pores (cm ³)	0.0607	0.0357
Closed Pores (cm ³)	0.1547	0.0663
Apparent Porosity (%)	28.39	34.35
Water Absorption (%)	14.86	21.17
Bulk Density (g/cm ³)	1.9129	1.6424

The pressure injected parts have a larger apparent porosity. The larger porosity indicates that more of the void content in the pressure injected composite is accessible to air or resin. The higher porosity makes pressure injected parts more suitable for a reinfiltration or reinjection process to minimize the void content.

The injection parts also have a lower closed porosity than vacuum manufactured parts. Closed porosity within the parts represents voids that will be difficult or impossible

to fill with resin. The resulting closed porosities indicate that the pressure injection parts will be capable of producing parts that have a lower void content than the vacuum infused parts. The reduction in void content will increase the strength and toughness of the composites.

6.2. MICROSCOPY

From scanning electron microscopy it was possible to see some promising characteristics developed within the high pressure injection matrix after the first pyrolysis cycle and some flaws within the vacuum matrix.

The vacuum infiltration process failed to wet out the fiber tows and produced a number of dry spots which can be seen as voids in Figure 6.1. Vacuum infiltration also produced large voids (Figure 6.2) which will not be possible to fill through future reinfiltration cycles. Within Figure 6.3 the matrix of the ceramic composite shows an uneven density and a large void.

Images of the high pressure injection samples showed improved qualities. Figure 6.4 shows an even matrix developed through the fiber tows. This fiber saturation will produce a part with fewer voids and higher strength. Within Figure 6.5 it can be seen that the matrix produced by pressure injection has a consistent structure which should allow for improved reinfiltration.

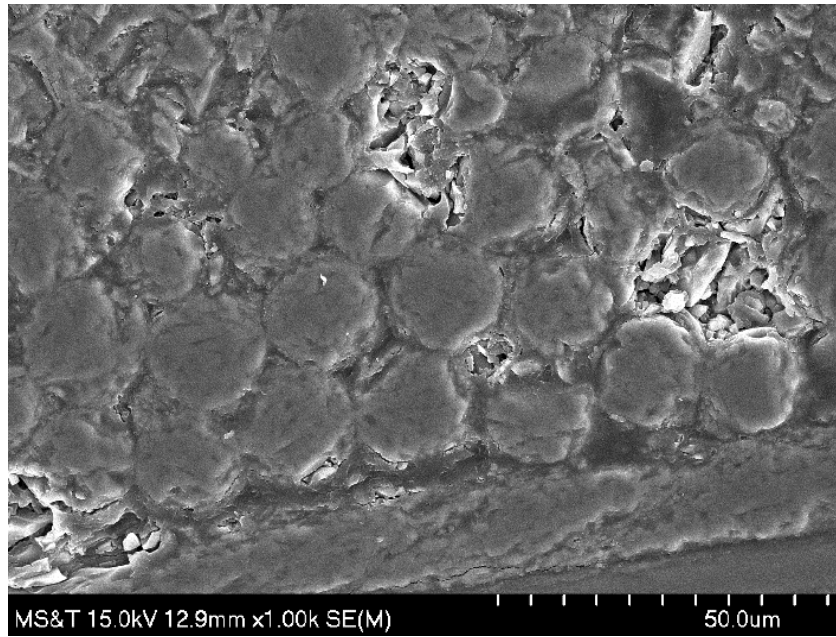


Figure 6.1. Image of Vacuum Matrix Showing Incomplete Fiber Wet Out

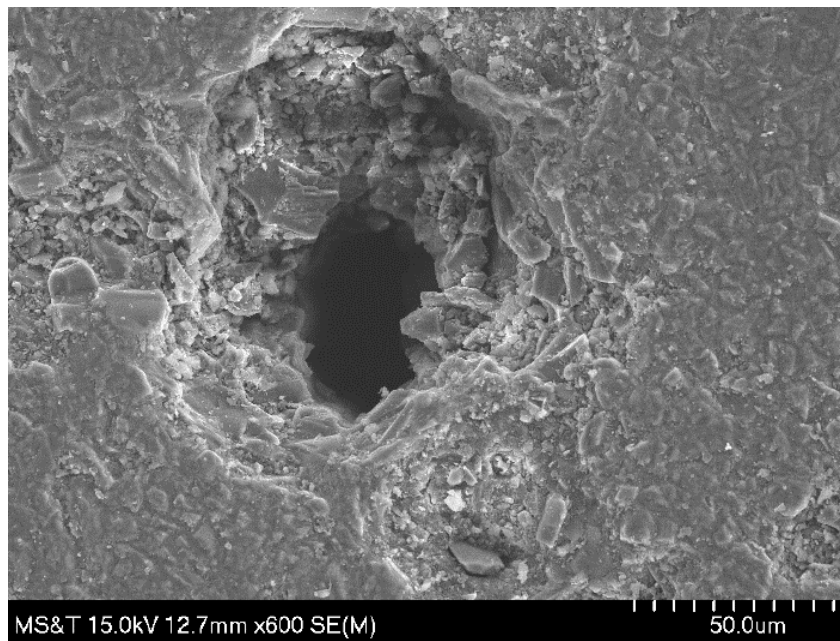


Figure 6.2. Image of Void in Vacuum Matrix

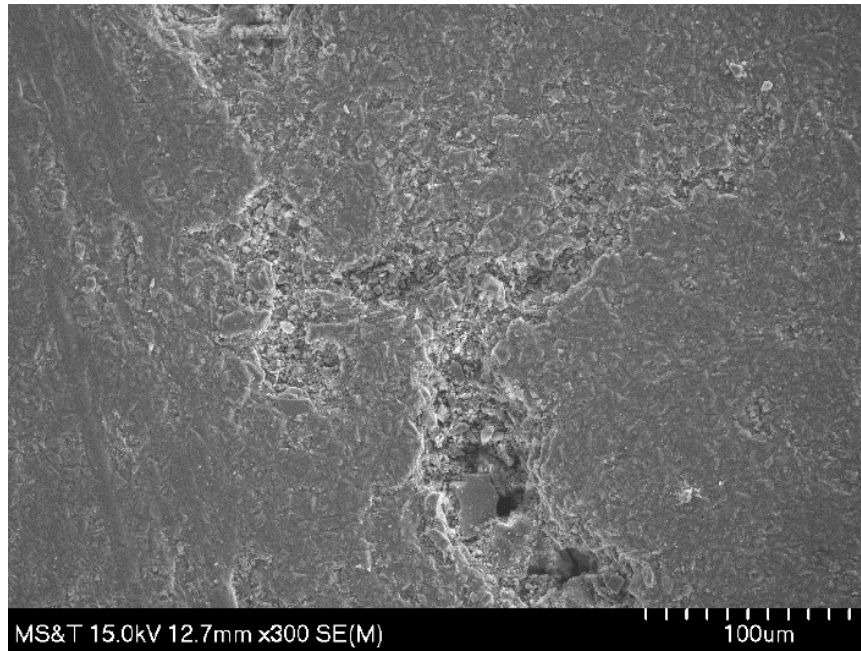


Figure 6.3. Image of Uneven Vacuum Matrix with Void

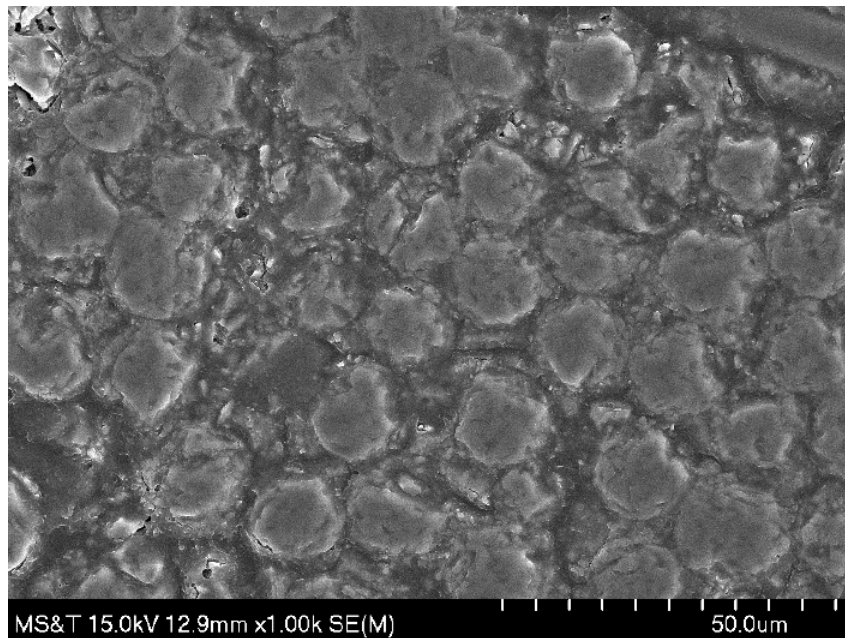


Figure 6.4. Image of Pressure Injection Matrix Showing Full Fiber Wet Out

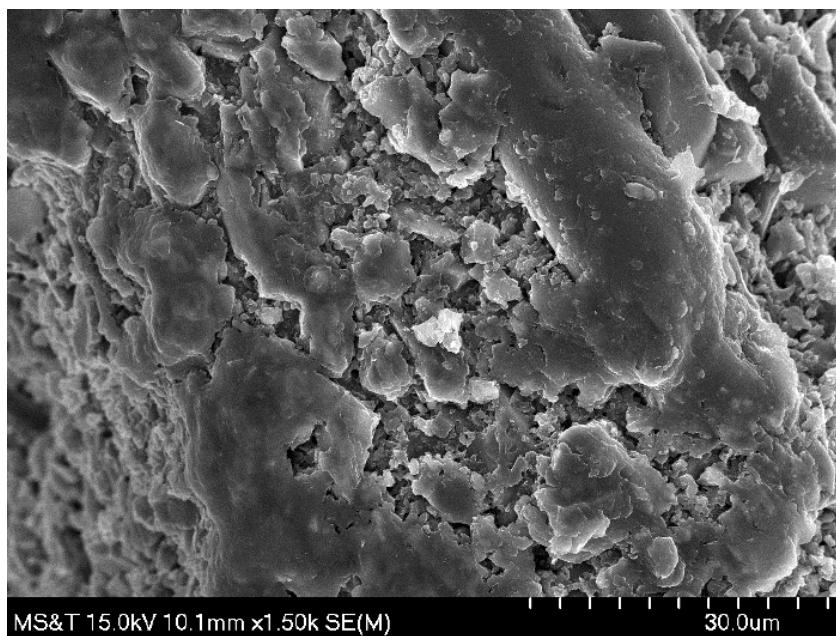


Figure 6.5. Image of Pressure Injection Matrix

7. CONCLUSIONS

This study has indicated that a high pressure injection process can produce SiC/SiC composites that contain a lower void content than traditional vacuum methods. The higher percentage of open porosity within the pressure injected samples allows for a reinfiltration process that will fill more voids than traditional vacuum forming.

Scanning electron microscopy of the samples provided information about the matrix microstructure that was formed. The vacuum matrix suffered from a high void content and lower fiber tow wet out. These defects would prove difficult to correct through traditional reinfiltration in part because the micro cracks leading to the voids would fill and close before the voids could be filled. The pressure injected matrix showed a thorough fiber wet out and lower void content. The matrix also had a consistency which would seem to be beneficial for reinjection to produce the lowest void content.

The pressure injection process developed improved part quality while drastically reducing the manufacturing time. The use of pressure injection reduces the time of reinfiltration or reinjection from weeks to hours.

8. FUTURE WORK

The results of this study have shown the use of high pressure injection can improve upon ceramic composite manufacturing processes using a preceramic polymer. Further studies must be conducted to investigate what effects manufacturing pressure may have on composite parts. Material testing of fully infiltrated parts at room and elevated temperature is needed to further evaluate pressure injected ceramics. Development and testing of additional sample geometries under high pressure injection processes for industrial adoption should be considered.

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VITA

Robert Raymond Meinders was born in Bellevue, NE to Steven and Linda Meinders on February 6, 1991. He received his Bachelors of Science degree in Aerospace and Mechanical Engineering in May of 2013 from the Missouri University of Science and Technology. He then joined the Master of Science degree program in Aerospace Engineering the following semester, and received his M.S. degree in December 2015.