

HIGH TEMPERATURE/ HIGH STRENGTH DISCRETE FIBER REINFORCED COMPOSITES

by

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ABSTRACT OF THE THESIS

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Most of the high temperature resistant composites are made using ceramic matrices. Typically these composites are processed at temperatures higher than the operating temperatures. The results presented in this thesis focus on the development of an inorganic matrix composite that can be processed at temperatures ranging from 80 to 400°C and can withstand temperatures up to 1500°C. The composites can be fabricated using inexpensive mold-cast techniques or vacuum bagging techniques. Short discrete fibers can be incorporated in the matrix to improve mechanical properties.

The composite is a two component system consisting of: potassium/ sodium silicate solution and a powder component containing; silica, alumina, fillers, fibers, flow enhancing additives and activators. The major parameters evaluated in this dissertation are: (i) influence of fiber type and fiber content, (ii) matrix composition in terms of silica/alumina ratio, (iii) fabrication techniques, (iv) influence of curing temperature and (v) influence of exposure to temperatures varying from 200 to 1500°C. The response variables were: the integrity of the samples after high temperature exposure and the mechanical property of the composite. The fiber types consisted of: economical bulk alumina fibers, alumina fibers in paper form and uniform-short alumina fibers. The fiber

content varied from 4 to 13 percent by weight of total matrix. Silica to alumina ratios were varied from 1 to 5. Fabrication techniques investigated include: compression molding using wetted alumina fiber papers and simple casting using a mold and vacuum bagging technique.

The major findings are as follows:

- Both mold-casting and vacuum bagging techniques can be effectively used for fabrication
- Optimum curing temperature is 400°C
- For composites with bulk-economical alumina fibers the maximum flexural strength is 65 Mpa and the maximum flexural modulus is 52 GPa
- These values can be increased to 130 MPa and 85 GPa by using high quality fibers
- The densities for composites with short fibers range from 2000 to 2800 kg/m³
- Typically higher density leads to higher strengths

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CHAPTER 1. INTRODUCTION

1.1 Scope and Objective of this Study

Advancements in automotive, aircraft and space shuttle industries have influenced the development of specialized materials. Metal alloys were one of the first materials affected by this industrial movement. Typically, metals exhibit high tensile strengths and ductility. However, the mechanical properties of metals are dramatically reduced in high temperature environments such as engines, turbines, thermo insulators and the like. Thus, interest grew in ceramic materials due to their high thermal resistance. Ceramics perform well in aggressive temperature environments, but they are very brittle materials. This brittle behavior causes catastrophic failure which makes ceramics unattractive to structural applications. As a result, a combination of several materials led to the development of composites to achieve greater performances.

The development of organic resins, such as epoxy and polyester advanced composites in many applications. Fibers, typically carbon and glass were combined with the resins to create the composite material. These composites proved to experience high mechanical properties and low densities, making them attractive in aerospace industries. However, these materials were again challenged to perform in high temperature environments beyond 1000°C. As a result, composites were advanced to metallic matrix, carbon/ carbon, and ceramic matrix composites. These composite matrices all exhibit high mechanical properties, but do not all perform at extreme temperatures. Another disadvantage of these materials is their high processing costs. Most require curing

temperatures above 1300°C and special equipment and processing techniques to protect the fiber reinforcement.

The primary focus of this research study is to develop a high temperature inorganic matrix composite (IMC) that can cure below 400°C. This inorganic matrix was combined with common ceramic matrix materials to perform in high temperature environments. The variables investigated include the following: (i) silica to alumina ratio, (ii) fiber type, (iii) fiber volume fraction, and (iv) fabrication techniques. Multiple matrix compositions have been reinforced with various fibers to create an inorganic matrix composite material that will cure at or near room temperature. In addition to the matrix formulation and fiber reinforcement studied, several fabrication techniques have been evaluated.

CHAPTER 2. LITERATURE REVIEW

2.1 Introduction

In past years much research and development has focused on the advancement of material science, especially in fields pertaining to engines, turbines, thermo insulators and the like. For most applications, lightweight high strength composites are made of carbon or glass fibers and organic matrices. In spite of the excellent mechanical properties, these composites cannot be used in high temperature applications. In certain cases such as aerospace and naval structure applications, exposure to high temperatures during accidents not only reduces the mechanical properties but also results in toxic fumes and smokes.

In applications that require more than 200°C exposure temperature, most organic matrix composites cannot be used. For these special conditions superior materials have been developed to be strong, light weight structural components that can withstand temperatures in excess of 1000°C. Currently, high temperature materials fall into three main categories: metallics, ceramics, and composites. The classifications of these materials are summarized in Table 2.1.

The success of composite materials is due to the combination of fiber reinforcement into various matrices such as metallics and ceramics. In most composite production high temperature curing, often exceeding 1000°C is required. Special high temperature resistant equipment is needed for fabricating these composites, thus contributing to the fabrication cost. In the case of ceramic matrices, the cost of fibers is

the main contributing factor. Since most ceramic matrices also require curing temperatures in excess of 1000°C, commonly used high strength fibers such as carbon or glass are not typically used. Specialized, very high temperature resistant fibers can cost as much as \$66,000 per kilogram (Hammell, 2000).

If the cost of the high temperature composite is reduced, then their demand could increase many fold, especially in automobile structures. The development of inorganic matrices provides an excellent opportunity to produce economical high temperature resistant composites. Select properties of the inorganic matrix composites (IMC) created for this study are compared against typical high temperature composites in Table 2.2. This table presents the density, bending strength, modulus of elasticity, curing and operating temperatures for these composites. The following will discuss the three contributing materials in high temperature environments, as well as, inorganic matrices and commercially available fibers.

2.2 High Temperature materials

2.2.1 Metallics

The advancement of metallics has led to the development of materials known as superalloys. These superalloys are based on a combination of nickel, cobalt and iron alloys. Cobalt-based alloys were the first metallics developed that showed high mechanical performance. Nickel-based alloys soon replaced cobalt due to their temperature capabilities. This has caused the use of nickel-based alloys to become major contributors to the manufacturing of jet engines. However, metallic alloys have failed to maintain their mechanical properties beyond 600°C.

Recent metallic research in Japan has led to further development of superalloys which can perform at temperatures close to 800°C. The effects of temperature on the mechanical properties of these materials are shown in Figure 2.1. Typical mechanical properties of commercially available nickel-based superalloys are tabulated in Table 2.3. These metallics exhibit creep rupture strengths that range from 300 to 700 MPa or more. High elongation percentages, typically around 20%, have also been experienced. However, it is very difficult to stabilize these alloys at temperatures beyond 700°C. Most of the available superalloys experience an extreme loss of strength between 40 and 60 percent after 700°C (Harada, 2006).

2.2.2 Ceramics

Ceramic materials are inorganic materials characterized by ionic, or in some cases partially ionic, bonds between metallic and non-metallic elements (Callister, 1994). The production and use of ceramic materials is not a new technology. For thousands of years, ceramic materials have been used for a variety of applications. Classical examples of ceramics are pottery, and bricks. Clay, which is composed of silica, alumina, and bonded water, is the primary material in these ceramics. Kaolinite Clay $[\text{Al}_2(\text{Si}_2\text{O}_5)(\text{OH})_4]$ is the most commonly used material. The clay needs to be dried to remove water and then fired to an extremely high temperature in order for bonding to occur. Portland cement can also be considered as a ceramic material. Clearly the most significant advantage of cement is that bonding occurs at room temperature, aided by the alkalinity of the slurry. Another classical example of a ceramic material is window glass, which is made primarily out of silica. Modern ceramic materials such as those previously mentioned all have an

important advantage over organic materials, which is their resistance to extreme heat, primarily beyond 1000°C (Hammell, 2000).

Non-oxide ceramic materials which are primarily based on silicon carbide and silicon nitride compositions have been developed for use in applications where there is a need for high temperature resistance and moderate strength (Tanaka, 2000). Ceramic materials have been used to manufacture a variety of products including insulators, coil forms, turbine nozzles, and even high strength alumina bolts and nuts. The brittle fracture behavior and low damage tolerance of ceramics has made them unattractive for many applications. One solution to this problem has been the addition of long fibers into the ceramic matrix which has lead to the development of ceramic composite materials.

2.2.2.1 Crystalline Ceramics

Ceramic material can be divided into two basic categories based upon the crystal structure of the material. The first category contains crystalline ceramics. These materials exhibit a well-developed crystal structure similar to that of metals. However, there are no metallic bonds in the structure. Rather the bonding is either purely or partially ionic. Rock Salt (NaCl) is an example of a ceramic in this category. Rock salt has a Face Centered Cubic (FCC) crystal structure which is the same structure that is found in copper, aluminum and gold (Hammell, 2000).

Advanced crystalline ceramic materials have a variety of uses from heat shielding material to piezoelectric sensors, computer chips and circuitry. These ceramic materials are called refractory because, as mentioned previously, they can withstand high temperatures, and they remain chemically inert in severe environments.

2.2.2.2 Glasses (Amorphous Ceramics)

The second category is comprised of non-crystalline ceramics, known as glasses. Glasses are ceramic materials with amorphous bonding patterns. This amorphous structure is what gives glasses their optical properties. As stated earlier, a good example of a material in this category is ordinary window glass, which is composed primarily of silica (SiO_2) with other metal oxides such as soda (CaO), lime (Na_2O), and alumina (Al_2O_3) to keep the system in a non-crystalline state and lower its glass transition temperature (Shriver et. al., 1990). The exact proportions of the metal oxides can be varied depending on the exact mechanical and thermal properties that are desired.

2.2.2.3 Glass-Ceramics

A process called devitrification is used to convert a glass into a crystalline ceramic (Hammell, 2000). During devitrification, glasses are heated to a temperature where nucleation and crystal growth are allowed to begin. This is done to remove residual stresses in the material. Normally, when a glass is devitrified, it forms a coarse polycrystalline structure, loses its optical properties and fundamentally ceases to be a glass since there is no amorphous region. However, if the material is doped with a nucleating agent (TiO_2 , Ta_2O_5 , or Nb_2O_5) and the devitrification process is closely controlled, an extremely fine polycrystalline structure can be achieved which allows the glass to maintain its “glassy” properties while relieving the residual stresses that existed in the amorphous phase. These materials are then considered glass-ceramics. The typical degree of crystallinity in glass-ceramics is between 50-90% (Lehman, 1995).

Glass-ceramics are often used as matrix material in CMCs because of the relative ease of fabrication. When conventional glasses are used in CMCs, softening of the amorphous glass limits the use temperature of the resulting composite. With glass-ceramic matrices, fabric reinforcement can be impregnated by conventional methods used with glasses, and thermal stability can be achieved through devitrification.

2.2.3 Composites

Typical high temperature composites can be divided into three classifications: metal matrix, carbon/carbon and ceramic matrix composites. Metal matrix composites (MMC), have shown increased strength and temperature resistance than metallic alloys alone. Carbon/carbon composites are known to experience high strengths at temperatures beyond 1500°C, however, this is extremely difficult to achieve in oxidizing environments. Ceramic matrix composites (CMC), or continuous fiber ceramic composite (CFCC) take advantage of the high temperature resistance provided by the ceramic matrix and added fibers to increase the toughness. The ceramic matrix also provides excellent protection to the fibers from oxidation. However, all three of these composite matrices require extreme curing temperatures, beyond 1000°C. Recent advancements have led to the development of inorganic matrix composites (IMC), which require much lower curing temperatures (400°C).

2.2.3.1 Metal Matrix Composites

Metal matrix composites (MMCs) have attracted much attention in recent years due to their increased thermal resistance, modulus, strength, and fatigue resistance

compared to the unreinforced metallic alloys. The concept of MMCs is based on the combination of two different materials. Ductility and toughness are provided by the metallics while the modulus and strength are provided by ceramic reinforcements. MMCs depend on the properties of matrix material, reinforcements, and the matrix-reinforcement interface (Pandey, 2003). Some physical and mechanical properties of select MMCs are presented in Table 2.4.

A variety of matrix materials have been used for making MMCs, while a major emphasis has been on the development of lighter MMCs using aluminum and titanium alloys. Most common aluminum alloys have maximum usage temperatures below 150°C. High temperature aluminum alloys are currently being researched for aerospace applications in hopes of achieving mechanical performance beyond 300°C. Titanium alloys are very attractive for MMC applications, due to their higher strength and temperature capability compared to aluminum alloys (Pandey, 2003). Despite the increased properties in titanium alloys, processing these composites are highly cumbersome. Special care and attention is required during processing to reduce damage and ensure quality control. These procedures can increase production cost substantially.

2.2.3.2 Carbon/ Carbon Composites

Carbon can be used as an effective matrix material for high temperature ceramic composites. In 1995, Turner, Speck and Evans investigated the mechanisms of plastic deformation and composite failure. The carbon matrix that was used for this study was created by Chemical Vapor Infiltration CVI processing, and had a modulus of only about

20 GPa (Hammell, 2000). However, the fiber/matrix interface properties still allowed the composite to be strong and stiff by effectively transferring load to the fibers.

Available data on the mechanical properties of 2-D woven C/C composites indicate a low tensile strength of 173 MPa, and an elastic modulus of 94 GPa (Kostopoulos and Pappas, 1998). Even with the low strength that is presented here, C/C composites are still being considered for a variety of applications because of their other properties such as low density (1.49 g/cm³), and resistance to aggressive thermal environments. Perhaps most important is the ability of the composite to withstand damage by using built in stress redistribution mechanisms such as matrix cracking and significant fiber/matrix interface debonding.

Additional mechanical tests were conducted by Kogo et. al. (1998) on Carbon/Carbon composites fabricated by a CVI process. They found that the interlaminar shear strength by Iosipescu test was 35-40 MPa, and the failure strain of this composite in tension was 0.35%. The in-plane shear strength of the composite as tested in $\pm 45^\circ$ tension was 31 MPa, and the fracture toughness K_{Ic} was 7.5 MPa m^{1/2}. Some mechanical properties for select carbon/ carbon composites are presented in Table 2.5.

2.2.3.3 Ceramic Matrix Composites

In ceramic matrix composites, fiber reinforcement is combined with a ceramic matrix to increase the toughness of an otherwise brittle material. The use of ceramic matrix composites is currently limited due to their high production costs, but is expected to decrease as demand increases. Most ceramic matrices require temperatures in excess of 1000°C to cure which requires special high temperature equipment to produce the

composite. The primary uses of these composites are in applications where materials are expected to encounter high temperatures; such as engine components, exhaust systems and fire barriers. The low density as compared to metals makes them attractive in applications where weight is a critical design parameter.

Ceramic matrix composites can have several different matrix compositions in respect to the base material. Table 2.6 shows several properties of common ceramic matrix materials. Alumina, Silicon carbide (SiC), Mullite, and Alumina/ Zircon are some of the more commonly used materials in ceramic matrix compositions. Table 2.7 presents the strength and toughness of some CFCCs compared with conventional monolithic ceramics.

In 1996, Kamino, Hirata and Kamata reinforced an alumina matrix with long alumina fibers by high temperature sintering, 1000-1400C. They found an increase in strength with an increase in sintering temperature. Figure 2.2 shows the flexural stress versus sintering temperature of their findings. Although these composites have flexural strengths beyond 100 MPa, the high temperature curing process fails to reduce production costs. Composites with silicon carbide fibers in a silicon carbide matrix can be manufactured using the CVI process. These samples used Nicalon silicon carbide fabric, which is the most commonly used silicon carbide fiber. Tensile testing on [0/90°] samples yielded a tensile strength of 200 MPa, a modulus of 230 GPa, and a failure strain of 0.3%. Flexural testing yielded a strength of 300 MPa and interlaminar shear testing strength of 40 MPa (Hammell, 2000).

The most important advantage of using SiC/SiC composites is their performance at elevated temperature. Tensile testing of these composites was conducted in nitrogen to

prevent oxidation. It was found that the sample still had a tensile strength of 230 MPa after 1 hour stabilized at 1300°C. Also, the failure strain of the sample was approximately 1.5% (Hammell, 2000). It is extremely difficult to achieve such strength and toughness at these temperatures. Hence, SiC/SiC composites have become popular in the aerospace industry as engine heat shields.

The aforementioned ceramic composites were found to have greater mechanical stability at extreme heat exposures compared to unreinforced materials. This is an immense achievement in the materials industry; however, production cost still pose problems. The high temperature sintering requirements and specialized procedures make CMCs unattractive on an economical scale.

2.3 Inorganic Matrix

In the civil engineering industry Portland cement is the most commonly used inorganic matrix. The main disadvantage of the Portland cement system is the size of the grains which are relatively large. This prevents the formulation of thin matrices.

In addition to Portland cement, other common room-temperature matrices are alluminosilicates and phosphate based compounds. Alluminosilicates are not 100% impermeable thus allowing the concrete surface to release the vapor pressure. They are also UV light resistant, resistant to high temperature and hard. Inorganic matrices do not need precautions because they are not toxic and they do not form toxic fumes. One of the inorganic resins which are currently available is a potassium alluminosilicate, or poly (sialate-siloxo), with the general chemical structure.



Where $z \gg n$. This resin hardens to an amorphous or glassy material, and is one of a family of inorganic Geopolymer materials (Davidovits, 1991). It has a pot life of more than three hours and is compatible with organic, mineral, and steel fibers. This low-cost, inorganic polymer is derived from naturally occurring geological materials, namely silica and alumina, hence the name Geopolymer (also known as polysialate). Geopolymer is a two-part system consisting of a potassium silicate liquid and a silica powder and cures at a reasonably low temperature of 150°C or hardeners can be added to achieve room temperature curing (22°C). Geopolymer can sustain temperatures in excess of 1000°C which provides potential for use where high temperatures are anticipated such as engine exhaust systems or where fire safety criteria are of concern.

Geopolymer have been evaluated for use with carbon, glass, nylon, steel fibers, and fabrics. The results are quite encouraging (Balaguru et al., 1996, Lyon et al., 1996, Foden et al., 1996, Foden et al., 1997). Table 2.8 presents the modulus of elasticity for matrices investigated by C. Papakonstantinou.

2.4 Fibers

The principal function of the reinforcement in the matrix is to resist most of the applied load acting on the composite system. The primary characteristics that are desirable for reinforcement of a ceramic are: high modulus, high strength, high thermomechanical stability, resistance to oxidation, small fiber diameter, low density, and low cost (Lehman, 1995). Clearly, each fiber will have some of these characteristics while lacking in others.

Since CMCs are most commonly used in thermally aggressive environments, emphasis is put on thermomechanical stability and resistance to oxidation. Thus, the use of common reinforcing materials such as carbon and aramid is not feasible because most high strength fibers cannot withstand temperatures above 600°C in oxidizing environments. This makes silicon carbide and metal oxides such as alumina establish themselves as viable reinforcement choices. Although several fiber types and formats such as aramid, silicon carbide, basalt and others can be used to reinforce CMCs, only select ceramic and carbon fibers were investigated in this study.

2.4.1 Carbon Fibers

Carbon fibers offer the highest modulus of all reinforcing fibers and are most commonly used with organic resin such as epoxy. Among the advantages of carbon fibers are their exceptionally high tensile strength-to-weight ratios as well as high tensile modulus-to-weight ratios. In addition, carbon fibers have high fatigue strengths and a very low coefficient of linear thermal expansion and, in some cases, even negative thermal expansion. This feature provides dimensional stability, which allows the composite to achieve near zero expansion to temperatures as high as 300°C in critical structures such as spacecraft antennae (Giancaspro, 2004). Carbon fibers are chemically inert and not susceptible to corrosion or oxidation at temperatures below 600°C. If they can be protected from oxidation above 600°C, then carbon fibers are stable up to 2000°C or more. However, it is extremely difficult to achieve a non-oxidizing environment which requires special processing techniques.

2.4.2 Ceramic Fibers

Ceramic fibers are composed of a significant alumina-silica percentage and only small percentages of other metal oxides. These fibers can withstand temperatures well in excess of 1200°C. This makes them the most thermally stable fibers currently available. These fibers have similar compositions to the common glass matrices used in inorganic matrix composites. Some common ceramic fibers used in composites are silicon carbide, silicon nitride, alumina and alumina/zirconia. A summary of information on the composition, fabrication method, manufacturer, density, fiber diameter, number of fibers in each tow, elastic modulus, strength, coefficient of thermal expansion and suggested maximum use temperature of these fibers and others is presented in Table 2.9.

The following observations can be made based on a review of Table 2.9. Ceramic fibers provide a much higher temperature range than oxidizing fibers such as carbon. Most of these fibers can sustain 1000°C as compared to about 400°C for carbon fibers. The tensile strength of ceramic fibers is usually lower than the strength of carbon fibers, but new advancements in ceramic fibers have proven otherwise. Carbon fibers are available with three moduli of elasticity of 300, 600 and 900 GPa. For ceramic fibers, the range is 190 to 470 GPa. Failure strains for carbon and ceramic fibers are about the same. Depending on the modulus of elasticity, failure strain of carbon varies from 0.004 to 0.015 as compared to 0.006 to 0.018 for ceramic fibers (Papakonstantinou, 2003).

2.5 Summary

Materials continue to be developed to meet more stringent design requirements especially for high temperature environments. Metallic superalloys and ceramic

materials are still under development to perform in temperature aggressive applications. Metal matrix and carbon/ carbon composites typically provide substantial mechanical properties between 500 and 1500°C. Ceramic matrix composites reinforced with silicon carbide fibers are becoming leading materials in composites. These materials provide increased toughness to ceramics as well as thermal resistant capabilities.

However, all of these materials require extreme temperatures to manufacture and/or precision processing techniques. These processes substantially affect the cost of the final composite part. The recent addition of Geopolymer into composites such as CMCs has given ceramic materials the ability to cure at temperatures of 150°C or below, thus greatly reducing processing temperatures. These composites have proven to perform well under extreme temperatures in excess of 1000°C and not experience such high losses in strength as seen in metallics.

Table 2. 1: A classification of the ultra-high temperature materials (Tanaka, 2000)

| | |
|------------|---|
| Metallics | <ul style="list-style-type: none"> * Intermetallic compounds (IMCs): TiAl, NiAl, MoSi₂, Nb₃Al, etc. * Refractory metals and alloys: W, Ta, Mo, Nb, etc. |
| Ceramics | <ul style="list-style-type: none"> * Oxides: Al₂O₃, SIALON, Al₂O₃/YAG directionally solidified eutectic (MGC Melt growth composite), etc. * Non-oxides: SiC, Si₃N₄, etc. |
| Composites | <ul style="list-style-type: none"> * Metal matrix composites (MMC): SiC/Ti, SiC/TiAl, W/Superalloy, etc. * Ceramic matrix composites (CMC): SiC/SiC, SiC/LAS, C/SiC, etc. * Carbon/carbon composites (C/C): C/C * Functionally graded materials (FGM): TiB/Ni, Al₂O₃+ZrO₂/Ni, etc. |

Table 2. 2: Select properties of typical High Temperature Composites (Pandey, 2003; Papakonstantinou, 2003)

| Composite | Density (kg/m ³) | Bending Strength (MPa) | E (GPa) | Curing Temperature (°C) | Operating Temperature (°C) |
|-----------|------------------------------|------------------------|---------|-------------------------|----------------------------|
| MMC | 2000-5500 | 600-1100 | 120-300 | 700-1300 | 20-650 |
| C/C | 1500-2000 | 335-527 | 76-100 | 1100-2700 | 300-600 |
| CMC | 3720-4000 | 345-635 | 100-230 | 1200-1700 | 1000-1300 |
| IMC | 1000-2800 | 50-130 | 60-90 | 80-400 | 1000-1500 |

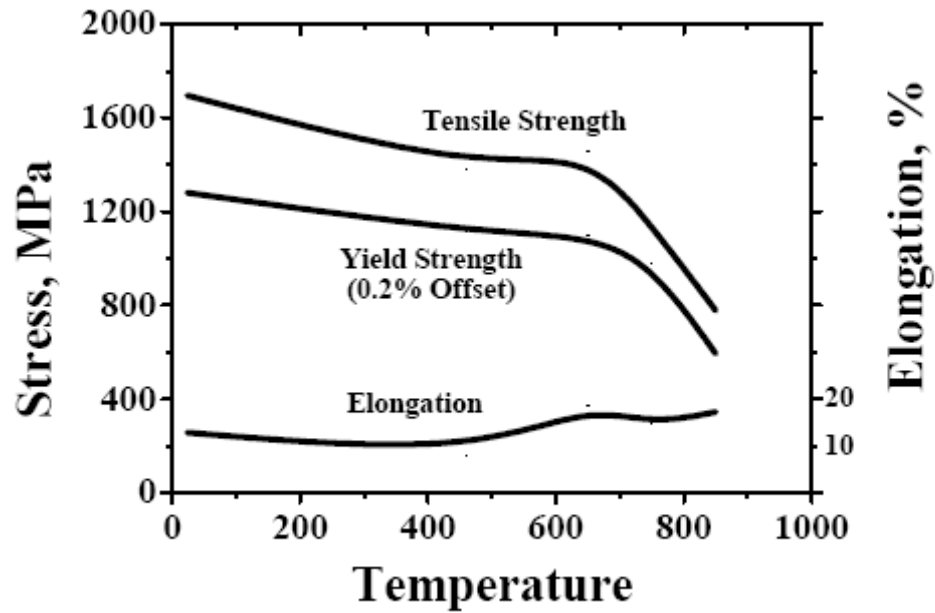


Figure 2. 1: Tensile Strength and Elongation of Nickel-Cobalt Base Superalloy (Harada, 2006)

Table 2. 3: Typical Mechanical Properties of commercially available Nickel base single Crystal Superalloy (Harada, 2006)

Creep Rupture Strength (TMS-75)

| Conditions | | Rupture life τ h | Elongation % | Reduction of Area % |
|--------------------|-------------|--------------------------|-----------------|------------------------|
| Temperature, °C(K) | Stress, MPa | | | |
| 900 (1173) | 392 | 961 | 23.0 | 30.3 |
| 1000 (1273) | 245 | 329 | 27.9 | 34.0 |
| 1100 (1373) | 137.2 | 227 | 14.0 | 26.2 |
| 1150 (1423) | 98 | 196 | 11.9 | 39.1 |

Creep Rupture Strength (TMD-103)

| Conditions | | Rupture life τ h | Elongation % | Reduction of Area % |
|--------------------|-------------|--------------------------|-----------------|------------------------|
| Temperature, °C(K) | Stress, MPa | | | |
| 900 (1173) | 392 | 519 | 10.8 | 17.9 |
| 1000 (1273) | 196 | 444 | 18.8 | 18.7 |
| 1040 (1313) | 137 | 884 | 18.6 | 32.3 |

**Table 2. 4: Some properties of carbon reinforced metal-matrix composites
(Kostikov, 1998)**

| <i>Property</i> | <i>Matrix</i> | | | |
|--|---------------|--------------|-----------|-----------|
| | <i>AL-9</i> | <i>AMG-6</i> | <i>Ni</i> | <i>Cu</i> |
| Filler | Kulon | Kulon | VMN-4 | VMN-4 |
| Tensile strength M_{min} , GPa | 2.5 | 2.5 | 2.0 | 1.5 |
| Elastic modulus, min, GPa | 40.0 | 40.0 | 25.0 | 23.0 |
| Coating | TiC | SiC | Ti(Zr)C | – |
| <i>Metal-matrix composite</i> | | | | |
| Density, ρ , kg m ⁻³ | 2000–2300 | 2200–2300 | 5200–5400 | 5200–5500 |
| Tensile strength, MPa | | | | |
| at 20°C | 900–1000 | 700–900 | 450–500 | 400–500 |
| at 400°C | 800–900 | – | – | – |
| at 800°C | – | – | 400–500 | – |
| Bending strength, MPa | 900–1050 | 800–1100 | 700–900 | 600–700 |
| Compressive strength, MPa | 800–1000 | 500–700 | – | – |
| Elastic modulus, E , GPa | 270–300 | 240–270 | 140–180 | 120–170 |
| Specific modulus, E/ρ , m ² s ⁻² × 10 ⁶ | 135 | 109 | 26 | 23 |

Table 2. 5: Mechanical properties of carbon/carbon composites (Papakonstantinou, 2003)

| Fiber/matrix | Tensile strength (MPa) | | Elastic modulus (GPa) | | Tensile failure strain (%) | | Flexural strength (MPa) | | In-plane shear strength (MPa) |
|--|------------------------|------------------|-----------------------|------------------|----------------------------|-------------------|-------------------------|---------|-------------------------------|
| | (0°) | (0/90°) | (0°) | (0/90°) | (0°) | (0/90°) | (0°) | (0/90°) | (±45°) |
| SiC/SiC-1 | NA | 255 | NA | 230 | 0.3 | 0.47 | NA | 300 | NA |
| SiC/CAS-1 | 924 | 220 | 120 | 110 | 0.82 | 0.83 | 950 | 500 | 65 |
| SiC/CAS-2 | 680 | 250 ^a | 125 | 105 ^a | 0.7 | 0.74 ^a | 680 | NA | NA |
| SiC/LAS | 455 | 285 | 139 | NA | 0.93 | NA | 600 | 380 | 24 |
| SiC/Al ₂ O ₃ | NA | 265 | NA | 150 | 0.65 | 0.83 | NA | 461 | NA |
| SiC-SiC-2 | NA | 260 | NA | 100 | NA | 0.45 | NA | 378 | NA |
| SiC/BMAS | NA | 237 | NA | 94 ^b | NA | 0.83 | NA | NA | NA |
| SiC/SiAlON | NA | NA | NA | NA | NA | NA | 605 | NA | NA |
| SiC/Zircon | NA | NA | 244 | NA | NA | NA | 700 | NA | NA |
| SiC/BN/Zircon | NA | NA | 235 | NA | NA | NA | 681 | NA | NA |
| SiC/BN/ Si ₃ N ₄ | NA | NA | NA | NA | NA | NA | 550 | NA | NA |
| C/Si ₃ N ₄ | NA | NA | NA | NA | NA | NA | 740 | NA | NA |
| C/BN/Si ₃ N ₄ | NA | NA | NA | NA | NA | NA | 410 | NA | NA |
| SNF/SNC | NA | 410 | NA | NA | NA | NA | >1000 | 620 | NA |
| SNF/SNC(A1) | NA | 330 | NA | 141 | NA | 0.44 | NA | 391 | NA |
| SNF/SNC (A2) | NA | 340 | NA | 143 | NA | 0.45 | NA | 488 | NA |
| SNF/SNC (A3) | NA | 419 | NA | 142 | NA | 0.56 | NA | 743 | NA |
| Alumina/glass | NA | NA | NA | NA | NA | NA | NA | 230 | NA |
| Alumina/tin/glass | NA | NA | NA | NA | NA | NA | NA | 190 | NA |
| SiC/PyC/SiC | NA | 250 | NA | 100 | NA | 0.55 | NA | NA | NA |
| SiC/PyC/SiC | 939 | 360 | 160 | 140 | 1.3 | 1 | NA | NA | NA |
| Saphikon/monazite/Al ₂ O ₃ | NA | NA | NA | NA | NA | NA | 230 | NA | NA |
| Saphikon/zirconia/Al ₂ O ₃ | NA | 110 | NA | 193 | NA | 0.1 | NA | NA | NA |
| Carbon/carbon (QI) | NA | NA | NA | NA | NA | NA | 335-514 | NA | 9.1-20.2 |
| Carbon/carbon (DPY) | NA | 225 | NA | 100 | NA | 0.25 | NA | NA | 30 |
| Carbon/carbon (HIP) | NA | 375 | NA | 140 | NA | 0.38 | NA | NA | 15-30 |
| Carbon/carbon (HTT) | NA | NA | NA | 120 | NA | NA | NA | 190-240 | NA |
| C/polysilate | 623 | 332 | 156 | 76 | 0.47 | 0.67 | 527 | 344 | 30 |

^a Bi-directional SiC/CAS-2 has 66.6% of fibers in 0° direction and 33.3% of fibers in 90° direction.^b Value taken from graph and not from table (136 GPa).

Table 2. 6: Properties of typical ceramic matrix materials (Amateau, 1998)

| Materials | Young's modulus (GPa) | Poisson's ratio | Modulus of rupture (MPa) | Fracture toughness (MPa m ^{1/2}) | Density (g cm ⁻³) | Thermal expansion (10 ⁻⁶ /°C) | Melting point (°C) |
|-----------------------------------|-----------------------|-----------------|--------------------------|--|-------------------------------|--|--------------------|
| LAS | 117 | 0.24 | 138 | 2.42 | 2.61 | 5.76 | – |
| Pyrex | 48 | 0.20 | 55 | 0.08 | 2.23 | 3.24 | 1252 |
| Al ₂ O ₃ | 345 | 0.26 | 483 | 3.52 | 3.97 | 8.64 | 2050 |
| Mullite | 145 | 0.25 | 186 | 2.20 | 3.30 | 5.76 | 1850 |
| ZrO ₂ PS | 207 | 0.23 | 648 | 8.46 | 5.75 | 7.92 | 2760 |
| ZrO ₂ FS | 207 | 0.23 | 248 | 2.75 | 5.56 | 13.5 | – |
| TiO ₂ | 283 | 0.28 | 83 | 2.53 | 4.25 | 9.36 | 1849 |
| Si ₃ N ₄ SN | 310 | 0.24 | 496 | 5.60 | 3.18 | 3.06 | 1870 |
| Si ₃ N ₄ RB | 165 | 0.24 | 303 | 3.41 | – | – | – |
| Si ₃ N ₄ HP | 310 | 0.24 | 827 | 5.60 | 3.19 | 3.06 | 1870 |
| SiO ₂ | 76 | 0.16 | – | 0.77 | 2.20 | 0.54 | 1610 |
| SiC Sn | 331 | 0.19 | 386 | 4.94 | 3.21 | 4.32 | 1980 |
| SiC HP | 414 | 0.19 | 462 | 4.94 | 3.21 | 4.32 | 1980 |
| B ₄ C | 290 | – | 310 | – | 2.41 | 3.06 | 2350 |
| TiB ₂ | 552 | 0.20 | 896 | 6.92 | 4.62 | 8.10 | 2900 |
| TiC | 427 | 0.19 | 248 | – | 4.92 | 8.46 | 3140 |
| TaC | 283 | 0.24 | 200 | – | 14.50 | 6.66 | 3880 |
| BeO | 359 | 0.24 | 234 | – | 3.00 | 5.76 | 2530 |
| WC | 669 | 0.20 | – | – | 15.80 | 4.50 | 2870 |
| Cr ₂ O ₃ | 103 | – | 262 | 3.85 | 5.21 | 7.56 | 2435 |
| Cr ₃ C ₂ | 386 | 0.20 | – | – | 6.70 | 9.67 | 1890 |
| BN _L | 34 | – | 76 | – | 1.94 | 6.66 | 2982 |
| BN _L | 76 | – | 110 | – | 1.94 | 0.36 | 2982 |
| NbC | 448 | 0.21 | – | – | 7.82 | 6.66 | 3499 |

Table 2. 7: Strength and toughness of some CFCC compared with those of corresponding monolithic ceramics (Tanaka, 2000)

| CMC, Ceramics | Flexural strength (MPa) | Fracture Toughness K _{IC} (MPa•m ^{1/2}) |
|--|-------------------------|--|
| SiC/LAS | 830 | 17 |
| LAS | 200 | 2 |
| SiC _W /Al ₂ O ₃ | 800 | 8.7 |
| Al ₂ O ₃ | 550 | 4~5 |
| ZrO ₂ (TZP) | 954 | 11.6 |
| SiC/SiC | 193 | 30 |
| SiC | 402 | 3.4 |
| (Pressureless sintered) | | |
| SiC _W /Si ₃ N ₄ | 800 | 15.6 |
| CVD-SiC/RBSN | 620 | – |
| HIP'ed Si ₃ N ₄ | 863 | 5 |

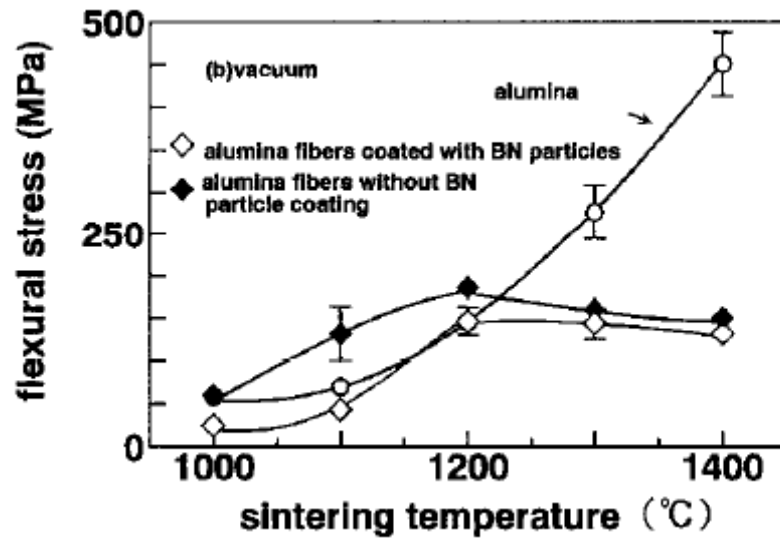


Figure 2. 2 Room temperature flexural strength of monolithic alumina and alumina fiber (22 vol%)/alumina matrix composites sintered in air or in vacuum (Kamino et al, 1996)

Table 2. 8: Modulus of matrices (Papakonstantinou, 2003)

| Matrix | SiC | CAS | Zircon | Glass (N51A) | Si ₃ N ₄ | Polysialate |
|-----------------------|-----|-----|--------|--------------|--------------------------------|-------------|
| Young's Modulus (GPa) | 400 | 120 | 195 | 72 | 193 | 10 |

Table 2. 9: Properties of available ceramic fibers (Papakonstantinou, 2003)

| Trade Name | Manufacturer | Composition (wt%) | Density (g/cm ³) | Diameter (μm) | Fibers Tow | Process | Elastic Modulus (GPa) | Strength (GPa) | Use Temperature °C | CTE (x10 ⁻⁶ /°C) | Comments |
|-----------------------|---------------|--|------------------------------|---------------|---------------|------------------------------|-----------------------|----------------|--------------------|-----------------------------|----------------------------|
| Nicalon 201,P | Nippon Carbon | 56.6%Si 31.7%C 11.7%O | 2.55 | 14 | 500 | polymer pyrolysis | 210 | 3.0 | <1300 | 3.20 | 1.4% elongation to failure |
| Nicalon 202,M | Nippon Carbon | 56.6%Si 31.7%C 11.7%O | 2.55 | 14 | 500 | polymer pyrolysis | 200 | 2.8 | <1300 | 3.20 | 1.4% elongation to failure |
| Nicalon 404,DCC-2,HVR | Nippon Carbon | 56.6%Si 31.7%C 11.7%O | 2.35 | 14 | 500 | polymer pyrolysis | 180 | 2.8 | <1300 | 3.20 | 1.4% elongation to failure |
| Hi Nicalon | Nippon Carbon | 62% Si, 32% C, 0.5% O | 2.74 | 14 | 500 | polymer pyrolysis | 270 | 2.8 | <1400 | 3.50 | 1.0% elongation to failure |
| Hi Nicalon-S | Nippon Carbon | 68.9% Si, 30.9% C 0.2% O | 3.10 | 12 | 500 | polymer pyrolysis | 420 | 2.6 | <1400 | 3.50 | 0.6% elongation to failure |
| Tyranno LoxM | Ube | 55.4% Si, 32.4% C 10.2 % O, 2%Ti | 2.48 | 11 | 800 | polymer pyrolysis | 187 | 3.3 | N/A | 3.1 | 1.8% |
| Tymno ZM | Ube | 55.3% Si, 33.9% C 9.8% O, 1.0% Zr | 2.48 | 11 | 800 | polymer pyrolysis | 192 | 3.3 | N/A | - | 1.7% |
| Sylramic | Dow-Corning | 66.6% Si, 28.5% C 2.3% B, 2.1% Ti 0.8% O, 0.4% N | 3.0 | 10 | 800 | polymer pyrolysis and sinter | 380 | 3.2 | >1400 | 5.4 | Developmental Fiber |
| SIBN(C) | Bayer | SIBN ₃ C with 1-3% O | 1.8-1.9 | ~14 | 300 | Melt spin polymer pyrolysis | 358 | 3.0 | - | 3.5 | Developmental Fiber |
| Tonen | Tonen | 58% Si, 37%N 4% O, trace C | 2.5 | 10 | - | polymer pyrolysis | 250 | 2.5 | N/A | - | - |
| SCS-6 | Textron | SiC on C | 3.0 | 140 | Mono-filament | polymer pyrolysis | 390 | 4.0 | >1400 | 4.6 | - |
| SCS-9A | Textron | 50% SiC, 50% C | 2.8 | 79±5 | Mono-filament | CVD on C | 307 | 3.45 | >1400 | 4.3 | - |

| Trade Name | Manufacturer | Composition (wt%) | Density (g/cm^3) | Diameter (μm) | Fibers Tow | Process | Elastic Modulus (GPa) | Strength (GPa) | Use Temperature $^{\circ}\text{C}$ | CTE ($\times 10^{-6}/^{\circ}\text{C}$) | Comments |
|-------------|---------------|--|-----------------------------|----------------------------|---------------|------------------------------|-----------------------|----------------|------------------------------------|---|----------------------|
| Fiber FP | DuPont | >99% $\alpha\text{-Al}_2\text{O}_3$ | 3.92 | 20 | 200 | slurry spinning | 380-400 | 1.38 | - | -9 | - |
| PRD-166 | DuPont | ~80% $\alpha\text{-Al}_2\text{O}_3$ ~20% ZrO_2 | 4.2 | 20 | 200 | slurry spinning | 380 | 2.3 | - | -9 | - |
| Nextel 312 | 3M | 62% Al_2O_3 , 24% SiO_2 , 14% B_2O_3 | 2.7 | 10-12 | 740-780 | sol-gel | 150 | 1.7 | <1000 | 3 | Melting point 1800 |
| Nextel 720 | 3M | 85% Al_2O_3 | 3.4 | 10-12 | 400 | sol-gel | 260 | 2.1 | <1000 | 6 | Melting point 2100 |
| Nextel 650 | 3M | 89% Al_2O_3 , 10% ZrO_2 , 1% Y_2O_3 | 4.1 | 10-12 | 750 | sol-gel | 358 | 2.55 | <1000 | 8 | Melting point 1710 |
| Nextel 610 | 3M | 0.2-0.3% SiO_2 0.4-0.7% Fe_2O_3 >99% $\alpha\text{-Al}_2\text{O}_3$ | 3.96 | 10-12 | 400 | sol-gel | 373 | 2.93 | <1000 | 7.9 | Melting point 2000 |
| Almax | Mitsui Mining | >99% $\alpha\text{-Al}_2\text{O}_3$ | 3.6 | 10 | 1,000 | slurry spinning | 210 | 1.8 | - | 8.8 | - |
| Saffil | ICI | 95% Al_2O_3 , 5% SiO_2 | 3.6 | 3 | - | - | 100 | 1.0 | - | - | - |
| Sigma 1140+ | Dera | $\beta\text{-SiC}$ +W core | 3.3 | 100 | - | - | 330-360 | 3.2 | - | - | - |
| Altex | Sumitomo | 85% Al_2O_3 , 15% SiO_2 | 3.2 | 16 | 1,000 | Sol-gel | 193 | 2.0 | - | 7.9 | - |
| Sumika | Sumitomo | 85% Al_2O_3 , 15% SiO_2 | 3.2 | 9 | - | Sol-gel | 250 | 2.6 | - | - | - |
| Saphikon | Saphikon | 100% Al_2O_3 | 3.96 | 125 | mono-filament | edge-defined film-fed growth | 470 | 3.5 | >1400 | 9 | Developmental fibers |

CHAPTER 3. FABRICATION TECHNIQUES

3.1 Introduction

Utilizing FRP material in a design usually assumes that the materials are joined together as a unified structure. Strength and stiffness predictions rely solely on the assumption that all materials are completely bonded together to form one cohesive element. Therefore, the bond between polymer matrix and the fiber reinforcement is critical in determining the mechanical properties of the resultant composite material. The impregnation process is the most significant way to achieve good adhesion and strong bonding. During fiber wetting, all surfaces of the reinforcement must be exposed to the resin. Otherwise, gas-filled bubbles, air voids, crevices, and other discontinuities or defects will remain, adversely affecting the mechanical properties of the finished composite.

It is well known that fiber and matrix type largely influence the overall mechanical properties of a composite. However, the end properties of these materials are also a function of the way in which the composites are prepared and processed. Table 3.1 shows a summary of common fabrication processes for CMC compositions. This chapter presents the methods in which the composite samples were prepared for this study including resin mixing, hand lay-up, hot pressing, vacuum bagging, and vibration methods. The mechanics and logistics of these methods are discussed in detail as well as other processing factors.

3.2 Resin Preparation

The base inorganic resin is a two-part inorganic system that consists of an amber-colored potassium silicate solution (Part A) and a white, amorphous, silica powder (Part B). If room temperature curing is desired, a hardener in powdered form can be added to the basic mix of Parts A and B. To facilitate better wet-out of the fibers, a liquid wetting agent can also be added to the base mix.

The components of the resin are mixed together in a small high-shear mixer containing serrated stainless steel blades. After 30 seconds of vigorous mixing at 1,500 RPM, the sides of the mixer are scraped to remove any clumps of powder not blended. The resin is mixed again for another 30 seconds and then placed into a freezer at approximately -1°C (30°F) for about 15 minutes to allow any entrapped air to escape. The polymer has a pot-life of approximately 2 hours at room temperature. If the hardener is added for room temperature curing, the pot-life is significantly reduced to about 1 hour or less. Since the pH of the polymer is almost 14, latex gloves are always worn during the fabrication process. However, the polymer is non-toxic and does not emit any toxins or fumes during the mixing process.

3.3 Fabrication Techniques

As mentioned previously, four different common practice fabrication techniques were investigated to create the CMC samples. These procedures were first developed for use with organic resins, such as epoxy. However, Geopolymer resins do not require new technologies and can take full advantage of processing techniques in use today. The following will discuss in detail the different fabrication methods considered.

3.3.1 Hand lay-up

The hand lay-up or wet lay-up technique is a very simple and widely used process. A schematic of the hand lay-up process is presented in Figure 3.1. Typically, this procedure is broken down into four steps: mold preparation, coating, lay-up, and curing. In other composite applications molds can be prepared and wrapped with the laminate to create various objects such as canoes. The mold can be constructed out of various types of materials and should be chosen by the designer.

However, specific molds are not required in all applications, such as laminates which can be free formed as well. The coating or impregnation process is achieved by pouring a specified amount of the prepared resin onto one ply of a woven fabric or chopped fiber sheet. Squeegees, brushes, and grooved rollers are used to force the resin into the fabric and to remove much of the entrapped air. The wet lay-up process layers one impregnated ply on top of the other until a predetermined thickness is achieved. Serrated hand rollers are used to compact the plies together to ensure the removal of air and excess resin. The curing process is usually accomplished at room temperature, but other curing techniques could be used as well.

3.3.2 Hot Pressing

Hot pressing is a simultaneous combination of consolidation and temperature curing process for CMCs. A matrix slurry is prepared and set on either a tool or into a mold. The tool is placed into a hydraulic or screw driven press which then compresses the material. Figure 3.2 displays an example of a hydraulic heat press. The press

simultaneously applies a predetermined force, usually between 20 to 40 MPa, while generating heat. Typical ceramic matrix composites are produced using temperatures between 1500°C to 1900°C.

This process compresses the slurry allowing air bubbles to escape as well as increasing the rate of densification of the compact. The densification joins the material together and stimulates bonding. The resultant composite has low porosity and is fairly uniform.

3.3.3 Vacuum Bagging

Vacuum bagging is an economical and effective method that has been used primarily for the manufacturing of aerospace structures. However, vacuum infusion has gained popularity in other fields as indicated in Table 3.2. In this process, a fiber reinforced matrix or fiber reinforcement layers are first impregnated with resin, then stacked together and placed inside a sealed bagging system. A vacuum pump is then attached to the bag, removing the air from the bag and allowing external atmospheric pressure to firmly press the FRP composite. The wet FRP layers are pressed tightly against the surface being covered so that the excess resin is squeezed out and soaked up in a disposable outer wrap. The vacuum-bagging system allows for predictable and consistent pressure application, providing control on FRP thickness, reducing void content, improving resin flow, and assisting in bonding. The most critical element of a vacuum bagging system is that a smooth surface must be provided around the perimeter of the bag to create an airtight seal (Nazier, 2004).

All vacuum bagging setups are essentially the same, with some minor variations depending on the specific application. The basic vacuum bagging system consists of several key elements as shown in the schematic in Figure 3.3. A firm tool, which is the surface the composite will be pressed to, is essentially the foundation of the system. This tool is usually in the form of a smooth flat plate, a metal sheet, or a deeply curved cowl form. The tool needs to be strong enough for ordinary handling, but does not need to withstand large forces as seen in a metal forge or a steel press.

Metal is usually chosen as the base of the system since its surface is non-porous, hard, and very smooth. A non-porous surface is essential to ensure a tight bond with the sealant tape, which is placed around the perimeter of the tool and is used to seal the bagging film to the metal tool. Sealant tape is a sticky, putty-like material, which comes in ½” wide rolls with a release paper on one side. The pliable sealant tape is pressed firmly against the tool, leaving the release paper on until the bag is ready to be sealed. The tape is usually applied after the composite laminate is layed up, especially if it is a wet lay-up. Forming a tight bond between the sealant tape and the metal tool is of paramount importance if the bagging system is to perform effectively; even the slightest opening will compromise the entire bagging system (Nazier, 2004). Figure 3.4 presents an example of an actual vacuum bagging system.

3.3.4 Vibration

The vibration process is a very simple method which requires minimal equipment. An open mold, which can be of various materials, is used to contain the aqueous composite compound. However, the mold should be made of a material that will not

adhere to the wet matrix. The slurry filled mold is then placed atop a vibration table. A rubber pad is placed in between the mold and table for better harmonic distribution and to protect the mold from cracking. The mold is properly secured to the vibration table to ensure it remains in direct contact with the table and transfers the vibrations into the wet matrix. This can be achieved by using bungee cords or straps. After the mold is secured to the vibration table the power can be turned on and left to run for a predetermined period of time.

This process allows for the compound to settle and take the shape of the mold. During the vibration process, entrapped air travels through the compound and is released at the surface. The mold is removed from the vibration table and the composite is left to cure at room temperature. However, this process can lead to inconsistent thicknesses if low viscosity matrix slurries are used.

3.4 Curing Temperature

Curing is the process of irreversibly changing the physical properties of a thermosetting resin usually by a chemical reaction. Curing can be achieved by the application of pressure and/or heat. Vacuum bagging is one technique in which pressure cures a compact.

Temperature curing can be achieved by two different methods. One, while the slurry or laminate is still wet, heat can be applied simultaneously during a molding process, such as hot pressing discussed above, to cure the composite. The second temperature curing process allows the slurry to harden at room temperature and is then placed in an oven to be heated to the required curing temperature.

Another curing process is room-temperature curing which is highly advantageous and currently attracting much research. This method eliminates the need of kilns or other ovens to heat composites to high temperatures to be cured. This is achieved by using additives or other powders into a matrix which induces chemical bonding and curing.

3.4 Summary

The fabrication cost of composites can be extremely high and not economical. Table 3.3 presents a cost estimation for some of the fabrication techniques discussed.

The following concluding remarks will discuss the advantages (A) and disadvantages (B) for each of the aforementioned fabrication processes:

- Hand lay-up – (A) Freedom of design, low cost, large sized parts possible (B) Labor intensive, operator-skill dependent
- Hot Pressing – (A) Fairly uniform fiber distribution, low porosity (B) Limited to size of tool, high cost
- Vacuum Bagging – (A) Low porosity, low cost, fairly uniform composite (B) Hard to form smooth surface, sensitivity to leaks
- Vibration – (A) Very simple, little skill required (B) High porosity, inconsistent fiber distribution, variation in thickness

Table 3. 1: Summary of CMC fabrication processes and examples of typical composite systems fabricated by these processes (Chawla, 2003)

| Process | Examples |
|---|---|
| Slurry infiltration (ply stacking and hot pressing) | SiC/glass ceramic, carbon/glass-ceramic, C/glass, mullite/glass |
| Powder processing and hot pressing | SiC/Al ₂ O ₃ , Al ₂ O ₃ /Al ₂ O ₃ |
| Gas-liquid metal reaction (Lanxide) | SiC/Al ₂ O ₃ , SiC/SiC |
| Sol-gel (infiltration and sintering/hot pressing) | C/glass, mullite/mullite |
| Chemical vapor infiltration (infiltration of a woven preform) | SiC/SiC, C/SiC |
| Polymer conversion (infiltration and pyrolysis) | C/C, C/SiC, SiC/Si-C-N |

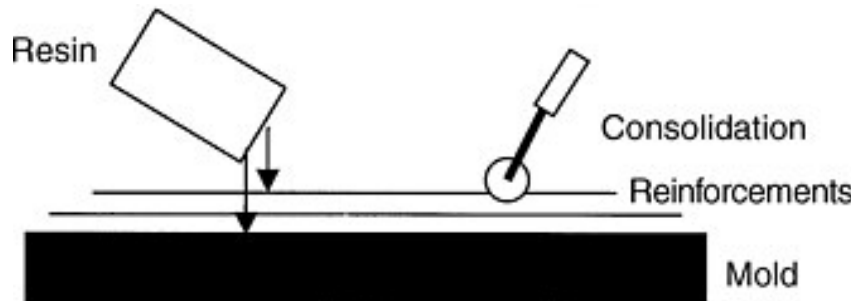


Figure 3. 1: Hand lay-up schematic (Andressen, 2003)

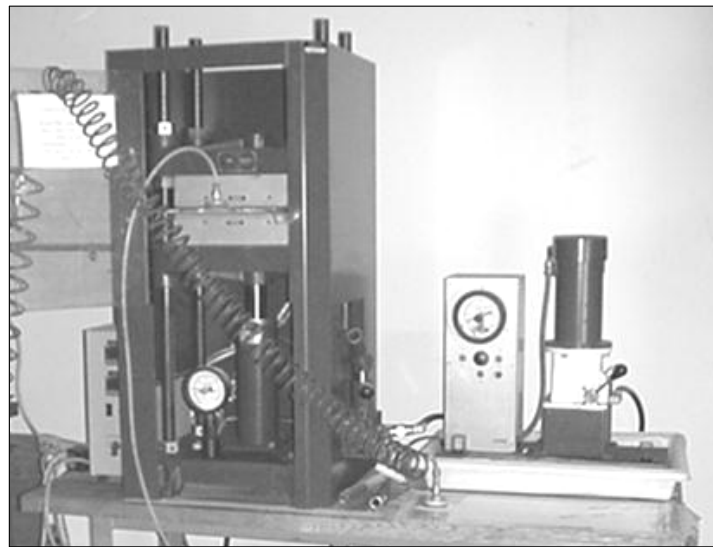


Figure 3. 2: Hydraulic press with heated platens used for hot pressing

Table 3. 2: Examples of applications of parts made with vacuum infusion (Hoebergen, 2003)

| Sector | Applications |
|----------------|---|
| Marine | Hulls, decks, and hatches of yachts, hulls and decks of recreational boats |
| Transportation | Roof and floor of refrigerated container, automotive exterior body panels, train fronts |
| Aerospace | Rudder of small aircraft |
| Industrial | Fan blades, part for fish counting unit, toilet bowl, oil separator |
| Energy | Solar cell housings, wind turbine blades, electrical insulation materials |
| Infrastructure | Lighting columns, bridge deck |
| Military | Hull of composite armored vehicle, hull of stealth corvette |

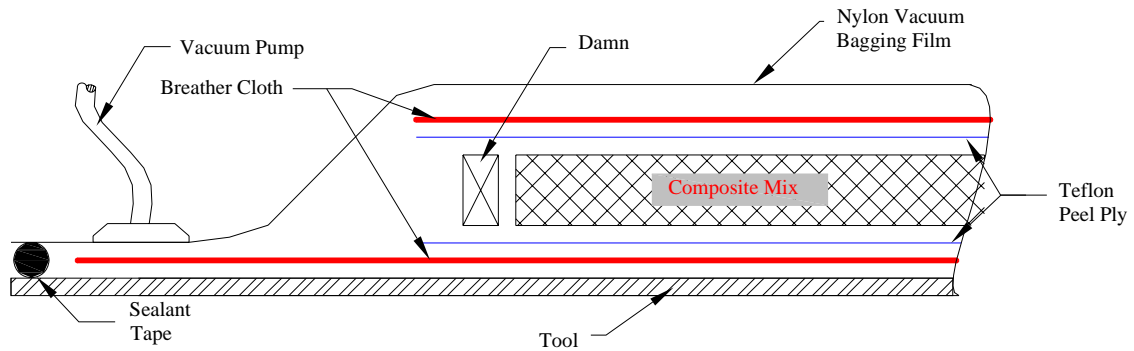


Figure 3. 3: Schematic of vacuum bagging setup

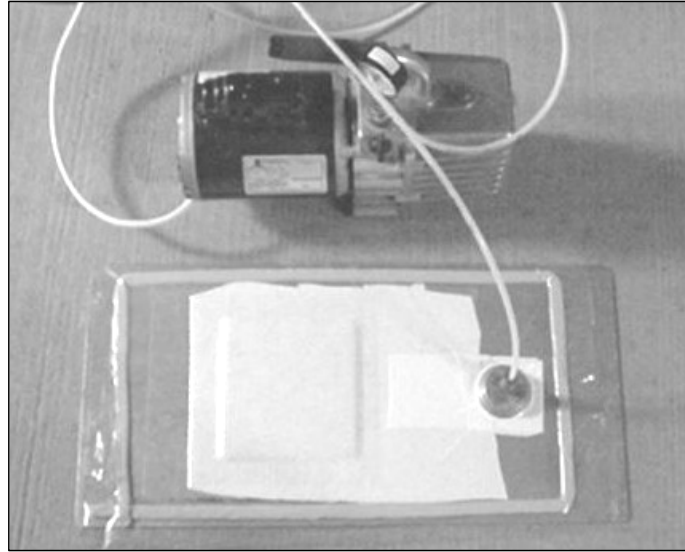


Figure 3. 4: Vacuum bagging assembly and pump

Table 3. 3: Comparison of different cost categories for different manufacturing processes (Hoebergen, 2003)

| | Vacuum infusion | RTM | Hand lay-up | Spray-up | Low P/T prepreg (a) | Autoclave prepreg |
|-----------------------|-----------------|----------|-------------|----------|-------------------------------------|-------------------|
| Workshop requirements | \$\$ | \$\$ | \$\$\$\$ | \$\$\$\$ | \$\$\$ | \$\$\$ |
| Equipment | \$\$ | \$\$\$ | \$ | \$\$ | \$\$\$ | \$\$\$\$\$ |
| Tooling | \$\$ | \$\$\$\$ | \$\$ | \$\$ | \$\$ | \$\$\$ |
| Ancillary materials | \$\$\$ | \$\$ | \$ | \$ | \$\$\$ | \$\$\$ |
| Raw materials | \$\$ | \$\$ | \$\$ | \$ | \$\$\$ | \$\$\$\$ |
| Labor | \$\$ | \$ | \$\$ | \$ | \$\$\$ | \$\$\$ |

CHAPTER 4. EXPERIMENTAL INVESTIGATION

4.1 Introduction

The fabrication process of any composite material is a critical procedure that can substantially affect the mechanical properties. The fiber format is a key factor when determining the fabrication technique to be used to create the composite. Woven fiber fabrics or sheets perform best using hand lay-up or vacuum bagging techniques. Short fibers or whiskers incorporated in wet free-form slurries can be molded using various techniques such as hot pressing. However, the designer should understand the level of skill and equipment required to master some of the composite fabrication processes. The following describes in detail how the various samples in this study were prepared. In order to understand the effects of different fabrication techniques, four separate series were investigated.

4.2 Fiber reinforcement

The following will discuss the commercially available fibers used in this study to reinforce the ceramic matrix composites produced.

4.2.1 Carbon Fibers

Carbon fibers can experience problems because of their poor wettability against metals (Ryu, 2000). To rectify this, the fibers are usually coated with a light metallic dusting. Nickel is one metal commonly used to coat the carbon fiber and enhance the

wettability of the strands. High modulus carbon fibers coated with nickel are commercially available in spools with multiple toe counts.

4.2.2 Ceramic Fibers

4.2.2.1 Reformatted Alumina

The reformatted alumina fiber is a flexible paper type material that is available in rolls or cut sheets. These fiber sheets are ideal for thermal insulation in various applications. Manufactures can use either an organic or inorganic binder to hold the fibers together. The density of the fiber ranges from 0.14 to 0.7 g/cc depending on the thickness of the sheet. This alumina fiber is rated for a maximum use temperature of 1650°C.

4.2.2.2 Bulk Alumina

Bulk alumina fibers are a material processed with short fiber lengths having a relatively cotton-like consistency. Again, these fibers make good thermal insulators, but can be used in vacuum forming of high alumina fibrous ceramic materials. The mean fiber length is 3.2mm with a density of 3.4 g/cc. This fiber is rated at 1700°C maximum use temperature.

4.2.2.3 Short Ceramic

Short ceramic fibers have been specifically developed for the reinforcement of metal and high temperature ceramic matrix composites. These fibers are fully crystalline which make them very chemically stable. The ceramic fibers can be converted into

textiles, papers, fabrics and many other applications where there are thermal requirements. Commercially available structural ceramics fibers by Nextel have densities ranges between 3.4 and 3.9 g/cc. These fibers have a melting point over 2000°C.

4.3 Specimen Preparation

4.3.1 Matrix Preparation

The ceramic matrix composition used the inorganic resin as the base binder. Table 4.1 presents all twenty-one of the matrix compositions prepared in this study. Alumina and silicate powders were then added to the base in predetermined ratios, by weight. Other fine sands and admixtures, such as water agents were also combined into the matrix. The water agents help the resin remain viscous, which increases the ability to wet the fibers. All of the matrix components were placed in a high shear mixer for about 1 minute. However, this process is dependant on the amount of material in the mixer resulting in longer mixing durations for larger quantities.

The fiber reinforcement can be added to the mixing container after the slurry is well incorporated. Nickel coated carbon, bulk alumina, and ceramic fibers were chosen as the discrete fiber reinforcement in this study. The time required to chop, wet and incorporate all of the fiber into the matrix is dependant on the fiber format and content. For example, bulk alumina fibers have a thick cotton-like consistency which requires a longer mixing time than short ceramic fibers. Adding the dry fibers at a slow rate will increase their wettability and reduce the total blending time. A scraper should be used to

clean the walls of the mixing container to ensure all of the fibers have been impregnated with the resin.

To achieve room temperature curing, a hardening powder is combined into the matrix. However, it is important to add the hardener at the end of the mixing cycles. This will allow the resin to remain viscous while wetting the fibers and achieve the longest possible pot life.

4.3.2 Fabrication Series

4.3.2.1 Series I

The first fabrication series investigated the use of reformatted alumina plies with thicknesses of 3.2 and 6.35 mm as the reinforcement. A hand lay-up technique was used to create the laminates. The alumina ply was cut into square pieces having a surface area of about 230 cm². The resin was prepared as described above without the addition of discrete fibers and refrigerated to increase the pot life. The inorganic based resin was then poured onto an alumina ply in predetermined amounts. Squeegees, brushes, and grooved rollers were used to maneuver the resin across the ply as well as impregnate the fiber. One side of the ply is completely wetted with the resin and is then flipped over to repeat the process on the other side. A fairly skilled hand is required during this process to ensure an evenly distributed coating of the resin and a well impregnated fiber ply. Multiple plies are prepared and stacked one on top of the other until the required thickness is achieved. Grooved rollers are used to force one ply to adhere to the other as well as removing much of the entrapped air between layers. The wet laminate was then

placed in a standard vacuum bagging system to achieve uniform pressure and remove entrapped air and excess resin. The vacuum bag system is then placed in a hydraulic heat press at a pressure of 48 MPa and temperature of 150°C for a minimum of 3 hours. Works done by J. Giancaspro et al determined the required pressure and temperature to properly compress and cure the laminate without damaging the fibers or compact. The composite is left to slowly cool down to room temperature in the press to prevent thermal cracking after the curing time has elapsed. The vacuum bag system can then be disassembled to reveal the final laminate plate. The ceramic plates are then cut into 12 mm wide by 63.5 mm long coupons using a wet-saw with a diamond tipped blade. Figure 4.1 shows an example of the wet-saw used for cutting. The coupons are placed in a low temperature oven at 90°C to fully dry the coupons and remove any absorbed moisture during cutting.

4.3.2.2 Series II

A hot-press technique was used to fabricate ceramic matrix composites with discrete fiber reinforcement. The inorganic resin was prepared as previously described with the addition of fiber reinforcement. The aqueous slurry is removed from the high shear mixer and placed directly on a Teflon[®] peel ply protecting the tool. A top peel ply is added and grooved rollers are used to spread the mixture as uniformly as possible across the tool. A second metal sheet is placed on top of the wet slurry to protect the hydraulic press. Metal shims are placed in between the tools at all four corners to regulate the thickness of the composite. The system is placed in a heated hydraulic press at a pressure of 48 MPa and temperature of 80°C. A lower temperature than Series I

above was used to slow the curing process when using wet slurries instead of laminates. A duration of 48 hours in this system is required to cure the composite. The plate was cooled to room temperature and removed from the hydraulic press. Smaller coupons are cut and dry in a similar process described above.

4.3.2.3 Series III

This series designates samples that were fabricated using only a vibration process. This process was a laboratory investigation to simplify equipment and fabrication cost. The resin was mixed with milled or short fibers using a high shear mixer as described above. The fiber reinforced slurry was placed in a plastic mold about 250 cm². A 6mm thick rubber pad was placed in between the mold and the vibrating table. A bungee cord was used to secure the mold to the table during vibration.

This technique allows entrapped air to travel up through the matrix and escape from the top surface. The matrix was left in the mold for 48 hours to cure at room temperature. After the designated curing time has elapsed, the composite plates were removed from the mold and placed in a low temperature oven at 200°C for 24 hours. This heating process is required to dry the composite and remove any residual moisture. The specimens are left to slowly cool down to room temperature before cutting into smaller coupons and again oven dried at 200°C.

Two main disadvantages were discovered during this technique. One, is the exposed surface is left with a rough finish due to the air bubbles escaping. This can lead to seating errors and false failures during testing. The other disadvantage is shrinkage

can occur during the curing process which cambers the composite. This occurs because of uneven curing due to one exposed and one protected surface.

4.3.2.4 Series IV

The last fabrication technique combined a vacuum bagging system with a heat-press process for the CMCs. Discrete fibers were mixed with the resin using a high shear mixer and poured onto a protected metal tool. A stainless-steel damn with approximate dimensions of 150 by 150 by 20 millimeters was used to contain the slurry in a set boundary. The damn was placed in a standard vacuum bagging system with the aqueous slurry. The bagging system was sealed and grooved rollers were used to gently distribute the wet mixture within the damn.

The vacuum pump was turned on and again the mix was rolled to ease the distribution. The entire system was then placed into a hydraulic heat press at a pressure of 48 MPa and temperature of 80°C. The plate would remain in the machine for a duration of four days. During this time period the vacuuming, applied pressure and heat were adjusted until the composite was completely cured. Again, the plate was left to cool to room temperature and then cut into smaller coupons. This process resulted in a much more uniform plate thickness with fewer imperfections which were disadvantages to the previous fabrication series.

4.4 Test Preparation

All of the samples within this study were cured at relatively low temperatures ranging from 80°C to 400°C depending on the matrix composition and fabrication

technique. After this curing process was achieved it was necessary to determine how the ceramic composites would perform in a high-temperature environment. A high temperature oven with maximum exposure temperature of 1300°C was used to heat the coupons to 400, 600, 800, and 1050°C for 1 hour time intervals.

Although the mechanical and physical properties of the composites were affected by the exposure temperature, the variation was minimal at 600°C and 800°C. A careful review of the results from this study led to the decision to heat samples to a maximum curing temperature of 400°C if hardeners were not added to the matrix to achieve room temperature curing. A maximum exposure temperature of 1050°C was used to simulate aggressive thermal applications

4.5 Test Method

The flexure tests were conducted over a simply supported span of 50.8 mm with a center point load in accordance with ASTM D790 (American Society for Testing and Materials, 1999). The span-to-depth ratios ranged from approximately 6:1 to 11:1, both of which fell within the acceptable limits of the standard flexure test. A schematic of the test setup is presented in Figure 4.2. The tests were conducted on an MTS TestWorks® system under deflection control with a mid-span deflection rate of 0.25 mm/min. Load and deflection readings were taken using a computer for the entire test duration.

4.6 Independent variables

Several variables were investigated during composite processing and post processing. The processing variables include the following: (i) silica to alumina ratio in

the ceramic matrix, (ii) fiber type, (iii) fiber volume fraction and, (iv) the fabrication technique. After the composite is fully cured the post processing thermal investigation can begin. The last variable considered was an extreme heat exposure at 1050°C for a 1 hour time duration.

4.7 Summary

Composite materials are highly dependant on their fabrication processes and are extremely vulnerable during manufacturing. Many imperfections and impurities can be introduced into the composite if proper techniques are not followed. The fabrication series investigated utilized several fiber formats with different processing techniques. Tables 4.2 and 4.3 present a summary of the fabrication techniques and fibers used in each experimental series.

Series I is a fairly labor intensive process which can be time consuming. However, it is possible to achieve relatively uniform end products. Series II and III are very simple processes, but can experience high levels of impurities. The vibration process resulted in composites with the most imperfections and inconsistencies. The last fabrication technique, Series IV, produced the most uniform composites with minimal defects.

Table 4. 1: Matrix Compositions

| Mix ID | Sample ID | Chemical Composition (gm) | | | | | Alumina Fibers | | Chopped Ceramic (gm) | Carbon Fiber (gm) |
|--------|-----------|---------------------------|--------|--------|-----------|----------|----------------|-----------|----------------------|-------------------|
| | | Potassium Silicate | Part B | Part D | Fine Sand | Hardener | Paper (ply) | Bulk (gm) | | |
| 1 | A1 | 50 | 60 | 5 | -- | -- | 3 | -- | -- | -- |
| 2 | A2 | 50 | 60 | 5 | -- | 5 | 3 | -- | -- | -- |
| 3 | A3 | 75 | 101.3 | 7.5 | -- | -- | 1 | -- | -- | -- |
| 4 | A4 | 75 | 101.3 | 7.5 | -- | 18.4 | 1 | -- | -- | -- |
| 5 | B1 | 50 | 20 | 30 | 68 | -- | -- | -- | -- | 8 |
| 6 | B2 | 50 | 10 | 40 | 68 | -- | -- | -- | -- | 8 |
| 7 | B3 | 50 | -- | 50 | 68 | -- | -- | -- | -- | 8 |
| 8 | B4 | 50 | 20 | 30 | 68 | -- | -- | 8 | -- | 2 |
| 9 | B5 | 50 | 10 | 40 | 68 | -- | -- | 8 | -- | 2 |
| 10 | B6 | 50 | -- | 50 | 68 | -- | -- | 8 | -- | 2 |
| 11 | B7 | 50 | -- | 50 | 68 | 5 | -- | -- | -- | 4 |
| 12 | B8 | 50 | -- | 50 | 68 | 5 | -- | 8 | -- | -- |
| 13 | C1 | 50 | 10 | 90 | 60 | 5 | -- | 10 | -- | -- |
| 14 | C2 | 50 | 10 | 90 | 60 | -- | -- | 10 | -- | -- |
| 15 | C3 | 50 | 10 | 90 | 60 | 5 | -- | -- | 15 | -- |
| 16 | C4 | 50 | 10 | 90 | 60 | 5 | -- | -- | 20 | -- |
| 17 | C5 | 50 | 10 | 90 | 60 | 5 | -- | 15 | -- | -- |
| 18 | D1 | 50 | 10 | 90 | 30 | 5 | -- | 20 | -- | -- |
| 19 | D2 | 50 | 10 | 90 | 30 | 5 | -- | -- | 20 | -- |
| 20 | D3 | 50 | 10 | 90 | 45 | 5 | -- | -- | 25 | -- |
| 21 | D4 | 50 | 10 | 90 | 40 | 5 | -- | -- | 30 | -- |

**Figure 4. 1: Diamond blade wet saw used to cut composite plates**

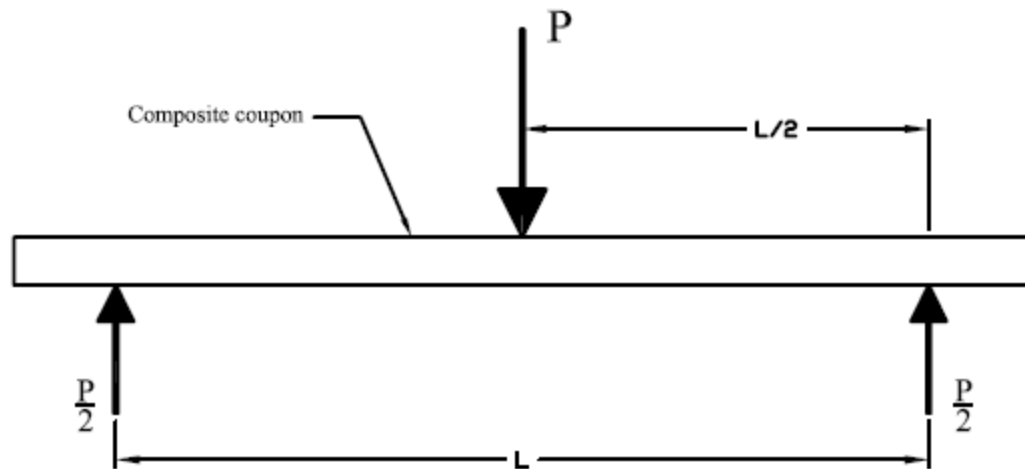


Figure 4. 2: Schematic of flexural test setup

Table 4. 2: Summary of Fabrication Series

| Fabrication Series | Hand Lay-up | Heat Pressing | Vacuum Bagging | Vibration |
|--------------------|-------------|---------------|----------------|-----------|
| I | ✓ | ✓ | ✓ | -- |
| II | -- | ✓ | -- | -- |
| III | -- | -- | -- | ✓ |
| IV | -- | ✓ | ✓ | -- |

Table 4. 3: Fiber Type associated with Fabrication Series

| Fabrication Series | Alumina Ply | Alumina Bulk | Carbon | Short Ceramic |
|--------------------|-------------|--------------|--------|---------------|
| I | × | -- | -- | -- |
| II | -- | × | × | -- |
| III | -- | × | -- | × |
| IV | -- | × | -- | × |

CHAPTER 5. TEST RESULTS AND DISCUSSION

5.1 Introduction

This chapter will present the test results and discussion of the ceramic matrix composite coupons studied in this thesis. The test variables investigated were the modulus of rupture and modulus of elasticity. Flexural tests were conducted on a minimum of three coupons from each experimental sequence resulting in a total of 180 samples. An average value of the three coupons was taken to represent the test results. However, many of the composite plates are represented by the results of one coupon. This is due to outlier data caused by coupon impurities. These impurities can be an affect of fabrication errors, such as entrapped air bubbles, or the unpredictable alignment of milled fibers which will be discussed in detail below.

5.2 Relevant Formulae

The load-deflection curves were converted to apparent flexural stress versus apparent fiber strain. This conversion makes it feasible to compare samples of varying thickness. The flexural stress, σ_f , for a given moment, M , was computed using:

$$\sigma_f = \frac{6M}{bh^2} \quad (5.1)$$

where b and h are the specimen width and thickness, respectively. The extreme fiber

strain, ε_t , was calculated using the following procedure. For the simply supported beam of span length, L , with center-point load, P , has a mid-span deflection of,

$$\delta = \frac{PL^3}{48EI} \quad (5.2)$$

or,

$$\delta = \frac{ML^2}{12EI} \quad (5.3)$$

Where E is Young's modulus and I is the moment of inertia. Since the strain at the extreme tension fiber is the curvature times one-half the thickness of the specimen, the extreme fiber strain becomes:

$$\varepsilon_t = \frac{M}{EI} \times \frac{h}{2} \quad (5.4)$$

Combining equations (5.3) and (5.4) yields the following relationship between deflection and strain:

$$\varepsilon_t = \frac{6\delta h}{L^2} \quad (5.5)$$

Uncracked sections were assumed for both equations (5.1) and (5.5), and these values should be used only for comparison of the various samples' load-deflection characteristics.

5.3 Test Results

The apparent stress versus apparent strain curves are presented in Figures 5.1 through 5.21. The stress-strain behavior of fabrication Series I coupons is presented in Figures 5.1 through 5.4. Stress-strain behavior of fabrication Series II and III are presented in Figures 5.5 through 5.12 and Figures 5.13 through 5.17, respectively. Figures 5.18 through 5.21 present the stress-strain behavior for fabrication Series IV coupons.

The Control curve presented in all of the stress-strain graphs represents sample A1, which is an alumina ply laminate. This composite was chosen as the control to represent previous works by others under the supervision of Dr. P.N. Balaguru. The reader should refer to references by J.W. Giancaspro and A. Foden for further discussion on their previous works.

A summary of all the specimens tested including the maximum apparent stress and corresponding apparent strain calculated from the load-deflection curves are presented in Table 5.1. The 'H' designation added to the sample identification was used to identify the samples that had been heated to an exposure temperature of 1050°C. Designations used to identify the specimens' type of fiber reinforcement include the following: (i) P for alumina ply 3.2mm thick, (ii) P* for alumina ply 6.35mm thick, (iii) C for nickel coated carbon, (iv) A for bulk alumina and (v) B for short ceramic. Samples shown with a fiber type A/C indicated a combination of both carbon and bulk alumina fiber reinforcement. Tables 4.1 from the previous chapter presented the fiber reinforcement included in each matrix composition. The "Fiber V_f ," in Table 5.1

indicates the fiber volume fraction by weight percent of reinforcement in each of the samples. For samples shown with a fiber volume fraction of 4.4/1.1 percent represent a combination of bulk alumina and carbon fiber reinforcement, respectively.

5.4 Discussion

The following will discuss the effects of the experimental variables on the specimens.

5.4.1 Stress-Strain behavior

A review of the apparent stress versus apparent strain curves presented previously leads to the following observations. All of the stress-strain curves behaved in linear elastically. The samples all failed by fracture of the extreme tension face of the coupon. This is due to the random alignment of the discrete fiber reinforcement. The modulus of rupture for the samples was found to be between 20 and 129 MPa. The extreme tensile strain for most samples was between 0.1 and 0.18%. The cause of this large gradient highly depends on the effects of the experimental variables which will be discussed in the following sections.

5.4.2 Effects of increasing Silica/ Alumina Ratio

. Figures 5.22, 5.23 and 5.24, present the effects of varying the silica to alumina ratio on the modulus of rupture, modulus of elasticity and density, respectively. Carbon and bulk alumina fiber reinforcement were investigated for the following silica to

alumina ratios: (i) 1:1, (ii) 1:2 and (iii) 1:5. The following will discuss the results at 400°C exposure temperature. The results at 1050°C will be discussed in a later section.

The carbon fiber reinforcement shows an increase in strength of 88% and 63% from a 1:1 to 1:2 and 1:2 to 1:5 ratios, respectively. While the bulk alumina fiber has a decrease in strength of 22% between 1:1 and 1:2 ratios and then increases by 16% at a ratio of 1:5. This decrease in strength can be a result of dry fibers. The maximum modulus of rupture is 52 MPa for carbon fiber at silica to alumina ratio of 1:5.

The modulus of elasticity of the carbon fiber coupons showed a similar response to the varying silica to alumina ratios. There was a continuous increase from 1:1 to 1:5 ratios for the carbon fibers showing a maximum modulus of 39 GPa. Similarly, the bulk alumina coupons decreased to 21.5 from 29 GPa at 1:2 and then increased to 27 GPa at 1:5. The results of the carbon fiber are expected since they are longer in length, 6 mm compared to the 3 mm, and also have a higher modulus than the bulk alumina fibers.

There was not a significant variation in the densities for either carbon or bulk alumina fibers due to the silica and alumina content. The increase in density is primarily due to the higher density of alumina than silica. The density is directly proportional to the alumina content and not the fiber type.

5.4.3 Effects of fiber reinforcement

As previously mentioned the fiber type and content greatly affect the composite properties. The maximum modulus of rupture, modulus of elasticity and density for the tested coupons are presented in Figures 5.25, 5.26 and 5.27, respectively. Again, only the

results at or below 400°C will be discussed here and the results at 1050°C will be discussed in a later section.

The alumina ply laminates proved to have the weakest strength at 26 MPa while the short ceramic fibers have a maximum modulus of rupture of 129 MPa. The short ceramic fiber more than doubled the strength of any other fibers investigated. Similar behavior was found for the modulus of elasticity. Again the alumina laminates and short ceramic fibers have the lowest and highest modulus, respectively. Typically, carbon fibers would excel at temperatures below 400°C. However, the mulching process during the slurry processing reduced the fiber length preventing full capacity to be achieved. Figure 5.27 presents a comparison of the fibers investigated to the maximum densities of the composites after curing. The select fibers investigated have similar densities, thus the relative stable result. However, the alumina laminates have a much lower density than the other CMCs. This is because only the Geopolymer resin was used to impregnate the reformatted plies compared to the dense ceramic matrix used elsewhere.

The effect of increasing the fiber volume fraction on the modulus of rupture is presented in Figure 5.28. A comparison of bulk alumina and short ceramic fibers is shown. By review of this curve one can deduce that CMCs reinforced with bulk alumina or short ceramic fibers will gain strength as the fiber percentage increases. However, the modulus of rupture increases at a much steeper rate for short ceramic fibers than bulk alumina.

5.4.4 Effects of increasing exposure temperature

The figures previously discussed, (Figures 5.22 to 5.24), presenting the effects of a varying silica to alumina ratio also provide results from heating the samples to a maximum exposure temperature of 1050°C. Again, there is an increase in strength as the alumina content is increased. However, due to the high heat exposure a loss of less than 10% of the flexural strength at a 1:5 silica/alumina ratio for carbon fiber is observed. The coupons reinforced with bulk alumina fibers increased in flexural strength by nearly 40%.

The Young's modulus increased for both carbon and bulk alumina fibers by 13 and 48 percent, respectively. At 1050°C the ceramic matrix sinters, strengthening the chemical bonds, but this is reducing the tensile strain capacity. The extreme heat exposure has a negative effect on the composite densities. At a 1:2 silica/ alumina ratio there is a loss in density between 10 and 20 percent for carbon and alumina fibers. However, at a 1:5 ratio there is only a loss in density of 1 to 5 percent. The ceramic matrix with larger alumina content proves to be more stable at 1050°C.

A review of the previously mentioned Figures 5.22 and 5.23 shows the overall effect on the fiber reinforcement at 1050°C heat exposure. The carbon and bulk alumina fibers experienced a loss in flexural strength of 10 and 23 percent, respectively. The short ceramic fiber experienced a loss of 43% bending strength, which was expected based on the manufacture specifications.

5.4.5 Effects of different fabrication techniques

The main advantages of one fabrication technique over another is the cost and quality assurance. The curing temperature for all of the samples tested was at or below 400°C. Samples cured at room temperature (22°C) were heated to 200°C to remove any

excess water remaining in the resin. The CMCs proved to have flexural strengths between 20 and 130 MPa as seen in the aforementioned figures and tables. These composites greatly reduce the processing cost and equipment needed.

A careful physical review of composite samples for each fabrication series investigated results in the following remarks.

Fabrication Series I: There were fairly dense and uniform laminates. However, it is difficult to control the wetting uniformity which would result in a composite with a low fiber to resin ratio. This creates weaknesses in the composite plate and could result in premature failure.

Fabrication Series II: These samples resulted in relatively dense, strong composites with a uniform plate thickness. However, air voids remained entrapped in the matrix causing weaknesses.

Fabrication Series III: This series further reduced processing costs by using very primitive equipment. Unfortunately the ease of processing created a trade off for quality and performance. There was a large inconsistency in plate thickness and large air voids in the finished composite.

Fabrication Series IV: This series had the most uniform and structural consistent samples. The application of vacuum bagging and molding removed entrapped air and contained the matrix. This resulted in dense plates that did not experience premature failure due to voids.

5.6 Summary

The inorganic matrix proved to reduce to the curing temperature to an optimum 400°C. An increase in the alumina content leads to greater thermal stability and higher flexural strengths. The various fiber reinforcement investigated prove to affect the mechanical properties of the composite material differently. The discrete fibers investigated provided reinforcement to the matrix and retained their mechanical properties at extreme temperatures within reasonable percentages. The high quality short ceramic fibers are twice as effective then the other reinforcement investigated.

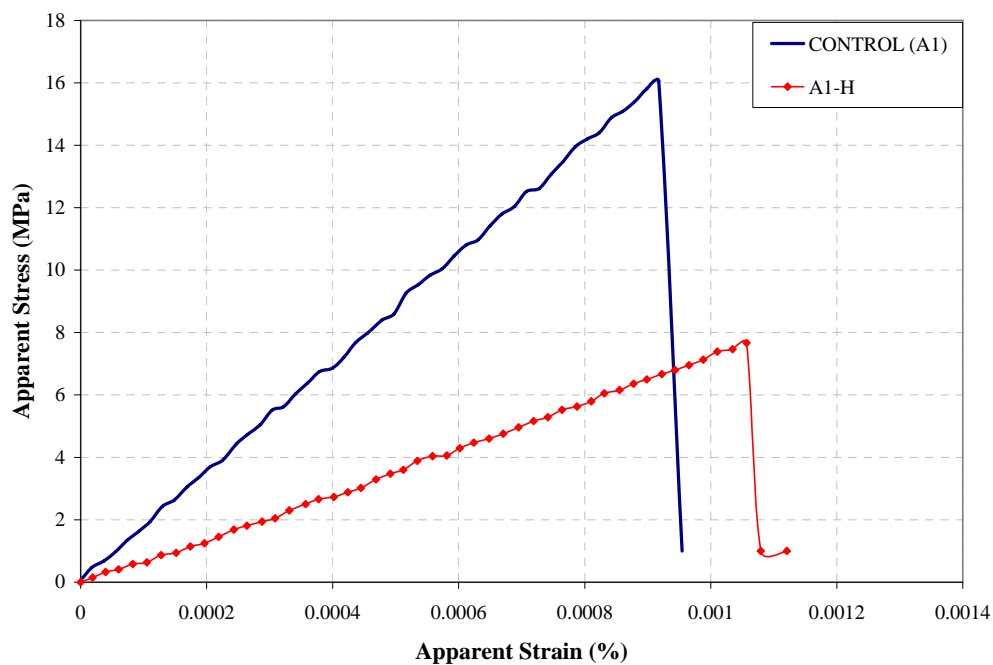


Figure 5. 1: Apparent Stress vs. Apparent Strain curve for 3-Ply Alumina fiber

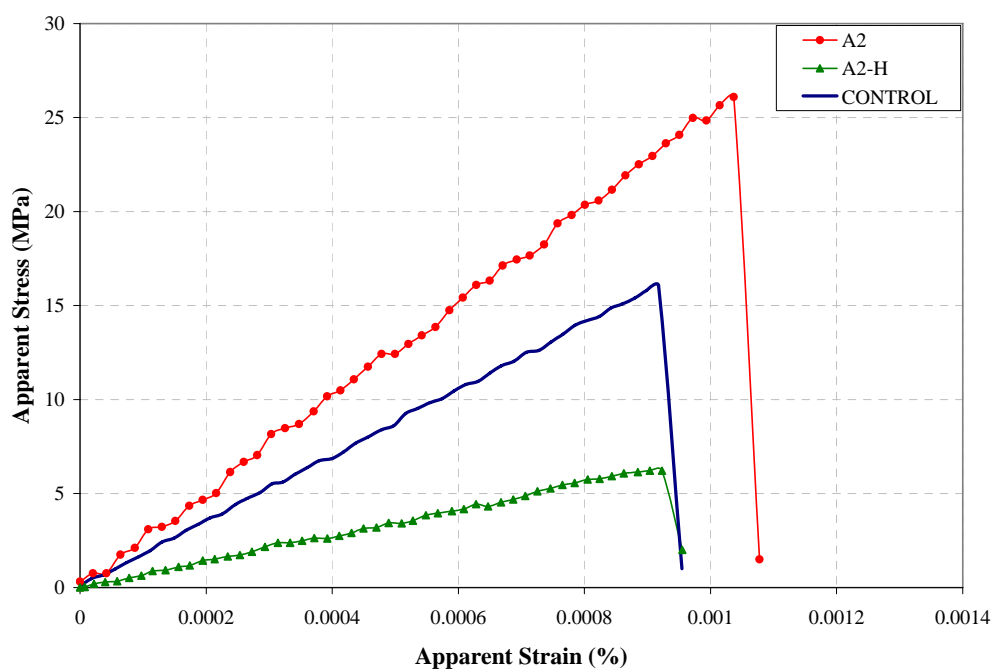


Figure 5. 2: Apparent Stress vs. Apparent Strain curve for 3-Ply Alumina fiber with hardener

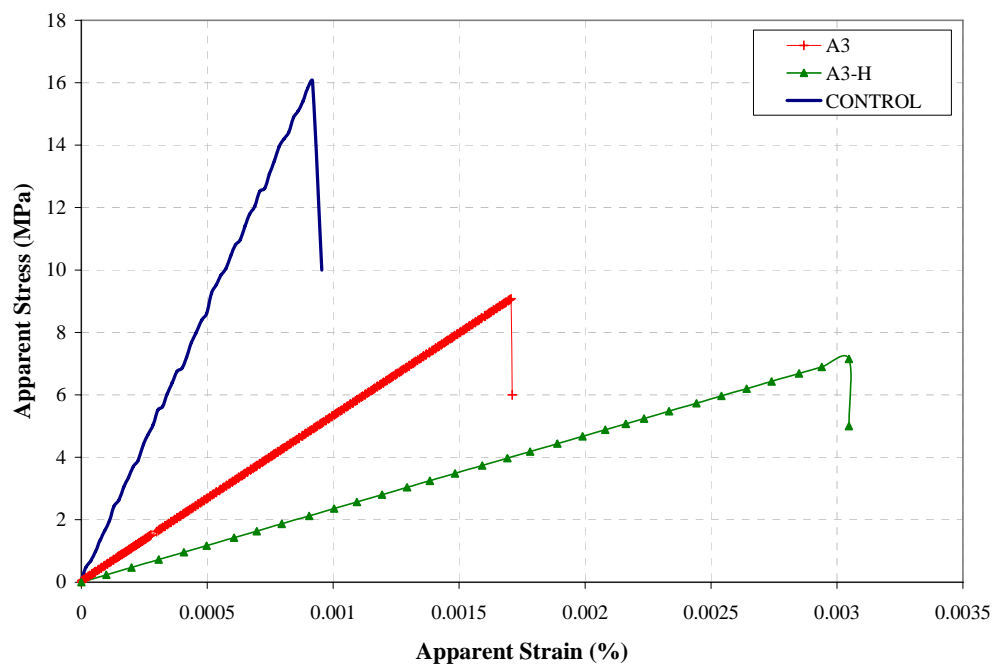


Figure 5. 3: Apparent Stress vs. Apparent Strain curve for 1-Ply Alumina fiber

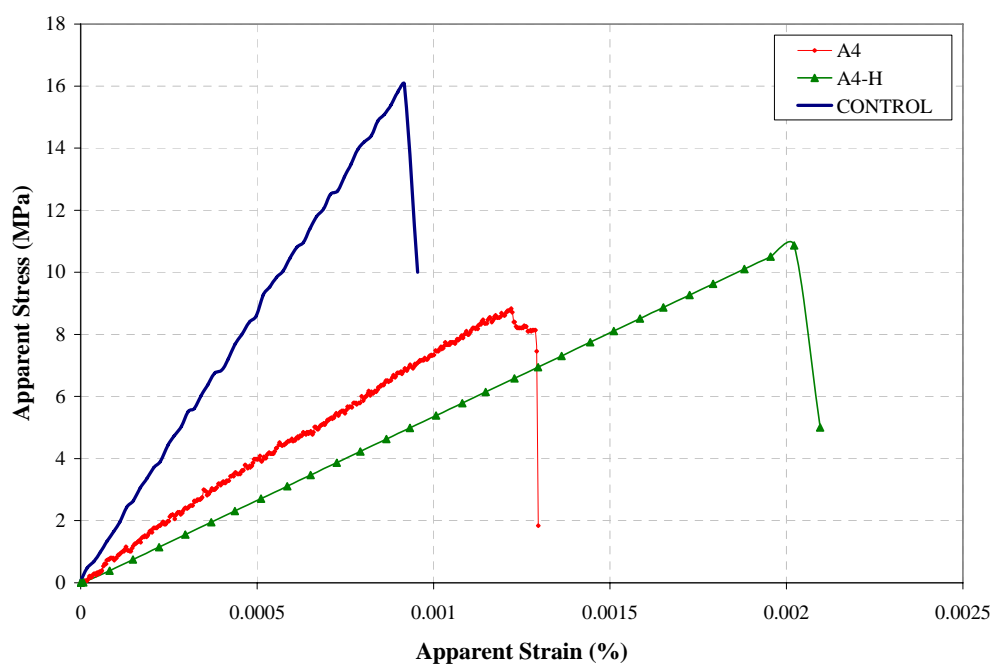


Figure 5. 4: Apparent Stress vs. Apparent Strain curve for 1-Ply Alumina fiber with hardener

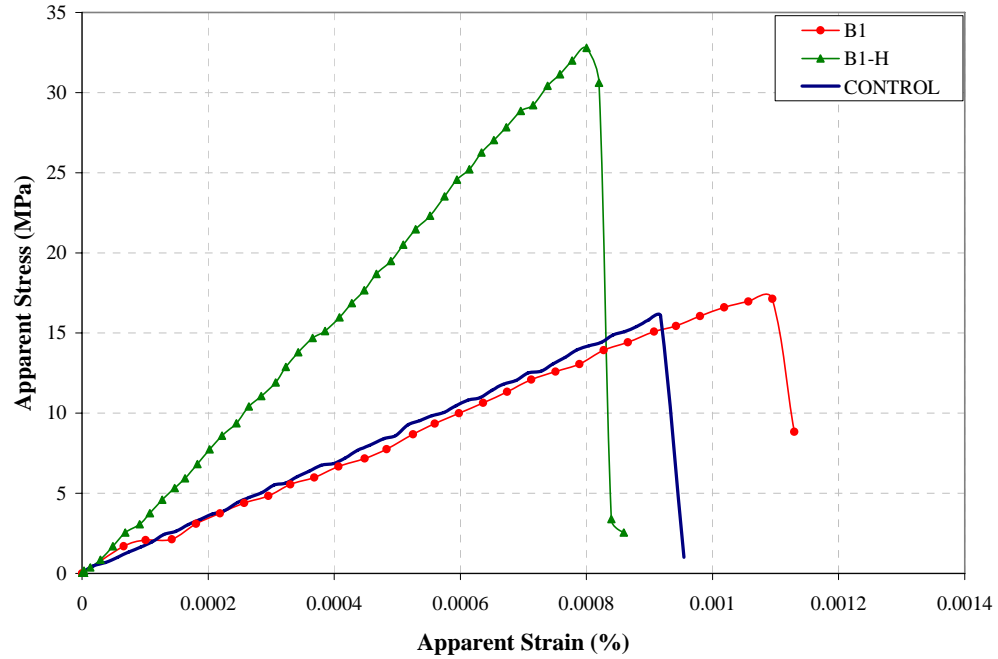


Figure 5. 5: Apparent Stress vs. Apparent Strain curve for Carbon fiber with 1:1 SiO₂/ Al₂O₃ ratio

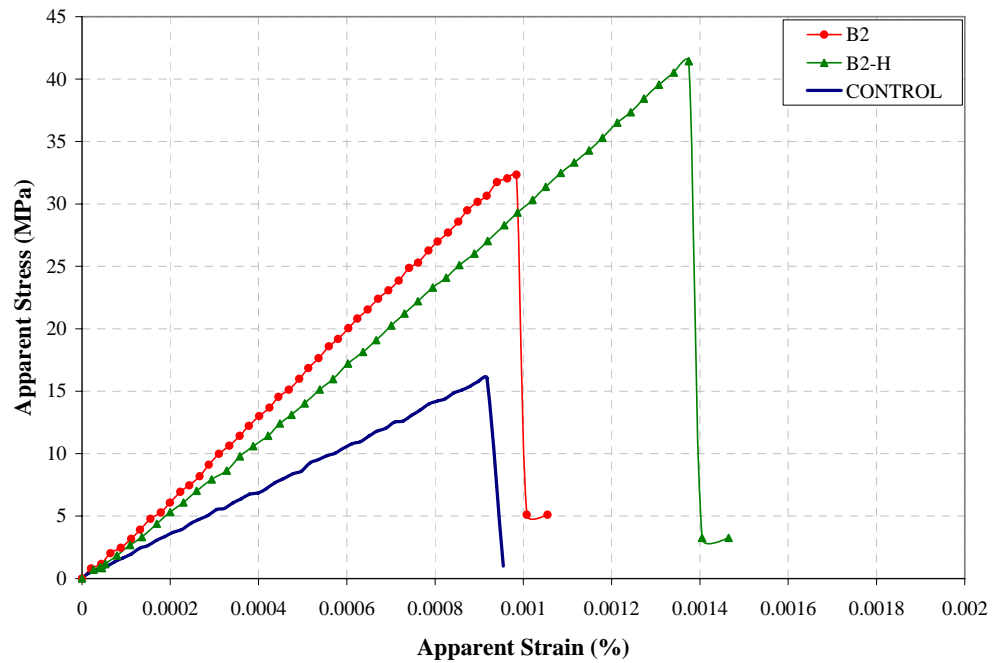


Figure 5. 6: Apparent Stress vs. Apparent Strain curve for Carbon fiber with 1:2 SiO₂/ Al₂O₃ ratio

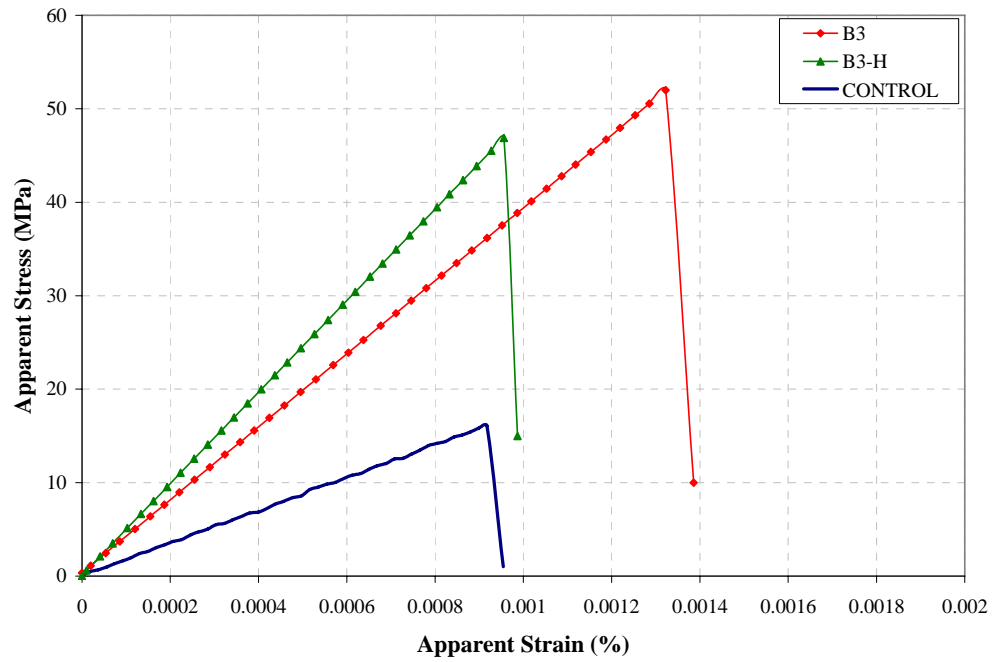


Figure 5. 7: Apparent Stress vs. Apparent Strain curve for Carbon fiber with 1:5 SiO₂/ Al₂O₃ ratio

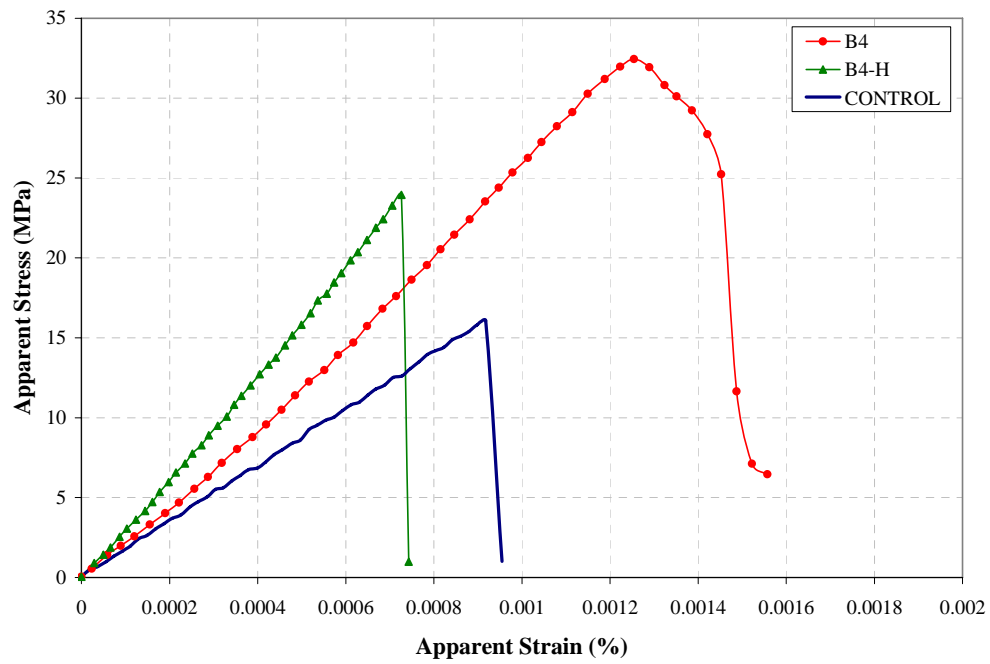


Figure 5. 8: Apparent Stress vs. Apparent Strain curve for Bulk Alumina & Carbon fiber with 1:1 SiO₂/ Al₂O₃ ratio

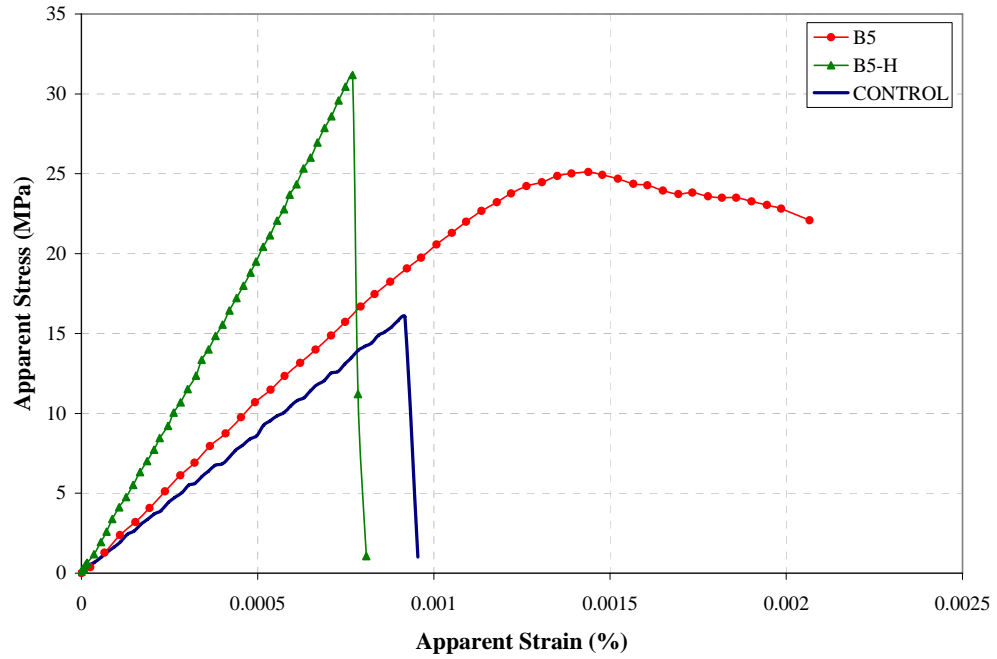


Figure 5. 9: Apparent Stress vs. Apparent Strain curve for Bulk Alumina & Carbon fiber with 1:2 $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio

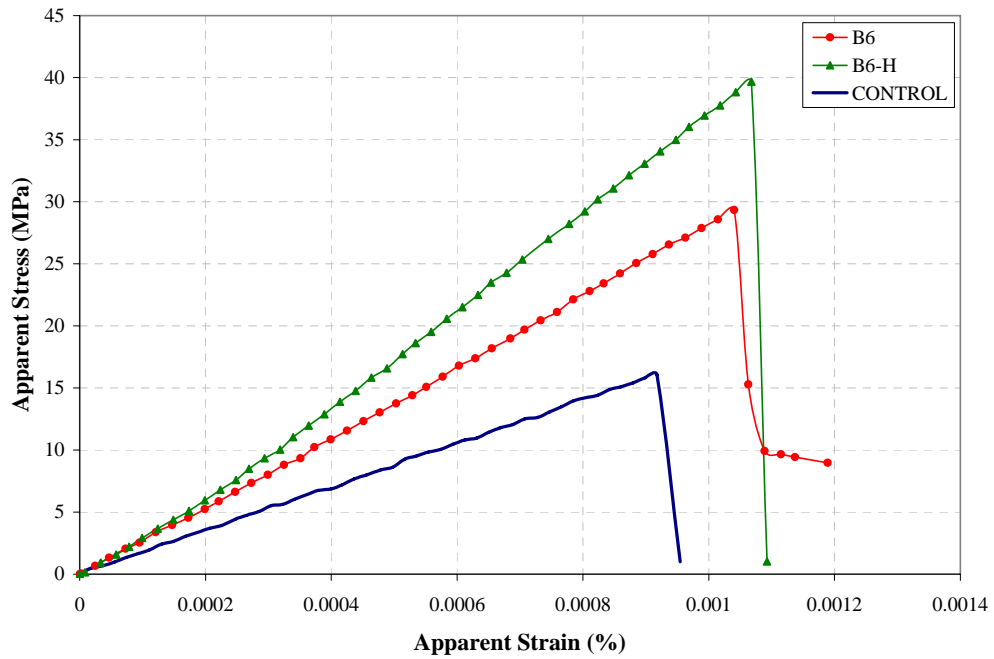


Figure 5. 10: Apparent Stress vs. Apparent Strain curve for Bulk Alumina & Carbon fiber with 1:5 $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio

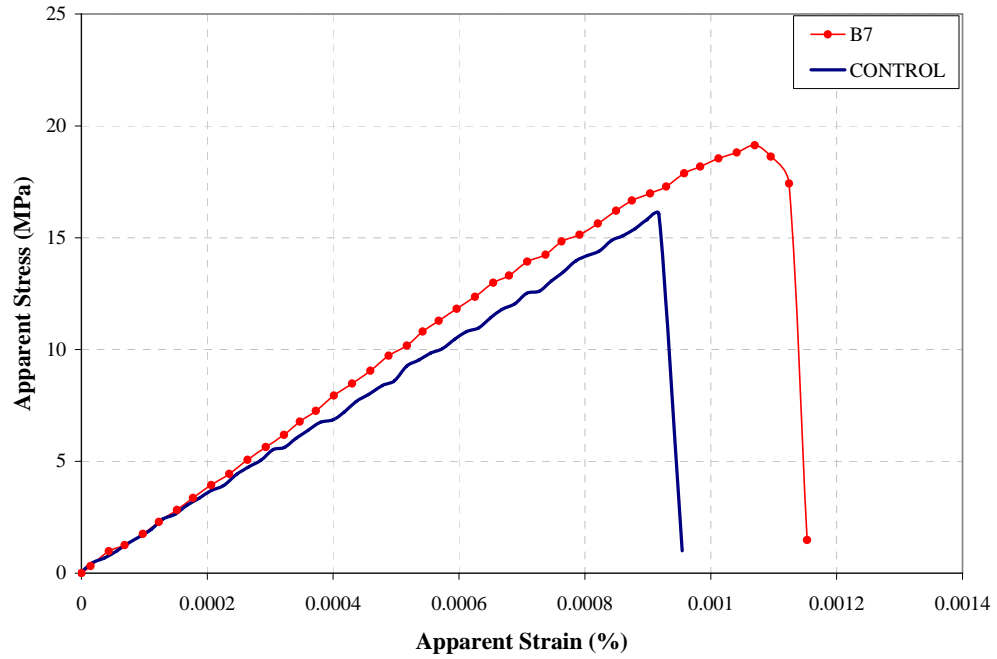


Figure 5.11: Apparent Stress vs. Apparent Strain curve for 4% Carbon volume fraction

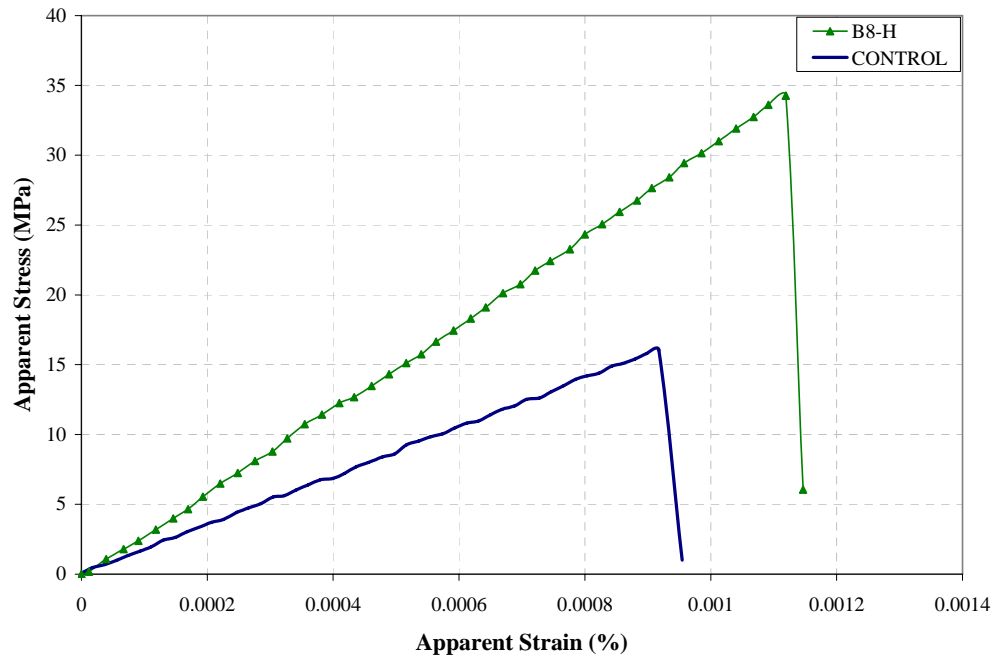


Figure 5.12: Apparent Stress vs. Apparent Strain curve for Bulk Alumina fibers

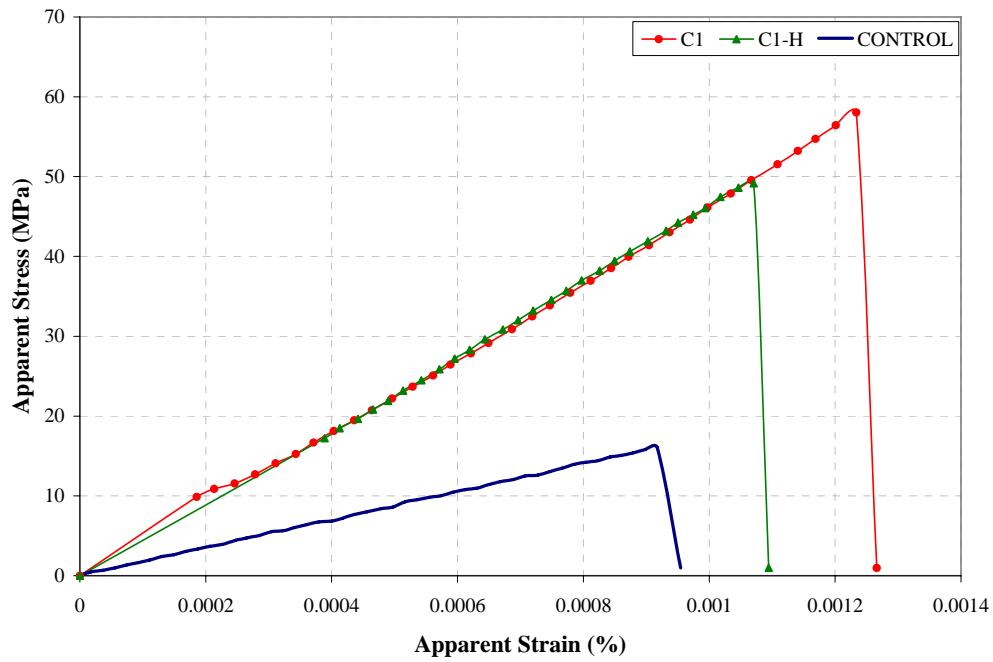


Figure 5. 13: Apparent Stress vs. Apparent Strain curve for Bulk Alumina fibers (2)

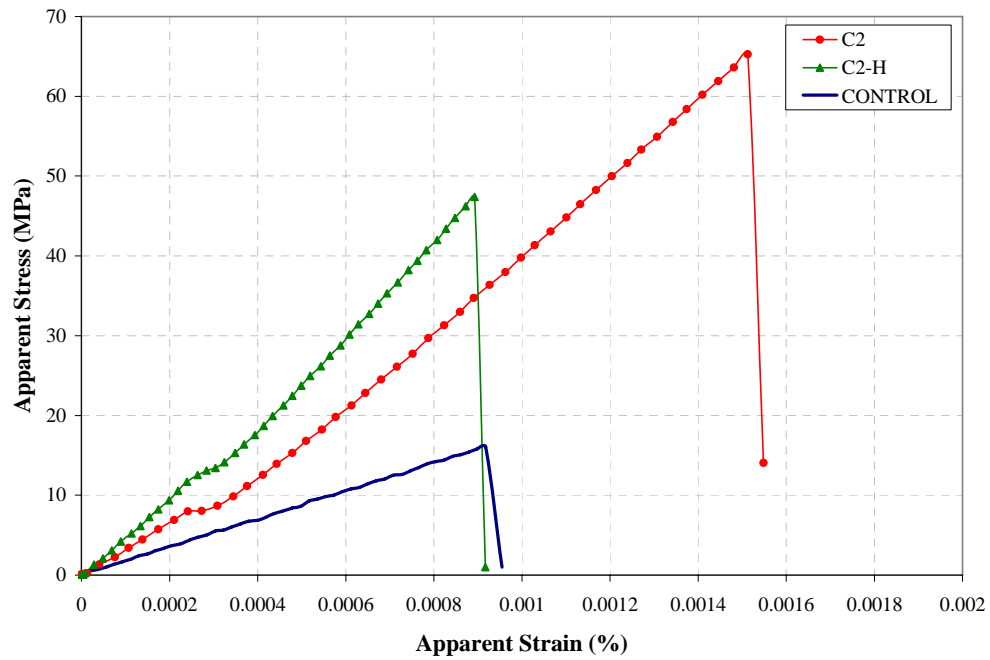


Figure 5. 14: Apparent Stress vs. Apparent Strain curve for Bulk Alumina fibers (3)

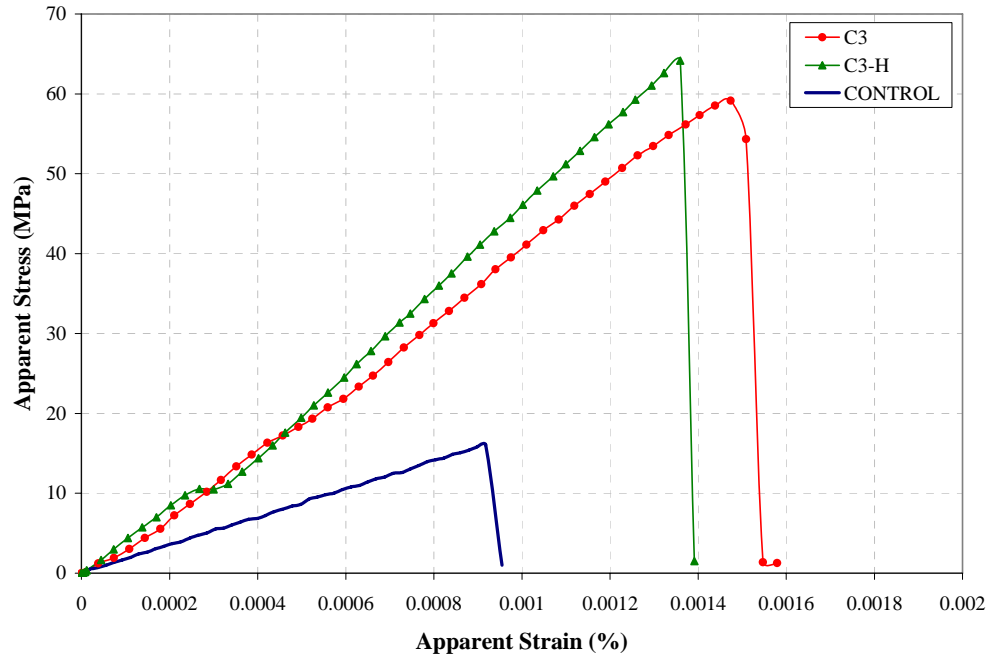


Figure 5. 15: Apparent Stress vs. Apparent Strain curve for Short Ceramic fibers

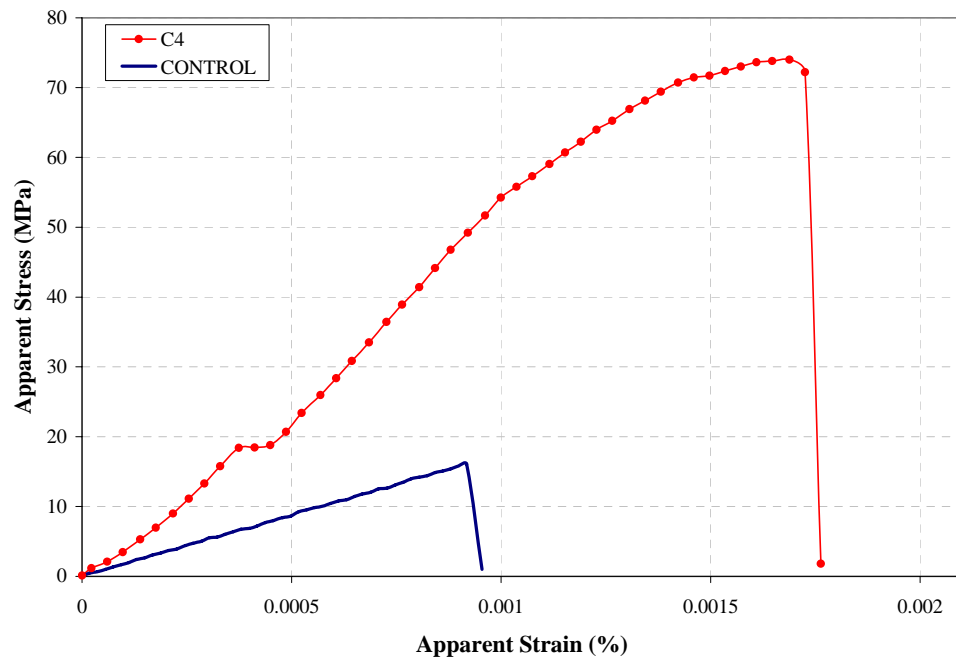


Figure 5. 16: Apparent Stress vs. Apparent Strain curve for Short Ceramic fibers (2)

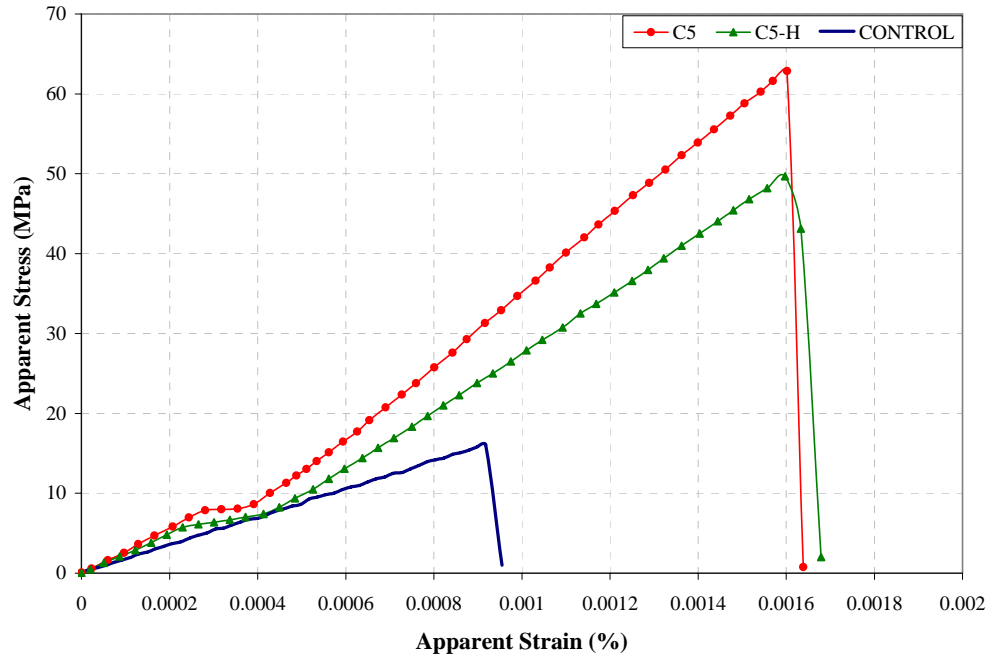


Figure 5. 17: Apparent Stress vs. Apparent Strain curve for Short Ceramic fibers (3)

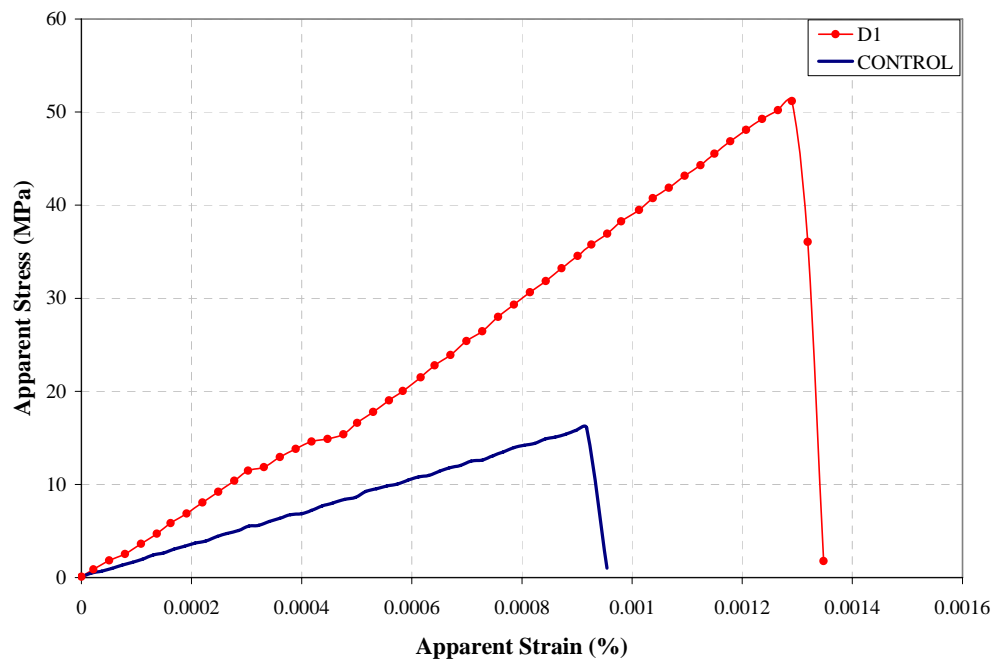


Figure 5. 18: Apparent Stress vs. Apparent Strain curve for 9.7% Bulk Alumina fiber volume fraction

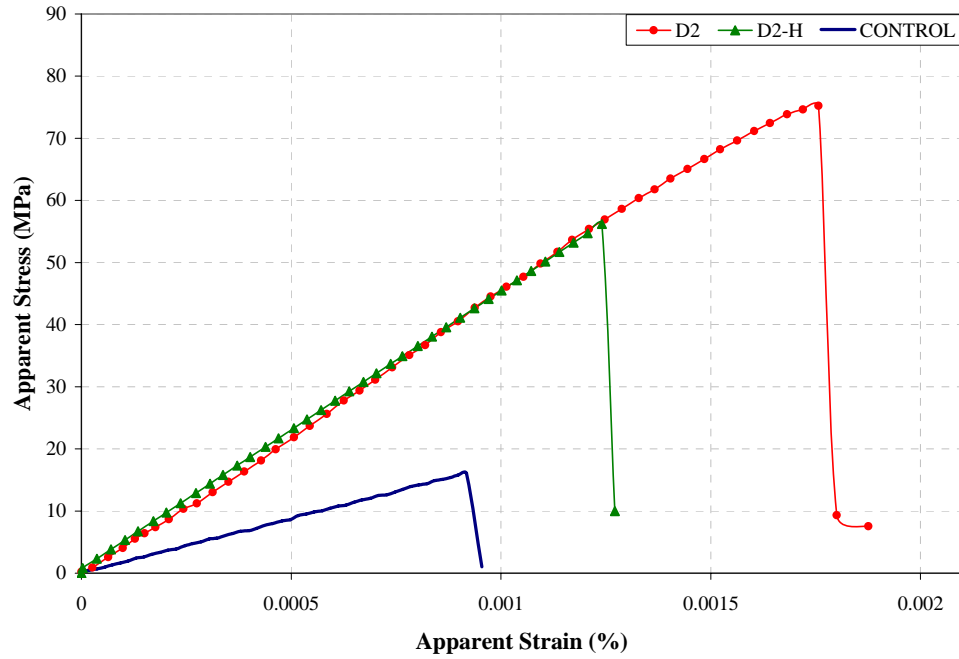


Figure 5. 19: Apparent Stress vs. Apparent Strain curve for 9.7% Short Ceramic fiber volume fraction

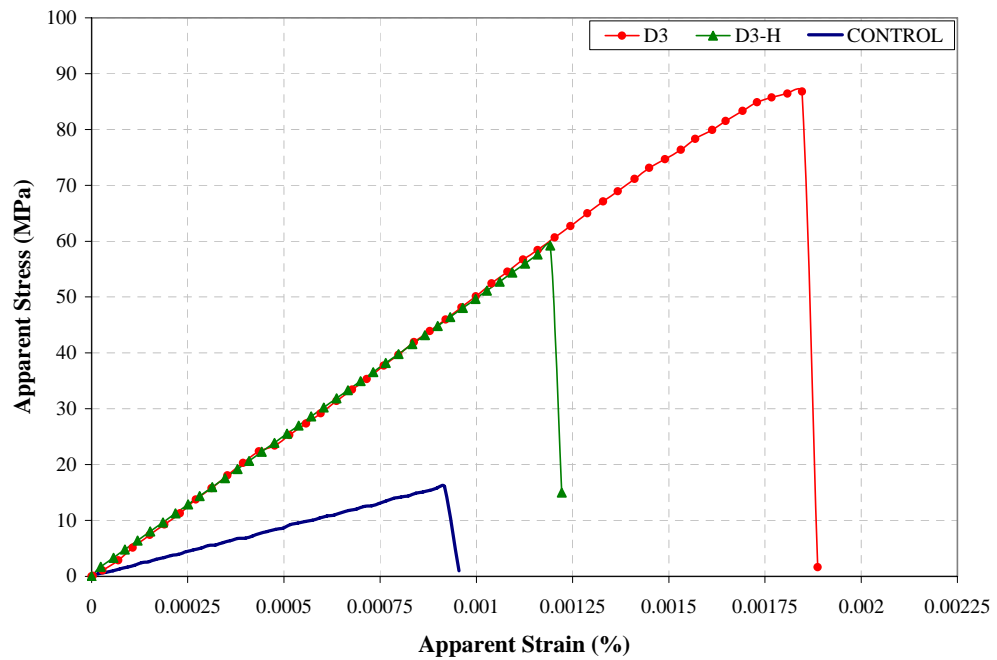


Figure 5. 20: Apparent Stress vs. Apparent Strain curve for 11% Short Ceramic fiber volume fraction

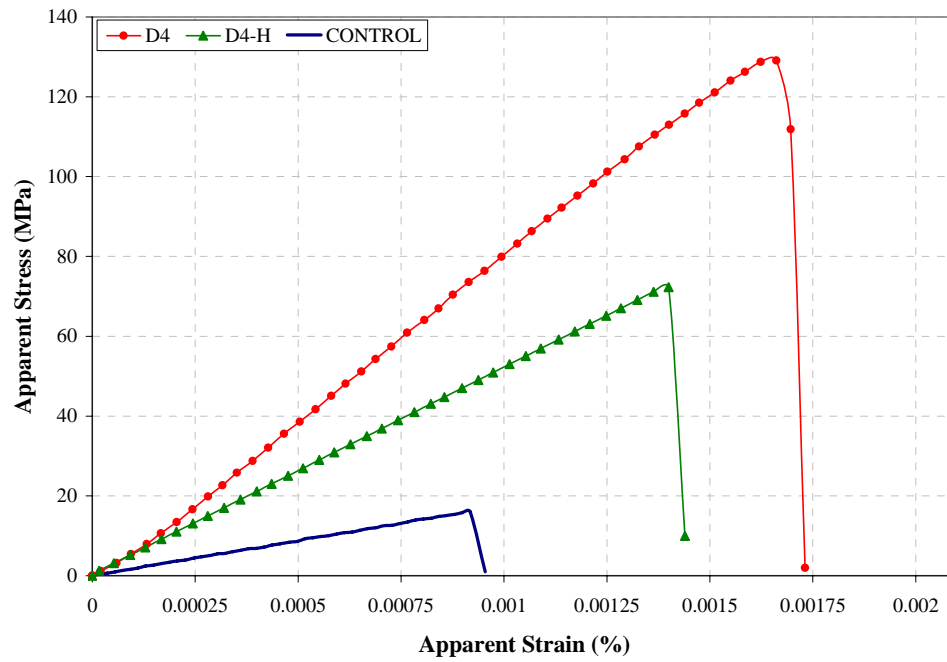


Figure 5. 21: Apparent Stress vs. Apparent Strain curve for 13% Short Ceramic fiber volume fraction

Table 5. 1: Summary of experimental results

| Sample ID | Density (kg/m ³) | Apparent | | E (GPa) | Fiber Type | Fiber V _f (Wt %) | Maximum Exposure Temperature (°C) |
|-----------|------------------------------|--------------|------------|---------|------------|-----------------------------|-----------------------------------|
| | | Stress (MPa) | Strain (%) | | | | |
| A1 | 1159 | 16 | 0.0918 | 19.0 | P | 20.0 | 200 |
| A1-H | 771 | 8 | 0.1057 | 7.0 | P | 20.0 | 1050 |
| A2 | 1717 | 26 | 0.1037 | 25.0 | P | 30.0 | 200 |
| A2-H | 934 | 6 | 0.0923 | 7.0 | P | 30.0 | 1050 |
| A3 | 1159 | 9 | 0.2536 | 4.0 | P* | 13.0 | 200 |
| A3-H | 765 | 7 | 0.4604 | 3.0 | P* | 13.0 | 1050 |
| A4 | 1586 | 9 | 0.1220 | 7.0 | P* | 13.0 | 200 |
| A4-H | 807 | 11 | 0.3132 | 4.0 | P* | 13.0 | 1050 |
| B1 | 2405 | 17 | 0.1095 | 16.5 | C | 4.5 | 400 |
| B1-H | 2451 | 33 | 0.0800 | 43.0 | C | 4.5 | 1050 |
| B2 | 2603 | 32 | 0.0984 | 35.0 | C | 4.5 | 400 |
| B2-H | 2084 | 41 | 0.1375 | 31.0 | C | 4.5 | 1050 |
| B3 | 2870 | 52 | 0.1322 | 39.0 | C | 4.5 | 400 |
| B3-H | 2820 | 47 | 0.0957 | 49.0 | C | 4.5 | 1050 |
| B4 | 2353 | 32 | 0.1254 | 29.0 | A/C | 4.4/1.1 | 400 |
| B4-H | 2033 | 22 | 0.0685 | 33.0 | A/C | 4.4/1.1 | 1050 |
| B5 | 2663 | 25 | 0.1438 | 21.5 | A/C | 4.4/1.1 | 400 |
| B5-H | 2325 | 31 | 0.0769 | 40.0 | A/C | 4.4/1.1 | 1050 |
| B6 | 2696 | 29 | 0.1041 | 27.0 | A/C | 4.4/1.1 | 400 |
| B6-H | 2553 | 40 | 0.1068 | 40.0 | A/C | 4.4/1.1 | 1050 |
| B7 | 2291 | 19 | 0.1070 | 20.0 | C | 2.2 | 400 |
| B8-H | 2451 | 34 | 0.1119 | 30.0 | A | 4.3 | 1050 |
| C1 | 2748 | 58 | 0.1234 | 48.0 | A | 4.4 | 500 |
| C1-H | 2667 | 49 | 0.1071 | 48.0 | A | 4.4 | 1050 |
| C2 | 2748 | 65 | 0.1513 | 49.0 | A | 4.5 | 500 |
| C2-H | 2618 | 47 | 0.0893 | 59.0 | A | 4.5 | 1050 |
| C3 | 2828 | 59 | 0.1474 | 43.0 | B | 6.5 | 200 |
| C3-H | 2711 | 64 | 0.1359 | 53.0 | B | 6.5 | 1050 |
| C4 | 2724 | 74 | 0.1688 | 67.0 | B | 8.5 | 200 |
| C5 | 2675 | 64 | 0.1403 | 52.0 | A | 6.4 | 200 |
| C5-H | 2538 | 50 | 0.1597 | 38.0 | A | 6.4 | 1050 |
| D1 | 2573 | 51 | 0.1290 | 41.0 | A | 9.7 | 200 |
| D2 | 2733 | 75 | 0.1757 | 47.0 | B | 9.7 | 200 |
| D2-H | 2675 | 56 | 0.1241 | 45.0 | B | 9.7 | 1050 |
| D3 | 2770 | 87 | 0.1846 | 50.0 | B | 11.0 | 200 |
| D3-H | 2823 | 59 | 0.1192 | 49.0 | B | 11.0 | 1050 |
| D4 | 2790 | 129 | 0.1661 | 85.0 | B | 13.0 | 200 |
| D4-H | 2781 | 72 | 0.1763 | 50.0 | B | 13.0 | 1050 |

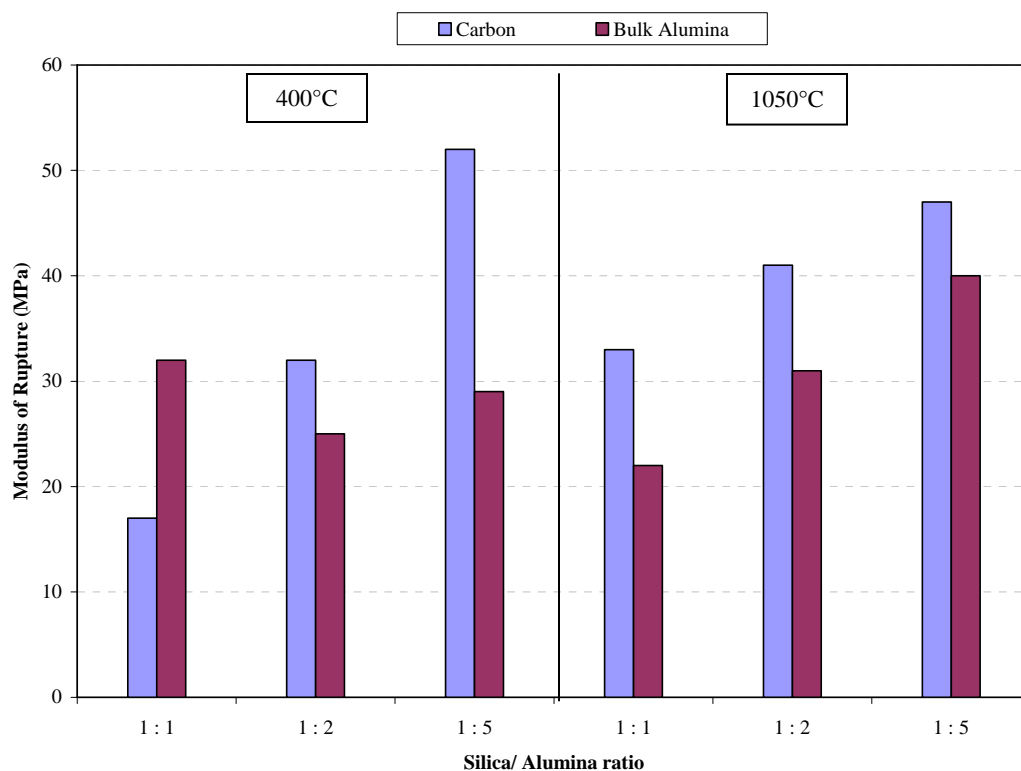


Figure 5. 22: Effect of Silica/Alumina ratio on Modulus of Rupture

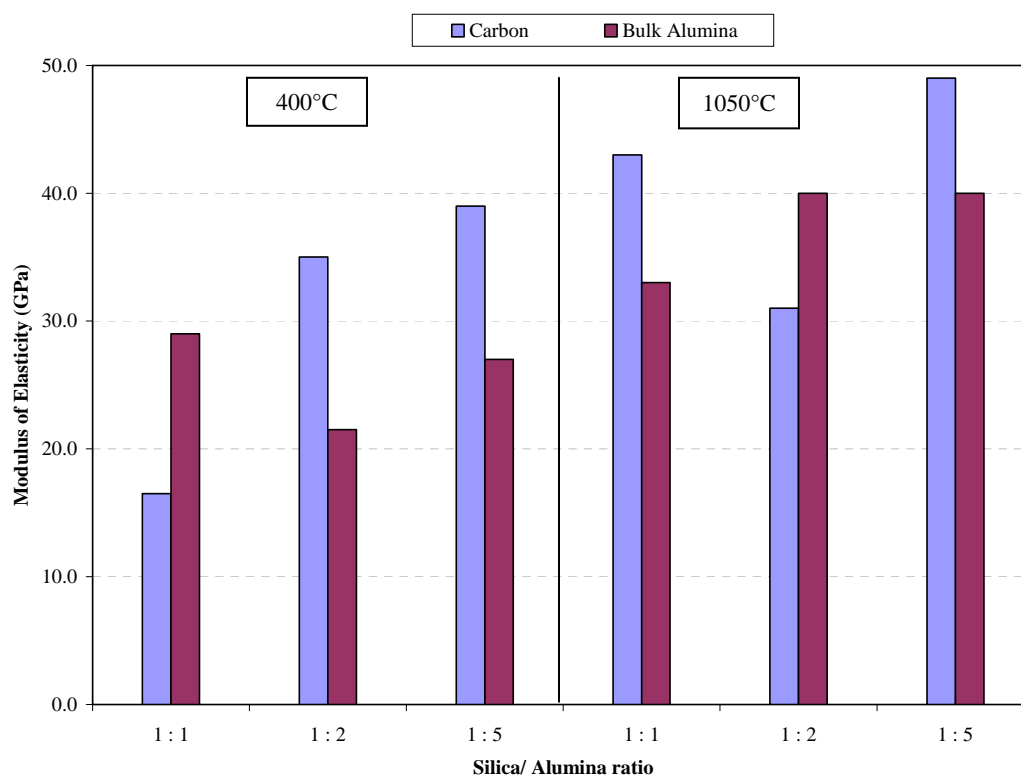


Figure 5. 23: Effect of Silica/Alumina ratio on Modulus of Elasticity

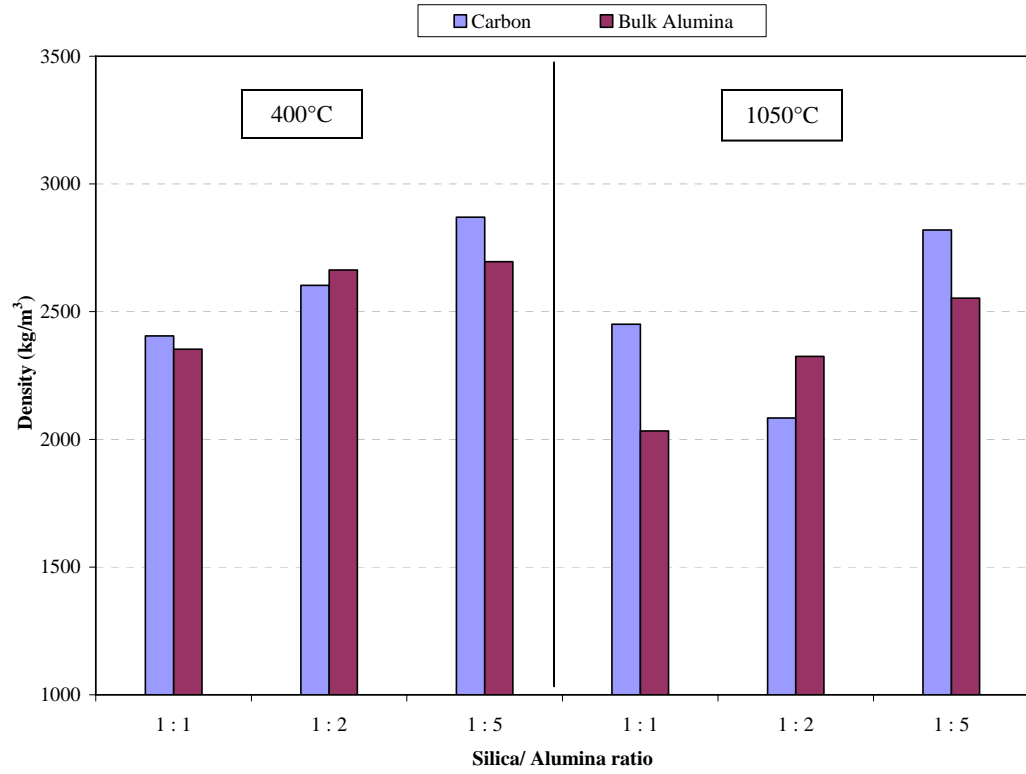


Figure 5. 24: Effect of Silica/Alumina ratio on Density

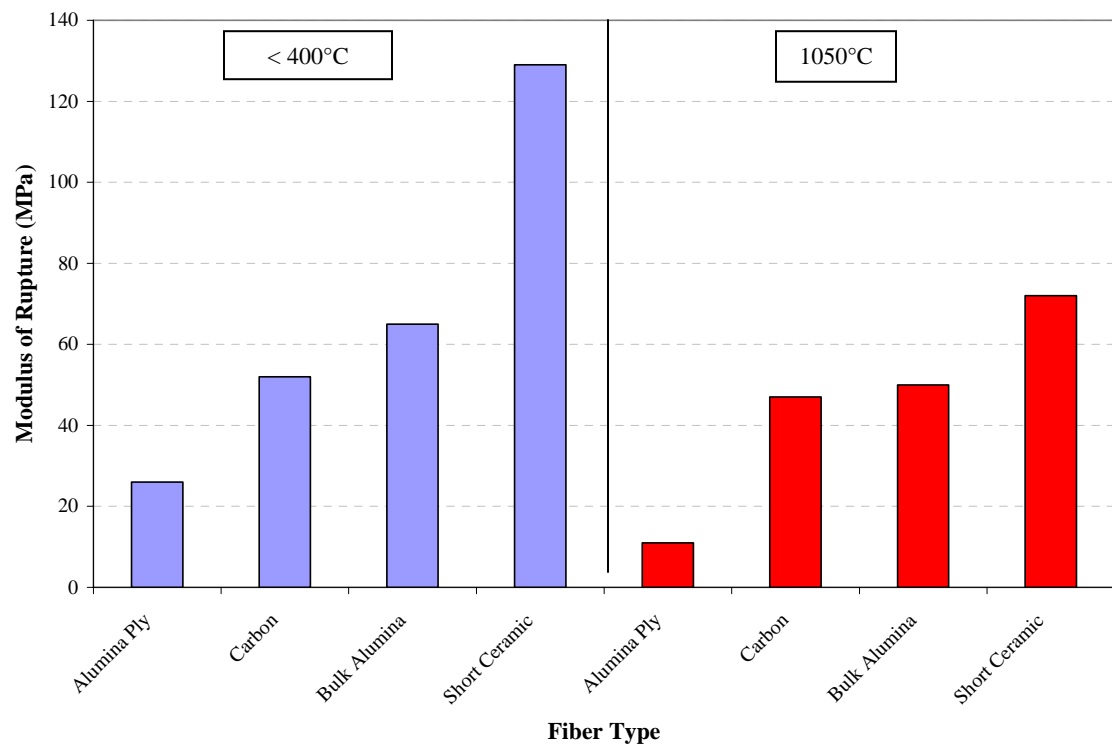


Figure 5. 25: Effect of Fiber Type on Modulus of Rupture

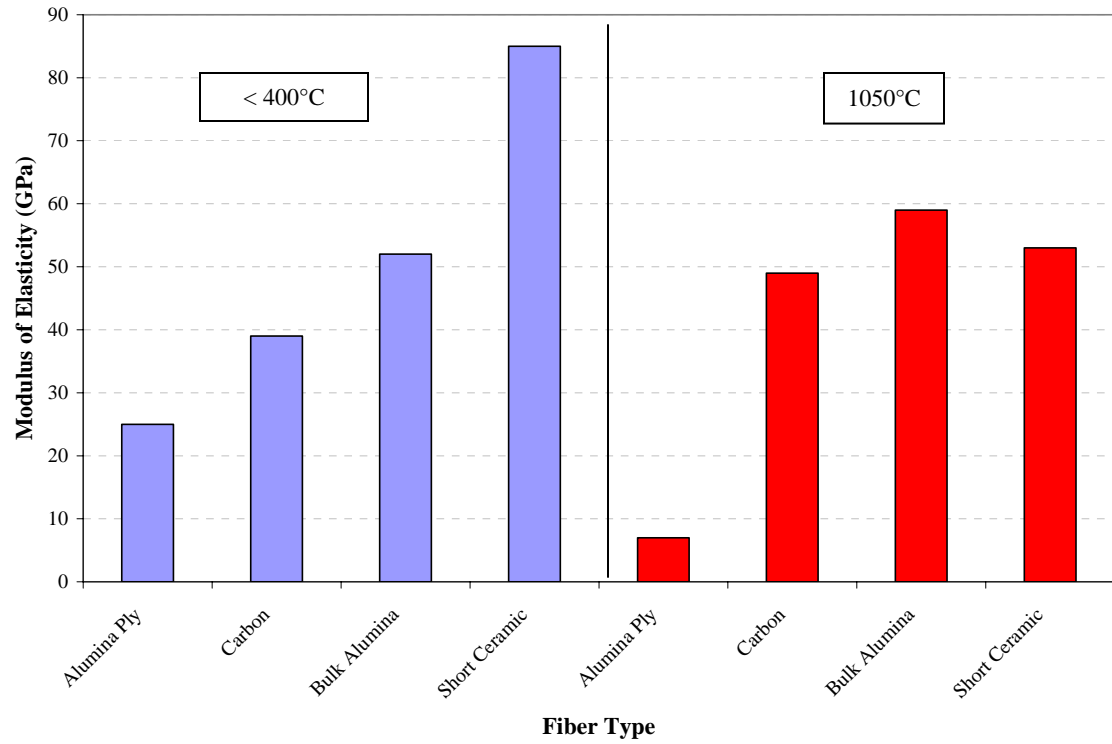


Figure 5. 26: Effect of Fiber Type on Modulus of Elasticity

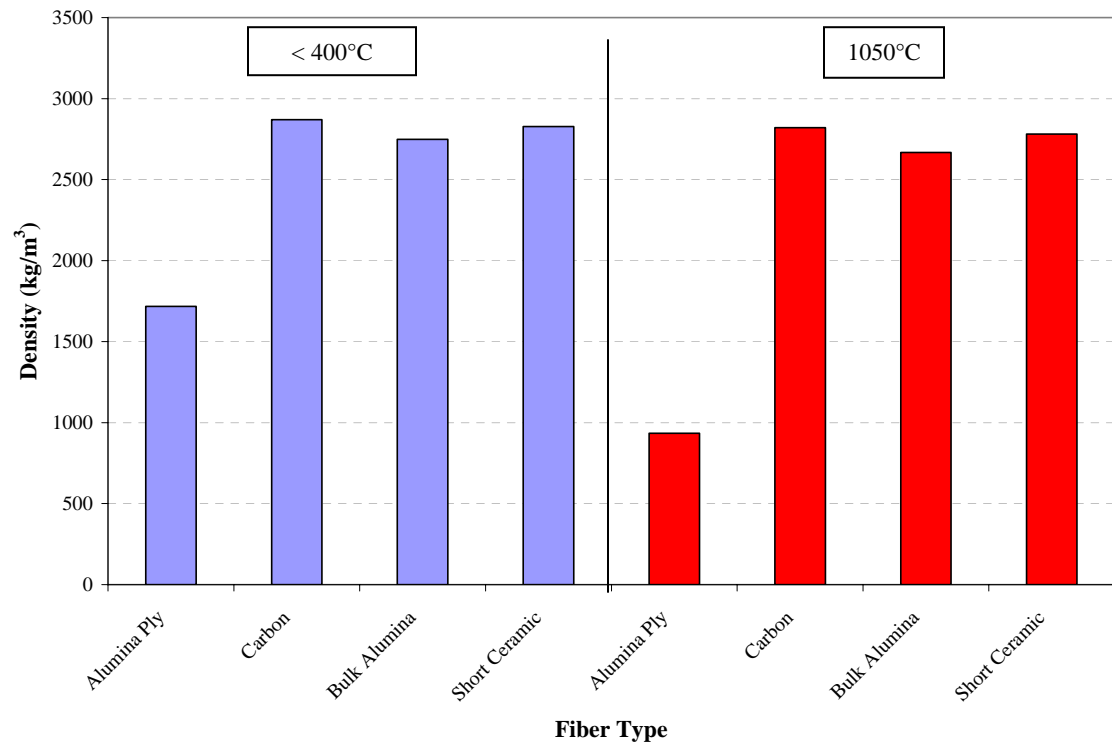


Figure 5. 27: Effect of Fiber Type on Density

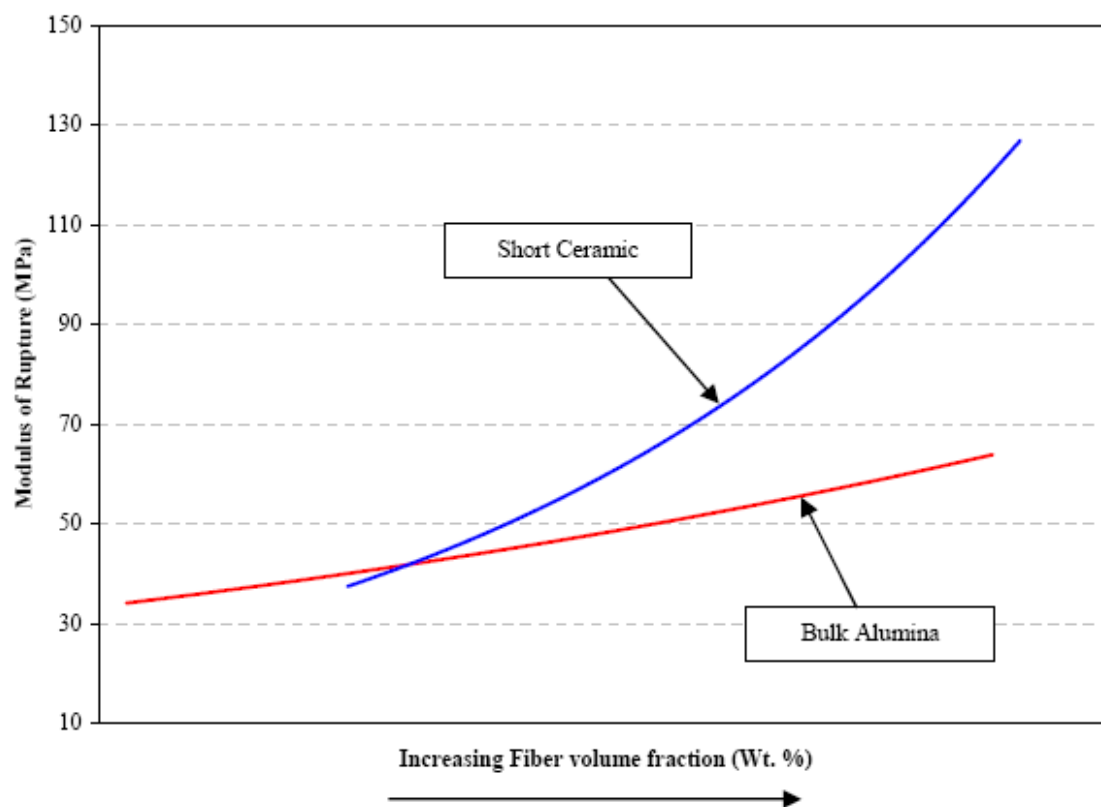


Figure 5. 28: Effect of Increasing Fiber volume fraction on Modulus of Rupture

CHAPTER 6. CONCLUSIONS

6.1 Conclusions

The results presented in the thesis focus on the use of an inorganic matrix composite to reduce the curing temperature and processing costs generally associated with typical high temperature composites. The following will highlight significant conclusions based on the results from experimental testing.

- The optimum curing temperature is 400°C for the inorganic matrix composites. This is a significant advantage compared to the 1200°C minimum curing temperature for typical high temperature composites.
- The low curing temperature makes it possible to produce high volume composites without the need for large processing equipment.
- The inorganic composites cure at low temperatures, but can perform at extreme temperatures beyond 1000°C.
- Typically, composites with higher densities experience higher flexural strengths.
- Increasing the exposure temperature decreases the density and flexural strength of IMCs
- An increase of alumina powder in the matrix results in a more thermally stable composite, as well as increased flexural strengths.
- A combination of hot-pressing and vacuum bagging fabrication techniques produces high quality composites with minimal impurities.

These composites have the least amount of air voids, highest densities and flexural strengths, as well as high product consistency.

- The stress-strain response of the coupons is linear elastic. In all of the tests the failure was brittle with no post peak resistance.
- Alumina ply laminates have the lowest flexural strength of 20 MPa at the cured temperature. At 1050°C the strength reduces to 11 MPa.
- High modulus carbon fiber IMCs have a flexural strength of 52 MPa and elastic modulus of 39 GPa. At 1050°C the flexural strength reduces by 10% while the modulus of elasticity increases by 25%.
- Bulk alumina IMCs have a flexural strength of 65 and 50 MPa at 400 and 1050°C, respectively. The elastic modulus ranges between 50 and 60GPa at these temperatures.
- The flexural strength of inorganic matrices reinforced with high quality short ceramic fibers is 129 and 72 MPa at 400 and 1050°C, respectively. The modulus of elasticity at these temperatures is 85 and 53 GPa.

6.2 Suggestions for Further Research

The following are recommendations for further research in the development of low temperature curing inorganic matrix composites.

- The long term mechanical behavior of the inorganic matrix needs to be studied. This will help understand if the composite will loss or gain strength as time progresses.

- The use of high quality ceramic fiber woven fabrics or mats for laminate processing reinforcement. These fabrics have high temperature capabilities as well as direct fiber alignment with the applied load.
- A combination of discrete and continuous fibers to reinforce the inorganic matrix
- The use of inorganic matrix composites as structural components in civil engineering infrastructure

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