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The Effects of Porosity on the Friction And Wear of Carbon-Carbon Composite Aircraft Brakes

By

Daniel E. E. Hayes, B.S., M.S.

A Doctoral Thesis

Submitted in partial fulfillment of the requirements for the award of the degree of Doctor of Philosophy of Loughborough University.

March 2002

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Publications and Presentation from this Work

Abstract

Six sets of subscale carbon-carbon composite rotors and stators for aircraft brakes were manufactured to provide friction and wear test samples at six different densities. The friction and wear tests used energies to represent the service energy of the Boeing 767 aircraft. A functional relationship between friction coefficient and porosity/density was made. This relationship was used to minimise manufacturing cost by providing the minimum densification of the carbon-carbon composite brake discs needed to meet design friction coefficient.

Weight and specific gravity measurements, optic microscopic image analysis, mercury porosimetry, and He pycnometry were used to measure the surface and bulk porosity/density of the brake discs before testing. Post-test porosity was measured by scanning electron microscope/quantitative image analysis to determine the area percentages of friction film, wear debris particulate, and empty pores on the surface after the test. This analysis showed that the ratio of percentage area of film coverage is equal to the ratio of the friction coefficients of the high and low porosity discs. For the highest and lowest porosity discs used in this study the ratios were both nearly equal to a value of 2.1.

Optical, SEM, and TEM microscopes, photo correlation spectography, X-Ray diffraction, and micro Raman spectroscopy were used to analyse the morphology of the worn friction surfaces. Theses studies indicated that for low porosity surfaces, where the wear debris has no place to leave the surface, the debris is shear worked into a very fine amorphous and non-ordered crystalline friction film. This fine film would increase the real area of friction surface contact and thus increase the friction coefficient. It was possible to relate the friction coefficient directly to the real area of surface contact. There are several features of the worn surface morphology discussed in this study. Micrograph collages were made for the same disc areas at 70, 120, 170, and 220 stops to analyse the changes in the surface during testing.
Acknowledgments

The author wishes to express sincere appreciation to Professors Patrick, and Brooks, and Doctor Walker for their role as my faculty supervisors. I wish to express special appreciation to John Patrick for his patience and understanding and his boundless knowledge of carbon. I wish to thank Neil Murdie and Frank Dillion for acting as my proxy advisors in the USA. My thanks to Su Chen Lui and her team at our corporate research centre for their help whenever I got stuck in need of a test I couldn’t do myself. I give my deepest appreciation to Catherine, my spouse, my best friend and companion for her support and love that has always inspired me to do anything that challenged me.
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1.1 The commercial impetus for research on the friction and wear of carbon composites

The aircraft brake manufacturing industry agrees that the wear of the carbon based materials used for brake discs for large aircraft is far greater in service than predicted by test data used in the development of these brake systems [1,2]. There is also general agreement that the reason for this increased brake wear is that the number of aircraft brake applications during aircraft taxi far exceed the number assumed in the criteria applied during the design and test stages of the brake development [3]. This mismatch between the predicted wear in the laboratory and the actual wear seen by airlines in the field could:

1.) Cost the brake manufacturers hundreds of millions of dollars in warranty claims

2.) Reduce the manufacturing capacity significantly, due to the unforeseen increase in frequency of brake disc replacement. For some models, such as the Boeing 777 the production capacity is reduced by as much as fifty percent.

Three possible solutions to premature wear exist. They are as follows:

1) Introduce a new material with a better wear rate.
2) Improve the wear rate of the current materials.
3) Improve the manufacturing cost of the brake discs so that the wear rate would have less impact on profit.

Allied Signal (now Honeywell) Aircraft Landing Systems is the largest producer of aircraft brakes and thus could face the largest reduction in profits and customer satisfaction. This company ran a propriety simulation to assess the risk/reward of the above options [4]. It is the conclusions of that simulation that spawned this research and indicated that pursuit of the above options 2 and 3
would yield the least risk and most value. The improvement of the current materials and/or the improvement of the manufacturing cost would most likely be the least costly options since the current manufacturing equipment could still be used; the in service overhaul and maintenance procedures would remain similar and the customers, designers, and production personnel would not require redesign or extensive retraining.

1.2 Summary of Research Objectives

The overall objective of this study is to understand how the current carbon based brake material wears and suggest a more economical manufacturing procedure for current materials. A cost model for the most expensive segment of the manufacturing process for carbon-carbon brake discs will be developed and minimised. As will be shown later the manufacturing process time can be reduced, however this results in an increase in the porosity of the carbon-carbon material. The effect of porosity on the wear and friction will be evaluated. Moreover, the friction film (wear debris) produced during braking will be analysed to determine its contribution to the wear phenomenon in braking. Dynamometer brake tests on sample materials will be carried out at typical aircraft service energies and the surface wear debris collected to determine its morphology as a function of manufactured brake disc porosity.

1.3 Review of Research Approach and Thesis Organisation

1.3.1 The Approach

The approach to understanding the effects of porosity on friction and wear will include a cost model. The model will be discussed in detail in chapter 4. Suffice it to state now that the reduction in cost of the current manufacturing conditions carries with it an increase in the material porosity. Thus, it is essential to understand the effects of material porosity on not just the wear, but also on all the important design properties for aircraft brakes such as friction performance.
and mechanical strength. The approach used to address this task was to produce three sets of subscale brake discs with different porosity values by varying the process conditions. These discs were then tested using a dynamometer at energies equivalent to the Boeing 767 service landing condition. The relationships between porosity and friction and wear were then established from the data. Finally, a characterisation of the wear film and wear debris produced during the wear process will be necessary to develop an understanding of these relationships and will be accomplished in this study.

1.3.2 The Layout of this Study

The layout of this study is as follows:

Chapter 2 provides a background for carbon materials and the methods to characterise carbons.

Chapter 3 provides a brief explanation of carbon-carbon composites, their manufacture, their properties and their application in aircraft brakes. These are followed by an extensive literature review of friction and wear models.

Chapter 4 provides the objectives, the cost model, and the gaps in the literature to be filled by this study. All the objectives of this study are listed in bold italics at the end of chapter 4.

Chapter 5 provides the experimental procedures and techniques used in this study.

Chapter 6 presents the results of the experimental procedures and characterisations presented in chapter 5.

Chapter 7 contains the discussion of results.

Chapter 8 contains the conclusions and recommendations.
Chapter 2 Structure and Formation of Carbons

2.1 Introduction

The following introduction to carbon is taken primarily from Introduction to Carbon Science, [5]. The element carbon has an atomic number 6 and an atomic weight of 12.011 amu. Its ground state electron configuration is 1s² 2s² 2p². According to hybridisation theory it is energetically favorable on bonding to promote a 2s electron to the empty 2pz orbital, [6]

\[ 1s^2 2s^2 2p^2 \rightarrow 1s^2 2s^1 3p^x 3p^y 3p^z \]  

(2.1)

The resulting electron configuration affords carbon the opportunity to form three possible hybrid orbitals, i.e. four sp³, three sp² and two sp¹ orbitals. These hybrid orbitals form the covalent (σ) bonding of carbon in all its forms. An sp³ hybridised carbon atom has four bonding orbitals at a 109.5° bond angle (e.g. CH₄). An sp² hybridised carbon atom has three in a 2-dimensional plane at 120° bond angles; the remaining p-electron (3pₓ) forms a π-bond with adjacent unpaired p-electrons (e.g. C₂H₄). An sp¹ hybridised carbon atom has two bonding orbitals at 180° bond angles, the remaining two p-electrons (3pₓ, 3pᵧ) form two π-bonds with adjacent unpaired p-electrons, e.g. C₂H₂. The ability of a carbon atom to form σ and π bonds with itself (i.e. catenation) and other atoms (e.g. hydrogen, nitrogen, oxygen, etc.) results in the myriad of compounds that are classified in one branch of the chemical sciences, i.e. organic chemistry. The bonding versatility of carbon is apparent from its two main allotropes, diamond and graphite. Additionally two other carbon allotropes are recognised, carbyne and most recently fullerenes.

With the exception of sp³ diamond, most forms of carbon utilised or generated in industrial applications are based upon the sp² form of carbon i.e.
graphite. Consequently it is this form of carbon that will be emphasised in the following review of the structure, origin and properties of carbons.

2.2 Crystallographic Structure of Carbon Allotropes: Graphite, and Diamond.

The element carbon exists in two allotropic forms, including diamond and graphite. Standard definitions accepted by the International Union of Pure and Applied Chemistry (IUPAC) exists and is given in Introduction to Carbon Technologies [7]. The following definitions are taken from that reference.

Diamond is an allotropic form of the element carbon with cubic structure.

Graphite is an allotropic form of the element carbon consisting of layers of hexagonally arranged carbon atoms in a planar condensed ring system (GRAPHENE LAYERS).

Apart from diamond, all other forms of carbon such as charcoal, coke, soot, carbon black, carbon fibre, CVD matrixes and even the new fullerenes are distorted versions of the graphite form [8]. In this study primary emphasis will be only on the hexagonal or graphite crystalline forms.

To understand carbon in the solid form, it is necessary to understand the structure of graphite, both pure hexagonal and perturbations of pure hexagonal. Graphite consists of stacked layers of hexagonally arranged carbon atoms in a planer trigonal condensed ring system (i.e. graphene layers.). Each carbon atom is sp²-σ-bonded to three other carbon atoms and π-bonded via delocalised 2pz electrons. The delocalised π-bonds are stacked parallel principally in an ABAB stacking sequence. This arrangement results in a hexagonal unit cell, Figure 2.1 [5].
The carbon atoms are arranged in basal layers and are closely packed hexagonally in each layer. The atomic spacing of the basal plane is 0.14nm. The bond in the basal plane is covalent with strength of 522 KJ mol\(^{-1}\).

![Hexagonal Unit Cell](image.png)

**Figure 2.1 Graphite Structure**

The low bond strength (17KJ mol\(^{-1}\)) between the layers is attributable to Van der Waals force of attraction [9,10]. The stacking arrangement shown in Figure 2.1 is normally ABAB, but because the interplanar Van der Waals force is low, the energy required to shear perpendicular to the c-axis is relatively low. This property of graphite allows an alternate stacking sequence (ABCABCA) giving rise to the rhombohedral cell also depicted in Figure 2.1. This form of graphite constitutes up to 30% of natural graphite but is usually absent from synthetic graphite [11]. It is thermodynamically unstable and at temperatures greater than 1700K is converted to the stable (ABABA) form. The ABCABC stacking sequence can be induced by grinding single crystal graphite [12]. The susceptibility of basal plane shear is utilised when graphite is used as a lubricant and on a friction material due to the generation of friction film. Graphite (synthetic and single crystal) always contains crystallographic imperfections such as dislocations, stacking faults, voids and interstitial defects [10].
Not all graphitic forms of carbon exhibit a perfect graphite lattice. Most carbon materials possess structures that are somewhere between isotropic (non-graphitizable) carbon and single crystal graphite. Figure 2.2 is a schematic diagram, from [5] that shows the changes in the carbon structure that takes place during the transformation of a graphitizable carbon to graphite. This transformation occurs by heat-treating the carbon to temperatures between 1000°C and 3000°C in an inert atmosphere, which results in the alignment and enlargement of the crystallites through the annealing of defects and the loss of impurities such as N, O, S, or hydrogen.

Figure 2.2. Schematic Diagram of Various Forms Carbon from Graphitizable Non-Graphitic Form to Graphitic Carbon [5]
2.3 Standard Nomenclature for Carbon Materials

As stated above, the structure of all industrial carbons, with the exception of diamond, is based upon the graphite crystal lattice. Consequently, most industrial carbons are classified either as graphitic or non-graphitic depending on how their structure approximates to that of single crystal graphite. The established nomenclature standard adapted by the IUPAC for carbon materials is also used in the following definitions [13,14].

**Carbon** is all varieties of substances that consist of elemental carbon in the allotropic form of graphite irrespective of the presence of structural defects

**Non-graphitic Carbon** is all variations of solids consisting mainly of elemental carbon with two-dimensional long-range order of the carbon atoms in planar hexagonal networks, but without any measurable crystallographic order in the third direction (c-direction) apart from parallel stacking.

Non-graphitic carbon can be further classified into graphitizable carbons and non-graphitizable carbons.

**Graphitizable Carbons** is a non-graphitic carbon that, upon graphitization heat-treatment converts into graphitic carbon.

**Non-graphitizable Carbon** is a non-graphitic carbon that cannot be transformed into graphitic carbon solely by the application of heat-treatment up to 3300K under atmospheric or low pressures.

The extent to which graphitization under heat treatment can happen depends on the starting structure of the heat treated carbon and has led to the commonly used terms anisotropic and isotropic carbon. The structure of polished carbons when viewed through a reflected polarised light microscope is described by one of these two terms.
Anisotropic Carbon is a graphitizable carbon whose optical properties is a function of its crystallographic direction, i.e., it is made up from discrete isochromatic units depending on their crystallographic orientation.

Isotropic Carbon is a non-graphitizable carbon whose optical properties is not a function of its crystallographic direction, it is monochromatic, i.e., purple when viewed through a reflected polarised light microscope.
3 Chapter 3 Carbon Fibre Reinforced Carbon Composites

3.1 Introduction

New composite materials that are typically light in weight, strong, stiff and suitable for structural applications are referred as advanced composites or "engineered materials". These composites are typically categorised according to their matrix. Some examples are metal matrix composites (MMC), polymeric matrix composites (PMC), and ceramic matrix composites (CMC). There is a special composite type called fibre reinforced composites (FRC). Fibre reinforced composites are generally categorised by stating their reinforcing component first followed by their matrix component (i.e., carbon-epoxy, glass-phenolic, etc.). A sub-class of fibre-reinforced composites is carbon fibre reinforced composites (CFRC). One of these, carbon-carbon composites has some form of carbon as both the reinforcing member and the matrix. The reinforcing component is usually a fibre made from one of the following precursors: pitch (coal tar/petroleum), mesophase pitch (synthetic, coal tar or petroleum derived), or polyacrylonitride (PAN, the old Dupont Orlon used for carpets, socks, etc.) or rayon (a cellulosic material made from wood pulp). Additionally, carbon fibres derived from cellulosic precursors or produced via catalysed chemical vapour deposition (ccvd) are also available, but are rarely used in brake material applications.

In the early 1960's, rayon (cellulosic) was a popular carbon fibre precursor and is presently used in the nose cap and exhaust nozzle of various aerospace vehicles, as in the space shuttle [15]. However, large quantities of carbon fibres are no longer routinely produced using rayon since they require stretching at a high temperature to produce fibres with optimum properties. This requirement when combined with the low carbon yield (35%) results in a process that is not commercially viable [16].

The matrix for carbon-carbon is generally made from organic precursors such as pitch, resin, hydrocarbon gas, or a hybrid of these. Typical properties of carbon-carbon composites that make them attractive substitutes for other
structural materials including metals and alloys include; high specific strength/stiffness, high temperature strength, high corrosion resistance, low thermal expansion, good friction and wear properties and high thermal and electrical conductivity. The diversity in properties displayed by carbon-carbon composites provides for a wide variety of applications including aircraft brakes, automobile brakes, rocket nozzles, nose cones as well as structural materials in the aerospace industry [9,17-21].

3.2 The Manufacturing Processes for Carbon-Carbon Composites

This section provides background information on carbon-carbon composites. The specific processes for carbon-carbon composite aircraft brakes will be discussed in Section 3.3

3.2.1 Reinforcing Components

Either PAN or pitch fibre reinforces most carbon-carbon composites. A summary of the major carbon fibre varieties available at the present time is given in Table 3.1 [22-31]. Table 3.1 shows how in general, the PAN fibres provide high strength and intermediate modulus properties while the pitch fibres provide high modulus intermediate strength properties. The fibre cross-section shape, the surface treatment, the microstructure as well as the process conditions used during the manufacture of the fibres influence these properties. For the manufacturing processes for PAN and pitch fibre the reader is referred to [32-35]. There have been several good reviews on the structures of carbon fibres, and the reader is referred to these [36-39]. In contrast to all other fibrous materials, only carbon fibres can be fabricated to exhibit a range of Young’s modulus values between 206 GPa and 689 GPa (30 msi and 100 msi) and strength values from 1759MPa-4137MPa (255 ksi and 600 ksi). The higher modulus fibres possess higher thermal conductivity, higher density and carbon assay and lower thermal expansion coefficients than the lower modulus fibres. Polyacrylonitrile (PAN) based carbon fibres tend to exhibit high tensile strengths
but medium Young's modulus values 4127 MPa (600 ksi) and 241 GPa (33 msi) respectively.

Table 3.1. Types of Carbon Fibres

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Manufacturer</th>
<th>Tensile Modulus (GPa) (Msi)</th>
<th>Tensile Strength (GPa) (Msi)</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pitch</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P-25</td>
<td>Amoco</td>
<td>159 23</td>
<td>1.38 200</td>
<td>1.90</td>
</tr>
<tr>
<td>P-55</td>
<td>Amoco</td>
<td>379 55</td>
<td>1.72 250</td>
<td>2.00</td>
</tr>
<tr>
<td>P-75</td>
<td>Amoco</td>
<td>517 75</td>
<td>2.07 300</td>
<td>2.04</td>
</tr>
<tr>
<td>P-100</td>
<td>Amoco</td>
<td>724 105</td>
<td>2.24 325</td>
<td>2.15</td>
</tr>
<tr>
<td>P-120</td>
<td>Amoco</td>
<td>827 120</td>
<td>2.24 325</td>
<td>2.15</td>
</tr>
<tr>
<td>E-35</td>
<td>DuPont</td>
<td>241 35</td>
<td>2.83 410</td>
<td>2.10</td>
</tr>
<tr>
<td>E-75</td>
<td>DuPont</td>
<td>517 75</td>
<td>3.10 450</td>
<td>2.16</td>
</tr>
<tr>
<td>E-105</td>
<td>DuPont</td>
<td>724 105</td>
<td>3.31 480</td>
<td>2.17</td>
</tr>
<tr>
<td>PAN</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-300</td>
<td>Amoco</td>
<td>231 34</td>
<td>3.24 470</td>
<td>1.79</td>
</tr>
<tr>
<td>T-2</td>
<td>PRChina</td>
<td>172 25</td>
<td>2.24 325</td>
<td>-</td>
</tr>
<tr>
<td>AS-4</td>
<td>Hercules</td>
<td>231 34</td>
<td>3.64 528</td>
<td>1.80</td>
</tr>
<tr>
<td>T-40</td>
<td>Amoco</td>
<td>290 42</td>
<td>3.45 500</td>
<td>1.78</td>
</tr>
<tr>
<td>HMS</td>
<td>Hercules</td>
<td>345 50</td>
<td>2.21 -</td>
<td>1.83</td>
</tr>
<tr>
<td>GY-70</td>
<td>BASF\Celan</td>
<td>517 75</td>
<td>1.86 153</td>
<td>1.96</td>
</tr>
<tr>
<td>Rayon</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>WCA</td>
<td>Amoco</td>
<td>69 10</td>
<td>- -</td>
<td>-</td>
</tr>
<tr>
<td>T-75</td>
<td>U. Carbide</td>
<td>538 78</td>
<td>2.62 380</td>
<td>1.80</td>
</tr>
</tbody>
</table>

Mesophase pitch fibres tend to exhibit higher modulus 827 GPa (120 msi), and density values (2.18 g/cm³) but lower strength values than PAN fibres [40-42]. The fibres are spun into tows of 3000 to 480,000 fibre filaments. These fibre tows are then utilised either as unidirectional chopped fibres, woven fabrics or non-woven felts. Chopped fibres, which consist of short sections of fibre
tows, are used in applications where the strength requirements do not necessitate
the use of costly woven fabrics (i.e., aircraft brakes)[17]. There are many types
of weaves used to make carbon fibre substrates [18]. Almost any weave from the
textile industry from plain weave to 5 and 8 harness satins are used in carbon
composites. Also needled sheets of unidirectional fibre are used for the fibre
substrates of some carbon-carbon composites, which are used in rocket nozzles
and recently the production of aircraft brakes.

3.2.2 Densification of Carbon Fibre Reinforced Composites

There are basically three methods of creating the matrix of carbon-carbon
composites. The first is to infiltrate a substrate of fibre with chemical vapour
deposition (CVD), also referred to as chemical vapour infiltration (CVI), of a
hydrocarbon gas, usually methane, propane, or a combination of both. This is
done by cracking the gas at low pressure (200-3000 Pa) and approximately
1000°C, and with gas flows approximately 100 volume changes of gas in the
reaction vessel per minute. The second method is to use a resin such as phenolic.
The fibre is usually dipped in the resin to create a prepreged yarn, which is then
chopped and used to mold a part under heat (approximately 180°C). The molded
part is heated to 250°C to cure the thermal set phenolic resin. The molded and
cured part is then heated to approximately 900°C to char the resin. The charred
part is sometimes heat-treated to open up the porosity and relieve residual stress.
After this initial heat treat the part is densified with CVD. The third method is a
hybrid, such as used in rocket nozzles, where the fibre substrate is rigidised with
CVD and then impregnated with liquid pitch or resin. The composite is then
heat-treated to carbonise the pitch. The steps of impregnation and carbonisation
may be repeated as many as 10 times to fully densify the composite [19].
3.2.3 Heat Treatment

After the composite is densified it is usually heat treated to a temperature greater than the end use temperature to ensure that no further changes in the composite properties will occur during application. Heat-treatment temperatures are often as high 2500 °C to graphitize the graphitizable carbon components and thus modify the composite properties. This graphitization process occurs readily in pitch and CVD matrices but can also occur in resins via stress graphitization. Phenolic resin is one of the few resins that can graphitize when stress is created during heat treatment [43]. This only occurs in the small area inside the fibre bundles, therefore the bulk of the resin remains isotropic. The graphitization process causes the crystallites of graphitizable carbons to increase in size while their interplanar d-spacing decreases. These changes in crystallinity cause dimensional changes within the composite that may lead to fibre-matrix debonding due to differences in the thermal expansion between the fibres and the matrix [44].

As a result of these changes in structure during graphitization, the properties of carbon composites are also modified. The strength and thermal expansion of carbon composites decrease while the strain to failure is increased with increased heat treatment temperature. Meanwhile, thermal conductivity and thermal stress resistance increase. Properties are less affected when non-graphitizable matrices (thermoset resins and isotropic pitch) are used. Differences between the coefficient of thermal expansion of the fibres and matrix generate internal stresses and stress cracks on cooling.

3.2.4 Oxidation protection

Carbon-carbon composites are excellent materials to use at high temperature as long as they can be protected from oxidation in air. At 500°C the oxidation of carbon becomes rapid and leads to an increase in porosity, weight loss, and lowering of mechanical properties [45,46]. Levels of 2 to 5% weight
loss due to oxidation leads to a substantial degradation (40-50%) of mechanical properties [47-49]. Therefore, although short-term high temperature applications (i.e. rocket nozzles and exhausts) may only require limited oxidation protection, long-term exposure normally requires that a surface barrier coating be present [50], or an inhibitor (such as the “getter” used in vacuum tubes to react with oxygen) must exist throughout the matrix.

3.3 The Mechanical Properties of Carbon-Carbon Composites

Some of the more important mechanical properties of carbon-carbon composites include the elastic modulus, the tensile strength, the inter laminar shear strength and the compressive strength. However, the importance of a given property is dictated by the application of the composite. Some of the important aspects related to the mechanical properties of carbon-carbon composites are described below.

The elastic modulus describes the stiffness of a composite. When measured parallel to the fibre direction, the elastic modulus of a unidirectional reinforced composite can be calculated from the rule of mixtures,

\[ E_C = E_m(1-V_f) + E_f V_f \]  \hspace{1cm} (3.1)

Where \( E_m \) and \( E_f \) are the modulus of the matrix and fibre measured parallel to the fibre axis; \( V_f \) is the fibre volume fraction. When the modulus of the matrix is much smaller than that of the fibres, equation (3.1) can be reduced to:

\[ E_C = E_f V_f \]  \hspace{1cm} (3.2)

The above expressions give reasonable values for resin and metal matrix composites. However, it has been shown [51-54], using a variety of fibre types
and matrices, that the measured modulus values for carbon-carbon can be much larger than might be estimated by substituting the appropriate values for \( E_f \) and \( V_f \) into equation 3.2.

For example Murdie et. al. [55] provided evidence of the large stiffness contribution of the matrix when they tested a unidirectional reinforced sample of PAN T-300 fibre reinforced pitch matrix composite that had been cut from a graphitized three dimensional composite. The composite sample contained about 50-volume percent fibre reinforcement and exhibited a modulus of 310 GPa (45 Msi). Since Becker [56] previously reported that the modulus of graphitized T-300 PAN fibres is approximately 400 GPa (58 Msi) it was inferred that the modulus of the carbon matrix in the direction of the fibre axis was 220 GPa (32 Msi).

These studies confirmed that the matrix carbons could make a significant contribution to the modulus of a composite. Such contributions have been shown to be caused by the preferred orientation of the basal planes in the graphitic crystallites of the matrix carbons. It was shown earlier that the crystallites in pitch, resin and CVD carbon matrices can all develop a preferred orientation relative to the fibre axis. Therefore, it is possible to tailor the modulus of carbon-carbon composites by varying the microstructure (degree of preferred orientation) of the matrix carbon.

The tensile strength of carbon-carbon composites defines the maximum tensile load that can be applied to the material before fracture occurs. According to the rule of mixtures, in order for the mechanical properties of a continuous fibre reinforced composite to be superior to an unreinforced material, it is necessary that the modulus and strength of the fibre reinforcement be greater than that of the matrix.

\[ E_f \gg E_m \]  

(3.3)

There must be chemical, physical or mechanical bonds formed between the fibres and matrix that are strong enough to transfer load between individual
fibres and between fibre layers. In a mechanically loaded composite, it is assumed that the strain in each part of the composite is identical and equal to the total strain on the composite. The failure strain of a polymer or a metal, is substantially larger than the failure strain of the reinforcing fibres, and failure of this type of composite occurs when the fibres fail. However, in the case of a brittle fibre/brittle matrix system as in the case of carbon-carbon composites, there is a deviation from the rule of mixtures. In carbon-carbon composites, the failure strain of the matrix is smaller than the failure strain of the fibres and, in a well-bonded composite, failure occurs when the matrix fails. In carbon-carbon composites the overall strength of the composite is controlled by the following parameters:

- Young's modulus of fibres
- Fibre matrix interface
- Fibre volume fraction

The effect of the fibre matrix interface on the mechanical properties of carbon-carbon composites has been studied in some detail [57-66]. Fitzer et al. [58] studied the effect of degree of fibre oxidation on the mechanical properties of resin composites reinforced with high modulus and high strength fibres. After carbonisation of the resin matrix they found that relative to material reinforced with nonoxidised fibres, increasing the fibre oxidation improved the strength of the material reinforced with the high modulus fibres, but decreased the strength of the composites containing high strength fibres.

Yasuda et al. [59] performed a similar experiment using surface treated and non-surface treated high modulus fibres and a furfuryl alcohol condensate resin matrix and found that after the initial carbonisation the strength of the composite containing the surface treated (oxidised) fibres was lower than that containing untreated fibres. However, following graphitization, the strength of the surface treated fibre composites increased by a factor of three or more whilst the non-surface-treated materials decreased by nearly the same amount. In
effect, Yasuda showed that the composite reinforced with the surface treated fibres exhibited the highest strength when graphitized. Yasuda explained these differences to the increased ductility of the graphitized matrix that improved the stress transfer from fibre to fibre [59].

There appears to be a synergistic effect between the carbon fibres and the matrix carbon, whereby the carbon fibres impart strength and stiffness to the composite whilst the matrix provides integrity by holding the fibres together as well as providing a medium through which stress transfer can occur from the matrix to the load bearing fibres.

The effects of the fibre-matrix interface on the properties of the composite are not well understood. However, the strength of the fibre matrix interfacial bond appears to determine the efficiency of the matrix to fibre load transfer mechanism and therefore influences critical properties including the Young's modulus, inter-laminar shear strength, compressive strength, as well as the mode of failure [60]. The load transfer mechanism between the fibre matrix interfaces is dependent on a strong fibre matrix interfacial bond, which resists failure [61].

Attempts to improve the strength of carbon-carbon composites have been achieved by arresting the movement of an otherwise catastrophically propagating crack by lowering the stress at its tip. Providing weak interfaces does this or cracks oriented approximately perpendicular to the plane of the advancing crack. The most obvious interface that can be made weak is the one that exists at the junction between the fibre and the matrix. Weak interfaces occur naturally in some matrix-fibre combinations, while strong interfaces will occur in others [62-64]. Specifically, the nature and strength of the bond appears dependent on the reactivity of the fibre (i.e. fibre type), the reactivity of the matrix, and the type and degree of fibre surface treatment. Crack branching improves the strength of the composite, and the frictional resistance associated with pulling fibres from the matrix imparts a pseudo-plasticity. Thus, both crack branching and fibre pullout are desirable qualities that improve the structural capability of an initially brittle composite. Unfortunately, the appropriately oriented weak interfaces
necessary to improve the strength parallel to the fibres will result in a degradation of the inter-laminar tensile and shear properties.

Nevertheless, an improvement in properties is achieved with densification. For instance, Fitzer [52] indicated that the strength of non-surface treated HM fibres could be increased from 10% after the initial carbonisation to 90% of expected after four densification steps. Conversely, non-surface treated 227 GPa (33 Msi) fibres increased to only about 40%. The inference being that reaction occurred naturally between the fibre and matrix in the case of 227 GPa (33 Msi) fibres whereas little reaction occurred at any stage of the process for the HM materials. Perry and others [51,68] working with phenolics and different fibres found a maximum of only 40% to 50% translation of strength indicating some degree of reaction; however, since their preparation technique involved a number of graphitizations, other factors may have contributed to the low strength values.

Fitzer et al. [52] showed that resins that form strong fibre-matrix bonds and also exhibit the highest shrinkage should be avoided if high translation of fibre strengths is required. They suggest the use of high carbon yielding precursors that do not form strong bonds and that exhibit minimum shrinkage. Data presented indicates that pitches (rather than resins) will produce better translation of fibre strengths for fewer densification cycles.

The addition of graphite powder is sometimes used to decrease the shrinkage of the resin matrix during carbonisation and to increase the char yield [58]. Since the decrease in the thermal contraction should also produce a corresponding decrease in the internal stress generated in the matrix on cooling from carbonisation temperatures, the strength of composites whose failure is matrix dominated should be improved. The results of Fitzer et. al. [67] confirmed this as they demonstrated that the strength translation of 227 GPa (33 Msi) fibre reinforced material increased from 40 to 60 % when 50% graphite powders were added to the resin matrix before carbonisation. Further work appears necessary to more fully characterise the improvements in strengths that might be obtained by matrix pre-stressing.
The matrix of the carbon composite has significant influence on the mechanical properties. The out-of-plane, off-axis and transverse properties of unidirectional reinforced composites (transverse modulus, and strength and inter-laminar shear strength) depend mostly on the properties of the matrix or the fibre-matrix bond. The results of Perry and Adams [51] showed that larger values of transverse strength, modulus and inter-laminar shear are obtained from composites reinforced with well-bonded fibres. Although several fibre types were investigated, it appears that bonding is controlled by the reactivity of the matrix and the fibre. All properties improved with densification up to the fourth cycle and, in general, heating to graphitization although degrading the transverse tensile strength caused no degradation in inter-laminar shear values. For instance, carbon fibre reinforced carbonised Borden SC-1008 phenolic exhibited a maximum transverse strength of about 6 MPa (870 psi) that degraded to about 4.8 MPa (700 psi) or less when heated to graphitization temperatures.

The apparent superiority in matrix dominated properties of the CVD densified material when compared to resin densified material is supported by the work of Walker et al. [69] who indicate that both transverse tension and inter-laminar shear of 2D carbon-carbon are superior if the matrix is prepared by chemical vapour deposition. These authors indicated inter-laminar tensile strengths of about 5.4 MPa (790 psi) and inter-laminar shear strengths of 16.1 MPa (2340 psi) for carbonised resin precursor material compared with 10.1 MPa (1460 psi) and 18.9 MPa (2740 psi) for CVD material. In this work, it was pointed out that the superiority of CVD was only true for thin materials, 2.8mm (0.1 inch), since reductions of both inter-laminar tensile and inter-laminar shear strengths occurred when the thickness of the CVD densified material approached 12.7mm (0.5 inches). The degree of difficulty in fully densifying thick sections using CVD was shown to be related to this property degradation.

It appears reasonable to summarise that the matrix dominated properties depend on: fibre type, surface reactivity, matrix type (resin precursor, CVI microstructure, pitch precursor, etc.), matrix reactivity, fibre volume fraction, processing condition, degree of densification, fibre orientation, and laminate
thicknesses. By optimising all of these parameters, the matrix-dominated properties can be maximised. Nevertheless, by far the greatest improvement in these properties is obtained by placing fibres in the appropriate directions.

Table's 3.2 and 3.3 from Reference [70] compares carbon-carbon composites mechanical properties obtained in the fibre direction to polycrystalline graphite and pyrolytic graphite's very high properties. Figure 3.1 also from Reference [70], compares the strength and stiffness of carbon-carbon composites with other aerospace materials at elevated temperatures.
<table>
<thead>
<tr>
<th>Properties</th>
<th>Test Direction</th>
<th>1-D CCC (P-100/Pitch Coke)</th>
<th>2-D CCC (T-300 HT 8HSW/ Phenolic Char)</th>
<th>3-D FWPF CC (HM Yarn-fabric/ Pitch Coke)</th>
<th>4-D Orthotropic CCC (HM Tow Rods/Pitch Coke)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³ (lb/in³)</td>
<td></td>
<td>1.87 (0.068)</td>
<td>1.63 (0.059)</td>
<td>1.95 (0.070)</td>
<td>1.89 (0.068)</td>
</tr>
<tr>
<td>Tensile Strength, MPa (ksi)</td>
<td>X</td>
<td>656 (82)</td>
<td>330 (48)</td>
<td>228 (33)</td>
<td>303 (44)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>14 (2.0)</td>
<td>4.5 (0.65)</td>
<td>172 (25)</td>
<td>103 (15)</td>
</tr>
<tr>
<td>Tensile Modulus, GPa (Msi)</td>
<td>X</td>
<td>303 (44)</td>
<td>117 (17)</td>
<td>83 (12)</td>
<td>37 (5.3)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>10 (1.5)</td>
<td>3.1 (0.45)</td>
<td>69 (10)</td>
<td>97 (14)</td>
</tr>
<tr>
<td>Tensile Ultimate Strain, %</td>
<td>X</td>
<td>0.23</td>
<td>0.33</td>
<td>0.25</td>
<td>0.31</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>0.21</td>
<td>0.19</td>
<td></td>
<td>0.42</td>
</tr>
<tr>
<td>Compressive Strength, GPa (Msi)</td>
<td>X</td>
<td>331 (48)</td>
<td>200 (29)</td>
<td>138 (20)</td>
<td>38 (5.5)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>32 (4.6)</td>
<td>255 (37)</td>
<td>117 (17)</td>
<td>200 (29)</td>
</tr>
<tr>
<td>Compressive Modulus, GPa (Msi)</td>
<td>X</td>
<td>269 (39)</td>
<td>110 (16)</td>
<td>83 (12)</td>
<td>15 (2.1)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>9 (1.3)</td>
<td>8.3 (1.2)</td>
<td>66 (9.5)</td>
<td>200 (29)</td>
</tr>
<tr>
<td>In plane Shear Strength, MPa (ksi)</td>
<td>X</td>
<td>34 (4.9)</td>
<td>41 (6.0)</td>
<td>9.7 (1.4)</td>
<td>62 (9.0)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermal Conductivity, W/m-K (Btu/ft-h-°F)</td>
<td>X</td>
<td>204 (118)</td>
<td>45 (26)</td>
<td>159 (92)</td>
<td>100 (58)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>17 (10)</td>
<td>4.7 (2.7)</td>
<td>130 (75)</td>
<td>195 (113)</td>
</tr>
<tr>
<td>Thermal Expansion Coefficient, RT to 1650°C (3002°F) ppm/°F</td>
<td>X</td>
<td>-1.8 (-1.0)</td>
<td>1.3 (0.73)</td>
<td>1.1 (0.63)</td>
<td>1.5 (0.85)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>5.9 (3.3)</td>
<td>1.2 (0.65)</td>
<td></td>
<td>1.3 (0.73)</td>
</tr>
</tbody>
</table>

Table 3.2. Typical Properties of Carbon-carbon composites Materials [70]

X = Parallel to with-grain in graphite or major fibre/fabric axes in composites

Z = Parallel to across-grain in graphite or perpendicular (transverse) to major fibre/fabric axes.

Room temperature values; not for design purposes
### Table 3.3. Typical Properties of Graphite and Composites Materials [70]

<table>
<thead>
<tr>
<th>Properties</th>
<th>Test Direction</th>
<th>Polycrystalline Graphite (ATJ-S)</th>
<th>Pyrolytic Graphite</th>
<th>2-D CCC (T-300 HT 8 SW/Phenolic Char)</th>
<th>3-D FWPP CCC (HW Yarn- Fabric/Pitch Coke)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³ (lb/in³)</td>
<td>X</td>
<td>1.83 (0.066)</td>
<td>2.22 (0.080)</td>
<td>1.63 (0.059)</td>
<td>1.95 (0.070)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>1.95 (0.070)</td>
<td>2.22 (0.080)</td>
<td>1.63 (0.059)</td>
<td>1.95 (0.070)</td>
</tr>
<tr>
<td>Tensile Strength, MPa (ksi)</td>
<td>X</td>
<td>37 (5.4)</td>
<td>124 (18)</td>
<td>330 (48)</td>
<td>228 (33)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>30 (4.4)</td>
<td>6.9 (1.0)</td>
<td>4.5 (0.65)</td>
<td>172 (25)</td>
</tr>
<tr>
<td>Tensile Modulus, GPa (Msi)</td>
<td>X</td>
<td>12 (1.7)</td>
<td>29 (4.2)</td>
<td>117 (17)</td>
<td>83 (12)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>7.6 (1.1)</td>
<td>10 (1.5)</td>
<td>3.1 (0.45)</td>
<td>69 (10)</td>
</tr>
<tr>
<td>Tensile Ultimate Strain, %</td>
<td>X</td>
<td>0.44</td>
<td>0.4</td>
<td>0.33</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>0.54</td>
<td>---</td>
<td>---</td>
<td>0.19</td>
</tr>
<tr>
<td>Compressive Strength, GPa (Msi)</td>
<td>X</td>
<td>52 (7.5)</td>
<td>97 (14)</td>
<td>200 (29)</td>
<td>138 (20)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>41 (6.0)</td>
<td>345 (50)</td>
<td>255 (37)</td>
<td>117 (17)</td>
</tr>
<tr>
<td>Compressive Modulus, GPa (Msi)</td>
<td>X</td>
<td>9.6 (1.4)</td>
<td>17 (2.5)</td>
<td>110 (16)</td>
<td>83 (12)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>7.3 (1.1)</td>
<td>23 (3.3)</td>
<td>8.3 (1.2)</td>
<td>66 (9.5)</td>
</tr>
<tr>
<td>In plane Shear Strength, MPa (ksi)</td>
<td>X</td>
<td>19 (2.8)</td>
<td>3.5 (0.5)</td>
<td>41 (6.0)</td>
<td>9.7 (1.4)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>121 (70)</td>
<td>415 (240)</td>
<td>45 (26)</td>
<td>159 (92)</td>
</tr>
<tr>
<td>Thermal Conductivity, W/m-K (Btu/ft-h°F)</td>
<td>X</td>
<td>161 (93)</td>
<td>415 (240)</td>
<td>45 (26)</td>
<td>159 (92)</td>
</tr>
<tr>
<td></td>
<td>Z</td>
<td>121 (70)</td>
<td>23 (13)</td>
<td>4.7 (2.7)</td>
<td>130 (75)</td>
</tr>
</tbody>
</table>

X = Parallel to with-grain in graphite or major fibre/fabric axes in composites

Z = Parallel to across-grain in graphite or perpendicular (transverse) to major fibre/fabric axes.

Room temperature values; not for design purposes
3.4 Carbon-Carbon Composite Aircraft Brakes

The remainder of this chapter is specific to carbon-carbon composite aircraft brakes. Properties of carbon-carbon composites that make them attractive as friction materials include good heat capacity and high strength at elevated temperatures [17,71-73]. The graphitic structure of carbon-carbon composites also provides low wear rates due to the ability of graphite to act as a self lubricant [74].
Carbon offers a number of advantages over these materials. These advantages, as mentioned above, include weight savings, extended life, smoother operation, survival of maximum energy stop, and absorption of higher energies per mass due to its higher heat capacity. But, since they are much more complex, more expensive to manufacture and maintain, why did the conversion to carbon-carbon take place? There were two reasons, one was technical because of the new performance requirements in some types of aircraft and the other was actually because of an economic reason [75,76].

3.4.1.1 The Technical Reason and New Requirements to change to Carbon-Carbon

To understand the technical reasons to use carbon for brakes, it is necessary to have an understanding of the range of energies that the new larger aircraft brake place e on the brakes to convert kinetic energy to heat during the stopping of an aircraft. Aircraft brakes operate in a wide and continuous range of energies, but for design purposes only three energy conditions are defined. They are taxi stops (snubs), landing stops (normal energy), and the rejected take off emergency stop (RTO). The actual energy of a stop is a function of the aircraft speed and its weight, which of course is kinetic energy. The taxi stops are the lowest energy stops and occur as the aircraft travels to and from the airport gate. The landing stop is a much higher energy than a taxi. A typical commercial aircraft may land at a speed of 290Km/h (180 mph), which provide brake disc sliding speeds of 15.4m/s-22.9m/s (50-75 ft/sec).

Higher energy landing stops are sometimes defined in design, to account for the rogue pilot that just has to make the first turn off the runway. The highest energy stop is the rejected take off (RTO), which must be designed into the brake system for safety. This is a design requirement by the CAA, FAA, and other national safety of flight organisations worldwide. The RTO occurs when a fully loaded aircraft, with maximum fuel for the flight, is at take off (or roll up) speed and must for some reason abort the flight. The brake system must be capable of stopping the aircraft before the runway ends and not allow the brake heat to transfer to the wheel and start a tire fire for 15 minutes following the stop. The
brakes must also be able to function following the RTO stop to allow the aircraft to be removed from the runway. A typical RTO for a Boeing 767 weighing 170,000kg and traveling at 322 km/h (200mph) would be about 670 MJ [72]. High bulk temperatures (>1500°C) are reached due to the high energy and the short stopping time during which it is converted. Friction surfaces have been reported to reach temperatures in excess of 3000°C [17], another reference says 1100°C bulk and surface temperatures exceeding 2400°C[6]. In either case the temperature are significantly higher now than in the past. All heat must be absorbed by the brake stack and dissipated slowly to prevent softening of nearby metal structures or causing a tire fire. This RTO requirement for the larger aircraft that were being built would require a great deal more mass for the metallic brakes then currently in use to absorb so much kinetic energy. This much heat sink mass would add greatly to the aircraft total weight. Larger brakes, larger gear retraction systems, and other design problems, such as space (envelope to put this large metallic brake), were reasons to look for a new lighter brake material.

Even though these new technical requirements indicated a new brake material was needed, Boeing still put cerametallic brakes on the 747 initially through the dash 400 version, because carbon-carbon brakes were not yet available in the quantities needed.

3.4.1.2 The Economic Reason for Changing to Carbon-Carbon Brakes

The main economic reason to look for a new brake material was the fossil fuel energy (jet fuel) crisis of the nineteen-seventies. All the aircraft manufacturers conference rooms were a buzz with “hockey stick” projections of fuel costs rising to over $4.00 per gallon. (It is still at this time only 57 cents). The business charts all showed the huge gains per flight for each pound of weight saved. Before carbon-carbon was chosen to reduce the weight there were some designs that used beryllium brake discs with carbon friction pads to act as the friction surface. The C-5A, saved brake weight of 726Kg (1600 lbs) over cerametallic linings with this beryllium-carbon brake for its twenty four wheel
sets. Others followed the beryllium-carbon brake route, the Lockheed S-3A, the Grumman F14, and the first space shuttle. These brakes had one huge problem, the interface between the carbon and beryllium would, with the brake heat, form beryllium carbide, which is very brittle and the pads would crack and fall off.

Today, all these aircraft have changed to carbon-carbon composite brakes. When the C-5B switched to carbon-carbon from beryllium, it saved an additional 180 kg (400 lbs) [77]. The reason that beryllium was dropped was not the safety issue of machining it, the brakes just did not work [75,76].

3.4.1.3 The switch to Carbon-Carbon for brake materials

Carbon is attractive to the aircraft brake industry because it is lighter than the metals that were utilised previously. The use of carbon-carbon composites in aircraft brakes resulted in 40%-60% weight savings over conventional steel brakes [17,72]. The weight saving were realised because carbon has a much better heat capacity than metals. With the threat of high fuel prices, the economics forced the industry to change.

The candidate material must have suitable heat capacity to absorb these new high energies, with less weight. The heat capacity of carbon is 2.5 times greater than that of steel [72]. During braking, the brake lining is heated rapidly, which makes carbon an attractive material since it is highly resistant to thermal shock. Sufficient friction must also be generated by the brake lining material surface to bring the aircraft to a stop. High mechanical strength is also required because the brake lining also acts as a structural material that transfers the frictional torque to the axles during braking. High strength must therefore be retained at the elevated temperatures that are reached during brake application. The strength of carbon is comparable to that of steel; at high temperature carbon is nearly twice as strong [17]. Thus, carbon could handle the new larger RTO energies with far less mass; this was the economic and some would say technical reason for the consideration of carbon-carbon. However, there are factors other than weight that favor carbon, it has lower volume of material and provided 3000 landings vs. 400 to 1000 landings the airlines were getting for the metallic brakes.
Today, more than 63% of all carbon-carbon composites produced are for the heat sink and friction material used in aircraft brakes [6]. Yet the energy crisis did not occur, the economics predicted did not happen, so carbon-carbon turned out to make sense for only large transport aircraft to save weight, and military aircraft for performance. The new Boeing 737-400 designed for short commuter flights is using cerametallic brakes today. Although there were some military experiments with carbon brakes in the 1960’s, the first commercially used carbon brake was for the Concorde provided by Dunlop in 1974[6].

The mother of nearly all the carbon-carbon brake production technology was the Super-Temp Division of Ducomen. B.F. Goodrich later purchased Super-Temp. Prior to that purchase that took place in 1984, Super-Temp assisted in the development of carbon-carbon brake production for AlliedSignal Aircraft Landing Systems (Then Bendix Wheel and Brake), Dunlop, Messier (SEP), and Hitec (who made brakes for Goodyear Aerospace, now Aircraft Braking Systems) and B. F. Goodrich. Super-Temp provided equipment and technology to all five of the major aircraft brake manufacturers that exist today. The reason there are now five independent manufactures is because of a United States State Department decision. The state department of the United States put carbon-carbon brakes on the weapons list, since carbon had improved the performance of military aircraft, restricting the export of this technology. Thus Ducomen was forced to pull out of England and France and sold all their equipment in Europe to Dunlop and SEP who became independent carbon-carbon manufacturing companies. When B. F. Goodrich purchased Super Temp from Ducomen, the other United States manufactures were then on their own to develop their carbon-carbon technology [75,76]. Each of the manufacturers has improved upon the Super-Temp technology primarily in the direction of lowering manufacturing cost. According to Savage [6], the cost of carbon-carbon brakes was around $170/pound when they were first introduced. Today, the cost is between $20 and $50/pound [6]. This reduction in cost per pound is more due to manufacturing process improvements than life cycle costs. The uneconomical side of carbon is that it fails because of vibration. Carbon-carbon brakes are not quiet, much
design time and resources have been expended to solve this problem. Since the energy crunch did not occur, it might be more economical to operate some aircraft now using carbon with the newer cerametallic linings. However, the reason that some of the lighter aircraft did not switch back to cerametallic materials is that the new landing gear retraction systems designed for the carbon brakes could not handle the retraction loads of a steel brake mass and over the years of using carbon-carbon brakes the envelope for the brake stack had changed considerably.

3.5 The Manufacturing process for Carbon-Carbon Brake Discs

Before understanding the properties of carbon-carbon composites used for brake applications, it is necessary to understand the processing steps used in their manufacture. Two typical processes for manufacturing carbon-carbon composite brake discs are the random fibre molded preform process and the non-woven preform process shown schematically in Figures 3.2 and 3.3.

Both of these processes can be broken down into three major steps, preform manufacturing, densification and post-densification processing. The major differences, in the two processes shown, are in the manufacturing of the preform. Each of the processes will be discussed followed by the processing steps common to both.

3.5.1 Random Fibre Process

For the random fibre process, Figure 3.2, the preform is made from resole phenolic resin impregnated chopped carbon fibre, both PAN and pitch. The resole phenolic resin is carbonised to about 40 weight percent. It is used primarily for its excellent carbon fibre wetting and moldable characteristics during hot molding. The resin is combined 50:50 by weight with PAN or pitch chopped carbon fibre. The resultant material is referred to as a prepreg. The prepreg fibre is tacked into a preform and then molded. The resin is then cured.
usually around 250°C for a thermal set, such as phenolic. The molded part is then carbonised at about 900°C. The carbonised preform is further heated to temperatures in the range of 1600-2400°C. This heat treatment of the preform relieves internal molding stress and opens the porosity through cracking on cooling. The heat treat temperature will also contribute to determining the shape, size, and distribution of the porosity of the preform, which will greatly influence the chemical vapour deposition rate to follow. This initial heat treatment could advance the graphitization of some types of carbon fibres, i.e. pitch.

![Diagram of the Random Fibre Manufacturing Process](image)

**Figure 3.2. The Random Fibre Manufacturing Process**

### 3.5.2 Non-woven Process

The non-woven fibre brake process is shown in Figure 3.3. It begins by first making unidirectional "fabric" from large (i.e., 360,000 filaments) oxidised PAN tows. Needling a continuous warp of tows produces this fabric. The needles are barbed to rip and entangle the filaments of the tow to form a sheet.
The sheet or "unidirectional fabric" can be needled in blocks and die cut to an angular shape or cut into segments which are then needled into a preform. This type of preform is referred to as Non-woven material. The advantage of the segments is load path control and reduction of scrap over making a non-woven block and cutting out an annular ring, as some manufacturers do.

The segments can be cut so that the fibre directions of the segments used to fabricate a brake disc are within the control of the designer. In the process for the samples used in this study, chordal and radial segments are laid up in alternate layers to produce load-carrying paths that are parallel and perpendicular to the sliding direction. This type of control of the architecture cannot be accomplished with the random fibre process. The other major advantage of non-woven preforms over random fibre preforms is the lack of resin. Thus, no molding, resin curing, and charring are required. The matrix structure is comprised of only CVD carbon that is graphitizable. The disadvantage is that without resin char, much more CVD carbon is required to densify the substrate, approximately four times as much, greatly reducing CVD capacity in the plant. The preforms are then carbonised at approximately 1000°C to 2600°C, converting the oxidised PAN to carbon fibre. About 53% shrinkage will occur during carbonisation. The preform is then die-cut to near net shape to reduce machining after densification.
3.5.3 CVD Densification

The subsequent densification process for random fibre and the non-woven processes is the same, chemical vapour deposition (CVD), which is the primary densification process for most commercial carbon-carbon composites. The CVD used in this particular process is isothermal CVD. During CVD it is possible to control the matrix microstructure of the CVD carbon through the process variables temperature, pressure, gas composition, and gas residence time. The CVD carbon microstructure can be characterised as isotropic, smooth laminar, and rough laminar [78]. There is no literature that proves which microstructure is best for brakes. However, since rough laminar structure will easily graphitize during heat treatment, the friction and wear properties can be influenced by the heat treatment temperature. Post CVD, the densified discs (1.70g/cm$^3$ to 1.90g/cm$^3$) are heat-treated to advance the graphitization of the CVD matrix, then machined to final configuration and finally protected on the non-friction surfaces with an application of an anti-oxidant. These final processing steps go a long way toward determining the final composite properties and the friction and wear performance.

3.6 Applications of Carbon-Carbon Composites as Friction Materials used in Aircraft Brakes

The remainder of this chapter will first provide an explanation of an aircraft brake system, and then the properties for aircraft brakes will be discussed. The majority of the literature review was limited to porosity and friction and wear, which are the emphasis of this study

3.6.1 Aircraft Brake System

Carbon-carbon aircraft brakes usually consist of multiple discs. A photograph of a Bendix carbon brake landing gear assembly produced by an
Allied Signal Corporation (Honeywell) finite element model is shown in Figure 3.4. The Boeing 777 carbon-carbon brake cut away is shown in Figure 3.5, followed by a schematic drawing of the same wheel and brake assembly in figure 3.6. A typical set of carbon-carbon discs are shown in Figure 3.7.

Figure 3.4. The Boeing 777 Truck with Four Carbon Brakes
Figure 3.5. Boeing Carbon Brake and Wheel Assemble Cut Away

Figure 3.6. Boeing 777 Brake Schematic
The stack of discs is made up of alternate rotors and stators, the stator on the ends of the stack are of a somewhat different design, since they only have one friction surface. The disc next to the pistons is called the pressure plate and the disc on the other end of the stack is called the backing plate. The stators are connected to the aircraft axle through a torque tube and the rotors are attached to the wheel through clips and drive lugs. The brake is actuated by hydraulic pressure applied to pistons that compresses the carbon-carbon heat stack. The friction couple at the rotor and stator interfaces provides the torque, which stops the aircraft. The largest portion of the kinetic energy of the aircraft is converted to heat of the brake stack and a small portion of the kinetic energy is converted to mechanical vibrations. The mechanical vibrations known by such names as squeal, judder, whirl, and other noises are undesirable and an attempt is made to keep them to a minimum. The pressure holding the stack faces together (referred to as "rotors tight" condition) minimises the presence of surface oxygen, thus oxidation of the friction surfaces is not very large. An oxidation inhibitor protects the non-friction surfaces. The thermal properties of the heat stack are
controlled by the type of carbon used in the matrix, the type of fibre used as the reinforcing material, the density and by the processing conditions. In addition to being a heat sink the carbon-carbon composite must serve two other purposes: It must provide the structural integrity to transfer torque loads from the carbon to the wheel and axle, and it must provide the friction force at the carbon surface to provide the torque to stop the aircraft.

3.7 Properties of Carbon-Carbon Brake Composites

Some of the properties that differentiate carbon-carbon brake composites from conventional engineering materials are the same as mentioned earlier for general carbon composites and are as follows: high thermal stability, high modulus, good specific strength, light weight, low thermal expansion, high heat absorption and good friction and wear properties. Carbon-carbon composites can be thermally cycled from sub-zero to 3000°C in an inert environment without cracking or fatigue failure. However, like all forms of carbon, carbon-carbon composites are susceptible to oxidative gasification at temperatures above 350°C and rapid oxidation above 500°C.

The properties of carbon-carbon brake composites are controlled by the matrix type, fibre type, and fibre architecture and conditions. The fibres and matrix microstructure generally control properties such as strength and stiffness. The processing conditions and the direction of the fibres in the preform architecture generally controls properties such as density, thermal conductivity, friction coefficient, and wear rate. Much has been written on the mechanical and thermal properties of carbon, the reader is referred to the discussion and references presented in section 3.3.
3.7.1 **Bulk Density**

Carbon-carbon composites are light in weight and thus suitable for aerospace applications. Densities of carbon-carbon brake composites used for brakes typically range from 1.6 g/cm$^3$ to 2.0 g/cm$^3$.

3.7.2 **Porosity**

An understanding of the porosity in carbon-carbon composites is necessary firstly to establish the background for this study, and secondly to properly assess the results of the various characterisations.

In addition to the crystallographic imperfections of carbon mentioned in section 2.2.1, manufactured carbon-carbon composites contain many types of flaws when compared to pure carbon crystals. Among these are voids, which range in size from atomic to large macro cracks (i.e. areas of no matrix in the composite). The effect of porosity and the use of fibre at 1.76 g/cm$^3$ are to reduce the density from the theoretical graphite crystal values of 2.262 g/cm$^3$ to values of 1.6 g/cm$^3$ to 2.0 g/cm$^3$ for carbon-carbon composites.

Since the porosity of carbon has such a large range in type and size, it is necessary to categorise them. Figure 3.8 from “Porosity in Carbon and Graphite” by McEnaney and Mays [79] depicts different types of pores by shape and location in a porous solid.
A classification based on Sing et al and presented in [80] was proposed by the International Union of Pure and Applied Chemistry, IUPAC and is as follows:

- Micropores - width less than 2nm
- Mesopores - width between 2 and 50 nm
- Macropores - width greater than 50 nm

The porosity of a carbon-carbon composite (indirectly the density) is a qualitative measure of several aspects of carbon-carbon composites. The porosity is related to the efficiency of the production, the quality of the material, the mechanical properties and in this study, the relationship to friction and wear will be established. The complexities of a carbon-carbon composite, with its various phases, microstructures, open and closed porosity, cracks and voids, necessitate the use of a number of techniques to produce a characterisation of the pore structure of the material [81,82]. The percentage of macropores has a much more significant effect on carbon properties than mesopores and micropores [82]. This study will concentrate on only the macropores and the appropriate techniques to characterise them described by McEnaney [82].

The physical property of porosity holds the greatest interest for this study and can be affected by several fibre architecture and process conditions. However, the effect of CVD time, which is the most expensive step in the
manufacturing process is the only process varied in this study. It can be assumed that any differences in the pore size or pore size distribution is due to the number of CVD cycles only, all other processing parameters were held constant for all the material used in this study.

3.7.3 Mechanical properties required for Carbon-Carbon Aircraft Brake

This has been covered thoroughly in sections 3.3 and 3.4. Only to add, that according to Patrick, the strength of carbon decreases as the porosity increases [83]. The mechanical properties for the materials used in this study will be measured to verify that this premise is true for these particular materials.

3.7.4 Thermal characteristics

Carbon-carbon composites are carbonaceous and thus their specific heat capacity is the same as traditional carbon materials. At room temperature the specific heat is 0.18 cal/g°C and about 0.5 cal/g°C at 1650°C.

3.7.5 Friction and Wear

The literature on carbon-carbon is extensive, but the literature on the characteristic of friction and wear in carbon-carbon composites is sparse. The friction and wear mechanisms of carbon-carbon composites are not well understood. Previous studies have indicated the importance of wear debris and moisture on the friction and wear performance of carbons [74,84]. The friction values for graphite are influenced by the presence or absence of water vapour that is adsorbed between the carbon basal planes. Thus under vacuum conditions graphite loses its lubricating properties and exhibits high coefficient of friction and high wear rates.

According to Lancaster [74], the low friction exhibited by graphite was first suggested by Bragg in 1928 to be due to easy shear between the carbon basal planes. It was later found that the friction of graphite remained low only in the presence of water or other condensable vapours [84]. In fact, under vacuum,
the friction of carbon was shown to increase by a factor of ten. Therefore, the low coefficient of graphite may not be intrinsic. Bryant et al. in 1964 [85] showed that in vacuum the inter-laminar bonding (cross-linking) of graphite is high. This inter-laminar bonding decreased in the presence of air or water. They also suggested that water might reduce the binding energy of graphite by bonding with the available electrons. They suggested that the lubrication properties of graphite occurred by the adsorption of moisture between the carbon basal planes.

Rowe [86] observed a similar effect but offered an alternative mechanism. He suggested that water or oxygen might intercalate between the carbon basal planes and reduce the pi electron bonding. Intercalation is the ordered bonding of oxygen or water molecules between the carbon basal planes. The low coefficient of friction of graphite could also be due to basal plane contact at the sliding surface. Lancaster [74] indicated that studies by Jenkins showed that there was preferred orientation of the graphite crystallites (basal planes) at the contacting surfaces. He believed the orientation of the carbon basal planes to be parallel to the sliding surfaces. Later studies [87-89] showed a slight tilt of the basal planes to the sliding surfaces. The crystallites were tilted so that the basal plane normals were slightly against the direction of motion at the sliding surface and correspondingly parallel to the resultant force of normal and frictional forces. According to Lancaster [74], the studies of Skinner, in which a tungsten stylus was in contact with single crystal graphite in a basal plane orientation, indicated low friction (0.005-0.02) even in high vacuum. This result seems to contradict the earlier work of Bryant et al. [85], but further investigations showed that with surface damage the friction coefficient was much higher. Skinner suggested that low friction was due to a film of debris and that once the film was disrupted, the coefficient increased.

Longley et. al. [89] found that the degree of graphiticity had little effect on friction. Interpretation of this result is difficult because of the surface modifications that occurred during sliding. Reorientation of basal planes and bulk movement of material between the surfaces resulted in loose debris or
surface films [90]. They believed that it was these surface films that determined the coefficient of friction. They also showed that surface film formation was affected by the carbon structure and the hardness of the counterface material. Strong, non-graphitic carbons required a hard surface to produce the debris to reduce the friction coefficient. Weak, graphitic carbon, on the other hand, formed debris even with a soft counterface material. Therefore, debris would more easily form between two graphitic carbon surfaces than when both carbons were non-graphitic.

Microstructural characterisation of carbon-carbon composites has also proved useful in explaining the friction and wear properties of carbon. Mechanisms of friction film formation and the detailed microstructure of the friction films is not certain. Several techniques have been used to examine the surfaces of worn carbon materials and characterise the structure of the friction films. Previous studies also indicated the importance of a debris film that forms on the friction surface during the wear process [54,55]. Therefore, factors which influence the formation and stability of this wear debris are critical to the understanding of the friction and wear mechanisms of these materials. Murdie et. al. [91] showed that at least two categories of wear debris can be identified at the friction surface of carbon-carbon composites. A Type I debris which has particulate characteristics acts as an abrasive, provides high friction coefficients and high wear rates, and is found predominantly under low energy conditions. Type II debris, which has film-like characteristics, acts as a solid lubricant to provide low coefficient of friction and low wear rates and is found predominantly under high energy conditions.

Murdie et. al. [91] investigated the wear debris of carbon-carbon composite disks used in simulated low and high energy aircraft braking. The disks utilised in their experiments consisted of randomly oriented chopped pitch fibres in a phenolic resin and CVD matrix. They distinguished two types of wear debris. Type I debris was particulate in nature while type II was a film type debris.
Their results showed that after high energy testing, type II film debris was more prevalent on the worn surface. They also showed that after low energy friction testing, the surfaces of the disks had a duller appearance and the debris tended to deposit in pores. This debris was particulate, type I debris [91]. The wear debris appeared purple in polarised light, indicating either basal plane orientation or that the debris was optically isotropic.

SEM examination indicated in his study that the type II film debris was comprised of sub-micron size particles that combined to form the friction film. These particles were too small to be resolved by the optical microscope and thus appear optically isotropic. The type II debris was comprised of small graphitic particles of fibre, resin and CVI.

Murdie et al. [91] also showed that the amount of fibre pullout and debris coverage was related to the orientation of the fibres. Longitudinally oriented fibre bundles had higher amounts of fibre pullout and debris coverage than transverse fibre bundles. The type I, particulate debris tended to collect in the depressions left from individual fibre pullout. In the normal oriented fibre bundles, the debris tended to cover the matrix.

According to the studies on friction and wear of graphites described above, it would appear that both fibre and matrix type are important considerations in friction applications of carbon-carbon composites (aircraft brakes). The structure and strength of these components as well as the bonding between them, are also critical to friction and wear performance.

Graphitic structures (pitch fibres, pitch matrix, anisotropic CVI and graphitized phenolic resins) usually have lower shear properties and weaker bonding characteristics than non-graphitic and turbostratic carbons. The weak bonding between these materials is thought to facilitate their easy removal. The removal of graphitic materials provides loose material for the formation of debris. Because these materials are graphitic, the debris formed has low shear strength and thus can easily be transformed from the particulate (type I) debris into the film (type II) debris.
Conversely, non-graphitic materials like PAN fibres, resins and isotropic CVD carbon have a more cross-linked structure, which provides stronger bonds and higher shear strengths. Removal of these materials through pullout is more difficult during the wear process and therefore these materials are not readily available for debris formation. When these materials are available they tend to remain particulate (type I), due to their high shear strength and therefore do not readily transform into type II debris.

Two known mechanisms of wear exist for carbon materials. These include rubbing (or mechanical) wear occurring from an abrasive mechanism and oxidation of the friction surfaces, both of which are discussed by Chung [92].

Possible mechanisms to lower the wear rate of carbon-carbon brakes include an increase in the friction surface sub layer strength and an increase in the available material undergoing mechanical or abrasive wear and participating in the formation of friction film. This material addition may modify the film layer acting as a solid lubricant, thus reducing material loss. Possible mechanisms associated with oxidative wear may include an effective reduction in the number of active sights at which oxidation takes place. This reduction in active sites may reduce material weight loss due to oxidation and may also contribute in reducing mechanical wear by maintaining structural integrity within the friction sub layer.
4 Chapter 4. Objectives

4.1 Introduction

By aircraft brake performance, friction performance and wear performance are implied. It is the objective in this study to determine if there is a relationship between porosity and the friction coefficient and the wear rate of carbon-carbon composite brake materials. It is also an objective to see if friction and wear are strong functions of the material porosity. The examination of these objectives forms the basis of this study.

4.2 The effects of porosity on friction of Carbon- Carbon composites

The friction models presented in the literature review of chapter 3, above do not address the explicit effects of porosity. It is an objective here to determine if the porosity has a first order effect on the friction performance through its influence on surface contact area. The morphology of the friction surface, wear debris, and friction film will be characterised with respect to porosity to establish the relationship of porosity to the friction and wear after testing subscale brake discs of varying densities at Boeing 767 service energies.

Consider the faces of two carbon discs brought into surface contact. Due to the asperities of the friction surfaces the real contact area of the surfaces is much smaller than the apparent friction surfaces of the discs. Such a contact model for friction in metals has been shown [93, pg74]. It is an objective here to determine if the lower porosity materials will have a larger real contact area and possibly generate a higher stable friction coefficient as compared to the higher porosity materials. If true this should be seen on the friction surfaces of the tested discs as the area of film coverage. If the results of the experiments prove that the stable friction coefficient is a direct result of the real contact surface area (area
covered with friction film) in direct proportion to the initial porosity then this will be proven. The results of Skinner presented by Lancaster [74, indicated that the presence of friction film will cause low friction values, and a disruption of that film will cause an increase in friction. Although it is agreed, the second statement, that disruption of friction film will increase friction, it is proposed here that the first statement is not the case for carbon-carbon composites brakes. It can be postulated that because of the porosity of the carbon material that as the friction film increases the real contact area will increase more for low porosity materials than high porosity materials and the friction will indeed increase.

4.3 Effect of Porosity on Manufacturing Cost

Wear rate is a property of carbon friction material that is significantly tied to the profitability of the related carbon brake, in the operational life cycle cost. Also, the relationship between wear rate and porosity may be practically applied to minimise material operating cost. Assuming a preform type with fixed heat treatment cycles and densification using isothermal CVD (chemical vapour deposition) material porosity is controlled by densification process time. If quantifiable relationships can be determined for material cost as a function of densification process time, wear rate as a function of material porosity and porosity as a function of CVD process time, it becomes possible to minimise material operating cost expressed as material cost per brake landing.

The operating cost expressed as material cost per brake landing is given by

\[
MCBL = \frac{MaterialCost}{Life} = \frac{f(DensificationTime)}{WP} \cdot \frac{WP}{WearRate} = \frac{f(DensificationTime)}{g(Porosity)}
\]  

(4.1)

Which leads to

\[
MCBL = \frac{f(DensificationTime)}{WP} \cdot \frac{WP}{g(h(DensificationTime))}
\]  

(4.2)
MCBL = Material Cost Per Brake Landing
WP = Brake Assembly Wear Pin in inches
f = functional relationship between densification process time and material cost

g = functional relationship between material porosity and material wear rate

h = relationship between densification time and material porosity

A brake assembly that is made up of several discs uses a wear pin of about 37 mm to track the wear. As the discs wear the exposed portion of the wear pin becomes shorter. Within this relationship, both numerator and denominator contain functions of CVD densification time. Therefore, if a local minimum of MCBL exists, it will represent the minimum material cost per brake landing with respect to densification time. One objective of this work is to find the functions, g, the functional relationship between material porosity and material wear rate. Note that the porosity of the brake material might reach a limit where it is uneconomical to continue to densify to reduce the porosity further. It is first necessary to know the friction (effectiveness) required for a level of aircraft stopping energy and at what minimum porosity this friction can be achieved. The effectiveness required for design is a function of each type of braking, such as, taxi, normal landing, and rejected take off. The design effectiveness is usually determined by the rejected take off (RTO) condition since it has the highest energy. The RTO test however destroys the brake material and surrounding brake system structure because of the extreme heat. Thus the pursuit of a function of effectiveness versus porosity in this study will be limited to normal service energies of the Boeing 767 aircraft to allow the testing to be of a reasonable cost. A more complex function would be required to predict the actual MCBL.

4.4 A Wear Model that Might Explain the Effect of Porosity

A wear model that might explain the wear differences (if any) between discs of differing initial porosity should come out of the extensive study of the
worn disk morphology. And the study of the formation and stability of the friction film.

4.5 Summary of Objectives

In summary, the objectives of this study is to establish if there is:

1) a functional relationship for the friction coefficient (effectiveness), versus porosity/density,

2) a functional relationship for the wear rate versus porosity/density,

3) a first order effect of porosity/density on friction coefficient (effectiveness) and/or wear rate,

4) a contact area model of real area versus apparent area, which can model the effect of porosity/density on the friction effectiveness by assuming the real contact area is the area of friction film. This will be proven by the friction surface and friction film morphology of the stable film coverage.

5) a link between the porosity/density and (the CVD processing time), the material cost per landing which can possibly be minimised,

6) a discussion of the morphology of the friction surface and friction film which might help to understand friction and wear mechanisms of carbon-carbon composite friction materials.
5 Chapter 5 Experimental Procedure

5.1 Introduction

The material chosen for this study is typical of non-woven substrate brake materials now used in the industry. The densification method was chemical vapour deposition and follows the manufacturing procedure given in figure 3.3, page 31, and described in section 3.5.2. The resulting carbon-carbon composite brake discs contains no resin. The fibre is PAN and the matrix is CVD with process conditions set to give a microstructure of rough laminar carbon.

5.2 Sample Brake Materials of Various Densities Used in This Study

5.2.1 The Approach to obtain samples

The general approach of this study is based on a simple regression analysis to determine an empirical relationship between material friction coefficient (also effectiveness) and porosity/density. To this end it was required to have brake material samples of a range of porosities consistent with that which could be obtained from the manufacturing plant. Controlling densification time varied material porosity/density, taken as the independent variable. Practically, three full size samples discs were obtained by removing them following 2\textsuperscript{nd} and 3\textsuperscript{rd} and 4\textsuperscript{th} CVD cycles and completing the final heat treatment cycle. Carbon-carbon composites with thicknesses of 1.5 cm or greater will typically have a density gradient from the higher density at surface to lower density in the centre. This property of CVD carbon was utilised to obtain more samples of different densities. Six sets of two dynamometer rings each were fabricated from each disk (a total of six sets of two rings). One set of two rings from each disk was obtained maintaining the “as manufactured” friction surface while a second set of two rings was obtained with the friction surface located near the axial centre line of the disk so as to utilise the density gradient obtained during CVD to provide two samples of similar bulk density and mass but different porosity.
5.2.2 Sample Preparation for Characterisation and Dynamometer Testing

The samples for dynamometer testing were fabricated in the following manner: The very dense exterior (flash) coating from the CVD densification was removed from each full size discs by grinding 0.508mm from each side. The resulting sample discs obtained for experimentation were identified as in Table 5.1.

<table>
<thead>
<tr>
<th>Disc #</th>
<th># Of CVD Cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>401</td>
<td>2</td>
</tr>
<tr>
<td>419</td>
<td>3</td>
</tr>
<tr>
<td>415</td>
<td>4</td>
</tr>
</tbody>
</table>

All three disks were ground to exactly $\frac{1}{2}$ of their original thickness by grinding only one side. The remaining disc surfaces had densities of full size brake disc friction surface and disc centre. The bulk density and thermal diffusivity of each new $\frac{1}{2}$ disk was then measured and the locations of the thermal conductivity measurements were marked for later use.

Figure 5.1. Cutting Diagram
The full size disc of ½ thickness were then cut into four dyno rings, four cores, and two slices using the configuration illustrated in Figure 5.1. One set of dyno rings would use the original friction surface of the ½ disc and the sample number would be appended T for top, while the other set of rings would use as its friction surface the original centre of the full disc, and thus these discs would have lower surface density, these sample numbers would be appended by a C for centre.

The non-friction surfaces of the dyno rings, cores and slices were ground to 10mm sample thicknesses.

The flats for the dyno rings were finally machined to 9.906mm.

The bulk densities of the cores and slices were measured.

Grinding .635mm off the friction surface side of each slice and repeating the measurement of the disc bulk densities led to the surface density. A sample of the friction surface side of one out of each core set was prepared for skeletal density measurement on the helium pycnometer. Another core from each set was polished in preparation for porosity measurement on the imaging microscope. Slices were used for mercury porosimetry.

5.3 Characterisation Techniques for Pre-dynamometer Test Material

5.3.1 Density Analysis

5.3.1.1 Full size disc Air/water Mass Method for Bulk Density

The bulk densities of the three full-size carbon disks were measured at full and half thickness. The densities were calculated using the air mass/water mass method using the following formula:

\[
\text{Density} = \frac{\text{air mass}}{\text{air mass} - \text{water mass}}
\]

(Note that this method works because the full scale disc surfaces are completely closed with CVD and the water can not enter the pores of the discs. The resulting weight will be referred to as "mass").
5.3.1.2 *Surface and bulk density of cores and slices*

The bulk density of the cores and slices was calculated from the dimensional and air mass measurements using the following formulas:

\[
\text{Cores density} = \frac{\text{air mass}}{\pi \times \text{diameter}^2 \times \text{thickness} / 4} \tag{5.2}
\]

\[
\text{Slices density} = \frac{\text{air mass}}{\text{length} \times \text{width} \times \text{thickness}} \tag{5.3}
\]

Each slice was then machined on its friction surface to remove .025” of material. The bulk density of the remaining material was then calculated by the same method as above. The surface density for each slice was then calculated from the pre- and post-machining mass and dimensional measurements using the following formula:

\[
\text{Surface density} = \frac{\text{mass}_{\text{pre}} - \text{mass}_{\text{post}}}{\text{length} \times \text{width} \times \text{thickness}} \tag{5.4}
\]

Where \( l \) is the length

\( w \) is the width

\( t \) is the thickness

5.3.1.3 *Helium Pycnometer Skeletal Density*

Core samples were used for the helium pycnometry measurements. Helium pycnometry measures the skeletal density of the carbon-carbon samples. The helium pycnometer used was Model MPX-1.

In helium pycnometry, the sample is placed in a sealed chamber of known volume. A second sealed chamber, also of known volume, is pressurised with helium to a measured pressure \( P_2 \) and the two chambers are connected. As the helium flows into the second chamber, which is at atmospheric pressure, a pressure drop from \( P_2 \) occurs and the equilibrium pressure \( P_1 \) is recorded. From the ideal gas law, the pressure is inversely proportional to the volume, thus the volume occupied by the helium in the sample chamber \( V_1 \) can be
determined from the ratio of the pressure drop ($P_2 / P_1$) and the known volume of the pressurised chamber ($V_2$) before the two chambers were connected:

$$V_1 = [(P_2 / P_1) - 1] V_2 \quad (5.5)$$

This volume is then subtracted from the known volume of the chambers ($V_c$) to give the volume of the sample ($V_s$):

$$V_s = V_c - V_1 \quad (5.6)$$

Since the gas easily fills even the smallest accessible pores, the method gives the absolute skeletal density of the sample:

$$\text{Absolute skeletal density} = \frac{\text{mass of sample}}{V_s} \quad (5.7)$$

5.3.2 Porosity Characterisation Techniques

Two porosity measurements techniques were utilised in this study. They are mercury porosimetry and optical microscopic image analysis. Each of the methods, the impetus for using them and the procedures of each methodology used will be discussed below. The results will be presented in chapter 6.

5.3.2.1 Mercury Porosimetry

The samples slices were used for the mercury porosimetry measurements. The mercury intrusion porosimeter used in the study is a Porous Material Inc. (PMI) Model PMI 60K-A-4. This instrument was used to measure the average pore size and pore size distribution in the carbon-carbon samples. The equipment measures pores in the 3nm to 300 $\mu$m range. The method gives the fullest amount of information, including values found in other methods used; however, it is somewhat less accurate than direct density measurements and will not detect some of the smaller mesopores and micropores that helium can enter [82].

52
however, it is somewhat less accurate than direct density measurements and will not detect some of the smaller mesopores and micropores that helium can enter [82].

Mercury porosimetry or mercury intrusion porosimetry is based on measuring the volume of mercury forced into the pores of the sample as a function of pressure. The pressure at which intrusion into the pores occurs is inversely proportional to the pore diameter. Given appropriate values for the mercury surface tension and interfacial contact angle, the pore diameter at any applied pressure can be determined. This technique is useful for measuring macrospores in carbon-carbon composites [82]. Mercury has a high contact angle of approximately $140^\circ$ and does not wet the carbon. It forms a meniscus at the pore opening. There must be hydrostatic pressure greater than the vapor pressure outside the meniscus to force the mercury into the pore. The relationship between pressure and pore radii was given by McEnaney and May as the Washburn Equation [95], written as:

\[ \Delta P r = 2v \cos \Theta \]

Where $\Delta P =$ hydrostatic pressure-meniscus pressure
$r =$ radii
$v =$ surface tension of mercury
$\Theta =$ contact angle of mercury

This model assumes the pores are cylindrical. Some limitations of this technique are detailed by Gregg and Sing (1982) and Scholten (1967) [95]: a) surface angle varies with contamination of the mercury; b) the small pores may be crushed; c) all pores are not cylindrical; d) pores shapes such as bottlenecks will be measured as having the radii of the entry to the pores, (see Figure 3.8, b and c) actually measured; e) contact angles range from 130-150°. Despite these limitations, it was shown by Bansal, R.C., et al given in [82] that for pores in the
range of 3.5 nm to 7.5 nm, mercury porosimetry agrees well with other diffusion methods for carbons.

5.3.2.2 Porosity from Optical Imaging Microscopy

An imaging microscope and the Quantimet 500+ software were used to measure the porosity of the friction surfaces and cross sections of samples cut from the cores. Samples from each disk were prepared in the following manner:

- Sample of the top friction surface cut parallel to the friction surface
- Sample of the centre friction surface cut parallel to the friction surface
- Sample of the cross-section perpendicular to the friction surfaces

Twenty-seven images were collected for each of the samples prepared parallel to the friction surfaces. Each set of images was made up of three rows of nine images. Each image and row was adjacent to the previous image and row. The images were "black sharpened" using software image enhancement, given a sample number and stored on the computer's hard drive. The images were then retrieved from the hard disk and "detected" to fill the black areas of the image, which represent the voids in the carbon. Filling the black areas to account for the voids is a qualitative process, which requires some practice. The scale of the detecting number is the same as the number of gray scale graduations (0 - 255).

If the detection is off by 2 or 3, then the error in void area fraction might be around 5 percent. If the detection is off by 10 errors will be closer to 10 percent. This, of course, depends on the number and size of the voids. An image with one large void will show much smaller error sensitivity to detection number than an image with several hundred small voids. Therefore, as long as the detection number is fairly close, the results will be acceptably accurate (±3 to 5). After detection, the image is measured and a line is appended to the detection summary. This provides the number, area fraction, and area percent of voids for that image.
Once all of the images of a particular sample are collected, enhanced, detected and measured, the detection summary provides a list of each image measurements and statistical information (mean, std dev, etc.) for each category of measured parameters.

The samples prepared perpendicular to the friction surfaces were analysed in the same manner as the parallel samples except that 16 images were collected from each friction surface. Each set of 16 images was collected in two rows of 8 images. In each case, the first row was along the friction surface, the second row was one image width way from the friction surface, third row two images from the surface and so forth. A summary of the image data is shown in Table 6.4.

5.3.3 Microstructure Characterisation

Although no change was anticipated, the microstructure of the dynamometer test rings was determined both before and after the friction and wear tests. The method used was to cut one of the cores in half top to bottom to obtain a cross section. One of the half cores was mounted in resin and polished for microscopic examination. Each sample was analysed at 1mm depth from the friction surface and 10mm depth from the friction surface. At each level 3 fields of view of 100x will be analysed at approximately 1mm intervals, figures 5.2 and 5.3.

![Figure 5.2. Bands Chosen for Analysis (Notch Identifies Friction Surface)](image)
Figure 5.3. Fields of View Chosen at Each Cross Section

In each field of view, the CVD microstructure (as quantified by its extinction angle and colour) was determined at 5 predetermined positions. A 10x10 eyepiece graticule is inserted into the body of the optical microscope. This provides a 10x10 grid (A-Jx1-10) to be superimposed over the optical image. The CVD microstructure at positions B2, G3, C6, E8, and H7 are recorded. Therefore 15 measurements are taken at each level giving 30 measurements per core. For six various density specimens, and a rotor and stator per specimen there was a total of 360 areas analysed and photographed.

The extinction angle provides a means of quantifying the preferred orientation of the crystallites in the CVD microstructure. Thus, the polarizer on the microscope can be used to measure the extinction angle. The procedure is as follows:

1). Set up the microscope at around 50x magnification and focus on a fibre with a CVD structure around it. This fibre should be orientated perpendicular to the view plane, i.e. it should look fairly round. See figure 5.4

2). Set the polarizer to 90 degrees. With the $\frac{1}{2} \lambda$ wave retarder plate in place, the fibres and other isotropic areas should look purple. Good rough
laminar (graphitic) CVD structures should have bright blues in the northeast and southwest quadrants, bright yellow-oranges in the northwest and southeast quadrants, and purple in between. This is shown schematically below in figure 5.5. The colours are not this vivid in actual micrographs.
3) Pull the $\frac{1}{2} \lambda$ retarder plate out. The blue and orange areas should turn to white or yellow. The purple areas should become dark.

4). Rotate the polariser in a clockwise direction until the northeast or southwest quadrants (formerly blue areas) are as dark as possible. The difference between the new polariser angle and 90 degrees is the extinction angle for the area under consideration.

The extinction angle is related to the morphology as shown below.

Table 5.2. Relationship between Extinction Angle and CVD Morphology

<table>
<thead>
<tr>
<th>Extinction Angle (Degrees)</th>
<th>CVD Morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-6</td>
<td>Isotropic</td>
</tr>
<tr>
<td>8-12</td>
<td>Smooth Laminar</td>
</tr>
<tr>
<td>15-23</td>
<td>Rough Laminar</td>
</tr>
</tbody>
</table>

Measurements of the CVD thickness can be obtained by comparing the deposits to the diameter of the fibre. The average fibre (PAN) has a diameter of seven microns. Intrabundle CVD layers tend to be between one and three microns thick, while interbundle structures tend to be between 6 and 15 microns thick after the third CVD cycle used in this study.
5.3.4 Thermal Diffusivity

The thermal diffusivity of each half thickness, full size disk was measured using the AlliedSignal (Honeywell) thermal diffusivity tester. This measurement is a thermo-analytical method which measures the thermal diffusivity of a solid brake disc at constant temperature while monitoring the temperature increase of the sample caused by a flash of Zeon light. A schematic diagram of the thermal diffusivity apparatus is shown in figure 5.6.

![Schematic of the Thermal Diffusivity Tester](image)

Figure 5.6. Schematic of the Thermal Diffusivity Tester

With this equipment, the irradiation time (shutter speed typically 15 seconds) is set to produce a two-degree rise in rear surface temperature. It often requires several retests to obtain the two-degree rise. The data, which are an average of all of the sample measurements, are presented in Table 6.3.4.1. The thermal diffusivity is related to the conductivity by
\[ \kappa = \alpha C_p \rho \]  
(5.9)

Where: \( \kappa \) is the thermal conductivity (W/mK),
\( \alpha \) is the thermal diffusivity (m\(^2\)/s),
\( C_p \) is the heat capacity (Ws/gK),
\( \rho \) is the density (g/m\(^3\)).

Knowing the heat capacity, density, and thermal diffusivity, thermal conductivity can thus be obtained.

### 5.3.5 Mechanical Properties Tests

The flexure, shear and compression strengths were measured for the high and low porosity samples. A diagram of the properties directions with respect to the dynamometer discs i.d. shown in figure 5.7
The mechanical testing followed ASTM D-790-00, for flexure, ASTM D-645-96, for compression, and an ASTM C 1292-95a for shear strengths. Each test was completed on 5 specimens for 401C and 415 T the highest and lowest porosity samples respectively. The range, average, and standard deviation were recorded and will be reported in section 6.3.5.

5.4 Friction and Wear Dynamometer Test and Post Test Characterisation

This section will describe the friction and wear test and all the characterisation completed after the first friction and wear test.
5.4.1 The Friction and Wear Tests

The material friction effectiveness and wear rates were determined by a subscale dynamometer test of a ring set 8.57 cm o.d and 5.4 cm i.d. on the Honeywell 43 cm inertia dynamometer utilising a single friction interface.

5.4.1.1 Test Equipment

The geometry of the subscale dynamometer test apparatus is depicted in Figure 5.8 schematically along with photographs in Figures 5.9 and 5.10.

![Schematic of The 43 cm Dynamometer](image)

Figure 5.8. Schematic of The 43 cm Dynamometer

The piston applies pressure to the stator through a load cell, thus the normal load is measured directly for each stop. The flywheel plates are the appropriate mass to give the required energy for the stop. The motor shaft is spun up to the required speed, and then the motor disengaged. The piston pressure is immediately applied to the stator to put the disc surfaces in contact for the stop. The torque arm (not shown) is on a pressure plate between the load cell and the stator. Velocity, pressure, stator temperature, stop time, and torque are all
measured and stored on the control computer at a sampling rate of 15 samples per second.

Figure 5.9. The Dynamometer

Figure 5.10. The Dynamometer Shaft with Test Specimens

5.4.1.2 Dynamometer Test Procedures

Six sets of dynamometer rings ranging in bulk density from 1.567 g/cm$^3$ to 1.757 g/cm$^3$ (Table 5.3) were friction and wear tested on the above dynamometer. A photograph of the rings is Figure 5.11
Table 5.3 Sample Ring Sets Used in Friction and Wear Tests

<table>
<thead>
<tr>
<th>Sample</th>
<th>CVD</th>
<th>Bulk Density (gram/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>401-centre</td>
<td>2$^{nd}$</td>
<td>1.567</td>
</tr>
<tr>
<td>401-top</td>
<td>2$^{nd}$</td>
<td>1.621</td>
</tr>
<tr>
<td>419-centre</td>
<td>3$^{rd}$</td>
<td>1.696</td>
</tr>
<tr>
<td>419-top</td>
<td>3$^{rd}$</td>
<td>1.722</td>
</tr>
<tr>
<td>415-centre</td>
<td>4$^{th}$</td>
<td>1.718</td>
</tr>
<tr>
<td>415-top</td>
<td>4$^{th}$</td>
<td>1.757</td>
</tr>
</tbody>
</table>

Figure 5.11. Dynamometer Test Rings

Each set of dynamometer rings completed 20 break-in stops and 200 service landing stops simulating the area loading and linear velocity at the mean friction radius of a 767-300, 100% service energy stop. The test conditions are provided in Table 5.4.
Table 5.4. Dynamometer Test Conditions

<table>
<thead>
<tr>
<th></th>
<th>Service</th>
<th>Energy landing</th>
<th>Stop</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Velocity at the MFR (km/h)</td>
<td></td>
<td>123</td>
<td></td>
</tr>
<tr>
<td>Area Loading (J/cm²)</td>
<td></td>
<td>2054</td>
<td></td>
</tr>
<tr>
<td>Mass Loading (kJ/kg)</td>
<td></td>
<td>463.3</td>
<td></td>
</tr>
<tr>
<td>Average Normal Load (N)</td>
<td></td>
<td>1334.5</td>
<td></td>
</tr>
<tr>
<td>Target Stop Time (sec)</td>
<td></td>
<td>25</td>
<td></td>
</tr>
</tbody>
</table>

MFR is the mean friction radius.

5.4.1.3 The Friction Measurement

In this study, only dynamic conditions are considered for the friction measurement; static friction is not calculated. The terms friction coefficient and system effectiveness, or just effectiveness will be synonyms. An axial or normal load is applied by hydraulic pressure through the stationary disk (stator) to the rotating disc (rotor) by the pressure plate. The relationship between torque and frictional force is given by the following expression.

\[ T = F_r R_a \]  \hspace{1cm} (5.10)

where \( T \) is the torque

\( R_a \) is the average radius of the test disc

\( F_r \) is the friction force

The average radius of the disc is

\[ R_a = \frac{\text{o.d.} + \text{i.d.}}{4} \]  \hspace{1cm} (5.11)

The frictional force is also proportional to the normal force (axial brake force), \( L \) and equal through a constant of proportionally \( \mu \) defined as the friction effectiveness.

\[ F_r = \mu L \]  \hspace{1cm} (5.12)

where \( L \) is the normal load
\( \mu \) is the effectiveness.

Substitution of equations 5.10 and 5.11 into equation 5.12 and solving for \( \mu \) determines the instantaneous effectiveness for the system.

\[
\mu = T / (R_a L)
\]  
\[(5.13)\]

5.4.1.4 Wear Measurements

The wear of the friction surfaces was determined by both sample weight loss (measured in mg) and linear wear (measured in inches).

5.4.1.4.1 Thickness Technique

The discs' thicknesses are measured in four quadrants of the discs, at 0, 20, 120, and 220 stops. The average of the four measurements is used as the thickness in the wear calculation; that is simply the average difference in thickness between the stops divided by the number of stops. The result is the wear rate in mm per stop.

5.4.1.4.2 Weight Technique

The discs are weighed at 0, 20, 120, and 220 stops. The number of stops to give the wear rate in terms of mg per stop divides the difference in weights between measurement stops.

5.4.2 Microscopic Characterisation after Friction and Wear Testing

The objective is to analyse, by image analysis, selected specimens of low and high porosity materials after friction and wear testing to measure the area \% porosity of filled, open and partially filled pores, and to characterise the morphologies after testing. Both optical and scanning electron microscopes were used. The technique used by McEnaney and Mays [82] for optical microscopic
image analysis, figure 5.12, is also used in this study for optical and SEM image analysis.

![Diagram of Image Analysis System](image)

**Figure 5.12. Schematic Diagram of Computer Image Analysis [82]**

### 5.4.2.1 Image Analysis after the Friction and Wear Testing

Both optical and SEM/QIA (scanning electron microscope/quantitative image analysis) were used. The percent porosity (measured as area %) of each sample was measured by image analysis using an optical microscope using the same image analysis used prior to testing, described above. The measurements were carried out using a 2.5x magnification objective lens, which imaged an area of 2.7 mm x 3.0 mm. Twelve images were acquired from each section and the area % porosity was measured for each image area. These values and standard deviations are depicted graphically in section 6.4.2.1.

The SEM analysis on the post-tested specimens was limited to the highest and lowest porosity specimens. The instrumentation used for the QIA study was the JEOL 840 SEM and the Noran (formerly Tracor Northern) Tn-8502 image analysis system. The selection of the area % is based on the gray scale of 0-255 shades of gray. The objective here is to separate the pores (filled or partially filled) the film and wear particulate to determine the makeup of the worn disc surfaces and cross sections. Images are also taken at 500x adjacent to the 100x
range. These image areas are to be used to determine the friction film coverage. The results of the image analysis are presented in section 6.4.2.1.

5.4.2.2 Microscopic Study of the Morphologies of the worn discs

The surface and cross section morphologies of the lowest and highest porosity sample, 401 C and 414 T were studied by both optical and SEM. The radial cross section morphologies of samples 401C and 415T were studied using optical microscopy. The sizes (area) of the polished sections were 15 mm from o.d. to i.d. not including key and 10 mm from surface to bulk (thickness). Three polished sections were studied: 415T rotor, 415T stator and 401C rotor. Each sample was cut from the respective worn sub-scale disc, embedded in epoxy and polished to a final finish of 0.04-micron silica.

Both the JEOL 840 SEM and The JEOL 6300F field emission SEMs were used to study the morphology of the samples after the completed dynamometer testing. The images were digitised and recorded on a computer disc for subsequent image analysis. The results of this morphology study are presented in section 6.4.2.2

5.4.3 Photon Correlation Spectrography (PCS)

The samples used in the PCS analysis were taken from the sample preparation for the transmission electron microscope (TEM) described below for the TEM analysis. The part of the sample that had formed a wafer was reserved for TEM studies while the loose material was used for the Photo Correlation Spectroscopy light scattering experiments.

Photo Correlation Spectroscopy (PCS) used to determine the translational diffusion coefficients of material dissolved or suspended in liquid median by measuring the time-dependent fluctuations of the light scattered by the material. Typically, these diffusion coefficients are used to determine the sizes of the particles causing the fluctuations. The ‘spectra’ consist of time autocorrelation
functions of the number of photons collected during fixed time intervals (from 0.1 to 1 sec). The decay constants of these spectra are directly proportional to the diffusion coefficient of the material; a Laplace transform is used to convert these spectra (sums of exponentials) into a diffusion coefficient distribution that can be converted into a size distribution. The technique is good for particle sizes ranging from 1 nm to 1 μm. The equipment was a model 1021 digital autocorrelation; with the 50mW HeNe laser replaced with a higher power water-cooled krypton laser.

5.4.4 Morphology by Transmission Electron Microscopy (TEM)

It is of interest to understand the morphology of the friction film from the lowest porosity and highest porosity samples. The TEM was used to determine if there were differences in the morphology of the friction films formed from friction and wear testing the various porosity samples. Friction film materials were typically made up of very fine (small particle sizes <1 μm) material that cannot be completely resolved by optical or scanning electron microscopes [91]. Both a Hitachi and a Jeol (high resolution) transmission electron microscope were used.

Stripping the friction surface of the lowest and highest porosity samples using replicating tape collected samples for TEM analysis. Standard cellulose acetate replicating tape was used. The friction surface was first wetted with acetone and then a sheet of the replicating tape was placed on top. Compressed air was blown on the film to press it gently against the friction surface. After allowing the acetone to dry for about 10 minutes, the film was stripped. The same procedure was performed a second time to obtain a second peel. Samples of friction film from 401 C (I) and 415 T (I) consisted of the first peeling from the disc friction surface while samples 401 C (II) and 415 T (II) resulted from a second peeling. To dissolve the replicating tape, the individual sheets were immersed in subsequent baths of clean acetone. After three or four baths the
material. It was noticed that in samples 401 C (I) and 415 T (I), the residue consisted of a thin cake or wafer as well as loose material. Presumably the wafer or cake had formed because of incomplete dissolution of the replicating tape. The part of the sample that had formed a wafer was reserved for the TEM studies. Since it was suspected that some of the replicating tape remained in the sample, the TEM samples were allowed to sit in acetone for several days. Then, after several additional rinses in clean acetone, the final acetone was replaced with methanol to obtain a final dispersion.

5.4.4 Electron Energy Loss Spectrograph (EELS)

The samples, used in TEM analysis were also used for an Electron Energy Loss Spectrograph (EELS) analysis. This was done to detect whether or not there was a presence of diamond. The EELS process and an explanation of the principles of operation can be found in [96-99]. The purpose for this study was only to compare the spectra of the friction film components to the spectrum for diamond.

5.4.5 X-Ray Diffraction (XRD)

Many good basic treatments of X-ray diffraction are given in Characterisation of Materials [100. chapter 32]. Samples of friction film and bulk carbon were examined by x-ray diffraction to determine the d_{002} spacing of interlayers, and L_e stack height.

5.4.6 Micro Raman Spectrography

The vibrational characteristics of carbonaceous material exhibited in Raman spectra allow distinct resonances from ordered and disordered regions of the material to be identified [101-104]. Ordered graphitic layers show resonance between 1580 cm^{-1} and 1600 cm^{-1}. This band narrows and moves closer to 1580 cm^{-1} (the resonance for graphite) with graphitization. A second resonance 1350 cm^{-1}-1380 cm^{-1} decreases with graphitization and is assigned as a defect band [5, p29].
cm\(^{-1}\)-1380 cm\(^{-1}\) decreases with graphitization and is assigned as a defect band [5, p29].

In this study, Raman data were collected using a micro-Raman spectrometer with a spatial resolution of 1 \(\mu\)m. The objective of this analysis was to obtain the degree of graphitization of the components (matrix, fibre, and interface) of the carbon-carbon composite brake discs. The analysis was performed on the optical microscope polished samples. Data was collected using a charge coupled device (CCD detector for collecting light and converting to digital data). Data collection times were 4 min. per spectrum.

The Raman spectrum of graphite consists of the two major peaks, mentioned above [5], 1580 cm\(^{-1}\) and 1360 cm\(^{-1}\). The first is the peak for graphite and the second represents defects in the plane. The ratio of the intensities of these two peaks is proportional to the \(L_a\) spacing (stack width) in randomly oriented samples. For Raman measurements, this procedure generates an average \(L_a\) value for the microstructure present in the area under the examination (this is typically 100 \(\mu\)m diameter). This cannot be said of the micro-Raman experiments that might be affected by the detailed geometry and orientation of the microstructure in the very small area under examination. It was first necessary to calibrate the measurements by using homogenous materials of known crystallite sizes. It is believed that the \(L_a\) would at least be relative for different surface morphologies, if not absolutely exact.

In this study, the differences of the degree of graphitization of the composite components: fibre, fibre matrix interface and matrix CVD were of the most interest. After testing, an average over a non-homogenous area, containing more than one carbon type, using standard Raman spectography, would not address this interest.

The attempt to measure the friction film and other surface particulate after testing did not work out; the procedures for the micro Raman were in early
development during this study. To do a micro Raman study of friction film and surface particulate is one of the recommendations for future work discussed in chapter 8.

5.5 The Second Friction and Wear Test

After the first friction and wear test it was felt that more evidence was needed to prove out the contact area model for the explanation of the higher stable friction coefficient for the lower porosity discs. Thus a second test was conducted to view the friction surface at various points during the test and not just at the beginning and end as was done in the first part of the study. This new test consisted of grinding down the lowest porosity and highest porosity dynamometer rings' friction surfaces that were not destroyed for other characterisations. Each disc set was notched on the ID and OD in two radial areas. One radial strip had fibre in the radial direction and the other radial strip had fibre in the chordal area.

The friction and wear test was then repeated. The test was stopped at 20, 70, 120, 170, and 220 stops. Sets of stereoscope photographs were taken at each of these stops for the lowest and highest porosity samples and in regions of chordal and radial fibre. The ID and OD notches guaranteed that the same radial strips would be photographed after each stop. Two complete sets of photographic radial strips from ID to OD for the chordal and radial fibre direction for each ring set of low and high porosity were then made into a complete radial collage. These collages were made up from the microphotographs taken at 5x and stitched together with the Adobe Photoshop 6.0 software. There were six to eight microphotographs in each collage. After each stop, great care was exercised to ensure that the friction surfaces would not be disturbed between the test segments and that the discs would be placed back in the dynamometer aligned just as they came off. These collages are presented in section 6.5 and discussed in detail in chapter 7.
6 Chapter 6 Results

6.1 Introduction

All of the experimental data presented in this chapter can be related to the corresponding section numbers used in chapter 5. By use of the corresponding section numbers, the reader can refer back to the procedures and equipment descriptions. The intent of this chapter is to present the data without discussion of these results. The procedures presented in chapter 5 were followed exactly as written unless noted with this data. The results are broken down into three main sections the same as in chapter 5: the pretest results, the post dynamometer results, and the results after the second test. Figures 6.24 through 6.31 define friction film and particulate by examples.

6.2 Sample Preparation

Samples were prepared as outlined in section 5.2.

6.3 Characterisation Results Prior to Dynamometer Testing

6.3.1 Density Results

Density was measured for the full size discs, after densification and final machining. The densities were measured for the test rings and property test samples immediately before the test or experiment for which they were intended.

6.3.1.1 Full size Discs

The pre test bulk density for each full size brake disc was measured and calculated as outlined in section 5.3.1.1 and listed in Table 6.1.
Table 6.1. Full Size Brake Discs

<table>
<thead>
<tr>
<th>Disc #</th>
<th>Density (g/cm³)</th>
<th>Number of CVD Cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>401</td>
<td>1.608</td>
<td>2</td>
</tr>
<tr>
<td>419</td>
<td>1.710</td>
<td>3</td>
</tr>
<tr>
<td>415</td>
<td>1.735</td>
<td>4</td>
</tr>
</tbody>
</table>

6.3.1.2 Surface and bulk densities of the cores and slices

The densities of the cores and slices, to characterise the pre test conditions, were measured/calculated using air/water weights as outlined in section 5.3.1.1 and 5.3.1.2 and listed in Table 6.2.

Table 6.2. Bulk and Surface Densities

<table>
<thead>
<tr>
<th>Sample</th>
<th>CVD cycles g/cm³</th>
<th>Bulk Density g/cm³</th>
<th>Surface density g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>401 Centre</td>
<td>2</td>
<td>1.567</td>
<td>1.572</td>
</tr>
<tr>
<td>401 Top</td>
<td>2</td>
<td>1.621</td>
<td>1.775</td>
</tr>
<tr>
<td>419 Centre</td>
<td>3</td>
<td>1.696</td>
<td>1.708</td>
</tr>
<tr>
<td>419 Top</td>
<td>3</td>
<td>1.722</td>
<td>1.873</td>
</tr>
<tr>
<td>415 Centre</td>
<td>4</td>
<td>1.718</td>
<td>1.733</td>
</tr>
<tr>
<td>415 Top</td>
<td>4</td>
<td>1.757</td>
<td>1.922</td>
</tr>
</tbody>
</table>

6.3.2 Porosity Characterization Results

6.3.2.1 Helium Pycnometry

The results of the measurement of the skeletal density from He Pycnometry followed the procedure of section 5.3.2.1 and are listed in the sixth column of Table 6.3.

6.3.2.2 Mercury Porosimetry

The results of the measurement of the mercury porosity are listed in Table 6.3. There were two samples tested from a slice from each half-thickness...
full size disc yielding a total of 12 samples, shown in Table 6.3. In this table the absolute density is calculated from mercury porosimetry, whereas the skeletal density is from He pycnometry. Also, the mean pore diameter is a volume average.

Table 6.3. Mercury Porosimetry and Helium Pycnometry of Pretest Carbon-Carbon

<table>
<thead>
<tr>
<th>Sample</th>
<th>Position</th>
<th>Pore Vol. cm$^3$/g</th>
<th>% Pore Vol.</th>
<th>Piece Density g/cm$^3$</th>
<th>Skeletal Density g/cm$^3$</th>
<th>Pore Area m$^2$/g</th>
<th>Mean Pore Diam. μm</th>
<th>Absolute Density g/cm$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>401C</td>
<td>Centre</td>
<td>0.151</td>
<td>23.2%</td>
<td>1.53</td>
<td>2.00</td>
<td>2.3</td>
<td>0.268</td>
<td>2.002</td>
</tr>
<tr>
<td>401C</td>
<td>Outer</td>
<td>0.142</td>
<td>22.5%</td>
<td>1.58</td>
<td>2.04</td>
<td>2.5</td>
<td>0.228</td>
<td>2.012</td>
</tr>
<tr>
<td>401T</td>
<td>Centre</td>
<td>0.137</td>
<td>21.6%</td>
<td>1.58</td>
<td>2.01</td>
<td>2.8</td>
<td>0.195</td>
<td>2.000</td>
</tr>
<tr>
<td>401T</td>
<td>Outer</td>
<td>0.113</td>
<td>18.6%</td>
<td>1.65</td>
<td>2.03</td>
<td>3.1</td>
<td>0.143</td>
<td>2.014</td>
</tr>
<tr>
<td>415C</td>
<td>Centre</td>
<td>0.078</td>
<td>13.5%</td>
<td>1.73</td>
<td>2.00</td>
<td>4.9</td>
<td>0.064</td>
<td>1.985</td>
</tr>
<tr>
<td>415C</td>
<td>Outer</td>
<td>0.071</td>
<td>12.4%</td>
<td>1.76</td>
<td>2.01</td>
<td>4.2</td>
<td>0.068</td>
<td>2.000</td>
</tr>
<tr>
<td>415T</td>
<td>Centre</td>
<td>0.069</td>
<td>11.9%</td>
<td>1.73</td>
<td>1.96</td>
<td>4.7</td>
<td>0.059</td>
<td>1.964</td>
</tr>
<tr>
<td>415T</td>
<td>Outer</td>
<td>0.062</td>
<td>10.7%</td>
<td>1.73</td>
<td>1.93</td>
<td>4.7</td>
<td>0.053</td>
<td>1.973</td>
</tr>
<tr>
<td>419C</td>
<td>Centre</td>
<td>0.095</td>
<td>16.2%</td>
<td>1.70</td>
<td>2.02</td>
<td>4.4</td>
<td>0.087</td>
<td>1.994</td>
</tr>
<tr>
<td>419C</td>
<td>Outer</td>
<td>0.087</td>
<td>15.0%</td>
<td>1.72</td>
<td>2.03</td>
<td>4.0</td>
<td>0.086</td>
<td>2.006</td>
</tr>
<tr>
<td>419T</td>
<td>Centre</td>
<td>0.086</td>
<td>14.6%</td>
<td>1.70</td>
<td>2.00</td>
<td>4.3</td>
<td>0.080</td>
<td>1.969</td>
</tr>
<tr>
<td>419T</td>
<td>Outer</td>
<td>0.074</td>
<td>12.8%</td>
<td>1.72</td>
<td>1.97</td>
<td>4.3</td>
<td>0.068</td>
<td>1.970</td>
</tr>
</tbody>
</table>

6.3.2.3 Porosity from optical imaging microscopy

The results of the optical imaging porosity measurements are Table 6.4.
Table 6.4. Porosity from Imaging Microscopy

<table>
<thead>
<tr>
<th>Sample</th>
<th>CVD</th>
<th>Orientation</th>
<th>Position</th>
<th># Frames</th>
<th>Area % Mean</th>
<th>Std Dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>401 C</td>
<td>2nd</td>
<td>Surface</td>
<td>Centre</td>
<td>27</td>
<td>50</td>
<td>7.7</td>
</tr>
<tr>
<td>401 T</td>
<td>2nd</td>
<td>Surface</td>
<td>Top</td>
<td>27</td>
<td>39</td>
<td>7.1</td>
</tr>
<tr>
<td>401 C</td>
<td>2nd</td>
<td>X-section</td>
<td>Centre</td>
<td>16</td>
<td>44</td>
<td>4.7</td>
</tr>
<tr>
<td>401 T</td>
<td>2nd</td>
<td>X-section</td>
<td>Top</td>
<td>6</td>
<td>33</td>
<td>3.3</td>
</tr>
<tr>
<td>419 C</td>
<td>3rd</td>
<td>Surface</td>
<td>Centre</td>
<td>27</td>
<td>41</td>
<td>9.5</td>
</tr>
<tr>
<td>419 T</td>
<td>3rd</td>
<td>Surface</td>
<td>Top</td>
<td>27</td>
<td>20</td>
<td>3.8</td>
</tr>
<tr>
<td>419 C</td>
<td>3rd</td>
<td>X-section</td>
<td>Centre</td>
<td>16</td>
<td>28</td>
<td>4.8</td>
</tr>
<tr>
<td>419 T</td>
<td>3rd</td>
<td>X-section</td>
<td>Top</td>
<td>16</td>
<td>18</td>
<td>2.8</td>
</tr>
<tr>
<td>415 C</td>
<td>4th</td>
<td>Surface</td>
<td>Centre</td>
<td>27</td>
<td>26</td>
<td>2.9</td>
</tr>
<tr>
<td>415 T</td>
<td>4th</td>
<td>Surface</td>
<td>Top</td>
<td>27</td>
<td>21</td>
<td>2.4</td>
</tr>
<tr>
<td>415 C</td>
<td>4th</td>
<td>X-section</td>
<td>Centre</td>
<td>18</td>
<td>25</td>
<td>5</td>
</tr>
<tr>
<td>415 T</td>
<td>4th</td>
<td>X-section</td>
<td>Top</td>
<td>19</td>
<td>23</td>
<td>4.8</td>
</tr>
</tbody>
</table>

6.3.3 Microstructure from optical microscope

A micrograph from the optical microscope is shown in Figure 6.1. The interference colours, blue, purple, and yellow are typical of rough laminar microstructure. There were 30 areas analysed for each of 12 specimens cut from the various density discs yielding 360 photographs. Each of the 360 measurements gave an extinction angle greater than 20 degrees. This is in the range of highly graphitic carbon known as rough laminar.
6.3.4 Thermal Diffusivity

The results of the thermal diffusivity measurements are shown in Table 6.5.

Table 6.5. Sample Thermal Diffusivity

<table>
<thead>
<tr>
<th>Sample</th>
<th>CVD</th>
<th>Thermal Diffusivity (cm²/sec)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>401</td>
<td>2nd</td>
<td>0.297</td>
<td>0.023</td>
</tr>
<tr>
<td>419</td>
<td>3rd</td>
<td>0.369</td>
<td>0.026</td>
</tr>
<tr>
<td>415</td>
<td>4th</td>
<td>0.320</td>
<td>0.032</td>
</tr>
</tbody>
</table>

6.3.5 Mechanical Properties

The mechanical properties measured for the highest and lowest porosity discs are given in Table 6.6
### Table 6.6. Mechanical

<table>
<thead>
<tr>
<th>Mechanical Property</th>
<th>Highest Porosity Disc</th>
<th>Lowest Porosity Disc</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean MPa</td>
<td>STD. DEV MPa</td>
</tr>
<tr>
<td>CS1</td>
<td>60.05</td>
<td>6.41</td>
</tr>
<tr>
<td>CS2</td>
<td>88.67</td>
<td>9.45</td>
</tr>
<tr>
<td>CS3</td>
<td>90.8</td>
<td>7.37</td>
</tr>
<tr>
<td>S1T</td>
<td>26.88</td>
<td>6.34</td>
</tr>
<tr>
<td>S3T</td>
<td>22.48</td>
<td>4.76</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FIT</td>
<td>86.5</td>
<td>6.55</td>
</tr>
<tr>
<td>F3T</td>
<td>54.29</td>
<td>5.65</td>
</tr>
</tbody>
</table>

*One statistical outlier eliminated

### 6.4 Characterisation Results after First Dynamometer Test

The surface appearance of a typical brake discs after the dynamometer test is shown in Figures 6.2 at 0.7x from a stereoscope. One disc set number 415-T during the first dynamometer test developed "record grooving" as shown in Figure 6.3.
Figure 6.2. Typical Worn Surface of 401 T Rotor after 220 Stops

Figure 6.3. “Record Grooved” Surface of Sample 415 T Rotor
Figures 6.4 and 6.5 show the torque, pressure, and velocity versus time curves at 60 and 200 stops for the lowest porosity disc 415 T during dynamometer testing. Figures 6.6 and 6.7 show the same stops (60 and 200) for the highest porosity material 401 C. There are similar curves for each stop and each disc tested. For the six sets of test rings representing six variations in porosity there are 1320 such graphs. These four were chosen to show the variation in torque and thus effectiveness for low porosity and high porosity brake discs. The effectiveness after stop 20, stop 120 and stop 220 is given in Table 6.7 for all tested materials. These representative graphs from stops 60 and 200 for each of the highest and lowest porosity materials were chosen to show the variations in torque as the friction film stabilised with number of stops. All the parameters are averaged over velocity to calculate an average effectiveness.
Figure 6.4. Low Porosity Friction and Wear Test at 60 Stops

Figure 6.5. Low Porosity Friction and Wear Test at 200 Stops
Figure 6.6. High Porosity Friction and Wear Test at 60 Stops

Figure 6.7. High Porosity Friction and Wear Test at 200 Stops
6.4.1 The Friction Test and Wear measurement Results

The friction performances of the discs with different surface and bulk densities are show in Table 6.7. Figures 6.8, graphically shows the relationship between bulk density and effectiveness (friction), along with the best cubic polynomial curve fit of friction as a function of density. Figure 6.9 shows the density vs. friction relationship for surface density. Figures 6.10 and 6.11 shows the same relationships between surface density and effectiveness with bar graphs, each set of bars represents the number of CVD cycles needed to achieve these densities. The number of cycles is 2 to 4 from left to right.

Table 6.7. Effectiveness at Specific Number of Stops

<table>
<thead>
<tr>
<th>Material</th>
<th>Bulk Density (g/cm$^3$)</th>
<th>Surface Density (g/cm$^3$)</th>
<th>Effectiveness at Stop Number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>20 stops</td>
</tr>
<tr>
<td>401C</td>
<td>1.567</td>
<td>1.572</td>
<td>0.268</td>
</tr>
<tr>
<td>401T</td>
<td>1.621</td>
<td>1.775</td>
<td>0.340</td>
</tr>
<tr>
<td>419C</td>
<td>1.696</td>
<td>1.708</td>
<td>0.351</td>
</tr>
<tr>
<td>419T</td>
<td>1.722</td>
<td>1.873</td>
<td>0.302</td>
</tr>
<tr>
<td>415C</td>
<td>1.718</td>
<td>1.733</td>
<td>0.399</td>
</tr>
<tr>
<td>415T</td>
<td>1.757</td>
<td>1.922</td>
<td>0.489</td>
</tr>
</tbody>
</table>
Figure 6.8. Effect of Bulk Density vs. Friction at 120 and 220 Stops

Figure 6.9. Effect of Surface Density on Friction at 120 and 220 Stops
Figure 6.10. Bulk Density (CVD Cycles) vs. Friction for 120 and 220 Stops

Figure 6.11. Surface Density (CVD Cycles) vs. Friction for 120 and 220 Stops
The results of the wear measurements are given as a function of density (Figure 6.12) by the weight method and (Figure 6.13) by the thickness method, again for 120 and 220 stops.

![Figure 6.12. Wear vs. Bulk Density Mass Technique](image)

![Figure 6.13. Wear vs. Bulk Density for the Thickness Technique](image)
6.4.2 Microscopic Characterisation Results

A SEM microscope was used to complete the porosity image analysis, which measured the porosity and pore type, and an optical microscopy, SEM, and TEM were used for the morphology analysis.

6.4.2.1 Image Analysis

6.4.2.1.1 Optical Image Analysis

The overall surface and cross section porosity was first measured by optical microscopy image analysis. Figure 6.14 contains optical micrograph montages taken from the polished cross sections showing the variations in pore structure from the worn surface to the non-friction surface of the discs.

Table 6.8 shows the percentage porosity measured by optical Image analysis. Figures 6.15, 6.16, and 6.17 show the distribution of % porosity across the sample from i.d. to o.d. and from the worn surface to the bulk. Figures 6.18, 6.19, and 6.20 show the statistical variations with respect to locations of pores within a sample. The friction surfaces are at the top of the photographs.

![Figure 6.14. Optical Micrograph Montages](image)

401 stator 415 stator 415 rotor
### Table 6.8. Percentage Porosity Measured by Image Analysis

<table>
<thead>
<tr>
<th>Area</th>
<th>415T Rotor</th>
<th>415T Stator</th>
<th>401C Rotor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.4</td>
<td>11.7</td>
<td>30.7</td>
</tr>
<tr>
<td>2</td>
<td>11.1</td>
<td>12.4</td>
<td>27.8</td>
</tr>
<tr>
<td>3</td>
<td>12.3</td>
<td>12.9</td>
<td>18.7</td>
</tr>
<tr>
<td>4</td>
<td>19.5</td>
<td>11.6</td>
<td>29.2</td>
</tr>
<tr>
<td>5</td>
<td>11.6</td>
<td>13.8</td>
<td>25.0</td>
</tr>
<tr>
<td>6</td>
<td>10.8</td>
<td>13.8</td>
<td>18.7</td>
</tr>
<tr>
<td>7</td>
<td>21.0</td>
<td>13.9</td>
<td>32.0</td>
</tr>
<tr>
<td>8</td>
<td>12.4</td>
<td>15.4</td>
<td>27.8</td>
</tr>
<tr>
<td>9</td>
<td>11.3</td>
<td>14.5</td>
<td>22.0</td>
</tr>
<tr>
<td>10</td>
<td>21.1</td>
<td>11.9</td>
<td>31.7</td>
</tr>
<tr>
<td>11</td>
<td>12.3</td>
<td>17.5</td>
<td>19.7</td>
</tr>
<tr>
<td>12</td>
<td>12.9</td>
<td>13.8</td>
<td>21.1</td>
</tr>
<tr>
<td>Average</td>
<td>14.3</td>
<td>13.6</td>
<td>25.4</td>
</tr>
<tr>
<td>Std. Dev.</td>
<td>3.9</td>
<td>1.7</td>
<td>5.1</td>
</tr>
</tbody>
</table>
6.4.2 Microscopic Characterisation Results

A SEM microscope was used to complete the porosity image analysis, which measured the porosity and pore type, and an optical microscopy, SEM, and TEM were used for the morphology analysis.

6.4.2.1 Image Analysis

6.4.2.1.1 Optical Image Analysis

The overall surface and cross section porosity was first measured by optical microscopy image analysis. Figure 6.14 contains optical micrograph montages taken from the polished cross sections showing the variations in pore structure from the worn surface to the non-friction surface of the discs.

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Figure 6.14. Optical Micrograph Montages
### Table 6.8. Percentage Porosity Measured by Image Analysis

<table>
<thead>
<tr>
<th>Area</th>
<th>415T Rotor</th>
<th>415T Stator</th>
<th>401C Rotor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.4</td>
<td>11.7</td>
<td>30.7</td>
</tr>
<tr>
<td>2</td>
<td>11.1</td>
<td>12.4</td>
<td>27.8</td>
</tr>
<tr>
<td>3</td>
<td>12.3</td>
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<td>11.9</td>
<td>31.7</td>
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<td>12.3</td>
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<td>19.7</td>
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<td>12</td>
<td>12.9</td>
<td>13.8</td>
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<td>Average</td>
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<td>13.6</td>
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</tr>
<tr>
<td>Std. Dev.</td>
<td>3.9</td>
<td>1.7</td>
<td>5.1</td>
</tr>
</tbody>
</table>
Figure 6.15. Percent Porosity Distribution for 415 T Rotor

Figure 6.16. Percent Porosity Distribution for 415 T Stator
401C Rotor, Radial Section

Figure 6.17. Percent Porosity Distribution for Sample 401 C Rotor

415T Rotor, Radial Section

Figure 6.18. Statistical Variation in Percent Porosity for 415T Rotor
Figure 6.19. Statistical Variation in Percent Porosity for 415T Stator

Figure 6.20. Statistical Variation in Percent Porosity for 401C Rotor
6.4.2.1.2 SEM/QIA

The selection of the area % is based on the gray scale of 0 - 255. In most of the image areas taken at 100x magnification, a distinct range of gray levels representing the film filled pores and film spread over the adjacent area of a pore was apparent. The area % for the empty pores (black) is very small, < 1%. The bright region of the scale, from 150-255, represents the pores filled with particulates. In most cases, only the edges of the pores are being included and not the entire pore area. For each of the image areas, a higher magnification, 500x, image was taken adjacent to the 100x image. This image area was used to determine the film coverage. I have used manual editing to select the film area. Therefore, the area % reported from the 500x image is more accurate than those reported from the 100x image. However, since the 500x image captures a much smaller image area and thus more image frames are necessary to obtain a good statistical average of the % film coverage. The Tables 6.9 and 6.10 give the results of pores filled with film, pores filled with particulate, and empty pores for the highest and lowest porosity sample respectively.

Table 6.9. SEM/QIA Study of 201 C Highest porosity

<table>
<thead>
<tr>
<th>Region</th>
<th>Area</th>
<th>Magnification</th>
<th>Film Filled Pore Area %</th>
<th>Particulate Filled Pore Area %</th>
<th>Empty Pore Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>OD</td>
<td>1</td>
<td>100</td>
<td>6.72</td>
<td>15.7</td>
<td>0.79</td>
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<tr>
<td>OD</td>
<td>2</td>
<td>100</td>
<td>6.79</td>
<td>13.9</td>
<td>1.24</td>
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<tr>
<td>OD</td>
<td>3</td>
<td>100</td>
<td>3.18</td>
<td>12.2</td>
<td>0.89</td>
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<tr>
<td>OD</td>
<td>1</td>
<td>500</td>
<td>12.2</td>
<td></td>
<td></td>
</tr>
<tr>
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<td>500</td>
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</tr>
<tr>
<td>OD</td>
<td>3</td>
<td>500</td>
<td>10.7</td>
<td></td>
<td></td>
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<tr>
<td>Center</td>
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<td>100</td>
<td>5.76</td>
<td>14.4</td>
<td>0.61</td>
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<td>Center</td>
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<td>100</td>
<td>10.25</td>
<td>14.5</td>
<td>0.56</td>
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<tr>
<td>Center</td>
<td>3</td>
<td>100</td>
<td>6.2</td>
<td>14.4</td>
<td>0.72</td>
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<tr>
<td>Center</td>
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<td>500</td>
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<td>Center</td>
<td>3</td>
<td>500</td>
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<td>5.87</td>
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<td>500</td>
<td>13.9</td>
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</table>
Table 6.10. SEM/QIA Study of 415T the Lowest Porosity Sample

<table>
<thead>
<tr>
<th>Region</th>
<th>Magnification</th>
<th>Area %</th>
<th>Film Filled Pores</th>
<th>Particulate Filled Pores (white)</th>
<th>Empty Pores (black)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OD</td>
<td>100</td>
<td>0.0</td>
<td>20.4</td>
<td>0.21</td>
<td></td>
</tr>
<tr>
<td>OD</td>
<td>200</td>
<td>0.0</td>
<td>15.2</td>
<td>0.65</td>
<td></td>
</tr>
<tr>
<td>OD</td>
<td>300</td>
<td>7.4</td>
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<tr>
<td>OD</td>
<td>1500</td>
<td>0.0</td>
<td>10.2</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>OD</td>
<td>2500</td>
<td>24.8</td>
<td>10.2</td>
<td>0.78</td>
<td></td>
</tr>
<tr>
<td>OD</td>
<td>3500</td>
<td>0.0</td>
<td>8.2</td>
<td>0.00</td>
<td></td>
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<tr>
<td>Center</td>
<td>100</td>
<td>4.7</td>
<td>7.6</td>
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<td>4.7</td>
<td>7.8</td>
<td>0.05</td>
<td></td>
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<tr>
<td>Center</td>
<td>300</td>
<td>4.1</td>
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<td>0.09</td>
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<td>13.5</td>
<td>0.00</td>
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<tr>
<td>Center</td>
<td>2500</td>
<td>0.0</td>
<td>11.4</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Center</td>
<td>3500</td>
<td>18.9</td>
<td>7.5</td>
<td>0.00</td>
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<td>0.00</td>
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</tr>
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<td>5.7</td>
<td>0.00</td>
<td></td>
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<tr>
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<td>0.8</td>
<td></td>
</tr>
<tr>
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<td>17.2</td>
<td>10.0</td>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>ID</td>
<td>3500</td>
<td>31.8</td>
<td>14.5</td>
<td>0.55</td>
<td></td>
</tr>
</tbody>
</table>

To obtain the above Tables for the highest and lowest porosity, eighteen gray scale images of each sample were analysed. Six samples were made at the i.d., centre, and i.d., three at 100x and three at 500x. Samples of one set of the grayscale images are Figures 6.21, 6.22, and 6.23.
Figure 6.21. Gray Scale of Empty Pores <1%

Figure 6.22. Gray Scale of Pores Filled with Friction Film
The following two surface images are for the high porosity disc. Figure 6.24 is at 100x and is typical of an area analysed with the SEM/QIA. Figure 6.25 is the same disc showing a portion at 10,000x magnification.
Figure 6.24. High Porosity Material No Film Coverage

Figure 6.25. Large Particulate in Bottom of Pore for High Porosity Sample
Figure 6.26 for the low porosity disc 415 C is at 100x magnification and typical of the area analysed by SEM/QIA. Figure 6.27 shows the same disc at 5000x magnification.

![Figure 6.26. Low Porosity Disc with High Film Coverage](image1)

![Figure 6.27. Low Porosity Disc with Coverage of Film and "Near Film" Very Fine Wear Debris](image2)
6.4.2.2 Microscopic Morphology.

The morphology of the worn surfaces was analysed with both optical and SEM microscopes.

6.4.2.2.1 Optical Microscope Morphology

The cross section photographs of Figure 6.28 are from the high porosity sample. In order to have enough detail to discuss these photographs they are placed on a separate page to follow. The cross sections for the low porosity disc are presented in Figure 6.29.
Figure 6.28. Typical Cross Sections of the High Porosity Sample (401 C)
Figure 6.29. Typical Cross Sections of the Low Porosity Sample 415 T
6.4.2.2 SEM Morphology

Figure 6.30 is the friction surface of the low porosity sample, 415 T. Each photograph making up this collage is 2500x on the SEM. Figure 31 is the friction surface of the high porosity disc, 401 C.

Figure 6.30. Composite Showing Friction Surface of Low Porosity Disc after Testing
Figure 6.31. Composite Showing Friction Surface of High Porosity Disc after Testing
Figure 6.32 shows a photograph of a pore in the low porosity disc filled with friction film. Needling the fabric caused this type of pore. Figure 6.33 shows a similar pore for the high porosity disc.

Figure 6.32. Low Porosity Sample Cross Section with Needle Pore

Figure 6.33. Wear Debris in Friction Surface Pore Caused by Fabric Needle
6.4.3 Photon Correlation Spectroscopy Particle Size/Morphology

The particle size distribution of the collected film is presented in Table 6.11. The distributions for the samples are given in Figures 6.33.

**Table 6.11. Distribution of Film Particle Sizes**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total</th>
<th>First Peak</th>
<th>Second Peak</th>
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<tbody>
<tr>
<td></td>
<td>$d_w$</td>
<td>$d_n$</td>
<td>$d_z$</td>
</tr>
<tr>
<td>401C-I</td>
<td>57</td>
<td>1.7</td>
<td>114</td>
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<tr>
<td>401C-II</td>
<td>60</td>
<td>1.7</td>
<td>159</td>
</tr>
<tr>
<td>419C-I</td>
<td>59</td>
<td>1.8</td>
<td>127</td>
</tr>
<tr>
<td>415T-I</td>
<td>49</td>
<td>1.9</td>
<td>131</td>
</tr>
<tr>
<td>415T-II</td>
<td>43</td>
<td>1.6</td>
<td>124</td>
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</tbody>
</table>

$d_w$ = Weight-Average Diameter (nm)

$d_n$ = Number-Average Diameter (nm)

$d_z$ = Z-Average Diameter (nm)

**Figure 6.34. The Distribution of Film Particulate Sizes**
6.4.4 TEM analysis of 401C and 415 T

Figures 6.35 through 6.47 are TEM micrographs used to discuss the morphology of the friction film.

Figure 6.35. TEM Micrograph of Typical Cluster of Small Particulate
Figure 6.36. TEM Micrograph of Smaller Particles in Clusters
Figure 6.37. TEM Micrograph of Smaller Particles with Diffraction Rings
Figure 6.38. TEM Micrograph of Graphite Like Structure
Figure 6.39. TEM Micrograph of Thin Film Protruding from Clusters
Figure 6.40. TEM Micrograph of Globules on Film
Figure 6.41. TEM Micrograph of Globules on Film at Higher Power
Figure 6.42. TEM Micrograph of Electron Diffraction of Thin Films

Table 6.12 shows the selected area diffraction from the REM study.

**Table 6.12. Selected Area Diffraction Data**

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<th>JEOL</th>
<th>Hitachi</th>
<th>Diamond</th>
<th>Graphite</th>
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<td>d(A)</td>
<td>d(A)</td>
<td>d(A)</td>
<td>d(hkl)</td>
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<td>2.12</td>
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<td></td>
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<td>1.23</td>
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<td>0.7</td>
<td>0.7</td>
<td>0.73</td>
<td>422</td>
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Figure 6.43. TEM Micrograph of Amorphous Structure of Film
Figure 6.44. TEM Micrograph of Crystalline Films
<200nm,>100nm

Figure 6.45. TEM Micrograph of Small Crystalline Particles
Figure 6.46. TEM Micrograph of Agglomerate Greater than Filter Size
Figure 6.47. TEM Micrograph of Crystalline Particles about 20nm in Size
6.4.5 *Electron Energy Loss Spectroscopy (EELS)*

The samples used for the TEM study were sent to Stevens Institute of Technology to obtain the core loss and low loss spectra for the film and film agglomeration (referred to as “cotton balls”). These spectra were compared to diamond and graphite spectra. The first two Figures 6.48 and 6.49 provide the core loss spectra and Figures 6.50 and 6.51 provide the low loss spectra.
Figure 6.48. The Core Loss Spectra for Diamond
Figure 6.49. The Core Loss Spectra for Graphite
Figure 6.50. The Core Spectra for Friction Film
Figure 6.51. Core Loss Spectra for Film or Film Agglomeration
Figure 6.33. The Low Loss Spectrum for Graphite

<table>
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<th>Energy (eV)</th>
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<tr>
<td>3.3E-01</td>
<td>1.33E+01</td>
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<tr>
<td>3.2E-01</td>
<td>1.42E+01</td>
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<tr>
<td>1.51E+01</td>
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<td>2.41E+01</td>
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<tr>
<td>2.50E+01</td>
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</tr>
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</table>

Low-loss: Graphite
Figure 6.54. The Low Loss Spectra for Friction Film
Figure 6.55. The Low Loss Spectra for Friction Film Agglomerate
6.4.6 X-ray Diffraction (XDR)

The following Table 6.13 is the x-ray diffraction data.

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<td>Angstrom</td>
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<tr>
<td>401C BULK</td>
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<tr>
<td>415T FF</td>
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<td>129</td>
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<tr>
<td>415T BULK</td>
<td>3.433</td>
<td>121</td>
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</tbody>
</table>

6.4.7 Raman Spectroscopy

Raman spectroscopy data was converted to convert to L_a. Table 6.14 shows the raw data, the conversion, first to nm and then to angstroms. The ratio was calculated from the graphitic peak (1580 cm\(^{-1}\)) and the defect peak (1350 cm\(^{-1}\)).
Table 6.14. Raman Fits to Polished C-C Brakes (401 & 415)

<table>
<thead>
<tr>
<th>Sample 401 C</th>
<th>Region</th>
<th>Raman Width (1580 cm⁻¹)</th>
<th>Convert to La (nm)</th>
<th>Convert to La (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>401BCV1X.SPC</td>
<td>CVD near fiber</td>
<td>40.209985</td>
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<td>CVD near fiber</td>
<td>38.045112</td>
<td>4.9</td>
<td>48.6</td>
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<td>401BCV3X.SPC</td>
<td>CVD</td>
<td>30.769734</td>
<td>6.7</td>
<td>67.0</td>
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<td>CVD</td>
<td>30.495311</td>
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<td>Convert to La (nm)</td>
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<tr>
<td>415BC4X.SPC</td>
<td>CVD near fiber</td>
<td>44.405999</td>
<td>3.9</td>
<td>39.3</td>
</tr>
<tr>
<td>415BCV5X.SPC</td>
<td>CVD near fiber</td>
<td>40.18897</td>
<td>4.5</td>
<td>44.9</td>
</tr>
<tr>
<td>415BCV6X.SPC</td>
<td>CVD near fiber</td>
<td>38.356266</td>
<td>4.8</td>
<td>48.0</td>
</tr>
<tr>
<td>415BCV7X.SPC</td>
<td>CVD</td>
<td>32.228184</td>
<td>6.2</td>
<td>62.3</td>
</tr>
<tr>
<td>415BF12X.SPC</td>
<td>Fiber</td>
<td>55.157022</td>
<td>3.0</td>
<td>30.3</td>
</tr>
<tr>
<td>415BF13X.SPC</td>
<td>Fiber</td>
<td>52.696477</td>
<td>3.2</td>
<td>31.9</td>
</tr>
</tbody>
</table>

6.5 The second test results

The follow Figures 6.56, 6.57, 6.58, and 6.59 are collages across the friction surfaces of the highest and lowest porosity specimens, in areas of mostly
chordal and radial fibre directions. Each of the Figures has 4 strips across the friction surface taken at 70, 120, 170, and 220 stops. These collages were made up from the second test microphotographs taken at 5x and stitched together with the Adobe Photoshop 6.0 software. There are six to eight microphotographs in each collage. The sets of photographs were taken at 70, 120, 170, and 200 stops for the lowest and highest porosity samples and in regions of chordal and radial fibre. There were notches cut into the i.d. and o.d. to insure the same region would be photographed after each test segment. Great care was exercised to insure that the surfaces would not be disturbed between the test segments. These Figures are placed on separate pages to provide large enough detail necessary for the discussion in chapter 7.
Figure 6.56. The Highest Porosity Disc Chordal Fibre Direction, 70 120, 170, and 220 Stops
Figure 6.57. The Lowest Porosity Disc Chordal Fibre Direction, 70, 120, 170, and 220 Stops
Figure 6.58. The Highest Porosity Disc Radial Fibre Direction, 70, 120, 170, and 220 Stops
Figure 6.59. The Highest Porosity Disc Radial Fibre Direction, 70, 120, 170, and 220 Stops
7 Chapter 7 Discussion of the results

This chapter discusses the results of the tests and characterisations presented in chapter 6.

7.1 The Pretest Data

7.1.1 Density

The bulk density of 415 T of 1.735g/cm\(^3\) for the completed processing of a full size brake disc (Table 6.1) is typical for production discs when densification with CVD. The other two full size discs of 1.608g/cm\(^3\) and 1.710g/cm\(^3\) are low as intended by using reduced CVD time to obtain a range of densities for the study.

A carbon-carbon composite with thickness greater than 0.05 inches densified with CVD, such as full size brake discs, will usually have a density gradient from top to centre. The pores closing before they are filled with CVD matrix material cause this gradient, this is referred to as “bottle necking”. The range of this gradient is a function of the CVD parameters, mostly, dependent on the rate of diffusion and the scarcity of molecules to diffuse into the pores. By splitting the discs at mid plane and using both the top and new centre surfaces for the friction surfaces of the test rings made use of this gradient to provide 3 additional sets of different density test rings.

The surface densities of the test discs shown in Table 6.2, provided a good range of densities from 1.57g/cm\(^3\) to 1.92g/cm\(^3\) for the effect of friction and wear as a function of porosity performed in this study. A wider range was not necessary since these densities are both below and above production aircraft brakes.

The skeletal density measured by He pycnometry ranged from 1.93 to 2.04g/cm\(^3\), shown in the sixth column of Table 6.3. The skeletal density measured by He pycnometry and mercury porosimetry were in very close agreement, Table 6.1. Absolute density by He pycnometry is done at
atmospheric pressure and is usually more accurate than skeletal density measured by mercury porosimetry because of the small atomic size of He gas. The density by mercury porosimetry is calculated at 52,500 psi (358.5MPa). The fact that the mercury porosimetry and He pycnometry are very close in value is a result of the compressibility of carbon-carbon composites being low.

The procedure for measuring compressibility with mercury porosimetry is to increase the mercury pressure from 10,000 psi (68.9 Mpa) to 50,000 psi (344.7) in steps and record changes in the porosity. For the material used in this study there were no changes, so compressibility could not be calculated. The compressibility of samples cannot be measured by mercury porosimetry if the samples are either too porous or the bulk modulus is too high.

7.1.2 Porosity

The porosity measured by mercury porosimetry ranged from an average of 23.2 volume percent for the highest porosity material to 11.9 volume percent for the lowest porosity material with the other sample falling nicely in between, Table 6.3.

The surface porosity measured by optical microscopic imaging, Table 6.4 shows the area percentage of the porosity on the surfaces and the cross sections. The average surface porosity for the highest porosity disc on the friction surface is 39 percent and the surface porosity average for the cross section is 33 percent. For the lowest porosity discs the surface porosities measured for the friction surface and the cross section were 21% and 15% respectively. The optical microscopic image analysis measures the surface porosities, whereas and mercury porosimetry methods yield volume values. These methods were all in good agreement.

The volume fraction of porosity can theoretically be calculated from the skeletal density given by mercury porosimetry or He Pycnometry given above, Table 6.3. If the volume fractions of the composite components are known the following calculation for volume fraction of porosity can be made. For example, if the volume fraction of PAN fibre is approximately 28%, the density of the
PAN fibre, at the heat treatment temperature used in production, is approximately 1.76g/cm³, and the density of the CVD rough laminar matrix microstructure, at an extinction angle of 21 degrees is approximately, 2.2g/cm³ and assuming that the composite consists of only two components, the volume fraction of the CVD matrix of the disc could be calculated. The skeletal density is the density of the solid portion of the sample tested. Assuming a 1 cm³ volume sample the skeletal density can be written in the following form

\[
\text{Skeletal Density} \times 1\text{cm}^3 = (\rho_f V_f + \rho_{\text{CVD}} V_{\text{CVD}})/(V_f + V_{\text{CVD}})
\]

where \(\rho_f\) is the density of the fibre

\(V_f\) is the unit volume of the fibre

\(\rho_{\text{CVD}}\) is the density of the CVD

\(V_{\text{CVD}}\) is the unit volume of the CVD

For sample 401 C, the Skeletal Density average of the two samples, Table 6.3, is \((2.002+2.012)/2\), or 2.007g/cm³. Substituting this skeletal density into equation 7.1 and solving for \(V_{\text{CVD}}\) yields a unit volume fraction of CVD of 36 percent. Using a volume fraction equation for a volume of 1 cm³ gives

\[
V_p = 1 - (V_f + V_{\text{CVD}})
\]

(7.2)

where \(V_p\) is the pore volume

\(V_f\) is the fibre volume

\(V_{\text{CVD}}\) is the matrix volume

1 cm³ is a unit volume 401 C

The specimen unit volume for 401C is 1 cm³, the matrix volume for CVD, from equation 7.1, is approximately 36 percent, the fibre volume is about 28 percent, then the result for pore volume from the from 7.2 for 401 C is calculated as 36 percent. The pore volume measured for this sample with mercury porosimetry was 22.85, Table 6.3. This calculated value is higher than the measured value because of the assumptions used in the calculation, the exact
distribution of density of the fibre is not known and more importantly, as will be discussed below, some of the CVD was of smooth laminar microstructure that is of lower density than rough laminar microstructure and would be take up a higher volume than rough laminar. Also there were closed pores observed in the micrographs that would not be measured by the mercury porosity or the He pycnometry. Thus the actual volume fraction of pores of the pre test discs lies some where between the calculation of 36 percent and the measured 22.85. As seen the approximation could contain too many assumed values and might not be accurate, so the measured values would be assumed more accurate. These values measured or calculated are not used further in this study. After the friction and wear tests, more accurate surface porosities are measured by SEM/QIA and are the important values used in this study.

7.1.3 Microstructure

The microstructure was predominately rough laminar structure seen in aircraft brakes. Seen in Figure 6.1 is a slight ring of smooth laminar microstructure just as CVD densification begins. This small ring around the fibre surface is typical of the CVD process. All of the samples had the smooth laminar ring followed by rough laminar CVD. The premise of this study was to keep all parameters constant except for porosity/density. The microstructure was measured at 30 areas on each of the six disc sets, 12 discs. Thus, 360 areas were analysed and photographed with the same results. This gives a high confidence that the discs were identical in structure except for the required difference in porosity/density.

7.1.4 Thermal Diffusivity

The results of the thermal diffusivity measurements, on the three full size ½ thickness discs are shown in Table 6.5. The thermal diffusivity for the lowest porosity disc 415 is 0.32 cm²/sec, which is higher than that measured for the highest porosity disc 401, 0.297 cm²/sec. This is as expected. However, the
middle density disc 419 had a higher value of 0.369 cm²/sec. The bulk densities of the samples 415 and 419 were very close being 1.735g/cm³ and 1.710g/cm³ respectively. Whereas, the bulk density for the high porosity sample 401 was 1.608g/cm³, quite a bit lower, Table 6.1. Therefore, these results are likely to be within the scatter of the thermal diffusivity measurements. The sensitivity and thus scatter of the thermal diffusivity provided in Table 6.5 was consistent with prior experience. Suffice it to say that the thermal diffusivity values and thus conductivity values of the carbon materials are higher for higher density material.

7.1.5 Mechanical Properties

The mechanical properties, given in Table 6.6, were as expected in that the higher the porosity the lower the properties, with the exception of S3T which was only slightly higher. The data for S3T for the low porosity specimen had one sample with a S3T shear of only 9.67 MPa, the remaining four samples ranged from 21.0 MPa to 25.2 MPa. Throwing out the low value would give an average S3T shear of 23.03 MPa. Making it higher than the high porosity sample and consistent with the rest of the test data. Patrick and Walker [83], report that interlayer and interbundle cracks in carbon-carbon composites are appropriately aligned to act as positions of initiation of failure in interlamellar shear or flexure, respectively. Furthermore, it is the flexure strength that correlates with the voidage [83]. These observations are consistent with the observed voidage and the shear and flex strength measurements. Unfortunately, since the flexure strengths for only two different porosity discs were measured a correlation of flexure to porosity could not be made.

7.2 The Post Test Discussion of Results
7.2.1 The Effects of Porosity/Density on Test Torque

As shown in equation 5.11, torque divided by the mean disc radius is the friction force, and is directly proportional to the friction coefficient. In this type of sample subscale dynamometer testing, the energy levels, brake pressure and
velocities can be setup with accuracy. However, because the surrounding structure does not simulate a brake system, the temperature at the surface of the discs is usually lower than found in an actual brake system. Since this holds true for all the porosities tested, the relative friction and wear values should not be affected. This is the standard premise used when doing subscale brake materials tests. If temperature effects are not being measured the tests are still valid in a relative sense. The actual values of torque, stopping time, friction and wear will be representative of full-scale brakes only in a relative sense. The pressure to give equal stop times of 28 seconds controlled all of the test stops.

The torque, pressure, and velocity versus time, Figure 6.4, shows the torque vs. time (blue curve) for the low porosity friction and wear test for 60 stops. The peak torque value of 12 ft lbs (16.27 J), which is directly proportional to instantaneous friction coefficient, and the torque curve shape for the low porosity disc had already stabilised before 60 stop. This is because the peak torque value and shape of the torque curve are the same in Figure 6.5 at 200 stops as they were at 60 stops.

In the early stops, the torque curves for the higher porosity discs, Figures 6.6, began at a value of less than 8 ft lbs (10.85 J), a third less torque than the low porosity discs, and then increased quickly at 10 seconds into the stop to 12 ft lbs (16.27 J), before decreasing rapidly to the end of the stop. In the later stops as the torque stabilised for these disc, In Figure 6.7, the torque began at the same low torque value of 8 ft-lbs (10.85 J), but did not rise, it would remain flat until 16 seconds into the stop and then decrease normally as the velocity decreased. In general the higher the porosity the lower the torque values and took more stops before the torque became stable. The average friction value after 60 stops remained constant for both the highest porosity samples and the lowest porosity samples. The explanation for this behavior will be discussed in section 7.3
7.2.2 The Relationships between Porosity/Density and Friction

The effectiveness versus porosity is given in section 6.4.1. The data was actually plotted as density. The best cubic fit to the bulk density data for 220 stops after the friction had stabilised for all samples is given in Figure 6.8 to be

\[ \mu = 247.09\rho^3 - 1237.1\rho^2 + 2064.2\rho - 1147.3 \]  
(7.3)

Where \( \mu \) is the friction (effectiveness)  
\( \rho \) is the density

The correlation coefficient for this data fit is 0.9849, which indicates a very good fit of the data. The same type of function for the surface density after 220 stops is given in Figure 6.9, where

\[ \mu = 33.8\rho^3 - 178.63\rho^2 + 313.88\rho - 183.39 \]  
(7.4)

Where \( \mu \) is the friction (effectiveness)  
\( \rho \) is the density

The correlation coefficient for the surface density data fit is 0.9991. These two relationships between bulk and surface density/porosity are shown again in Figure 6.10 and Figure 6.11 as bar graphs. Each set of bars from left to right represent the number of CVD cycles from 2 to 4.

Thus objective one of chapter 4 that a functional relationship between effectiveness and porosity could be found was proved. Objective 3 that the effectiveness is a first order function of porosity/density was also demonstrated.

As an example of how the porosity affects cost, assume that the effectiveness required for service landing energy is 0.4; solving the cubic equation 7.3 for \( \rho \) gives a density value of 1.578 g/cm\(^3\). If equation 7.4 is used instead the result is nearly the same, 1.573 g/cm\(^3\). From Figure 6.10 this would fall below the first bar that represents 2 CVD cycles. So the standard four cycles would be reduced by two cycles. Whatever the cost per disc per CVD cycle is, this would cut the standard CVD manufacturing cost by half.
However, this is not the whole story. To obtain the material cost per brake landings (MCBL), one must return to equation 4.1 and in this study assume that the manufacturing cost will be fixed except for the cost of CVD. This material cost must be divided by the quotient of the wear pin length and the wear per landing. The wear pin is a device in the brake system that moves inward as the brake discs wear. This pin indicates how much mass is left in the brake stack. The length is a given design parameter necessary to ensure enough mass for a rejected take off (RTO). The wear is taken from Figure 6.13. Using the density after two CVD cycles of 1.62 g/cm$^3$, the wear is 0.2 μm per stop. For a wear pin length of arbitrarily 30 mm, the quotient of wear pin length to wear is $30000/3.6$ or 8333 landings before replacing the brake. The reason that 18 times the wear rate is in the denominator is that for a full brake stack wear pin, the wear of all 18 friction surfaces of the stators and rotors of a Boeing 767 must be included. The fact that this number of landings is higher than the actual field service landing of approximately 1200 or the design number of landings of 2500 is that only one energy was tested. Much of the wear occurs in the taxi condition, which was not tested. Also there is no accounting for oxidative wear since as pointed out previously the temperature of the surfaces of subscale tests are not reflective of full-scale brakes. Full-scale brakes could reach bulk temperatures of 1200°C whereas the subscale test only had bulk temperatures of about 600°C to 650°C. Recall also that all numbers are relative and the cost per CVD is proprietary. To continue with the explanation of the material cost per landings the actual calculations will not be made since the CVD cost per disc is proprietary and would be unrealistically low due the high number of landing. Dividing the total cost per disc for 2 cycles of CVD by the number of landings in the brake life gives the MCBL. For the MCBL for one brake system this number would be multiplied by the total number of brakes per aircraft, when all the landing gears and wheels per landing gear are considered, i.e. for a 4 wheel per landing gear, 4 landing gear aircraft the MCBL for one brake system would be multiplied by 16 to give the total cost savings, of 2 less CVD cycles, per landing per aircraft. Of course a particular airline fleet size and landings per fleet would
also multiply the savings. So it is possible that the cost savings for an airline could easily be a substantial value.

If wear was a steep function of porosity then the cost might be dictated by wear and a lower porosity would be required. This possible procedure for reducing cost only works because wear is a weak function of porosity and friction is a strong function of porosity. The example chosen here used the lowest wear measured. The explanation for this follows in the next section.

7.2.3 *The relationships between density/porosity and wear*

The relationship between porosity and wear was relatively flat after the friction and wear had stabilised. As seen in Figure 6.13 the wear after 220 stops is approximately 0.2 μm per stop for all porosities. There is a rise in wear around 1.72g/cm³, this is the wear for sample 415 C that was observed to be severely grooved in Figure 6.3 as compared to the other test disc which had smoother surfaces as shown in Figure 6.2. “Record grooving” is not fully understood, but is believed to be caused by mechanical wear (plowing) of hard particles, probably causes by fibre pullout [91], since Pan fibre is harder than the rough laminar CVD. The cause of the grooving is a study in itself. Because of this grooving the wear number will be very high since the material was lost from the creation of the grooves. The wear data as a function of density/porosity was not statistically made because with this small number of data points, one corrupted point would make any mathematical fit meaningless. Thus the probability of the hypothesis of objective 2 still exists, that there is a direct functional relationship between wear and porosity/density. It was not calculated in this study, but only shown graphically.

In the above example, a design friction value was chosen that would avoid the point on the wear curve for 415 T. It is assumed here since the majority of the actual full scale brake discs returned from service do not have this type of grooving, that wear is not a strong function of porosity in the range of energies and porosities chosen for this study and that the point for 415 T is not typical.
A much more complex model of the material cost would need to be completed that includes the entire mission mix of the aircraft landing energy spectra so as the friction and wear versus porosity test results would be more representative of field service. However, by just using the service energy for this study, it is clear that the cost of processing can be reduced by not always requiring the same number of CVD cycles.

Processing methods and costs should be part of the design process and not just a set of default processing parameters used for all aircraft carbon brakes. Typically, in this industry the manufacturing process and manufacturing cost are not considered in the design phase. This study indicates that including manufacturing process design and cost should be a necessary step in initial aircraft brake design. The importance of this conclusion has been demonstrated in this study.

7.2.4 The post test porosity of the friction surface study

7.2.4.1 Optical microscope image analysis

A collage of the cross-sections used in the optical microscopy is presented in Figure 6.14. The 401 C rotor appeared to have more pores as well as larger pores than those observed from the two 415 T samples. The porosity values were 25.4 percent for the high porosity rotor of 401 C, whereas the porosities of the lowest porosity disc were 14.3 percent and 13.6 percent for the rotor and stator of 415 T, respectively. More strikingly in Figure 6.14, the subsurface region of the 401C sample had a distinct band of large pores. The pores near the surface of the low porosity disc were filled with friction film and wear debris.

Several observations can be drawn from the optical microscope image analysis, when analysed statistically, Figures 6.15, 6.16, and 6.17. This statistical analysis of the porosity image analysis is given in Figures 6.18, 6.19, and 6.20. The 415 T stator has the least amount of variation within a cross section (13.6 % ± 1.7%) and smaller standard deviations, while 401C rotor has the largest amount of variation within a cross section (25.4% ± 5.1%) with larger
standard deviations). For all three samples, there are more variations from the worn surface to the bulk (5-10%) than from o.d. to i.d. (2-3%). The sub-surface region of the 401C disc has the highest amount of porosity (>30%). This amount may be under-estimated since many of the pores were filled with embedding resin during mounting the specimens and the gray levels of the epoxy resin were lighter than that of the pores (appeared dark). This was measured more accurately in the SEM/QIA at analysis, at 500x magnification to follow.

7.2.4.2 SEM/QIA Study

Typical gray scale images from the SEM/QIA are shown in Figures 6.21 to 6.23. These images have four quadrants, the upper left quadrant is the actual image, and the lower left quadrant is the gray scale region selected for the particular feature of interest, the total gray scale is 0 to 255. This feature is shown on the image in yellow in the upper right quadrant and again in white in the lower right quadrant. The selection of the gray scale areas is qualitative and chosen for the best visual fit. Figure 6.21 shows almost no empty pores, Figure 6.22 shows the area of pores filled with friction film, and Figure 6.23 shows the pores partially filled with particulate.

In Tables 6.9 and 6.10, the area percentages of filled pores with particulate, filled pores with film, and empty pores are presented for the 100x magnification and the 500x magnification analyses of the highest and lowest porosity discs. For the 100x analysis the pore outlines were recorded not the pore areas, this is since the mounting resin filled the pores and caused the gray scale for these pores to different than normal, it was difficult to tell film from particulate in the bottom of the pores. Thus the 500x study was used to determine the actual area of film coverage for each specimen.

For sample 401C-Rotor, the high porosity material, the image analysis, Table 6.9, shows little or no film coverage, an average of 6.91% for the 100x magnification analysis and an average of 11.14 percent with the 500x magnification analysis. Most of the wear debris coverage areas that were measured for the high porosity samples were inside the pores. There was only a small amount of film coverage on the fibre-matrix surface and these were at
areas with radial fibres. This is since a small amount of debris seems to pile up against the radial fibres and then was sheared into fine particulate. The depth of field focus on the SEM indicates that the radial fibres are higher than the matrix. This is plausible since the graphitic rough laminar CVD matrix is softer than the Pan fibres, so would be more easily worn away. For the 500x image, there were only a small distinction between the gray scale of the fibre and the film, showing almost complete coverage of the bottom of the pores with debris.

For sample 415 T, the lowest porosity disc, Table 10, the average area percentages of the film coverage are 15.9 percent for the 100x magnification analysis and an average of 24.55 percent for the 500x analysis.

A sample friction surface of one of the 9 areas at 100x magnification analysed for film coverage and particulate coverage for 401 C is shown in Figure 6.24. The areas of fibres and CVD with no film coverage can clearly be seen in this photograph, also, note the pores that are partially filled with large particulate. Figure 6.25 shows this large particulate at the bottom of one of these pores at 10,000x magnification.

In Figure 6.26, of the low porosity sample, a sample 100x image area is shown to have nearly the entire surface covered with film or very fine particulate. The pores do not contain large particulate but fine film debris. This coverage can be seen more clearly in Figure 6.27, a portion of the area in 6.26 at 5,000x magnification can be seen to be nearly clear of large particulate. Note that the film and fine debris cannot be resolved at the 5,000x magnification. The morphology of this film will be discussed thoroughly in the next section.

7.2.5 Morphology of the Worn Discs

For the Morphology of the Worn Discs several techniques were employed: optical microscopy, SEM, PCS, TEM, EELS, XRD and Micro Raman Spectroscopy. The results of each characterisation will be discussed in this section.
7.2.5.1 Morphology from Optical Microscope

Typical cross sections of the high porosity sample are shown in Figure 6.28. Here, in cross section, the large particles can be seen into the pores. In the upper right photograph, the film actually had enough adhesion to bridge the pore. In the other three photographs, it can be seen that the large particulate drops in the large pores first and then is followed by increasingly smaller particle sizes as the pore fills with particulate that has under gone more and more shear work.

Typical cross sections of the low porosity disc is shown in Figure 6.29, which also contains a pore created by the fabric needling as the substrate is manufactured. Note that there was much shear work done on the particles before they dropped into the pores. This will be discussed fully in the SEM study.

7.2.5.2 Morphology by SEM

Figure 6.30, is a composite of the worn friction surface of the low porosity disc, 415 T. Each micrograph in this collage is at 2500x magnification. Note the areas of friction film coverage. This particular area was selected for the micrograph because it contained all the components of the worn surface and is less typical of the total surface area because of scarcity of film coverage. The fibres, CVD, pores with larger particulate and friction film are all indicated on the collage.

Figure 6.31, is a similar collage of the high porosity disc, 401 C, at 2500x magnification. There is very little film coverage and what film there is collects against fibres running along the surface. It is possible that had the wear debris not been trapped by the longitudinal fibre; the particles would have fallen into a pore and not received enough shear work to grind them into a friction film. At the bottom of the micrograph, where the CVD deposited on a longitudinal fibre, is shown that a small portion of the CVD had broken off exposing the fibres beneath.
The cross section of the low porosity sample, Figure 6.32, shows a close up of a pore created during the needling of the fibre substrate and is filled with fine film particulate. The contents of the pore is fine film since the small number of pores not filled or closed by CVD is small, thus, a great deal of shear work is done before the particles find an empty pore. In contrast, the cross section, Figures 6.33, of the high porosity sample, for the same type of pore caused by needling, has a gradient of particulate sizes from the bottom to the top of the needle pore. This is because the number of pores not filled or closed with CVD is great enough that the debris falls into the pores before they receive enough mechanical shear work to grind them to fine film debris. In the discussion of the second test, it will be shown that for the highest porosity discs, new pores open because of wear before the particulate becomes worked into film and that for the low porosity sample the new pores that open during wear are immediately filled with friction film, so that the particles have no where to go. For the high porosity disc there is a higher probability that the particulate will find a pore in which to fall. For the low porosity disc, where the probability of finding a pore in which to rest is lower, the particles have time to be highly worked into friction film before they fall into a pore. These statements are of course generalisations.

7.2.5.3 Morphology by PCS

The friction film particle size distribution results from the PCS light scattering studies, Figure 6.34 and Table 6.11, showed that sample 401 C consisted of only one peak in its distribution at around 60 nm, but sample 415 T consisted of two peaks at around 30 nm and 130 nm. Thus the low porosity sample had friction film size distributions peaks of both half the particle size and twice the particle size than the size distribution peak for the high porosity samples.

It was of interest to see if TEM would in fact detect a bimodal size distribution or two different morphologies for the low porosity samples and whether the two different porosity samples had the same morphology.

Also, in an earlier publication a Russian group of scientists had reported evidence of a bimodal distribution in the friction film. Their claim was that the
film contained both graphite and a thin diamond film [105]. Their claim being that during full scale aircraft braking it is possible to have local asperity temperatures greater than 3000°C and pressures greater than 10Mpa, which they claimed was sufficient to produce diamond.

It was therefore of interest, because of this bimodal distribution of particle sizes, to see if the presence of a different morphology, such as diamond film, could be confirmed in either 401 C or 415 T by using the TEM samples for EELS analysis.

7.2.5.4 Morphology by TEM

The morphologies observed in samples 401 C (I) and 415 T (I) were very similar. In both cases clusters of smaller particles were found. The clusters were several microns in size. A typical cluster is shown in Figure 6.35. These clusters exist in both 401 C and 415 T. At higher magnification, the smaller particles that compose the individual clusters can be seen in Figures 6.36 and 6.37. These particles have a "cotton-ball" appearance and they are on the order of 100 nm to less than 50nm. Selected area diffraction patterns from these "cotton-balls" showed characteristic amorphous haloes, Figure 6.37, indicating a high degree of disorder and not preferred orientation of the crystallites. Even though the untested disc had highly graphitic CVD and well ordered carbon fibres, once sheared into fine friction film the resulting material is amorphous.

In addition to the amorphous "cotton ball" particles, small amounts of residue of particles with a graphite-like structure were also seen in both cases (see arrows, Figure 6.38). The diffraction patterns from these structures (inset, Figure 6.38) and the characteristic morphology is in good agreement with those seen in the bulk of pristine brake discs and are somewhat reminiscent of graphitic CVD matrix material.

What are clearly fractions of a thin friction film can occasionally be seen protruding from the edges of some of the clusters (Figure 6.39). While in the present study these were only seen in sample 415 T, it is suspected that these are also present in 401 C. At higher magnifications the thin films are rather continuous. Small nearly circular structures, 20nm to 50nm in size are resolved
(Figures 6.40 and 6.41). These are the black nearly round structures. Electron diffraction from these thin films (Figure 6.42) show that they are polycrystalline in nature and at least from this orientation, they show little or no evidence of preferred orientation. The measured spacings 0.212, 0.123, 0.108, 0.08 and 0.07 nm shown in Table 6.12, are in good agreement with those expected from parallel graphite-like sheets viewed along the normal to the plane of the sheets. With the exception of the spacing of 0.212 nm, the other d-spacings are also in good agreement with those from a diamond lattice.

Because light scattering experiments in sample 415T showed two well-differentiated peaks at about 30 nm and 130 nm, it was of interest to see if TEM would find differences in morphology or size that could explain the light scattering results. For this analysis, filtrates were examined (from peeled samples) with the following nominal particle sizes R:

- R > 200 nm,
- 100 < R < 200 nm
- R < 100 nm

For R > 200 nm, the results show morphologies similar to those discussed above. We find clusters of "cotton-ball" particles that give Selected Area Diffraction (SAD) patterns typical of amorphous structures. The clusters are shown in Figures 6.43. The clusters are typically 100nm or larger in size while the cotton balls of fine particles making up the clusters are less than 50 nm in size. In addition also seen are relatively large portions of crystalline films (see arrow in Figures 6.44) that give the same type of diffraction patterns that were discussed above (see also Table 6.12).

In the case of 100 < R < 200 nm, we again do not see marked differences, Figures 6.45, showing crystalline material and Figure 6.46 showing agglomeration of crystalline particulate. Large clusters are seen with many of them larger than 500 nm in size. Since the filter pore size had an upper limit of 200 nm, the results indicate that agglomeration has taken place. Some of the clusters show evidence of crystallinity, just as in of some the previous samples. In some areas, Figure 6.45, see arrows, small (10 nm) crystalline particles were
found, but the corresponding diffraction patterns neither corresponded to graphite nor diamond. It is possible that these represent some contamination or not fully dissolved replicating tape. This sample seemed to contain less of the crystalline film than the previous sample. It is possible that the thin film, which is comparatively extensive in nature, is filtered out, because of the agglomeration of the fine particles, Figure 6.46.

In the case of R<100 nm, there were relatively few particles that could be associated with the filter size material. There are also a few areas containing crystalline particles about 20 nm in size (Figure 6.47, see arrow). The diffraction patterns are definitely not associated with a carbon phase, possibly replicating tape or contamination.

As far as the bimodal distribution of particle sizes seen by light scattering, the TEM results do not provide any evidence of a different morphology or a different particle size that could be associated with these observations. At the present time it must be assumed that the results from light scattering was due to different degrees of agglomeration of the fine particle into clusters of various sizes, often larger than the filter size. The reason the bimodal distribution of particle sizes was only seen in the low porosity sample might be that the larger amount of finer particles in the low porosity sample more readily agglomerates into clusters.

7.2.5.5 Morphology of thin film (EELS Results)

A group of Russian scientist reported that a diamond film was present in friction film of carbon friction materials [105]. Their conclusion was based on diffraction patterns that appear to match those that were obtained on 401 and 415, Table 6.12. However, because there is severe overlap of diamond and graphite-like reflections of the type (hkl) when l≠0; it was felt that EELS studies were required to properly differentiate between these two phases. The same samples analysed with TEM. Core loss spectra and low loss spectra from pure diamond, graphite, the friction film, and the amorphous agglomeration (="cotton
balls") in question were obtained. The spectra are shown in Figures 6.48 through 6.55. It can be seen that the core-loss and low loss spectra for diamond are clearly different from those obtained from graphite, from the thin film, or from the agglomerates. The latter two contain a band at about 285 eV that is absent in the diamond spectra. This band would not be expected in the diamond spectra. The low loss spectra and core loss spectra for the film and agglomerates matched that of graphite and did not match diamond. The higher energy bands in both the graphite and the thin film spectra are rather broad compared to those in diamond. As to the nature of the thin film and agglomerate, the EELS results indicate that they are graphite in character and no evidence of diamond was found.

7.2.5.6 Morphology by XRD

The XRD data, Table 6.13, was consistent with carbon d_{002} and L_α spacing, and showed no difference in spacing between the bulk carbon and the film. The x-ray data is based on such a large average area and provides average crystallinity of possibly two or more components (CVD, fibre) that it doesn't seem to be a good tool for understanding the morphology of individual components of the surface carbons. Thus it was decided to include micro-Raman spectroscopy to view the crystallinity of the individual components.

7.2.5.7 Morphology by Micro-Raman Spectroscopy

The micro-Raman Spectroscopy was used to separate the crystallinity of components, CVD at the fibre interface, CVD in the bulk matrix, and the Fibre. The results of the micro-Raman measurements are presented in Table 6.14. Micro-Raman spectroscopy was able to differentiate between the smooth laminar ring of CVD and the bulk rough laminar CVD. However, the attempt to measure the crystallinity of the film was unsuccessful. The method was just under development at the time of this study. It is recommended that micro-Raman be tried on the friction film again in future work. The resulting L_α (stack width) had to be calibrated to known homogeneous crystals. The correction factor was applied to the ratio of the graphitic peak to the defect peak to obtain L_α. For the
non-graphite PAN fibre the stack width range was 29.8 Å to 31.9 Å, for the isotropic smooth laminar CVD near the fibre the stack width range was 36.4 to 48.8, and for the bulk rough laminar graphitizable CVD, L, was between 52.4 to 67.9. So the micro-Raman was able to provide three distinct non over lapping ranges for the three of the types of surface carbon that made up the worn discs, without any characterisation of particulate or wear debris. This study showed that the micro-Raman spectroscopy test might have promise.

7.3 The Second friction and wear test to add to the evidence for the friction model based on contact area.

The friction versus density model, in Figure 6.8 or Figure 6.9, showed that neither the highest porosity sample nor the lowest porosity sample studied changed its friction value from 120 to 220 stops as the test proceeded. The expectation was that the friction level would rise and stabilise but not as soon as only 120 normal energy stops. Because of this observation it was decided to run the second test described in section 5.5, with the resulting collages shown in section 6.5.

The collages in Figures 6.56 through 6.59 were “painted” with low opacity colours to enable a discussion. Red is new pores forming, magenta is pores partially filled, and yellow is pores filled. The scale of these stereoscope photographs are at 0.7x so only the macro is observed.

Figure 6.56, for the high porosity sample in the area of chordal fibres, show little or no filling of the macropores. The red band beginning after 70 stops shows new pores opening and not filling through the remainder of the test. Although there are minor changes from one radial strip to the next, no significant change in the percentage or size distribution of the macro porosity on the surface is apparent. Figure 6.57 is the low porosity sample also in the area of chordal fibre. The observation here unlike the high porosity sample is that the red bands of new pores fill completely to yellow from 70 to 120 stops. There are some new pores that open after 170 stops (red) and are either partially or completely filled after 220 stops (yellow and magenta).
For the areas of radial fibre Figures 6.58 and 6.59 are the collages for the high and low porosity samples respectively. For the high porosity sample, Figure 6.58, again the pores do not completely fill. One large pore on the i.d. never fills throughout the test. After 120 stops a group of new pores open and then fill by 170 stops, but reopen after 220 stops. Once again visually the overall surface porosity or size distribution do not show an apparent change after 70 stops throughout the testing. The low porosity sample, Figure 6.59, surface fills the pores from one collage to the next. New pores that open due to wear are filled quickly. Because of this nature of quickly filling any new pore that opens, the surface porosity after 70 stops seems to remain the same throughout the testing for this sample as well. Since for both samples the surface porosity does not change in a macro sense, the friction coefficient has stabilised by the end of only 60 stops. The measured friction coefficient was higher for the low porosity sample and lower for the high porosity sample, but did not change appreciably for either after 60 stops. So, it is postulated that for both samples their friction coefficients remained constant yet different (became stable), since the surface area in contact (real contact) area remains the same after 70 stops and further postulated that the amount of real contact area was different and varied with the porosity which determined the friction coefficient. Thus objective 4 of chapter 4, that a real contact area model can model friction coefficient vs. porosity, was demonstrated. This model requires further discussion. The contact area model will be given in the following section.

7.4 The Friction/Contact Area Model

The areas of pores filled with film from the low and high porosity samples are shown in Tables 6.9 and 6.10. The average areas of filled pores for the highest porosity sample are 11.14 percent at 500x and 6.92 percent at 100x. For the lowest porosity sample the areas of filled pores are 24.55% at 500x and 15.5% at 100x. The ratio of low porosity to high porosity of filled area at 500x is
24.55/11.14 equals 2.2. The ratio at 100x is 15.5/6.92 equals 2.24. This is excellent agreement of the two image analyses. It is important to note that the ratio of stable friction coefficient for the two samples is 0.649/0.308 equals 2.11. Thus the friction coefficients and the areas of film coverage ratios are approximately the same. The 500x images were completed to approximate the real area of friction film, since only the pore outlines were used at 100x due to the specimens mounting resin giving false shades of gray for the pores. The friction coefficient

\[ F = \mu L \]  

(7.5)

Where \( F \) is the friction force

\( L \) is the normal load

\( \mu \) is the coefficient.

According to Rabinowicz [93], friction can also be written as

\[ F = \tau_{av} A_r \]  

(7.6)

Where \( \tau_{av} \) is the average shear stress

\( A_r \) is the real area of contact.

If the total area of real contact was known exactly, the average surface shear stress could be calculated from this equation. Although the ratio of film coverage was the same for 100x image analysis and 500x image analysis, the value of percent of the area actually in surface contact cannot be assumed to be only the areas covered with friction film. There still might be other asperities in contact and doing work.

The friction force is known as the torque/mean radius and in these tests the normal load was measured by a load cell between the piston and the stator. The friction coefficient was calculated by using the velocity-averaged torque over the entire stop. The instantaneous real area is not known, the friction film coverage, even if assumed to be the real contact area, can only be measured at
the end of a stop. Thus the instantaneous shear stress on the surface cannot be calculated from this relationship.

Some believe that the friction coefficient decreases as the friction film builds. They reason that as the film builds the surface becomes flatter, thus the asperity stress to lift one asperity over another is lower, thus causing the friction coefficient to be lower as the disc flattens. It was shown in the first and second friction and wear tests to be the opposite, the reason being that as the friction film builds the real area of contact becomes greater and therefore the friction increases. In Figure 7.1, Rabinowicz [93], presents data for a metal that shows that depending on the RMS of the surface, the friction will increase with increasing contact area and will also increase with higher roughness.

![Figure 7.1. RMS roughness vs. Friction Coefficient [93]](image)

This plot of friction vs. roughness shows a negative slope region caused by excessive junction growth, (i.e. increase in friction film) and a positive region caused by the interlocking of asperities. In between, friction is independent of roughness [93].
It seems to follow that the friction film consisting of very small nm-scale particles will still do a lot of work during sliding. Thus can still produce a great deal of friction due to a huge number of smaller asperities. The friction force, being dependent on the real area of contact, might be explained if the number of smaller particles rubbing against each other is orders of magnitude greater than the original number of larger asperities.
8 Chapter 8 Conclusions and Recommendations.

8.1 Objective 1, Relationship between Friction and Porosity/Density

There was established a functional relationship between density/porosity and friction coefficient for the surface and the bulk densities/porosities. The friction coefficient as a function of either surface or bulk density yields the same result if solved for required density given a design friction coefficient. This is an inverse relationship. As porosity increases the friction coefficient decreases.

8.2 Objective 2, Relationship between Wear and Porosity/Density

The wear was measured by both weight and thickness methods. The weight method is best to use in the early number of stops before the discs fully break in (become flat). The thickness measurement is a good method after the discs are fully mated and parallel. Because sample 415 T develop "record grooves" which produced excessively high wear, the wear function vs. density/porosity was not statistically established. The wear rate is slightly lower for the lower porosity material, however the porosity effect on wear in this study was second order at best. This conclusion is based only on service energy stops. The porosity may have a greater effect on wear if the full spectrum of energies was included in the friction and wear testing, such as normal landing energy and taxi energy. It is well known that taxi snubs have a tendency to knock off the wear film and then additional wear is necessary to reestablish a stable film.
8.3 Objective 3, Porosity/Density Has a First Order Effect on Friction Coefficient (effectiveness) and/or Wear Rate,

It was established that the friction is affected strongly by the porosity/density. The relationship between wear and porosity/density was not established statistically, but seemed to be a weak function graphically.

8.4 Objective 4, A Contact Area Model of Real Area Can Model the Coefficient of Friction

The ratio of percent area film coverage of the high and low porosity samples was equal to the ratio of the friction coefficients. The friction coefficient increased with increasing film area coverage. This was consistent with the model for metals given by Rabinowicz [93]. Thus for the energy used in this study the contact area was a good correlation to friction coefficient, thus the objective was met.

8.5 Objective 5, A Link between the Porosity/Density and the Material Cost per Landing

It was shown that controlling porosity can control the friction coefficient. Controlling the densification time for the CVD process determined the porosity and thus, influenced the cost of manufacturing. To lower cost of manufacturing it is not necessary to require more density for the brake material then the design friction coefficient (effectiveness) required. Thus, densification time can be minimized to produce the lowest cost for a required effectiveness. If a high effectiveness is not needed in design, discs with more porosity are more cost effective.

Only requiring the density necessary to produce the design friction coefficient minimizes the CVD time and thus the manufacturing cost. This conclusion indicates that manufacturing process and manufacturing cost should
be included in the initial design phase. This practice is not now the aircraft brake industry standard.

8.6 Objective 6, A Discussion of the Morphology of the Friction Surface and Friction Film

The objective was to discuss the morphology of the friction surface and friction film, which might help to understand friction and wear mechanisms of carbon-carbon composite friction materials.

The effectiveness stabilizes quickly for the lowest and highest porosity material. For the lower porosity material produced few new pores opening during wear and there was sufficient friction film and fine wear debris to fill them quickly, therefore keeping the stable real friction contact surface constant and higher than the higher porosity material.

For the highest porosity material there are enough surface pores, so the particulate could easily find a pore in which to fall, not allowing shear work on the particles to produce friction film. The exception to this was when the matrix, which is softer than the fiber, wore away, allowing the fibers to be higher than the matrix. Some of the particulate would become blocked by the longitudinal fibers; this particulate could not fall into an open pore so became sheared into a fine friction film.

As the wear progresses the shear forces continue to break down the particulate into smaller and smaller particle sizes. The resulting friction film is mechanically ground under heat and stress to produce an amorphous or crystalline material that is not well ordered. The friction film material eventually becomes so fine it is below the crystallite size of the original carbon components and could not be adequately resolved by TEM. The finest particles in the friction film give evidence that the actual new disc non-worn carbon-carbon composite crystallites have been broken.
There is a gradient of particulate size from the bottom to the top of a deep pore indicating continual mechanical grinding under heat and pressure. There are apparently large shear forces acting on the wear debris if it stays on the surface and does not fall into a pore.

In the TEM and PCS analyses the friction film was shown to agglomerate into clusters larger than the filter sizes used to obtain the samples. This gave the illusion, when measured by light scattering (PCS) that there was a bimodal particle size distribution for the lowest porosity samples. No such bimodal distributions of particle sizes or different morphologies were found with TEM.

The TEM analysis indicated by the Selected Area Diffraction and measured crystallite sizes that diamond might be present in the film. An EELS analysis proved that only graphite is present in the friction film and the agglomerations of the friction film.

8.7 Recommendations

This work was done using only the service energy of the Boeing 767 aircraft. It would be of interest to study the friction and wear tests of full-scale brakes with a complete load spectrum, which would include normal landing, taxi, and RTO energies.

The preform manufacturing could take many forms, completing this study for various carbon-carbon composites might lead to different results.

The effect of additives, such as SiC, would more than likely change the morphology of the friction film. Also, contaminates such as hydraulic fluid, deicing fluids, washing fluids, fresh water and seawater. It is assumed the absorption and adsorption would be influenced by the porosity size and distribution.

The friction film should be explored with the micro Ramon spectroscopy as soon as the procedures for the method are complete.
A follow on project is now under way at Notre Dame University to gain a better understanding of the friction couple at the disc surfaces. The study is including full scale mating rotors and stators returned from field service.
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