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**FABRICATION AND CHARACTERISATION OF A HOOP REINFORCED,
CONTINUOUS SiC FIBRE, TITANIUM MATRIX COMPOSITE PRODUCED
BY FILAMENT WINDING AND HOT ISOSTATIC PRESSING**

by

Karl W. Griffin, *B.Eng*

A thesis submitted to
the Faculty of Graduate Studies and Research
in partial fulfilment of the requirements for
the degree of

Master of Engineering

Department of Mechanical and Aerospace Engineering

Ottawa-Carleton Institute for
Mechanical and Aerospace Engineering
Carleton University
Ottawa, Ontario
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February 1998

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Thesis Supervisor

Chair, Department of Mechanical and Aerospace Engineering

Carleton University
March 1998

ABSTRACT

The high specific strength and service temperatures of metal matrix composites has engendered the interest of the aerospace industry. The potential application of metal matrix composites in aerospace components depends on the development of practical fabrication techniques to produce the required geometries. Ring geometries are of interest to the gas turbine industry as reinforcements for compressor disks and spacers.

This thesis describes the development of a viable laboratory scale fabrication technique to produce continuous fibre, metal matrix, composite rings. The technique developed simultaneously winds Ti-6Al-4V matrix wires and SiC fibre onto a mandrel which is then vacuum encapsulated and hot isostatically pressed. Subsequent modification of the matrix microstructure of the consolidated ring, through secondary heat treatment, is presented.

An unexpected microstructure results from β annealing. The potential factors leading to this microstructural anomaly are presented and it is concluded that residual stresses play an important role in the development of the β annealed microstructure.

A technique for determination of fibre-matrix interfacial shear strength using a nanomechanical probe is described and the results of the fibre push out tests on the composite samples produced are summarised. Although the load capacity of the nanomechanical probe is insufficient to determine the interfacial shear strength of all microstructural conditions, the results suggest that the thermal stresses produced during rapid cooling strongly influence titanium matrix composite properties.

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This thesis is dedicated to my nephew Kyle and the dream of a better future for the next generation.

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Nomenclature

α	hexagonal close packed phase of titanium
α'	hexagonal close packed martensite phase of titanium
α -SiC	amorphous phase of SiC
β	body centered cubic phase of titanium
β -SiC	hexagonal close packed phase of SiC
$\dot{\epsilon}_s$	steady state creep rate
σ	applied stress
σ'	average metal flow stress
σ_b	Weibull fibre bundle strength
σ_c	ultimate strength of the composite
σ_f	ultimate strength of the fibre
σ_m	ultimate strength of the matrix
σ_{mc}	stress necessary for matrix cracking
σ'_m	stress of the matrix at fibre failure strain
σ_s	plastic contact stress of two spheres
σ_y	yield stress
ψ	Weibull scale parameter

τ	interfacial debond shear strength
ς	density dependant scale plastic yielding factor
ASTM	American Society for Testing and Materials
b	Burger's vector
d	grain size
D_{gb}	self-diffusion constant
EBED	electron beam evaporation and vapour deposition
EDAX	energy dispersive analysis of x-rays
F	ratio of average metal flow stress and HIP pressure
FPO	fibre push out
HCF	high cycle fatigue
HIP	hot isostatic press
HySTP	Hypersonic System Technology Program
IHPTET	Integrated High Performance Technology Program
k	Boltzmann's constant
K	parabolic growth rate constant
K_0	material growth rate constant
LCF	low cycle fatigue
m	Weibull shape factor

MMC	metal matrix composite
MCF	matrix coated fibre
MROM	modified Rule of Mixtures
N	number of fibres in the sample
NMP	nanomechanical probe
NRC	National Research Council of Canada
NASP	National Aero-Space Plane
Q	activation energy
r	fibre radius
R	ideal gas constant
ROM	Rule of Mixtures
SEM	scanning electron microscope
STA	solution treat and age
STOA	solution treat and overage
t	time
T	absolute temperature
TMC	titanium matrix composites
V_{cnt}	critical fibre volume for useful strengthening
V_f	fibre volume fraction

w sample thickness

x reaction layer thickness

The next generation of aerospace vehicles will require increases in overall performance to fulfill their mission roles. The devolution of the National Aero-Space Plane (NASP) program into the Hypersonic System Technology Program (HySTP) [1,2] delineates the importance of the development and implementation of novel, effective and economically feasible technologies by the aerospace industry. While progress in all aspects of aviation is necessary, an area where advancements are vital for the success of future initiatives is that of propulsion. By increasing the thrust and reducing component weight of gas turbine engines significant gains in aircraft performance can be achieved. The Integrated High Performance Turbine Engine Technology program (IHPTET) and similar initiatives have been launched to realize this potential.

The goal of the IHPTET program is to double the turbopropulsion capability of gas turbines rated in 1985 by the year 2003 without significant detrimental effect to engine life and maintainability [3]. This is ambitious; the required improvement in specific thrust (thrust to weight ratio) of gas turbines is significantly greater than the historical trend. The program plans to achieve its goals in three phases; phases one and two having been completed on schedule [3]. This thesis is part of a series of overlapping, but independent, Canadian initiatives to advance gas turbine design.

The required improvements are well beyond the capabilities of the monolithic alloys currently utilised in gas turbines and it is unlikely that future improvements in

traditional materials will be capable of meeting the IHPTET phase 3 target [3]. Metal matrix composites (MMCs) have been proposed as a possible replacement material for monolithic alloys in several aerospace applications. MMCs are multi-phase materials consisting of ceramic fibres or whiskers suspended in a metallic or intermetallic matrix. The resulting material has the high strength and stiffness of the ceramic fibres and retains much of the ductility of the original matrix alloy. In this they are similar to thermoset and thermoplastic polymer matrix composites that are currently used in the aircraft industry with one additional caveat; they retain these properties at elevated temperatures. This makes them attractive for gas turbine applications. In several of these applications the loads are essentially applied in one direction hence unidirectional continuous fibre reinforcements can be utilised. This will both maximize the weight savings and give the greatest increase in the strength of the material in the direction of the reinforcement. Thus a considerable amount of research has been devoted recently to the processing and properties of continuous fibre reinforced MMCs. The key processing technologies will be further discussed in chapter 2.3.

A prime example of a MMC system that can be used to speed the necessary improvements in gas turbine applications are titanium alloys reinforced with silicon carbide fibres (TMCs). They are stronger, stiffer and lighter than monolithic titanium alloys and have been proposed as replacements for conventional titanium in a multitude of areas. TMCs have also been suggested as replacements for nickel and steel compressor stages that operate at temperatures and stresses above that of titanium alloys [4, 5]. Titanium matrix composites have also been proposed as structural material on

the postponed NASP aerospace plane [6] and as replacements for MAR aged steel in landing gear assemblies [7, 8]. The only known production aerospace application to date, however, is the control linkage for the convergent-divergent nozzle for the F-22 air superiority fighter shown in Figure 1.1.

In gas turbine applications, several prototype TMC components have been manufactured. TMC compressor rotors, spacers, turboshafts and internal reinforcement rings for centrifugal compressor impellers, depicted in Figure 1.2, have all been postulated to replace current monolithic parts [3, 9, 10, 11]. Specifically, continuous fibre TMC components for axisymmetric rotating components have elicited interest. In these applications the primary load experienced is the centrifugal force developed by the rotation. This type of application is tailored for the anisotropic properties of TMCs. Experiments with reinforcing rings in model axial compressors stages has raised the burst speed from 40,000 rpm for monolithic stages to 76,000 rpm, an increase of 190% [12]. However, the greatest weight reductions would be from the radical redesign of the axial compressor stage. TMC reinforcement rings could potentially provide enough strength and stiffness to axial compressor stages that the axial compressor disk's central hub and web could be eliminated as sketched in Figure 1.3 [13]. The potential weight reductions utilizing TMC components, shown schematically in Figure 1.4 [14], have been estimated to be 75% or greater than that of current turbo-engines.

Additionally, the properties of TMCs compare favourably with both current and developing high temperature materials. In Figure 1.5 [6] it can be seen that various

TMC systems compare well to monolithic titanium, titanium aluminides and nickel based superalloys. However, despite the promise of this composite system, its induction into service has been limited to the above mentioned control linkage lagging predictions that TMCs would be seeing increasing service from 1997 onwards, seen in Figure 1.6 [15].

There are several reasons for the lack of TMC parts in industry. Significantly, the innate conservatism of the aerospace industry is a major obstacle to the introduction of TMC parts onto aerospace vehicles. This reluctance is well justified; despite the efforts of several research groups and industrial initiatives the quantification of TMC properties is nascent. Several different alloys and silicon carbide fibres have been investigated, but there is as yet no fully coherent database for any matrix fibre system. This is complicated by the fact that TMC properties are dependent upon the processing route and there is no standard method for constructing axisymmetric TMCs. Also, the processing methods under current investigation rarely consider economic issues. Before TMCs can be implemented into new, high performance designs they must have well defined standards, including a baseline manufacturing method that is both reliable and economical.

The National Research Council of Canada (NRC), in collaboration with Carleton University, has undertaken a program that may help define realistic economic methods of manufacturing axisymmetric TMC geometries that can be used in gas turbine applications. After a thorough economic study [16], two routes were identified as the

most economically feasible. This thesis explores the technical viability of one such method.

1.1 Scope and Outcome of Thesis

The aim of this thesis is to develop the processing capability for the manufacture of TMC axisymmetric rings on a laboratory scale. The method developed winds co-mingled titanium matrix wires with Textron SCS-6TM silicon carbide fibre onto a translating and rotating mandrel. This preform is consolidated utilizing a hot isostatic press (HIP). Details of the filament winding apparatus and consolidation parameters are presented in chapters 4 and 5.

As stated, the primary objective of this project is to demonstrate the technical viability of the co-mingled winding process. An important secondary objective is the characterisation and quantification of the parts produced. Additionally, whenever feasible, efforts were made to model the various aspects of the composite.

The thesis research demonstrates that co-mingled filament winding and HIPing is a viable method of producing hoop reinforced TMC components. Microstructural adjustment of the matrix through heat treatment to optimize mechanical properties is possible; however the application of conventional heat treatments for titanium alloy matrices result in microstructures that do not fully comply with the accepted norms. On the basis of fibre push out tests and models for estimating the composite strength it was

determined that heat treating the TMC increases the interfacial strength and hence the composite strength.

This chapter is designed to impart a basic understanding of the various factors involved in the fabrication of TMC components. It covers a broad scope of topics, starting with matrix and fibre characteristics and the chemical reaction between the fibre and the matrix. It then describes fabrication techniques for TMCs with an emphasis on solid state consolidation parameters. Finally, a short review of the methodologies for used assessing and predicting composite strength has also been included.

2.1 Constituents

2.1.1 Titanium and Titanium Alloys

Titanium is a high strength, low density material that has been used in the aerospace industry for over fifty years. Table 2.1 [17] lists the characteristic properties of pure titanium. Pure titanium has an allotropic transformation temperature of $882\text{ }^{\circ}\text{C} \pm 2^{\circ}\text{C}$, from a hexagonally close packed alpha phase (α) to a body centered cubic beta phase (β) [17]. Hence, the mechanical properties of titanium alloys are strongly dependent on the microstructure present. Microstructural adjustments are made through a combination of alloying elements and numerous heat treatment routes. There are a multitude of titanium based alloys, but most can be grouped into three alloy families based on the predominant room temperature phase present. Intermetallic titanium based alloys (typically Ti-Al and Ti-Al-Nb systems) exist and have been considered as

future matrix alloys, but are themselves experimental and are thus beyond the scope of this thesis.

The α or near α alloys typically have a microstructure consisting primarily of the α (hcp) phase at room temperature. Since α is the predominant equilibrium room temperature phase, this series of alloys includes commercially pure titanium. α alloys have α stabilizing alloying elements (typically Ga, Al and Sn [17]) added to raise the β transus temperature. Thus α and near α alloys have stable microstructures at higher temperatures than other titanium alloys. This class of alloys are characterised by good toughness and weldability, but have inferior strength [18] and poorer forgeability [19] when compared to other titanium alloys. More important, for gas turbine applications, is the excellent property retention and superior creep resistance exhibited at high temperature due to their increased stability. These properties can be enhanced further in most near α systems by additions of minute (0.1 wt%) amounts of silicon [18]. Thus, most new titanium alloys introduced for gas turbine applications belong to the near α family, especially when operating temperature is the critical parameter i.e. high pressure compressor stages [20].

The β alloys are typically defined as alloys that are capable of retaining 100% of the high temperature β (bcc) allotrope at room temperature upon quenching from above the beta transus [18, 21]. By using β stabilizers (transition metals) the transus is reduced and the phase diagram is shifted so that martensitic α' is not produced upon quenching.

These β alloys fall into two categories: *stable*, in which enough beta stabilizers have been added to the alloy that the β microstructure will remain stable at elevated temperatures; and *metastable*, in which the β phase will decompose into transformed α at elevated temperature. A phase diagram illustrating the change from metastable β alloys to stable β alloys is given in Figure 2.1 [21]. All known alloy systems in use are metastable [17] and are thus not typically utilised in elevated temperature applications [19]. β alloys have the highest strength to weight ratio, they can be hardened and are easily forged and cold formed [19]. They are also the most resistant to corrosive environments due to their high alloying content [22]. However, the transition metals used as stabilizers are heavy elements, hence β alloys possess the highest density of the titanium alloys. Welds with acceptable strength and ductility are difficult to achieve due to precipitation of the alloying elements. Finally, β alloys are prone to ductile/brittle transitions at low temperature due to their body centered cubic structure [17].

The $\alpha + \beta$ alloys are generally alloy systems that contain between 10% to 50% of the high temperature β (bcc) phase present at room temperature [17]. They have both α and β stabilizers as alloying elements and display an excellent blend of characteristics. The $\alpha + \beta$ alloys are somewhat more thermally stable than beta alloys and are used in applications that experience service temperatures up to 400 °C [18]. The properties of $\alpha + \beta$ alloys can be modified by heat treatment and/or hot working [23]; weldability, strength and toughness are highly dependent on grain morphology and amount of β present at service temperature [17, 18, 23-26]. $\alpha + \beta$ alloys are the most widely utilised

alloys in the aerospace industry with both static and rotating gas turbines components constructed from this alloy family [18].

2.1.1.1 Matrix Alloy: Ti-6Al-4V

Ti-6Al-4V (Ti-6-4) is an $\alpha + \beta$ alloy with excellent strength and toughness in its mill annealed form [17]. This alloy is the standard utilised in most aerospace applications and accounted for 60% of the total titanium production in 1995 [27]. It has been the most widely used grade of titanium since 1961 [28]. Ti-6-4 is produced in several different grades which differ primarily in the amount of interstitial elements (O, C, N, H) present, including a high purity extra low interstitial grade (ELI) for cryogenic applications [17, 28]. Ti-6-4 alloy is produced under vacuum either by the conventional consumable electrode, triple-arc melt method or cold hearth melting of sponge and alloy additions to produce ingots that are free of type I or type II imperfections [19, 27]. Type I imperfections consist of nitrogen and oxygen rich regions of α stabilized titanium that possess high hardness but low ductility and are associated with voids and cracks in service. Type II imperfections are caused by segregation of the alloy's α stabilizers creating stringers of abnormally stabilized alpha that can extend across several β grains [19]. Both defects are unacceptable for aerospace applications.

Ti-6-4 is available in wrought, cast and powder metallurgy forms. It is commonly used in the milled annealed condition, which has an excellent blend of strength, ductility, toughness and fatigue strength [17]. As an $\alpha + \beta$ alloy with a lean β (less than 20% β

phase at room temperature) content it is weldable [17, 28]. It is useable for applications ranging from -69 °C to 399 °C [28]. In gas turbine compressors it is used extensively in the first four to five stages as both disc and fan blade material. Typical service temperatures do not exceed 315 °C in these applications [18, 28].

2.1.1.2 Physical Metallurgy of Ti-6Al-4V

The physical metallurgy of Ti-6-4 has been extensively researched in literature. Thus only a summary of its characteristics is within the scope of this thesis. The 6 wt% aluminum acts as an α stabilizer increasing both $\alpha \rightarrow \alpha + \beta$ and $\alpha + \beta \rightarrow \beta$ transus temperatures and strengthens the alloy through solution strengthening. The 4 wt% V acts to mitigate the effect of the aluminum addition on the $\alpha \rightarrow \alpha + \beta$ transformation temperature and stabilizes the β phase as shown in Figure 2.2 [28]. The net effect of these additions is to raise the β transus for Ti-6-4 to 995 ± 15 °C [17]. The alloying additions also alter the lattice parameters of both phases. The vanadium addition, by stabilizing the β phase, improves the forgeability and strength of the alloy. The complete composition of Ti-6-4 (grade 5) alloy wire according to ASTM standards is given in Table 2.2 [32].

The properties of Ti-6-4 also depend on the amount of interstitial elements present. Oxygen and nitrogen vary, depending upon the grade, from 0.08 to ~ 0.2 wt% for oxygen and up to 0.05 wt% for nitrogen. The higher the concentration of these interstitial elements the higher the strength. However, the increase in strength occurs at

the cost of ductility, fracture toughness and stress-corrosion resistance [17]. The carbon content limit is 0.10 wt%. Higher levels of interstitial elements will raise the β transus temperature [28], thus the ELI grade of this alloy has a lower β transus than the standard grade [17].

2.1.1.3 Heat Treatment of Ti -6Al -4V

The microstructure of Ti-6-4 is highly tailorable depending on the heat treatment temperature, the cooling rate and the amount of hot working. Figure 2.3 [17] illustrates the typical microstructures produced by varying the cooling rate after heating to different regions in Ti-6-4 phase diagram. The heat treatments are used to accentuate different characteristics of the alloys. Several common heat treatments are given in Table 2.3 [28] and are summarized below.

Stress Relief cycles for Ti-6-4 are widely used to remove residual stresses incurred in the final forming stages without adversely affecting strength and ductility [23]. A nomograph has been developed for this alloy system, presented as Figure 2.4 [17] allowing for precise selection of thermal treatments to reduce stress levels. This is important as Ti-6-4 suffers from the *Bauschinger Effect* such that a slight tensile strain results in a drastic reduction in compressive strength [28]. This effect is healed by stress relief annealing. Stress relief annealing is also used to remove warpage and springback [28] from parts. While stress relief cycles have been studied at temperatures

as low as 260 °C [17] stress relief is generally performed at 480 °C to 650 °C for 1 to 4 h [23].

There are several annealing cycles for Ti-6-4. Most components made from Ti-6-4 are in the *mill or full annealed* form. This heat treatment produces a stable microstructure of equiaxed α in a matrix of β , shown in Figure 2.5a [28]. Full annealed microstructures have balanced properties with good ductility and strength [17].

Recrystallisation annealing is used to optimize the damage tolerant aspects of the alloy at the cost of a slight loss of strength compared to the full annealed state. The microstructure consists of large α grains with small β grains at the triple grain boundary intersections, as illustrated in Figure 2.5b [28]. Another annealing route used to promote damage tolerance is ***duplex annealing***. This is accomplished by heating the alloy high into the $\alpha+\beta$ region of Figure 2.2. Upon cooling the beta transforms into a lamellar alpha structure giving a duplex structure of lamellar alpha surrounded by equiaxed alpha grains seen in Figure 2.5c [28]. This structure has excellent fracture toughness and crack growth resistance, but reduced fatigue strength [17]. The most extreme annealing operation is ***β annealing***, for which the temperature is raised into the fully transformed β zone. As the alloy is cooled the microstructure, shown in Figure 2.5d [28], transforms into fully lamellar α . This maximizes both damage tolerance and creep resistance of the alloy.

As stated in chapter 2.1.1, it is possible to strengthen β and $\alpha + \beta$ titanium alloys. The key to this is the metastable nature of the β phase. By heating at a higher temperature in the $\alpha + \beta$ phase range more β is present than the room temperature equilibrium volume fraction. This extra β is transformed into a martensitic α' upon quenching and upon subsequent aging at an elevated temperature the α' phase decomposes into very fine, randomly aligned, needle like α grains in a matrix of β [17, 23]. The fineness of the grains impedes dislocation movement and this lowers ductility and increases strength. The hardenability of the component thus depends strongly on the amount of β stabilizers in the alloy and the temperature from which it is quenched. Ti-6-4 is considered have shallow hardenability because it is lean in β stabilizers [17]. Full hardening of Ti-6-4 components is only possible in pieces of one inch thickness or less. The standard *solution treat* (ST) for Ti-6-4 is performed high in the $\alpha + \beta$ region (~ 50 °C below the β transus) for good strength and adequate ductility. If improved creep resistance and damage tolerance is required (at the loss of tensile ductility) then the treatment is performed in the β region or *β quenched* [17].

The final step, an elevated temperature *aging* (A) of the quenched Ti-6-4 is performed at 538 °C for 4 h. At this temperature the α' phase has enough energy to decompose into lamellar α , thus strengthening the alloy. By heating Ti-6-4 higher into the $\alpha + \beta$ zone it is possible to *overage* (OA) the component, improving the fracture toughness and ductility with a modest reduction in strength. Microstructures of STA and STOA Ti-6-4 samples are given in Figure 2.6a and 2.6b [23] respectively.

2.1.1.4 Mechanical Properties of Ti-6Al-4V

The mechanical properties of Ti-6-4 are highly dependent on the processing route and type of stock. Ultimate tensile strength, yield strength, elongation and reduction in area for Ti-6-4 (Grade 5 titanium alloy) are specified by ASTM standards [32-36] for various titanium products. These values are developed utilizing tensile tests described in ASTM standard E-8 [37]. The guaranteed properties of the various mill annealed forms of Ti-6-4 are given in Table 2.4 [32-36]. Typical stress-strain curves of full annealed Ti-6-4 plate and bar are given in Figure 2.7 [28].

The effect of *solution treating and aging* (STA) treatments on the tensile properties of Ti-6-4 sheet is demonstrated in Table 2.5 [17]. As stated previously, Ti-6-4 has shallow hardenability and tensile strength falls off rapidly with section thickness. The modulus of elasticity for both full annealed and STA treated bar are shown in Figure 2.8 [28]. The deleterious effects of quench delays in STA treatments are given in Figure 2.9 [28].

The effect of *β Annealing's* on the tensile properties of Ti-6-4 sheet given in Table 2.5 [28]. The improvement in creep rupture life of beta treated Ti-6-4 when compared to mill annealed stock is illustrated in Figure 2.10 [28].

2.1.1.5 The Effect of Stress State on Titanium Alloy Microstructures

Thermomechanical processing techniques are often applied to Ti-6-4 and other $\alpha + \beta$ alloys to influence the microstructure and physical properties of the alloy while achieving shape design requirements [17]. Conventional forging for Ti-6-4 occurs below the β transformation temperature and alters α grain morphology through dynamic recrystallisation and residual strain. This forging operation is typically used to produce an equiaxed α structure in a matrix of β that maximizes tensile ductility [28]. β forging is used when a high level of deformation is required or increased fracture toughness is desired [28]. This process occurs above the β transus and statically recrystallises Ti-6-4 [17] and is, hence, a non-cumulative process.

Another interesting use of stress to alter the microstructure of $\alpha + \beta$ alloys is to create a duplex microstructure through cold working the surface of the alloy followed by heat treatment. These techniques are used when conventional thermomechanical processing is not practical or desirable. One example of this technique is to induce residual compressive stresses in the alloy surface through shot peening prior to heat treating [17]. An example of a resultant microstructure is shown in Figure 2.11 [17], with a creep resistant lamellar core and a fine equiaxed, low cycle fatigue resistant, surface layer.

2.1.2 Silicon Carbide Fibre

Ceramic fibres are generally used in most aerospace metal matrix composite systems [15]. Ceramic fibres have high strength, low density and high stiffness. Alumina, carbon, sapphire and glass are among the ceramics that have been utilised in polymer matrix composites. Boron fibre has enjoyed some use in metal matrix composites, most notable being the boron/aluminum reinforcement tubes on the Space Shuttle orbiter doors [38]. However, boron fibre is expensive to produce, reacts rapidly with molten aluminum and degrades at elevated temperatures [10]. The search for an alternative fibre that is relatively inert, inexpensive and retains properties at high temperatures led to the development of silicon carbide (SiC) as a reinforcement material. Silicon carbide was first utilised in whisker form and an aluminum/SiC composite was used as a piston ring insert by Toyota [38]. For high end applications, continuous fibres have been developed. There are essentially two primary methods for producing continuous silicon carbide fibre.

The first method spins a precursor fibre from a molten polycarbosilane (PCS) developed from the pyrolysis of dodecamethylcyclohexasilane. The filaments are then oxygen cured to render them infusible. The green fibres then undergo a two step pyrolysis at temperatures ranging from 200 °C to 500 °C for the first pyrolysis and a final pyrolysis at 500 °C to 900 °C. During this phase the filament loses its organosilicon character and becomes a ceramic fibre [39, 40]. This method produces microcomposite fibres with a microstructure of minute (~ 2 nm) β -SiC crystals and carbon clusters surrounded

by an amorphous silicon oxycarbide at the grain boundaries [41]. Fibres prepared by this method are strong, of small diameter (10-20 μm) and can be woven. Two firms market polycarbosilane derived fibre; Nippon Carbon (Nicalon fibre) and UBE (Tyranno fibre). However PCS derived fibres are not suited for TMCs. This is because titanium is both very reactive at high temperatures and sensitive to the oxygen (see section 2.1.1.2) present in PCS fibres in the silicon oxycarbide phase. Additionally, fibres produced by this method degrade when exposed to temperatures above 1100 °C-1200 °C [42] since the silicon oxycarbide phase decays causing growth of the SiC crystals and the evolution of gaseous by-products [43]. There have been recent efforts to increase the thermal stability of PCS derived fibres by substituting the oxygen infusion with a physical cure, thus reducing the oxygen content of the fibre [40, 43]. However, this work is developmental and no PCS fibres produced by this method are commercially available.

An alternate method of producing silicon carbide fibres is through the use of chemical vapour deposition (CVD). In this method a carbon or tungsten monofilament is heated and exposed to silane and hydrogen gases. The silane gas decomposes to form β -SiC continuously on the monofilament substrate [10, 44]. Fibres prepared by this method are typically of large diameter (>100 μm), though recent advances in deposition techniques by Textron have resulted in a new, SCS-X fibre with a diameter of $\sim 50 \mu\text{m}$ [45]. Fibres produced by CVD are thermally stable compared to PCS derived fibres and retain properties at elevated temperatures. Significantly, for MMCs, they can receive an

additional, protective coating through the CVD process [44, 46]. There are two major producers of CVD SiC fibre, Textron (SCS series) and British Petroleum/ DRA Sigma (Sigma series). For titanium matrix composites the fibres developed for use are the Textron SCS-6 fibre with a 3 μm thick carbon coating and the Sigma SM-1240 with a duplex coating of 2 μm thick TiB_x over a base coat of 1 μm carbon [46].

2.1.2.1 Microstructure of the Textron SCS-6TM fibre

The SCS-6 fibre has several layers or regions, created through differences in the deposition parameters of the CVD process. These layers have been discretized through analysis of TEM foils of fibres consolidated in polymer matrices [47, 48]. A widely accepted explanation of the microstructure of the SCS-6 fibre separates the fibre into six distinct regions, shown schematically in Figure 2.12 [47].

The core, or carbon fibre substrate, consists of blocks of turbostratic carbon of 1-5 nm, randomly oriented throughout the filament. Turbostratic carbon is essentially an isomer of graphite, the main difference being that the layers of graphite are randomly shifted an at angle ϕ about the normal axis to the basal plane. A protective layer of pyrolytic carbon is then deposited upon the substrate. This layer can be differentiated from the substrate by an increase in size (30-50 nm) and orientation with the basal axis oriented roughly in the radial direction [47].

As illustrated in Figure 2.12 the silicon carbon can be split into four separate layers [47].

The three inner layers (SiC-1 through SiC-3) are carbon rich, with a C to Si ratio of 2:1 at the inner carbon coating/ SiC-1 interface. The carbon concentration diminishes with radial thickness until it reaches the mid-radial region between the inner and outer carbon coatings where it stabilizes at the stoichiometric ratio [48]. The differences in the layers is thus a function of the free carbon present though the shift between regions is fairly distinct [47].

The inner most SiC region consists of a distribution of β -SiC ranging from equiaxed grains at the inner carbon coating and changing to columnar SiC grains trapped in a matrix of amorphous carbon [48]. The columnar grains are 5 -10 nm in length with an aspect ratio of ~ 10 [47] and are arranged in a 'herringbone' pattern [49].

As the SiC sheath thickness increases, the length of the SiC rods increases to 50-150 nm and the orientation of the grains shift so that the rod axis lies along the radial direction of the fibre [47]. These elongated grains grow preferentially along the $\langle 111 \rangle$ direction of the hexagonally close packed β - SiC microstructure.

In the last carbon rich region, the SiC-3 layer, the excess carbon diminishes to $\sim 5\%$ above the stoichiometric ratio [47] and the SiC grains grow to micron length. The grains are heavily faulted, with the faults lying normal to the rod axis (in the columnar plane) [48].

At the midradial boundary between the inner and outer carbon coatings the SiC layer becomes stoichiometric or slightly rich in silicon [48]. The SiC grains remain columnar but their size approximately doubles. However, the grain size then diminishes, the diameter (width) of the grains reducing from 248 nm to 90 nm as the outer layer is built up [47]. This reduction in grain size effectively smoothes out the surface of the fibre reducing possible stress concentrations.

The final layer of the SCS-6 fibre is the 3 μm thick outer protective coating. This layer increases the strength and durability of the fibre by reducing the stress concentrations caused by any uneven deposition of the SiC layers and acts as a sacrificial coating. This sacrificial coating can be separated into three sub-regions. The inner and outermost of these regions contain minute β -SiC crystals in a matrix of turbostratic carbon blocks 1-5 nm in diameter. The middle region is entirely composed of minute turbostratic carbon with no SiC crystals evident [47].

Understanding the microstructure of the SCS-6 fibre is extremely important as it intimates the failure mechanisms of the fibres when consolidated in composites. The middle region in the outer carbon coating should be the weakest area of the fibre due to its lack of SiC nanocrystals. This has been confirmed by numerous fibre push out tests in composite systems where the fibre- matrix interface is strong. In these tests the fibre shears in the outer carbon coating as the weak Van der Waals bonding between the basal layers is broken [47].

The fracture mechanisms for the fibre can also be explained by the microstructural characteristics of the fibre. The most common failure of the SCS-6 fibre is a planar crack initiating from the fibre - matrix interface (see section 2.2). This is consistent with traditional cleavage mechanics as the crack advances along the close packed plane which is normal to the fibre axis. Another, complex, fracture mode initiates from the core region of the fibre and propagates in a non-planar fashion until it reaches the stoichiometric SiC-4 layer where it deflects to a planar fracture. One explanation for this phenomenon [48] is that the small SiC grains at the fibre core/ SiC sheath interface act as stress concentration sites causing crack initiation. However, the excess carbon located in the inner silicon carbide layers causes a gradient in the modulus. The modulus of the fibre increases as the excess carbon concentration decreases causing the crack to deflect as it travels until it reaches the outer SiC layer where the modulus remains constant. At this point the crack reverts to a planar cleavage crack.

2.1.2.3 Physical Properties of the SCS-6 Fibre

The SCS-6 fibre is strong, creep resistant and thermally stable at temperatures up to 1300°C when compared to PCS derived fibres. Fibre strength deterioration with temperature is given in Figure 2.13 [49]. The physical and mechanical properties of the fibre are given in Table 2.6 [10].

2.2 Fibre - Matrix Interface

In the previous section the properties and physical chemistry of both the matrix and fibre reinforcement, respectively, were detailed. However, the performance of composite systems is dominated by a third quantity, the interface. The interfacial region affects most properties of the resultant composite and successfully tailoring the interface has become an important step in the development of composite systems [50].

The ideal interface is theoretically designed at a point where several independent qualities are all maximized for peak composite performance. However in most systems the interfacial zone is a compromise between several mutually exclusive properties [50-54], including:

- the interfacial zone should be strong enough to transfer load effectively from the matrix to the fibres,
- the interface must be able to promote stress relaxation within the composite through matrix- fibre sliding before the fibre reaches its failure limit,
- the interface should be resistant to environmental attack,
- the interface should be chemically and thermally stable at service temperatures,
- the interface should not adversely affect the component properties desired, i.e. the matrix ductility should not decrease nor fibre strength,

- the fibre should experience complete wetting by the matrix with no voids present at the interface, and
- the interface should not create stress concentrations.

In TMCs, the interfacial morphology is extremely important and can, when taken into account with matrix microstructure, affect the choice of fibre and the protective coating utilised.

2.2.1 Chemistry and Morphology of Ti/ SiC Interfaces

Silicon carbide is considered to be a very stable compound and CVD SiC coatings have long been utilised as a protective layers on more reactive ceramic substrates [54], commercially produced examples being BORSIC and SICABO (amorphous α - SiC deposited on a boron substrate)[52, 55]. However, titanium is a reactive transition metal and even a relatively stable reinforcement such as SiC undergoes significant attack at elevated temperatures. The reaction has a large thermodynamic driving force and will continue at moderately high temperatures until the fibre is fully consumed by the matrix. Diffusion occurs in both directions, the carbon and silicon diffuses through the interface to react with the matrix and the titanium will diffuse through the interface to react with the fibre [31, 55]. Thus, the reaction layer does not act as an effective barrier to further chemical attack until very thick interfaces have been achieved. Typically at this point the fibre surface area has been reduced to such an extent that the fibre could no longer effectively reinforce the composite [31]. The stoichiometric reaction is:

Under isothermal conditions the reaction obeys the parabolic growth law and the thickness of the reaction zone should satisfy the relation [31, 55, 56]:

$$x = K \cdot \sqrt{t} \quad \text{-(2.2.2)}$$

where x is the reaction layer thickness, t is the time at the elevated temperature and K is the parabolic growth rate constant. The constant, K , is highly dependent on temperature and is generally determined using an Arrhenius type relationship [55, 56]:

$$K = K_0 \cdot \exp\left(-\frac{Q}{2 \cdot R \cdot T}\right) \quad \text{-(2.2.3)}$$

where R is the gas constant ($R = 1.9872 \text{ cal K}^{-1}$), T is the absolute temperature and K_0 and Q (the activation energy) are material constants dependent upon initial reaction layer thickness, alloying additions to and carbon saturation of the titanium matrix, crystallography of the titanium alloy (as the reaction proceeds quicker in β titanium than in α) and the composition of the outermost fibre layer. Several values for K_0 and Q under different conditions are given in Table 2.7 [31, 56].

The reaction also occurs in non-stoichiometric ratios resulting in concentrations of TiC_{1-x} in the interface region. When the titanium and stoichiometric SiC are brought into intimate contact the interfacial morphology produced is complex. The interfacial

region typically has a thin ternary phase immediately adjacent to the fibre surface consisting of nonstoichiometric forms of the compound Ti_3SiC_2 . The interaction subzone immediately about this ternary region consists of the products derived from the reaction in eq. (2.2.1), with the titanium silicides directly in contact with the ternary layer and the TiC crystals forming concentric rings around the fibre. The outer subzone tends, for unprotected SiC fibres, to be formed of Ti_5Si_3 [56].

Most typical (Al, V, Nb) alloying elements for the titanium matrix do not segregate to the fibres and are rejected by the interface zone [56, 57]. These additions pile up in the titanium alloy at the interfacial boundary and slow the reaction kinetics. This is most noticeable with Al, [56] and the increase in activation energy for the reaction, Q , is noticeable even in a lightly alloyed matrix such as Ti-6-4 (see Table 2.7).

2.2.2 Protective Fibre Coatings

In addition to alloying elements a variety of protective coatings on most TMC fibres have been employed to protect the fibres from exposure to titanium at elevated temperatures [46, 53]. The most commonly used coating is a thick layer of pyrolytic carbon. Prototype coatings of pure carbon were found, however, to be consumed more quickly than stoichiometric silicon carbide under a similar parabolic law to that given in equation 2.2.2 [55]. Textron SCS-6 fibre coatings, as discussed in chapter 2.1.2.1, currently consist of a complex coating of two binary layers of carbon and silicon carbide that sandwich a layer of turbostratic carbon. Whether or not this adequately

protects the fibre is controversial. While the activation energy for the coating is lower than both stoichiometric SiC and pure carbon it is within the same magnitude [31]. Some researchers believe that as long as the temperature is below 700 °C [31], the kinetics of the reaction are slow enough that the composite will be protected indefinitely, while others note degradation in composite strength at temperatures as low as 600 °C from oxidation attack when TMCs are exposed to atmosphere [58].

In any event, the protective coating changes the morphology of the outer zone of the interfacial region [55]. Rather than the rings of small, irregular Ti_5Si_3 crystals that are produced in the interface with unprotected SiC fibres, large grains of TiC precipitate, protruding out into the matrix [55]. The TiC grains act as stress concentrators and cracks initiate in the interface region or on the boundary between the interfacial zone and the matrix, shown in Figure 2.14 [59]. However, the coating has been reported to blunt the crack tip, thus preventing cracks from advancing into the fibre [50]. This allows TMCs with SCS-6 fibre reinforcements, in some cases, to prevent failure through fibre bridging of the crack [60].

However, the reaction at the interface is extremely detrimental to composite properties in both coated and uncoated fibres. The reaction products are brittle and most cracks initiate at the interface. The fracture toughness of Ti/SiC composites is known to decrease with increasing reaction zone thickness shown in Figure 2.15 [50, 53], and extremely thick reaction zones promote delamination and cavitation of the fibre which prevent effective load transfer [53, 61]. Additionally, there is a 5-7 % volume

contraction [50, 53] associated with the reaction which counteracts the compressive stress imposed by the thermal contraction of the matrix onto the fibre and results in a residual tensile fibre stress as depicted in Figure 2.16 [53].

Whether the competing beneficial and detrimental effects of the coating aid TMC performance seems to depend on the crystallography of the titanium matrix. In one study SCS-6 fibre TMCs were fabricated from different alloys, Ti-6-4 and Ti-15V-3Cr-3Al-3Sn (Ti-15-3) [59]. The Ti-6-4 had an equiaxed $\alpha + \beta$ structure with an average grain size of $\sim 9 \mu\text{m}$. The Ti-15-3 alloy microstructure consisted of equiaxed β grains with diameters approaching the fibre radius ($\sim 48 \mu\text{m}$). Under fatigue testing cracks were observed to grow from the interface until failure. The Ti-6-4 TMC had superior low cycle fatigue performance, probably due to the smaller matrix grain size. More importantly the researchers concluded that Ti-6-4 composite failure was dominated by cracks that initiated at damaged fibres, whereas in the Ti-15-3 TMC crack propagation from fibre damage was not significantly faster than those initiating at the interface. It was suggested that since the fibre diameter was similar in magnitude to the grain diameter in the Ti-15-3 composite that even the growth of cracks from fibre damage would be retarded by microstructural barriers. However, the Ti-6-4 grains were nearly an order of magnitude smaller than the fibres, thus cracks initiated due to fibre failure had already achieved the critical length necessary for rapid crack propagation. Hence, in Ti-15-3 composites it is possible that it is advantageous to utilise unprotected SiC fibres as the stress concentrating TiC particles would not have formed and acted as

crack initiation sites. It is noted that this is speculative as unprotected fibre TMCs were not included in the study.

Other protective coatings have been developed [46, 53]. Recently, the commercially available BP Sigma SM1240 fibre has been marketed with an inner coating of 1 μm pyrolytic carbon and an outer coating of TiB_x in an attempt to reduce coating thickness and improve thermal stability [62, 63]. However, it has been found that at high temperatures the TiB_x coating reacts with the titanium to form TiB needles at the TiB_x /Ti matrix interface and the carbon in the inner coating then migrates into the reduced outer coating creating an irregular TiC layer at the inner layer/ outer layer interface [63]. The TiB needles act as failure initiation sites [62] and some titanium alloying additions, such as V, will segregate to the needles [63]. Hence, is not clear whether this coating scheme is an improvement on that of the SCS-6 fibre [46].

Another coating developed for use in TMCs is sputter deposited yttrium/yttria [50, 53, 61]. This coating acts an effective diffusion barrier and can self- repair through the gettering of oxygen from the titanium matrix. This has the added benefit of removing, oxygen, an interstitial contaminant from the matrix. The coating also acts a laminate and will deflect and blunt cracks propagating into the fibre [64]. However, despite promising results there is currently no commercially available fibre produced with this coating.

2.3 Current Manufacturing Techniques of Axisymmetric TMCs

The challenge to develop reliable processing methods for various TMC geometries has not gone unnoticed; many teams of researchers have undertaken to address this issue. Much of this research has been technically successful and the quality of TMC parts has increased substantially in only a few years [50, 65]. All of the techniques under development attempt to achieve the following goals:

- full consolidation with no matrix porosity is highly desired,
- complete fibre matrix wetting with no interfacial void areas,
- minimal interfacial reaction zones, and
- uniform fibre distribution throughout the composite.

The most prominent methods for constructing TMC components are described below. Methods most suitable for the construction of axisymmetric composites have received more scrutiny, however an effort has been made to cover other techniques that may affect the scope of this thesis.

2.3.1 Liquid Melt Infiltration

The consolidation of composite materials by the forced infiltration of a liquid matrix through a fibre preform followed by subsequent matrix solidification or curing has been previously used in polymer matrix composites [66]. This method remains an attractive route for producing complex shapes and has been adapted to MMC production. It is

used for composite systems with low matrix liquidus temperatures and limited interfacial attack between the constituents. Al-SiC and Mg-SiC composites are two systems where this method is used extensively [57].

However, titanium is considered to be too virulent a matrix material for traditional melt infiltration techniques. This was verified by the liquid infiltration of titanium in a preform of carbon fibres [67]. The fibres were found to have undergone severe dissolution during consolidation.

A modification of traditional melt infiltration techniques for titanium matrices has been developed [68]. This process, rapid infrared manufacturing (RIM), involves rapidly heating the matrix and fibre above the matrix liquidus in an infrared furnace, holding the temperature constant during a short infiltration period and quenching the resultant composite to moderate temperature. The short infiltration time, 1-2 min, reduces interfacial reactions and complex shapes can be potentially produced.

However, only low melting Ti-Ni alloy matrices have successfully used RIM to cast TMC parts [68]. A Ti-6-4 TMC was also produced but only after modifying the SCS-6 fibre's protective coating by depositing an additional 2 μm thick TiC protective coating and adding an additional 0.5 wt% C to the alloy. Carbon is an interstitial contaminant for Ti and even a small addition will reduce matrix ductility. Additionally, the TMC sections produced were quite narrow, scaling up the process for larger sections would slow the heating/ cooling rate of the process and increase fibre degradation. For the

narrow Ti-6-4 composite section produced the reaction zone thickness was determined to be 1-2 μm which is comparable to other methods [68]. However, this does not include the already existent 2 μm thick TiC protective layer.

2.3.2 Matrix Coated Fibres

An attractive method of creating MMCs is to coat the fibres with a layer of matrix material, lay up the preform of coated fibres and then consolidate the composite using diffusion bonding and hot pressing techniques. Several methods to produce matrix coated fibres (MCF) have been explored. Two of these techniques show high potential for producing quality composites with uniform fibre distribution, high (>79 %) fibre volume fractions and little fibre damage [69].

Electron beam evaporation and vapour deposition (EBED) is a MCF technique that shows great promise [64, 70]. An electron beam gun delivers a stream of electrons onto a source rod of titanium alloy. The alloy melts into an 'evaporation' pool geometry and vaporizes. The vapour then condenses on the fibre forming the coating, shown schematically in Figure 2.17 [69]. The rate of evaporation and deposition is directly dependent on the vapour pressure of the element and the power of the electron beam gun. With high power (~ 579 kW) guns deposition rates of 3000 $\mu\text{m}/\text{min}$ can be achieved with Ti based matrices [69]. This method has the advantage of depositing coatings at low temperatures and high rates minimising interfacial attack. The coating of matrix material can be quite thin and composite preforms can be made up of mixtures

of coated and uncoated fibres before consolidation resulting in very high volume fractions, demonstrated in Figure 2.18 [64]

However, there are practical limitations to this technique. Each element has a different vapour pressure and co-evaporation of the multiple elements in a single source rod is only feasible if the vapour pressure of the individual elements are within two orders of magnitude. A chart of the vapour pressures of various elements is given in Figure 2.19 [64]. Several of the alloying elements for titanium alloys have high liquidus temperatures and cannot be co-evaporated. This difficulty can be avoided by using multiple evaporation sources to produce complex compositions [70] provided the vapour flux rates for the different alloys allow complete mixing of the various streams. Even utilising multiple evaporation sources the composition of alloys deposited using this technique can vary significantly. The composition of a Ti-6-4 alloy deposited on a SiC substrate was found to vary between Ti-3Al-5V to Ti-5Al-4V [68, 69] and it is to be expected that more complex alloys would be more difficult to control compositionally. Additionally, the fibre must be carefully handled to prevent an eccentric coating, shown in Figure 2.20 [69]. It was also discovered that SCS-6 fibres consolidated with less than a 30 μm inter-fibre spacing experienced radial cracking upon consolidation, hence limiting the maximum fibre volume of the composite [70].

Another MCF technique that has displayed promise is magnetron deposition or sputter deposition [69]. This technique utilises a cathode of the source alloy which fires ions of the coating onto a substrate (the anode) through an inert gas. A magnetic field is

deployed normal to the surface of the cathode, containing the ions and increasing the efficiency of the process [71, 72]. A schematic illustration of the process is depicted in Figure 2.21 [71]. A major advantage of this technique is that it produces coatings of the required composition with negligible variation [71] as the sputtering rates for most elements vary only within a factor of ~ 2.5 [69]. Another advantage is that this method deposits the matrix at a lower processing temperature than EBED, reducing interfacial attack and allowing metastable microstructures to be deposited. However, due to the higher deposition energy more coating flaws may be present and the residual stresses present may be higher [64]. The deposition rate for magnetron sputtering is far less than that of EBED [70]. A 30 μm coating of Ti-6-4 on an SCS-6 fibre was accomplished in ~ 5 to 10 minutes by EBED processing; the same coating took between 1 and 12 hours using magnetron sputtering [69].

A concern shared by both methods is contamination from the residual atmosphere in the deposition chamber. This causes oxygen embrittlement of the matrix [69]. EBED suffers less from this phenomena as the deposition time and thus exposure time is much shorter. Oxygen pick up during magnetron sputtering has been reported but it can be controlled by reducing deposition time and altering the geometry of the sputtering (see Figure 2.22) [69]. Recent developments in magnetron sputtering of titanium matrices onto SiC substrates indicate that oxygen contamination can be eliminated by strict control of the deposition chamber [73].

2.3.3 Thermal Spray Techniques

The high cost and low deposition rates of matrix coated fibre techniques has engendered interest in other methods of matrix deposition [68, 74]. Spraying a fine mist of molten matrix droplets onto a substrate preform has captured the attention of several researchers. Several coating techniques have been modified to produce the desired results.

Electric arc spray forming is one technique that has been adapted to produce TMC monotapes [74, 75]. Electric arc spraying consists of two conducting source wires that are fused in an electric arc generated between the wire tips. The molten metal at the wire tips is atomised by a stream of high velocity gas which propels the molten metal onto the drum wound fibre preform, shown in Figure 2.23 [75]. The droplets infiltrate the preform and produce a TMC monotape. The monotape is then cut and hot pressed to form a fully dense composite.

Electric arc spraying is efficient and has lower equipment costs than MCF and competing thermal spray techniques. Electric arc spray can operate in a positive pressure environment [74]. TMC monotapes were successfully manufactured at 2 kPa above atmospheric pressure. This is advantageous as the positive pressure prevents ingress of air and hence reduces the possibility of oxygen contamination of the matrix.

Electric arc spraying parameters must be strictly controlled. The fibre to fibre spacing, the atomising gas pressure, the distance from arc to target, and the arc current must all

be optimised. If not the molten metal droplets will either be too small and freeze before impinging on the fibre, thus, failing to adhere effectively [74], or they will be too large and remain molten on the fibre long enough for significant interfacial attack to occur [75]. Porosity in arc sprayed composite monotapes can be as high as 48% if organic binders are used to hold the substrate in place.

Another thermal spray technique used to produce TMC composites is vacuum plasma spray or VPS. There are two forms of plasma spray, direct current plasma spray and induced plasma deposition [76]. The methods use different techniques to produce the plasma, each with ramifications to the spray process. Direct current plasma spray applies a potential difference across two fixed electrodes. An inert gas flows between the electrodes turning into the plasma source. Titanium powder is fed into the plasma stream where it is melted and flung at the substrate, illustrated in Figure 2.24 [77]. This is a high velocity plasma stream and hence uses fine ($\sim 20\text{-}99\text{ }\mu\text{m}$) powders that will melt quickly. An optimised direct current spray can have matrix porosities as low as $\sim 1\text{-}3\%$ [78].

Induced plasma spray uses an inductively coupled radio frequency plasma generator to produce the plasma [79]. Titanium powder is fed into the plasma to provide the molten matrix, however, since the plasma stream generated by this technique is much slower, coarser powder may be used [76]. The matrix porosity in induced plasma deposition is typically greater (5-25%) than that of direct current plasma deposition. However, the interstitial contaminant concentrations are much lower due to the larger powder size and

the droplets impact at a lower velocity thus reducing fibre damage. Interfacial reactions in the initial as sprayed condition are negligible utilising either technique [76-79].

The gas generally utilised in VPS to supply the plasma is typically Ar/H₂, but, this was found to promote brittleness in TMC monotapes [78]. An alternate plasma gas mixture that can be utilised in the production of TMCs is Ar/N₂.

Both thermal spray and MCF techniques succeed in depositing matrices onto the SiC fibre without significant interfacial reaction occurring. However, to obtain a fully dense composite ring the monotapes/ MCF must be laid up into a preform and diffusion bonded [76, 77]. During this portion interfacial attack occurs in the composite negating the advantage of this technique over foil/fibre diffusion bonding methods [79]. Attempts to eliminate the diffusion bonding stage have been attempted by independent teams of researchers in the United Kingdom and Canada [80, 81]. In these processes the VPS deposition of the titanium matrix is continuous and the silicon fibre is wound onto a mandrel. In one set of experiments the fibre was wound continuously throughout the spray process [81], while trials of continuous, semi-continuous and discontinuous winding strategies were performed in the second program [80]. The two methods also used different strategies for the deposition of the matrix. The British researchers used a direct current plasma gun [79] while the Canadian initiative favours inductive plasma deposition [81]. These programs had limited success; multiple layer composites were produced without significant fibre damage, however, matrix porosity from 'shadow'

spray effects necessitated the hot pressing/ hot isostatic pressing of the rings [80, 81]. Still this methodology remains promising.

2.3.4 Powder Fibre Processing

An economically advantageous method is to create a composite tape of SiC fibres filled with matrix powder. This tape is then carefully laid up and diffusion bonded to produce a consolidated part. Textron and Atlantic-IMT have individually developed powder monotapes [82, 83] for consolidation into composites. Despite the economy of this method it has severe limitations. In order to hold the powder in place on the monotape an organic binder must be applied liberally throughout the composite. Despite off-gassing and binder burn off pre-processing stages, the binder chars leaving deposits that contaminate the composite part. There is also significant porosity in the fabricated composites rendering this method unsuitable for primary axisymmetric TMC components.

2.3.5 Foil Fibre Foil Method

The current benchmark processing method for constructing TMC components is the foil fibre foil methodology. In this method foils of the matrix metal are sandwiched with alternating layers of fibre, typically in an organically bound fibre mat. The resulting preform is diffusion bonded under vacuum to produce a fully consolidated composite. Diffusion bonding techniques such as hot isostatic pressing (HIP'ing), hot pressing, and triaxial hot pressing have been successfully adapted to this processing

method [84]. TMC components produced by this technique range from flat plates to corrugated cores [85]. Major advantages of this technique are the simplicity of the apparatus necessary to produce the TMCs and the lack of porosity in the finished composite.

Foil fibre foil methods have several disadvantages. The cost of titanium alloy foil is high negating the economic advantage gained in the initial apparatus cost [69, 70]. The fibres often 'swim' or debuckle during consolidation leading to poor fibre distribution and fibre touching [70]. Finally, the fibre mats are typically bound with an organic binder. This binder typically leaves char residue during the consolidation phase which is detrimental to the final composite properties [86]. The fibres can also be aligned using titanium or molybdenum wires. However it has been determined that these aligning wires lead to severe interface reactions detrimental to the composite during consolidation [87]. Another disadvantage that proponents of alternate methods often cite is the formation of interface reaction products during the diffusion bonding phase of the process. While this is true, all the methods currently in use utilise a diffusion bonding step to achieve full consolidation. The interface layer developed with optimised diffusion bonding envelopes and the foil fibre foil method are not appreciably greater than that of the other methodologies [79].

The foil fibre foil techniques are most appropriate for flat geometries. However, production of axisymmetric reinforced components has been demonstrated by Textron [86]. This variation of the foil fibre foil method utilises a 'doily' of organically bound

hoop wound fibres. These doilies are then sandwiched between thin plates of titanium foil to produce the composite. A new development on this process removes the 'doily'. This new technique scratches the titanium foil disk in a phonograph pattern. The fibre is then laid down in the groove on the disk and fixed into place with an organic binder. The advantage of the 'phonograph' technique over that of the 'doily' method is the reduction in binder necessary to place the fibre in the desired orientation [83].

Another technique derived from the foil fibre foil approach is the wire wrapping technique developed by Atlantic-IMT. This technique fixes a layer of titanium wire onto a mandrel, then a layer of silicon carbide fibre is laid on the interstices between the matrix wires. The layers are then wrapped onto the mandrel creating the composite preform which is subsequently hot isostatically pressed. A schematic sketch of the wrapping process is shown in Figure 2.25 [88]. This method does not require significant amounts of binder and the titanium wire utilised is cost effective when compared to titanium foil. Prototype rings produced by this method have uniform fibre distributions. However the width of the composite part is dominated by the number of filament spools mountable on the creel system and currently rings 16 fibres wide are the maximum realistically produceable [82]. Furthermore the fibre volume fraction of this method is limited by the titanium wire diameter. Hence, the extremely high volume fractions demonstrated by the MCF and thermal spray techniques (< 79%) are difficult to obtain with this method.

2.4 Solid State Consolidation of TMCs

Due to the high reactivity of titanium with the silicon carbide reinforcement, consolidation of TMCs is generally done at temperatures well below the liquidus for titanium [80]. Hot pressing and HIPing of green pre-forms of fibres and matrix is the common consolidation method. As stated in chapter 2.3, the preforms can be laid up utilizing thermal spray monotapes, wound MCF, the traditional foil-fibre-foil technique and the co-mingled filaments explored in this thesis.

The consolidation parameters are limited by several independent concerns. The consolidation temperature and time must be minimized to reduce the detrimental reactions at the fibre-matrix interface. The consolidation pressure should be raised to accomplish this, but excessive fibre swimming and fibre damage/breakage is possible at the higher pressures [89] thus limiting the applicable pressure. Finally, to obtain a fully dense TMC geometry both the temperature and pressure must be high enough to promote diffusion bonding of the titanium matrix. The competing trade-offs are presented graphically in Figure 2.26 [5].

2.4.1 Mechanisms of Solid State Consolidation of Metal

The mechanisms that control the joining and consolidation of metals are highly dependent on the temperature and pressure applied. The major mechanisms can be discretized into time independent e.g. plastic deformation, and time dependent phenomena consisting of transient creep and steady state creep.

2.4.1.1 Plastic Yielding

As the discrete surfaces impinge on each other at the HIP temperature and pressure they begin to deform elastically. The deformation of two elastic surfaces is non-linear and plastic deformation has been determined to commence for contact between two spherical particles when:

$$\sigma_s = \zeta \cdot \sigma_y \quad \text{-(2.4.1)}$$

where σ_s is the stress at the contact area between the two spheres, σ_y is the yield strength of the material and ζ is approximately 3 in this case. Constant ζ is density dependent in a real system however, and most HIP models [91] take into account either the density of asperities or the coordination number of the discrete powder particles in contact with each other in the system.

Plastic deformation is time independent as it is dependent on the stress state in the material, the yield strength of the material at temperature, and work hardening and frictional effects.

2.4.1.2 Transient Creep

Creep is defined as the plastic flow of a metal under constant stress at an elevated temperature [91]. A typical creep curve is shown in Figure 2.27 [91]. The curve can be broken into three discrete stages, primary or transient creep, secondary or steady state creep and tertiary creep.

Transient creep begins with a high strain rate which then reduces until it reaches the secondary creep phase. The high initial rate is thought to be caused by the stress induced movement of dislocations through barriers [91]. As the strain increases the number of barriers that can be readily overcome by the dislocations is reduced and they are pinned at the barriers. This work hardening phenomenon impedes the movement of the dislocations and the different recovery mechanisms dominate secondary creep behaviour.

2.4.1.3 Steady State Creep Mechanisms

Steady state creep is achieved when the work hardening of the material is compensated by one of or a combination of recovery mechanisms. The dominant recovery mechanism depends upon the microstructure, applied stress and temperature to which the material is exposed.

At low stresses and high temperatures diffusion mechanisms are the dominant forms of creep. Diffusional creep occurs through the migration of vacancies from grain boundaries experiencing tensile stresses to those experiencing compressive stresses. This leads to the elongation of the grains and metal flow as the atoms flow in the reverse direction. This type of creep is termed Nabarro-Herring diffusional creep and can be described by [92]:

$$\dot{\epsilon}_s \approx \frac{7 \cdot \sigma \cdot D_{gb} \cdot b^3}{k \cdot T \cdot d^2} \quad -(2.4.2)$$

where $\dot{\epsilon}_s$ is the steady state creep stress, σ is the applied stress, D_{gb} is the grain boundary diffusion constant, b is Burger's vector, T is the absolute temperature, K is Boltzmann's constant, and d is the grain size.

A related diffusional creep process, termed Coble creep, involves atomic diffusion along grain boundaries and can be described by [92]:

$$\dot{\epsilon}_s = \frac{50 \cdot \sigma \cdot D_{gb} \cdot b^4}{k \cdot T \cdot d^3} \quad -(2.4.3)$$

Both diffusional mechanisms are proportional to the grain boundary area, hence the diffusional creep rates are inversely proportional to grain size.

At higher temperatures and pressures creep is dominated by the dislocation escape from sub-grain boundaries. This may occur either through the cross slip of screw dislocations or climb of edge dislocations away from dislocation barriers. Dislocation climb requires the greater activation energy of the two processes and thus controls the creep rate in the material [91]. This process is controlled by the vacancy density and self-diffusion of the material, however, it is differentiable from the above processes in that it is not sensitive to grain boundary area. This form of creep is termed dislocational creep and can be described by [92]:

$$\dot{\epsilon}_s \approx \frac{A \cdot D \cdot G \cdot b}{k \cdot T} \cdot \left(\frac{\sigma}{G} \right)^5 \quad -(2.4.4)$$

where D self-diffusion constant, G is the shear modulus and the other variables are as defined for equation 2.4.2.

Another mechanism that occurs at high temperatures is grain boundary sliding. This is not an independent phenomenon and follows from both diffusional and dislocation creep mechanisms. For instance the diffusion of atoms from grain boundaries in compression to grain boundaries in tension would cause the grains to separate. Grain boundary sliding is thus necessary to retain contiguity. This mechanism, grain boundary sliding with diffusional accommodation is thought to control superplastic forming of metals [92].

2.4.2.1 Modelling the Solid State Consolidation of Foil-Fibre-Foil Preforms

As discussed in section 3.5 of this chapter, the foil-fibre-foil technique has been fairly well developed by industry. Despite this, consolidating parameters are rarely detailed. In the open literature several teams of researchers have modelled the behaviour of foil fibre layups. The consolidation process was discretized into three distinct stages by modifying the models for the consolidation of powders : (1) instantaneous plastic deformation of the foil, (2) time dependent deformation of the disparate foils until they touch, and (3) creep of the matrix material to close the voids at the sides of the fibres, shown schematically in Figure 2.28 [93]. The creep consolidation of the latter two stages is dominated by the diffusion controlled movement of dislocations. Utilising the commercial FEM software ABAQUS to model the consolidation stages, densification maps for temperatures of 789 °C, 864 °C, and 938 °C were developed for different

pressures, presented in Figure 2.29 [94]. These maps gave good agreement with experimental results [94, 95].

An alternate methodology models the consolidation as one stage [96]. Using the FEM program, ALPID, the metal foils were assumed to deform based on the power law creep model and the fibres were modeled as rigid dies. From the resultant metal flow patterns an area weighted average effective strain and the time to achieve consolidation of 96% of the lay-up was developed. This allowed the calculation of an average metal flow stress, σ^* . Utilizing this concept to determine a ratio, F , between the HIP pressure and the flow stress for a given fibre array, the effect of several independent parameters on consolidation time was estimated. This development was used to correlate the effects of geometry (fibre to fibre spacing and foil thickness), rate sensitivity and the frictional shear factor between the fibres and the foils. It is postulated, from the data developed, that F was relatively insensitive to both the creep rate sensitivity and the geometry of the lay ups, while it is extremely sensitive to the frictional shear factor [96]. Further work determined that in some alloy/ fibre systems that the lot to lot variations in material properties and grain size of the alloy could affect the consolidation time. However, it was concluded that for an alloy produced in large quantities, such as Ti-6-4, material property variations were small and thus variations in consolidation time due to material chemistry and grain size would be fairly small [97].

The fibre array geometry has been observed to affect the consolidation behaviour of multilayer composites. The consolidation of the ear formed at the triple junction at the

fibre and the two foils is dependent on whether the fibre array is rectangular or hexagonal as seen in Figure 2.30 [94]. The ear is caused by different mechanisms. In triangular or hexagonal arrays the ear is attributed to matrix bending. In rectangular arrays the ears have been theorized to form from an earring defect caused by matrix incompressibility. If the matrix contact is defined by a contact angle ϕ , defined in Figure 2.31 [98], then the critical contact angle can be defined by [98]:

$$\phi_c = \arccos\left(\frac{R}{h}\right) \quad -(2.4.5)$$

,if the foil material is allowed to be ejected sideways and,

$$\frac{h}{R} \cos \phi_{cc} + \frac{R}{b_0} \sin^3 \phi_{cc} = 1 \quad -(2.4.6)$$

,if the foil material is constrained by dies, where the symbols are defined in Figure 2.31 [98]. In addition to the earring defect, matrix barrelling is caused by shear resistance to matrix flow caused by friction at the fibre- matrix interface. This can be seen in the micrograph displayed in Figure 2.32 [98]. The ear defect can be seen at the arrows and the barrelling effect is evident by the extended convex curvature of the foil surface. The implications of this work is that to close the ears caused in a rectangular array the strain experienced by the matrix must be much higher than that in a triangular array. Thus consolidation of rectangular fibre arrays takes longer to perform. This was confirmed by the FEM simulations discussed above.

Once consolidation has been completed, control of the cool-down phase of the process is vital to reduce residual thermal microstresses within the composite. The thermal coefficient of expansion of titanium and silicon carbide is nearly an order of magnitude different, thus the residual thermal stresses can significantly affect the thermal fatigue life of the composite. Work in developing the ideal cooling rate/ pressure envelope for MMCs has been conducted utilising non-linear programming algorithms to assess the effect of the several independent parameters involved. The algorithm optimizes the cooling envelope by inducing plastic yielding in the matrix to remove the residual stresses. Using the envelopes developed, the residual microstresses in a copper / graphite composite was reduced by 15%. However, application of an optimized cooling envelope to a Ti-15-3-3-3 / SiC system provided negligible reductions in the thermal microstresses [99]. It was theorized that the baseline cooling envelope in this case had already been optimized to an extent by trial and error. Further work to reduce residual stresses has been performed; however, in this case post fabrication cycles were performed. Each cycle consisted of a heat treating the samples while subjecting them to either longitudinal, transverse or hydrostatic stresses [100]. Qualitative residual stress reduction was achieved; however, the reduction in stress did not agree with the values predicted by FE analysis. One possible explanation for this is that stresses developed from interface products were not accounted for in this study.

2.4.2.2 Modelling the Solid State Consolidation of Thermal Spray Monotapes

Thermal spray monotapes of titanium onto SiC fibres present different modelling challenges. The final consolidation of the “ears” at the fibre foil juncture, which is approximately half the consolidation time [97], is unnecessary with plasma sprayed monotapes. The monotapes, as a result of the spraying process, generally have one smooth surface and one rough surface. The rough surface is the outside spray surface while the smooth surface is the surface originally in contact with the drum. Thermal spray monotapes also exhibit considerable porosity due to the high solidification rate of the powder. The liquefied powder freezes before it can fully spread out and thus creates voids [91]. Thus, consolidation of monotapes involves the bonding of the rough surface of one monotape to the smooth surface of another and the healing of the internal porosity (see Figure 2.33)[91]. Modelling of the consolidation has been performed [91, 101] by modifying methods developed for the consolidation of powders. By defining a geometrically representative volume element, GRVE, that is representative of either stage of consolidation, i.e. the impingement of asperities onto a smooth surface or the compaction of a spherical void, it is possible to determine the strain rate of the element. In both cases the strain rate is a combination of three mechanisms; plasticity, dislocation creep and diffusional flow (i.e. Nabarro-Herring creep or Coble creep).

Modelling of the bonding of the surfaces between monotapes was simplified into the impingement of hemispherical asperities onto a flat surface. This is a fairly standard method of determining adhesive and diffusive properties of materials [102]. Modeling

of the densification is performed by utilising statistical methods to account for the variation in asperity height [91] or by approximate solutions developed for the compaction of powder [101]. The densification caused by plastic flow is determined while the dislocation creep and diffusional mechanisms have their densification rates modelled. Healing of porosity and densification of the last 0.08% of the porosity of the composite is defined geometrically as the collapsing of a spherical void within a thick walled sphere of the material. From these assumptions HIP pressure and temperature maps can be developed. However, the generalisations involved in the mathematical modelling such as the geometry of the porosity, material creep parameters and the effect of the fibre on the consolidation process leads to inaccuracies.

2.5 Methods of Estimating Composite Properties

The implementation of composites into engineering designs requires an understanding of their mechanical behaviour. In this vein several models predicting composite performance and corresponding methods to develop the necessary parameters have been developed. Each model is based on certain underlying assumptions, hence the accuracy of the model depends on its efficacy in describing the composite system studied. There are a multitude of composite models and a full review is not within the scope of this thesis. However, the standard methods and the models with relevance for MMC systems will be described in this section.

2.5.1 Voight Averaging

Voight averaging, commonly referred to as the **Rule of Mixtures (ROM)**, is widely used in literature to estimate the strength and elasticity of composites. This methodology assumes that both the matrix and the fibre experience equivalent strain when the composite is stressed. Thus, an efficient transfer of load between the matrix and the fibre is required [51] and end effects are ignored. The load on the composite to produce a strain is the summation of the load on the fibre plus the load on the matrix. From this the composite strength can be derived as [66]:

$$\sigma_c = \sigma_f \cdot V_f + \sigma_m \cdot (1 - V_f) \quad -(2.5.1)$$

and similarly the composite's modulus of elasticity can be defined [51]:

$$E_c = E_f \cdot V_f + E_m \cdot (1 - V_f) \quad -(2.5.2)$$

where σ_c , σ_f , σ_m , E_c , E_f , & E_m are the ultimate strengths and modulus of elasticities of the composite, fibre and matrix respectively, and V_f is fibre volume fraction.

2.5.2 Modified Rule Of Mixtures

The ROM model predicts that all the fibres will fail at the same time and that the matrix will reach its ultimate tensile strength before fibre failure. However, the fibre is generally more brittle than the matrix and can fail well before the matrix has reached its

failure strain [66]. If σ_m' is defined as the stress in the matrix at fibre failure strain then [66]:

$$\sigma_c = \sigma_f \cdot V_f + \sigma_m' \cdot (1 - V_f) \quad -(2.5.3)$$

This modification of the ROM model (MROM) implies that there is a minimum volume fraction that must be introduced for useful strengthening of the matrix to occur. This fibre volume fraction is defined as [66]:

$$V_{crit} = \frac{\sigma_m - \sigma_m'}{\sigma_f - \sigma_m'} \quad -(2.5.4)$$

2.5.3 Global Load Sharing

The assumption that all the fibres reach the same failure strength is inaccurate and to account for this the distribution of fibre failure strengths is often fitted to a two parameter Weibull distribution. The general form, defining the probability of fibre failure of a fibre of length L under an applied load σ is [103]:

$$P(\sigma) = 1 - \exp(-L \cdot \psi \cdot \sigma^m) \quad -(2.5.5)$$

where ψ is the Weibull scale parameter, and m is the Weibull shape factor. If the mean (48% failure) fibre failure strength, σ_0 , is known for a gauge length L_0 then ψ can be replaced [104]:

$$P(\sigma) = 1 - \exp\left(-\left(\frac{L}{L_0} \cdot \left\{\frac{\sigma}{\sigma_0}\right\}^m\right)\right) \quad -(2.5.6)$$

From this probability profile the expected strength of a bundle of the fibres can be determined [103]. It is assumed that when a fibre fails, that the load is redistributed equally among the remaining fibres (i.e. no stress concentrations on the fibres close to the broken fibre), and that the matrix/ fibre interface will delaminate with the cracks propagating into the matrix before fibre failure occurs (a typical assumption for MMC and ceramic matrix composites). Then it can be assumed that the response of a composite can be estimated by equation 2.5.1 until a critical matrix cracking strength is reached. At this point a matrix crack completely propagates through the sample. In the plane of a crack the fibre is assumed to carry the entire load, recalling that delamination of the interface prevents the crack from propagating into the filament. Thus, sliding of the fibres is assumed to occur and the load is transferred to the matrix through the uncracked portion of the interface through shear along a critical length [104]:

$$L_s = \frac{r \cdot \sigma_{mc}}{2 \cdot \tau} \quad -(2.5.7)$$

,where r is the fibre radius, σ_{mc} is the stress necessary for matrix cracking, and τ is the interfacial debond shear strength. This continues until the matrix cracks are all spaced such that σ_{mc} can no longer be fully transferred to the matrix through shear [104]. The fibres then begin to fail. Along any matrix crack the definition of unbroken fibres is

that the fibre cannot have failed along the critical length, as illustrated in Figure 2.34 [104]. The force balance for unbroken and broken fibres within a critical length in the composite can then be performed. Applying the Weibull distributions of fibre strength the bundle strength of the filaments can be expressed as [105]:

$$\sigma_b = \left[\frac{\sigma_0^m \cdot \tau \cdot L_0}{r} \right]^{1/(m+1)} \cdot \left[\left(\frac{2}{m+2} \right)^{1/(m+1)} \cdot \left(\frac{m+1}{m+2} \right) \right] \quad -(2.5.8)$$

The load carried by the matrix material varies. For a metal matrix it has been hypothesized that the amount of load carried by the matrix cannot be greater than its yield strength. If residual stresses on the matrix are neglected, then the total composite strength will be [103, 104]:

$$\sigma_c = V_f \cdot \sigma_b + (1 - V_f) \cdot \sigma_y \quad -(2.5.9)$$

where σ_y is the matrix yield strength. Table 2.8 [104] shows a comparison of yield strengths derived for several different TMCs utilising equations 2.5.3 and 2.5.9 compared to experimental results. The Global Load Sharing method tends to be a superior method of estimating composite strength.

2.5.4 Local Load Sharing

Another method of predicting MMC strength is to account for matrix plasticity and residual thermal stresses induced in the composite. It also factors in stress

concentrations for fibres adjacent to broken fibres and is thus a local load sharing model. In this methodology fibres nearest to the broken fibres are assumed to carry more of the load. A schematic of the model geometry is given in Figure 2.35 [106]. The broken fibres are assumed to form a core, surrounded by a sheath of matrix material. The surrounding row of unbroken fibres are assumed to form a concentric cylinder about the core of broken fibres. Using force equilibrium equations and solving for the displacement of the broken fibres, the unbroken fibres, and the matrix, the elastic and plastic stress concentration factors for fibres surrounding the broken fibre can be determined. The first fibre failure can be determined from [106]:

$$\sigma_1 = \left(\frac{1}{N \cdot L_0} \right)^{1/m} \cdot \sigma_0 \quad -(2.5.10)$$

where N is the number of fibres in the sample, and the remaining variables are the Weibull parameters defined in section 2.5.3. From this the average interfacial shear strength can be determined. If the average interfacial shear strength is greater than the debond shear strength, debonding of the fibre matrix interface occurs. If this happens then plastic stress concentration factors are used to calculate subsequent failures, otherwise elastic concentration factors are utilised. Subsequent fibre failures can then be determined [106]. Results of this method were compared to experimental measurements for a Ti-24-11 / SCS6 TMC with good agreement.

Another Local Load Sharing model predicts that the fibre contribution to the composite can be described as [107]:

$$\sigma_r = \sigma_0 \left(\frac{L_0 \cdot (m-1)}{L \cdot N \cdot m} \right)^{1/m} \quad -(2.5.11)$$

This model suggest that the larger the composite volume, the lower the fibre strength contribution will be to the composite because the probability of a severe fibre flaw increases [107].

It has been noted in the literature [105] that Global Load Sharing techniques are accurate for composite systems that have low interfacial debond strength while Local Load Sharing mechanisms are more accurate for systems with a high interfacial debond strength.

2.6 Methods of Measuring Bond Strength

Experimental tensile testing of fabricated composites is the most commonly utilised method of determining TMC strengths. However, tensile testing of composites requires the fabrication of tensile test specimens that meet standard test requirements [37]. For fabrication methods dedicated to ring geometries, such as thermal spray winding or the co-mingled filament winding developed in this thesis, the production of these coupons is not trivial. Mechanical testing of ring geometries is currently limited to burst testing in which a disk is spun at speed until failure [12]. These tests are wasteful as only one test can be performed per ring. Thus, for statistical averaging of the results several

rings of varying fibre fractions must be manufactured. Another method to determine composite strength is to utilise the models discussed in section 5 of this chapter.

However, to utilise these methods several parameters must be determined, notably the mean strength σ_0 for a gauge length L_0 and the interfacial bond strength τ_s .

2.6.1 Fibre Pull Out Testing

Fibre pull out testing consists of extracting a fibre that is half embedded in the matrix through the application of a tensile load [50]. The load necessary to remove the fibre from the matrix is then recorded as either a function of time or fibre displacement.

From this data the maximum debond strength of the composite, σ_d and the initial frictional pull out stress, σ_{fr} [108] can be determined. This method is extensively used for polymer matrix composites; however, the difficulties in specimen preparation limit the applicability of the procedure for MMCs [53]. The test is also not easily adapted for ring geometries.

2.6.2 Full Fragmentation Testing

Another test devised for developing interfacial shear strength properties is the full fragmentation test or the Fraser mechanical test. In this test a single fibre is introduced into the matrix. The specimen is then subjected to a large tensile plastic strain [53].

The mean aspect ratio of the broken fibre fragments is then analyzed. From this data the Weibull strength distribution of the fibre, the critical fibre length, and the interfacial debond shear strength can be determined [109]. This test has been used to determine

MMC properties [109], however, the need to prepare special samples for the test, some controversy over the theoretical analysis [50] to obtain the interfacial shear strength, and the inability to learn the frictional shear strength [109], hamper the popularity of test.

2.6.3 Fibre Push Out Testing

The most widely utilised method of measuring the interfacial strength of MMCs is the fibre push out test where the fibre is pushed out of a thin slice of the matrix material using an indenter [53]. In most cases a conventional Vickers microhardness indenter is used to develop the data by varying the indentation load until fibre push out has been achieved. Figure 2.36 [109] demonstrates the set up for one such test. Typically negligible sample bending and constant shear stress is assumed throughout the sample with the load being related to the interfacial debond shear stress by the relation [109]:

$$P_{cr} = 2 \cdot \pi \cdot r \cdot (w - \xi) \cdot \tau_s \quad -(2.5.1)$$

where P_{cr} is the critical load to push out the fibre, r is the fibre radius, w is the sample thickness, τ_s is the interfacial debond shear strength and ξ is a factor to account for surface effects. The interfacial frictional shear stress can be estimated by pushing the fibre back into the composite [50].

Recently more sophisticated equipment has been developed for fibre push out tests. These apparatuses can measure the displacement of the loaded end of the fibre with the

load to determine the onset of fibre debonding and the frictional shear stresses. A graph of the load vs displacement for a fibre push out is presented in Figure 2.37 [109].

3.1 Selection of Matrix Alloy

The Ti-6-4 alloy was chosen as the matrix alloy for the composite part for several reasons. As the most commonly used titanium alloy, many of the components that could potentially be replaced by TMCs are currently being manufactured from Ti-6-4. The benefits of adding continuous fibre SiC reinforcements to this alloy allow direct comparisons to be made on a case by case basis. Ti-6-4 is also used extensively in low pressure, low temperature compressor stages, applications for which TMCs will probably gain initial acceptance in the gas turbine industry. While TMCs are being postulated as materials for high pressure compressor stages [11, 29, 30], with service temperatures reaching 800 °C, current TMCs show degradation of properties when exposed to temperatures of 500 °C [31] and interfacial reactions at elevated temperatures remain a concern (see chapter 2.2). Finally, the majority of work performed in the TMC field has utilised Ti-6-4 as the matrix alloy allowing for direct comparisons to be made with results from literature.

Ti-6-4 wire was procured from Titanium Industries Limited. The wire has a nominal diameter of 0.006" $\pm$ 0.0004" (152 $\pm$ 60 μ m) and complies with ASTM B-345 grade 5 alloy wire (Ti-6-4) compositional and strength requirements. The chemical and mechanical properties of the wire are given in Table 3.1.

3.2 Selection of Fibre Reinforcement

Textron SCS-6 fibre was utilised in this thesis for several reasons. It is a mature fibre that has been used extensively for titanium composites [62]. The only other SiC fibre produced for TMC's, British Petroleum's Sigma SM-1240, is a relatively new fibre and is still undergoing characterisation. Also, the interfacial reaction of the protective coating of the Sigma fibre with titanium is complex and it has not been determined whether the TiB_x coating is an effective shield [111] or if it will lead to the degradation of the composite properties. Finally, at the initiation of this thesis the Sigma SM-1240 was not readily available.

The SCS-6 fibre was procured from Textron Specialty Materials. The fibre has a nominal diameter of 0.004" (143 μm) and has the guaranteed properties published by Textron Specialty Materials discussed in chapter 2.

4.1 Design Concept

The goal of the Filament Winding Apparatus (FWA) is to wind a mandrel with SCS-6 silicon carbide fibres and titanium wires to produce a composite preform for consolidation. The FWA utilises a winding strategy to build up the layers of the combined filaments. This differs from the method developed by Atlantic-IMT discussed in chapter 2, section 3.5 in that it has a translational component, as well as a rotational component. The advantage offered by this strategy over that of the filament wrap methodology is that pieces of variable length can be manufactured without a large creel system. Hence rings of longer lengths can be potentially produced utilising a filament winding approach. A goal of this thesis was to demonstrate that composite ring preforms could be produced by wire winding and that the method was viable for producing hoop reinforced TMC rings.

The FWA must have both rotational and translational elements in order to wind the mandrel. There are two possible methods of achieving this:

1. Rotational and translational assemblies are separate, with the mandrel rotating and the translational movement being developed by moving the filament guide the required distance.

2. Rotational and translational assemblies integrated, either with a stationary mandrel and a rotating and translating fibre pay out or a stationary fibre guide and a rotating and translating mandrel.

The design methodology selected was that of a rotating and translating mandrel with a stationary guide. It was thought that this concept was the simplest to design and produce in the short time allotted to this portion of the project. Another concern that was paramount to the design was the control of the filament orientation during the wind. It was thought that this would be most easily achieved by fixing the filaments' orientation for as long as possible. Also it was believed that the motion of the guide would facilitate slippage if the winding speed was increased.

Design constraints for the FWA were: (1) the design had to be of as low a cost as possible, (2) the apparatus had to fit on a table top of approximately 91.4 cm (3') by 364.76 cm (12'), and (3) full winding of a ring preform should not take more than 8 hours. The first constraint was the most important and the use of existing equipment from previous NRC projects was promoted throughout the design.

The initial fibre orientation was 'rectangular', with one SCS-6 fibre surrounded by three Ti-6-4 alloy wires as illustrated schematically in Figure 4.1(a). This orientation was selected, despite the fact that it was less stable than a 'triangular' orientation as shown in Figure 4.1 (b), because it could be laid down continuously without stopping the

winding process. Also in this orientation the SCS-6 fibres could not come into contact with other fibres during consolidation even if considerable fibre swimming occurred.

4.2 Major Component Selection and Design

4.2.1 Mandrel

Three mandrels upon which the fibres would be wound were manufactured from austenitic stainless steel. The ideal mandrel material was Ti-6-4, the matrix material. The mandrel also acted as the vacuum can during the consolidation stages and had to be capable of withstanding the consolidation temperature of 800°C to 1000°C without melting. Stock Ti-6-4 of the necessary dimensions was not readily available. Stainless steel was the cheapest available alternative, hence stock of 10.16 cm (4") dia. was obtained and machined into the correct dimensions. A photograph of a completed mandrel is given in Figure 4.2. The outer diameter of the mandrels was initially 7.62 cm (3.00") with an inner diameter of 5.08 cm (2.00"). To conserve SCS-6 fibre the outer diameter of the mandrel was reduced to 2.5". The spool length of the mandrels was 1.91 cm (0.75"). The mandrel had two lips 0.635 cm (0.25") wide and a motor connection 0.95 cm (0.375") in diameter and 2.54 cm (1.0") long.

4.2.2 Rotational Motor

The pre-existing rotational motor obtained was a SLO-SYN synchronous stepping motor with controller and mechanical coupling. The operational requirements of the rotational motor was:

- a rotational speed of 33 rpm &
- an output torque of 0.54 N·m (76.8 oz.-in.)

A mount for the motor was machined from aluminum sheet such that it could be mounted on the Thompson Super Slide described in the next section.

4.2.3 Translational Slide

Several concepts were examined for the translational portion of the apparatus. The relevant parameters were flexibility in stroke length, lack of eccentricity and backlash in the stroke, velocity control and the capability to react and mount the rotational mount, motor and mandrel. The slide also had to be capable of withstanding the overturning torque caused by the rotational portion of the mandrel.

A Thompson Super Slide model 2AB-08-ARB-B-L18 was purchased to fulfill the translational stroke requirements. The linear roller screw slide has several advantages over cam linear travel systems in that there is no eccentricity in the stroke and that it can react heavy loads. The slide was deliberately purchased with a 22.9 cm (9") stroke thus allowing for the future manufacture of longer rings.

The translational side was mounted initially on a wooden block. The wood mount was chosen to reduce vibration from the rotational motor. The rotational motor, mount, coupling and the mandrel were then mounted to the slide. The lead of the ball screw of the translational slide is 0.5 cm (0.1969").

4.2.4 Translational Motor

The calculated stroke velocity of the mandrel for a tight wind was 0.016 cm/sec (0.00625 in/sec). In order to achieve this the roller screw on the Thompson slide had to be turned at a speed of 1.84 rpm. The motor also had to be able to reverse direction at the end of the stroke. To meet these particular requirements a pre-existing DC motor was obtained at NRC. The motor had a fixed rotational speed of 2 rpm. To calibrate it to the required speed the motor was hooked in series with a variac. By varying the resistance of the variac the speed of the DC motor could be controlled. The motor was also wired into a reversing circuit to achieve directional reversal at the end of the desired stroke. The stroke length was determined by placing micro-switches at the desired positions. As the mandrel hit the micro-switches it activated the circuit reversing the DC motor.

4.2.5 Back Tension Controller

An important variable that had to be determined was the tension on the filaments necessary to ensure that the filaments are laid out correctly. A pre-existing tension sensor and controller was obtained. The sensor used in the FWA was a MAGPOWR SSTS-F50 Tension Sensor capable of sensing 222.4 N (50 lb) of tension connected in a circuit with a brake capable of applying 36.5 N·m (25 ft·lb) of torque to produce the correct line tension.

4.2.6 Feed System

The feed system consists of the spools of titanium wire and SCS-6 fibre necessary for the commingling of the filaments and a system to mount these spools. Several methods to mount the spools were considered, however most involved significant design, procurement and manufacture time. The final design consisted of a steel plate with four steel rods for mounting the spools welded to it. The major advantage of this concept was simplicity and ease of manufacture. Possible concerns were that binding of the spools on the mounting rods would create high tensile forces on the filaments that would cause fibre or wire breakage during winding. It was found during testing that placing Teflon discs under the spools prevented binding from occurring. This resulted in a significant savings in time and resources.

4.2.7 Guides

The problem of guiding the individual filaments was a major technical challenge. Initially it was thought that a series of steel spring loaded rollers could be used to control the fibre alignment. However it proved too difficult to align them in a fashion that would produce a repeatable filament orientation.

Another method considered for controlling the fibres was to drill holes in synthetic rubies. This option was quickly abandoned due to the cost of the raw material and the difficulty of machining guide holes of a small enough diameter to guide the fibre.

Similarly plans to machine the guides out of metal were abandoned due to concerns about machinability and possible damage to the fibres during the winding process.

Finally it was decided to machine the guides out of a softer material. TeflonTM was selected as the guide material due to its low cost and its low frictional coefficient. It was hoped that this would allow for experimentation with different guide hole configurations and methods of drilling the guide holes. It was also thought that the ductile nature of the guide material would prevent fibre damage during the winding procedure.

Four 152 μm (0.006") diameter holes were initially machined into 0.635 cm (0.25") thick TeflonTM sheet by laser drilling. However, the holes produced by this method were irregular and it was difficult to thread the filament through the guides. Also, during initial testing it was found that the guides wore out quickly due to excessive friction. New guides were fashioned from 0.318 cm (0.125") thick TeflonTM by conventional drilling using a very fine dimension drill bit. The holes produced by this method were very well defined and the wear rate of the guides during testing was reduced significantly.

The guides were mounted in a vise that could be rotated to ensure proper alignment from the tension meter/ controller to the mandrel and thus reduce wear of the Teflon guides.

Side and end views of the full apparatus are given in Figure 4.3.

4.3 Winding Trials

4.3.1 Calibration & Set-Up

Calibration of MAGPOWR back tension sensor was performed by feeding a fibreglass tow through the system. The bundle was then loaded incrementally with a set of dead weights. The tension gauge was then adjusted to read a filament tension on a scale 0 to 44.48 N (10 lb) by adjusting the gain and zero of the meter.

The translational and rotational motors were also calibrated. The calibration was first attempted by using automated equipment for the purpose. A photo-tachometer and an optical tachometer were employed to fix the motor speeds; however, the desired set speeds for the motors were below the calibration limit of 50 rpm for the tachometers. Eventually the motors were calibrated using optical methods.

To attach the filaments to the mandrel an ethyl cyanocrylate (Loctite 495) binder was used. This binder was selected for several reasons. It has a thin glue line which is very useful for binding the fine filaments. It binds both metal and ceramics and is thus capable of fixing both the SCS-6 fibre and the Ti-6-4 wires. It sets very quickly and volatilizes completely at 250°C to 350°C and hence is easily removed before HIP consolidation.

4.3.2 Procedure

The procedure developed for the filament winding process is as follows. The filaments were wrapped around the brake and run through the tension controller. They were threaded through the Teflon guide and attached to the stainless steel mandrel with the Loctite 495 binder. The micro-switches were adjusted so that they were precisely the length of the mandrel plus the required stroke apart. The MAGPOWR tension controller was then turned on and the translational and rotational motors were then turned on simultaneously. After the wind was completed, or when the filament orientation began to degrade, the motors were shut down.

4.3.3 Modifications to Apparatus

After the first winding trials several minor modifications were made. The wooden base on which the translational slide was initially mounted was replaced with steel. This was to prevent the slide from being displaced by the winding torque. To damp the motor vibrations insulating material was placed under the steel plate. Insulating material was also placed under the rotational motor and its mount to the translational slide.

The initial laser drilled Teflon guides were replaced with conventionally drilled guides. The initial guides had very poorly drilled and spaced holes. The new guides had far better tolerances. This allowed superior filament placement.

The MAGPOWR brake could not accurately control the tension on the filaments. This was due to the fact that the brake was devised for fibre tows and not for single filaments. To ensure that enough tension was placed on the filaments 0.91 kg (2 lb) brass weights were machined and placed on the feed spools. The resulting frictional force was sufficient to create a tensile force of ~ 3.34 to 4.45 N (0.75 to 1 lbf) on each filament which was sufficient back tension for the winding.

The Teflon guides were also moved 5.08 cm (2 in) closer to the mandrel. This placed them within 1.27 cm (0.5") of the mandrel.

4.3.4 Ring Winding Results and Discussion

In the winding trials the sensitivity of the winding process to various parameters was determined. These parameters included:

1. the filaments must experience enough back tension to lay correctly,
2. the guides must be very close to the mandrel to ensure proper orientation,
3. vibration and sudden jarring of the motors must be minimised, and
4. translational and rotational motor speeds must be precise.

Despite considerable modification and improvement of the apparatus a perfect wind pattern was never achieved during the trials. The main reason for this was that the motor speed control was inadequate and vibration from the rotational motor was excessive.

These factors would result in an accumulated error in the wind path until the filament orientation was disturbed sufficiently enough to produce an irregular fibre layout. Eventually it was decided that the rectangular orientation was untenable and a triangular pattern was adopted as illustrated in Figure 4.1 (b). To achieve this, a discontinuous winding strategy had to be adopted. One layer consisted of a SCS-6 fibre spaced by a Ti-6-4 wire and the following layer consisted of two Ti-6-4 wires. Utilising this strategy several layers were built up on the mandrel with good distribution until eventually error in the wind spacing created irregularities in the fibre distribution. The wind was then finished by the continuous winding of Ti-6-4 wire until a thickness of 0.25" of wire and fibre had been achieved as is illustrated in Figure 4.4.

Chapter 5.0: Post Wind Experimental and Analytical Procedure

Upon successful winding of the TMC preform a series of consolidation and post-consolidation experimental steps were performed. The procedures developed in performing this work are presented in this chapter. The results of this work are detailed in chapter 6 and the relevant discussion follows in chapter 7.

5.1.0 Vacuum Canning of TMC Preform

As discussed in chapter 4, section 3, upon completion of winding the fibre layers, several layers consisting of solely Ti-6-4 wire were wound on the mandrel. This was done to provide a control region of Ti-6-4 so that the influence of the fibre on the microstructure of the composite could be assessed. The wire was then spot welded to a lip in the mandrel so that the wire would remain tight during the canning operation.

A stainless steel sleeve of the same alloy as the mandrel was machined out of bar stock. This sleeve formed the outer wall of the vacuum can and had an inner diameter of 6.36 cm (2.505") with a wall thickness of 0.25 cm (0.100"). A 1.27 cm (0.50") diameter hole was drilled into this sleeve to provide an interface for the attachment of an evacuation tube. A steel pipe, ~45.7 cm (18") long with an outer diameter of 1.59 cm (0.625") was used for the evacuation tube. The end of the tube was machined to fit in the hole of the stainless steel sleeve. The assembly of the stainless steel tube and sleeve is depicted in Figure 5.1. The sleeve was fitted over the wound mandrel and welded in place. The evacuation tube was then welded to the sleeve. A photograph of

the welded assembly is given in Figure 5.2. To ensure that consolidation would occur in all directions a 4.57 cm (1.8") diameter hole was machined into the center of the can as shown in Figure 5.3. This gave an inner and outer radial wall thickness of 0.25 cm (0.100") and a 0.63 cm (0.250") wall thickness at the top and bottom of the can.

The can was then off gassed to remove the ethyl cyanocrylate binder and other organic contaminants. This was achieved by heating the mandrel under vacuum in a series of steps:

1. 4 hours at room temperature (vacuum pressure of 1.8×10^{-5} mbar was achieved),
2. 1.25 hours at a temperature of 200 °C (vacuum pressure of 1.2×10^{-5} mbar was achieved),
3. 20 hours at a temperature of 400 °C (vacuum pressure of 1.2×10^{-5} mbar was achieved),
4. 24 hours at a temperature of 600 °C (vacuum pressure of 1.0×10^{-5} mbar was achieved), and
5. 2.5 hours at a temperature of 850 °C (vacuum pressure of 1.2×10^{-5} mbar was achieved).

The final burn off cycle temperature of 850 °C was performed as a precaution to disassociate and remove any possible Teflon wear particles picked up from the filament guides.

After removing the organic contaminants the vacuum can was temporarily sealed and placed within a vacuum chamber. The evacuation tube was filled with 60 μm diameter Ti-6-4 powder and a fibreglass filter as a precaution to prevent the collapse of the weld at the tube/ sleeve interface. The vacuum can was subjected to another 48 hour burn-off at 400 °C to remove oxide contaminants from the powder. Finally, the vacuum can was sealed for HIP'ing.

5.1.1 HIP Envelope

The HIP temperature, pressure and consolidation time were selected by adapting the consolidation times and pressures presented in references 93-96. The consolidation parameters were as follows:

1. consolidation temperature of 875 °C,
2. consolidation pressure of 100 MPa, and
3. consolidation time of 90 minutes.

The consolidation time of 90 minutes is 150% of the time estimated to ensure full consolidation. It was felt that consolidation at this time, pressure and temperature would not degrade the composite through extensive fibre matrix reactions. The full HIP cycle is given in Figure 5.4.

5.1.2 Preparation of Metallographic Samples

After confirmation of full consolidation of the TMC ring, several radial sections were cut out of the ring using a low speed diamond saw. These specimens were mounted in bakelite and polished for optical microscopy. The polishing procedure for the TMC specimens is as follows:

1. the specimens were mounted in the automatic polisher with 4 lb. of pressure per specimen,
2. the specimens were ground using 180 grit SiC paper until the specimens were flat and all surface scratches were removed,
3. successive grades of sand paper 240, 320, 600 were used to reduce scratches, and
4. final polishing was performed with 9 μm , 6 μm and 1 μm diamond paste polishing wheels.

The samples were etched in Kroll's solution for 17 seconds to reveal the microstructure of the matrix.

5.2.0 Secondary Heat Treatment Regimen

To assess the effect of the introduction of fibres into the matrix alloy a series of secondary heat treatments were performed. The secondary heat treatments selected were standard Ti-6-4 heat treatments discussed in chapter 2, section 1.1. The heat treatments performed were:

- duplex annealing,
- recrystallisation annealing,
- solution treating and aging, and
- β - annealing.

Details of the heat treatment regimens are given in Table 5.1. The first three heat treatments were performed below the β transformation temperature and the fourth above it. The goal of the heat treatments, discussed in greater detail in chapter 7, is to increase ductility and toughness of the matrix, however, a solution treat and age was included in the program for comparison purposes. This was to produce a microstructure of the TMC ring where the α and β phases at temperature could be clearly distinguished, with the β regions distinguishable from lamellar α grains. The solution treatment temperature for Ti-6-4 is the same as that of the duplex and recrystallisation annealing.

5.2.2 Heat Treatment Sample Preparation

Four samples, each with an arc length of ~ 0.63 cm (0.25"), were cut from the TMC ring utilising a high-speed diamond saw. The specimens were then prepared for heat treatment. Table 5.1 summarizes the heat treatment and encapsulation information for each sample. One sample was quartz encapsulated. The quartz tube was evacuated of air to a vacuum of 2.5×10^{-3} torr and back filled with argon gas pressure of 200 torr to prevent sample oxidation. However, this procedure requires considerable time and expense. Therefore, a novel method of encapsulating heat treatment specimens was

used for two of the other samples. In this method a heated ceramic urn is coated with Corning glass powder and water paste. As the water evaporates the glass is precipitated out as a film in the urn. This glass film is built up until a glass base has been formed. The sample is then placed on this base and more paste is applied until the sample has been completely embedded in the glass film. The sample is then left to dry out for at least 24 hr. The glass film impedes oxidation attack during heat treatment and is effective for the relatively short treatment times and temperatures of interest [112].

Finally, the last sample (the recrystallisation annealed sample) was heat treated in a vacuum furnace. This was done as this heat treatment requires a long time at the recrystallisation temperature and it has been previously determined that the Corning powder encapsulation method does not effectively protect the sample from oxidation when exposed to atmosphere for long exposure times [112]. Additionally recrystallisation annealing requires a fairly complex cooling envelope which could be more easily performed in the vacuum furnace.

5.3 EDAX Analysis

To determine whether microstructural differences between the heat treated TMC samples and the standard Ti-6-4 microstructures, discussed in detail in the following chapter, were caused by compositional variances a series of energy dispersive analysis of x-rays (EDAX) analyses were performed within a scanning electron microscope (SEM). Two forms of EDAX spot analyses were performed, a qualitative *thin window*

analysis to determine the relative carbon concentration as a function of distance from the fibre, and a quantitative *beryllium window* analysis to determine if variation of alloying element concentrations occur with distance from the fibre.

The samples were polished following the procedure outlined in section 1.2 of this chapter and, unless otherwise noted, were not etched to improve the accuracy of the spot EDAX analysis. Spot analysis was performed, instead of a line scan analysis, because of its greater accuracy. The SEM utilised also had an automated feature allowing for a line of spots to be analyzed in one step.

The SEM parameters were:

- an excitation voltage of 20 kV, and
- a 100 sec dwell time at each spot.

5.4 Fibre Push Out Test Set Up and Procedure

Fibre push out (FPO) tests were performed to determine the interfacial shear strength of the TMC ring. This was performed with a nanomechanical probe (NMP). The NMP is a high resolution depth indentation instrument and is capable of generating the load vs. displacement plots discussed in chapter 2, section 6 [113].

FPO thin slice samples were cut from the as HIP'ed and heat treated specimens using a low speed diamond saw. The thickness of these slices was 0.4-0.6 mm. The sample thickness was carefully reduced to 0.20 mm without warping the specimens.

The FPO tests were performed following the procedure outlined in Appendix A. The indenter used during the trials was a conical diamond indenter with an included angle of 70° and a flat bottom of $100\text{ }\mu\text{m}$. The upwards stage velocity was a constant $1\text{ }\mu\text{m/sec}$ throughout the stroke. A schematic diagram of the apparatus set up is given in Figure 5.5. To minimise bending during the FPO test the sample was clamped between two stainless steel plates with a 3.18 mm hole through which the tests were conducted.

6.1 Results of HIP'ing

A photograph of the post-HIP'ed vacuum can is given in Figure 6.1. It can be seen that the can has deformed, thus indicating that consolidation has occurred. After HIP'ing the vacuum can was machined away to reveal the TMC ring, depicted in Figure 6.2, with an inner diameter of 4.95 cm (1.95"), an outer diameter of 6.48 cm (2.55") and a thickness of 2.12 cm (0.835"). These dimensions include a steel wall thickness of ~ 0.64 mm (0.025") on all sides. To determine if the composite was fully consolidated the ring was cut longitudinally by electron discharge machining. The resulting section, illustrated in Figure 6.3, confirms that full consolidation has occurred and that the fibres have survived the HIP process.

6.1.1 HIP'ed Microstructure

A photograph of the polished cross section is given in Figure 6.4. The TMC ring is fully consolidated. The fibre consists of three layers with an even distribution and no fibre-fibre contact. After this initial area the fibre distribution deteriorates significantly with voids and fibre-fibre contact. Examples of voids from poor fibre spacing and fibre-fibre contact are given in Figure 6.5.

The observed microstructure, shown in Figure 6.6 a of the Ti-6-4 alloy matrix is fine grained, equiaxed, α with a grain size 4-6 μm in a matrix of β . The observed

microstructure of the alloy does not change with distance from the fibre, however, there is a region of discontinuous microstructure near the stainless steel sleeve area of the Ti-6-4 control region shown in Figure 6.6 b. This is most likely due to iron contamination.

6.2 Results of Secondary Heat Treatments

6.2.1 Duplex Annealed Sample

The duplex anneal failed to produce the desired microstructure when performed on the TMC sample. The observed microstructure remained similar to that of the as fabricated sample, shown in Figure 6.6, as evidenced in the micrograph of Figure 6.7.

6.2.2 Recrystallisation Annealed Sample

The microstructure of the TMC after recrystallisation annealing is shown in Figure 6.8 a. The desired recrystallised microstructure of large equiaxed α grains with β forming at the triple grain boundaries has been achieved with an approximate α grain diameter of 10-12 μm . There are no microstructural changes or discontinuities with distance from the fibre surface as is shown in Figure 6.8 b.

6.2.3 Solution Treated and Aged Sample

Micrographs of the STA sample are presented in Figure 6.9 a and b. The microstructure is fully equiaxed with light regions consisting of α grains and darker regions consisting

of the martensitic α' phase. The microstructure was not observed to change with radial distance from the fibre.

6.2.4 β Annealed Sample

The final heat treatment, the β anneal, was performed at 1035 °C, well above the β transus temperature of the matrix. The microstructure of this heat treatment is presented in Figure 6.10. The microstructure of the matrix in the region remote from the fibre corresponds to the expected in β annealed Ti-6-4 with lamellar α platelets in a matrix of β as shown in Figure 6.10 a. However, the Ti-6-4 matrix in a 50 μm radius around the SCS-6 fibre failed to transform and retained its original microstructure of fine equiaxed α grains in a matrix of β , shown in Figure 6.10 b. The matrix transforms to the β annealed microstructure at a distance of approximately 50 μm . This leads to a mixed matrix microstructure with regions of equiaxed, fine grains immediately about the SCS-6 fibres and lamellar α between the fibres.

6.3 Results of the EDAX Investigation of the Microstructures

6.3.1 Results of Thin Window Analysis

The β annealed sample was placed in the SEM and backscatter electron images were taken at several areas around the fibre. The backscatter images show changes in phase due to the compositional differences between the α and the β phase. Figure 6.11 shows

an example of one of the backscatter electron images. Thin window spot analyses were performed at distances of 1 μm , 3 μm , 10 μm , and 100 μm from the fibre in both α and β grains. The generated spectra are presented in Figure 6.12. The carbon concentration becomes negligible after 3 μm indicating that the carbon does not diffuse away from the fibre into the matrix.

6.3.2 Results of the Beryllium Window Analysis

Quantitative analysis was performed on the as HIP'ed, β annealed, and the STA samples. Using backscatter images to differentiate the phases, spot analyses were performed on α and β grains 10 μm and 100 μm away from the fibre for the β annealed and as HIP'ed samples. The compositions are presented in Table 6.1. There are no significant differences in the composition of the α and β phases in the two samples.

To determine if compositional variances occur with radial distance away from the fibre, a line of spot analyses were performed for the STA and β annealed samples. The β annealed sample was lightly etched in Kroll's solution for 10 seconds to reveal the change in microstructure. Figure 6.13 illustrates the set up of the analysis points. The points were spaced ~ 4 to 5 μm apart. This corresponds to the area illuminated by the electron beam, such that a continuous analysis of the matrix along the line was performed. The variance of the various alloy elements is given in Figure 6.14 and Figure 6.15 for the β annealed and STA samples respectively. The STA sample has a slightly sinusoidal shape depending on whether the spot analysis is of α or α' grains.

The β annealed shape is discontinuous, this is apparently a function of whether the spot analysis is of the α or β phase.

6.4 Reaction Layer Thickness

After completing the optical microscopy SEM images were taken of the various samples. This was done to assess the degree of interfacial reaction during the heat treatment process. The measured reaction layer thickness' are presented in Table 6.2. These experimental values were obtained from measuring the reaction layers at several points on SEM secondary electron images, presented in Figure 6.16. Theoretical values for the reaction thickness were calculated based on equations 2.2.2 and 2.2.3 and the values from Table 2.7, and are presented with the experimental data in Table 6.2.

6.5 Results of the Fibre Push Out Testing

The post HIP'ed specimens were successfully pushed out using the nanomechanical probe. Eleven fibres were pushed out and the critical loads for fibre debonding are presented in Table 6.3. The force vs. probe displacement curve for the post HIP'ed specimen generated by the nanomechanical probe is given in Figure 6.17. The average critical load, P_{cr} , taken at the peak of Figure 6.17, necessary for fibre push out is 4.498 ± 1.0 N. Utilising this data and equation 2.6.1, it was determined that this load corresponds to an interfacial debond shear strength of 50.41 Mpa assuming an $\xi=1$. The

frictional shear strength of the composite could not be determined as the load began to increase immediately after debond occurred, indicating that the indenter had begun to impinge on the surrounding matrix material.

SEM micrographs were taken of the pushed out fibres from the push out side presented in Figure 6.18 and from the indenter side, given in Figure 6.19. From the micrograph of the pushed out side it can be seen that the fibre is intact, indicating that the fibre debonded before it reached its failure stress.

The heat treated samples could not be pushed out with the NMP. A typical force displacement curve for a heat treated specimen is given in Figure 6.20. This force was generated over a 110 μm stroke which indicates that the sample had deflected this distance without failure. A longer stroke would have produced a greater load, but the resultant sample bending would have made interpreting the data difficult. A stiffer load cell could have produced the necessary force with a lesser stroke, however, there was no appropriate load cell available during the testing period.

7.1 Discussion of HIP'ed TMC Ring

As stated in chapter 6, section 2 the fibre alignment of the HIP'ed ring is regular for the first three layers and then deteriorates as the winding progresses. This is most likely due to the poor wind pattern produced by the filament wind apparatus. The voids and fibre contact present in the composite are due to the poor wind pattern. Fibre swimming does not appear to be an important contributing factor to the composite defects as the regular orientation in the first three layers (see Figure 6.4) does not experience significant fibre movement.

Another interesting feature of the composite is the rectangular nature of the regular layers. The expected pattern should be approximately square, indicating that the alloy flowed away from the fibres radially which, in turn, spread the fibres further apart in the horizontal direction. One explanation for this phenomenon is the variation in the can thickness' in the radial and the longitudinal direction. The thickness of the can radially was 0.25 cm (0.100"), while the length of the can was 3.175 cm (1.25"). Thus the radial stiffness of the can was significantly less than the longitudinal stiffness. The deformed vacuum can, shown in Figure 6.1, has a bowed shape suggesting that the metal flow at the center of the can, where the composite had more void volume, was greater than at the solid ends. The ratio of the horizontal fibre distance to radial fibre distance to the ratio of can thickness' reveals that the former is 1.7 and the later is 9.6.

7.2 Discussion of the Secondary Heat Treatment Regimen

7.2.1 Aim of Secondary Heat Treatment Regimen

The matrix microstructure of the as HIP'ed TMC ring has several advantages. Fine, equiaxed, α grain Ti-6-4 microstructures are associated with excellent strength and ductility. However, in TMC components the strength along the fibre direction is dominated by the SCS-6 fibres. Thus microstructural optimisation can be performed to maximise the matrix dominated properties of the composite at the cost of matrix properties that are dependent on the fibre in the TMC.

Low cycle fatigue (LCF) strength is a crack initiation dominated phenomenon. As discussed in chapter 2, section 2, in TMC components crack initiation typically occurs at the fibre/matrix interface layer. Hence the LCF life of the TMC is not likely to be strongly influenced by the microstructure of the Ti-6-4 matrix. Damage tolerance, however, is controlled by crack propagation from the initiation sites through the matrix material and is thus controlled by matrix microstructural features [93]. Similarly, the ductility of the composite is dependent on the matrix ductility. Hence, the purpose of the heat treatments undertaken in this project is to attempt to produce a microstructure which should increase matrix ductility, fracture toughness and creep resistance without reducing fibre integrity significantly though interfacial attack. Typically, either a coarse grain, equiaxed microstructure or a randomly aligned lamellar microstructure would meet most of the requirements.

7.2.2 Duplex Anneal

The duplex annealed failed to produce the desired mixed microstructure. This is possibly due to the small grain size of the matrix before it underwent duplex annealing. The 4-6 μm diameter of the grains may have impeded formation of the coarse acicular α that may have formed within larger equiaxed grains.

7.2.3 Recrystallisation Anneal

The resultant microstructure is finer than that desired, however, this varies as a function of the initial grain size. Still, the microstructure closely resembles what is typically obtained [23] for this type of heat treatment. The time that this sample was subjected to the recrystallisation temperature was 4 hr. This can be increased, thus allowing the matrix to recrystallise further and thus increase the overall grain size. The practical limit of recrystallisation annealing for TMC components is the interfacial zone thickness that would occur during the treatment. After a certain time the ductility and toughness benefits of recrystallisation would be offset by the degradation of fibre properties.

7.2.4 Solution Treat and Age

The solution treat and age (STA) was performed to provide an insight into the effect of the heat treatment temperature on the microstructure of the TMC. The micrograph of the STA, shown in Figure 6.9 a and b, illustrates the microstructural differences imposed by the small grain size of the as HIP'ed TMC ring. As previously stated, the

microstructure consists of equiaxed α and α' grains. This microstructure confirms that the matrix is precipitating out the β phase at the solution temperature. However there are no acicular regions visible, even in SEM micrographs (see Figure 6.5 c), hence suggesting that the failure of the duplex anneal is not due to compositional variances, but is rather related to the grain size of the alloy.

7.2.5 β Anneal

The β annealed sample produced an unexpected duplex microstructure that varied with the radial distance of the matrix from the fibres (refer to Figure 6.10). There are several possible reasons for this mixed microstructure:

1. The fibre coating is contaminating the matrix with carbon. Since carbon is an interstitial contaminant it would increase the β transus temperature directly around the fibre which could affect the matrix behaviour at the heat treatment temperature.
2. The alloying elements of Ti-6-4 are either segregating to the interface or are being rejected by the interfacial zone. In chapter 2.2, it was previously reported that aluminum is rejected by the interface. This opens the possibility that the matrix directly around the fibre is aluminum rich. As aluminum is an α stabilizer this would raise the β transus temperature and inhibit the formation of the β annealed microstructure.
3. The irregular interfacial layer may impede the formation of lamellar grains. This could be possibly caused by nucleation sites at the interface altering the energy state of the matrix and favouring the formation of equiaxed grains.

However, as stated in chapter 6.2.4, the equiaxed area of the matrix remains stable for a region 50 μm from the fibre suggesting that this mechanism is not responsible for the duplex microstructure.

4. Another strong possibility is that the residual stresses caused during the cooling of the TMC sample from the heat treatment temperature created a stress field around the fibres which favoured the formation of equiaxed grains to reduce the stress state of the matrix. The coefficients of thermal expansion of the matrix and the fibre are an order of magnitude different. Upon air cooling from the β annealing temperature the differences in thermal contractions of the matrix and the fibre would have produced considerable residual tensile thermal stresses in the matrix surrounding the fibres. When the secondary stabilizing heat treatment was performed the residual stresses may have caused a microstructural change. The effects of heat treating residually stressed $\alpha + \beta$ titanium alloys in altering α grain morphology was discussed in chapter 2, section 1.1.5.

Further work, discussed in section 7.3, was performed to determine which of the above factors controlled this microstructural change.

7.3 Discussion of the EDAX Investigation of the TMC Microstructures

The goal of the EDAX analysis was to determine if either the carbon contamination or variation in alloying elements of the matrix caused the duplex microstructure presented in section 2.5 of this chapter. The thin window spectra presented in Figure 6.12 show the change in carbon concentration with distance from the fibre. Qualitatively it can be inferred from the height of the carbon peaks that the carbon concentration is fairly high in the interface and in the matrix at a distance of 1 μm from the fibre (Figure 6.12a and b). After that the concentration drops to background levels. From this work it can be

concluded that no significant carbon contamination from the fibre into the matrix occurred.

The beryllium window results are less definitive. The alloying elements in Figures 6.14 and 6.15 vary considerably from one point to another. However, when the concentrations of the alloying elements are compared to the values for the different phases developed in Table 6.1 it becomes evident that the changes in concentration compare well with the differences in concentration for the α and β phases for the β annealed sample. There is some variation in the V concentration with radial distance in Figure 6.14, specifically an anomalous peak is observed at a radial distance of approximately 5 μm away from the fibre periphery, but this change is not in the region of the microstructural change. There is also a slight change in V concentration of the α phase in the region of the microstructural change with the mean concentration increasing slightly. However, the change is extremely small and it is within the standard deviation of analysis. It also must be remembered that this is a spot analysis. In the fine lamellar α region the probability of the spot area containing a portion of a β grain is much higher and would raise the V content slightly. The almost identical V concentration in the β phase supports this hypothesis. In any event the minimal changes in concentration would not cause the drastic change in microstructure observed.

In the STA graphs of Figure 6.15 the concentration differences are less extreme. This is due to the low amount of β present in this sample. Slight increases in V concentration denote α' grains with the lower concentrations indicating the α phase. In the three trials

for the STA sample there are points of high V content, but, the location of the anomalies with radial distance from the fibre are fairly random and are thus likely areas where β grain formation is commencing. Hence, from this work it can be concluded that the change in microstructure is not likely to be due to compositional changes of the Ti-6-4 alloy.

7.4 Interfacial Reactions

A comparison of the reaction layer thickness given in Table 6.2 shows that the experimental reaction layer was typically less than the theoretical thickness calculated. This could be due to continued improvement of the protective coating which would change the K_0 and Q for the composite system. Importantly, the amorphous carbon rich coating was not completely consumed in any one of the heat treated conditions. Hence the fibre integrity should not be degraded. This suggests that the strength of the TMC ring should not be significantly compromised by any of the standard heat treatment routes studied in this thesis.

7.4 Discussion of Fibre Push Out Testing

The mean interfacial shear strength calculated for the post HIP'ed specimen was 50.41 MPa. This is significantly lower than the values obtained from literature [53, 109]. It has been noted, however, that FPO slices of 0.200 mm or less may not produce accurate results [109-110] due to surface effects. Thicker specimens were not pushed out because the load carrying capacity of the NMP is insufficient to push out fibres from

thicker samples. The frictional shear strength could not be obtained as the force data continued to rise after the fibre debonded. This occurred as a result of the impingement of the indenter into the matrix after the fibre debonded. This is due to the conical geometry of the diamond indenter.

Figure 6.18 shows the pushed out fibres from the pushed out side. The failure is smooth, suggesting that the fibres failed in the carbon coating. Figure 6.19 shows the pushed out fibres from the indenter side of the sample. The matrix around the fibre has been damaged confirming that the indenter has impinged on the matrix material.

The heat treated samples could not be pushed out with the available load cell. This implies that the interfacial debond strength of the heat treated samples was considerably higher than that of the as HIP'ed sample. This is most likely due to the high residual stresses induced by the rapid cooling of the heat treated samples. The coefficients of thermal expansion of the fibre and the matrix are approximately an order of magnitude different. All of the secondary heat treatments subjected the TMC to a final air cool, a relatively fast cooling procedure, where a rapid change in temperature of at least 500 °C occurs. This would set up considerable residual thermal clamping stresses as the matrix would shrink to a greater extent than the fibre. The as HIP'ed sample was cooled at a much slower rate under hydrostatic pressure. As stated in chapter 2, section 4.2.1, this process has been found to reduce the residual thermal stresses in TMC composites. Thus it is likely that the heat treated samples have a greater clamping stress on the fibre than the post HIP'ed sample. This is an important point as it suggests that the stresses

induced during cooling of the samples are a significant factor in the properties of the TMC. The stress field induced by the rapid cooling would hence be great enough to induce microstructural transformations in the matrix in the region of the fibre. The effect is similar to the thermomechanical treatments discussed in section 2.1.1.5 with the duplex structure caused by the residual stresses. This effect is the most probable reason for the altered β anneal microstructure in the fibre region.

The composite strength for the post HIP'ed sample was calculated. To utilise the formulae presented in chapter 2.6 several simplifying assumptions were made. The assumptions were that the TMC had a regular orientation 8 fibres wide and 3 fibres thick with a volume fraction of 27%. For calculation purposes the fibres were also assumed to be discrete and comprise one complete revolution of the ring. A schematic of the assumptions is presented in Figure 7.1. The fibre length was assumed to be the longest circumferential length as this is the most conservative assumption. The Weibull shape factor, m , was averaged from data presented in literature [104] to be 9.9 for a gauge length of 2.54 cm. The fibre strength for this gauge length was assumed to be 85% of the value presented in Table 2.6. This value accounts for fibre damage during the winding process and from interfacial attack during consolidation. The composite strengths are given in Table 7.1. The actual composite strength should fall between the Local and Global load sharing models. The low interfacial shear strength indicates that the Global load sharing model strength of 1322.3 MPa is probably closer to the actual strength.

This thesis research demonstrates that filament winding followed by HIP'ing is a viable method for producing axisymmetric TMC shapes. A Ti-6Al-4V matrix/ SiC fibre ring was produced through the winding of matrix wires and reinforcement fibres onto a mandrel. However, the composite fibre distribution was poor as the filament orientation could not be controlled with the laboratory filament wind apparatus. Since a regular distribution was achieved for the first three layers of the composite this could be overcome by more accurate equipment. However, the benefits of this method compared to the wire wrap method developed by Atlantic-IMT are debatable. The Atlantic IMT wrapping method cannot be easily adapted to wide cylindrical shapes without complicating the fibre/ wire feed creel, however, it circumvents the difficulties in producing a regular wind orientation that were encountered with the FWA. Finally, the maximum volume fraction obtainable by this method is 27%, which is considerably less than the MCF and thermal spray techniques. While filament winding is a technically feasible fabrication technique, it is not a suggested method for future development.

The mandrel was vacuum canned and hot isostatically pressed for 90 minutes at 875 °C. The TMC was fully consolidated and the fibres survived the process indicating that the consolidation of matrix wire with reinforcing fibre does not differ significantly from the parameters developed for the foil fibre foil fabrication technique.

The TMC was subjected to standard Ti-6-4 secondary heat treatments to optimise matrix dominated properties. It is concluded from the results of these heat treatments that standard heat treatments for monolithic Ti-6-4 cannot be used to alter TMC matrix properties. However, it is also concluded that the matrix microstructure can be altered by heat treatment to produce damage tolerant microstructures. Therefore, the heat treatments will have to be modified from those usually applied to Ti-6-4.

One of the standard heat treatments, the β anneal, revealed an unexpected microstructural transformation with radial distance from the fibre. EDAX analysis was performed to determine whether this was due to a variation in the chemical composition of the matrix alloy. The result of this analysis was negative indicating, using a process of elimination, that the microstructural transformation occurs due to thermomechanical mechanisms.

Interfacial shear strength data was developed by performing fibre push out tests using a nanomechanical probe. Critical loads for the as HIP'ed composite were determined experimentally, however the heat treated samples could not be pushed out with the available load cell. One possible reason is that the compressive residual thermal stresses induced through the air cooling of the samples increased the interfacial debond stress. The as HIP'ed sample had lower thermal residual stresses due to its slow cooling cycle which known to reduce the thermal residual stress. This indicates that the thermal residual stresses strongly influence TMC strength and failure characteristics as they affect matrix grain morphology and the interfacial debond strength.

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TABLES

Table 2.1: Selected properties of pure titanium. [17].

Property	
Atomic Number	22
Atomic Weight	47.88
Density : α β	4.57 g/cm ³ @ 20 °C 4.35 g/cm ³ @ 885 °C
Lattice Parameters (α) (β)	c = 0.468 nm a = 0.295 nm a=0.332 nm @ 900 °C
Liquidus Temperature	1670 $\pm$ 5 °C
Boiling Point	~3260 °C
β Transus Temperature	882 $\pm$ 2 °C
Coefficient of Thermal Expansion	8.41 x 10 ⁻⁶ / °C
Young's Modulus	100 - 110 GPa
Poisson's Ratio	0.32 - 0.36
Ultimate Tensile Strength	235 MPa
Tensile Yield Strength	140 MPa

Table 2.2: Chemical requirements for Ti-6-4 (Grade 5) alloy wire [32].

Element	Composition (%wt.)
Nitrogen, max	0.05
Carbon, max	0.10
Hydrogen, max	0.015
Iron, max	0.40
Aluminum	5.5 to 6.75
Vanadium	3.5 to 4.5
Tin	----
Palladium	----
Molybdenum	----
Nickel	----
Residuals, max. each	0.1
Residuals, max. total	0.4
Titanium	remainder

Table 2.3: Standard Heat Treatments for Ti-6-4 [17].

Heat treatment	Procedure	Comments
Stress relief annealing	2 to 4 h 595 °C (1100 °F), air cool	Relieves residual stresses from welding, forming, etc. This cycle only provides a partial stress relief. A full anneal must be used for full stress relief. Low strength, good ductility
Full annealing (mill annealing)	2 h, 735 ± 15 °C (1350 ± 25 °F), air cool	Most common heat treatment, has good overall properties combinations. Low strength, good ductility
Annealing for continuously rolled sheet	5 min 870 °C (1600 °F), rapid furnace cooled, plus 5 min 595 °C (1100 °F), air cool	Approximately the same strength and ductility as above, but with improved fracture toughness
Recrystallization annealing	4 or more h, 925 °C (1700 °F), furnace cool to 760 °C (1400 °F) at 55 °C (100 °F)/h (or slower), cool to 480 °C (900 °F) at 370 °C (670 °F)/h (or faster), air cool	Usually used for ELI material. Strength comparable to above conditions, but improved damage tolerance (fracture toughness, stress-corrosion resistance, reduced crack growth rates). Strength is lower for ELI material
Duplex annealing	10 min, 940 °C (1725 °F), air cool, plus 4 h 675 °C (1250 °F), air cool	Improved damage tolerance
Beta annealing	30 min, 1035 °C (1900 °F), air cool, plus 2 h, 730 °C (1350 °F), air cool	Used to maximize damage tolerance properties. These properties are attained with a slight loss of ductility and a significant fatigue debit. A preliminary treatment followed by other treatments such as annealing
Beta STA	30 min, 1035 °C (1900 °F), water quench plus 4 h, 510 to 675 °C (950 to 1250 °F)	...
Beta STOA	30 min, 1035 °C (1900 °F), water quench plus 2 to 4 h, 675 to 730 °C (1250 to 1350 °F)	Similar to beta anneal with improvement in fatigue performance may be preferable to beta anneal because of improved fatigue properties, but at a cost in damage tolerant properties
Solution heat treatment	10 min, 940 °C (1725 °F), water quench	Used as an intermediate step for forming material ultimately to be used in the STA condition. Not to be used as a final condition due to instability
Solution heat treatment and aging	10 min, 940 °C (1725 °F), water quench, plus 4 h, 510 to 540 °C (950 to 1000 °F), air cool	Highest strength condition, but less ductility, stress-corrosion resistance, and fracture toughness than annealed
Solution heat treatment and overaging	10 min, 940 °C (1725 °F), water quench, plus 4 h, 675 °C (1250 °F), air cool	Intermediate strength between annealed and STA, but improved ductility and damage tolerance properties compared to STA

Table 2.4: Guaranteed Tensile Requirements of Ti-6-4 products [32-36].

Property	
Tensile Strength (MPa)	895
Yield Strength (MPa)	828
Elongation (%)	10
Reduction in Area (%)	25

Table 2.5 : Effect of heat treatment on the tensile strength and toughness of various flat-rolled products[28].

Heat Treatment Type	Tensile Yield Strength ksi	Toughness K_{IC} ksi (in) ^{1/2}
Annealed (continuously rolled sheet)	132, 142 transverse	128, 140 transverse
β annealed (plate)	131	134
β STA age @ 1250 °F	128	150
β STA age @ 1000 °F	143	120
STA age @ 1250 °F	137	105
STA age @ 1000 °F	159	80

Table 2.6: Physical and Mechanical Properties of SCS-6 fibre [10].

Property	
Tensile Strength	3400 MPa (500ksi)
Tensile Modulus	400 GPa (60 msi)
Density	3.045 g/cm ³ (0.11 lb/in ³)
Coefficient of Thermal Expansion	1.5 x 10 ⁻⁶ /°C (2.7 x 10 ⁻⁶ /°F)

Table 2.7: Reaction constants for the interfacial reaction between Titanium and Ti-6-4 with various SiC reinforcements [31, 57].

Fibre	Matrix	Q (cal mol ⁻¹)	k ₀ (cm sec ^{-1/2})
SiC (Si+C coating)	Ti	52400	0.37
SiC (C coating) I	Ti	56600	1
SiC (C coating) II	Ti	63000	1.3
SiC (Si+C coating)	Ti-6-4	59260	0.41
SiC (C coating) I	Ti-6-4	58180	0.31
SiC (uncoated)	Ti-6-4	62700	0.52
SCS-6	Ti-6-4	73100	varies with temp.

I- coating is not fully consumed
II- coating is consumed

Table 2.8: Prediction of composite strengths compared to experimental results [104].

Fibre	Matrix	Experimental Strength (MPa)	R. O. M. (MPa)	G. L. S. (MPa)
SCS-6	Ti-24-11 V _f =0.30	1324	1557	1442
SCS-6	Ti-24-11 V _f =0.22	1140	1647	1543
SCS-6	Ti-6-4 V _f =0.27	1556	1644	1566
SCS-6	Ti-6-4 V _f =0.34	1670	1832	1726
SCS-6	Ti-6-4 V _f =0.34	1490	1832	1726
SCS-6	Ti-6-4 V _f =0.43	1900	2080	1944

Table 3.1: Chemical composition and mechanical properties of the Ti-6-4 wire.

TITANIUM INDUSTRIES CENTRE OF EXCELLENCE				TEST REPORT							
715, chemin Sfrancois Montreal, Que. H4S 1W6 Tel: 334-6777 Fax: 334-4280				7888 Alderbridge Way Richmond, B.C. V6X 2A5 (604) 276-8433 Fax: (604) 276-8483							
				DATE: 10/17/85 OUR ORDER / ITEM NO.: MTL-720/1 CUSTOMER ORDER NO.: 45(F)23527							
CUSTOMER NAME: RUSSEL STEEL 181, DALTON AVENUE KINGSTON, ONTARIO K7K 6C2											
MATERIAL DESCRIPTION				NO. PCS.	LINEAR FEET/WEIGHT		HEAT NO.				
3.06" DIA. (+.0004") X SPOCL				2	1.338		DF-87				
SPECIFICATION											
ASTM B-346 GRADE 5											
CHEMICAL ANALYSIS											
N	C	H	Fe	Ox	Al	V	Sn	Pd	Mo	Zr	Ni
0.0080	0.0300	0.0032	0.1300	0.1100	5.9200	3.9300					
RESIDUALS EACH	RESIDUALS TOTAL	Ti	Cr	Ta	Cb	Y	O PLUS Fe	Mn	Si	Mg	
		BAL									
MECHANICAL PROPERTIES											
ENSILE	L	154,000	L	HARDNESS							
TENSILE STRENGTH KSI	T		T								
YIELD STRENGTH	L	134,800	L	GRAIN SIZE							
SI	T		T								
ELONGATION	L		L								
	T		T								
REDUCTION	L		L								
IN AREA	T		T								
BEND TEST				OTHER DATA							
STATIC NOTCH STRESS RUPTURE				ANNEALED							
ULTRASONIC TEST											
TENSILE TEST				ELONGATION: 4.82							
IMPACT TEST											
FLATTENING TEST											
HYDROSTATIC TEST											
EDDY CURRENT TEST											
PNEUMATIC TEST											
REVERSE FLATTENING TEST											
DYE PENETRANT											
RADIOGRAPHIC (X-RAY) TEST											
MICROSTRUCTURE PERFORMED											
MICROSTRUCTURE COMPLIANCE											
MICROSTRUCTURE PERFORMED											
MICROSTRUCTURE COMPLIANCE											

DATE 10/11/85

SIGNED

TOTAL P.02

Table 5.1: Details of the heat treatment routes of the TMC samples.

<u>Sample</u>	<u>Method of Encapsulation</u>	<u>Heat Treatment</u>
Duplex Anneal	1. Corning glass powder encapsulation 2. Corning glass powder encapsulation	1. 10 min @ 940 °C, AC 2. 4 hrs @ 675 °C, AC
Recrystallisation Anneal	Vacuum Furnace (all steps)	1. in 15 min, 23 to 940 °C 1 hrs 15 min @ 940 °C 2 940 to 760 °C, FC @ 55 °C/hr 3 760 to 480 °C, FC @ 370 °C/hr 4 480 °C, AC
Solution Treat & Age	1. Quartz Encapsulation 2. Vacuum Furnace	1. 10 min @ 940 °C, WQ 2. 4 hrs @ 525 °C, AC
β Anneal	Corning glass powder encapsulation (all steps)	1. 30 min @ 1035 °C, AC 2. 2 hrs @ 730 °C, AC

Table 6.1 Composition of Ti-6-4 phases in TMC sample.

Sample	Region & Phase	Ti (wt%)	Al (wt%)	V (wt%)	Fe (wt%)	Si (wt%)	Total (wt%)
as HIP'ed	10 μ m from fibre - β phase	84.55	3.76	10.51	0.79	0.13	99.74
as HIP'ed	100 μ m from fibre - β phase	81.01	3.06	12.65	0.95	0.15	97.82
as HIP'ed	10 μ m from fibre - α phase	89.49	5.60	3.80	0.00	.15	97.82
as HIP'ed	100 μ m from fibre - α phase	89.58	5.66	2.67	0.00	0.10	98.01
β Anneal	10 μ m from fibre - β phase	82.86	3.30	12.21	0.67	0.62	99.66
β Anneal	10 μ m from fibre - β phase	83.05	3.63	10.72	0.73	0.14	98.27
β Anneal	10 μ m from fibre - β phase	86.32	4.13	8.90	0.54	0.13	100.02
β Anneal	10 μ m from fibre - α phase	91.04	5.86	2.11	0.00	0.51	99.52
β Anneal	10 μ m from fibre - α phase	91.21	5.67	2.37	0.00	0.14	99.39

Table 6.2: Interfacial reaction zone thicknesses.

<u>Sample</u>	<u>Experimental Reaction Layer Thickness</u>	<u>Theoretical Reaction Layer Thickness</u>
As HIP'ed	0.72 μm	0.46 μm
Recrystallisation Anneal	1.44 μm	2.84 μm
	0.73 μm	0.81 μm
β Anneal	0.86 μm	2.36 μm

Table 6.3: Critical loads to push out fibres from post HIP'ed composite sample.

Run	P_{cr} (N)
1	3.378
2	4.505
3	3.216
4	5.800
5	4.430
6	4.240
7	5.163
8	4.253
9	4.802
10	4.249
11	5.439

Table 7.1: Predictions of composite strength.

Model	Composite Strength (MPa)
Rule of Mixtures	1384.7
Global Load Sharing	1322.3
Local Load Sharing	1037.6

FIGURES

Figure 1.1: Titanium Matrix Composite control linkage for the convergent divergent nozzle for F-22 Raptor Advanced Tactical Fighter. The SiC fibres are oriented longitudinally along the cylindrical member. Figure courtesy of Atlantic-IMT.

a)

b)

c)

Figure 1.2: Various gas turbine components that have been proposed or successfully manufactured, a)TMC turbo-shaft [10],b) TMC axial compressor rotor [3], and c) Centrifugal compressor with internal reinforcement ring [11].

Figure 1.3: Sketch of traditional compressor disk (left) with sketch of proposed compressor ring (right) made possible by reinforcing TMC ring shown in inset at upper right [13].

Figure 1.4: Progressive weight savings of new compressor designs compared to current compressor rotors. Maximum weight savings are realized with TMC rings and spacers [14].

Figure 1.5: Relative temperature vs. specific strength capabilities of various materials considered for gas turbine applications. Note that TMC's display superior qualities in the intermediate temperature range [5].

Figure 1.6: Projected breakdown of advanced aerospace material use in the year 2010. TMCs are expected be used in 20% of most aerospace designs [13].

Figure 2.1: Titanium / β stabilizer phase diagram illustrating the change in titanium alloy microstructure with increasing β stabilizer concentration [21].

Figure 2.2: Portion of the Titanium / Vanadium phase diagram illustrating the effect of the vanadium addition on the Ti- 6Al- 4V alloy microstructure [28].

Figure 2-3: Matrix of Ti- 6Al- 4V microstructures produced by heating the alloy into different regions in the Ti-V phase a diagram and varying the cooling rate [17].

Figure 2.4: A stress relief nomograph for the Ti- 6 Al- 4 V alloy system [17].

a) Full Anneal microstructure

b) Recrystallized Anneal

c) Duplex Anneal

60 μ m

d) Beta Anneal

Figure 2.5 (a-d): Various heat treated annealed microstructures of Ti- 6 Al- 4 V alloys [28].

a)

b)

Figure 2.6: Micrographs of solution treated and *a)* aged microstructure *b)* over-aged microstructure[23].

Figure 2.7: Typical stress strain curve for annealed Ti- 6Al- 4V alloy at different temperatures [28].

Figure 2.8: Typical stress-strain curve for STA Ti- 6Al- 4V bar at different ambient temperatures [28].

Figure 2.9: Deleterious effect of quench delay on the tensile strength and yield strength of STA Ti- 6Al- 4V alloys [23].

Figure 2.10: Larson Miller plot showing the increased creep resistance imparted by β processing Ti- 6Al- 4V [17, 28].

Figure 2.11 Example of a duplex microstructure produced through shot peening and subsequent heat treatment with an equiaxed surface and a lamellar core [17].

Figure 2.12: Schematic sketch of the various regions within the SCS-6 fibre [46].

Figure 2.13: Degradation of SCS-6 fibre strength with increasing temperature. Fibre retains much of its strength up with temperature up to ~1500 °F (800 °C) [48].

Figure 2.14: Crack growth from the reaction layer/matrix interface in Ti-6-4/SCS-6 composite specimens a) from the reaction layer, and b) initiating at the unbroken reaction layer [59].

Figure 2.15: Graph of fracture energy as a function of reaction layer thickness in a Ti/SiC composite [50].

Figure 2.16: Stress distribution for a single SiC monofilament (with no core) in a Ti matrix after cooling through 500 K [53].

Figure 2.17: Geometry of fibre holder and deposition geometry for the EBED coating of SCS-6 fibres with Ti-6-4 matrix [69].

Figure 2.18: Ti-6-4 / SiC composite with an 80% volume fraction of fibre produced through EDED MCF and HIPing [64].

Figure 2.19: Temperature necessary to evolve 133 kPa of vapour pressure from several common alloying elements [64].

Figure 2.20: Eccentric matrix coating of a SiC fibre due to poor process control of the EBED method [69].

Figure 2.21: Schematic sketch of a set up for the coating of SCS-6 fibre using the magnetron sputtering fabrication method [71].

Figure 2.22: Table of oxygen contamination during the coating of SiC fibres through various MCF techniques [69].

Figure 2.23: Schematic sketch of an electric arc spraying apparatus [75].

Figure 2.24: Schematic sketch of the deposition of a Ti matrix onto a substrate using a direct current plasma spray gun [76].

Figure 2.25: Schematic diagram of Atlantic-IMT's wire wrapping process [88].

Figure 2.26: Process window for the consolidation of a TMC component [5].

Figure 2.27: Typical creep curve for a metal illustrating the three regions of creep [92].

Figure 2.28: Schematic illustrating the 3 consolidation stages of the hot pressing of a foil fibre foil composite section [93].

Figure 2.29: Densification map for complete consolidation of a foil fibre foil preform with different inter-fibre spacings (L) [94].

Figure 2.30: Examples of a rectangular and triangular (hexagonal) fibre array [94].

Figure 2.31: Diagram for the definition of the contact angle ϕ [98].

Figure 2.32: Micrograph of a halted consolidation illustrating the barrelling and earing defects that have to be removed for full consolidation to take place [98].

Figure 2.33: Diagram illustrating the compaction developed for the consolidation of thermal spray monotapes [90].

Figure 2.34: Schematic of composite with one matrix crack. Fibres with no breaks within L_f are unbroken (I) and fibres with breaks within L_f are considered broken (B) [104].

Figure 2.35: Schematic of composite cylinder with a central core of broken fibres used to calculate the failure criteria of the composite through the Local Load Sharing Model [105].

Figure 2.36: Schematic diagram of a fibre push out test using a Vicker's hardness indenter [109].

Figure 2.37: Schematic of the force vs. displacement plot for a single fibre push out test [108].

a)

b)

Figure 4.1: Schematic sketch of the two possible filament winding orientations. a) Is initial rectangular orientation and b) is the stable triangular orientation.

Figure 4.2: Photograph of a completed stainless steel mandrel.

Figure 4.3a) Front view of the filament wind apparatus: 1. feed spool system, 2. tension meter, 3. mandrel 4. rotational motor, 5. translational slide.

Figure 4.3 b) side view of the filament wind apparatus: 1. mandrel, rotational motor and translational slide, 2. guide vise, 3. tension meter, & 4. feed spool system.

Figure 4.4: Fully wound green preform mandrel. This mandrel was wound using a semi-continuous method.

Figure 5.1: Photograph of the vacuum sleeve and evacuation tube (side view).

Figure 5.2: Welded can assembly on mandrel.

Figure 5.3: Welded can assembly with 4.47 hole bored through center of mandrel to aid HIP consolidation (top view).

HIP envelope

Figure 5.4: Variation in temperature and pressure during the HIP cycle used to consolidate the TMC.

Figure 5.5: Schematic diagram of the Fibre Push Out Test set up.

Figure 6.1: Vacuum can after HIP consolidation has occurred.

Figure 6.2: TMC ring after vacuum can has been machined away. a) Top view and b) side view.

Figure 6.3: Micrograph of TMC ring section demonstrating that consolidation had occurred. Note that the matrix and fibre damage occurred from the EDM cutting.

Figure 6.4 Micrograph of the consolidated TMC cross section. There are three layers of good fibre distribution followed by several layers where the fibre distribution has deteriorated.

Figure 6.5 Flaws in the TMC. a) A micrograph illustrating the formation of an hourglass type void in the poorly oriented portion of the TMC ring. b) Fibre/ fibre contact in the same region of the composite.

Figure 6.6: Post HIP'ed microstructure. a) Next to fibre, b) near steel can wall, and c) in the Ti-6-4 control zone.

Figure 6.7: Micrograph of the duplex annealed microstructure. The desired microstructure of mixed equiaxed and lamellar α did not form.

Figure 6.8: Micrograph of the recrystallisation annealed sample. a) Large equiaxed α grains with β grains growing at the triple grain boundary junctions, and b) micrograph showing that the microstructure remains homogeneous throughout the sample.

Figure 6.9: Microstructure of the STA TMC sample. a) The microstructure is similar to the forecast microstructure except for the small grain size and no formation of lamellar α , and b) micrograph depicting the stability of the microstructure away from the fibres.

Figure 6.10 Micrograph of the β annealed sample. a) Micrograph away from the fibre depicting the desired β annealed microstructure, and b) micrograph of the matrix in the vicinity of the fibre. Note that the microstructure is fine grained, equiaxed α close to the fibre before reverting to the desired microstructure $\sim 50 \mu\text{m}$ away.

Figure 6.11: Back scatter electron image of the TMC sample. The light region is the β grain boundary and the dark region is the α grain.

a) interface

b) α phase 1 μ m away from fibre

c) α phase 2-3 μ m away from fibre

d) α phase 10 μ m away from fibre

e) β phase 10 μ m away from fibre

f) α phase 100 μ m away from fibre

g) β phase 100 μm away from fibre

Figure 6.12: Thin window EDAX analysis of the β annealed sample. Carbon concentration (far left peak) falls to background levels at 3 μm distance from the fibre.

Figure 6.13: Secondary SEM image illustrating the automated spot analysis: a) STA b) β anneal.

Figure 6.14: Compositional variances of alloying elements with radial distance away from the fibre in the β annealed sample.

a)

b)

Figure 6.15: Compositional variances of alloying elements with radial distance away from the fibre in the STA sample.

a)

b)

c)

d)

Figure 6.16: Sem micrographs of the various TMC samples. In these pictures the interfacial reaction layer is clearly visible as the white region bordering the SCS-6 fibre.
a) as HIP'ed, b) recrystallisation annealed, c) STA, and d) β annealed.

Figure 6.17: Force vs. displacement plot of a successful fibre push out test.

a) b)
Figure 6.18: SEM micrographs of a pushed out fibre. The clean surface of the fibre indicates that the fibre sheared in the carbon coating.

a) b)
Figure 6.19: SEM micrographs of a pushed out fibre from the indenter side. The ragged interface in b) suggests that the indenter impinged on the matrix.

Fibre Push Out (Recrystallisation Anneal 1)

Figure 6.20 Force vs. displacement plot of a heat treated specimen. The fibre could not be push out.

a)

b)

Figure 7.1: Schematic sketch of the geometry assumed for TMC strength calculations. a) Side view, and b) cross section.

Appendix A: Procedure for Long Stroke Fibre Push Out Testing (LSFPO testing)

References:

1. Microsense 3046A Gaging System - Operation and Maintenance. ADE Corporation Literature, Rev. G, 05/15/1986
2. R. Berriche, "Design and Initial Development of the Nanomechanical Probe: A Depth Sensing Indentation and Scratch Testing Facility". National Research Council Report #: LTR-ST-1903, November 1992
3. R. Berriche, "Manual for the NanoMechanical Probe. National Research Council of Canada Internal Document, Ver. 4, April 1997

A.1.0 Aim

This report is intended as an addendum to [3] and redevelops and expands NRC's capability to perform fibre push out testing on various composite samples. The following test differs from the previous fibre push out test, documented on page 18 of [3], in that it enables strokes of greater than 20 μm to be attained. This allows for greater loads to be applied to the composite sample and can potentially acquire data for the complete push out of a fibre from the mounted specimen.

N.B.: This document assumes that the user is thoroughly conversant with the operating principles of the nanomechanical probe (NMP) and [3]. It is highly suggested that the user perform the standard depth sensing indentation (DSI) testing previous to attempting LSFPO testing.

A.2.1 NMP Preparation

Several modifications have to be made to the NMP before running the LSFPO test. Please ensure that you have the permission of authorized personnel before making these modifications. It is highly recommended that all modifications be reversed upon the completion of LSFPO testing.

The first modification is to the capacitance probe set-up. Normal operation consists of CP1 acquiring displacement data from the movement of the PZT and CP2 acquiring the force history from the load cell. There are two supplemental capacitance probes, CP3 and CP4, mounted on the NMP. In LSFPO testing CP3 is used to monitor the z-axis travel of the motor stage.

CP3 is typically used to monitor x-axis travel of the motor stage during depth sensing scratch testing (DSS). To verify whether or not the capacitance probe is set up for LSFPO simply look at CP3. If the normal of the capacitance probe's sensing plate is

horizontal it is set up for DSS, if it is vertical then it is prepared for LSFPO. If CP3 is set up for DSS rotate the probe so that its sensing plate is pointing upwards.

Hook the input and output coaxial cables for CP3 into the PROBE A terminal on the back of the top 3046 Microsense unit [1]. Turn the 3046 unit on. Set the TOLERANCE to A (the range knob). Set the MODE to A (mode knob). The needle of the 3046 should be off scale to the left. Turn the unit off.

Move to the triple junction connecting the CP1 output to the servo box, the data acquisition card and multimeter. Disconnect the output coaxial cable from CP1 and reconnect it with the output coaxial cable from CP3.

Remove the calibration multimeter connected to the MODE OUTPUT of the bottom 3046 unit and connect it to the MODE OUTPUT of the top 3046 unit.

Turn on the top 3046 unit. The CP1 multimeter and the calibration multimeter should now be reading the CP3 voltage. This should be between -12 and -14 VDC as there should no surface for CP3 to sense.

The sensing attachment can now be attached to the stage. This attachment consists of a metal link with a reference (a spring loaded bolt) at one end and a mounting hole on the other side. Mount the attachment to the stage with an appropriate screw. **Ensure that the reference is not directly over the sensing plate of CP3 (angle it away).**

Mount the flat punch indenter and the appropriate load cell in the NMP as described in [3]. Power up the NMP normally but do not turn on the servo-box, the function generator or the Burleigh amplifier. These items comprise the PZT circuit which is superfluous for LSFPO tests.

A.2.2 LSFPO Test Procedure

Before testing can proceed the indenter must be centered in the optical microscope viewscreen as described in [3]. The sample should also be at least 20,000 steps (1.0 mm) below the indenter.

1. Mount sample in sample holder.
2. Mount sample holder in the NMP.
3. Move the sample underneath the optical microscope. Bring the sample into focus.
4. Use the manual jog feature of the motor controller program to center the punch over a fibre.

5. Drop the stage 30.000 steps (3pr-30000). At this point make a note of the step count using the 3tp command. Alternately, exit from the program and restart it to zero the count at this point.
6. Carefully move the sample under the indenter.
7. The sample should be ~25, 000 steps below the indenter. Move the sample up 3pr10000. Check the clearance between the sample and indenter. Repeat 3pr10000 command. Then move stage up 6 times in steps of 3pr500. Then continue to move the stage up 3pr5, monitoring the CP2 voltage for contact. When contact occurs move the stage down 3pr-20, then up 3pr10 then 3pr1 until the exact contact point is found. Make note of the contact point.
8. Drop the stage down 3pr-150.
9. Change the 3 axis motor speeds as described in [3]. Suggested motor speeds for an acquisition rate of 18 pts./sec. is a slow speed of 216 and high speed of 432. Leave the acceleration at 75000. **Before the speed change is accepted by the motor controller program the stage must be manually jogged.** Use the manual jog feature to move the stage down 50 steps and then move it back up 50 steps. This will reset the motor speed parameters to the new variables.
10. Move the stage up 3pr140. Then move up the stage 3pr1 until the contact point is found.
11. Swing the reference over CP3. Adjust the capacitance probe voltage by unscrewing the reference until the multimeter reads +0.5 to +1.5 V.
12. Type in 3pr(stroke). Note that stroke in steps = stroke in μm / 0.05. **Don't hit enter.**
13. Start the ACQ.EXE program. Gains should 1 for both channels. If more accuracy is necessary then try increasing the gains and the number of points acquired per second. Note that if the gain is set too high the data acquisition card will cut out and a constant voltage will be input as data.
14. Run the ACQ program. After about 30 to 50 points hit enter on the terminal command. Let the run finish.
15. At the end of the run type in 3pr-(stroke).
16. **Angle screw away from CP3.**
17. Reset z-axis motor speed to its default settings.

18. Return stage to start point. Move the sample underneath the optical microscope and verify that the fibre has been pushed out.
19. Repeat as necessary, selecting a new fibre each trial.
20. Run analysis as described in page 18 of [3]

Revision: A
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6 February, 1998

IMAGE EVALUATION TEST TARGET (QA-3)

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