

IMPROVING INTEGRALLY HEATED COMPOSITE TOOLING THROUGH COLD SPRAYED COPPER COATINGS AND HEAT TRANSFER SIMULATIONS

by

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This thesis is dedicated to the memory of my grandfather
François Gosselin

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Abstract

Integrally heated composite tooling (IHCT) is seen as a low cost alternative to autoclave manufacturing of polymer matrix composites. IHCTs consist of a composite tool heated by surface heaters; temperature distribution is ensured by a thermally conductive metallic sheet. The main original contributions of this thesis was the development of a new method for applying copper coatings onto carbon fibre/epoxy PMCs, the production of larger size samples, and the characterisation of the performance of the coatings and laminates obtained. It was shown that this method shows potential for producing the thermally conductive layer in an IHCT. Another contribution was the characterisation of parameters affecting temperature distribution across IHCTs through heat transfer simulations, leading to guidelines for IHCT design.

Work aimed at fulfilling the following objectives: 1) produce copper coatings on carbon fibre/epoxy composites using Pulsed Gas-Dynamic Spraying, 2) characterise the mechanical and thermal behaviour of the coated composites, 3) manufacture an IHCT with coated composites and comparing it to other types of heated tooling, and 4) identify parameters affecting temperature distribution in IHCT using heat transfer simulations.

Throughout the work, it was showed that Pulsed Gas-Dynamic Spraying could be used effectively for applying copper coatings on composites. This was made possible by co-curing an intermediate layer on the surface of the composite prior to spraying. Coating density was maximised by varying the spray gas pressure and temperature of the coating process.

Mechanical characterisation was performed through Vickers hardness testing, short-span bending, and low frequency cycling. It provided strength values for coated composites and showed that the coating were brittle but harder than bulk copper. Additionally, thermal characterisation showed that coated composites could sustain temperatures up to 250°C.

An IHCT was fabricated using coated composites. Comparison with other heated tooling revealed that the copper coating significantly improved in-plane conductivity and

temperature uniformity compared to bare composite tooling. The coating was slightly less conductive than the copper sheet of a conventional IHCT.

Simulation work showed that temperature uniformity across IHCTs can be improved by mitigating convective losses, increasing the copper thickness, and by distributing the heat source over a greater surface.

In the end, the work demonstrated that coated composites can be effectively used as IHCT materials, leading to a composite tooling viable for small production series. The work also provided key parameters for improving temperature distribution in IHCTs, leading to potential improvements for composite parts manufacturing consistency using IHCTs.

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Nomenclature

Latin Symbols

A	Area [m ²]
A_{Picture}	Area of an image [pixel·pixel]
A_s	Surface area [m ²]
$A_{\text{Threshold}}$	Filtered area of an image [pixel·pixel]
b	Width [m]
c	Speed of sound [m/s]
C_p	Specific heat capacity [J/g·K]
d	Average diagonal lengths for Vickers hardness [m]
E	Modulus of elasticity [GPa]
E_B	Bending modulus [GPa]
$\dot{E}_{\text{st}}$	Energy storage rate [W]
$\dot{E}_{\text{in}}$	Rate of energy entering a domain [W]
$\dot{E}_{\text{out}}$	Rate of energy leaving a domain [W]
$\dot{E}_g$	Rate of energy generated inside a domain [W]
F	Applied force [N]
G	Shear modulus [GPa]
H	Energy absorbed by or released from a chemical reaction [J]
HV	Vickers hardness
h	Convection coefficient [W/m ² ·K]
h	Thickness [m]
I	Second moment of area [m ⁴]
k	Thermal conductivity [W/m·K]
L	Supported beam span [m]
L	Lorenz number [W·Ω/K ²]
L_f	Load [kgf]
m	Mass [kg]
M	Molar mass [g/mol]
M_B	Bending moment [N·m]
M_e	Mach number at the exit of a nozzle

M_s	Mach number of the PGDS shock-wave
P	Gas pressure [MPa]
P_{Gas}	Stagnation pressure of the spraying gas [MPa]
q	Heat flux (W)
Q	Heat [J]
R	Radius of curvature [m]
$\bar{R}$	Universal gas constant [J/mol·K]
R_{th}	Absolute thermal resistance [K/W]
s	Standard deviation of the surface temperature standard deviation at each time step [°C]
SS	Sum of squares of a contrast
t	Time [s]
T	Temperature [°C or K when specified]
T_d	Polymer degradation temperature [°C]
T_g	Glass transition temperature [°C]
T_{Gas}	Gas heating temperature [°C]
T_p	Powder pre-heating temperature [°C]
t	Time [s]
u	Gas velocity [m/s]
U_d	Substrate displacement rate [mm/s]
v	Volume fraction
v_f	Fibre volume fraction
V	Volume [m ³]
V_s	Shear force [N]
$\bar{y}$	Average standard deviation of surface temperature [°C]
y	Vertical distance [m]

Greek Symbols

α	Coefficient of thermal expansion [m/m·°C]
γ	Specific heat ratio
Δh	Difference in height [m]
ΔT	Difference in temperature [°C]
ε	Strain [mm/mm]

ρ	Density [kg/m ³]
σ	Normal stress [MPa]
$\sigma_{\text{electrical}}$	Electrical conductivity [S/m]
σ_{std}	Standard deviation
τ	Shear stress [MPa]
υ	Beam deflection [m]
ϕ	Spherical inclusion content

Abbreviations

CPMC	Coated Polymer Matrix Composite
CTC	Control Thermocouple
DSC	Differential Scanning Calorimetry
IAR	Institute for Aerospace Research
IHT	Integrally Heated Tooling
IHCT	Integrally Heated Composite Tooling
LP-CGDS	Low-Pressure Cold-Gas Dynamic Spraying
NRC-AMTC	National Research Council of Canada - Aerospace Manufacturing Technology Centre
NRC-IAR	National Research Council of Canada - Institute for Aerospace Research
OHTC	Overheat Thermocouple
OOA	Out-of-Autoclave
PGDS	Pulsed Gas-Dynamic Spraying
PMC	Polymer Matrix Composite
SSB	Short-Span Bending
TC	Thermocouple
TGA	Thermogravimetric Analysis
UDF	User-defined function for FLUENT

Chapter 1

Introduction

1.1 Background

Quality and reproducibility of structural polymer matrix composite (PMC) parts are closely related to their manufacturing process. Traditionally, the aerospace industry has mainly used autoclaves for processing PMC parts [1–4]. However, with the advent of bigger PMC structures such as the wings and sections of the fuselage in the Boeing 787, Airbus A350 and Bombardier CSeries [5–7], operational costs of autoclave manufacturing are becoming prohibitive. This ushered in a strong focus on the development of out-of-autoclave manufacturing processes that aim at achieving comparable quality and consistency as autoclave manufacturing while being more affordable [1–4, 8–11].

The National Research Council Canada - Institute for Aerospace Research (NRC-IAR) has developed a prospective out-of-autoclave process featuring integrally heated composite tooling (IHCT) [4, 12, 13]. The process consists in consolidating PMC parts on specially designed self-heated tools, i.e. IHCT, which enables precise control the cure temperature. The IHCT is primarily composed of a PMC structure used as a mould, or tooling. PMC parts are manufactured on the tool inner surface. The outer surface of the IHCT is lined with a copper sheet for improving in-plane temperature conductivity, onto

which a series of surface heaters are mounted for providing the energy necessary to cure PMC parts. The PMC structure of the IHCT offers accurate part dimensioning by alleviating the issue of mismatched coefficients of thermal expansion between the tooling and the parts to manufacture. The surface heaters can provide non-uniform heat flow for achieving essentially uniform polymer cure temperatures at any given time regardless of PMC/manufacturing irregularities such as non-uniform part thickness.



Figure 1.1: Backseat integrally heated composite tooling (IHCT) designed by NRC-IAR [4]

A practical difficulty related to producing the IHCT is to ensure proper bonding between the copper sheet and the PMC structure. Inadequate contact between the two materials will prevent heat from effectively being transferred to a PMC part during manufacturing, leaving colder zones that affect cure and thereby PMC part quality. Currently, for relatively flat structures the copper sheet can be formed manually to the shape of the tooling, but for highly curved tooling the copper sheet or sheets require expensive forming processes to ensure intimate contact. In the latter case, this solution becomes impractical for IHCTs that are usually produced in small series. Instead, it would

be preferable to spray a metallic coating directly onto the outer face of PMC structures, to alter this surface property.

Metallic coatings can be deposited quickly on metallic surfaces using thermal spray and kinetic spray processes. However, using these processes on PMCs may result in several issues such as poor adhesion, delamination, erosion, melting and thermal degradation [14–22]. This is particularly true for epoxy-based PMCs which are the most common PMCs in structural applications. Nevertheless, in recent years Robitaille et al. showed that they could deposit a zinc coating on the surface of carbon fibre and epoxy-based PMCs using the pulsed gas-dynamic spray (PGDS) process [22]. Coating deposition was made possible by co-curing metallic particles on the surface of PMCs to prevent PMC erosion and to promote coating adhesion. A similar approach for depositing copper coatings on PMCs should be investigated to improve their thermal conductivity, which would be desirable for the IHCT. The advantages of the coating route include: 1) the need for metal forming is eliminated and 2) coatings can easily be applied to any curved surface given spray accessibility. Furthermore, like other spray processes the PGDS process deposits coatings linearly. This feature brings the possibility of changing the thickness of the coating locally, providing a simple method of producing copper layers of non-uniform thickness which can alter heat flow. Such layers which are presented in this work are subsequently referred to as “patterns”.

In addition to developing copper coatings for PMCs that can be used in an IHCT application, there is also a need to optimise the design of the IHCT. Work has been done in the past by students Michel Houde [23], Tariq Maksoud [24], Mélanie Brillant [25] and Imane El Karafi [26] at the University of Ottawa to understand the heat transfer mechanisms in IHCT using heat transfer simulations. However, additional work is required for identifying design parameters that can increase temperature uniformity across the IHCT. Furthermore, it would also be interesting to assess the potential of IHCTs featuring copper patterns.

1.2 Objectives

The objectives of the thesis consisted in showing that copper coatings can be deposited on PMCs using PGDS, and that coated PMCs (CPMCs) can be used for manufacturing IHCTs that may be used for producing PMC parts. Pursuit of these objectives resulted in the following milestones:

- Producing CPMCs by depositing a copper coating on PMCs using PGDS;
- Characterising the physical, mechanical and thermal behaviours of CPMCs;
- Manufacturing an IHCT prototype out of CPMC (IHCT-CPMC);
- Comparing an IHCT-CPMC with other types of IHCTs; and
- Identifying influential IHCT design parameters from heat transfer simulations made using FLUENT.

The thesis is organised in 7 chapters. Chapter 1 introduces the subject and states the objectives that were sought in the research project.

Chapter 2 discusses important concepts and prior engineering work relevant to the thesis. The chapter investigates first the differences between autoclave and out-of-autoclave manufacturing for PMCs and why conductive heating as offered by IHCTs may be a viable solution for reducing defects in PMCs. Afterwards, methods for producing copper coatings are presented, with a primary focus on properties of the copper coatings made using thermal spray and kinetic spray processes. A discussion about the viability of these families of processes for coating PMCs follows. Finally, work related to heat transfer simulation in IHCTs is presented. This last topic discusses previous work done to understand heat transfer in IHCTs and mathematical models required for predicting the behaviour of the simulated materials.

Chapter 3 presents materials, equipment and procedures used in the accomplishment of the thesis work. The chapter begins with a description of the materials used for manufacturing CPMCs. Afterwards, the manufacturing processes are detailed. Focus is on the autoclave and out-of-autoclave processes used for manufacturing PMCs and on the

kinetic spray processes used for coating the PMCs. A description of the physical, mechanical and thermal characterisation of the materials and IHCTs is then presented. The chapter concludes with the procedures and tools used for simulating heat transfer in IHCTs using commercial software FLUENT.

Chapter 4 discusses the deposition of copper coatings on PMCs using kinetic spray processes and the characterisation of CPMCs. The chapter begins by analysing the effect of spraying parameters on the deposition of copper on PMCs using PGDS. The coatings made from PGDS are then briefly compared to coatings made with low-pressure cold gas-dynamic spraying (LP-CGDS). The characterisation of CPMCs manufactured using optimal coating parameters follows. First, the machinability of the coatings is assessed. Then, the temperature limit of CPMCs is discussed. Finally, the chapter concludes with the mechanical characterisation of CPMCs. The characterisation consisted of coating hardness measurements, CPMC short-span three-point bending and CPMC low-frequency cycling.

Chapter 5 discusses the potential of CPMCs for an IHCT application. The chapter begins with the manufacturing of an IHCT-CPMC prototype and its characterisation. Afterwards, temperature distribution across an IHCT-CPMC is investigated during heating. The temperature uniformity across its outer surface is compared with other types of IHCTs to evaluate its temperature distribution capabilities. The chapter concludes with an analysis of the temperature distribution across the IHCT-CPMC when subjected to temperature profiles mimicking a curing operation.

Chapter 6 investigates the potential of heat transfer simulations as a tool to optimise IHCTs. The chapter begins by discussing the accuracy of temperature predictions obtained from heat transfer simulations by comparing the results with empirical IHCT temperature distributions. Afterwards, a parametric study using heat transfer simulations for identifying the most influential parameters for the IHCT surface temperature uniformity is presented. The dominant factors are then further investigated in simulations, leading to recommendations for designing IHCTs.

Chapter 7 presents a brief discussion of the findings made during the thesis. A preliminary assessment of CPMCs and IHCTs-CPMC is presented. The chapter concludes with recommendations regarding future work on CPMCs and IHCTs.

1.3 Contributions

The main original contributions of this thesis was the development of a new method for applying copper coatings onto carbon fibre/epoxy PMCs, the production of larger size samples, and the characterisation of the performance of the coatings and laminates obtained. As demonstrated in this thesis, the new copper coated composite material shows great potential for IHCT applications. In such cases, the copper coating could replace the thermally conductive metallic sheet bonded on the PMC in high curvature areas leading to simpler manufacturing and potentially better contact between the coating and PMC.

Another contribution is the proposed guidelines for improving temperature uniformity across IHCT which were obtained from heat transfer simulations. Knowledge can be used for helping design tools that can provide more uniform polymer cure conductions.

Results of the thesis were the subject of three oral presentations in conferences (CASI 2011 [27], ASC 2011 [28] and SEICO 2012 [29]), and was accepted for oral presentation at ICCM 19.

Chapter 2

Review of literature

In this chapter, the state of the art in integrally heated tooling (IHT) and in spray coating processes is discussed. First, a review of PMC processing is presented which explains the shift towards out-of-autoclave processes for which the IHCT was developed at NRC-IAR [4]. Afterwards, potential coating processes for replacing the copper sheet in the IHCT are presented, leading to the selection of kinetic spray processes. Finally, modelling strategies for IHT are examined. A review of previous IHT modelling work is presented, as well as mathematical models that can be used for describing the physical behaviour of IHTs.

2.1 Composites manufacturing

2.1.1 Energy equation

Manufacturing epoxy-based PMCs involves cross-linking a liquid or semi-solid epoxy resin and its hardener around a reinforcement to obtain a solid composite material. Conversion occurs when sufficient energy is provided to achieve the chemical cross-linking reaction. This reaction can be activated by heating; convective heating is used in

autoclave manufacturing and out-of-autoclave manufacturing using ovens [30, 31] while conduction heating is used in IHTs [4, 32–34]. A brief review of these heating mechanisms follows and all important heat transfer parameters are defined. It should be noted that Monaghan et al. also observed an important contribution (~30%) of radiant heating in autoclave [35]. However, this mode of heating will not be discussed here as it was noted at operating temperatures (~400°C) much higher than those required for epoxies, and in an environment with very low convection (heat transfer convection coefficient $h \approx 10 \text{ W/m}^2 \cdot \text{K}$).

The general energy balance equation which is valid for all manufacturing processes states that the rate at which heat is stored in a domain (i.e., combination of the PMC structure and tooling) is equal the sum of the rate of energy that enters the domain and is generated within it minus the rate of energy that:

$$\dot{E}_{\text{st}} = \dot{E}_{\text{in}} - \dot{E}_{\text{out}} + \dot{E}_{\text{g}} \quad (2.1)$$

where $\dot{E}_{\text{st}}$ is the rate at which energy is stored, $\dot{E}_{\text{in}}$ is the rate of energy flowing in, $\dot{E}_{\text{out}}$ is the rate of energy flowing out and $\dot{E}_{\text{g}}$ is the rate of energy generated.

Energy storage term $\dot{E}_{\text{st}}$ is defined as:

$$\dot{E}_{\text{st}} = \rho V C_p \frac{dT}{dt} \quad (2.2)$$

where ρ is the density, V is the volume, C_p is the specific heat capacity at constant pressure and dT/dt is the time derivative of the temperature. This term governs the heating rate of both the PMC and the tooling used for manufacturing [36]. Lower values of density and specific capacity of the domain will lead to quicker heat transfer, providing greater control over the cure temperature of PMCs.

During PMC manufacturing, there is an energy generation term $\dot{E}_{\text{g}}$ that originates from the exothermic reaction of the epoxy conversion or cure. This was not considered in the present work because of the minimal heat generated by thin laminates [37, 38].

However, future work should investigate including heat generation as exotherms cannot be neglected when laminates increase in thickness [37–40] and their modelling becomes imperative in designing proper control systems for IHCTs.

Finally, $\dot{E}_{in} - \dot{E}_{out}$ refers to the total rate of energy provided to cure PMCs. This external heating can come from various sources, the most common heat transfer modes being convection in autoclaves and ovens, and conduction in IHCTs.

For convection, the heat transfer rate $q_{Convection}$ is equal to the difference between heat entering and heat leaving a surface due to heat transfer by a circulating fluid:

$$\dot{E}_{in} - \dot{E}_{out} = q_{Convection} = hA_s\Delta T_s \quad (2.3)$$

where h is the convection coefficient, A_s is the surface area of the domain in contact with the convective fluid and ΔT_s is the temperature difference between the solid surface of the domain and the convective fluid. From this it can be concluded that the heat transfer coefficient is the parameter governing the heat transfer rate.

Conversely, for conductive heating the heat transfer rate $\dot{E}_{in} - \dot{E}_{out}$ relates to the difference between the heat flux provided by surface heaters q_{Heater} and the heat losses q_{Loss} with the surrounding environment. The net heat transfer rate into an IHT is:

$$\dot{E}_{in} - \dot{E}_{out} = q_{Heater} - q_{Loss} \quad (2.4)$$

For cases with sufficient thermal insulation, the heat losses can be neglected, making the system only dependant on the heater power.

Moreover, in the case of IHTs using localised surface heaters, the heat flux is not uniformly applied across the entire tooling. It is thus important to consider the heat flux inside IHTs. The equation governing the conductive heat flux $q_{Conduction}$ gives:

$$q_{Conduction} = -k\Delta T \quad (2.5)$$

where k is the thermal conductivity, A is the cross-sectional area of the IHCT in which heat flows and ∇T is the temperature gradient. It can be seen that a key design requirement for tooling with localised heating such as the IHCT is the ability to distribute heat evenly, necessitating a high in-plane thermal conductivity.

The implications of both heat transfer mechanisms for manufacturing will be discussed further in following sections which cover convection in autoclaves and ovens, and conduction in IHT.

2.1.2 Autoclave and out-of-autoclave manufacturing

Current standard procedure for manufacturing aircraft PMC structures is based on vacuum bagging of unidirectional fibres or fabrics pre-impregnated with resin (prepreg) which are then cured in an autoclave [1, 2, 4, 30, 31]. In this process, the prepreg is put under vacuum before cure for removing entrapped air that could result in voids. Cure is induced inside an autoclave that acts as a large pressure cooker filled with an inert gas. Gas is pressurised at around 7 bar [1, 9, 31, 41, 42] providing great compaction, enabling fibre volume fractions (v_f) typically above 60% in PMCs [1, 42]. Cure is achieved by heating the gas to the desired temperature which then heats the prepreg by forced convection.

The coefficient of convection in pressurised autoclaves varies greatly depending on autoclave model and operating conditions [30, 31, 35]. Generally, for autoclaves pressurised at 7atm the convection coefficient is greater than $80\text{W/m}^2\cdot\text{K}$ and can reach $180\text{W/m}^2\cdot\text{K}$ [30, 31] ensuring high heat transfer rates to the prepreg. However, effectiveness of heat transfer varies significantly with location inside an autoclave. Slesinger et al. showed that uneven gas circulation (Figure 2.1) can result in 50% difference in heat transfer rates [30]. Nevertheless, high heat transfer rates ensure reproducible and complete cure.

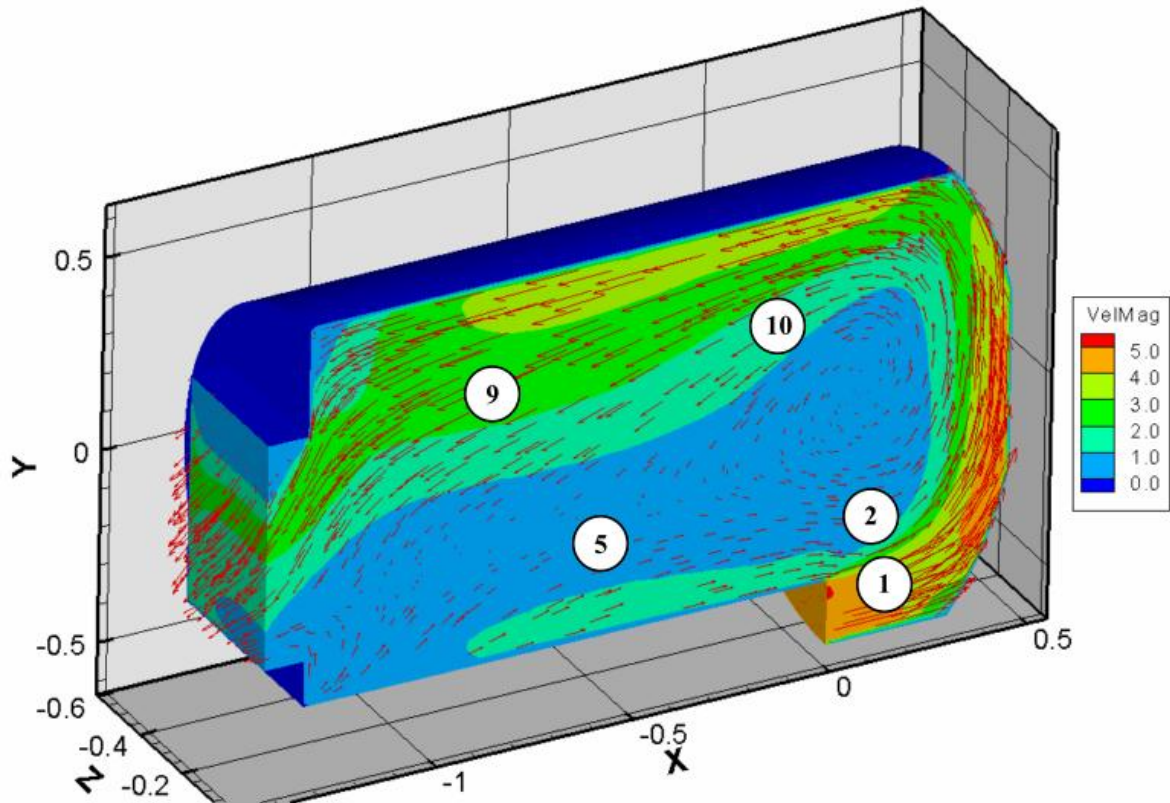


Figure 2.1: Gas velocity inside an autoclave [30]

Despite the key advantages of autoclaves for producing PMCs with high fibre volume fractions and minimal defects, the investment required in acquiring and maintaining an autoclave is relatively high. Hence, in the recent decades engineers and scientists tried to find alternative manufacturing solutions. A solution was to retain the prepreg vacuum bagging process but using an oven for curing [1, 9–11]. In this case the external pressure applied by the autoclave is replaced by a vacuum that consolidates the prepreg under a pressure of approximately 1atm. The absence of external pressure typically results in PMCs featuring lower fibre volume fractions [1]. Furthermore, early trials without autoclave pressure led to PMCs showing greater porosity since there was little resin pressure to collapse any remaining entrapped air [1, 39]. For example, Davies et al. used a Hexply 6376 autoclave prepreg for manufacturing out-of-autoclave and in-autoclave PMCs with resulting void contents of 8.9% and 0.6% respectively [1]. This was later addressed by prepreg manufacturers that developed out-of-autoclave “breathing” prepreg

materials that were designed for removing entrapped air and volatiles, yielding comparable porosity to autoclave PMCs [8–10, 43].

Regardless of advances in out-of-autoclave prepregs a practical limitation to ovens persists. Ovens are affected by low convection coefficients that hinder heat transfer. Johnston showed that removing the autoclave pressure of 7atm reduced the convection coefficient from $180\text{W/m}^2\cdot\text{K}$ to $30\text{W/m}^2\cdot\text{K}$ [31]. Reducing the pressure meant lowering the gas atomic density supporting heat transfer. Literature values for forced convection coefficients in unpressurised autoclaves, i.e. essentially ovens, were measured at 10 to $38\text{W/m}^2\cdot\text{K}$ [31, 35] which may cause issues in cure consistency, mostly when factoring in the non-uniform air circulation.

Limitations to convection in ovens can be mitigated somewhat by improving the heat transfer rate with the tooling. Abdullah et al. tested the effect of adding different textured surfaces and arrays of fins on the heat transfer rate in an aluminium tool [44]. In the best case, the increased surface area from fins that increased the mass by 20% improved heat flow by around 30%. Unfortunately, this remains inferior to autoclave heat transfer rates, and incorporating fins to tooling may not always be practical. It was also shown by the same researchers that the benefits of using fins vary greatly depending on the convective air flow orientation.

Another solution in addressing the low heat transfer rates in out-of-autoclave processes is to replace the air with a heat exchange liquid such as glycol. The greater density of the liquid yields greater convection coefficients than those found in autoclaves, resulting in faster cure [1, 2]. The concept, patented by Graham [45], relies on having the bagged and vacuumed prepreg as well as the tooling inserted between two impermeable bladders containing the heat transfer fluid. The commercialised QuickstepTM process can achieve heating rates as high as $15^\circ\text{C}/\text{min}$ [1, 2]. Researchers showed that high heating rates inherent to the process coupled with specific cure cycles, resulted in PMC parts showing interlaminar shear strengths comparable to those produced in autoclaves, and 35% greater than those made in an oven [1]. However, the cost of operating such equipment and

of the energy required for heating the fluid remains high. Walczyk and Kuppers estimated that the operating cost is 40% lower than that of an autoclave but the initial investment is much higher [33]. In addition to cost there are manufacturing limitations including part size and geometry due to the bladder elasticity limit. Therefore, work has been done to change the heating method. Instead of relying on convection, other solutions relied on conductive heating with integrally heated tooling.

2.1.3 Integrally heated tooling

Conductive heating is a prospective solution for out-of-autoclave PMC manufacturing [4, 32–34, 46]. It is often achieved by integrally heated tooling (IHT), i.e. tooling having surface or internal heaters. In contrast to convection, in IHT heat is directly supplied to the tool, eliminating the need for heating the surrounding environment and lowering energy consumption significantly. Direct contact with the heat source also increases heating rates, leading to shorter processing times [4, 32, 33]. Thus, a business case for IHTs can likely be made on energy savings alone compared to autoclaves or ovens. Additional savings can be made through reduced manipulation by curing the PMC part directly on the layup tool, which also reduces bottle-necks caused by a limited number of autoclaves or ovens.

Moreover, IHTs sometime offer the possibility of altering locally the heat flux. This is particularly beneficial for PMCs with varying thicknesses as requirements for cure energy will differ with location or when providing heating that adapts to resin infusion in liquid moulding processes [12]. This enables IHTs with greater control over cure temperatures compared to convective heating.

Aluminium and steel tooling constitute simple solutions for IHTs used in PMC manufacturing. They offer good wear resistance which enables multiple production runs. For aluminium, its high thermal conductivity (237 W/m·K at 20°C [47]) ensures good temperature uniformity across the tool when heated locally. Walczyk and Kuppers used an aluminium IHT that was heated on its outer face through a silicone heater [33]. They analysed the operational cost of their IHT for PMC manufacturing and found that the IHT

only used 1.2% of the energy required by an autoclave. Similarly, the initial investment towards a metallic IHT was estimated at less than 1% of that of an autoclave. Therefore, there are substantial savings to be made with IHTs for PMC manufacturing.

Despite their good durability and relatively high thermal conductivity, most metallic tools lack the manufacturing accuracy required for ensuring tight tolerances in parts having complex geometries, which is caused by a mismatch in thermal expansion coefficients with PMCs [46, 48]. Aluminium and steel have thermal expansion coefficients at room temperature of 23.6×10^{-6} and 11.7×10^{-6} m/m·K respectively [49], which are much greater than the $2\text{--}3 \times 10^{-6}$ m/m·K of a cross-ply PAN-based carbon fibre PMC or the negligible coefficient along the fibre direction in a unidirectional PMC [50, 51]. This difference in thermal expansion between a metallic tool and a PMC part results in greater shrinkage of the tool compared to the PMC after the cure. For male tools, thermal expansion can be addressed through tool design. However, in the case of female tools, a great difference in thermal expansion can prevent part fabrication because tool shrinkage would crush a part (Figure 2.2).

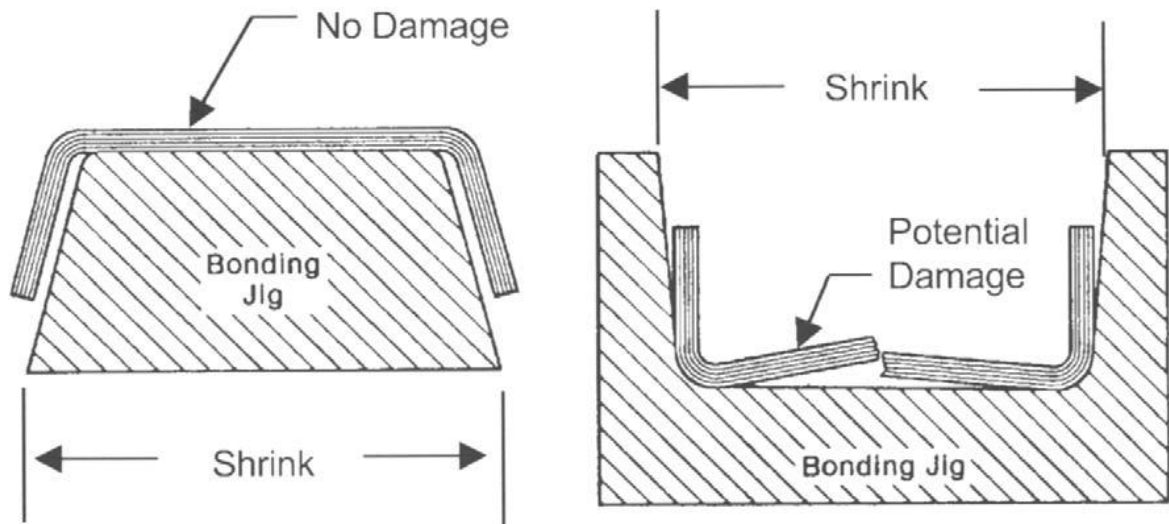


Figure 2.2: Potential effects of tool shrinkage on part quality [52]

Consequently, in early 1980s the aerospace industry adopted carbon/epoxy tooling to mitigate the mismatch in thermal expansion coefficients with carbon/epoxy parts [48].

However, in the later 1980s, the industry looked at a replacement technology for composite tooling due to lack of durability required for high volume manufacturing. Proper PMC tools could be used for approximately 200 cycles, which was 70% less runs than steel tools and 35% less runs than aluminium tools [53]. A more durable metallic replacement material was found with Invar (36% nickel and 64% iron), which also has a very low thermal expansion coefficient (1.6×10^{-6} m/m·K [49]) that nearly matches that of a PMC. With the advent of monolithic PMCs for large primary aircraft structures, Invar is slowly losing its place in the industry because of initial tooling investment costs, weight of the tooling, and operation costs. Another drawback for the operation was the relatively high volumetric heat capacity of the material ($4000 \text{ kJ/m}^3 \cdot \text{K}$ [49]) due its high density, which led to long cure cycles as the tooling was absorbing much of the heat, which is also a problem with all metallic IHTs. Hence, the current trend in industry is a return to PMC tooling for large monolithic structures [46, 48]. The relatively low density ($\sim 1800 \text{ kg/m}^3$ [54]) and volumetric heat capacity ($\sim 1800 \text{ kJ/m}^3 \cdot \text{K}$ [54]) of carbon fibre PMCs are also desirable for IHTs. Another advantage of PMC tooling for large structures is its lower manufacturing cost. A metallic tool requires machining which is especially expensive for harder metals such as Invar and steel, whereas PMCs can be formed easily to a net shape.

Despite having desirable characteristics such as a low coefficient of thermal expansion, low weight and simple forming, PMCs cannot readily be used as IHTs because of the low thermal conductivity of PMCs. Hind and Robitaille measured in-plane thermal conductivity values for carbon fibre PMCs made with unidirectional tape and plain weaves fabricated in autoclave and in a hot press [55]. Results for cross-ply laminates having fibre volume fractions between 55% and 62% yielded in-plane thermal conductivity values of $2.6 \text{ W/m} \cdot \text{K}$ to $3.9 \text{ W/m} \cdot \text{K}$ and through-thickness values of $0.45 \text{ W/m} \cdot \text{K}$ to $0.74 \text{ W/m} \cdot \text{K}$. It was seen that heating PMCs directly can results in hot spots [25, 56] which are undesirable for PMC cure.

Gliesche et al. proposed a solution for minimising temperature variation across the surface of PMC IHTs. This consisted in integrating an electrically resistant carbon fibre roving inside the tool that looped over the entire area [34]. The roving was positioned

accurately using fibre placement technology and was stitched to a glass fabric for electrical shielding. The resulting fabric may be co-cured with the rest of the PMC tooling. Tool heating was achieved by passing an electrical current inside the roving which provided a uniform heat flux of 0.24W/cm^2 . Temperature variation across the tool was measured at 3°C which makes this a viable solution for out-of-autoclave manufacturing. However, fibre placement of the roving and stitching along a precise path leads to manufacturing complexity.

The NRC-IAR IHCT solution that was presented in Chapter 1 avoids the complexity of fibre placement. Instead, a heat transfer tile, consisting of a thermally conductive metallic sheet and localised surface heaters, can simply be bonded to the outer surface of standard PMC tooling, reducing manufacturing costs. Originally, NRC-IAR used an aluminium sheet in the heat transfer tile. However, aluminium was replaced with copper for its greater malleability which simplified the forming process over curved tools.

Raizenne et al. built a IHCT demonstrator (Figure 2.3) for manufacturing a backseat part as a proof of concept [4]. The IHCT consisted of a 6.5mm thick carbon fibre/epoxy structure on which a 0.75mm malleable copper sheet was bonded. Several surface heaters were fixed to the copper sheet and provided power with a density of 0.24W/cm^2 . The IHCT was capable of a 5°C/min heating rate (Figure 2.4). For comparison they heated the same PMC laminate (6.5mm) in an oven; 40min were required for the PMC surface to reach a dwell temperature of 80°C , resulting in an average heating rate of 1.4°C/min , much lower than what is achievable with IHCTs. The main issue with convection is that the heat flux is proportional to the temperature gradient between the heat transfer fluid and the tooling; this is not the case with conductive heating. Moreover, the IHCT ensured good temperature uniformity, displaying a temperature variation of 3°C across its surface at a dwell temperature of 100°C . However, the authors noted that temperature variation can vary significantly depending on the heater arrangement, up to a difference of 20°C .

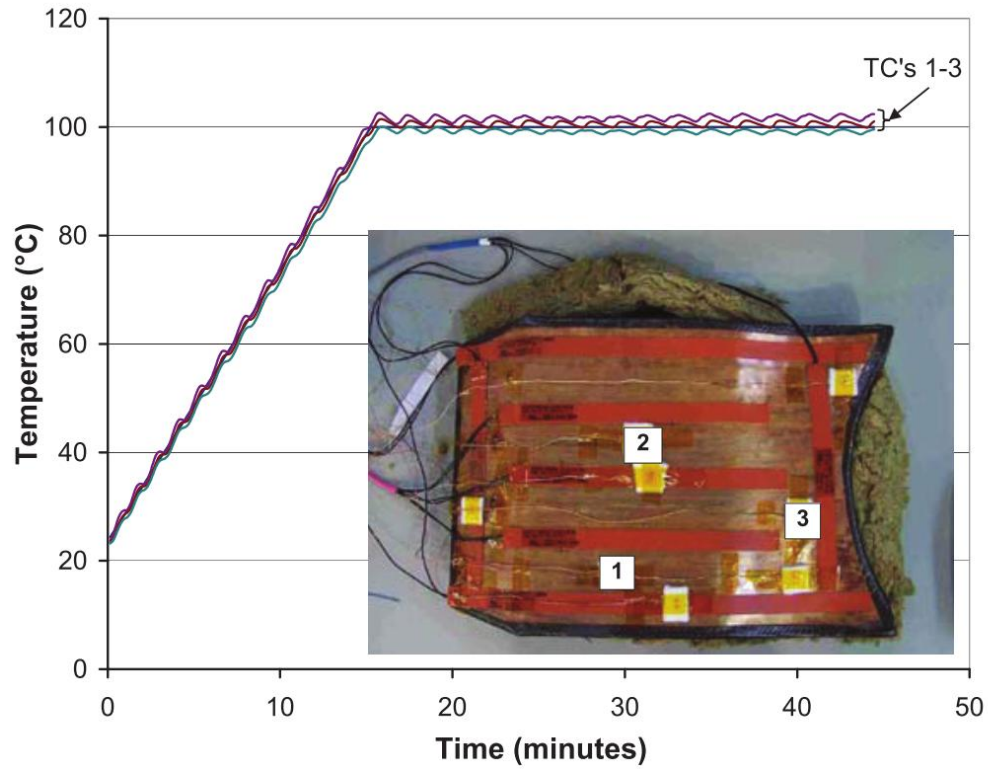


Figure 2.3: Temperature distribution for the inner surface of a backseat IHCT [12]

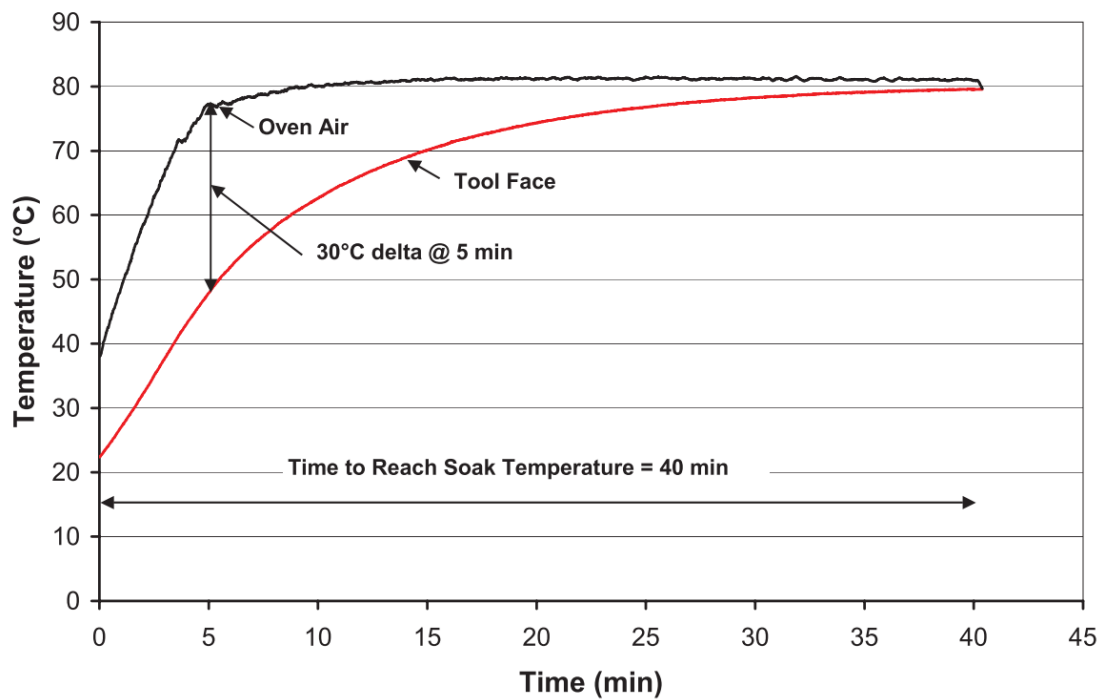


Figure 2.4: Temperature distribution for the inner surface of a PMC tool [12]

From the results of Raizenne et al. above [4] , it can be seen that IHCTs offer an attractive solution for out-of-autoclave PMC manufacturing because they can match the coefficient of thermal expansion of PMC parts. IHCTs are also much lighter than metallic tools, which is a desirable feature for easier manipulation, faster heating rates, lower energy consumption and smaller eggcrate substructure requirements. IHCTs can also provide uniform surface temperatures. Furthermore, the PMC structures of IHCTs can be formed to net shape easily, without machining. However, IHCTs do have a lower durability than metals but this can be addressed by applying a thin Invar lining on the PMC tool surface [48].

The specific characteristics of IHCTs sparked interest at the University of Ottawa regarding parameters affecting heat transfer in IHCTs. This is discussed in section 2.3 [23–26] and Chapter 6 of this work. In addition to heat transfer simulation, this work focused on replacing the thermally conductive layer in IHCTs with a sprayed coating. Although this constitutes a more expansive solution, a metallic coating could be used in tool areas of complex curvatures and tight radii which would require extensive forming for a bonded sheet otherwise. This would be especially beneficial for multi-curvature IHCTs that are manufactured in small series. Additionally, coatings could potentially be used for repairing very valuable IHCTs. The coating material selected was copper because it was the sheet material used in recent IHCTs and it has a high thermal conductivity. Coating methods for depositing such copper layers will be discussed in the following section.

2.2 Metallic coatings

This section begins with an introduction of spraying processes that can be used for forming metallic coatings. Then, properties of copper coatings made with these processes are analysed. Finally, the possibility of coating PMCs using these processes is explored.

2.2.1 Processes

Thermal spray processes are used for applying metallic coatings onto various materials. Even though several distinct processes exist (Figure 2.5), they all achieve coating by consolidating discrete amounts of metallic particles, molten or not, together on the surface of a substrate. Based on operational temperatures, thermal spray processes can be subdivided into two main groups: conventional (hot) processes, thereafter named thermal spray processes, and kinetic (cold) spray processes. The latter group will be presented more thoroughly given that it is one of the main aspects of the thesis.

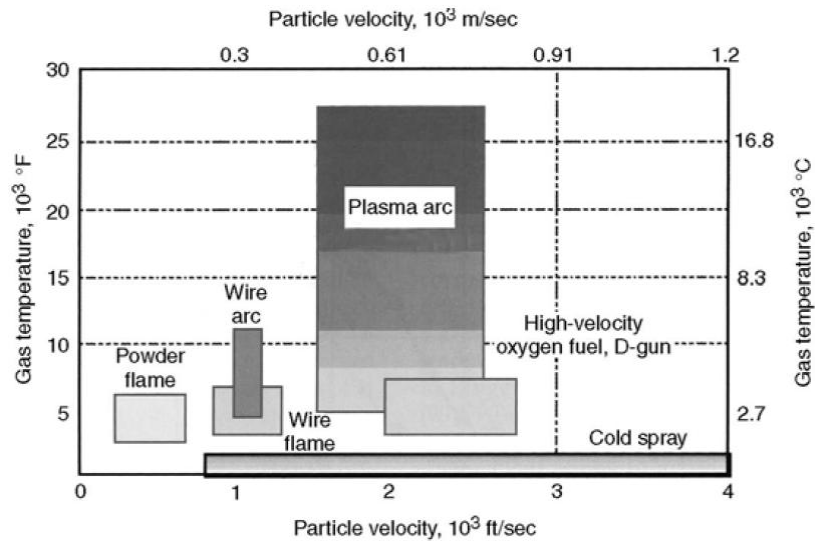


Figure 2.5: Temperature and velocity regimes for several thermal spray processes [57]

2.2.1.1 Conventional thermal spray processes

Thermal spray processes, or hot spray processes, involve spraying metallic droplets on the surface of a substrate. Upon contact with the substrate the droplets are cooled and solidify to form a coating. Bonding is partially attributed to true metallurgical bonding that arises from atomic diffusion induced by the hot impinging droplets. However, bonding is also due to mechanical interlocking of the metallic droplets with a rough surface, as well as to weak physical bonding from van der Waals forces between solidified particles [57]. A concern with these thermal spray processes is that temperatures involved tend to oxidise the surface of the droplets which reduces metallic bonding. Moreover, the high temperatures involved generate tensile residual stresses from contraction of the particles during solidification and phase transformation from the initial feedstock material [58–60].

Thermal spray processes are differentiated by the way they melt the metal and accelerate the resulting droplets. The main spray processes are: 1) flame [15, 57, 61, 62], 2) wire arc [16, 17, 57, 60, 61, 63], 3) plasma [16, 19–21, 57, 64] and 4) high-velocity oxygen fuel (HVOF) [14, 15, 57, 60–63].

Flame spray commonly uses oxyacetylene torches. In this process, a powder, wire or rod feedstock metal is fed into the flame and is molten into droplets. The droplets are then propelled towards a substrate using the expanding torch gasses.

Wire arc spray uses two consumable wire electrodes that are connected to a high-current DC voltage source. Proximity between the two electrodes produces an electric arc that melts their tips. The molten metal is then atomised by a stream of compressed air or inert gas and it is propelled towards a substrate. Unlike other thermal spray processes, the carrier gas is not heated directly, making this a desirable process for temperature sensitive substrates.

Plasma spray uses a plasma for melting feedstock powder. In this process, a DC electrical arc between a pair of electrodes ionises an inert gas (e.g. argon) to a plasma state.

Gas temperatures can range from 6000°C to 15000°C at the powder injection region, which is significantly higher than the melting temperature of known materials [57].

HVOF is similar to flame spray but features much higher gas flow velocities. The process mixes high volumes of gas and fuel (e.g. hydrogen, propane, acetylene) in a pressurised combustion chamber. The combustion results in supersonic gas flow in which discrete amounts of powder are injected. The powder can either be molten or partially molten depending on process parameters. In the latter case, coating deposition is possible due to the great kinetic energy, similar to kinetic spray processes presented next.

2.2.1.2 Kinetic spray processes

Kinetic spray processes, unlike thermal spray processes, rely on kinetic energy for producing metallic coatings. Due to the absence of particle melting these solid-state processes have the advantages of retaining the chemical properties (i.e. absence of oxidation) [59, 65–67] and some microstructural properties [68–70] of the powders being sprayed. The two main processes that will be analysed in this thesis are the Cold Gas-Dynamic Spray (CGDS) and Pulsed Gas-Dynamic Spray (PGDS) processes. Before going into specific details about individual spray process, a general analysis of coating deposition using kinetic energy is presented.

In kinetic spray processes, coating deposition is achieved by accelerating small particles to very high velocities. In the case of CGDS and PGDS this is done by injecting particles in a supersonic gas flow. Upon impact with a surface, the kinetic energy of the particles is converted into highly localised pressures that yield intense plastic deformation and interfacial heat, enabling bonding to the substrate. During the impact, particles flatten to conform to the surface of the substrate. It is widely accepted that bonding essentially results from shear instabilities between the substrate and impinging particles interfaces, which creates mechanical interlocking [68, 71–73]. The high deformation rates that are induced by an impact produce adiabatic heating at the interfaces which can yield temperatures near the melting point [68, 72–74]. Consequently, at the interfaces the effect of particle softening can surpass particle hardening resulting in material flow. Assadi et al.

estimated from strain-stress simulations that this behaviour is localised in a 2 μm thick layer originating from the impact interface [73]. Physically, shear instabilities can produce vortex-like mixing of the substrate and particle materials as shown by Ajdelsztajn et al. for nickel particles deposited on aluminium (Figure 2.6)[71]. This increases interfacial strength due to large contact surfaces. Another sign of shear instability takes the form of material jets that emanate from the particle-to-substrate interface (Figure 2.7) [68, 74–79]. Moreover, deformation involved during impacts tends to elongate and flatten the grains inside sprayed particles. The effect is more prominent near particle interfaces where there may even be some recrystallisation [75, 78]. King et al. showed that grain refinement of copper along the interface can be in the sub 100nm range [75].

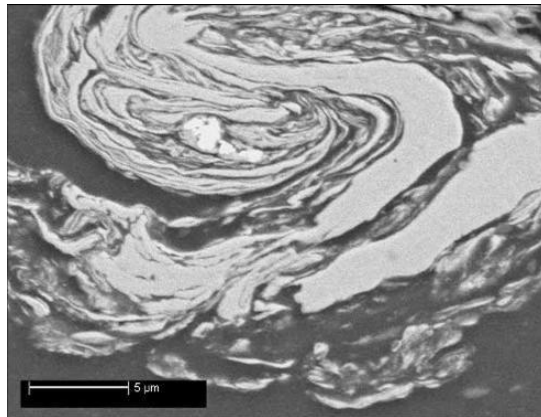


Figure 2.6: Image of interfacial instabilities resulting from sprayed nickel powder on aluminium using CGDS [71]

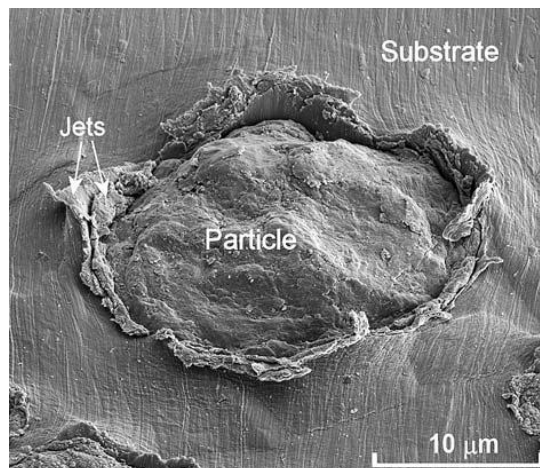


Figure 2.7: Copper particle sprayed onto a copper substrate using CGDS [75]

Despite the high interfacial temperatures reached during impacts, metallic bonding between the sprayed particles and the substrate is limited due to low amounts of atomic diffusion [72, 73, 79]. Grujicic et al. used simulations to show that the plastic strain rate and the peak of temperature caused by a particle impacting a substrate lasted approximately 20ns for copper and aluminium particles travelling at 400m/s to 1000m/s [79]. This was found to be faster than the inter-atomic diffusion. Hence, metallic bonding is not dominant in kinetic spraying. Nevertheless, Kuivuluoto et al. produced copper coatings using CGDS and observed metallic bonding using fractography that revealed ductile fracture [76]. However, scant occurrence of metallic bonding in kinetic spray coatings results in generally weaker mechanical properties than those of the bulk material [78], and fracture is often characterised by inter-particle failure [80]. This can be addressed by heat treatments that increase metallic bonding and yield coating properties similar to those of the bulk [60, 81].

Kinetic spray coatings retain residual stresses similarly to thermal spray coatings. However, particle impinging generates compressive surface stresses similar to shot peening resulting in surface stress values ranging from 30MPa to 60MPa [58, 63, 81]. The peening effect can be beneficial for coating consolidation. It was suggested from the greater surface porosity of kinetic spray coatings that particle impacts from following spray passes induce further deformation of the already deposited coating layers, increasing bonding and reducing defects [58, 71].

Coating deposition and bonding strength greatly depend on the amount of kinetic energy that can be imparted to sprayed particles [74]. It was proposed that a minimum particle velocity is required for deposition, and that greater velocities improve adhesion [73, 78, 79]. Predicting a minimum deposition velocity is complex as this is affected by various parameters including 1) spraying gas velocity, 2) processing temperatures, 3) spraying powder composition and morphology, and 4) substrate composition and roughness [63, 73, 79, 82].

Improvements to coating bond strength can be achieved by directly increasing the kinetic energy of the sprayed particles [74]. The most basic solution is to inject the particles

in a higher velocity gas flow [79, 80, 83]. Another solution is to lengthen the flight duration of the particles so that they can reach velocities closer to that of the gas flow. For example, Champagne et al. calculated that copper particles having a mean diameter of $19\mu\text{m}$ in a helium gas flow will be subjected to an increase in exit velocity from approximately 500m/s to 600m/s by lengthening the nozzle from 10cm to 20cm [73].

Ductility of the spray particles also affects deposition efficiency. Harder particles undergo less deformation, increasing coating porosity [69]. From this it was shown that particle softening from pre-heating helped increase deposition due to the lower energy requirement towards plastic deformation [65, 73].

Characteristics of the feedstock powder also play an important role in coating deposition and properties. The effect of particle shape was investigated by Jodoin et al. through measurement of spray particle velocity for both spherical and irregular shape aluminium alloy powders [69]. The higher drag coefficient of the latter particles enabled a velocity increase of 6% to 11% compared to spherical shape particles.

Moreover, spray particle size has a strong impact on deposition efficiency and bond strength [59, 67, 69, 73, 75, 83, 84]. Bigger particles when accelerated have more momentum than smaller particles, providing greater amount of kinetic energy for deformation. However, above a certain particle size of $25\mu\text{m}$ King et al. observed that inertia limited the acceleration of particles, lowering particle velocity compared to that of the gas flow [75, 77]. For example, Champagne et al. estimated that the velocity at the exit of a 9cm nozzle for a copper particle in a helium gas flow reduced from 500m/s to 100m/s when particle diameter increased from $10\mu\text{m}$ to $40\mu\text{m}$ [73]. Experimentally, Raletz et al. observed that the mean particle size in their CGDS coatings was $6.5\mu\text{m}$ as opposed to $16.2\mu\text{m}$ in their feedstock powder, implying that bigger particles did not adhere [83]. Conversely, smaller particles can be accelerated to higher velocities [71] but their low mass limits the amount of kinetic energy and subsequent deformation [77]. In addition, a bow shock is produced when a supersonic gas flow reaches a surface. This bow shock decelerates the gas to stagnation conditions. Bigger and denser particles can cross that bow

shock and retain velocity sufficient for deformation, but smaller and lighter particles are decelerated to velocities that limits plastic deformation and prevents bonding [72, 77].

Regarding the effect of the substrate, Stoltenhoff et al. showed a significant influence of the substrate material on the coating bond strength [63]. The authors measured the bond strength of a copper coating on a steel substrate at 10MPa while it increased to 40MPa on aluminium. The difference is likely due to the hardness of the steel which limited deformation for adequate mechanical interlocking. In addition, roughness is known to have an impact on the coating adhesion strength [74]. Kumar et al. showed experimentally that grit blasted substrates improved aluminium and copper deposition efficiency and increased coating bond strength [74]. They also investigated this effect using simulations of a particle impacting surfaces that are smooth, or roughened by a crest or trough. Particles impacting at the centre of the roughened features revealed that slight roughness improved plastic deformation due to an increase in impact contact time, contact area and interface temperature. Optimal roughness was found to be equivalent to spray particle diameter. They also noted that large roughness features showed similar behaviour to that of a flat surface, which was to be expected due to the greater radius of curvature. From these results, it would be interesting to observe the effect of non-centred particle impacts on crests and troughs.

Further discussion relating to kinetic spray processing parameters is featured in the following CGDS and PGDS sections.

2.2.1.3 Cold gas-dynamic spray process

The kinetic spray coating phenomenon was discovered in the 1980s at the Institute of Theoretical and Applied Mechanics of the Siberian Branch of the Russian Academy of Sciences (ITAM of RAS) while studying supersonic two-phase flow around objects [85]. Research showed that an unheated supersonic gas flow (400-450m/s), that included fine aluminium particles (1-40 μ m), around a steel cylinder target lead to the deposition of a continuous coating. Further research at the institute resulted in the first United States patent for a cold gas-dynamic spray (CGDS) system assigned to Papyrin in 1994 [86].

CGDS uses a converging-diverging nozzle (de Laval nozzle) to accelerate a steady state gas flow to supersonic velocity (500m/s to 1200m/s) in which powder is injected [68, 78] (Figure 2.8). The converging section expands the gas so that it reaches sonic velocity at the throat. The subsequent diverging section further expands the gas to greater velocity. Gas expansion converts enthalpy into kinetic energy. In other words, the increase in gas velocity is followed by a decrease in gas pressure and temperature. Consequently, a lower gas temperature cools the sprayed particles in flight [73]. This is inconvenient because it prevents particle heating and particle softening which would be desirable for reducing energy requirement for plastic deformation.

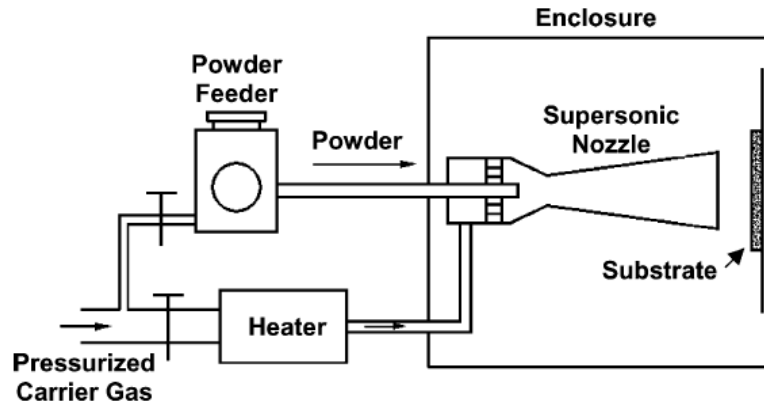


Figure 2.8: Schematic of a typical CGDS system [68]

Two different configurations of CGDS exist: high-pressure CGDS (HP-CGDS) and low pressure (LP-CGDS) [82]. The most important distinction between the two is the powder injection point. For HP-CGDS, the powder is fed in the high pressure zone before the nozzle while in LP-CGDS it is fed in the low pressure zone after the throat of the nozzle. In this thesis work, LP-CGDS was used.

For CGDS, gas velocity in a converging-diverging nozzle is strongly affected by the nozzle profile. A properly designed nozzle should achieve sonic gas velocity at the throat and the pressure at the exit of the nozzle should be equal to the outside pressure. For an isentropic (reversible and adiabatic) process, the theoretical Mach number M_e of the gas in such nozzle is obtained by the ratio of the stagnation pressure P_o and outside pressure P_e [87]:

$$M_e = \sqrt{\frac{2}{\gamma-1} \left[\left(\frac{P_o}{P_e} \right)^{\frac{\gamma-1}{\gamma}} - 1 \right]} \quad (2.6)$$

where γ is the specific heat ratio. The actual velocity is obtained by multiplying the Mach number by the speed of sound c :

$$u_e = M_e c = M_e \sqrt{\frac{\gamma \bar{R} T}{M}} \quad (2.7)$$

where $\bar{R}$ is the universal gas content, T is the temperature and M is the molar mass of the gas. In reality these optimal velocities are not reached because of many influential parameters, and great work is still needed for nozzle optimisation [59, 71, 73, 84, 88]. Nevertheless, it follows from Equations (2.6) and (2.7) that the pressure difference, gas temperature and type of gas can affect velocity.

The effect of gas pressure was the subject of many studies [58, 59, 65, 67, 70, 73, 80]. The conclusions are that increasing the stagnation pressure is beneficial for increasing deposition efficiency, lowering defects and improving coating adhesion. This is attributable to the increase in enthalpy that can be converted in additional kinetic energy. Similarly, pre-heating the gas increases enthalpy. Studies have shown that it is one of the most influential parameters for good coating adhesion [59, 67, 70, 73].

The type of gas also influences gas velocity [59, 60, 73]. From computational estimations, Champagne et al. calculated that 20 μ m particle velocity could be increased from 500m/s to 890m/s by using the lower molecular mass of helium ($M = 4\text{kg/mol}$) compared to nitrogen ($M = 28\text{kg/mol}$) [73].

Another factor that was investigated was the standoff distance which is the distance between the nozzle and the substrate. Stoltenhoff et al. suggested from simulations that a standoff distance between 10mm and 80mm has negligible impact on the sprayed particles [59]. Experimentally, Tushinsky et al. showed that with their CGDS system, reducing the standoff distance from 40mm to 5mm marginally improved the adhesion and cohesion of

an aluminium coating [58] whereas Phani et al. noted that a standoff distance between 5mm and 25mm offered the lowest porosity [66].

2.2.1.4 Pulsed gas-dynamic spray process

PGDS is a kinetic spray process that was developed at the University of Ottawa by Jodoin and patented in 2006 (Patent no. CA 2607550) [89]. Similarly to CGDS, PGDS forms coatings by consolidating high velocity particles onto a surface using impact energy. However, PGDS is characterised by its unsteady gas flow that accelerates the particles. This unsteady flow is produced by a series of shock-waves that are generated at a rate of 10Hz to 30Hz, making it a semi-continuous spraying process. The process was used successfully for producing aluminium, copper, zinc, amorphous iron, nanocrystalline material and metal-ceramic coatings [22, 90–96].

The operation of PGDS is reminiscent of a shock tube. It consists in having a tube filled with two gases, a high and a low pressure gas, which are separated by a valve. Upon valve opening the great pressure difference between the gases generates a shock-wave that propagates into the low pressure gas, accelerating and heating it in the process. Theoretical gas velocities from 1D-gas dynamic equations and from computational fluid dynamics range between 300m/s to more than 1700m/s depending on the gas type, gas pressure and gas temperature [92, 93]. Experimentally this translated to a gas velocity of 1095m/s when using pressurised helium at 3MPa and 100°C to accelerate air at ambient pressure and temperature. The gas velocity was 65% of the theoretical value due to the slow valve motion that impeded shock-wave generation [93]. The major advantage that PGDS has over CGDS is that the shock-wave heats the gas flow which can increase ductility of sprayed particles, enabling deposition at lower velocities. This feature was useful in enabling deposition of hard amorphous iron on an aluminium substrate, which was not possible using CGDS [93].

Another interesting feature of PGDS is that particles travel in clusters due to the unsteady nature of the gas flow (Figure 2.9). These clusters can trap particles that would

otherwise bounce off the substrate. This enabled Yandouzi et al. to deposit metal-ceramic composite coatings with a higher ceramic content than with CGDS [91].

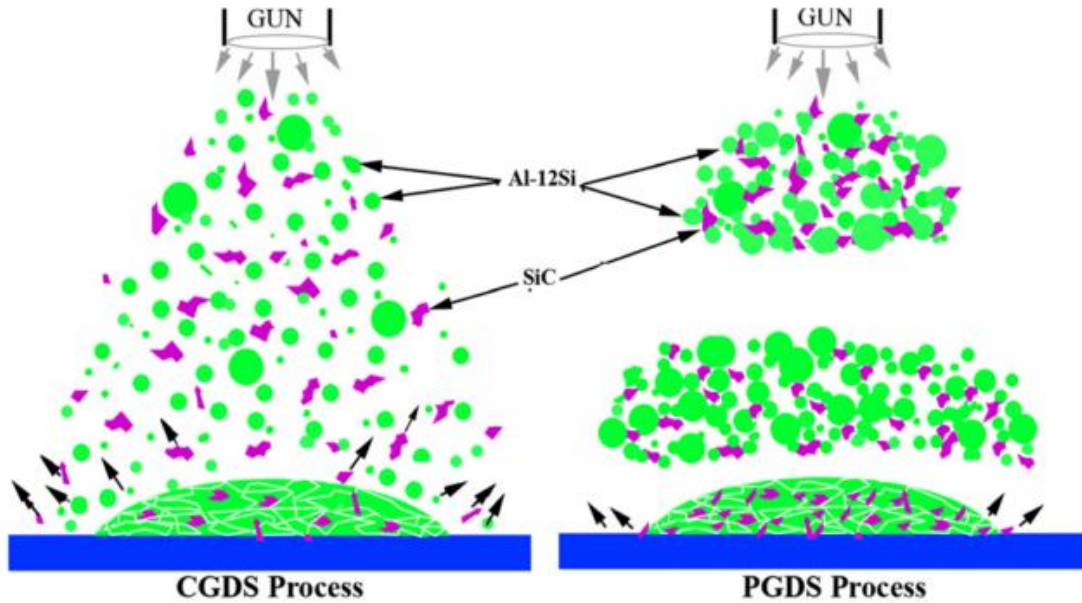


Figure 2.9: Schematic illustrating continuous and pulsed deposition for CGDS and PGDS respectively [91]

The cluster aggregation of sprayed particles also enabled Assaad et al. to deposit a coating on a wire mesh, something that was not possible using CGDS [96]. Success was attributed to high particle density in the sprayed clusters that clogged the gaps between the wires of the mesh. Filling all the gaps offered a relatively flat surface to deposit a coating.

Despite all its advantages in comparison to CGDS, PGDS requires greater control over powder injection in the gas. The unsteady nature of the gas flow results in regions having different gas properties. From unsteady gas-dynamics [87], 5 gas regions are created in a shock tube when the valve between the high and low pressure gases opens (Figure 2.10). Regions 1 and 5 represent the low pressure and high gases at rest, respectively. As the shock-wave propagates in the low pressure gas, it increases gas velocity and temperature (region 2). Concurrently with the shock-wave propagation, an expansion fan (region 4) travels into the high pressure gas to set it into motion, filling the

void left by the gas leaving the tube. This expands the high pressure gas, increasing velocity and reducing temperature (region 3). This region is undesired due to the lower temperatures that reduces particle ductility, as in CGDS. From this it can be seen that there is only a small timeframe, in the form of gas region 2, where it is desirable to accelerate particles. The actual feasibility of PGDS lies with injecting discrete amounts of powder in the tube ahead of the shock-wave for ensuring that particles are all accelerated in gas region 2. After particle acceleration, the valve is closed and the system is returned to its initial state of rest. Particles are then re-injected in the tube and the valve is re-opened to repeat the process.

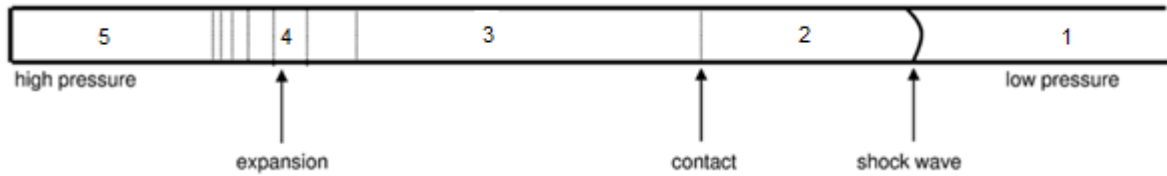


Figure 2.10: One dimensional shock tube model for an unsteady gas flow, adapted from [92]

From unsteady gas-dynamics, the gas velocity in regions 2 and 3 is dependent on the initial pressure ratio between the gases, gas type and speed of sound. The mach number of the shock-wave has to be solved from the implicit function [87]:

$$\frac{P_5}{P_1} = \frac{2\gamma_1 M_s^2 - (\gamma_1 - 1)}{\gamma_1 + 1} \left[1 - \frac{\gamma_5 - 1}{\gamma_1 + 1} \frac{c_1}{c_5} \left(M_s - \frac{1}{M_s} \right) \right]^{\frac{2\gamma_5}{\gamma_5 - 1}} \quad (2.8)$$

where P represents the gas pressure, γ is the heat capacity ratio, c is the speed of sound and M_s is the Mach number of the shock. The Mach number for the shock can be used for calculating the gas velocity u_2 in regions 2 and 3 [87]:

$$u_2 = \frac{2}{\gamma_1 + 1} c_1 \left(M_s - \frac{1}{M_s} \right) \quad (2.9)$$

Following the velocity equation above, it can be seen that deposition efficiency and bond strength of PGDS particles depend on similar gas parameters than in CGDS. Experimentally, Yandouzi et al. showed that increasing the stagnation gas pressure and temperature results in greater spray particle velocities [94], which follows the same trend seen with CGDS.

2.2.2 Copper coatings

Copper coatings were deposited using both thermal and kinetic spray processes [60, 63, 65–67, 70, 73, 75, 78, 81, 82, 84, 88, 93] and their properties are affected by processing parameters as presented in section 2.2.1. A summary of the mechanical and electrical properties found in literature for such coatings is presented in Table 2.1.

For the mechanical properties, Gärtner et al. compared the tensile behaviour of coatings made with wire arc, HVOF and CGDS (nitrogen and helium) [60]. For comparison, samples were cut in the shape of miniature tensile test specimens. Results showed that all as-sprayed coatings had low deformation at rupture. Thermal spray coatings were characterised by brittle failure and their ultimate tensile strength was weaker (93-151MPa) than the bulk (230MPa) due to the presence of oxides at particle-to-particle interfaces. CGDS coatings processed with nitrogen (3MPa and 305°C) resulted in brittle failure and showed the weakest resistance in tension (26-46MPa) due to interfacial defects. Conversely, higher spray velocities in CGDS using helium (2.5MPa and 300°C) produced a coating of highest ultimate tensile strength (453-472MPa), similar to highly cold worked sheets of copper (Table 2.1). In the case of CGDS with helium, intense impact pressures produced intimate contact between particles, limiting interfacial defects. Hence, kinetic spray processes can produce stronger as-sprayed coatings than thermal spray processes given sufficient particle velocities, and due to the absence of oxidation.

Gärtner et al. annealed the same coatings at various temperatures and analysed change in mechanical behaviour (Figure 2.11) [60]. For thermal spray processes (wire arc and HVOF), annealing resulted in rearrangement of oxides. This lowered slightly the ultimate tensile strength of the coatings and had negligible effect on the elongation at

failure. Conversely, annealing of the CGDS coatings permitted atomic diffusion that closed some particle-to-particle interfaces and allowed recrystallisation, made possible by the absence of oxides that would otherwise pin the grain boundaries. This increased cohesion strength in coatings processed with nitrogen and increased ductility significantly in coatings made using helium. In the latter case, the coatings showed strength comparable to annealed bulk copper and elongation at failure increased from 2% up to 24% by annealing for 1h at 600°C. From this, it is clear that kinetic spraying offers the most potential for producing copper coatings having properties similar to those of the bulk copper.

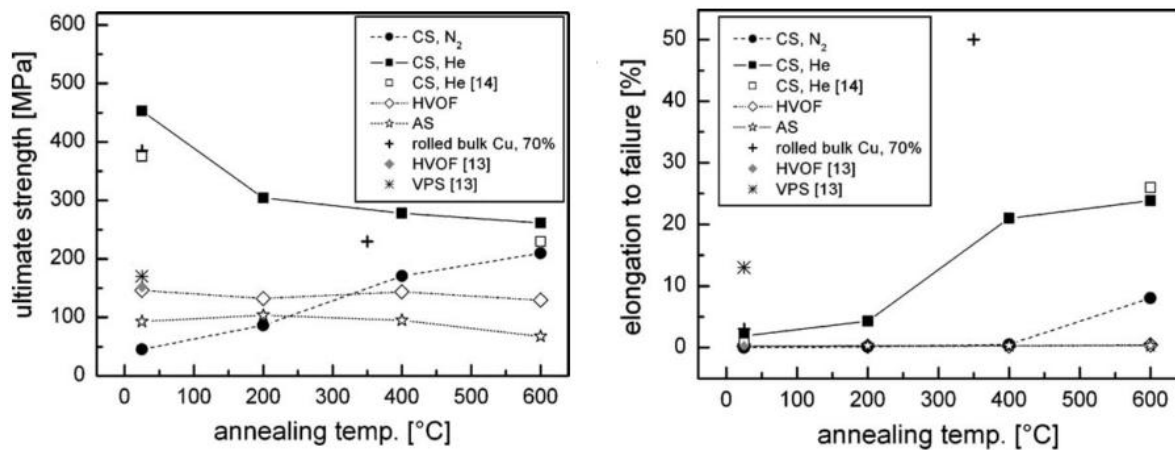


Figure 2.11: Effect of annealing on the tensile strength (left) and elongation (right) at failure of copper produced using various spraying processes [60]

Thermal conductivity is vital for temperature uniformity in IHCTs. Unfortunately, limited data about the thermal conductivity for kinetic spray coatings exist in the literature [97]. Seo et al. measured the through-thickness thermal conductivity of copper coatings made under various CGDS processing conditions and one thermal spray process, High Velocity Air Fuel (HVOF) which is similar to HVOF [97]. As-sprayed conditions resulted in thermal conductivities varying between 120W/m·K to 210W/m·K for CGDS and 290W/m·K for HVOF. All values fall short of the 401W/m·K value for bulk copper [47]. For CGDS, optimal spraying conditions were the use of helium pressurised at 0.6MPa without any gas heating. Annealing was performed for improving thermal conductivity. Similar to the mechanical properties, annealing did not have an effect on the thermal spray

coating. Annealing CGDS coatings above 200°C improved the thermal conductivity to a range between 300W/m·K to 340W/m·K. Thus, it can be concluded that annealing would be desirable for an IHCT application.

Additional information regarding thermal conductivity behaviour can be accessed by analysing the electrical conductivity for which greater interest was shown [63, 66, 88]. Both conductivities can be related by the Widemann-Franz law [49]. This is attributable to metallic bonding in copper in which electrons are responsible for the electrical conductivity and most of the thermal conductivity. The relationship tells that:

$$k = \sigma_{\text{electrical}} L T \quad (2.10)$$

where k is the thermal conductivity, $\sigma_{\text{electrical}}$ is the electrical conductivity, L is the Lorenz number ($\sim 2.44 \times 10^{-8} \text{ W}\Omega/\text{K}^2$) and T is the temperature. From this equation, any electrical conductivity improvement seen in coatings should also be reflected to thermal conductivity.

Stoltenhoff et al. compared the electrical conductivity of coatings produced using CGDS, HVOF and wire arc spray processes and analysed the effect of annealing [63]. Results appear in Figure 2.12. As-sprayed conductivity values were 63%, 39% and 19% of bulk copper conductivity for CGDS, HVOF and wire arc respectively. Annealing at various temperatures increased the overall electrical conductivity. Improvements were caused by diffusion at particle-to-particle interfaces and recrystallisation. The highest electrical conductivity values were obtained after annealing at 600°C for 1h and these values were 92%, 62% and 41% of bulk copper conductivity for CGDS, HVOF and wire arc respectively. These results are similar to the trends presented above for thermal conductivity, where kinetic spray coatings offered the best conductivity.

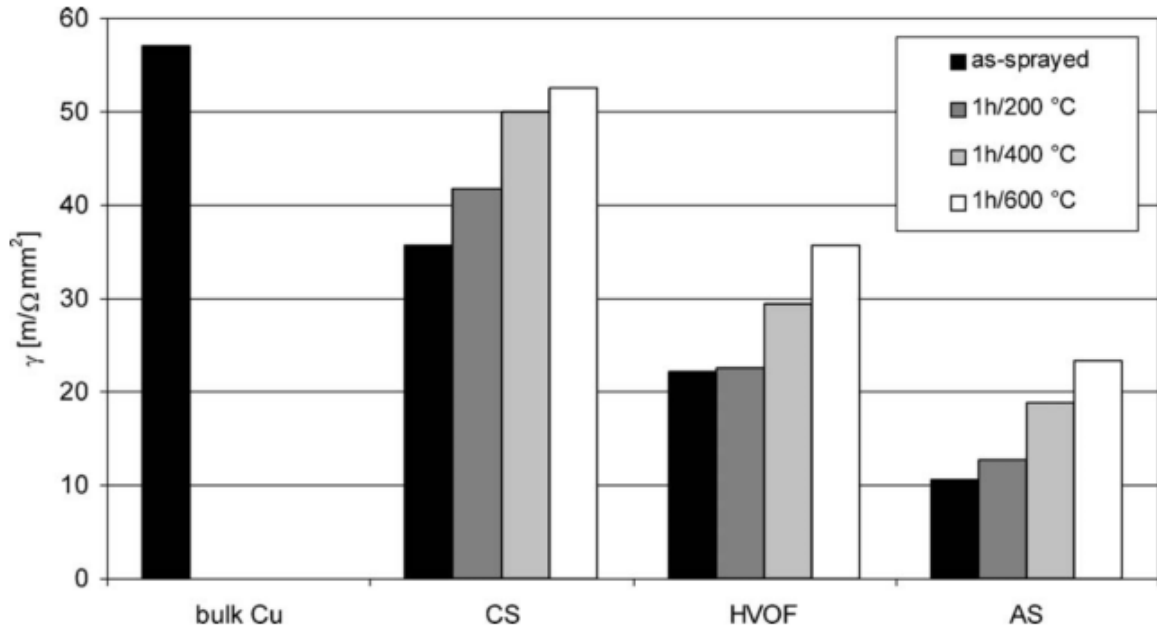


Figure 2.12: Electrical conductivity of kinetic spray (CS), HVOF and wire arc spray (AS) copper coatings, as-sprayed and annealed [63]

Phani et al. observed similar results for electrical conductivity of a CGDS copper coating [66]. As-sprayed and annealed values were 40% and 90% of the electrical conductivity of bulk copper, respectively. It was conjectured that the lower values compared to bulk copper were caused by strain hardening of the particles and interfacial defects between coating particles. Çetinarslan showed that in bulk copper, cold work of 50% lowered electrical conductivity by 2% due to lattice distortion [98]. Furthermore, Williams observed a reduction of 11.5% in thermal conductivity of copper due to 50% cold work [99]. From these, it can be concluded that interfacial defects are likely the dominant factor in the lower conductivity of kinetic spray copper coatings.

From results presented in the literature it can be concluded that despite the interfacial defects, kinetic spray coatings offer copper properties that are the most bulk-like. This is a desirable asset for producing IHCTs. In the following section we will investigate the potential of the spraying processes for coating PMCs.

Table 2.1: Mechanical and electrical properties of copper produced by various techniques

Property	Bulk annealed	Bulk cold work	Plasma	CGDS	Wire arc	HVOF
Yield strength [MPa]	63 [60]	378 [60]		N/A [60]	N/A [60]	N/A [60]
Ultimate tensile strength [MPa]	230 [60]	390 [60]		N ₂ : 26-46 [60] He: 453-472 [60]	93-102 [60]	140-151 [60]
Elongation at failure [%]	44 [60]	2.6 [60]		N ₂ : <0.1 [60] He: ~2 [60]	0.3 [60]	0.23 [60]
Compression strength [MPa]				60-120 [81]		
Elastic modulus [GPa]	126 [81]		25-37 [81]	108 [81]		
Hardness (Vickers)	83 HV [99]	112 HV [99] 125 HV _{0.3} [88]		130-150 HV [88] 140-160 HV _{0.1} [59] 100-135 HV _{0.1} [66] 140 HV _{0.1} [100] 80-150 HV _{0.3} [60] 100-175 HV _{0.3} [97] 150 HV _{0.3} [63]	90-100 HV [88] 90 HV _{0.3} [63]	125 HV _{0.3} [63]
Hardness (Knoop)	140-160 [81]			140-160 [81]		

Table 2.1: (Cont.): Mechanical and electrical properties of copper produced by various techniques

Property	Bulk annealed	Bulk cold work	Plasma	CGDS	Wire arc	HVOF
Bond strength [MPa]	N/A	N/A		35 [80]	10 [88]	28 [63]
				35-40 [59]		
				40-80 [74]		
				48-52 [58]		
				30-40 [78]		
				10-20 [88]		
				20-55 [70]		
Electrical resistivity [$\mu\Omega\cdot\text{cm}$]	1.7 [81]		5.45 [81]	2.4 [81]	6.35 [81]	
				1.9 [59]		
Electrical conductivity (%IACS)	100%	94%[98]		63% [63]	20% [88]	39% [63]
				60% [88]		
				40% [66]		
Thermal conductivity [W/m·K]	401 [47]	[66]2 [99]		120-210 [97]		
	386 [99]					

2.2.3 Coatings for composites

The potential of spray processes for coating PMCs was studied and yielded various degrees of success [14–22, 61, 62, 64, 100–103]. Research was mostly aimed towards improving wear resistance and thermal protection of PMCs in gas turbines [14–17, 61, 62, 64, 101]. This section begins with the assessment of conventional thermal spray coatings on PMCs and it is followed by the assessment of kinetic spray processes.

Thermal spray processes were used for depositing metallic coatings on PMCs [14, 16, 17, 19]. However, PMCs offer some practical difficulties that were not present with metallic substrates. First difficulty, surface preparation is vital for coating adherence on PMCs as, for structural applications, PMCs are often composed of thermosetting polymers. Thermosets are brittle and cannot undergo plastic deformation which is required for the intricate mechanical interlocking resulting from shear instabilities as seen with metals in section 2.2.1.2. Hence, for PMCs, grit blasting was used for increasing the surface area and promoting interlocking between substrates and coatings [14–17, 21, 61, 64]. It was shown that grit blasting is more effective than sand paper and that both methods induce PMC damage such as fibre breakage [15, 17]. Etching methods were also investigated for increasing adhesion [20, 21].

Second difficulty, thermal spray processes use molten metals for forming coatings on substrates. The melting temperature of metals is generally much higher than the melting or degradation temperature of polymers. Hence, thermal spray processes are mostly used with high temperature resistant polymers such as polyimide [15–17, 21, 61, 62] and PEEK [20]. Some work showed the possibility of applying coatings on epoxy-based PMCs, but no information was divulged regarding the coating material [64, 101].

Liu et al. used wire arc and plasma processes to spray aluminium, zinc, copper and nickel alloy (Ni3Al) on a carbon fibre/polyimide PMC [16]. Spraying the copper and nickel alloy, i.e., metals with high melting temperatures, resulted in the deposition of coatings that did not adhere to the substrate. Significant PMC damage was observed in the form of polyimide oxidation. Despite the temperatures involved, Nagai et al. managed to deposit

nickel-based copper coatings on PMCs using plasma spray, and also using other types of coatings [19]. It was noted that the difference in thermal expansion coefficients between the PMC and coating plays an important role in coating delamination. It can thus be concluded that the high melting temperature of copper and its great coefficient of thermal expansion ($17.0 \times 10^{-6} \text{ m/m}\cdot\text{K}$) prevents the use of thermal spray processes for spraying copper coatings directly on PMCs, even more so on PMCs with resins characterised by lower degradation temperatures such as epoxy.

A solution for producing copper coatings on PMCs would consist in first applying a bridging coating layer, i.e. bond coat, to protect against the high temperatures of molten copper. This technique was often used for applying hard metal-ceramic coatings on PMCs [16, 62]. In addition to protecting the PMC from erosion or high temperatures of a top coating, the bond coat also aims at bridging the thermal expansion coefficients of the substrate and top coating, reducing the risk of thermal shock during the thermal spray process due to the high processing temperatures [16].

Liu et al. considered aluminium and zinc for bond coats [16]. Coatings were produced by wire arc and plasma spray processes on polyimide PMC and gave shear strengths ranging from 7MPa to 12MPa. The lower melting temperatures of these metals prevented thermal damage. Moreover, Dixit et al. managed to spray a nickel bond coat on Avimid N (polyimide) using HVOF [14]. They found that the greater kinetic energy imparted by the process enabled the nickel to infiltrate into the PMC, ensuring interlocking. Compressed air was continuously blown on the substrate to prevent excessive thermal damage. Hence, this showed potential for kinetic spraying.

Polymer-based bond coats may also be used for applying subsequent metal-ceramic coatings [19, 64, 101], but often offer poorer performance against erosion compared to a metallic bond coat [61, 64, 101]. Strait and Jamison used plasma spray for depositing PEEK on carbon fibre/PEEK PMC [20]. Results showed that pre-heating the substrate helped adhesion significantly (15MPa to 23MPa). It was conjectured that the pre-heating reduced moisture and thermal stresses, while it bettered bonding conditions. A similar effect was not observed with metallic coatings [16]. Ivocevic et al. worked on the

development of the functionally graded WC-Co coating of a carbon fibre/polyimide PMC [15, 61, 62]. The idea consisted in spraying a first layer of pure polyimide using HVOF and adding subsequent layers with ever increasing WC-Co content until the top coat was 100% WC-Co. The authors observed that failure in the functionally graded coating resulted mostly from delamination at the interface between the PMC and the pure polyimide coating. The adhesive strength was reduced by more than 50% compared to the PMC (17.6MPa) due to damage from grit blasting.

From the literature it can be concluded that copper cannot readily be applied using thermal spray processes and that surface preparation and coating can induce damage in PMCs. Moreover, if a copper coating was to be applied over a metallic bond coat it would suffer from poor conductivity due to oxidation as reported in section 2.2.2.

Kinetic spraying processes were used for coating PMCs [18, 22, 100]. Lupoi et al. attempted to spray copper coatings on glass fibre/epoxy PMCs and on neat polymers with limited success [18]. The authors used CGDS with a nitrogen carrier gas at room temperature and varied the gas pressure from 0.5MPa to 3MPa. Lower gas pressures enabled some particle embedment in the polymers, but without actual coating formation. Higher pressures provided more kinetic energy which resulted in erosion of the substrates. This had a greater effect on the PMC as it exposed fibres and yielded fibre breakage. The authors nonetheless succeeded in depositing a tin coating on ABS, polyimide and polystyrene. It was proposed that coating formation is more likely to happen in materials with lower deformation energy, such as tin and lead.

Zhou et al. used CGDS with nitrogen at 1.2MPa and 300°C to deposit an aluminium coating on a carbon fibre/PEEK with a fibre volume fraction of 30% [100]. During the first spray pass aluminium particles embedded into the substrate. Upon subsequent spraying, aluminium formed a coating. One of the coatings was then polished with 1500-2000 grit paper and a copper coating was deposited on top. This is essentially equivalent to using a bond coat for applying a copper coating. The adhesion strength of the coating was 2.3MPa which is much lower than realised on a metallic substrate (section 2.2.2).

Robitaille et al. used PGDS to spray zinc on a carbon fibre/epoxy PMC [22]. They used helium as the carrier gas and tested various spraying parameters, including the variation of gas pressures from 1MPa to 3MPa and gas temperatures from 200°C to 400°C. The authors also pre-heated the zinc up to 200°C. All spraying cases were characterised by an absence of coating and by PMC erosion which sometimes exposed the carbon fibres. Deposition of a zinc coating on neat epoxy was feasible but adhesion was limited as the coating could be removed manually, suggesting that fibre inclusions in the PMC reduced the coating potential. A solution to mitigate the shortcomings of the coating process was proposed. The authors added a ductile layer on the surface of the PMC on which sprayed particles may deform and adhere. The layer consisted of spherical copper particles embedded in an epoxy matrix that was co-cured with the PMC. Spraying trials revealed that zinc coatings could be deposited on top of the co-cured layer (Figure 2.13). The shear strength of the zinc coating was measured at 14MPa which is higher than what was reported above by Liu et al. for thermal sprayed zinc coatings on polyimide [16].

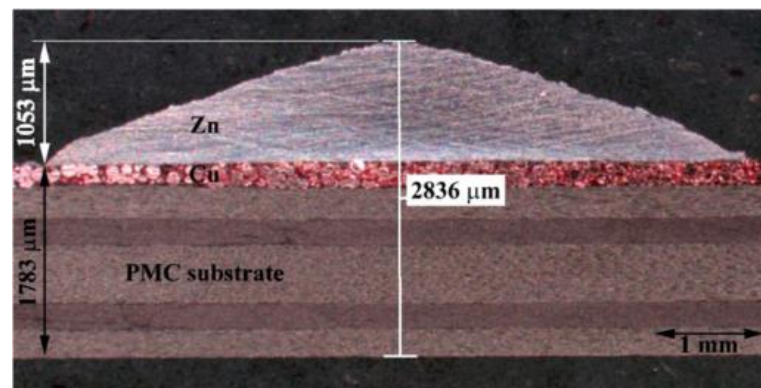


Figure 2.13: Zinc coating on a copper/epoxy layer co-cured on a carbon fibre/epoxy PMC [22]

Work on kinetic spray coatings for PMCs revealed that similar to what has been observed with thermal spray processes, copper cannot be sprayed directly on structural carbon fibre PMCs due to erosion. A metallic bond coat can be used for preventing erosion from sprayed copper but adhesion is weak. The solution that shows the best potential is the use of a co-cured layer featuring ductile particles on which coatings can adhere, as it prevents erosion and promotes adhesion. In this latter case, kinetic spraying also has the

advantage of producing copper coatings with low oxide content which are desirable for the sought IHCT application, explaining why this configuration was selected for the present thesis work.

2.3 Modelling

This section is structured in two parts. First, a review of previous heat transfer modelling work in IHCT is presented. In the second part, mathematical models for predicting the thermal and mechanical behaviour of coated PMCs that will be used for the fabrication of an IHCT demonstrator are reviewed.

2.3.1 Heat transfer in integrally heated composite tooling

Heat transfer in PMCs is an ongoing research interest of the University of Ottawa. Work in collaboration with NRC-IAR has been done for characterising the thermal conductivity of PMCs [55, 104], heat transfer during PMC repair [56] and heat transfer in IHCTs [23–26] which was introduced in section 2.1.3. Models of IHCTs presented in this thesis loosely originate from these previous studies.

Morton analysed temperature distributions during the repair of PMC parts [56]. He used computational fluid dynamics (CFD) software FLUENT to replicate a PMC repair operation where a heating blanket is used for curing a repair patch. The effects of the convection coefficient, glue thickness, PMC conductivity and part geometry were assessed. Even though this study is not directly related to IHCTs, it is worth noting that the results show that convection significantly hindered temperature uniformity. Hence, for repairs, it was suggested to minimise heat losses to the environment, which can be achieved through insulation; a concern which is also applicable to IHCTs.

Houde was the first to investigate optimisation parameters in IHCTs at the University of Ottawa [23]. Optimisation was performed using CFD software FLUENT. Goals for his project were to increase the heating rate across the moulding face of the tooling, minimise energy consumption and reduce the amount of metal in the conductive layer. His heat transfer model comprised a PMC part on top of a flat IHCT which featured layers for the surface heaters, copper sheet, adhesive for bonding the sheet, and PMC structure. Optimisation was performed through a parametric study featuring 17 factors including dimensions of the layers, convection coefficients, thermal conductivity values, and ambient

and processing temperatures. The responses analysed were the heating (up to 75°C) and cooling times, temperature difference in the domain and temperature variation in the thickness of the IHCT and PMC part. A Taguchi design of experiment was used for reducing the number of simulations from 2^{17} for a full factorial to 32 runs, consequently reducing the time required for identifying influential factors. A summary of findings is presented in Table 2.2.

Table 2.2: Influential factors affecting a given response for IHCT [23]

Response	Influential factors
Heating time	Processing temperature Heater power IHCT width and adhesive thickness
Cooling time	Convection coefficient Processing temperature
Temperature difference in the domain	Adhesive thickness Heater power Ambient temperature Processing temperature
Temperature variation in the PMC part	Thickness of PMC part Ambient temperature

Maksoud also worked on heat transfer in IHCTs [24]. His study was based on experimental investigation and simulation (FLUENT) of unsteady heat transfer in a flat IHCT. The effect of various factors was based on the comparison of the heating rate measured by 4 thermocouples located on the IHCT and heater. He first assessed the effect of orientation, i.e. horizontal or vertical, on heat transfer rate. Results showed that orientation had a negligible effect on the convection coefficient, which was estimated at $10\text{W/m}^2\cdot\text{K}$ in the laboratory. Others factors were then analysed. Results are presented in Table 2.3. The main finding is that increasing the thickness of layers results in slower heating rates for the inner tool face of the IHCT. However, it was also noted that experiments showed that the thickness of the adhesive used for bonding the copper sheet onto the IHCT had an effect on the heat flow, which was absent in simulations. It was

believed that inconsistent application of the adhesive and in some cases application defects altered the heat flux through the IHCT. It can be conjectured that a coating may offer more intimate contact between the copper and the PMC than an adhesive, which would improve heat flux uniformity. Moreover, Maksoud noted that the control thermocouple of the heater should not be placed directly on the heater as temperatures measured at that location are much higher than IHCT surface temperatures.

Table 2.3: IHCT factors affecting heating rates [24]

Factor	Effects on the heating rate
Orientation	No influence on the convection coefficient
Increasing thickness of adhesive for the heater	Heater temperature increases IHCT temperature is not affected
Increasing thermal conductivity of heating element	No effect
Increasing thickness of adhesive for the copper sheet	Slower heating rates for the moulding surface of the IHCT
Through-thickness thermal conductivity of PMC	Faster heating rates for the moulding surface of the IHCT

Brillant studied, through experiments and simulations (FLUENT), the effect of using several thinner sheets of copper instead of a thicker single sheet for heat distribution purposes in IHCTs [25]. This was investigated because thinner sheets have the advantage of offering greater formability, reducing the risk of improper contact between the copper and PMC. Several flat IHCTs were built using a certain number of copper sheets inserted between PMC plates, and were heated using 1 and 2 50W surface heaters. The IHCTs were heated for 90min and the temperature across their surfaces was measured using 8 thermocouples. Results showed that the location of the copper sheets had negligible impact on the temperatures at the inner tool face of IHCTs. However, placing the copper sheets directly under the surface heaters enabled lower operation temperatures for the heater,

which is a desirable feature. It was also noted that increasing the number of copper sheets reduced temperature variability across the inner tool face of the IHCT, but at a cost of greater energy consumption. It was also shown that increasing the number of heaters could improve temperature uniformity. Finally, Brillant developed a script to replicate the control of the heater (ON/OFF) in the CFD software FLUENT. The script provided a heating behaviour comparable to experimental measurements [25].

El Karafi worked on complex geometry IHCTs [26]. Her work focused on using a 3D digitizer for producing a mesh for a seatback tooling that was used afterwards for heat transfer analysis. Different types of meshes were imported in FLUENT and it was seen that the element size is vital for modelling heat transfer in complex IHCTs accurately. Regarding heat transfer, a comparison between the use of a single heater and 5 heaters on the IHCT was made. Results were in accordance with those of Brillant [25] and showed that increasing the number of heaters is beneficial for temperature uniformity across the IHCT.

The work performed on IHCTs greatly improved knowledge regarding their heat transfer mechanisms. However, there is a key element missing in the studies. One of the prime objectives of IHCTs is to offer a uniform cure temperature for PMC parts. This is achieved by having a uniform temperature across the moulding surface of IHCTs. Hence, to complement these studies an analysis of the factors affecting the inner tool face temperatures on IHCTs would be desirable. Such study is part of this thesis in Chapter 6.

2.3.2 Mathematical models

Heat transfer simulations for IHCTs presented in this thesis are based on the energy equation presented in section 2.1.1 and on the recommendations discussed in section 2.3.1. These simulations require specific input including geometry, boundary conditions and material properties; it is thus important to use input data that reflects reality accurately. Finding representative material properties can be difficult for composite materials. Often experiments can be devised for measuring the desired properties, but sometime this is not

practical or even feasible. In such cases, mathematical models describing material behaviour can be used.

This thesis presents coated PMCs which consist of three main materials: 1) carbon fibre PMC, 2) co-cured layer having copper particles embedded in epoxy and 3) a copper coating. Mathematical models for predicting thermal conductivity, specific heat capacity and mechanical properties for some of these materials without literature data are presented below. Models are for thermal conductivities and specific heat capacities. This section also includes a model for the elastic moduli of multiphase materials which will be used for predicting the mechanical behaviour of coated PMCs in Chapter 4.

2.3.2.1 Thermal conductivity

Thermal conductivity values for bulk copper and kinetic spray coatings are available in literature (presented in section 2.2.2) while for PMCs, values can be measured experimentally using the method developed by Hind [104]. However, no value is available, nor can it be measured easily, for the co-cured layer. A mathematical model is thus required for predicting the thermal conductivity of spherical copper inclusions embedded in an epoxy matrix.

Progelhof et al. made an extensive review of the various models that exist for predicting the thermal conductivity of a composite material having a dispersed filler phase inside a continuous matrix phase [105]. He compared the values predicted by the models with experimental values of magnesium oxide and glass spheres inside a polyethylene matrix. Results showed that the Lewis-Nielsen model gave the most reliable predictions. Similarly, Pal showed good agreement between experimental values and the Lewis-Nielsen model for spherical copper particles in polyimide, up to a filler content of 60% [106].

The Lewis-Nielsen model is a semi-empirical formulation that accounts for filler shape and packing type. The equation for the effective thermal conductivity of the system k_e is:

$$k_e = k_c \left[\frac{1 + AB\phi}{1 - B\phi\psi} \right] \quad (2.11)$$

where k_c is the bulk thermal conductivity of the continuous phase, ϕ is the dispersed phase content, A is the geometry factor (1.5 for spherical particles), and B and ψ are parameters defined as:

$$B = \frac{\frac{k_d}{k_c} - 1}{\frac{k_d}{k_c} + A} \quad (2.12)$$

$$\psi = 1 + \left(\frac{1 - \phi_m}{\phi_m^2} \right) \phi \quad (2.13)$$

where k_d is the thermal conductivity of the dispersed phase and ϕ_m is the maximum concentration of the dispersed phase for a given type of packing. A ϕ_m value of 64% should be used for randomly closed packed mono-dispersed spheres [105][106].

2.3.2.2 Specific heat capacity

The specific heat capacity of homogeneous materials can often be found in literature or it can be obtained through simple experiments. However, for heterogeneous materials like the PMC and the co-cured layer, a mathematical model is required for assessing the effect of each phase.

Jäckel showed that for glass fibre/epoxy composites a simple rule of mixture based on the volume fraction (v_d) of the discrete phases can be used for estimating the average composite specific heat capacity [107].

$$C_{p, \text{Composite}} = v_d C_{p, \text{Discrete}} + (1 - v_d) C_{p, \text{Continuous}} \quad (2.14)$$

The author observed that the equation overestimates the composite specific heat capacity by 10% to 20%. Since the specific heat capacity refers to the energy necessary to

excite mass, i.e. an atom, then a better approximation would be to use a rule of mixture based on mass (m) instead of volume (V).

$$C_{p, \text{ Composite}} = \frac{[mC_p]_{\text{Discrete}} + [mC_p]_{\text{Continuous}}}{m_{\text{Total}}} \quad (2.15)$$

For practicality it is easier to work with an equation based on the volume fraction of the discrete phase. The equation can be developed based on the density and volume of phases:

$$C_{p, \text{ Composite}} = \frac{[V\rho C_p]_{\text{Discrete}} + [V\rho C_p]_{\text{Continuous}}}{V\rho_{\text{Total}}} \quad (2.16)$$

After simplification of the volume and knowing that the average density can be obtained by a rule of mixture, the equation becomes:

$$[C_p]_{\text{Composite}} = \frac{[v\rho C_p]_{\text{Discrete}} + 1 - v_{\text{Discrete}} [\rho C_p]_{\text{Continuous}}}{v\rho_{\text{Discrete}} + 1 - v_{\text{Discrete}} \rho_{\text{Continuous}}} \quad (2.17)$$

which is the equation that is used throughout the thesis.

2.3.2.3 Elastic modulus

Similarly to the thermal conductivity, a mathematical model is required for estimating the elastic modulus of the co-cured layer having spherical copper inclusions in an epoxy matrix. The estimated values will be used for calculating stress distributions in coated PMC materials in Chapter 4.

Zimmermann investigated the Mori-Tanaka model [108]. The model was in good agreement with the experimental bulk elastic modulus of a sintered glass with spherical pores and a borosilicate glass with spherical tungsten inclusion.

The Mori-Tanaka model for spherical inclusions inside a matrix predicts that the effective elastic modulus of the composite material $\bar{E}$ is [109]:

$$\bar{E} = E_M \left[1 + \frac{\nu_I \left(\frac{E_I}{E_M} - 1 \right)}{\nu_I + 1 - \nu_I \left(1 + \frac{3 E_I - E_M}{3E_M + 4G_M} \right)} \right] \quad (2.18)$$

where E_M and G_M are the elastic and shear moduli of the matrix respectively, E_I is the elastic modulus of the inclusion and ν_I is the volume fraction of the inclusion.

Chapter 3

Materials, equipment and procedures

This chapter elaborates on the materials, equipment, procedures and simulation tools employed in the realisation of the project. First, a description of the material used for manufacturing the CPMCs and IHCTs is presented. This is followed by an explanation of the manufacturing procedures. Then, experiments for the thermo-mechanical characterisation of CPMCs and IHCTs are described. Finally, the chapter concludes with the description of the approach used in modelling heat transfer in IHCTs, aiming at understanding the governing factors affecting temperature uniformity.

3.1 Materials

In this section, the materials used for manufacturing CPMCs and IHCTs are presented. The CPMCs and IHCTs all consist of three distinct material layers: a structural carbon fibre/epoxy PMC inner layer, a thermally conductive copper outer layer, and an intermediate layer that connects the inner and outer layers together (Figure 3.1). For

IHCTs, heat is provided by a series of electrical heating elements bonded on their thermally conductive layer. The order in which materials are presented relates to their layer material category. Before describing each type of material, a short explanation of the actual composition of the CPMCs and IHCTs is presented.

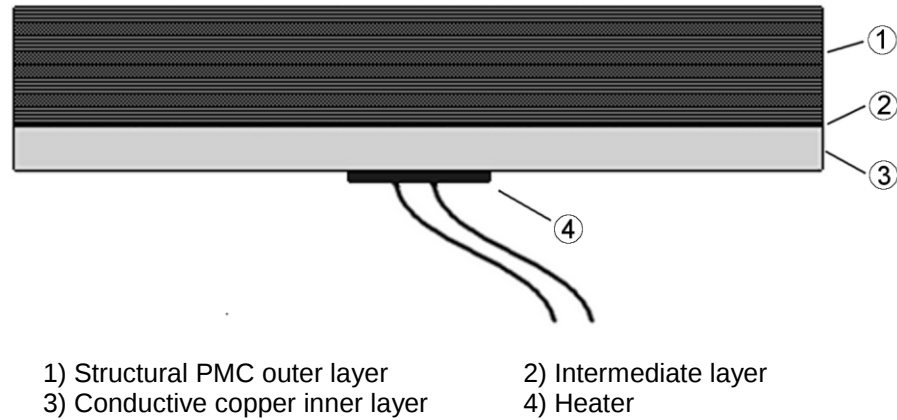


Figure 3.1: Diagram of an IHCT

The CPMCs used in this project had a carbon fibre/epoxy PMC inner layer, an intermediate layer made of metallic particles embedded in epoxy and a kinetically sprayed copper coating outer layer. The PMC layer offered structural rigidity as well as a low coefficient of thermal expansion. When using CPMCs as an IHCT material, the latter characteristic is particularly important for minimising the thermal expansion differences with the PMC parts to be made. The intermediate layer was used for bridging the outer coating and the inner PMC layers. This layer was composed of copper particles embedded inside the epoxy matrix which was co-cured on the PMC surface, and named henceforth the “co-cured layer”. Its main usage was to protect the PMC against erosion from direct exposition to the cold spray coating process and also to promote coating adhesion, as reported by Robitaille et al. [22]. The combined material system made from the PMC and co-cured layer was named the CPMC substrate, a PMC substrate on which a metallic coating was deposited. Finally, the last layer of the CPMC consisted of a kinetically sprayed copper coating that promotes in-plane thermal conductivity. The resulting CPMCs were then used as the basis for the IHCT-CPMC.

Two other types of IHCTs were compared to the IHCT-CPMC in Chapter 5: a heated PMC tooling plate (IHCT-PMC) and a NRC-developed IHCT (IHCT-NRC) using a bonded copper sheet for heat distribution. The IHCT-PMC did not feature any thermally conducting layer or intermediate layer. On the other hand, the IHCT-NRC had a thermally conductive copper sheet that was bonded to the PMC by using a silicone adhesive, or by co-curing the copper sheet with the PMC.

3.1.1 PMC materials

Two different prepreg materials were used for manufacturing PMCs and CPMCs. Most laminates were manufactured using a low cure temperature carbon/epoxy prepreg (SE-70, Gurit Ltd.) designed to be processed through out-of-autoclave vacuum bagging. However, some work was done with AS4/3501-6 prepreg plates consolidated in an autoclave for comparing with previous CPMC work done by Robitaille et al. [22].

In addition to the prepreps, resin films (SA-70, Gurit Ltd.) were sometime used for increasing the epoxy content in the laminate and for embedding a thicker layer of copper particles onto the CPMC substrates. When used, the film was added at the surface of the prepreg stack.

3.1.1.1 Gurit SE-70 prepreg

SE-70 (Gurit Ltd.) is a low cure temperature and high toughness unidirectional prepreg designed for out-of-autoclave manufacturing. The material is composed of aligned unidirectional PAN-based carbon fibres reinforcing an epoxy matrix. The nature of the resin system is proprietary to Gurit Ltd., while the fibres are PYROFIL™ TR50S produced by Grafil Inc. The properties taken from the manufacturer data sheet for consolidated SE-70 laminates are presented in Table 3.1.

Table 3.1: Properties of SE-70 unidirectional prepreg cured at 70°C for 16 hours as stated by Gurit Ltd.[110]

Property	After 16-hour cure at 70°C
Fibre surface weight	200 g/m ²
Prepreg surface weight	317 g/m ²
Prepreg resin content	37 wt% $\pm$ 3%
Cured ply thickness	0.2 mm
Fibre volume fraction	57.7%
Tensile modulus	136.2 GPa
Tensile strength	2480 MPa
ILSS	68.6 MPa

Properties of the carbon fibres are presented in Table 3.2. The TR50S carbon fibres have a longitudinal thermal conductivity of 7W/m·K [111]. As for the properties of the epoxy matrix, it is information undisclosed by Gurit Ltd.

Table 3.2: Mechanical and thermal properties of PYROFIL™ TR50S carbon fibres as stated by the manufacturer [111, 112]

Property	Value
Yield	15K fibres per tow
Tensile strength	4900 MPa
Tensile modulus of elasticity	240 GPa
Density	1820 kg/m ³
Coefficient of thermal expansion (parallel to fibres)	0.5 μ m·m/K
Specific heat capacity	740 J/kg·K
Thermal conductivity	$\parallel$ 7 W/m·K $\perp$ No data

The prepreg system can be cured at temperatures as low as 70°C for a period of 16 hours or as quickly as 25 minutes at a temperature of 120°C. In addition to cure duration, the cure temperature also affects the glass transition temperature (Table 3.3).

Table 3.3: Glass transition temperature of SE-70 based on the cure temperature [110]

Cure temperature (°C)	Dwell time (Hours)	Glass transition temperature (°C)
70	16	89
80	8	101
95	4	110
110	0.5	126

3.1.1.2 Hexcel AS4/3501-6 prepreg

Autoclave AS4/3501-6 prepreg (Hexcel Corp.) was selected for comparison with out-of-autoclave SE-70 prepreg. The selection was based on previous work done at the University of Ottawa related to CPMCs by Robitaille et al. [22] and on IHCTs by Maksoud [24]. The prepreg is composed of aligned unidirectional AS4 PAN-based carbon fibres made by Hexcel reinforcing a 3501-6 epoxy matrix. The epoxy system is composed of tetraglycidyl methylenedianiline (TGMDA) (56.5wt%), alicyclic diepoxy carboxylate (9wt%), epoxy cresol novolac (8.5wt%), diaminodiphenyl sulfone (DDS) (25wt%), and boron trifluoride (BF₃) (1wt%) [113–115]. The first three compounds make the epoxy resin whereas the two others are curing agents. The mechanical and thermal properties of the prepreg constituents and cured laminate are presented in Table 3.4.

For heat transfer modelling it is important to know the thermal conductivity and specific heat capacity of the PMCs as a function of temperature. In the case of AS4 fibres, the specific heat capacity varies from 711J/kg·K at 20°C up to 1210 J/kg·K at 200°C [116]. Conversely, Chern et al. measured the specific heat capacity of epoxy 3501-6 at different degrees of cure as depicted in Figure 3.2. The figure shows that in the useful temperature range, i.e., below the glass transition, specific heat capacity of the 3501-6 epoxy system increases linearly with temperature.

Table 3.4: Mechanical and thermal properties of AS4/3501-6 composites

Property	HexTow [®] AS4	Epoxy 3501-6	Unidirectional AS4/3501-6 composite
Fibre volume fraction			62% [117]
Cured ply thickness [mm]			0.13 [117]
Tensile strength 0° [MPa]	4,433 [118]	45.5 [42]	2,100 [117]
Tensile modulus of elasticity [GPa]	231 [118]	4.2 [42]	140 [117]
Density [kg/m ³]	1,790 [118]	1,260 [117]	1,590
Coefficient of thermal expansion [μm/(m·K)]	-0.63 [118]	38 [42]	-0.3 [54] ⊥ 28.1 [54]
Specific heat capacity at: 20°C [J/kg·K]	711 [116]	1,260 [117]	
200°C [J/kg·K]	1,210 [116]		
Thermal conductivity [W/(m·K)]		0.24 [115]	6.87 [55] ⊥ 0.74 [55] [0/90] _s 3.78 [55]
Glass transition temperature [°C]		193 [113]	199 [113]

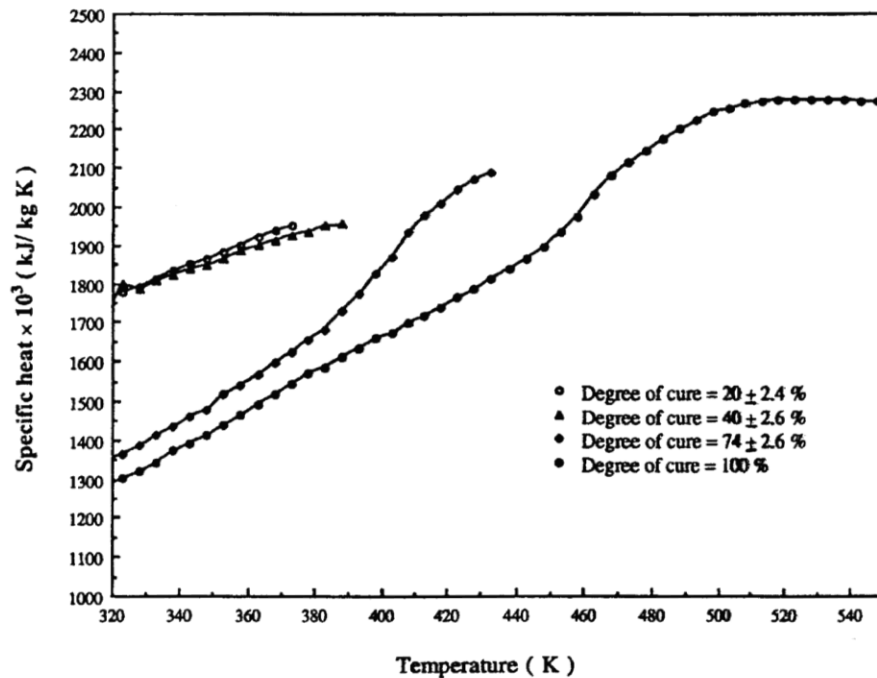


Figure 3.2: Specific heat capacity of epoxy system 3501-6 for various degrees of cure [115]

Combining both specific heat capacities of the carbon fibres and epoxy resin with the fibre volume fraction suggested by the manufacturer (~62%), one can approximate the specific heat capacity based on the equation presented in the literature review:

$$C_{p, \text{Composite}} = \frac{v_f [\rho C_p]_f + 1 - v_f [\rho C_p]_r}{\rho_f v_f + \rho_r 1 - v_f} \quad (2.17)$$

For the case of the AS4/3501-6, the specific heat capacity of the epoxy was obtained from a linear regression of the measurements presented in Figure 3.2. The regression was made from 320K to 410K, in the linear domain prior to the glass transition. Due to the lack of data for the AS4, the specific heat capacity was assumed to vary linearly with temperature between the two values reported in Table 3.4 from [116]. Linear regressions that were obtained for the specific heat capacities are:

$$C_{p, \text{AS4}} = 2.77T - 182 \quad [\text{J/kg} \cdot \text{K}] \quad (\text{For } 295\text{K} < T < 475\text{K}) \quad (3.1)$$

$$C_{p, \text{3501-6}} = 4.61T - 101 \quad [\text{J/kg} \cdot \text{K}] \quad (\text{For } 320\text{K} < T < 410\text{K}) \quad (3.2)$$

where T is in Kelvin. Combining Equations (3.1) and (3.2) with Equation (2.17) and a v_f of 62%, the specific heat capacity of the AS4/3501-6 laminate yielded:

$$C_{p, \text{AS4/3501-6}} = 3.3T - 158.5 \quad [\text{J/kg} \cdot \text{K}] \quad (\text{For } 320\text{K} < T < 410\text{K}) \quad (3.3)$$

3.1.1.3 Gurit SA-70 resin film

A resin film was also used in selected experiments which required additional epoxy for infusing a thicker co-cured layer. The selected material was a SA-70 epoxy adhesive film from Gurit Ltd. The SA-70 is intended to be compatible with the SE-70 prepreg and has a similar cure cycle [119]. The film has a resin surface density of 150g/m² and is supported by a 25g/m² glass carrier. Similarly to the SE-70, there is no divulged information from the manufacturer regarding the thermal properties of that epoxy system, but for the sake of the project they was assumed to be approximately the same as those of SE-70 epoxy due to the compatibility between the two resins.

3.1.2 Thermally conductive layer

The thermally conductive layer of CPMCs was made of consolidated copper powder. The two types of powder tested were: 1) Cu-105-3 and 2) SST-C5003. Conversely, the NRC-IHCT used a pure copper sheet as its thermally conductive layer.

3.1.2.1 Copper powders

Copper coatings were produced by compacting fine metallic particles together using kinetic spray processes. For the project, two different powders were tested. Work began with Praxair Cu 105-3 powder which was a material well known at the University of Ottawa coating laboratory for kinetic spray processes. Mid-way into the project the powder was changed as the supplier stopped producing Cu 105-3. From this point onward, the coatings were made using Centerline Ltd. SST-C5003 powder. Occasionally, both powders are compared in this work.

Praxair Cu 105-3 is a pure copper powder (>99wt% Cu). Its high copper content is desirable for promoting thermal conductivity as impurities in the crystal structure can disrupt lattice vibrations and thereby impair the thermal conductivity. The copper particles were produced by water atomisation, which may induce slight oxidation. SEM images of the powder showed that the particles have an irregular shape with a sub-angular structure (Figure 3.3). The grape-like structure seen in the SEM images suggests some surface oxidation which may be due to more than two years of powder aging in the laboratory.

The powder was sent to Vac Aero International Inc. for a particle size analysis. The characterisation was done with a laser-based system (Beckman Coulter LS™ 200). The analysis was performed in a methanol environment with 60 second duration for the run. The results are presented in Figure 3.4. The mean particle diameter was measured as $34.77\mu\text{m} \pm 10.65\mu\text{m}$.

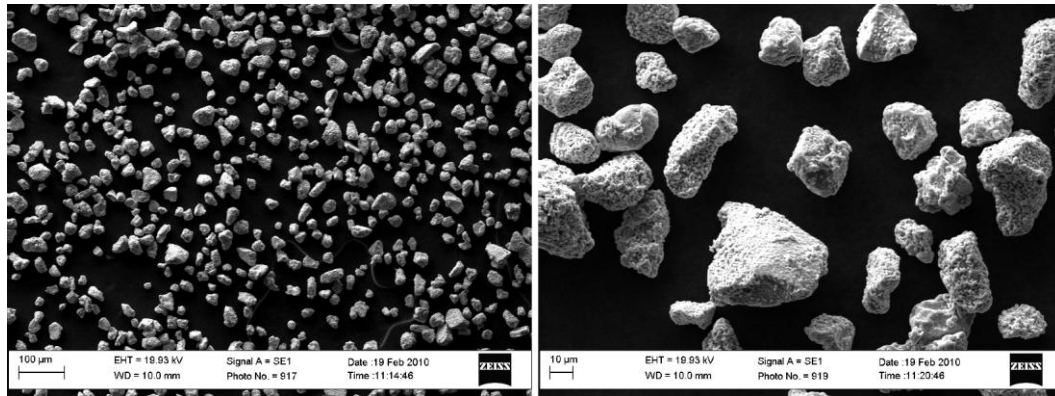


Figure 3.3: SEM images of copper powder Cu 105-3 at magnifications of 100X (left) and 500X (right)

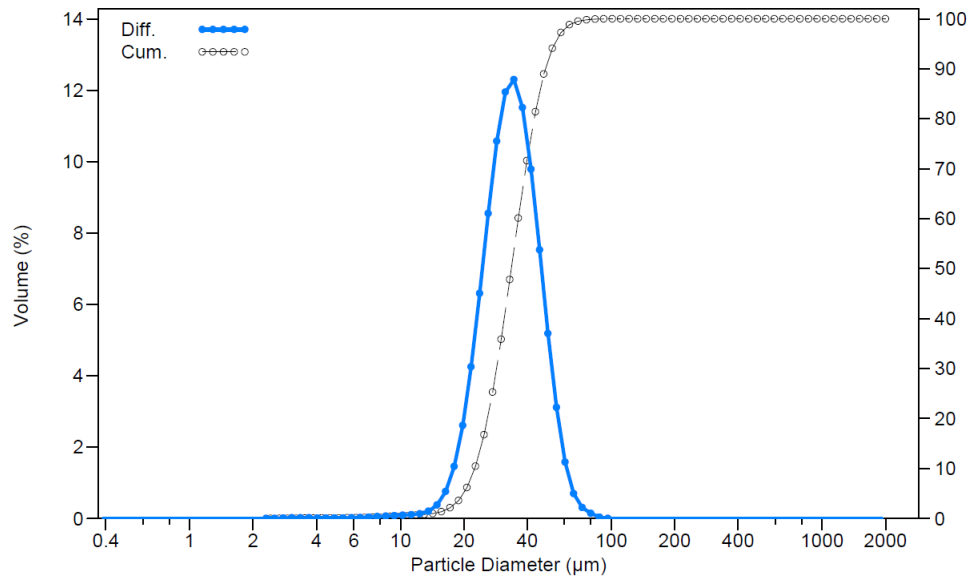


Figure 3.4: Differential and cumulative particle size analysis of Cu 105-3

The second type of powder that was tested was SST-C5003 from Centerline Ltd. It is a pure copper powder (>99.7%) with particle sizes ranging from 5μm to 45μm [120]. SEM images of the powder (Figure 3.5) showed that the particles have a grape-like and dendrite structure indicative of an electrolytic formed powder. It was also seen that there was a significant difference in particle size, where tiny particles were mixed with bigger ones. During the project, it was observed that filtering the sub 20μm particles significantly

helped adhesion of the particles to substrates. Hence, for the project the powder was sieved to retain all particles of a size greater than 20 μm .

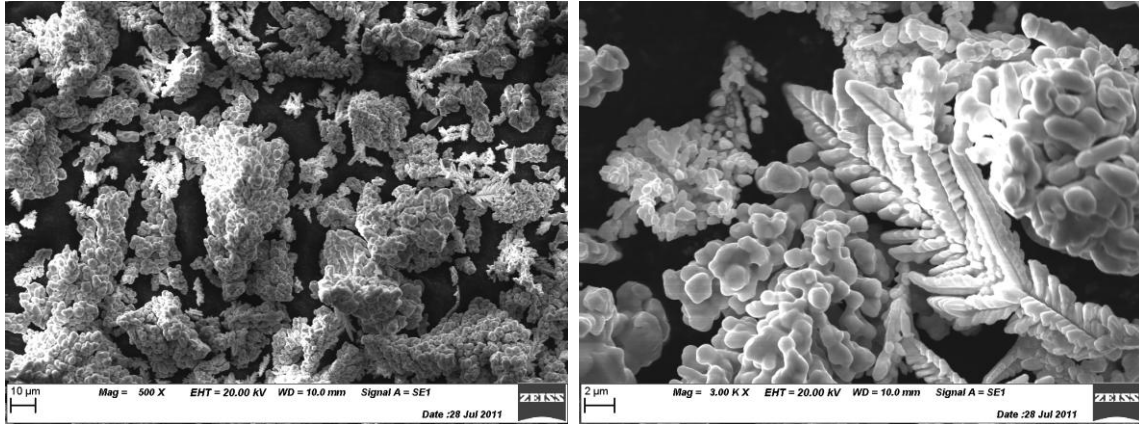


Figure 3.5: SEM images of copper powder SST-C5003 at a magnification of 500X (left) and 3000X (right)

3.1.2.2 Copper sheet

Copper sheets used for the IHCT-NRC were made of C10100, which is an oxygen-free electronic copper alloy with a copper content of 99.99%. The high purity of copper in the alloy provides thermal and mechanical properties similar to that of the pure metal. The properties of the pure metal can be found in Table 3.5.

Table 3.5: Mechanical and thermal properties of pure copper [121]

Property	Value
Density	8940 kg/m ³
Coefficient of thermal expansion at 20°C	16.7 $\mu\text{m}/\text{m}\cdot\text{K}$
Specific heat capacity at: 20°C	386 J/kg·K
150°C	400 J/kg·K
Thermal conductivity at: 0°C	401 W/m·K
127°C	392 W/m·K
Cold drawn hardness	37 HRB (96 HV _{0.1} [122])
Tensile modulus (annealed)	125 GPa
Shear modulus (annealed)	46.4 GPa
Tensile strength (annealed)	209 MPa

3.1.3 Intermediate layer

The intermediate layer was the medium that connected or bonded the PMC to the thermally conductive outer layer. For the CPMCs, the intermediate layer consisted of copper particles co-cured onto the PMC. For the IHCT-NRC, the copper sheet was either bonded with a high-temperature silicone adhesive or was co-cured.

3.1.3.1 Co-cured layer

The co-cured layer consisted of deformable particles that were embedded in an epoxy binder. Based on results obtained from [22], copper powder Cu 159-2 from Praxair Inc. was selected and co-cured on the surface of the PMC using excess resin within the prepreg. The powder certificate stated that it was composed 99.8% of copper with small amounts of oxygen, and that 95% of particles had diameters between 75 μ m to 125 μ m. From Figure 3.6, it is seen that the copper particles were characterised by a smooth surface and a spherical shape. The powder was made from fairly large particles with smaller satellites that are often attached onto them. The satellites and the spherical morphology of the particles indicate that the powder was likely processed by gas atomisation. The resin matrix used for embedding the copper particles came from the prepreg, and/or from SA-70 resin films when stated.

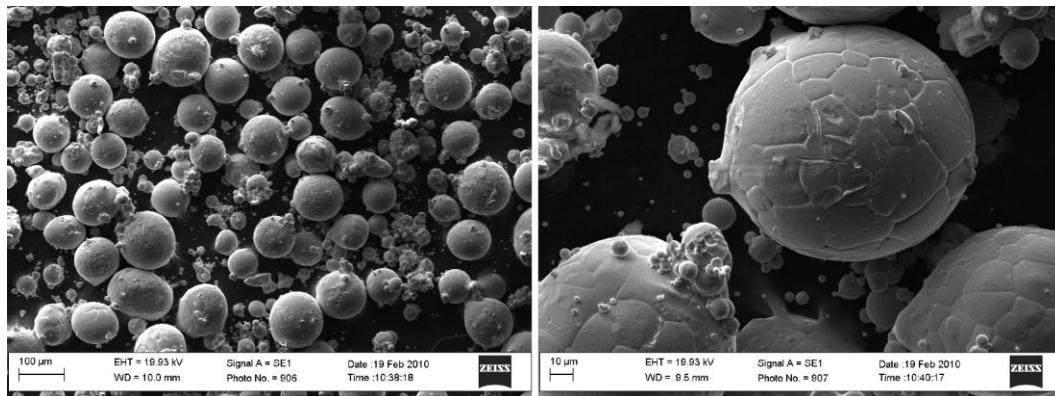


Figure 3.6: SEM images of copper powder Cu 159-2 at magnifications of 500X and 3000X

3.1.3.2 High temperature silicone

Copper sheets in the NRC-IHCTs were bonded using a high-temperature silicone adhesive that vulcanises at room temperature. Properties of the silicone are presented in Table 3.6.

Table 3.6: Properties of the high-temperature silicone adhesive [123]

Property	Value
Density	1470 kg/m ³
Thermal conductivity	0.18 W/m·K
Specific heat capacity	1465 J/kg·K

3.1.4 Flexible electric heaters

IHCTs were heated using flexible electric heaters from MINCO Inc. The heaters are made of silicone rubber reinforced by glass fibres and can be operated to temperature up to 235°C [124]. For the project, the heaters were bonded to the IHCTs using PSA #12 adhesive from MINCO Inc. and were cured in the oven at 150°C for 30min as recommended [125]. Two types of rectangular heaters were used, a low voltage heater labelled HR5466R8.6L12D and a high voltage heater labelled HR6600R576L24A. Their dimensions and electrical characteristics are presented in Table 3.7. Their power output was controlled by the supplied voltage using a Variac (variable autotransformer). The power outputs that were used in the project ranged from 15W to 25W.

Table 3.7: Description of the flexible electric heaters [124]

Model	Width (mm)	Length (mm)	Thickness (mm)	Electrical resistance (Ω)	Effective heating area (mm ²)
HR5466R8.6L12D	76	102	0.7	8.8	6540
HR6600R576L24A	25	127	1.5	576	2845



Figure 3.7: Flexible electric heaters HR5466R8.6L12D (top) and HR6600R576L24A (bottom)

The effect of heater temperature on the power output was investigated for heater HR6600R576L24A. For the experiment, the heater was attached to IHCT plate HT-02 described in section 6.3. Power was supplied directly from a 120V electrical outlet without the Variac. The voltage and current were measured using multimeters, providing the power supplied to the heater. The current was relatively constant with heater temperature at approximately $0.193\text{A} \pm 0.02\text{A}$. Slight variations in voltage and power were observed (Figure 3.8), but there seemed to be no correlations between heater temperature and power output. Nevertheless, voltage measurements taken directly at the outlet suggested that the variations were caused by voltage fluctuation from the utility. Hence, temperature did not appear to affect the flexible electric heaters significantly below 100°C . It was assumed that heaters of the other type had a similar behaviour due to product similarities.

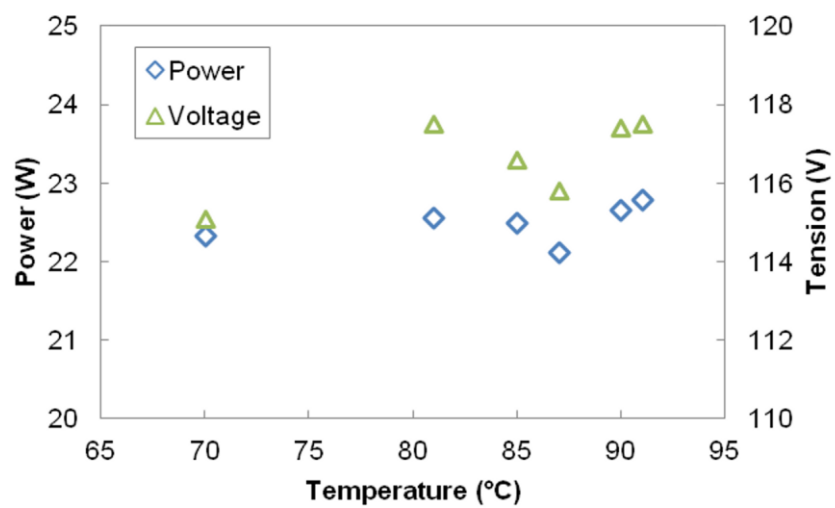


Figure 3.8: Variation in electrical power and tension with heater HR6600R576L24A temperature

3.2 Fabrication process

This section is separated along two main topics. The first topic of discussion (section 3.2.1) is the manufacturing method for PMC laminates and CPMC substrates. The second topic (section 3.2.2) is the description of the spraying processes used for depositing a coating on CPMC substrates.

3.2.1 PMC manufacturing

PMC laminates and CPMC substrates used in this study were mostly manufactured via out-of-autoclave vacuum bagging; a limited number were also made in an autoclave. Unless specified, the preparation procedure used for both in- and out-of-autoclave manufacturing was identical. This section details the entire manufacturing process which consists of stacking prepreg plies, adding a powder bed for CPMC substrates, vacuum bagging and curing.

3.2.1.1 Stacking prepreg plies and powder bed

The procedure used is depicted in Figure 3.9 and it first consisted in cutting prepreg plies to the desired dimensions from a feedstock roll. The plies had a square shape with an edge length that varied between 150mm to 300mm depending on the laminate. The plies were stacked on a flat tool that was covered in release film to prevent adhesion. The plies were oriented orthogonally with mid-plane symmetry. Stacks were made of 12 plies for the CPMCs ($[0, 90]_{6s}$) and 20 plies for the IHCTs ($[0, 90]_{10s}$). After laying each ply, the stacks were compacted manually with a roller for removing air entrapped between plies. When required, a single ply of SA-70 resin film was placed on top of the stack to increase the resin content. For the autoclave cured PMCs, debulking was performed using a vacuum bag once every 3-4 plies were added to the stack.

For CPMC substrates, a copper powder bed was laid on top of the stack. The powder bed was laid manually by sprinkling copper particles over the stack. Its mass was controlled through mass measurements to obtain a copper surface density of 0.2g/cm^2 to

0.5g/cm² which depended on the amount of excess resin available in the stack. Particles of the copper bed were embedded onto the PMC during cure by seepage of excess resin. It should be noted that for out-of-autoclave manufacturing, the bed was consolidated using a vibrating table which is discussed in the next section.

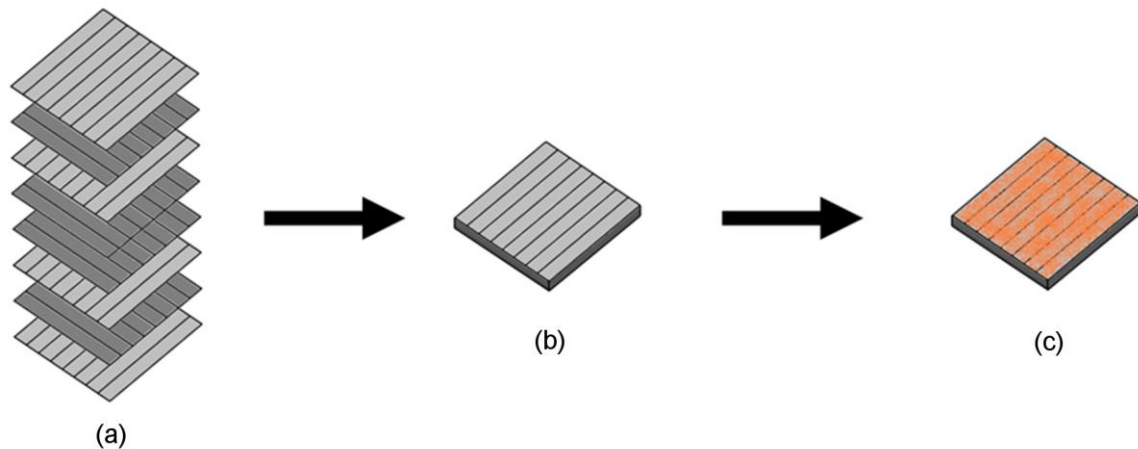


Figure 3.9: Stacking procedure for the substrates of CPMC: a) stacking plies with mid-plane symmetry, b) compacting prepreg stack, and c) adding the copper powder bed to the prepreg stack

3.2.1.2 Vibrating table and co-cured layer consolidation

A vibrating table was built for distributing the dry copper powder bed onto the out-of-autoclave prepreg stacks evenly before consolidation. The objective was to use vibrations to fluidise the powder bed and to let the gravitational forces distribute the powder, leading to a uniform powder bed thickness. The vibrating table (Figure 3.10) was composed of a flat plate that had each corner resting on a spring, ensuring sufficient mobility of the table and minimal vibration damping. Vibrations were generated by two 18V electric motors with off-centred weights. The motors were placed at opposite locations from each other on the table to distribute the vibrations evenly. The frequency and amplitude of the vibrations were controlled by pulse width modulation (PWM) of the motor voltage supply, and by displacing the centre of mass of the rotating weights. The weights were wooden blocks of 28g each with dimensions of 117mm in length, 32mm in

width and 19mm in thickness. Holes were drilled at various locations on the blocs for enabling attachment to the motor shafts.

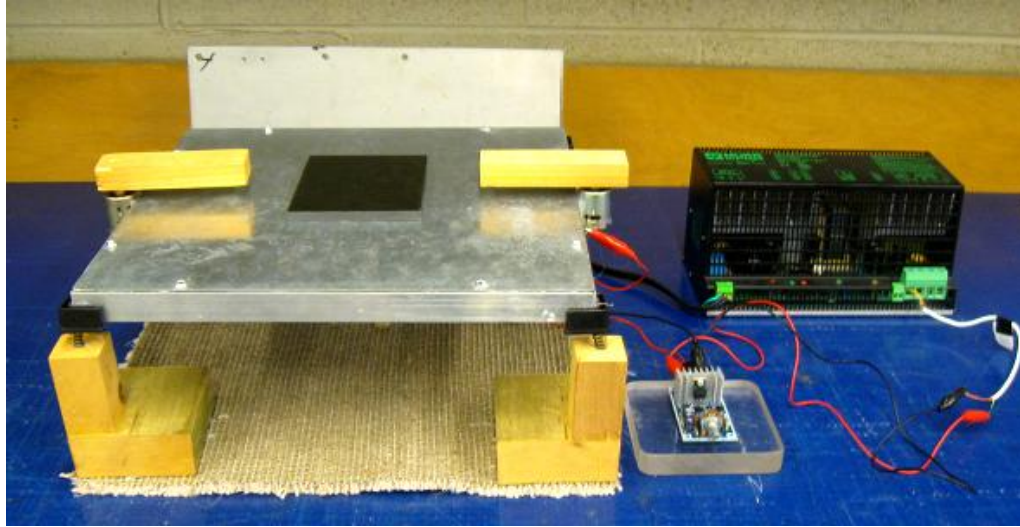


Figure 3.10: Vibrating table with PWM controller and power supply

Preparation for powder bed fluidisation consisted in placing a prepreg stack on a layer of release film previously taped on the vibrating table. The adhesion provided by the epoxy in the prepreg prevented the stack from moving under vibration. Pressure sensitive tape was placed on the edges of the stack, forming surrounding walls that prevented powder from falling off after fluidisation. The adhesive in the pressure sensitive tape resulted in contamination of the edges of the stack. This contamination was deemed negligible considering that the edges of the resulting laminate were cut prior to future use. However, in the future the walls will likely need to be made of a non-intrusive material, such as an aluminium frame wrapped in release film. Then the copper powder was distributed manually across the entire surface of the prepreg stack. Once preparation was completed the motors were activated and the powder bed was fluidised for a few seconds, which resulted in the flattening of the powder bed (Figure 3.11). Afterwards, the pressure sensitive tape was removed and the stack was cured through vacuum bagging in an oven as described in the next section.

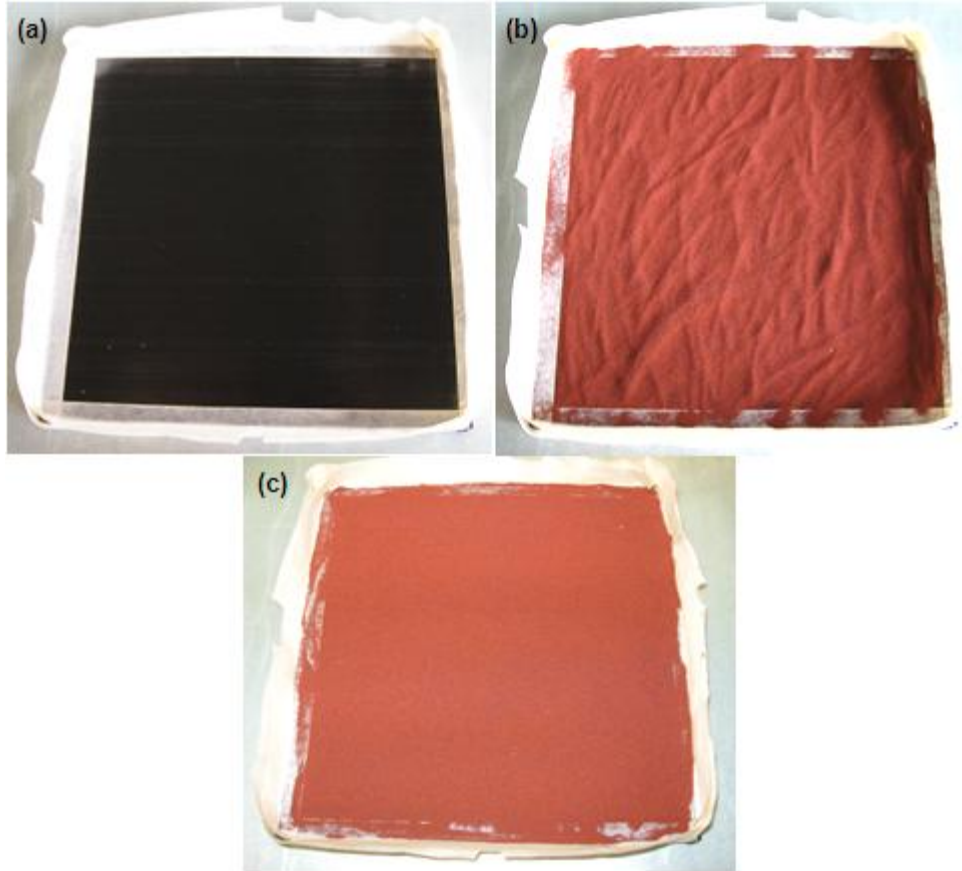


Figure 3.11: Prepreg stack a) without the powder bed, b) after spreading the copper powder, and c) after vibration

3.2.1.3 Vacuum bagging and cure

PMCs and CPMC substrates were consolidated by vacuum bagging in- and out-of-autoclave. Both curing methods employed the same vacuum bagging procedure depicted in Figure 3.12. It consisted in laying the prepreg stack on a flat 6.5mm thick aluminium tool that was protected with a release film (Dahlar® Release Bag 125, Airtech International Inc.) to prevent adhesion of PMC to the tool. A 6.5mm thick aluminium caul plate wrapped in release film was placed on the prepreg stack for providing uniform pressure distribution and obtaining a flat top surface for the PMC laminate or CPMC substrate. The stack and caul plate were covered by a perforated film (P-3 Dahlar® Release Bag 125, International Inc.) and by a bleeder ply (Econoweave 44, Airtech International Inc.) that absorbed excess resin. A vacuum bag (Dahlar® Release Bag 125, International Inc.) covered the assembly and the bag was sealed with high temperature sealant tape (SM 5126 Tacky Tape®,

Schnee-Morehead Inc.). A valve positioned on the centre of the caul plate connected the vacuum bag to a vacuum pump through a flexible hose (Econoflow 59R, Airtech International Inc.).

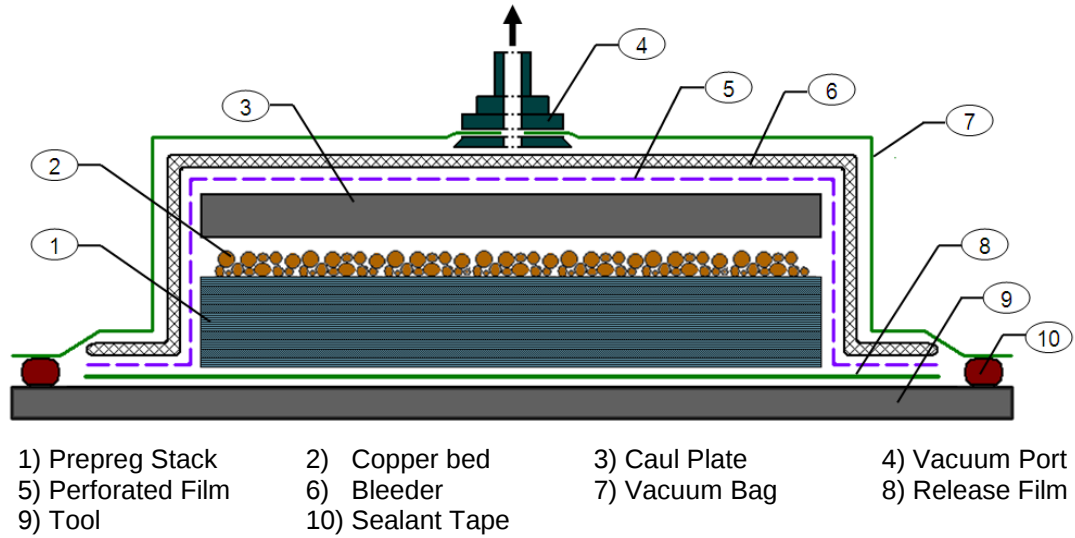


Figure 3.12: Vacuum bagging method for manufacturing PMC substrates

Out-of-autoclave PMC laminates and CPMC substrates were cured in a convection oven (PF120, Carbolite Inc.). It should be noted that the oven thermocouple was repositioned to minimise lag between the setpoint temperature and the actual chamber temperature (Appendix A). A vacuum of 0.98bar was applied by a diaphragm vacuum pump (DAA-V715A-EB, Gast) during the entire cure cycle which is presented in Figure 3.15. It consisted of heating the prepreg stack at a rate of 2°C/min until it reached 120°C. The temperature dwelled for 30min, after which the cured PMCs or CPMCs were cooled to room temperature.

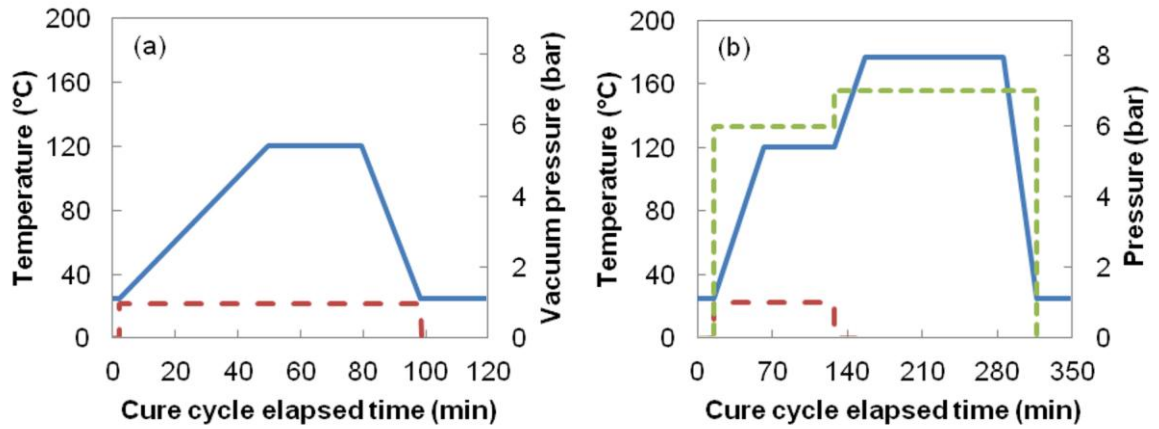


Figure 3.13: PMC processing through vacuum bagging in an oven



Figure 3.14: GAST vacuum pump used for vacuum bagging

Autoclaved PMCs were processed in a BAC-46 Baron-Blakeslee autoclave at NRC-IAR. The cure cycle used is presented in Figure 3.15. The prepreg stack was first heated to 120°C at a rate of 2°C/min. After a 65min dwell, the temperature was increased to 177°C and kept constant for a 130min post-cure. The cured plates were then cooled to room temperature.



Legend: — Temperature — Vacuum pressure — Applied pressure

Figure 3.15: Cure cycle for a) out-of-autoclave SE-70, and b) autoclave AS4/3501-6



Figure 3.16: NRC-IAR BAC-46 Baron-Blakeslee autoclave with vacuum bagged PMC plates [104]

3.2.2 Coating processes

Two kinetic spraying processes were tested for applying conductive coatings on PMCs: 1) Pulsed-Gas Dynamic Spraying (PGDS) was the main coating process used in this work, while 2) Low-Pressure Cold-Gas Dynamic Spraying (LP-CGDS) was mostly used as a mean of comparison. These processes were presented in section 2.2.1.

3.2.2.1 PGDS coating process

PGDS coating process was performed using various Waverider™ prototypes co-developed by University of Ottawa and Centreline Ltd. Three main versions of the system were tested during the course of the project, with their principle of operation remaining the same. A diagram of the general PGDS systems is presented in Figure 3.17. The systems were composed of a pressurised gas tank that fed helium gas to a plenum chamber through a pressure regulator. The plenum chamber was connected to a long heated barrel using a valve. Heat was provided to the barrel by a heated coil section. Upon valve opening, a shock-wave was generated from the high pressure difference between the plenum and the ambient. The shock-wave propagated into the lower pressure zone and it compressed and accelerated the carrier gas in the heated barrel to supersonic velocities. Compression also heated the gas. In a parallel process, metallic particles were injected into the barrel from a heated particle feeder. The carrier gas accelerated the particles to high velocities from the high drag forces. The particles were propelled towards a substrate where they formed a coating upon impact due to significant plastic deformation of the particles that leads to mechanical interlocking as described in section 2.2.1. The valve is closed after a certain time, enabling refill of the plenum and allowing the gas in the barrel to return to ambient pressure. After cool down, the valve is opened again for another shock-wave cycle. Shock-waves were generated at a frequency of 10Hz in this work.

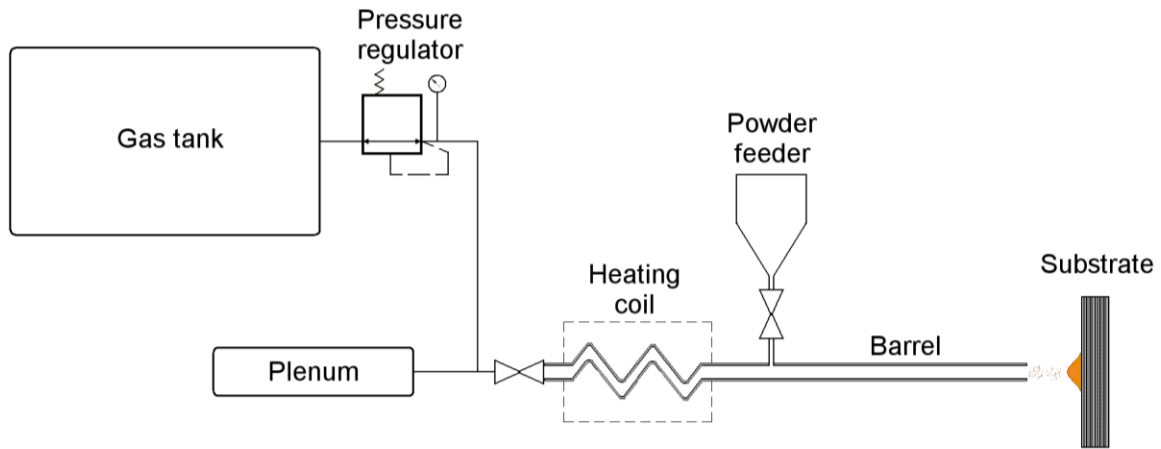


Figure 3.17: Diagram of a PGDS system

The three versions of the PGDS system that were tested differed in the way that powder was injected into the barrel, in how the shock-wave generating valve operated, and in the type of X-Y table used for displacement during spraying. For PGDS system #1 (Figure 3.18), gravity-based powder feeding was used in conjunction with a solenoid valve. For gravity-based feeding, a finite amount of powder was placed into a 150cm^3 stainless steel cylinder that could be heated using a heating jacket. When the shock-wave travelled down the barrel, it also went into the cylinder. Gas in the cylinder was put into motion and carried particles with it into the barrel. Powder flow was adjusted by a micrometer valve and a ball valve. Flow control was effectively accomplished with the ball valve as even when the micrometer valve was closed, large amounts of powder could flow through it due to valve erosion and high powder flowability. Control of the flow through the ball valve was approximative due to the lack of a gauge indicating valve opening. Furthermore, it was seen that the flow rate depended on the quantity of powder left in the cylinder. For a relatively constant flow rate, the valve required constant adjustment during spraying, which could lead to excessive or lack of powder feeding. Manual feeding control often offered greater and quicker control over powder feeding compared to the forced feeding system of PGDS #3.

In addition, PGDS system #1 used a solenoid valve (Asco Series 8223) for shock-wave generation. The valve was activated by a computer-controlled relay. During

operation, the valve was opened for 10ms before closing and the cycle was repeated at a rate of 10Hz. The solenoid valve procured precise control over valve operation, mostly over the ratio between the opened time and closed time. However, the fast cycle rate left the valve prone to significant fatigue wear, resulting in a short lifespan before valve leaking occurred. Valve leaking can weaken the strength of the shock-waves and yield lower particles velocity.

In-plane substrate motion was achieved using an X-Y table from Aerotech (Figure 3.19). The table was controlled by a computer through a Labview program written by previous students and technician Léo Denner. The table could initially move 50mm along both X- and Y-axes at respective velocities of 50mm/s and 5mm/s. The table was protected from powder dust by a sealed plastic bag, but powder still managed to get into the bag and into the motors. Early in the project, the Y-axis motor failed from powder infiltration in the circuitry. From that point on the Y-axis was controlled by a manual crank inside the chamber. The protected X-Y table with fixtures for holding the coating substrates appear in Figure 3.19.



Figure 3.18: PGDS system #1

PGDS system #2 was the same as PGDS system #1 but with a different gas valve. The solenoid valve was replaced with a rotary valve that featured a high toughness PTFE polymeric seal (Rulon®) to reduce wear. The valve split the flow into three identical channels for mitigating vibrations induced during rotation. Control over the shock-waves was achieved through the rotating speed of the valve and opening size of the channels. The valve was powered by a DC motor controlled with a variable DC power supply (0V-24V). As for the size of the channels, it was predetermined by the valve seat that was used. Valve seat #N3 (Centerline Ltd.) was selected with a rotational speed yielding shockwaves at approximately 10Hz.

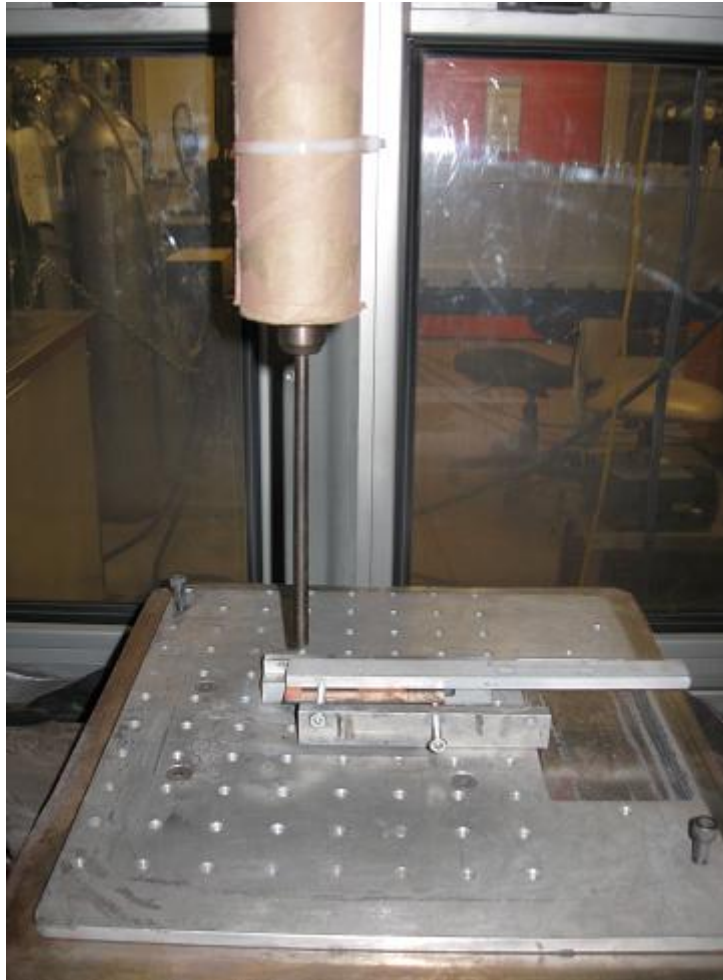


Figure 3.19: Inside the spraying chamber of the PGDS systems #1 and #2

PGDS system #3 was used at the end of the project for coating larger plates as it offered a fully controllable X-Y table. The system used the same rotary valve for shock-wave generation as PGDS system #2 but the gas and valve controllers were of a newer generation. The latest PGDS system was installed in the same spraying chamber as the LP-CGDS (Figure 3.21). Instead of gravity-based feeding, an automatic powder feeder (Praxair Inc.) was used with PGDS system #3. The feeder featured a sealed canister filled with powder, and a metering wheel to regulate powder distribution. The metering wheel injected powder into the injection line that was connected to the PGDS barrel. The amount of powder injected was controlled by the rotation speed of the metering wheel, and by the size and number of slots on the wheel. The powder was forced into the barrel by feeding helium in the canister at a rate of 10ml/min.

3.2.2.2 LP-CGDS coating process

A LP-CGDS system from Centerline Ltd. was used (Figures 3.21-3.22) for comparison with PGDS. A diagram of the system is presented in Figure 3.20. The system used a helium gas tank that fed the carrier gas into a heated plenum at a specific pressure controlled by a pressure regulator. The carrier gas was accelerated to supersonic velocity by a de Laval converging-diverging nozzle. Sprayed powder was fed in the expanding carrier gas and projected towards a substrate where it formed a coating by plastic deformation. Powder feeding was achieved using the same automatic powder feeder (Praxair Inc.) described for PGDS system #3. Contrarily to the PGDS systems, the coating process was performed by motion of the spraying nozzle instead of substrate motion. The nozzle was positioned on an X-Y gantry controlled by commercial computer software and controller. Coating characteristics were controlled through the plenum gas pressure, plenum gas temperature, powder feeding rate and nozzle displacement rate.

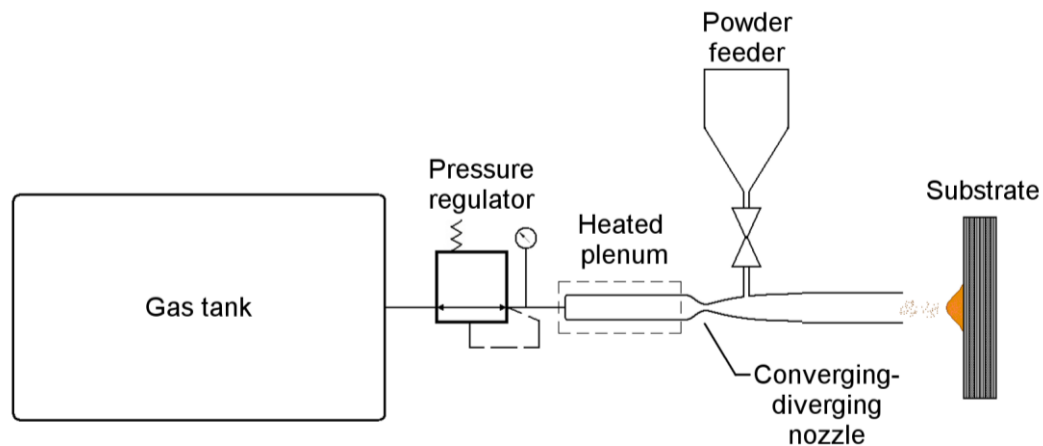


Figure 3.20: Diagram of a LP-CGDS system



Figure 3.21: Centerline LP-CGDS system at the University of Ottawa



Figure 3.22: Inside the LP-CGDS spraying chamber

3.3 Characterisation

Characterisation of the materials was realised through several processes. The methods and apparatus used were separated in three categories: 1) physical, 2) mechanical, and 3) thermal. Physical characterisation was related to inspection of the quality of the materials and to the measurement of their intrinsic properties. It consisted of:

- Visual inspection and microscopy (optical and SEM);
- Porosity measurements;
- Thermal conductivity measurements;
- Differential Scanning Calorimetry (DSC); and
- Thermogravimetric Analysis (TGA).

Mechanical characterisation was related to the mechanical behaviour of the materials under specific loading and temperatures. They were comprised of:

- Hardness measurements;
- Short-span three-point bending; and
- Low frequency cycling.

Thermal characterisation was based on the thermal stability of CPMCs and the temperature distribution in IHCTs when heated. It consisted of:

- Resistance to elevated temperatures; and
- Temperature distribution capability.

3.4 Physical characterisation

3.4.1 Microscopy

Microscopy was performed on PMC laminates, CPMCs and coatings to obtain a preliminary assessment of manufacturing quality. This method was used for investigating the interactions between the materials forming coated composite (i.e. carbon, epoxy, and

coating) and to look at manufacturing defects like voids and delaminations. Three types of microscopes were used:

- 1) Low-magnification stereo-microscope (Ancansco);
- 2) High-magnification metallographic optical microscope (Olympus XJP-3A); and
- 3) Scanning Electron Microscope (EVO MA-10, Carl Zeiss NTS GmbH).

Except when using the stereo-microscope, all samples had to be prepared prior to microscopy. Sample preparation consisted of cutting the samples, mounting and polishing.

The cutting process was essential for obtaining a flat sample surface which was required for appropriate image focus at high magnifications. Cutting was achieved with a precision saw (Secotom-10, Struers) using a 200mm diameter diamond blade turning at 2200rpm with a feed rate varying between 0.02mm/min to 0.10mm/min depending on the thickness of the PMC and coating (Figure 3.23).



Figure 3.23: Secotom-10 precision saw

Cut samples were mounted in a polymer matrix. This provided a stable base for polishing and for focusing during microscopy. Due to the low temperature limit of the PMCs, a cold mounting process was used. The samples were mounted in a two-part epoxy system (supplied by Anamet Inc.). The epoxy resin was mixed with the hardener manually,

using a spatula, with a resin/hardener mass ratio of 7:1. Samples were placed in 25mm diameter mounting cups, after which the mixed epoxy was poured. Samples were then placed in a vacuum chamber for 10min to remove any air entrapped in the epoxy. Cure was done at ambient temperature for 24h. A mounted sample is presented in Figure 3.24.

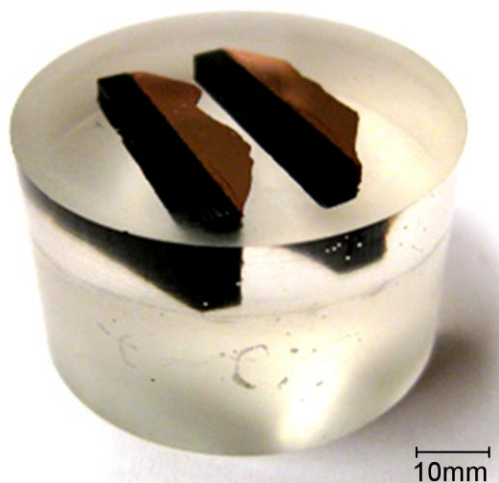


Figure 3.24: Cold mounted CPMC samples

Mounted samples were polished using an automatic polishing machine (Tegrapol-31, Struers) shown in Figure 3.25. The desired sample surface finish was obtained through multiple grinding and polishing operations that sequentially used finer abrasives, up to $0.04\mu\text{m}$ colloidal silica suspension. Between each grinding/polishing operation the sample was cleaned with soapy water to remove impurities, dried with cold air and inspected with a stereo-microscope to verify surface quality. If the sample passed inspection, e.g., adequate size of remaining scratches, then the next polishing operation was performed. Most surface preparations in this study were made following the procedure used for analysing copper coatings, presented in Table 3.8.

Table 3.8: Polishing procedure for copper coatings

Step	Polishing surface	Abrasive	Lubricant	Polishing time (min)
1)	MD-Piano #220 (Struers)	None	Water	1
2)	#500 SiC-Paper	None	Water	1
3)	MD-Largo (Struers)	9 μ m diamond suspension	None	4
4)	MD-Mol (Struers)	1 μ m diamond suspension	None	3
5)	MD-Chem (Struers)	0.04 μ m silica suspension	None	2
6)	MD-Chem (Struers)	None	Water	1



Figure 3.25: Tegrapol-31 automatic polishing machine

For SEM, a thin layer of gold was applied to the surface of samples, aiming at dissipating the electric charge caused by impinging electrons. The fine conductive layer was applied using a sputtering machine (Denton Vacuum, Desk IV).

After surface preparation the samples were observed using the various microscopes and pictures taken were analysed using an image analysis software. Pictures from the

stereo- and high magnification microscopes were taken using a digital camera (Clemex, 3 megapixels) while SEM images were re-constructed with the machines' own software and equipment.

3.4.2 Image analysis and porosity measurements

Image analysis of pictures taken with the microscopes was performed using the Clemex Vision Lite software. The software was pre-calibrated for calculating the lengths from point-to-point on an image depending on picture magnification, and the software was capable of area analysis based on contrast threshold. Length measurements were made for assessing the thickness of the various phases, such as coating or PMC ply thickness, and also particle size. Area analysis was used mostly for estimating the porosity of a coating, but also for estimating the copper volume fraction in the co-cured layer.

Porosity and volume fraction measurements were made through contrast analysis. This consisted in filtering the darker zones of a picture, notably the pores/voids, based on a brightness contrast threshold defined by the user. A porosity value ϕ was obtained by comparing the image area below the contrast threshold $A_{\text{Threshold}}$ to the total area of the image A_{Picture} :

$$\phi = \frac{A_{\text{Threshold}}}{A_{\text{Picture}}} \quad (3.4)$$

Porosity measurements were made at a magnification of 400X. Minimising the estimation error on porosity was achieved by taking at least 15 measurements located arbitrarily over the entire coating (centre, surface and substrate-to-coating interface). However, it is important to note that the porosity measurement method is dependent on the threshold selection made by the user, and therefore it cannot provide an exact measurement of porosity. Instead the method should be viewed as a comparison tool only.

3.4.3 Thermal conductivity measurements

In-plane and transverse thermal conductivities of the materials were measured with THISYS and THASYS instruments from Hukseflux Inc. respectively, at NRC-IAR in Ottawa, with the assistance of Research Council Officer Simon Hind. Both devices were designed for measuring the thermal conductivity of thin plates that are 70mm wide and 110mm long, with a thickness between 0.1mm to 6mm. Unfortunately, high conductivity samples such as copper coatings cannot be tested using the instruments due to sensor precision [126]. Nevertheless, the devices can measure both the in-plane and transverse conductivities of PMCs, as shown by Hind et al. [55].

In this section, a description of the in-plane and transverse thermal conductivities measuring apparatuses is provided. Additionally, thermal conductivity results for PMCs and CPMC substrates are presented.

3.4.3.1 In-plane thermal conductivity measurements

In-plane thermal conductivity was measured with the THISYS instrument (Figure 3.26). The apparatus heats a thin plate at the centre of its faces and measures the heat flowing to its edges. This is achieved by placing a single sample between two sensor blocks. The assembly is immersed in glycol, reducing contact resistance. Each sensor block consists of a small central heater and heat sink. The heater is insulated from the heat sink by a cavity filled with air, which forces heat to flow into the sample. The heat flows from the centre of the sample to its edges where it is absorbed by the aluminium heat sink of the sensor block. Heat is then guided towards a thermopile. The difference in temperature between the heater and thermopile can be converted into an in-plane thermal conductivity. Each measurement is conducted for 1 hour for ensuring steady-state. The measurements obtained from the apparatus have a reported accuracy of $\pm 6\%$ [126].

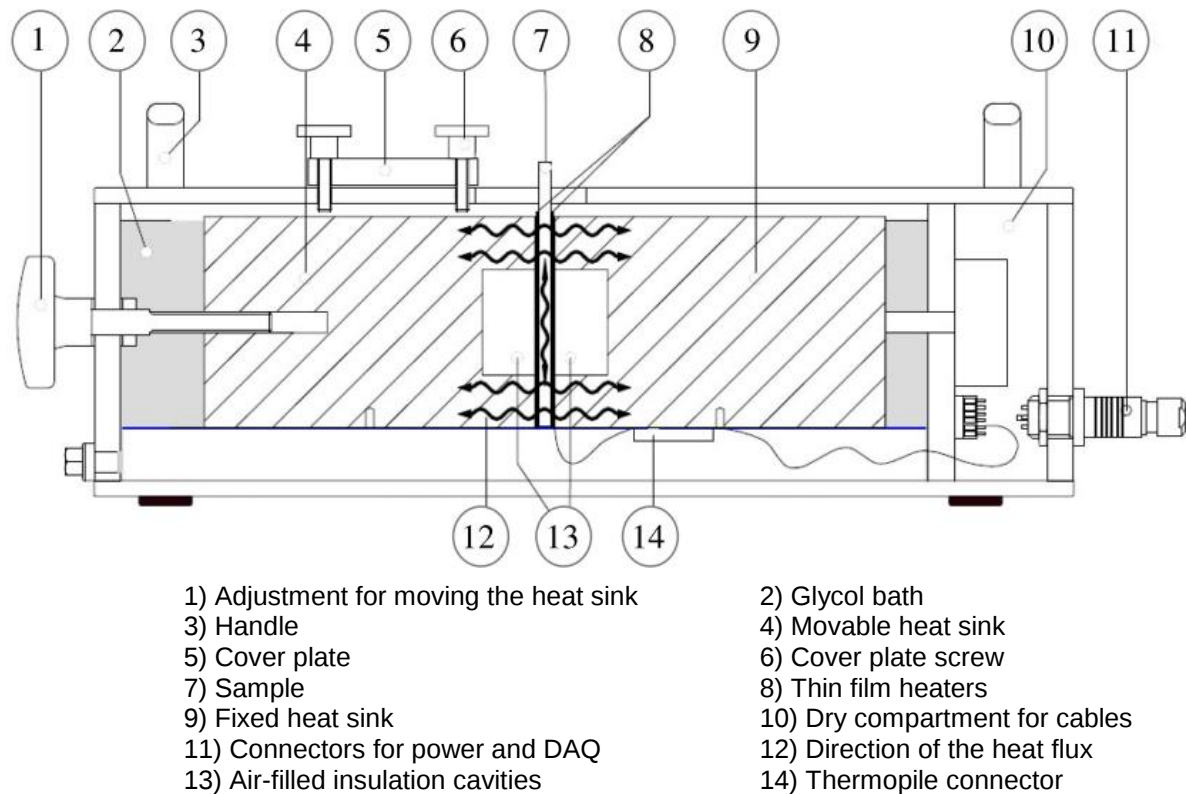


Figure 3.26: Diagram of THASYS instrument for in-plane thermal conductivity measurement [126]

3.4.3.2 Transverse thermal conductivity measurements

Transverse thermal conductivity was measured with the THASYS instrument (Figure 3.27), which complies with the ASTM 1114-98 standard. The operation of the apparatus consists in heating the surface of a thin plate and measuring the heat flowing through the thickness. This is achieved by placing two identical samples in the apparatus, with a thin film heater inserted between them, and securing the samples between two sensor blocks. Heat flows from the heater into the samples, and then into the sensor blocks. The aluminium sensor blocks act as a heat sink and direct heat towards a thermopile. The difference in temperature between the heater and thermopile can be converted into a transverse thermal conductivity. Similar to the THASYS, the apparatus is filled with glycol to reduce contact resistances between the samples and sensor blocks. Each thermal conductivity measurement was conducted for 1 hour to ensure steady-state. The measurements obtained from the apparatus have a reported accuracy of $\pm 3\%$ [127].

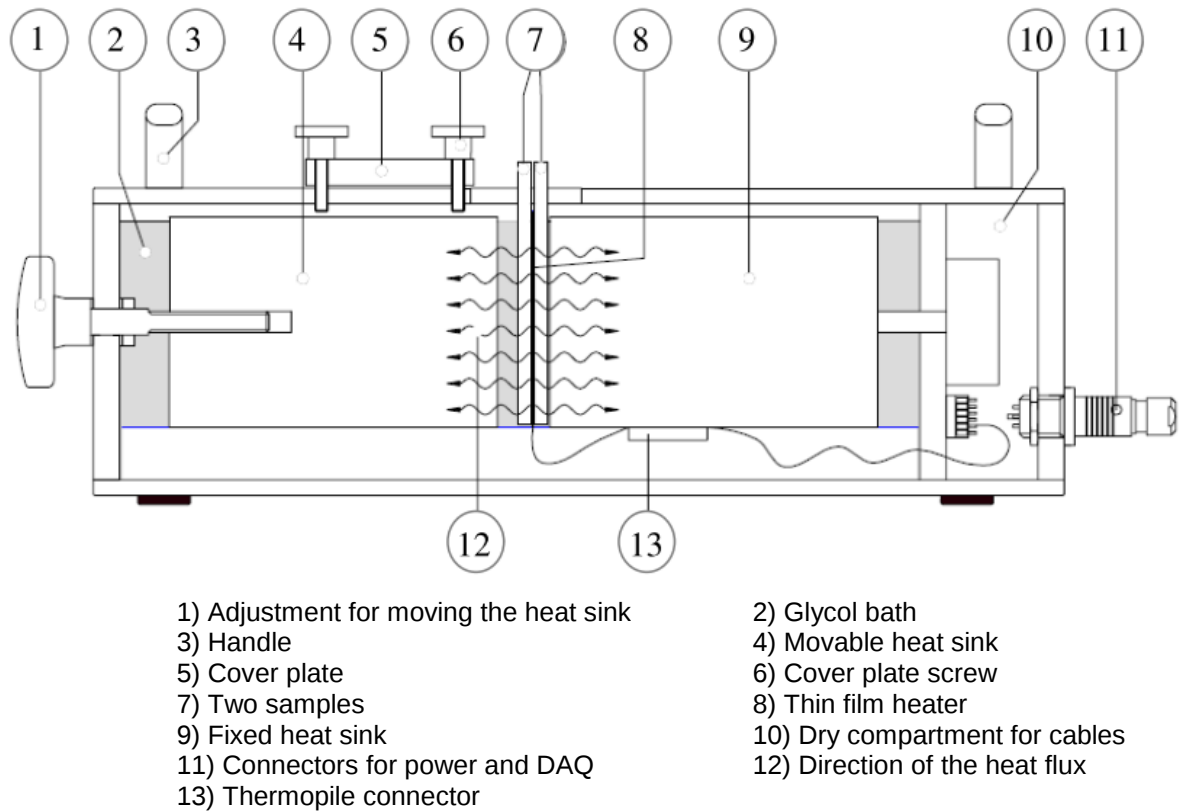


Figure 3.27: Diagram of THASYS instrument for transverse thermal conductivity measurement [104]

3.4.3.3 Thermal conductivity experiment and results

In-plane and transverse thermal conductivities of SE-70 PMCs and CPMC substrates were measured using THISYS and THASYS. One plate measuring 300mm by 300mm was manufactured for each material, for generating thermal conductivity samples. All plates were made of 12 plies of SE-70 prepreg. For thermal conductivity measurements, 4 samples (#1, #2, #3 and #4) were cut from each plate (Figure 3.28). The SE-70 PMC samples had smooth surfaces whereas the CPMC substrate samples had a smooth surface on the PMC side and a rough co-cured surface due to exposed copper particles. The average thickness for each sample is presented in Table 3.9. The thickness was obtained by averaging 8 measurements taken with a digital calliper. Unsurprisingly the CPMC substrate showed more variability in thickness due to the roughness of the co-cured layer.

Table 3.9: Thickness measurement of thermal conductivity samples

Sample #	Thickness (mm)			
	SE-70 laminate		SE-70 laminate with co-cured layer	
	Average	Standard deviation	Average	Standard deviation
1	2.58	± 0.03	2.98	± 0.12
2	2.63	± 0.01	3.01	± 0.11
3	2.55	± 0.07	3.01	± 0.09
4	2.55	± 0.05	3.01	± 0.10
Average	2.57	± 0.04	3.00	± 0.11

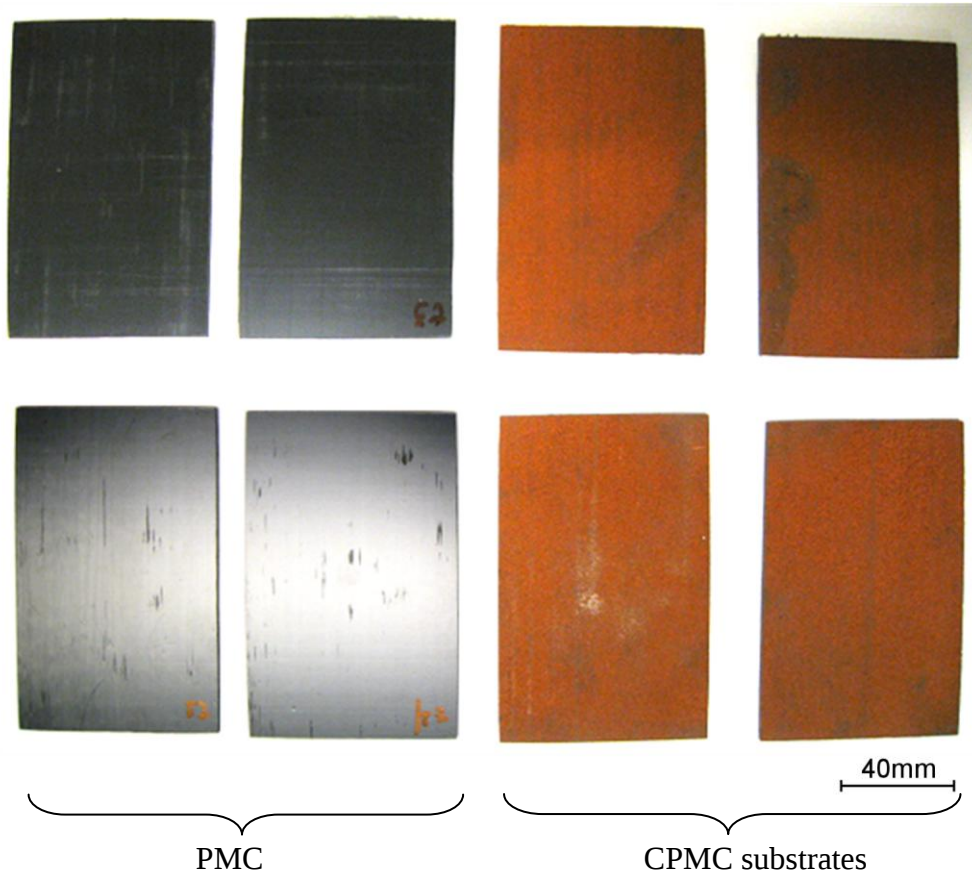


Figure 3.28: Thermal conductivity samples

In-plane conductivity data are presented in Table 3.10. Similar variability was observed between the PMCs and the CPMC substrates. The effect of the uneven surface on CPMC substrates seemed to be mitigated by the glycol. Moreover, values for the SE-70

laminate were comparable to those of similar cross-ply carbon/epoxy PMCs [55], suggesting that the measuring method was adequate. For the CPMC substrates, the measured in-plane thermal conductivity was slightly lower. Thus, the in-plane thermal conductivity for copper particles embedded in epoxy is lower than that of a SE-70 cross-ply PMC. The CPMC substrate was also tested for directionality of the heat flow flipping the sample by 180° (i.e., having the co-cured layer facing the opposite direction). Measurements indicated marginal difference, so samples can be tested on either surface.

Table 3.10: In-plane thermal conductivity data

Material	Sample #	In-plane thermal conductivity (W/m·K)				
		Trial #1	Trial #2	Trial #3	Average	Standard deviation
PMC: cross-ply SE-70	1	2.35	2.32	2.32	2.33	0.02
	2	2.26	2.27	2.26	2.26	0.01
	3	2.34	2.36	2.34	2.35	0.01
	4	2.34	2.33	2.34	2.34	0.01
	Average				2.32	0.04
CPMC substrate: cross-ply SE-70 with co-cured layer	1	2.25 (2.30)	2.25 (2.30)	2.25 (2.31)	2.28	0.03
	2	2.25	2.25	2.25	2.25	0.00
	3	2.22	2.22	2.22	2.22	0.00
	4	2.27	2.29	2.29	2.28	0.01
	Average				2.26	0.03

Note: Values in brackets are for specimens that were flipped 180°

Transverse conductivity data are presented in Table 3.11. Lower variability was observed compared with in-plane measurements, even when flipping the CPMC by 180° . The only exception is the presence of an outlier that occurred for the second measurement, which used SE-70 PMC samples #3 and #4. Nevertheless, values for the SE-70 were still comparable to those of similar cross-ply carbon/epoxy PMCs [55]. Results also showed that the transverse thermal conductivity of the co-cured layer was higher than the transverse thermal conductivity of the SE-70 laminate. Hence, from the in-plane and

transverse measurements, the thermal conductivity values of the thermally isotropic co-cured layer should be between 0.57W/m·K and 2.26W/m·K. This is much lower than the 401W/m·K of pure copper as the epoxy prevents direct contact between copper particles. Hence, the coating will be vital for improving the thermal conductivity.

Table 3.11: Transverse thermal conductivity data

Material	Samples #	Transverse thermal conductivity (W/m·K)				
		Trial #1	Trial #2	Trial #3	Average	Standard deviation
PMC: cross-ply SE-70	1 & 2	0.45	0.45	0.45	0.45	0.00
	2 & 3	0.47	0.47	0.47	0.47	0.00
	3 & 4	0.49	0.85 [Outlier]	0.49	0.61 [0.49]	0.21 [0.00]
	Average				0.51 [0.47]	0.09 [0.02]
CPMC substrate: cross-ply SE-70 with co-cured layer	1 & 2	0.57 (0.56)	0.57 (0.56)	0.57 (0.56)	0.57	0.01
	2 & 3	0.57	0.58	0.56	0.57	0.01
	3 & 4	0.56	0.57	0.57	0.57	0.01
	Average				0.57	0.01

Note: 1) Values in brackets () are for specimens that were flipped 180°
2) Values in bracket [] do not consider the outlier of measurement #2, for SE-70 sample #3 & #4

3.4.3.4 Thermal conductivity of the co-cured layer

For modelling purposes, a more precise thermal conductivity value for the co-cured layer was required. It was estimated from both the in-plane and transverse measurements above. To achieve this, thermal conductivity equations for the CPMC substrates were developed based on the electrical circuit analogy for solving 1D heat transfer problems. The analogy states that the thermal resistance is equal to:

$$R_{th} = \frac{l}{kA} \quad (3.5)$$

where R_{th} is the thermal resistance, l is the length of material parallel to the heat flow, k is the thermal conductivity and A is the surface area.

For the in-plane scenario (Figure 3.29), the total resistance is:

$$R_{th, Total} = \frac{R_{th, Co-cured} R_{th, PMC}}{R_{th, Co-cured} + R_{th, PMC}} \quad (3.6)$$

where the subscripts are defined as such: _{Co-cured} represents the co-cured layer, _{PMC} is the SE-70 layer, and _{Total} is the combined co-cured and SE-70 layers. Considering that heat travels the same distance in both media and that the width is identical, the equation is only dependent on the thermal conductivities of the media and area A , which is proportional to the thicknesses h of the media ($A \propto t$). Equation (3.6) can be simplified using Equation (3.5) to yield the thermal conductivity of the co-cured layer:

$$k_{Co-cured} = \frac{k_{Total} - k_{PMC}}{1 - \frac{h_{PMC}}{h_{Total}}} \quad (3.7)$$

Similarly, for the transverse scenario (Figure 3.29), the total resistance is:

$$R_{th, Total} = R_{th, Co-cured} + R_{th, PMC} \quad (3.8)$$

In this case, the total resistance is dependent on the distance travelled by heat in a medium which is equivalent to medium thickness ($l = t$), and on the thermal conductivities. Since the area does not change it can be ignored and Equation (3.8) can be reduced to:

$$k_{Co-cured} = \frac{\left(k_{Total} k_{PMC} \left(1 - \frac{h_{PMC}}{h_{Total}} \right) \right)}{k_{PMC} - k_{Total} \frac{h_{PMC}}{h_{Total}}} \quad (3.9)$$

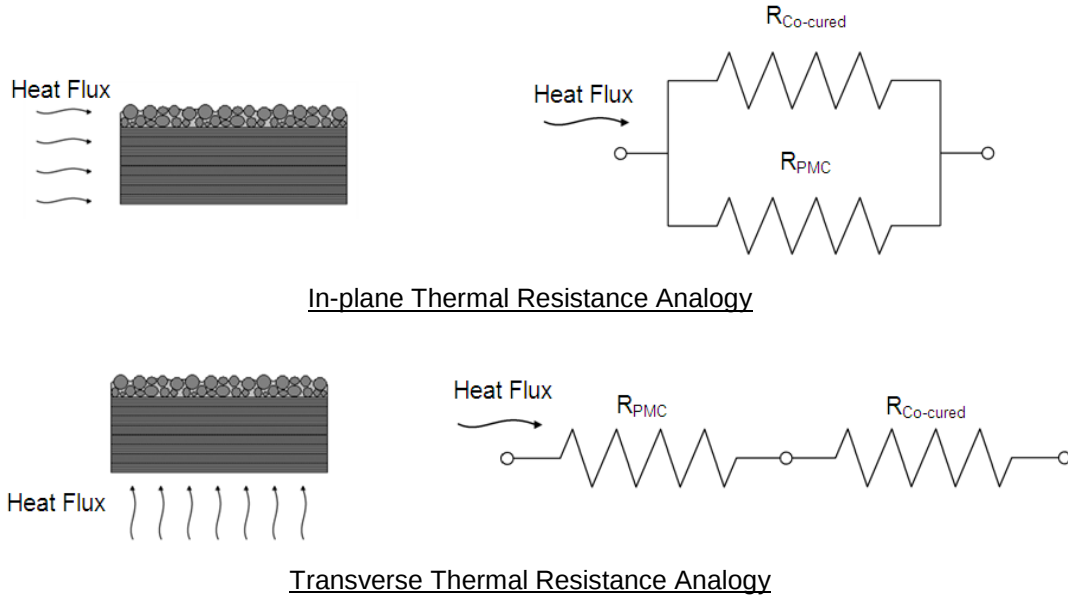


Figure 3.29: Electrical circuit analogy for the in-plane (above) and transverse (below) thermal conductivity of the CPMC substrate

Equations for the thermal conductivities of the co-cured layer can be solved with the measured values of the SE-70 and of the CPMC substrate, if the thicknesses of the layers are known. In this case there was insufficient information regarding the actual thickness ratio of the layers. However, this can be addressed as the co-cured layer should be isotropic, being composed of randomly dispersed copper spheres in an isotropic matrix, meaning that the transverse and in-plane conductivities should be equal. An estimated value of the thermal conductivity was thus obtained by plotting both equations of thermal conductivities based on the thickness ratio $t_{\text{PMC}}/t_{\text{Total}}$ and finding the intersection of the curves. From the plot (Figure 3.30) it can be seen that thermal conductivities are equal when the PMC composes 77% of the material, and equate to 2.06W/m·K.

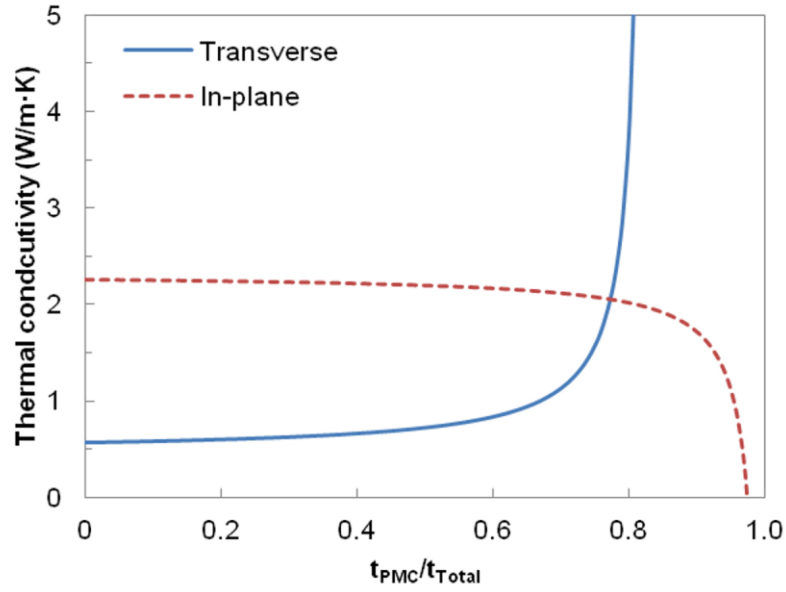


Figure 3.30: Estimation of the in-plane and transverse thermal conductivities of the co-cured layer based on the thickness ratio of the PMC layer

An estimate of the copper content of the co-cured layer can be obtained from the Lewis-Nielsen model for thermal conductivity of randomly packed spheres in a continuous matrix as presented in section 2.3.2.1. Using the material properties presented in section 3.1, a value of 1.5 for constant A and a maximum random packing density ϕ_m for spheres of 0.64 [105, 106], a copper volume content of 55% is obtained. If this is representative of the reality, maximum packing density was not reached even with the help of the vibrating table. This will be further discussed in section 4.1.

3.4.4 Thermogravimetric analysis

TGA was used for characterising the degradation temperature T_d of epoxy, i.e. the theoretical maximum temperature that the material can withstand. It was performed to assess the temperature limits of PMCs for kinetic spray processes.

TGA consists in analysing the change in weight of a material due to a change in temperature. At such temperature, chemical chains of the epoxy are severed, creating volatiles that leave the bulk material. This translates in an important drop in weight as depicted in Figure 3.31. The degradation temperatures reported were obtained from the intersection of a line tangent to the weight loss curve before any noticeable drop in weight was observed with the line tangent to the fastest weight loss rate.

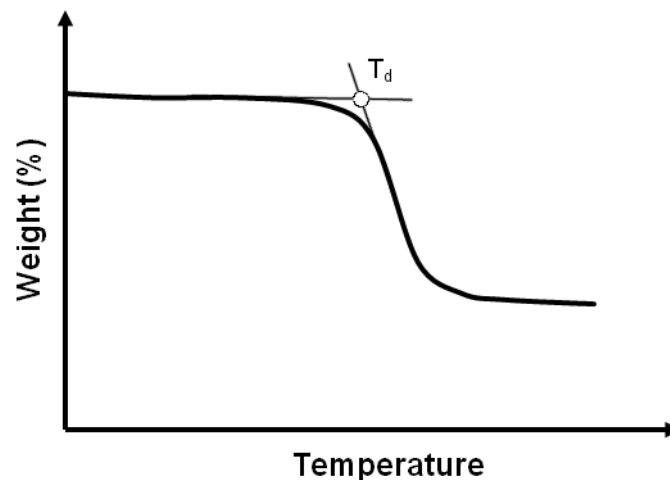


Figure 3.31: Representative TGA curve with method for obtaining the degradation temperature

Tests were conducted by NRC-AMTC in Montreal by technician Hugo Laurin using a TGA Q500 (TA Instruments) with a heating ramp of 10°C/min. A nitrogen purge gas with a flow rate of 50ml/min was used for preventing oxidation.

TGA was performed for a single sample of cured SE-70 and a single sample of cured SA-70. Results for both samples are presented in Table 3.12. The TGA curve for the SE-70

is presented in Figure 3.32, while the curve for the SA-70 is presented in Figure 3.33. The results indicate that the degradation temperature for both epoxy systems occurs around 400°C.

Table 3.12: TGA results for SE-70 and SA-70

Material	Initial mass (mg)	Degradation temperature (°C)	Weight loss (%)
SE-70	11.070	395.3	30.03
SA-70	10.012	398.7	52.89

From the TGA curves, it is interesting to note that significant weight loss begins near 300°C, which would be the onset of degradation of the materials. Any weight loss (~2%) occurring before 300°C is likely moisture evaporation as reported in [113]. It can also be noted that the SA-70 loses more weight than the SE-70. This difference is mostly attributable to the presence of carbon fibres in the SE-70 compared to the nearly neat resin of the SA-70.

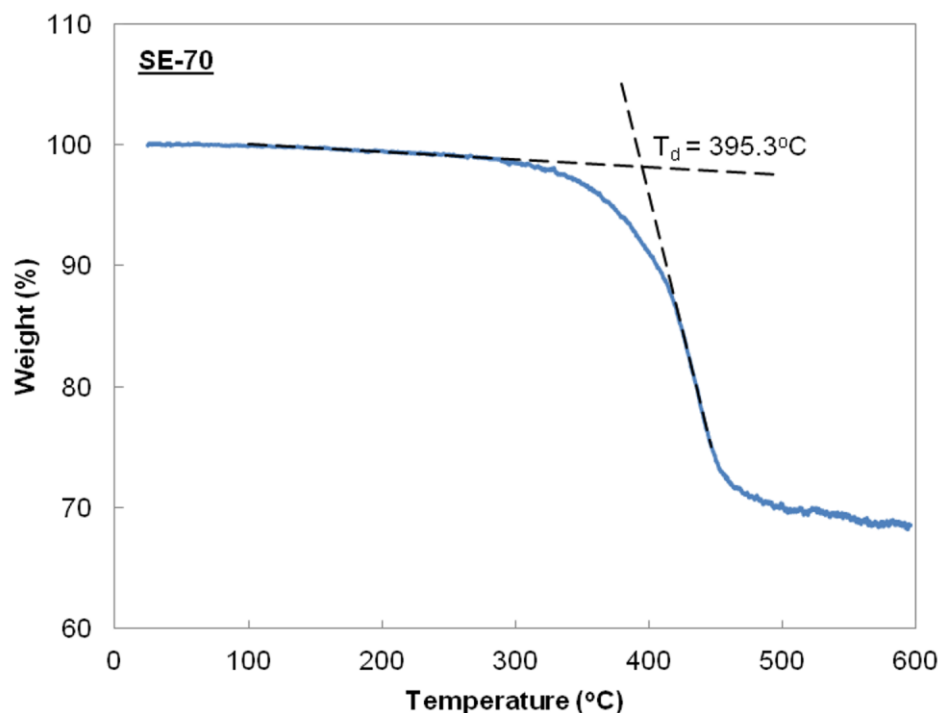


Figure 3.32: TGA curve for SE-70

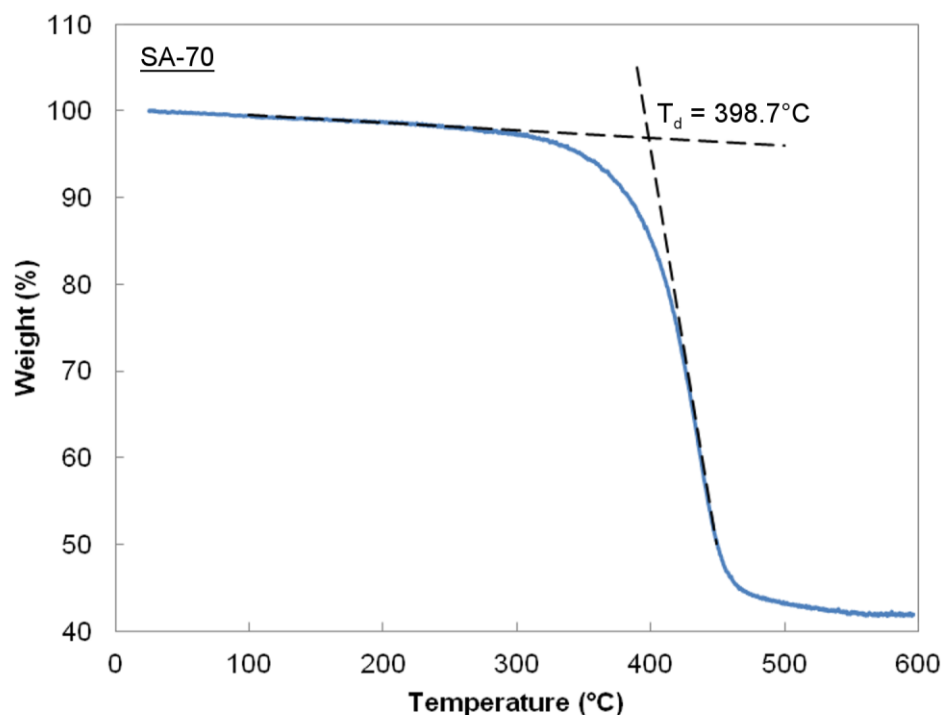


Figure 3.33: TGA curve for SA-70

3.4.5 Differential scanning calorimetry

DSC was used for characterising the specific heat capacity at constant pressure C_p of the materials as a function of temperature, and to estimate the glass transition temperature T_g of the resins. Specific heat capacity data provides useful information regarding the energy required to heat the material, which is essential for modelling heat transfer. Conversely, T_g gives the operational temperature limit of the PMC for structural applications.

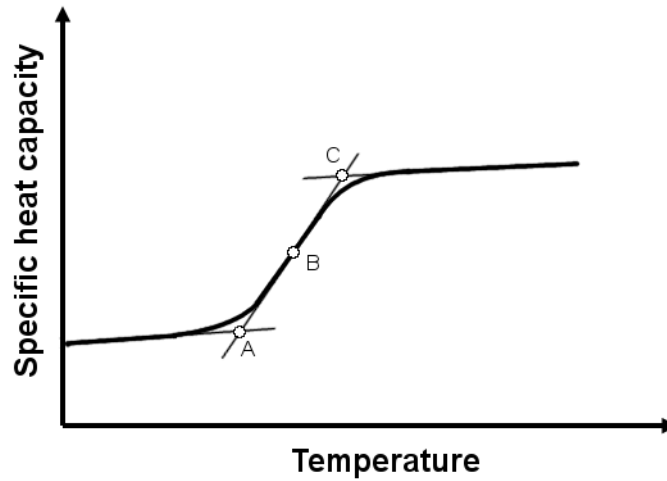
A DSC experiment for obtaining the specific heat capacity consists in measuring the difference of heat that is absorbed or released by a sample compared to an inert reference with a known specific heat capacity during non-isothermal heating. Before testing, the sample is encapsulated inside a sealed aluminium pan to prevent volatiles from escaping. During the test, the heat flux required for heating the sample is governed by:

$$\frac{dQ}{dt} = mC_p \frac{dT}{dt} + H_{T,t} \quad (3.10)$$

where dQ/dt is the heat flow, m is the sample mass, dT/dt is the rate of change in temperature, and $H_{T,t}$ is the exothermic or endothermic reactions occurring inside the samples. If there are no chemical reactions, the heat flux becomes solely dependent on the mass of the sample, on the heating rate and on the specific heat capacity. It should be noted that the mass of the sample and heat rate have a significant impact on measurement accuracy. Hartmann et al. suggested using a heating rate of 10-20°C/min [128], while J. McHugh et al. [129] opine that the optimal mass of the sample should be:

$$m_{\text{sample}} = \frac{m_{\text{reference}} C_{p, \text{reference}}}{C_{p, \text{sample}}} \quad (3.11)$$

The glass transition temperature T_g can be obtained from the specific heat capacity curves of a polymer, by looking at any step increase as seen in Figure 3.34. In this study, the glass transition temperature is taken as the inflection point of the step (point B).



A: Onset B: Inflection point C: Endset

Figure 3.34: Standard glass transition range

3.4.5.1 DSC experiment

DSC tests were undertaken by NRC-AMTC in Montreal by technician Hugo Laurin using a Q1000 (TA Instruments) with a heating ramp of 10°C/min and nitrogen as purge gas with a flow rate of 50ml/min. Cross-ply SE-70 prepreg and SA-70 samples both uncured and cured were tested. Samples masses are given in Table 3.13. The samples were heated from room temperature to 150°C; then they were cooled to room temperature and heated to 250°C.

Table 3.13: Mass of samples used in DSC testing

Sample	Mass (mg)
SA-70 uncured	57.8
SA-70 cured	5.8
SE-70 uncured	11.3
SE-70 cured	11.1

3.4.5.2 DSC results

Results obtained for the resin cannot be compared easily with literature data as the thermal properties of the epoxy is strongly dependent on parameters such as the type of resin, type of hardener, curing time, the curing temperature, and cross-linking density[128, 130].

Specific heat capacity curves for uncured SA-70 and SE-70 samples are shown in Figures 3.35-3.36, respectively. In both cases, a great exotherm from the curing reaction was observed on the first cycle to 150°C, with onset of the reaction appearing around 80°C. Due to the similarities observed between the resins of SA-70 and SE-70, it was expected that the exotherm would be greater in SA-70 than in SE-70 as in the prior the mass is all epoxy as opposed to the latter which contains inert carbon fibre, but this was not the case. It is proposed that an error was could have occurred during measurement of the mass of the SA-70 sample, affecting the magnitude of the specific heat capacity. This was conjectured

as a 57.8mg sample seems large. Hence, uncured SA-70 results were only used in commenting trends seen between cycles.

The first cycle results also show that cure was incomplete at the end of the cycle. Moreover, it can be seen that the heat capacity curve goes down with each successive cycle which may indicate an increase of the degree of cure as reported by [115].

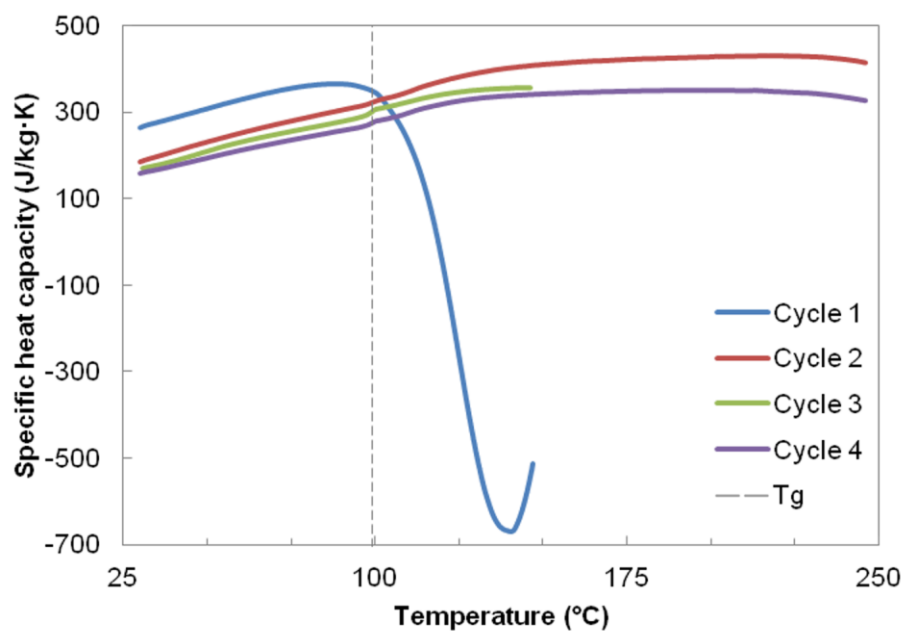


Figure 3.35: Specific heat capacity results for uncured SA-70

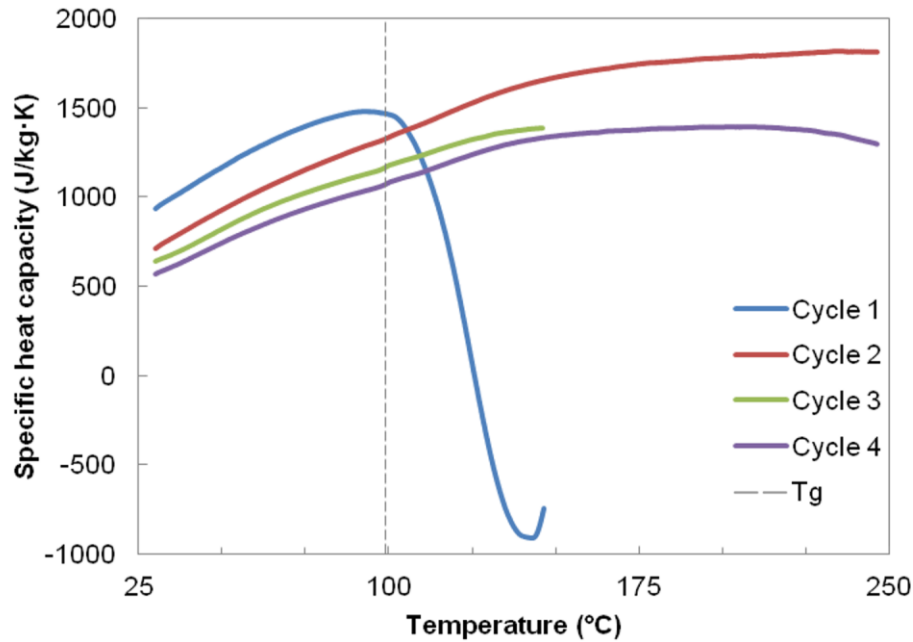


Figure 3.36: Specific heat capacity results for uncured SE-70

Specific heat capacity curves for the cured SA-70 and SE-70 samples are shown in Figures 3.37-3.38, respectively. In contrary to previous results, no exotherm was observed because the samples were already cured. Interestingly, the specific heat went down with each successive cycle, suggesting incomplete cure. However, it is questioned whether this can be attributed to incomplete cure only. Results for the SE-70 appear to be close to those of the DSC-cured sample, albeit with slightly higher ($\sim 20\%$) specific heat capacity which may be attributed to different cure conditions. As for results of SA-70 samples, they appear appropriate as the specific heat capacity values were $\sim 25\%$ greater than those of the SE-70, which is caused by the lower specific heat capacity of the reinforcement in the latter ($740\text{J/kg}\cdot\text{K}$) compared to the resin ($\sim 1000\text{J/kg}\cdot\text{K}$ at 25°C and $\sim 2000\text{J/kg}\cdot\text{K}$ at 120°C). It is also interesting to see that below the glass transition temperature the specific heat capacity of the epoxies increased nearly linearly with temperature. After crossing that temperature the heat capacity increased at a slower rate and even went down with a further increase in temperature.

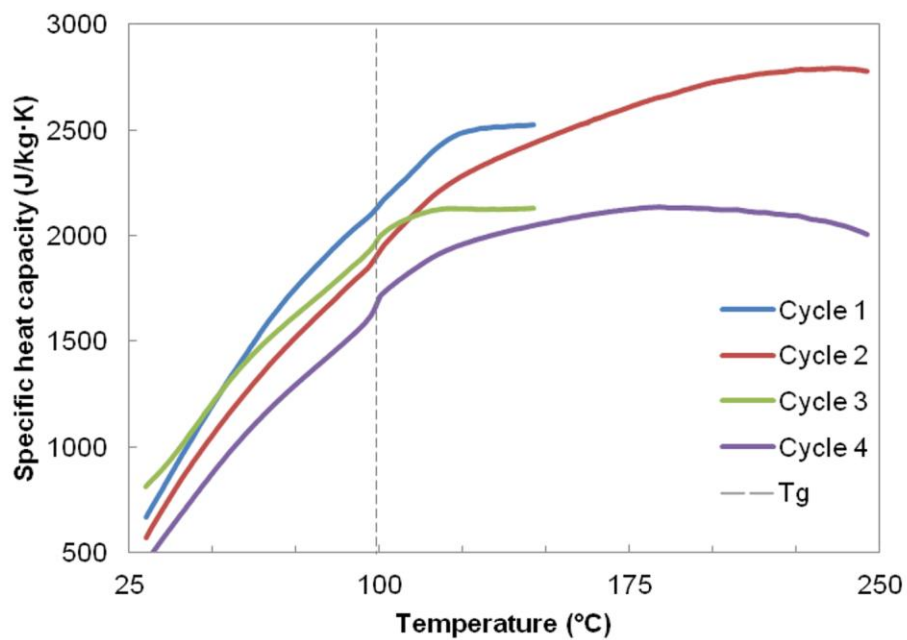


Figure 3.37: Specific heat capacity results for cured SA-70

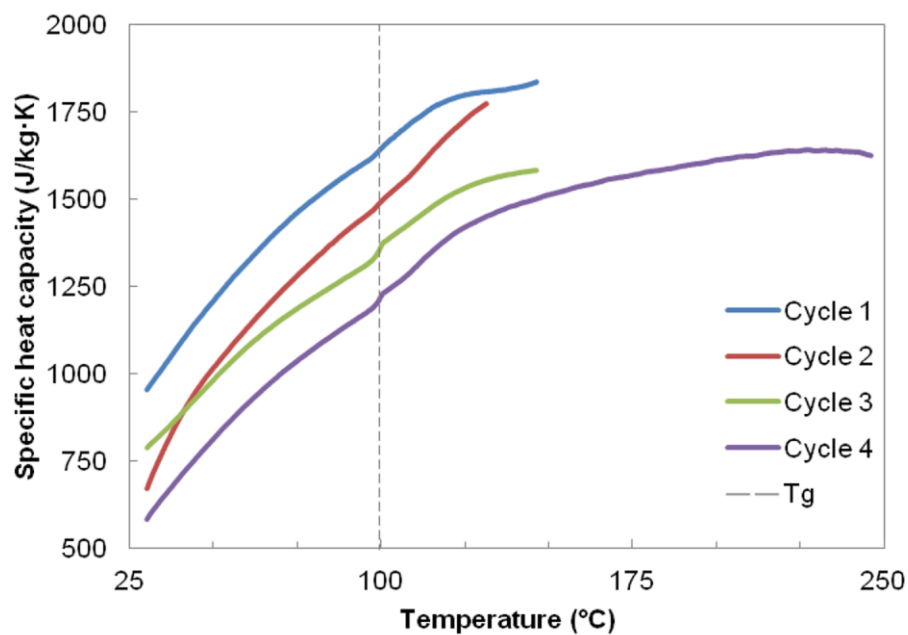


Figure 3.38: Specific heat capacity results for cured SE-70

The glass transition temperature T_g of the samples was obtained from the inflection point of the step increase in specific heat capacity. The heat capacity step increase for the samples was small and it was difficult to see on the previous plots, but it could be clearly seen on the re-scaled plot of the cured SE-70 (Figure 3.39). Results indicated that the magnitude of the step increased with the number of cycles, suggesting that there was not an increase in the degree of cure, as the difference in specific heat capacity at the glass transition is known to reduce with the degree of cure [131]. Nevertheless, the glass transition temperature for all cases was estimated at around 99°C (Table 3.14). This clearly indicated that the SA-70 and SE-70 materials used highly similar epoxies, or the same epoxy.

Table 3.14: Glass transition temperatures for SA-70 and SE-70

Sample	Glass transition temperature (°C)
SA-70 DSC-cured	99.0
SA-70 pre-cured	99.0
SE-70 DSC-cured	99.0
SE-70 pre-cured	99.5

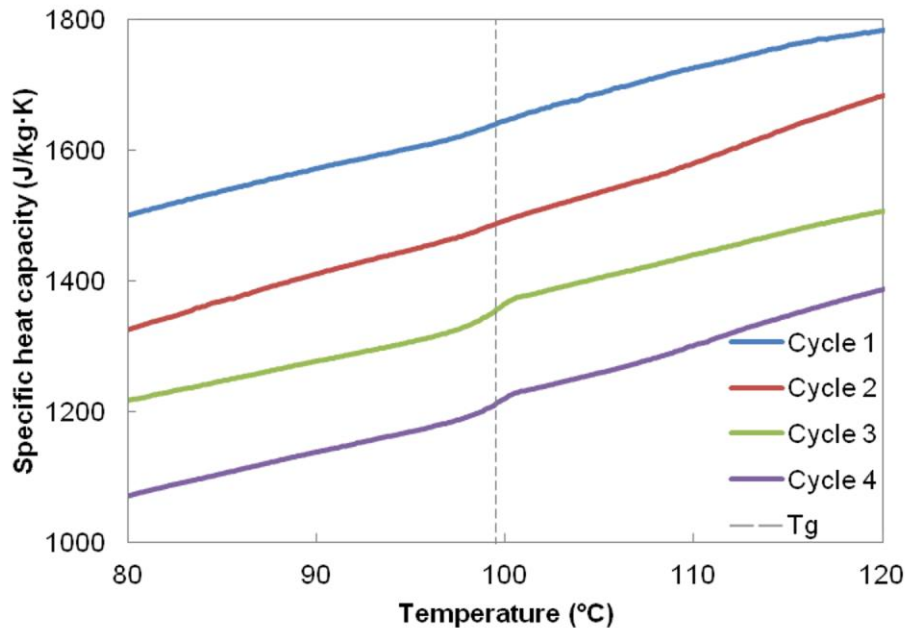


Figure 3.39: Glass transition temperature for cured SE-70

For the purpose of heat transfer modelling, the specific heat capacity of the materials was estimated in the 30°C to 120°C range, which is within the typical processing temperatures of comparable materials. Considering that there is limited change in specific heat capacity across the glass transition, a linear regression could be used in deriving a first approximation. The regression was performed for oven-cured samples for their first and last DSC cycles. Cycle #1 one was selected as it followed the actual cure recommendations while cycle #4 was selected due to its higher degree of cure. The regressions are plotted in Figures 3.40-3.41. The resulting specific heat capacity models are:

$$\text{Cycle \#1:} \quad C_{p, \text{SA-70}} = 19.403T - 5074 \text{ J/kg} \cdot K \quad (\text{For } 300\text{K} < T < 390\text{K}) \quad (3.12)$$

$$C_{p, \text{SE-70}} = 9.0147T - 1709 \text{ J/kg} \cdot K \quad (\text{For } 300\text{K} < T < 390\text{K}) \quad (3.13)$$

$$\text{Cycle \#4:} \quad C_{p, \text{SA-70}} = 16.301T - 4401 \text{ J/kg} \cdot K \quad (\text{For } 300\text{K} < T < 390\text{K}) \quad (3.14)$$

$$C_{p, \text{SE-70}} = 8.4726T - 1932 \text{ J/kg} \cdot K \quad (\text{For } 300\text{K} < T < 390\text{K}) \quad (3.15)$$

where T is the temperature in Kelvin.

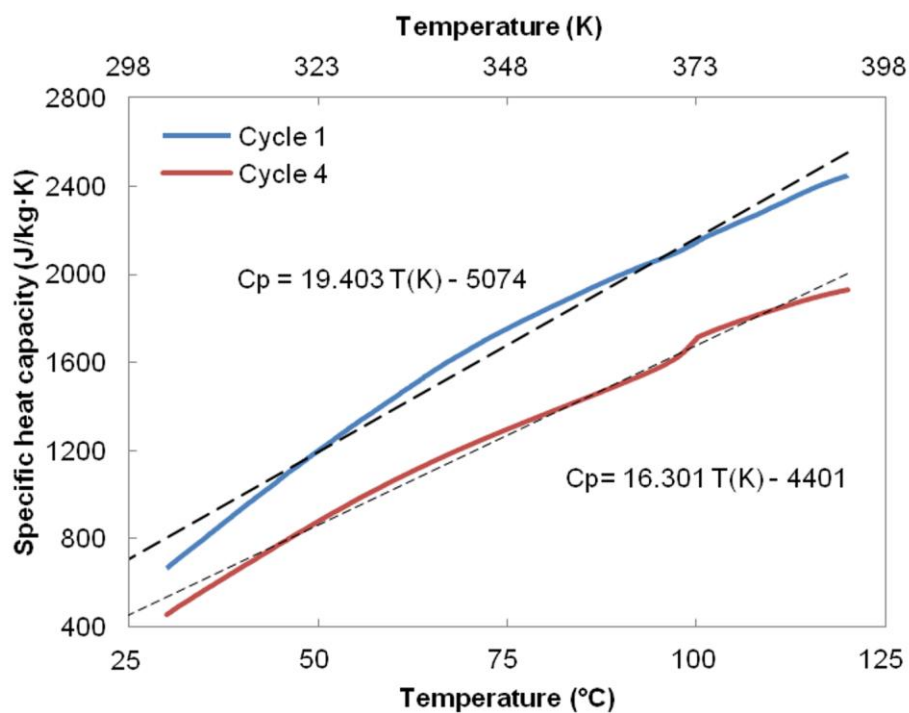


Figure 3.40: SA-70, linear regression of specific heat capacity in the 30°C to 120°C range for cycles #1 and #4

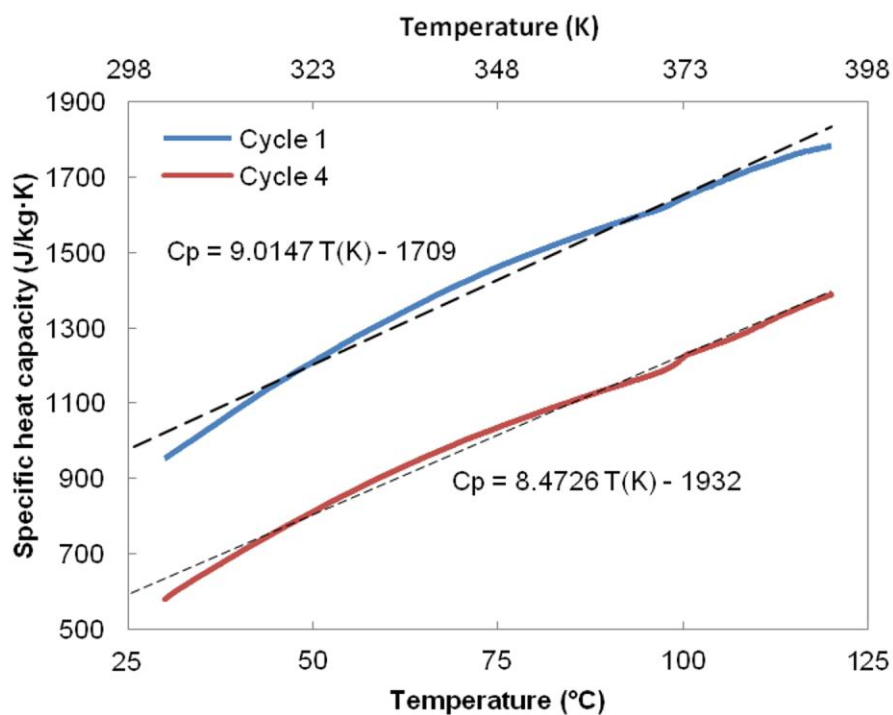


Figure 3.41: SE-70, linear regression of the specific heat capacity in the 30°C to 120°C range for DSC cycles #1 and #4

Another indication of similarities between the resin systems was obtained by calculating the specific heat capacity of the SA-70 of cycle #4 with a fabric reinforcement, using the rule of mixture and the previous specific heat capacity equation, Equation (2.17). Combining Pyrofil TR50S carbon fibres (740J/kg·K) at a fibre volume fraction of 38% with the SA-70 which is nearly neat resin, gave a specific heat capacity curve that matched measured data for the SE-70 perfectly (Figure 3.42). It should be noted that the low fibre volume fraction may be due to the local composition of the small DSC specimens, and that the equations can be used for estimating cases with different fibre volume fractions.

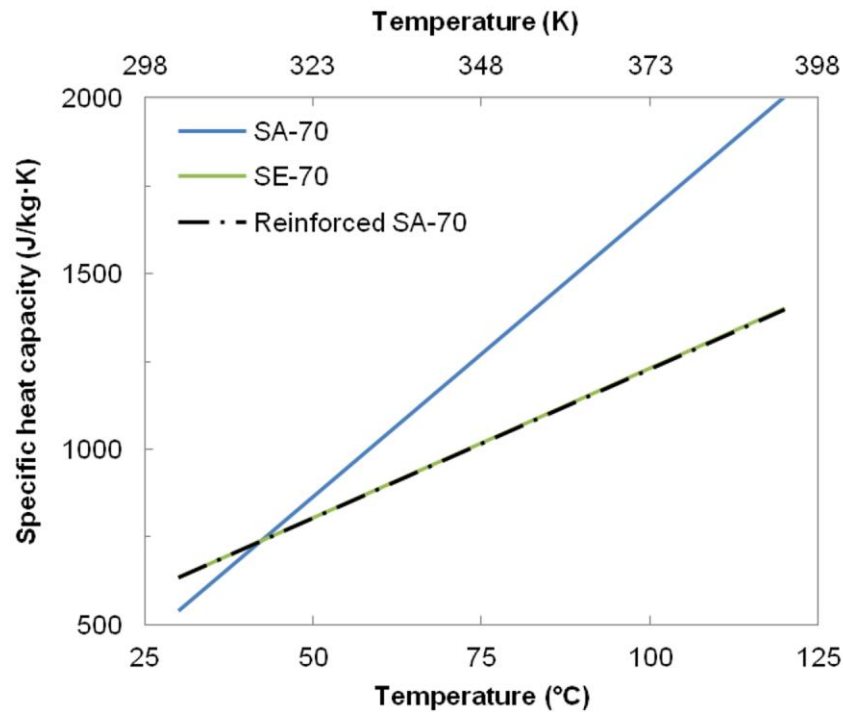


Figure 3.42: Comparison of the estimated specific heat capacity curves of the SA-70, SE-70 and reinforced SA-70 ($v_f = 38\%$) based on DSC cycle #4 data

3.5 Mechanical characterisation

3.5.1 Micro-hardness measurements

CPMC copper coating hardness was measured by micro-indentation using a Duramin micro-Vickers tester (Figure 3.43). Testing consisted in applying a prescribed load on a flat material surface through a diamond shape indenter having a face angle of 136° . All tests were performed with a load of 0.1kgf and an indentation time of 10s. An impression of the indenter was left on the material surface (Figure 3.44). The size of the impression depends on level of plastic deformation that occurred in the material, which is related to material hardness. The micro-Vickers hardness (HV) is calculated from the length of the two diagonals of the diamond shape impression using the following equation [132]:

$$HV_{\text{Vickers}} = \frac{1.8544L}{d^2} \quad (3.16)$$

where L is the load in kilogram force and d is taken as the average of the two diagonal lengths. Interference between measurements is prevented by making indentations at least 5 diagonal lengths from away each other.

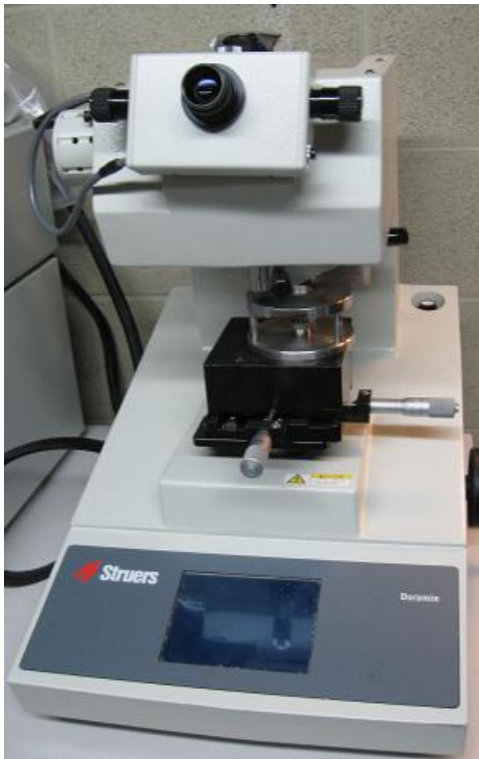


Figure 3.43: Micro-Vickers hardness testing machine (left) with indenter (right)

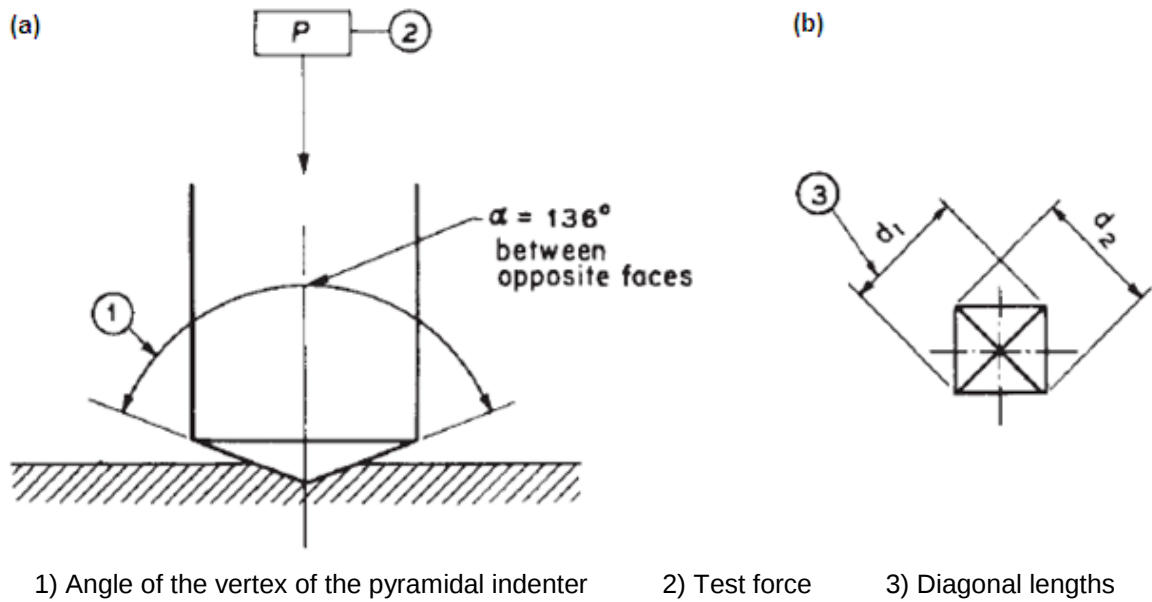


Figure 3.44: Vickers hardness testing: a) cross-section of indenter and b) resulting indentation [132]

3.5.2 Short-span three-point bending

3.5.2.1 Experimental

Critical failure modes and strength at rupture of CPMCs were assessed through short-span three-point bending (SSB) as depicted in Figure 3.45. The method was selected because it can produce interlaminar shear at material interfaces near the embedded copper layer of the samples while loading the samples in a fashion comparable to an IHCT during typical manipulation. The method also results in flexural deformation, inducing tensile and compressive stresses which give rise to several other modes of failure. This complicates the estimation of the failure stresses but it provides a quick assessment of the critical mode of failure via a single experimental method.

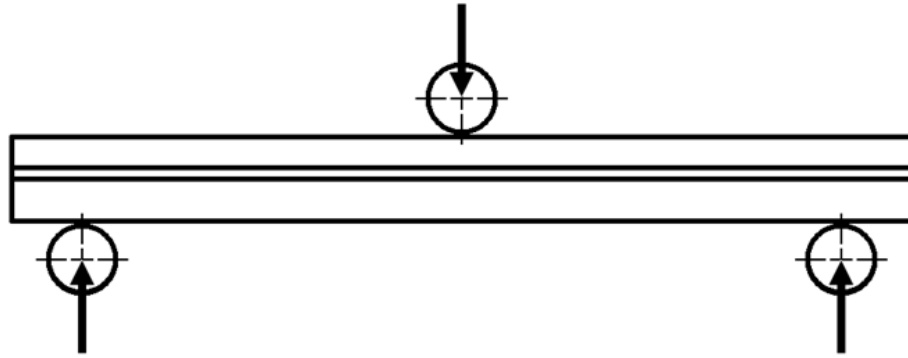


Figure 3.45: Three-point bending of CPMC samples

Tests were performed based on ASTM D2344 standard [133] which is used for determining the maximum short-beam strength of PMC laminates. An Instron 4482 load frame equipped with a three-point bending fixture was used for the tests (Figure 3.46). Specimens used measured 30mm to 40mm in length and had a rectangular cross-section with a width of 11.4mm to 12.7mm and a thickness of 4.8mm to 5.1mm. They were supported by rounded supports having a 6.3mm radius and a span of 25.5mm. Load was applied at mid-span between supports using a 6.3mm radius loading nose that moved at a rate of 1mm/min until specimen fracture ensued. Forces were monitored by the 100kN load cell of the load frame whereas displacements were measured by the cross-head motion. From these, stresses were calculated with equations developed for multilayered materials as presented in the next section.

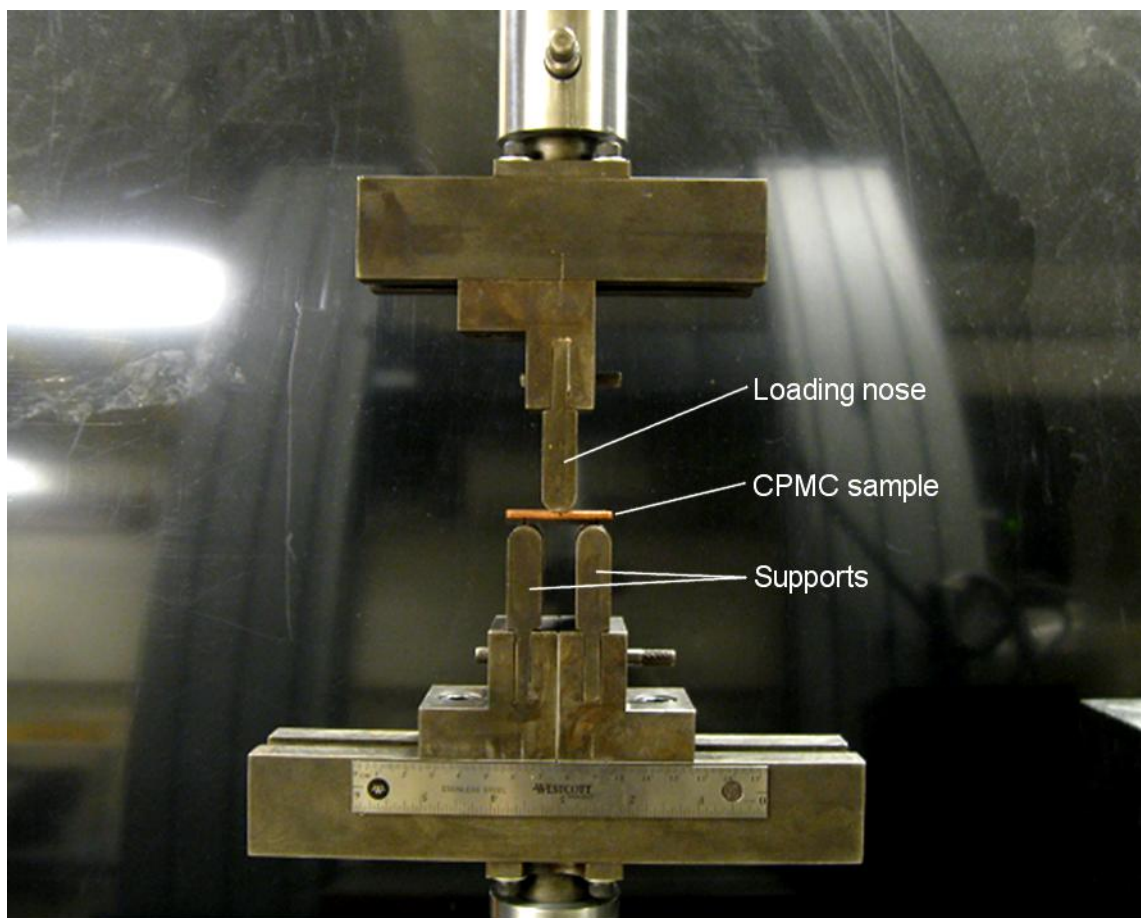


Figure 3.46: Short-span three-point bending fixture

Tests were made at different temperatures to assess change in mechanical behaviour due to heating, which is essential for mould applications. This was achieved by using an environmental chamber (Instron 3111) on the load frame (Figure 3.47). The temperature of the samples was made uniform by placing them in the chamber at least 2 hours prior to mechanical testing. The selected temperatures were 25°C, 70°C and 120°C. The two high temperatures were chosen based on the lower and higher tool temperature required in the fabrication of low cure temperature SE-70 laminates.

It should be noted that the flexural modulus of the SE-70 was estimated from standard three-point bending using the same set-up as SSB but with a longer span of 75mm. Results appear in section 4.5.2.

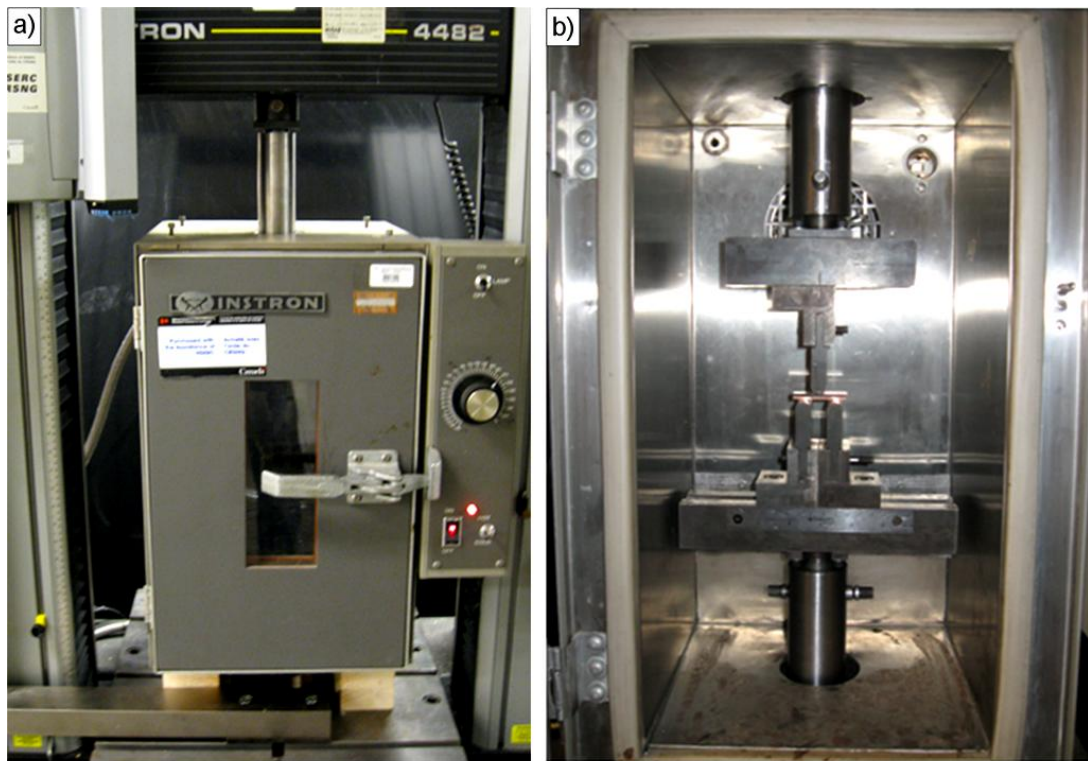


Figure 3.47: Environmental chamber a) on the load frame, and b) inside with bending fixtures

3.5.2.2 Short-span three-point stress equations

Both axial stresses and shear stresses are present during SSB testing, and they vary in the thickness. In homogeneous and isotropic materials, maximum and minimum axial stresses are found at the upper and lower surfaces and vary linearly through the thickness. Conversely, maximum shear is located on the neutral plane, while shear stresses are null on the outer surfaces and vary parabolically through the thickness. For multilayered materials with distinct moduli, the stress field differs based on the stiffness of the layers and location of the neutral plane. The mathematical derivation of the bending equations used in estimating these stresses is presented in Appendix B. The equations assume that the layers are in perfect contact with each other.

In bending, stresses are all dependent on the distance from the neutral axis. The location of this neutral axis can be evaluated from the following:

$$y_n = \frac{\sum_{i=1}^n E_i y_i A_i}{\sum_{i=1}^n E_i A_i} \quad (3.17)$$

where y_n is the location of the neutral axis from the lower face of the sample, y_i is the location of the centre of mass of material layer i from the lower face of the sample, E_i is modulus of elasticity of layer i , A_i is the cross-sectional area of layer i , the subscripts i and n representing a specific material layer and the total number of layers, respectively. The axial stress σ_x at a given height y from the neutral axis of a multilayered beam is equal to:

$$\sigma_x = -\frac{M}{\sum_{i=1}^n E_i I_i} y E_i \quad (3.18)$$

where M is the bending moment, and I_i is the second moment of area. The shear stress τ_{xy} at a given height y from the neutral axis of a multilayered material is given by:

$$\tau_{xy} = -V \frac{\sum_{i=1}^n \left[\int E_i y dy \right]_i}{\sum_{i=1}^n E_i I_i} \quad (3.19)$$

where V is the shear force and the integral in the bracket is evaluated over the control length of material layer i .

A C++ program was written for computing the SSB stresses for multilayered materials at mid-span. The sample geometry, material properties and applied force were entered manually. The program calculated the stresses at different heights within the beam. Integrations were done numerically with steps of 0.01mm. The source code for the program is in Appendix C.

3.5.3 Low frequency cycling

Low frequency cycling in short-span bending was performed on CPMC samples for simulating field use, such as different moulding and de-moulding cycles for a tooling application. Samples used were identical to the SSB samples presented in section 3.5.2, i.e., length of 30mm to 40mm, thickness of 4.8mm to 5.2mm and width of 11.5mm to 13.4mm. Samples were supported by rounded supports having a 6.3mm radius with a span of 25.5mm. Before cycling, the initial position of the 6.3mm radius loading nose was set based on a pre-load of 40N, which prevented sample motion during testing. Each cycle consisted in loading the sample at a rate of 1mm/min until a given force was reached. The force was based on a ratio F_r of the maximum SSB strength, Equation (3.20). After reaching the set force, the load was removed at the same rate of 1mm/s until the loading nose returned to its initial position. The cycle was repeated 100 times and similarly to the “SSB” experiment, cycling was performed at 25°C, 70°C and 120°C.

$$F_r = \frac{F_{\text{Cycling}}}{F_{\text{Max}}} \quad (3.20)$$

3.6 CPMC thermal characterisation

3.6.1 Resistance to elevated temperatures

CPMCs were subjected to thermal cycling which was deemed important due to the difference in thermal expansion coefficients between its various materials (see section 3.1 for values). This difference in thermal expansion coefficients may generate significant internal stresses that could in turn induce coating spallation.

A heating cycle (Figure 3.48) consisted in heating a sample in an oven at a rate of $2^{\circ}\text{C}/\text{min}$, up to a certain dwell temperature. The dwell temperature was held for 1 hour. Afterwards, the oven was turned off and the sample was cooled slowly to a temperature of 120°C , avoiding thermal shock. Then the oven door was opened, letting the sample to cool in ambient air. Following the heating cycle, the sample was inspected using the stereo-microscope. In the absence of noticeable damage, the sample was subjected to further thermal cycling with an increased dwell temperature. This was repeated until damage was observed.

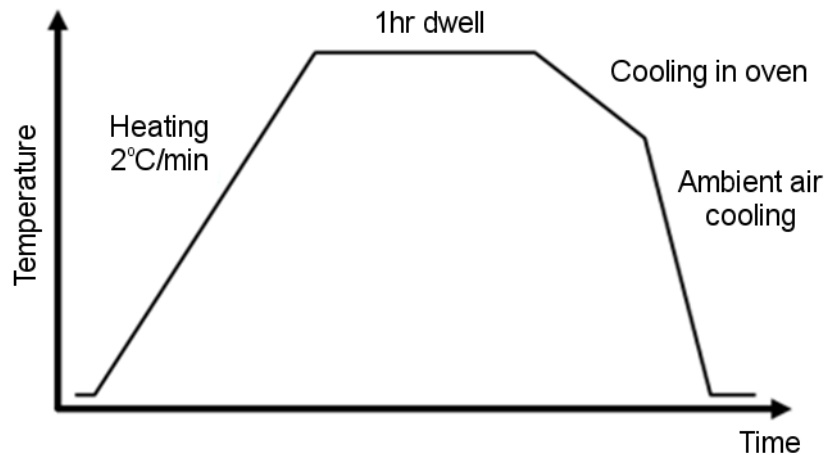


Figure 3.48: Standard heating cycle used for the temperature resistance experiments

3.6.2 Temperature distribution

The temperature distribution capability of IHCTs was assessed through thermometry. The IHCTs were heated using their flexible electric heater and temperatures across their inner tool face were monitored using both thermocouple and infrared thermal images. Heat losses were minimised by covering the IHCTs with insulation blankets made of 140mm thick mineral wool (Roxul, R24).

Heating experiments were made with a constant or variable heater power, the latter for imposing a temperature profile consisting of heating, dwell and cooling phases. Heating was controlled through a computer with a LabVIEW (v8.2) program that was written during the thesis work. The LabVIEW program reads the temperature of a control thermocouple CTC positioned next to the heater on the IHCT through a data acquisition board (M 6221, National Instruments), and compares it to a target temperature profile. The program activates and deactivates the heater through a relay, maintaining the control thermocouple temperature within $\pm 1^{\circ}\text{C}$ from the target temperature. The program also monitored a thermocouple (OHTC) for preventing over-heating of the heater. The power to the heater was cut when the temperature reached above 200°C . The system appears in Figure 3.49.

Temperature measurements made through thermocouples used the same LabVIEW program as above. The program monitored 12 K-type thermocouples including the CTC and OHTC. The data acquisition board sampled thermocouple readings at a rate of 1kHz and the program averaged the data for each thermocouple every 0.5 seconds.

Temperature measurements from infrared thermal imaging were made using a FLIR i7 infrared camera. The camera had a resolution of 140x140 pixels and a temperature sensitivity of 0.1°C [134]. The emissivity of the IHCT was set to match the temperatures of the thermocouples. For taking infrared images, the insulation blanket had to be removed momentarily. Infrared images were analysed using software ThermaCAM Researcher Pro 2.10 which offered statistical analysis tools including standard deviation of temperature.

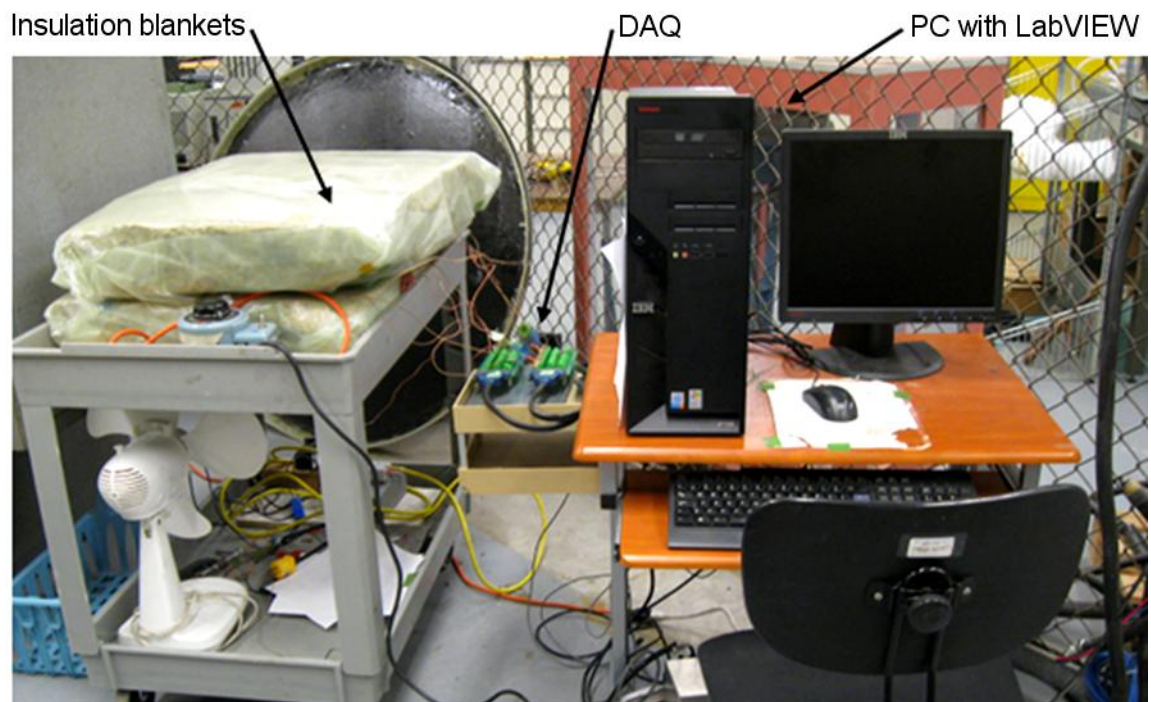


Figure 3.49: System for monitoring temperature distribution across IHCTs

3.7 Heat transfer simulations

Heat transfer simulations were made for characterising temperature distribution in IHCTs when heated. The simulations modelled the heat conduction inside IHCTs, heat generation in the heater and convective heat transfer at the external boundaries of IHCTs. In this section, only a brief explanation of the modelling and solution is presented. Simulations that are presented in Chapter 6 will discuss modelling strategies further.

Gambit v2.4.6 was used for creating the 3D geometry and mesh of IHCTs. The 3D geometry represented the inner PMC structure, outer copper layer, and heater. The heater had a volumetric source term for generating the heat flux. The interfaces between the PMC and copper, such as the co-cured layer or silicone were modelled as thin-walls, which are solved only for through-thickness conduction. The mesh used hexahedral elements with 8 nodes.

ANSYS FLUENT v11 and v12 were used for solving the steady-state and transient energy equations for IHCTs simulations. The software uses finite volume methods (FVM) for solving the partial differential equations. A pressure-based solver was used. The *First-order upwind* scheme was selected for spatial discretisation of the energy equation and gradients were calculated with the *Least-squares cell-based* method. This scheme was used because it offers fast convergence and it is suited for hexahedral meshes [135]. A convergence criterion for the energy equation of 1×10^{-6} was used for monitoring residuals, which governed the number of iterations performed.

For modelling the heater controller, a user-defined function (UDF) was used based on the work of Brillant [25]. The UDF function models the control thermocouple and over-heating thermocouples by reading the temperatures at their given location, on every iteration. The source term of the heater was activated/deactivated by the UDF for keeping the modelled control thermocouple temperature within $\pm 1^\circ\text{C}$ of the target temperature. The source code for the UDF appears in Appendix D.

Chapter 4

Material system:

Fabrication and characterisation

The main original contribution of this thesis was the development and characterisation of a copper coating for carbon fibre/epoxy PMC using Pulsed Gas-Dynamic Spraying for improving in-plane thermal conductivity of PMCs.

In this section, development of the CPMCs proceeded in three main phases. The first phase was the investigation of the manufacturing process for the co-cured layer of CPMC substrates. The second phase focused on identifying the spraying parameters leading to the deposition in a coating on PMCs without any damage to PMC structures. The third phase consisted of the thermo-mechanical characterisation CPMCs made using the suitable spraying parameters found in the second phase.

4.1 Vibrating table and co-cured layer

This section investigates the vibrating table as a mean towards producing co-cured layers for relatively flat out-of-autoclave coated PMCs (CPMCs). First, the operation of the vibrating table is discussed. This is followed by the characterisation of the behaviour of the vibrated powder that eventually forms the co-cured layer. Finally, CPMC substrates are presented with an analysis of the consolidated co-cured layer.

4.1.1 Vibrating table

The vibrating table used two motors rotating eccentric masses for generating vibrations. Power was sent to individual motors, enabling the selection of electrical polarity. This enabled two modes of operation, one where the motors rotated in the same direction and another where the motors were rotating in opposite directions. The types of motions resulting from these two modes of vibration were analysed by taking pictures of the rotating masses during operation of the vibrating table.

For the case of the motors rotating in the same direction, the eccentric masses were initially positioned in three different configurations based on phase shift (i.e. orientation) between the masses (Figure 4.1). The first configurations had both masses oriented in the same direction, resulting in a phase shift of 0° . The second configuration consisted in having a phase shift of 180° . Finally, the third configuration featured a phase shift of 90° between masses. Upon beginning rotation of the masses, it was found that the 180° phase shift configuration had the smoothest start. However, after a few seconds of vibration, the vibrating table reached a steady state level of vibrations for all configurations. Surprisingly, pictures showed that the steady-state configuration was unrelated to the initial phase shift of the masses. For all configurations the masses repeatedly positioned themselves with a phase shift of 180° (Figure 4.1). From a balance of force (Figure 4.2), it was deduced that the resulting motion of the vibrating table was an oscillating motion between a clockwise and counter-clockwise rotation. It was conjectured that this mode of motion would result in greater powder motion at the edges of the vibrating table due to an increase in radial acceleration with the distance from the centre of rotation.

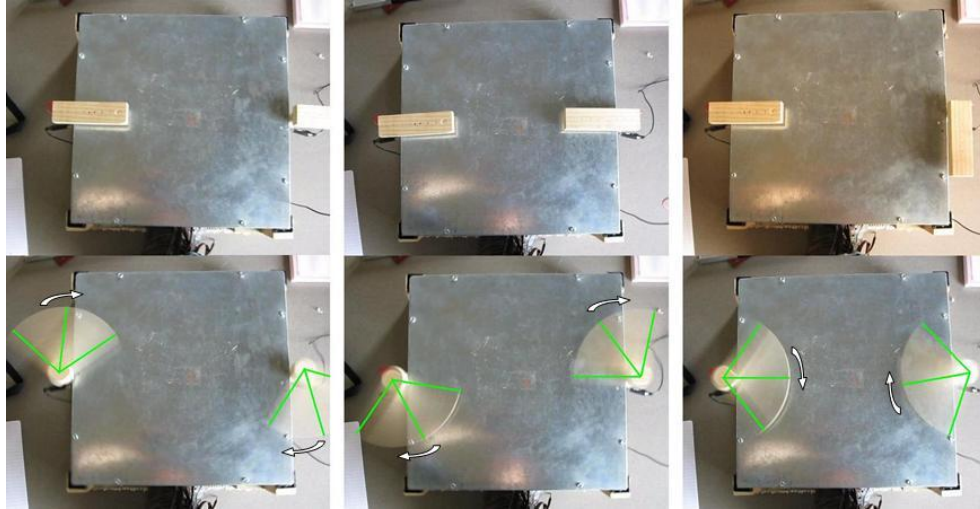


Figure 4.1: Steady-state motion for masses rotating in the same direction with an initial phase shift of 0° (left), 180° (centre), and 90° (right)

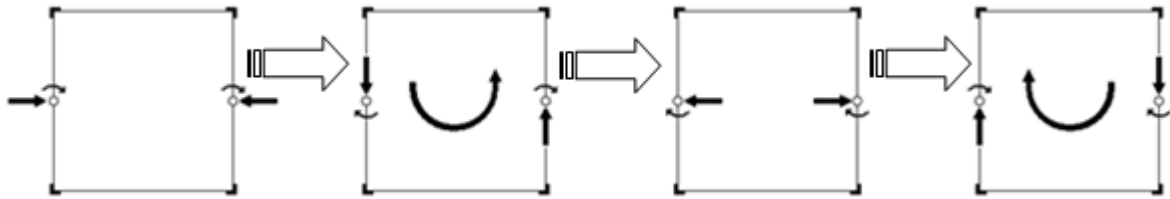


Figure 4.2: Forces acting on the vibrating table from eccentric masses rotating in the same direction and having a phase shift of 180°

A similar test was performed with the masses rotating in opposite directions. The same three mass configurations were tried. Upon initial rotation of the masses, the 180° phase shift configuration provided the smoothest start. For all configurations after a couple of seconds of motion, the vibrating table stabilised itself into steady-state motion. Pictures of the vibrating table (Figure 4.3) showed that the steady-state orientation of the masses was a phase shift of 180° . From a balance of force, it was deduced that rotations produced a back-and-forth motion in the vibrating table (Figure 4.4).

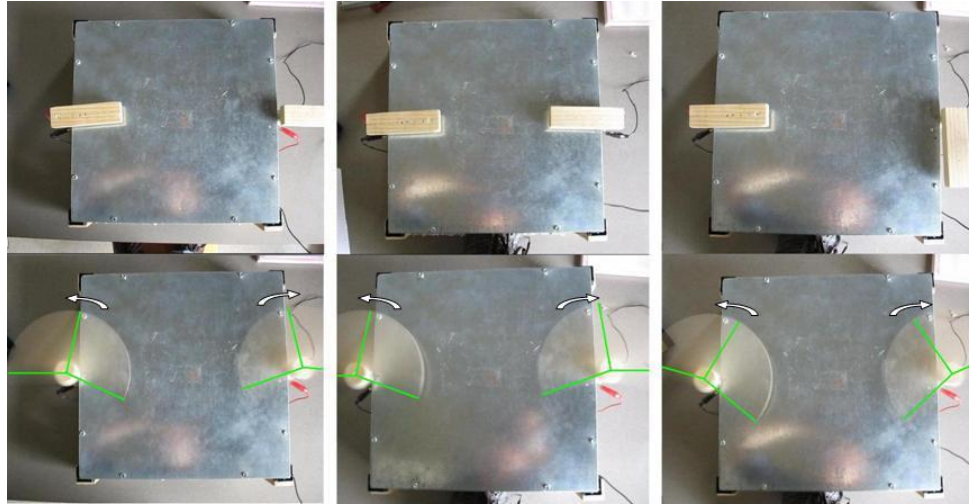


Figure 4.3: Steady-state motion of masses rotating in opposite directions with an initial phase shift of 0° (left), 180° (centre), and 90° (right)

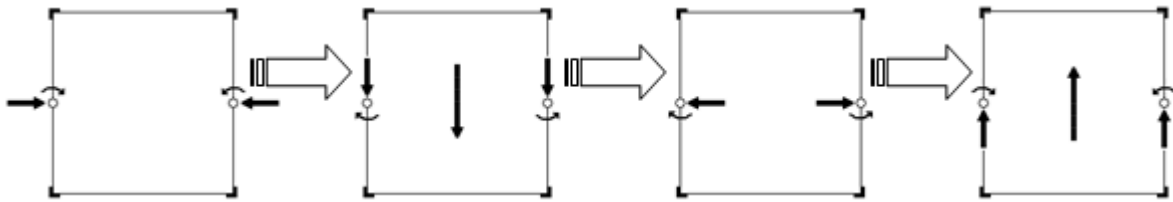


Figure 4.4: Forces acting on the vibrating table from eccentric masses rotating in opposite directions and having a phase shift of 180°

4.1.2 Vibration-induced powder motion

The two possible vibration modes of the vibrating table were tested for powder distribution. Discrete heaps of powder were placed on the vibrating table and vibrated a few seconds. Displacement of the powder was assessed visually afterwards. The tests were performed with the masses initially aligned along a 180° phase shift as this provided the same type of vibrations as steady-state motion. The amplitude and frequency of the vibrations were controlled by carefully increasing the power sent to the motors until the onset of powder fluidisation.

Vibrations induced by the motors rotating in the same direction resulted in the radial expansion of the powder heaps (Figure 4.5). Due to powder fluidisation, the gravitational forces flattened the heaps and pushed the powder away from the heap centre. The test also confirmed that the forces acting on the powder were stronger near the edges than near the centre of the vibration table. This behaviour is not favourable as uniform fluidisation of the powder bed is desired.

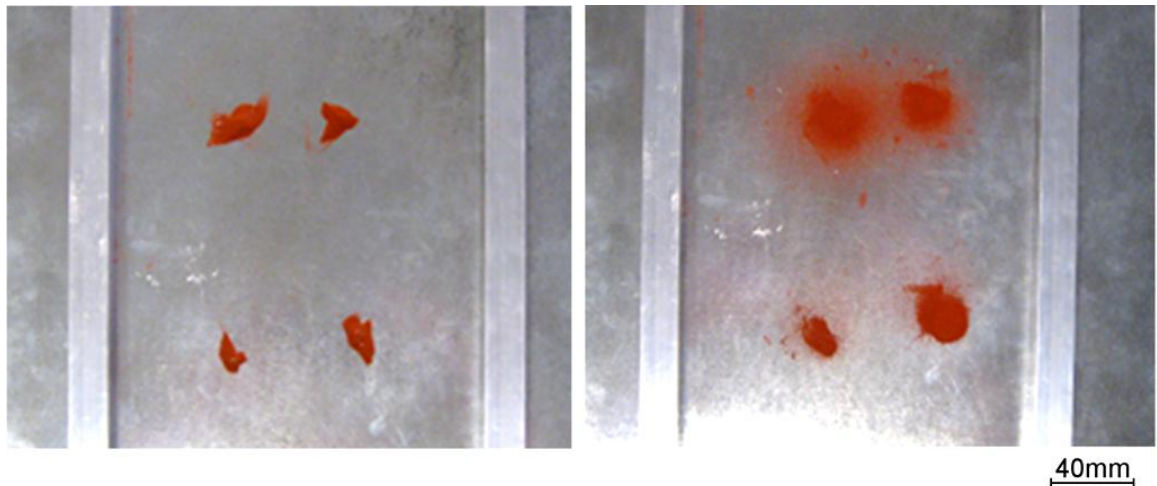


Figure 4.5: Powder before (left) and after (right) vibrations produced by eccentric masses rotating in the same direction

Vibrations induced by motors rotating in opposite directions also resulted in the flattening of the powder heaps. However, the back-and-forth motion of the vibrating table expanded the powder heaps solely along one axis (Figure 4.6). The forces acting on the powder were found to be constant throughout the entire vibration table which is a noticeable advantage compared to the other mode of motion. Hence, this mode of motion was selected for the project despite powder being only distributed along one axis.

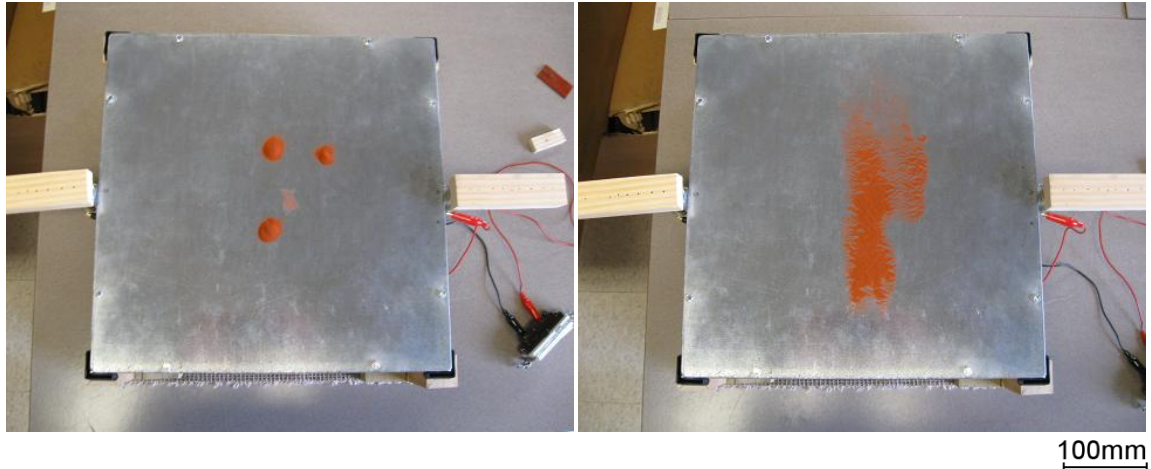


Figure 4.6: Powder before (left) and after (right) vibrations produced by eccentric masses rotating in opposite directions

The next phase was to assess the behaviour of a vibrated powder bed in a constrained environment that would replicate the prepreg stack. Powder heaps were placed over double-sided tape to reproduce the tack of prepreg (Figure 4.7). Edges of the tape were bridged with aluminium walls for preventing powder leaking. The powder was subjected to vibrations until the copper particles covered the entire tape.



Figure 4.7: Powder heaps placed on double-sided tape

Results showed that vibrations could be used effectively for distributing powder across the entire surface of the tape. However, contrast analysis of a powder bed image revealed that light reflection from the powder bed varied significantly (Figure 4.8). The

reflected light density appeared to be linked to the initial locations of the powder heap. Microscopy of the surface showed that the higher reflected light density was caused by particle segregation. The zones that reflected more light were formed by the smallest particles, while other zones mostly featured greater size particles. It is conjectured that vertical segregation likely occurred at the onset of vibrations, pushing smaller particles under the heap, as reported in [136, 137]. These small particles adhered to the tape and vibrations distributed the remaining unbound particles across the surface. Since it was not known whether particle size affected coating deposition, uniform powder size distribution was desired for the CPMC substrates.

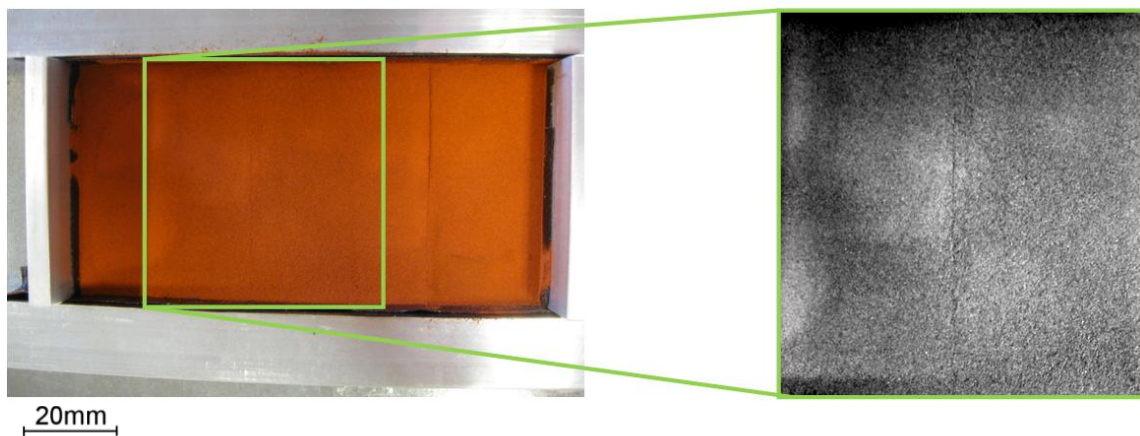


Figure 4.8: Variation of light reflection from vibrated powder bed

4.1.3 Comparison of co-cured layers

The CPMC substrates manufactured during the project are listed in Table 4.1. The co-cured layer of the out-of-autoclave CPMC substrates was produced following the recommendations of the preceding section. Prior to PMC consolidation, powder was distributed manually over the entire surface of the prepreg stack to limit particle segregation. The resulting powder bed was vibrated for a few seconds to distribute the powder evenly. Vibrations were produced through eccentric masses rotating in opposite directions.

Table 4.1: Description of CPMC substrates used in the project

Substrate	Manufacturing method	Number of prepreg plies	Additional resin plies	Size (mm)	Surface density of copper (g/cm ²)
IA-01	In-autoclave	N/A	No	300 by 300	N/A
OOA-01	Out-of-autoclave	12	No	150 by 150	0.16
OOA-02	Out-of-autoclave	12	No	150 by 150	0.20
OOA-03	Out-of-autoclave	12	No	150 by 150	0.20
OOA-04	Out-of-autoclave	12	Yes	100 by 100	0.50
OOA-05	Out-of-autoclave	12	No	150 by 150	0.21
OOA-06	Out-of-autoclave	12	Yes	100 by 100	0.46
OOA-07	Out-of-autoclave	12	No	150 by 150	0.20
OOA-08	Out-of-autoclave	12	No	150 by 150	0.25
OOA-09	Out-of-autoclave	12	No	100 by 100	0.50
CPMC IHCT	Out-of-autoclave	20	No	180 by 180	0.40

Selected CPMC substrates were inspected for assessing variability of the co-cured layer thickness and copper content. Three types of substrates were analysed: 1) out-of-autoclave substrates (OOA-02, -07, -11 and -12), 2) out-of-autoclave substrates with an extra resin ply (OOA-04 and OOA-06) and 3) an in-autoclave substrate (IA-01). Three cuts per substrate were taken at distinct locations, and were inspected by microscopy. Copper content measurements were obtained from the cuts by contrast analysis of microscopic images taken at equidistance.

Microscopy revealed that the substrate cured in autoclave had greater copper concentration in its co-cured layer than out-of-autoclave substrates (Figure 4.9). Measurements presented in Table 4.2 give a copper content of 64.9% for IA-01 and approximately 58% for the others. It can be concluded that the greater pressure from the autoclave was more effective for compacting the powder bed than the vibrations used in the fabrication of out-of-autoclave substrates. It is also worth noting that the copper content value of 55% estimated in section 3.4.3 from the thermal conductivity for the out-of-autoclave substrate is close to the measured value of 58%. Inspection also showed that

there are significantly more satellites to copper particles in the co-cured layer of the out-of-autoclave substrates, likely resulting from the batch of powder used. From microscopy, satellites appear to be detrimental to the copper content but this can be negated through sieving.

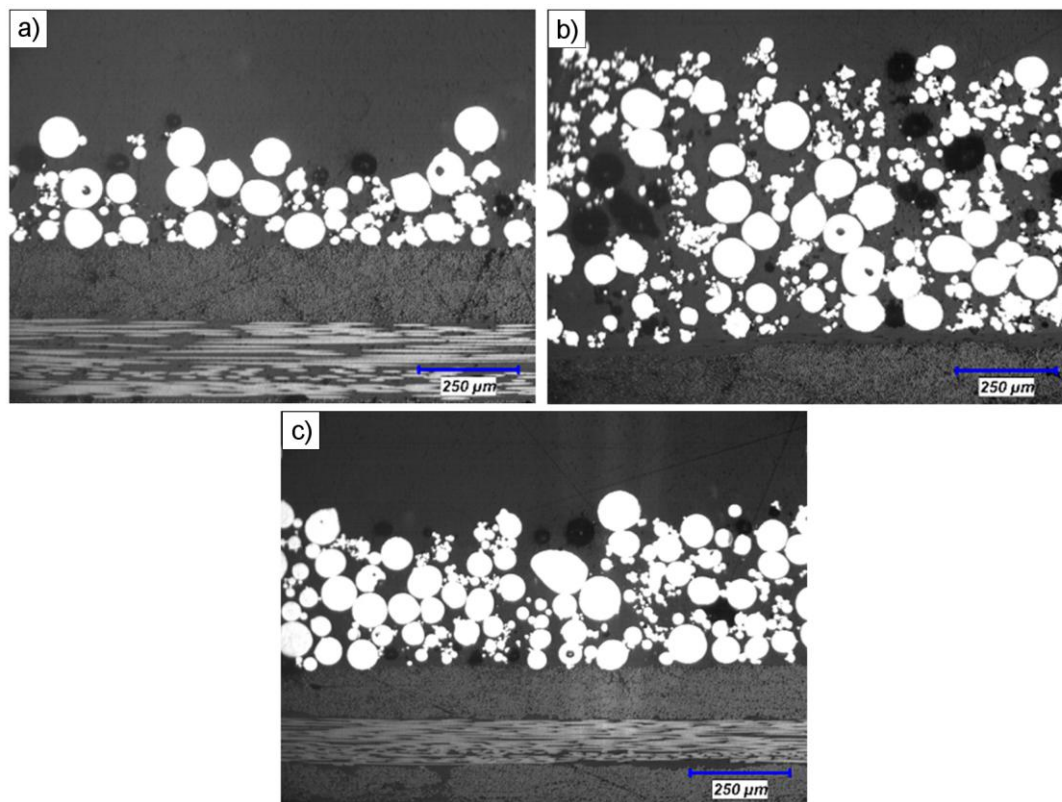


Figure 4.9: Co-cured layer for the a) out-of-autoclave substrates, b) out-of-autoclave substrates with an extra resin ply and c) autoclave substrate

The measured average thickness of the co-cured layer for the out-of-autoclave substrates was approximately 350μm but significant local variability was observed (Table 4.2). Thickness ranged from one to several stacked particles. The addition of an extra resin ply doubled the co-cured layer thickness but variability remained. Furthermore, the use of vibrations for obtaining a flat powder bed before co-curing did not translate into a more uniform co-cured layer as seen by the comparable variability in the autoclave substrate. This non-uniform thickness appears to result from irregularities in excess resin flow. Thus, the co-cured layer thickness is governed primarily by resin infusion and not by the initial powder bed distribution.

Table 4.2: Measurements of co-cured layer thickness and copper content of selected substrates

Substrate	Additional resin ply	Co-cured layer thickness (μm)				Copper content (%)	
		Average	Standard deviation	Minimum	Maximum	Average	Standard deviation
OOA-02	No	319	± 132	84	700	58.9	± 6.1
OOA-03	No	402	± 126	167	686	58.1	± 7.3
OOA-07	No	356	± 124	98	668	58.9	± 6.9
OOA-08	No	318	± 108	88	531	55.2	± 7.9
OOA-04	Yes	689	± 144	394	1033	55.3	± 5.6
OOA-06	Yes	700	± 125	438	909	58.8	± 6.9
IA-01	No	573	± 154	294	995	64.9	± 5.9

4.2 Coating PMCs

Coating of out-of-autoclave CPMCs substrates was performed using the PGDS process. Various parameters were investigated to develop knowledge about coating deposition and density. Several experimental programs were devised for evaluating the effect of spraying parameters:

- Experimental program “DR”: substrate displacement rate (U_d);
- Experimental program “GP”: gas pressure (P_{Gas});
- Experimental program “GT”: gas heater temperature (T_{Gas});
- Experimental program “PH”: spraying powder pre-heating temperature (T_P);
- Experimental program “SF”: surface finish; and
- Experimental program “LP”: difference between pulsating (PGDS) and continuous (LP-CGDS) spraying.

As part of these experimental programs, coatings on out-of-autoclave CPMC substrates were sometime compared to coatings made on in-autoclave substrate IA-01 and on an aluminium 6061 substrate. Information regarding the substrates used in the section can be found in Table 4.1. It should be noted that most experimental programs were limited to spraying Cu 105-3 spray powder as its coating density varied with spray parameters, providing a quantitative property for comparison. Conversely, the SST-C5003 powder always yielded coatings that were nearly fully dense, having a porosity much lower than 1%, under any given spray conditions that enabled deposition.

Prior to the presentation of results from the experimental programs, a short section discussing general observations relating to coatings on PMC is presented. The section discusses observations that apply to all experimental programs.

4.2.1 General coating observations

In this section, the main observations resulting from the experimental programs are presented. First, a description of PGDS spraying outcomes is discussed, followed by general coating characteristics.

PGDS on CPMC substrates lead to three different outcomes. Upon using high gas pressures and temperatures as discussed in experimental programs “GP” and “GT”, PGDS usually yielded erosion of the substrate (Figure 4.10). The sprayed particles removed the co-cured layer and damaged the PMC underneath, eroding several plies. Conversely, under low gas pressures ($<2\text{MPa}$) the co-cured layer protected the substrate from erosion but no coating deposition ensued. In this case, particles hit the substrate but bounced off it, resulting in light grit blasting of exposed copper from the co-cured layer (Figure 4.11). Finally, under certain spray conditions that are presented in the experimental programs, thick copper coatings could be formed on the surface of the co-cured layer (Figure 4.12).

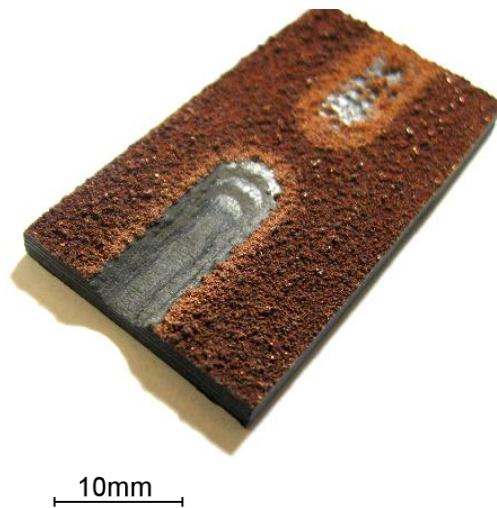


Figure 4.10: Erosion of an out-of-autoclave substrate

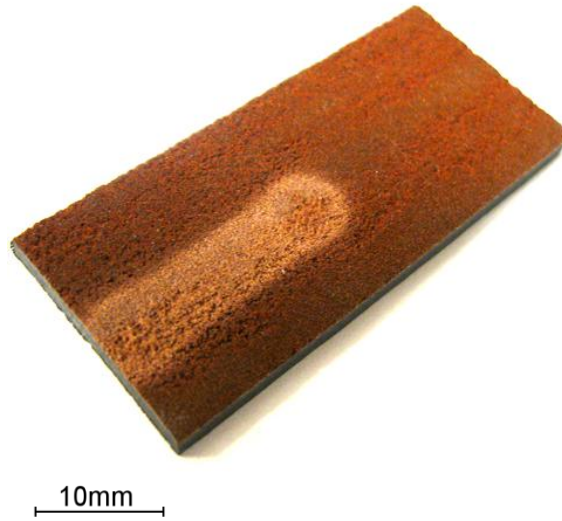


Figure 4.11: Absence of coating deposition or damage to the PMC due to protection from the co-cured layer

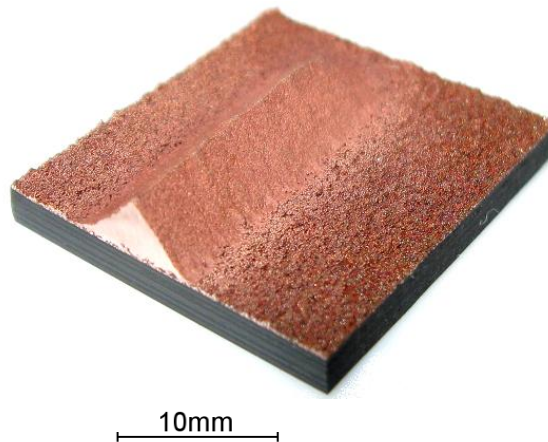


Figure 4.12: Copper coating deposited on the out-of-autoclave substrate

Coatings that were formed on the CPMC substrates had a rough surface finish similar to most cold sprayed coatings. This was caused by overlaid particle splats that mechanically interlocked together to form the coatings as seen in Figure 4.13.



Figure 4.13: Microscopic image of the coating surface where particle splats are observed

Moreover, it was observed that the sprayed powder morphology had an effect on the coatings. When spraying Cu 105-3 (Figure 4.14.a), the impact with the substrate resulted in significant deformation of the copper particles in the co-cured layer. However, little deformation was observed in the sprayed particles, as suggested by the numerous pores present between them. In contrast, in SST-C5003 coatings, copper particles of the co-cured layer underwent limited deformation while the sprayed particles were heavily deformed as suggested by the very low coating porosity (Figure 4.14.b). In the latter, it is thought that the sprayed powder was more ductile than the co-cured particles and the Cu-105-3. Unfortunately, SST-C5003 powder showed an adverse structural behaviour in that its dendritic particle morphology makes the particles prone to breakage. Sometimes when a particle impacted the substrate it shattered and left non-deformed dust particles at the substrate-to-coating interface, hindering further adhesion (Figure 4.14.c).

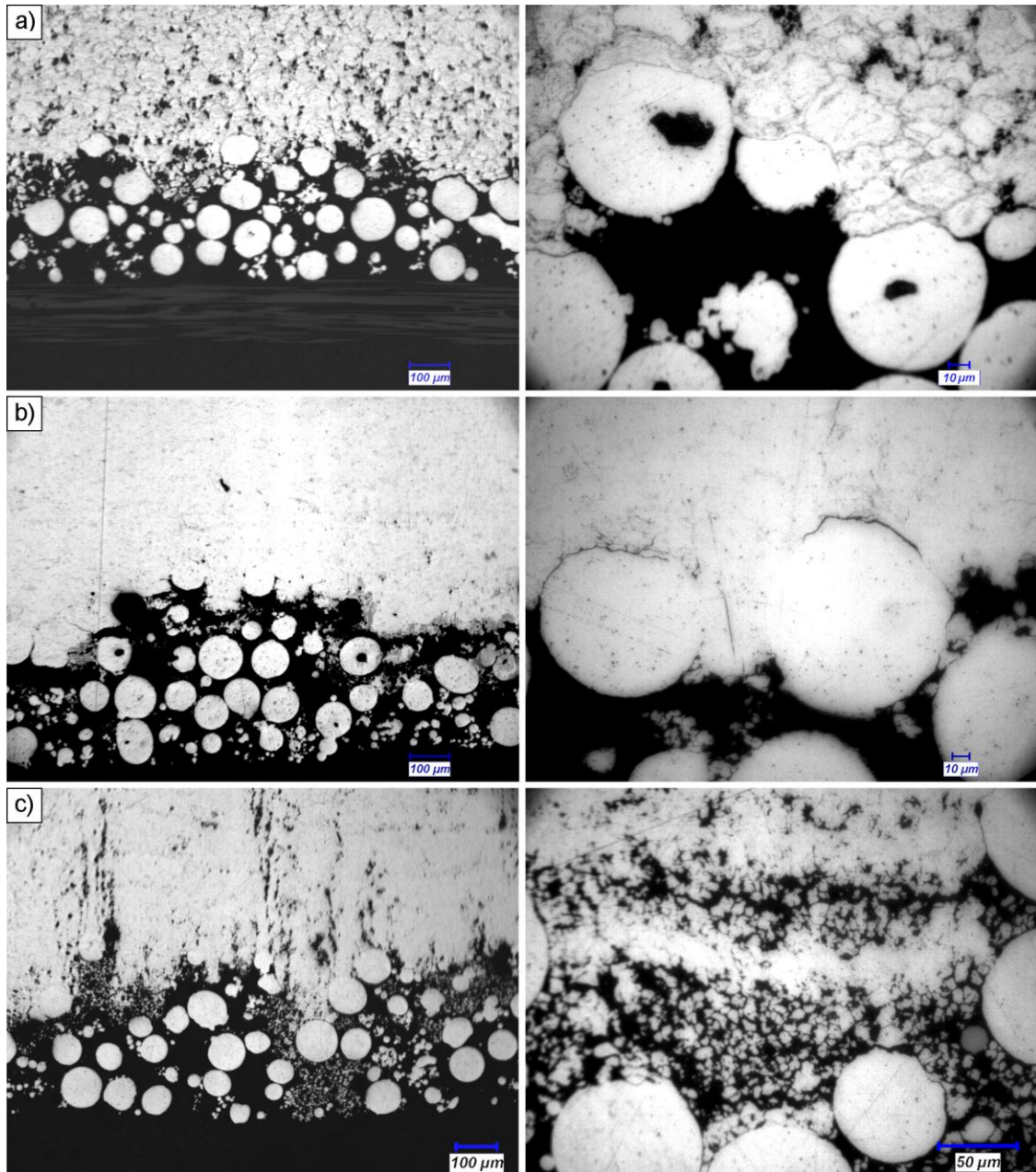


Figure 4.14: Cross-sections at magnifications 100X (left) and 400X (right) of coatings on the co-cured layer with a) Cu-105-3, b) properly deposited SST-C5003 and c) SST-5003 that underwent breakage

Consolidation of particles inside the coating varied with spray parameters and also with location. In cold spray processes, coatings generally feature higher porosity near the surface of the coating because further consolidation may be bestowed by particle impact of subsequent spray passes. Since the last sprayed layer does not get additional consolidation,

it is less dense. Similarly, CPMCs featured greater porosity at the coating surface but they also featured a zone of greater coating porosity near the co-cured layer (Figure 4.15). This phenomenon will be explained in section 4.2.6.

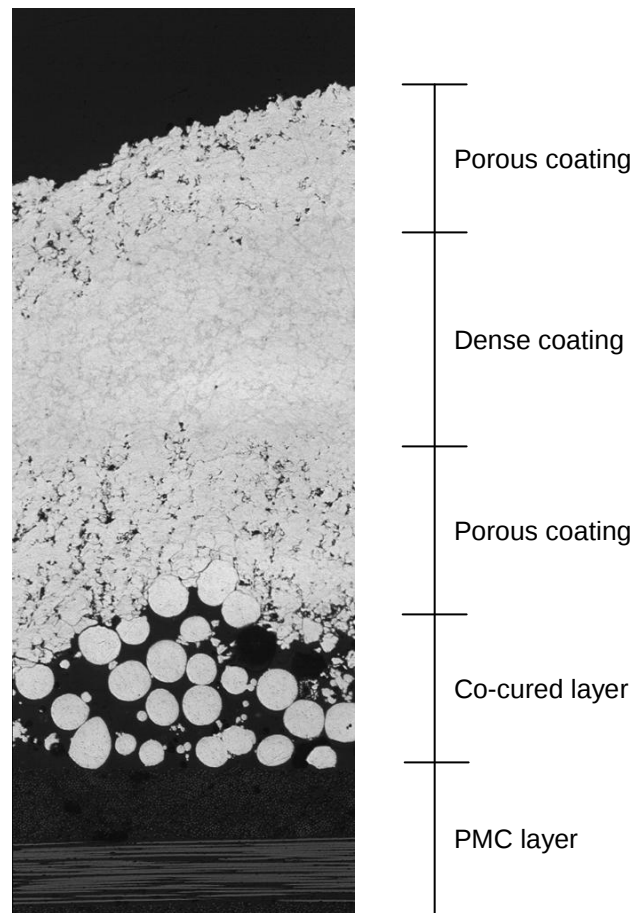


Figure 4.15: Typical zones in a CPMC from cross-section of GT21

4.2.2 Experimental program “DR”: effect of substrate displacement rate

Experimental program “DR” aimed at characterising the effect of the substrate displacement rate on coating deposition. Qualitative visual observations were made for assessing deposition. Coatings were made on substrate OOA-02 and on aluminium using PGDS system #1.

Spray parameters and observations appear in Table 4.3. Displacement rate varied between 5mm/s, 25mm/s and 50mm/s. For all velocities, deposition on aluminium was

possible. Conversely, deposition was only possible on OOA-02 at 5mm/s and 25mm/s. Hence, slower displacement rates are better for producing CPMCs.

Table 4.3: Spray parameters and visual observations for the experimental program “DR”

Sample	Substrate	Powder	Gas	P_{Gas} (MPa)	T_{Gas} (°C)	U_d (mm/s)	T_p (°C)	Observations
DR01	Al	Cu 105-3	He	3	RT	5	25	Deposition
DR02	OOA-02	Cu 105-3	He	3	RT	5	25	Deposition
DR03	OOA-02	Cu 105-3	He	3	RT	25	25	Deposition
DR04	Al	Cu105-3	He	3	RT	50	25	Deposition
DR05	OOA-02	Cu 105-3	He	3	RT	50	25	Barely any deposition

4.2.3 Experimental program “GP”: effect of gas pressure

Experimental program “GP” investigated the effect of gas pressure which influences the strength of the PGDS shock waves. Helium gas pressure varied from 1MPa to 4MPa and sprays were performed on substrates OOA-02, IA-01 and aluminium using PGDS system #1. Spray parameters and visual observations appear in Table 4.4.

Table 4.4: Spray parameters and visual observations for experimental program “GP”

Sample	Substrate	Powder	Gas	P _{Gas} (MPa)	T _{Gas} (°C)	U _d (mm/s)	T _p (°C)	Observations
GP01	Al	Cu 105-3	He	1	25	25	25	No deposition
GP02	IA-01	Cu105-3	He	1	25	25	25	No deposition
GP03	OOA-02	Cu 105-3	He	1	25	25	25	No deposition
GP04	Al	Cu105-3	He	2	25	25	25	Deposition
GP05	IA-01	Cu 105-3	He	2	25	25	25	Barely any deposition, speckles of copper
GP06	OOA-02	Cu105-3	He	2	25	25	25	Insufficient co-cured copper which led to erosion
GP07	Al	Cu 105-3	He	3	25	25	25	Thick deposition
GP08	IA-01	Cu105-3	He	3	25	25	25	Deposition
GP09	OOA-02	Cu 105-3	He	3	25	25	25	Deposition
GP10	Al	Cu105-3	He	3.5	25	25	25	Thick deposition
GP11	IA-01	Cu 105-3	He	3.5	25	25	25	Deposition
GP12	OOA-02	Cu105-3	He	3.5	25	25	25	Deposition but erosion on coating edges
GP13	Al	Cu 105-3	He	4	25	25	25	Thick deposition
GP14	IA-01	Cu 105-3	He	4	25	25	25	Deposition
GP15	OOA-02	Cu105-3	He	4	25	25	25	Deposition

The experiments showed that without any gas heating the Cu 105-3 powder could start depositing at 2MPa on aluminium but that a greater pressure was required to effectively deposit on OOA-02 and IA-01. Gas pressures between 3MPa and 4MPa resulted in powder deposition on all substrates. Coatings made on the OOA-02 and IA-01 substrates looked similar, while coatings on aluminium were always thicker for an identical number of spray passes.

It is also worth noting that spraying at elevated pressures ($>3\text{MPa}$) on substrate OOA-02 resulted in slight erosion at the periphery of the coating (Figure 4.16). Conversely, substrate IA-01 did not show such signs of erosion up to the tested pressures. It appears that the substrate matrix plays a vital role in the structural integrity of the co-cured layer and in this case the epoxy with the higher glass transition temperature performed best.

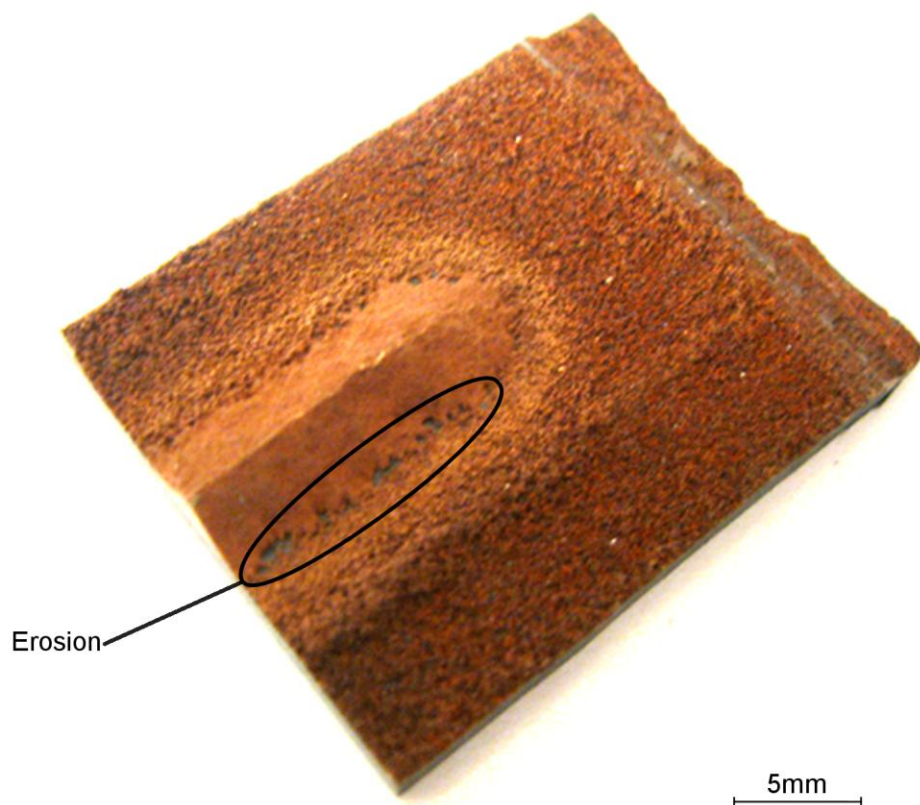


Figure 4.16: Spray-induced erosion around coating of GP12

Microscopy and porosity measurements were performed on selected samples (Table 4.5). Inspection showed greater consolidation in the middle of all coatings (Figure 4.17), but sprayed particles were never significantly deformed. At a gas pressure of 4MPa all substrates showed similar coating porosity of ~3%. This suggests that once the first few coating layers are deposited using PGDS, the coating density of subsequent layers is governed mostly by the spraying parameters and not so much by the composition and roughness of the substrate.

Table 4.5: Coating porosity for experimental program “GP”

Sample	Substrate	P_{Gas} (MPa)	Porosity (%)				Figure
			Minimum	Maximum	Average	Standard deviation	
GP08	IA-01	3.0	1.14	8.81	3.69	± 1.96	N/A
GP09	OOA-02	3.0	2.66	16.00	6.83	± 3.24	Figure 4.17.a
G11	IA-01	3.5	2.19	6.92	3.90	± 1.38	N/A
GP12	OOA-02	3.5	1.08	7.17	3.27	± 1.62	Figure 4.17.b
GP13	Al	4.0	0.01	9.41	3.33	± 2.27	N/A
GP14	IA-01	4.0	1.05	8.15	3.18	± 1.99	N/A
GP15	OOA-02	4.0	0.15	9.47	3.21	± 2.16	Figure 4.17.c

Increasing the gas pressure reduced the porosity of coatings made on substrate OOA-02, mostly from 3.0MPa to 3.5MPa. Significant improvement was also noted in the minimum porosity measurements, where porosity went from 2.66% at 3.0MPa to 0.15% at 4.0MPa. Conversely, no significant change in porosity was observed for the coatings made on substrate IA-01, which was unexpected considering the results on substrate OOA-02. Nevertheless, the lowest porosity for substrate IA-01 was seen at 4.0MPa, confirming that high gas pressures are beneficial to coating consolidation. However, the major milestone of experimental program “GP” was that deposition could be achieved without heating the gas or powder, leading to coatings with minimal oxide content; a desirable asset for thermal conductivity.

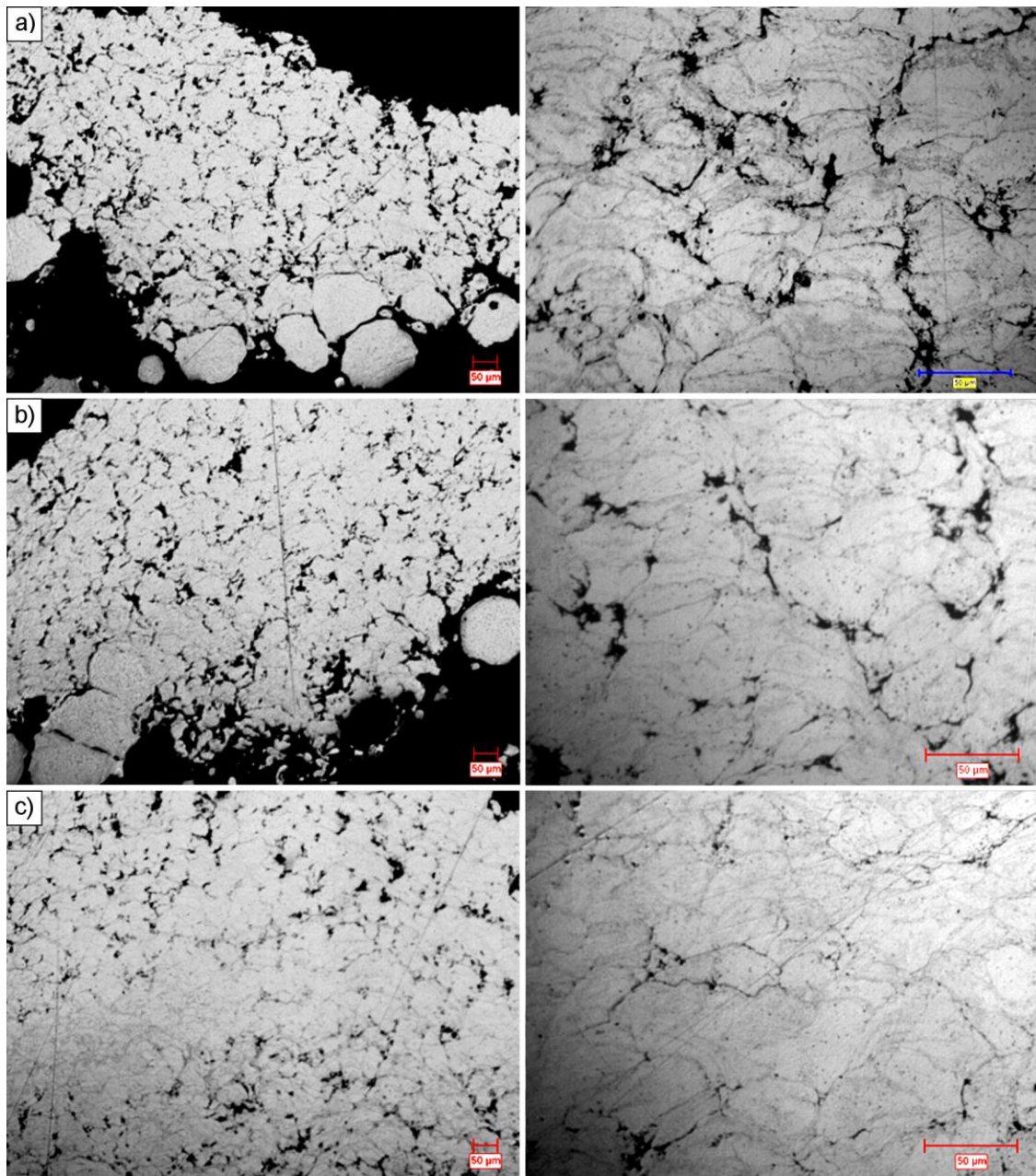


Figure 4.17: Cross-sections of a) PG09, b) PG12 and c) PG15 at magnifications 100X (left) and 400X (right)

4.2.4 Experimental program “GT”: effect of gas temperature

Experimental program “GT” aimed at assessing the effect of gas temperature on coating density. The rationale behind this experimental program is that providing more heat to a gas flow results in greater gas velocity and potentially to particle softening. In this

experimental program, temperature of the gas heater was varied from 100°C to 200°C while testing different gas pressures. Sprays were made on substrates OOA-01, OOA-02, IA-01 and on aluminium using PGDS system #1. Spray parameters and observations appear in Table 4.6.

Coating was achieved on aluminium with a pressure of 1MPa and gas heater temperature of 150°C. Coating was also achieved on substrate IA-01 using a gas pressure of 1MPa, but the gas heater temperature had to be set at 250°C. Conversely, gas heating did not seem as effective for the out-of-autoclave CPMC substrates. It was not apparent that gas heating could help lower the gas pressure required for deposition due to erosion resulting from limited heating. Below 3MPa, the maximum usable gas heater temperature was 150°C, while above 3MPa it was 100°C. The temperature limit was not a clear boundary but a transition zone as several tests conducted at 100°C yielded different outcomes: substrate erosion (TG19), deposition (TG21) or a mixture of both (TG20). From these tests at higher gas pressures, it appears that the gas heater temperature limit is related to the 99°C glass transition temperature of the SE-70 epoxy.

Cross-sections of the two successful depositions on OOA-02 appear in Figure 4.18. Porosity estimates are compared to coatings of experimental program “GP” in Table 4.7. At 3MPa, heating the gas resulted in significant improvement in coating density. Average porosity dropped to 4%, which is close to values obtained for coatings made with a gas pressure of 4MPa without heating. Additionally, relatively large zones of less than 1% porosity were achieved in the centre of the coatings. Increasing the pressure along with the heating did not show great promise of improvement. Minimum and average porosities were nearly identical to the unheated coatings of experimental program “GP” at higher gas pressures. However, there was a significant reduction of the maximum porosity. Thus, gas heating is recommended for improving coating density, mostly with higher temperature resistant polymer matrices such as the 3501-6 of the IA-01 substrate.

Table 4.6: Spray parameters and observations for experimental program “GT”

Sample	Substrate	Powder	Gas	P _{Gas} (MPa)	T _{Gas} (°C)	U _d (mm/s)	T _p (°C)	Observations
GT01	Al	Cu 105-3	He	1	150	7	25	Thin deposition
GT02	IA-01	Cu 105-3	He	1	150	7	25	No deposition
GT03	OOA-01	Cu 105-3	He	1	150	7	25	No deposition
GT04	Al	Cu 105-3	He	1	200	7	25	Thin deposition
GT05	IA-01	Cu 105-3	He	1	200	7	25	Slight erosion
GT06	OOA-01	Cu 105-3	He	1	200	7	25	Slight erosion
GT07	Al	Cu 105-3	He	1	250	7	25	Thin deposition
GT08	IA-01	Cu 105-3	He	1	250	7	25	Very thin deposition
GT09	OOA-01	Cu 105-3	He	1	250	7	25	Erosion
GT10	Al	Cu 105-3	He	2	150	7	25	Deposition
GT11	IA-01	Cu 105-3	He	2	150	7	25	Deposition
GT12	OOA-01	Cu 105-3	He	2	150	7	25	Weak adhesion and local erosion
GT13	IA-01	Cu 105-3	He	2	150	25	25	Deposition
GT14	OOA-01	Cu 105-3	He	2	150	25	25	Erosion
GT15	Al	Cu 105-3	He	2	200	7	25	Deposition
GT16	IA-01	Cu 105-3	He	2	200	7	25	Thin deposition
GT17	OOA-01	Cu 105-3	He	2	200	7	25	Severe erosion
GT18	Al	Cu 105-3	He	3	100	15	25	Deposition
GT19	OOA-02	Cu105-3	He	3	100	15	25	Severe erosion
GT20	OOA-02	Cu 105-3	He	3	100	15	25	Local deposition and severe erosion
GT21	OOA-02	Cu105-3	He	3	100	15	25	Deposition
GT22	Al	Cu105-3	He	3	150	15	25	Deposition
GT23	OOA-02	Cu 105-3	He	3	150	15	25	Erosion
GT24	Al	Cu 105-3	He	4	100	15	25	Deposition
GT25	OOA-02	Cu105-3	He	4	100	15	25	Mostly deposition with local erosion
GT26	OOA-04	Cu105-3	He	4	150	15	25	Erosion
GT27	Al	Cu 105-3	He	4	200	15	25	Deposition
GT28	IA-01	Cu 105-3	He	4	200	15	25	Deposition
GT29	OOA-02	Cu105-3	He	4	200	15	25	Severe erosion

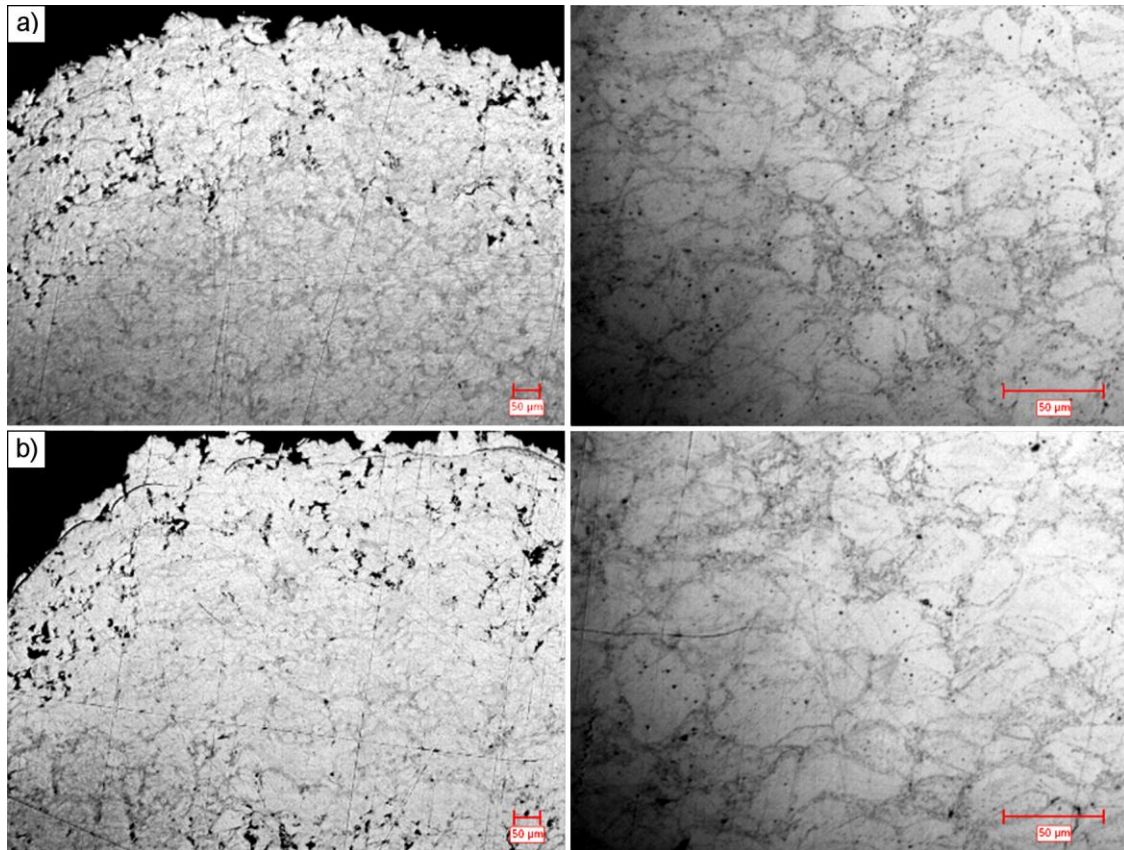


Figure 4.18: Cross-sections of a) GT21 and b) GT25 at magnifications 100X (left) and 400X (right)

Table 4.7: Coating porosity for experimental program “GT”

Sample	Substrate	P_{Gas} (MPa)	T_{Gas} (°C)	Porosity (%)				Figure
				Minimum	Maximum	Average	Standard deviation	
GP09	OOA-02	3.0	25	2.66	16.00	6.83	± 3.24	Figure 4.17.a
GT21	OOA-02	3.0	100	0.39	11.54	4.04	± 3.14	Figure 4.18.a
GP15	OOA-02	4.0	25	0.15	9.47	3.21	± 2.16	Figure 4.17.c
GT25	OOA-02	4.0	100	0.20	5.89	3.36	± 2.04	Figure 4.18.b

4.2.5 Experimental program “PH”: effect of powder pre-heating

Experimental program “PH” consisted in assessing the effect of spraying powder pre-heating on coating density. Sprayed powder was pre-heated in the powder feeder at temperatures between 100°C to 200°C for at least 1 hour prior to spraying under different gas pressures. The goal was to soften the sprayed particles and promote plastic deformation by heating at or near the annealing temperature. Spray trials were made on substrates OOA-02, IA-01 and aluminium using PGDS system #1. Spray parameters and observations are outlined in Table 4.8.

Pre-heated powder sprays were performed using the same gas pressures that were successful for deposition in experimental program “GP”. Deposition occurred in all trials, and in most cases the coatings were very thick and looked better than those obtained before. However, upon inspection of coating cross-sections it was found that the external appearance of the coating did not relate well to coating density. At 3MPa, average porosity did not change much from pre-heating the powder. At higher pressures there was a great increase in porosity with increasing pre-heating temperature. Porosity was mostly located near the surface of the coating and at the substrate-to-coating interface. However, large regions of fully dense coating were observed when spraying at 4MPa with powder pre-heated at 200°C.

Table 4.8: Spray parameters and observations for experimental program “PH”

Sample	Substrate	Powder	Gas	P _{Gas} (MPa)	T _{Gas} (°C)	U _d (mm/s)	T _p (°C)	Observations
PH01	Al	Cu 105-3	He	3.0	25	15	100	Thick deposition
PH02	IA-01	Cu105-3	He	3.0	25	15	100	Thin deposition with local erosion
PH03	OOA-02	Cu 105-3	He	3.0	25	15	100	Thin deposition with local erosion
PH04	Al	Cu 105-3	He	3.5	25	15	100	Thick deposition
PH05	IA-01	Cu105-3	He	3.5	25	15	100	Deposition
PH06	OOA-02	Cu 105-3	He	3.5	25	15	100	Deposition
PH07	Al	Cu105-3	He	4.0	25	15	100	Deposition comparable to 3.5MPa
PH08	IA-01	Cu 105-3	He	4.0	25	15	100	Deposition comparable to 3.5MPa
PH09	OOA-02	Cu105-3	He	4.0	25	15	100	Deposition comparable to 3.5MPa
PH10	Al	Cu105-3	He	3.0	25	15	200	Thick deposition
PH11	IA-01	Cu 105-3	He	3.0	25	15	200	Thick deposition
PH12	OOA-02	Cu105-3	He	3.0	25	15	200	Thick deposition with local erosion ^a
PH13	Al	Cu 105-3	He	3.5	25	15	200	Thick deposition
PH14	IA-01	Cu105-3	He	3.5	25	15	200	Thick deposition
PH15	OOA-02	Cu 105-3	He	3.5	25	15	200	Thick deposition
PH16	Al	Cu 105-3	He	4.0	25	15	200	Thick deposition
PH17	IA-01	Cu105-3	He	4.0	25	15	200	Thick deposition
PH18	OOA-02	Cu 105-3	He	4.0	25	15	200	Thick deposition

Note: ^a Erosion attributed to thin co-cured layer

Table 4.9: Coating porosity for experimental program “PH”

Sample	Substrate	P_{Gas} (MPa)	T_p (°C)	Porosity (%)				Figure
				Minimum	Maximum	Average	Standard deviation	
GP09	OOA-02	3.0	25	2.66	16.00	6.83	± 3.24	Figure 4.17.a
PH03	OOA-02	3.0	100	3.54	8.61	5.75	± 1.71	Figure 4.19.a
PH12	OOA-02	3.0	200	1.33	13.62	7.07	± 2.85	Figure 4.19.b
GP12	OOA-02	3.5	25	1.08	7.17	3.27	± 1.62	Figure 4.17.b
PH06	OOA-02	3.5	100	1.36	7.98	5.11	± 1.61	Figure 4.19.c
PH15	OOA-02	3.5	200	2.63	23.24	9.07	± 4.38	Figure 4.20.a
GP15	OOA-02	4.0	25	0.15	9.47	3.21	± 2.16	Figure 4.17.c
PH09	OOA-02	4.0	100	1.17	9.09	4.18	± 2.00	Figure 4.20.b
PH18	OOA-02	4.0	200	0.02	15.70	6.65	± 3.90	Figure 4.20.c
PH16	Al	4.0	200	0.01	17.14	5.07	± 4.02	N/A

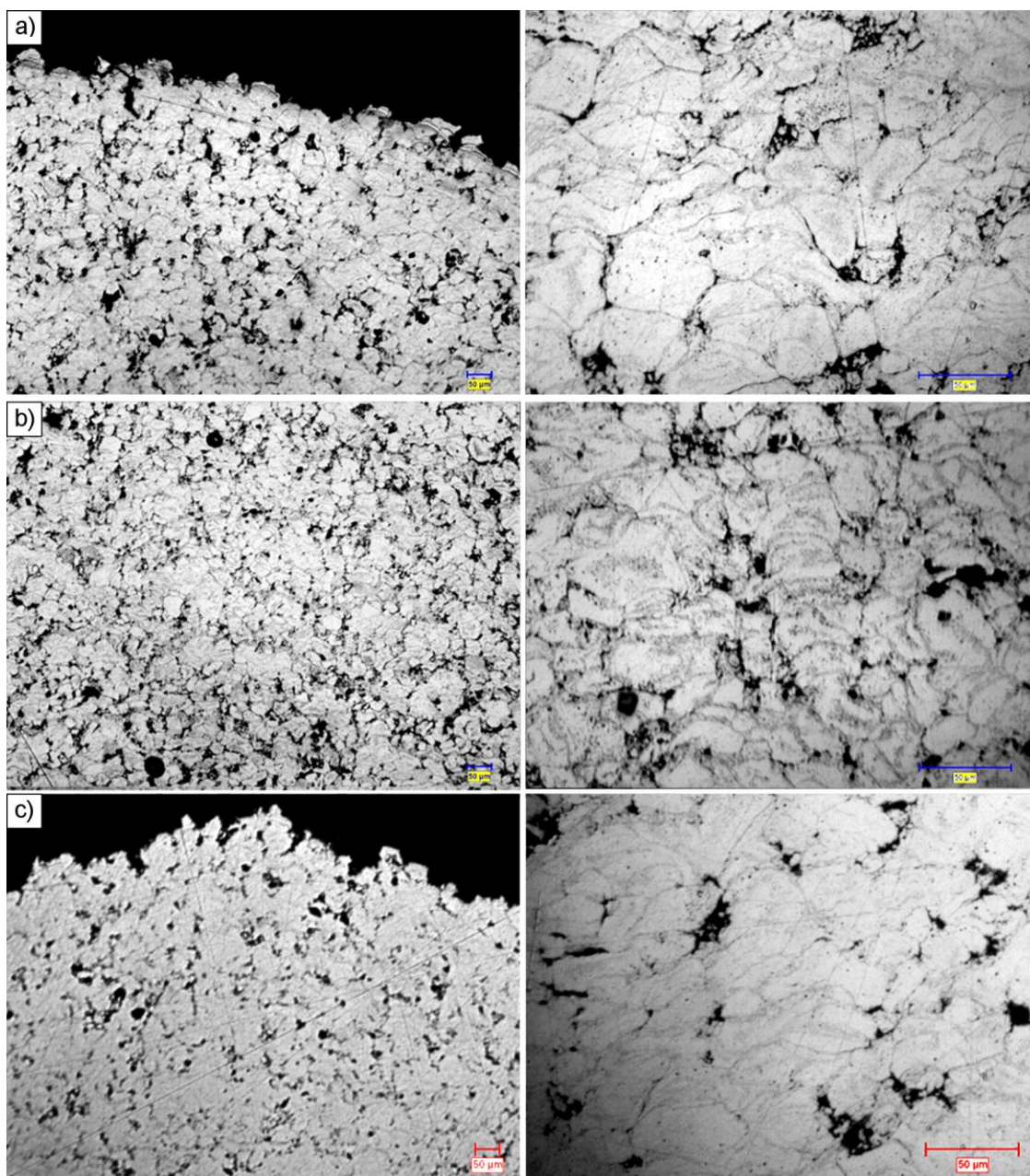


Figure 4.19: Cross-sections of a) PH03, b) PH12 and c) PH06 at magnifications 100X (left) and 400X (right)

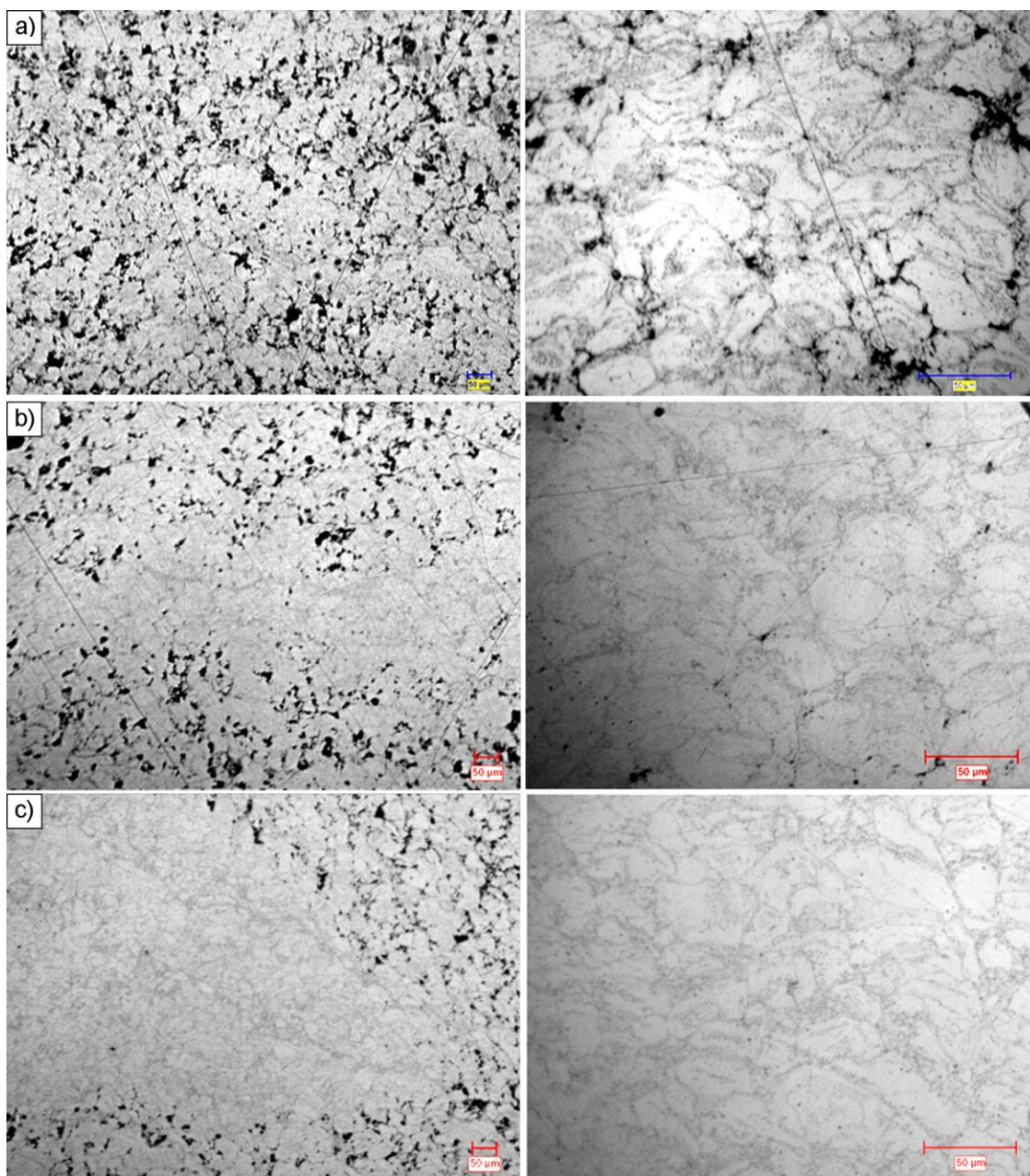


Figure 4.20: Cross-sections of a) PH15, b) PH09 and c) PH18 at magnifications 100X (left) and 400X (right)

In general, increases in porosity may be attributed to spray powder oxidation during heating, where copper particles oxidise more quickly in ambient air above 65°C [138]. In this experimental program, oxidation was observed in the powder remaining in the feeder which turned to a dark red/purple colour (Figure 4.21) reminiscent of Cu_2O and CuO mixtures. The hard oxide scale that formed limited plastic deformation of the particles, increasing coating porosity. However, gas pressures of 4MPa likely imparted sufficient kinetic energy to break the oxide scale and regions of fully dense coating could occur due to the increase in powder ductility from pre-heating at 200°C. Therefore, if pre-heating was achieved in an inert environment, increased spray particle ductility without the oxide scale could likely improve coating density.

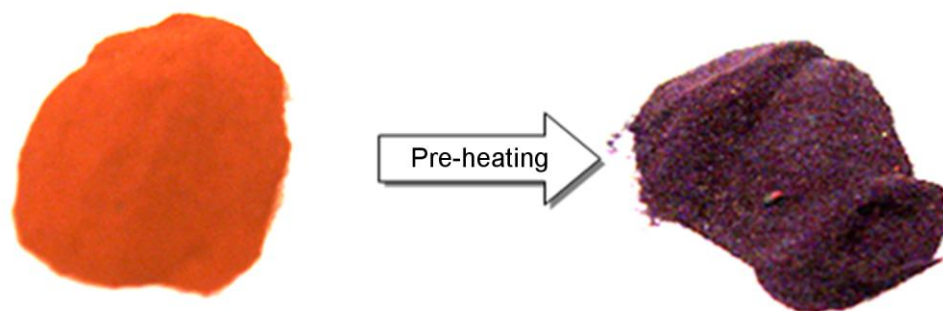


Figure 4.21: Non-oxidised copper powder before the experimental program “PH” (left) and oxidised powder after the program (right)

4.2.6 Experimental program “SF”: effect of surface finish

In previous experimental programs, CPMCs always showed higher porosity near the coating-to-substrate interface; this was not the case with the smooth aluminium. Thus, the effect of the surface roughness was studied in experimental program “SF”. Some CPMCs were grinded and polished with 500 grit silica paper. Sprays were performed on these substrates and deposition was compared with deposition on unaltered substrates.

Sprays were made on out-of-autoclave substrates OOA-03 and OOA-04, and aluminium using PGDS system #1. Substrate OOA-04 featured a thicker co-cured layer to prevent excessive thinning of the protective layer during surface preparation. Spray

parameters and observations appear in Table 4.10. It should be noted that no gas or powder heating was used in this experimental program.

Table 4.10: Spray parameters and observations for experimental program “SF”

Sample	Substrate	Powder	Gas	P _{Gas} (MPa)	Surface preparation	U _d (mm/s)	Observations
SF01	Al	Cu 105-3	He	4.0	None	15	Thick deposition
SF02	OOA-03	Cu105-3	He	4.0	None	15	Deposition
SF03	OOA-04	Cu105-3	He	4.0	Polished	0 (Spot)	Deposition
SF04	Al	Cu 105-3	He	5.0	None	15	Thick deposition
SF05	OOA-03	Cu105-3	He	5.0	None	15	Deposition
SF06	OOA-04	Cu105-3	He	5.0	None	15	Deposition
SF07	OOA-04	Cu 105-3	He	5.0	Polished	15	Erosion
SF08	OOA-04	Cu105-3	He	5.0	Locally polished	15	Deposition on rough surface and erosion on polished surface
SF09	OOA-04	Cu 105-3	He	3.0	Polished	0 (Spot)	Poor deposition

As before, coatings were easily deposited on aluminium and looked best based on visual inspection. Deposition was also achieved on substrates OOA-03 and OOA-04 without surface preparation, even at 5MPa. However, substrate OOA-04 with surface preparation was harder to coat. At 3MPa and 4MPa deposition was possible but erosion was present at 5MPa, even with the thicker co-cured layer.

Porosity measurements are presented in Table 4.11. As expected the porosity was reduced by increasing the pressure from 4MPa to 5MPa. Interestingly, SF03 with surface preparation had lower porosity at 4MPa than SF05 without surface preparation at 5MPa.

This was explained by the slightly lower overall porosity, but also by the absence of the higher porosity zone near the substrate-to-coating interface. Therefore, surface roughness does indeed hinder coating consolidation. Since a rough surface generally is not locally perpendicular to impinging particles, it is conjectured that particles lose significant amounts of kinetic energy in friction instead of it being used for plastic deformation, due to a surface that is not perpendicular to their trajectory (Figure 4.22). It should be noted that porosity values reported are higher than those obtained in previous experimental programs. This was likely caused by the deterioration and leaking of the solenoid valve used in system #1.

Table 4.11: Coating porosity for the experimental program “SF”

Sample	Substrate	P_{Gas} (MPa)	Surface Preparation	Porosity (%)				Figure
				Minimum	Maximum	Average	Standard deviation	
SF02	OOA-03	4.0	None	2.01	10.92	5.68	± 2.52	Figure 4.24.a
SF03	OOA-04	4.0	Polished	0.95	6.28	2.96	± 1.39	Figure 4.24.b
SF05	OOA-03	5.0	None	1.41	9.8	4.69	± 2.03	Figure 4.24.c

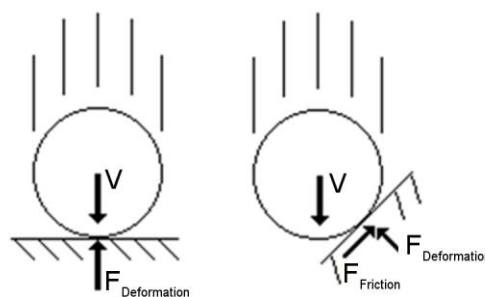


Figure 4.22: Forces during impact acting on a particle that has a perpendicular (left) and inclined (right) trajectory relative to the surface of the substrate

Finally, for comparison purposes, sample SF08 which had 3 different topological zones was sprayed. The sample had a polished surface, a surface not subjected to surface preparation featuring exposed copper particles, and a resin rich surface (Figure 4.23). Upon spraying at 5MPa, a coating was deposited on the unaltered surface with exposed copper

but erosion afflicted the other two zones. It is concluded that surface preparation clearly weakens the substrate against impinging particles. Also, it appears clearly that resin rich surfaces are prone to erosion, even with protective metallic particles underneath. Nevertheless, even if it results in lower resistance to impinging particles, substrate polishing lowers coating porosity by seemingly eliminating the adverse effect of surface roughness. In the future, manufacturing flatter surfaces should be investigated, possibility though greater control of the co-cured layer thickness and usage of flatter or smaller copper particles reinforcements.

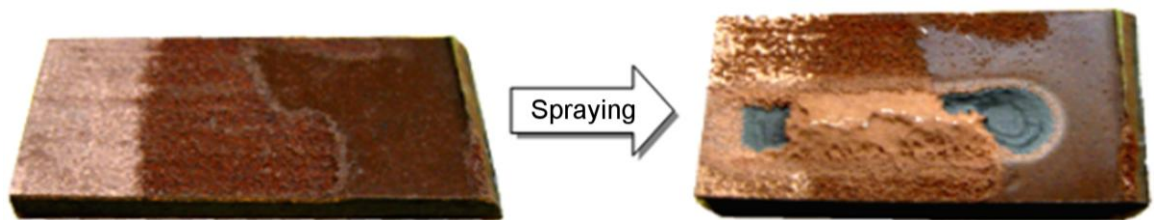


Figure 4.23: Effect of surface finish on deposition on sample SF08 that had a polished surface, an unaltered rough surface and a resin rich surface

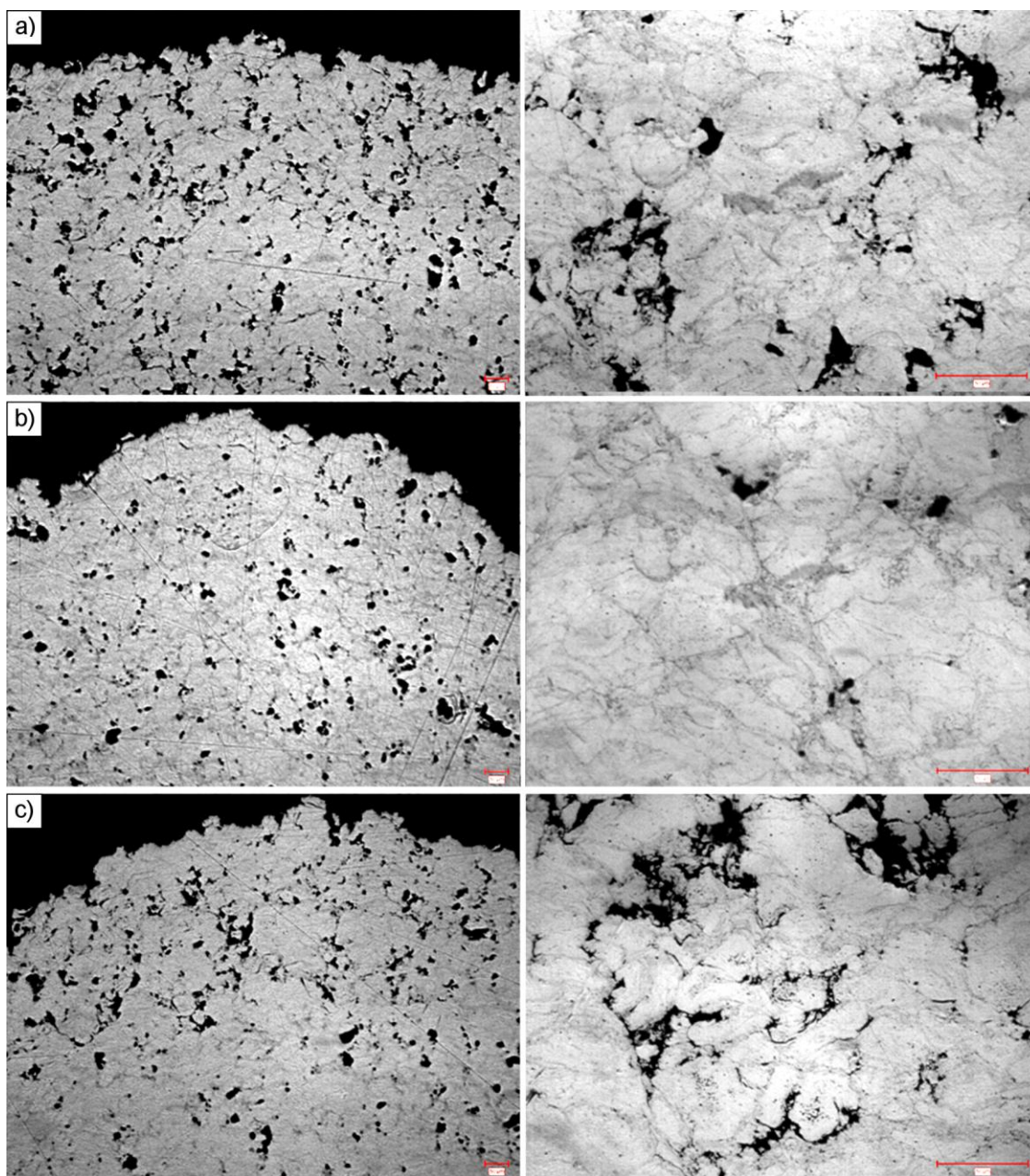


Figure 4.24: Cross-sections of a) SF02, b) SF03 and c) SF05 at magnifications 100X (left) and 400X (right)

4.2.7 Experimental program “LP”: pulsating and continuous spraying

Work presented in previous experimental programs was conducted using the PGDS process. That process is still in development and its usage is not widespread. On the other hand, the LP-CGDS process has had much greater visibility with several units commercially available. Hence, it was desired to see whether CPMCs can be produced using this more common kinetic spray process.

Sprays were performed on bulk copper and on OOA-05 substrate using powder SST-C5003 with the LP-CGDS system. The spray parameters, which are presented in Table 4.12, were selected as close as possible to those of trials done with PGDS. Helium at 90°C was the carrier gas, there was no powder pre-heating, and the substrate was moved at low velocity. The major difference is that a lower gas pressure of 1.7MPa (250psi) had to be used due to system limitations. Nevertheless, spray parameters yielded thick deposition on the copper substrate.

Table 4.12: Spray parameters and observations for experimental program “LP”

Sample	Substrate	Powder	Gas	P_{Gas} (MPa)	T_{Gas} (°C)	U_d (mm/s)	T_p (°C)	Observations
LP01	Cu	SST-C5003	He	1.7	90	10	25	Thick deposition
LP02	OOA-05	SST-C5003	He	1.7	90	10	25	Very rough and thin deposition
LP03	OOA-05	SST-C5003	He	1.7	90	5	25	Poor deposition with pits

Unfortunately, using the same spray parameters for the first out-of-autoclave substrate (sample LP02) did not yield positive results. The co-cured layer protected against erosion, but only a very thin and rough coating was achieved (Figure 4.25). The coating amplified the surface roughness of the substrate. Even so, potential was still seen as a relatively smooth surface coating was deposited before the spray nozzle began to move. Hence, it is suggested that coatings may be deposited using LP-CGDS if the displacement rate is sufficiently slow.

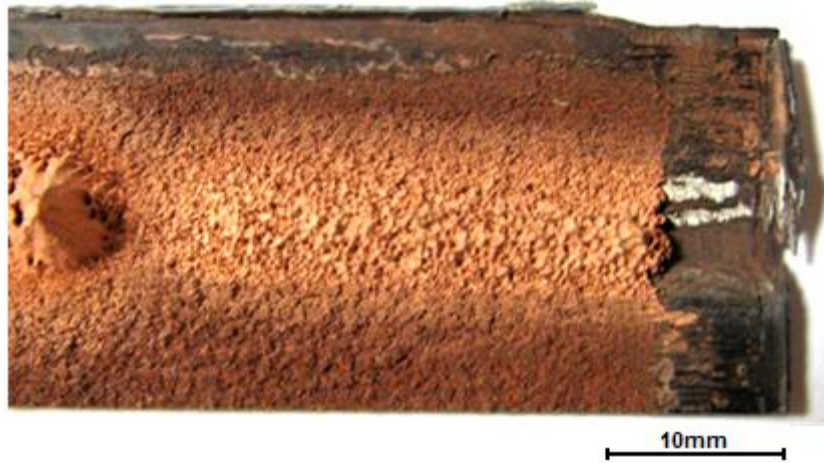


Figure 4.25: LP-CGDS on sample LP02

Spray on LP03 was performed using a slower substrate displacement rate. In this case, a thicker coating build-up was possible with some coating zones having a smooth surface, but most of the coating was covered with huge pits (Figure 4.26). As seen in Figure 4.26, LP03 lead to an improvement over the previous attempt but it is still far from providing the surface quality obtained using PGDS. Slower displacement rates may help, but would lead to very thick coatings.

Closer inspection of LP-CGDS CPMCs revealed that the pits were closed by thin copper walls (Figure 4.27). Cross-section micrographs appearing in Figure 4.28, show that the sprayed particles only adhered onto exposed copper particles of the co-cured layer, on which they continued to accumulate upon further spray. This resulted in a columnar coating deposition with poor lateral consolidation as hinted by the porosity distribution. It is also interesting to note that deposition seemed to occur mostly on the highest peaks and not around them, creating defects like gaps and pits. It is conjectured that such defects were caused by particles following the gas flow which bypassed pits due to stagnation zones inside them. Nevertheless, LP-CGDS is not suitable for coating PMC materials with the current method because coatings are too dependent on the surface roughness of the substrate.



Figure 4.26: Sample LP03 coated with PGDS (left) and by LP-CGDS (right)

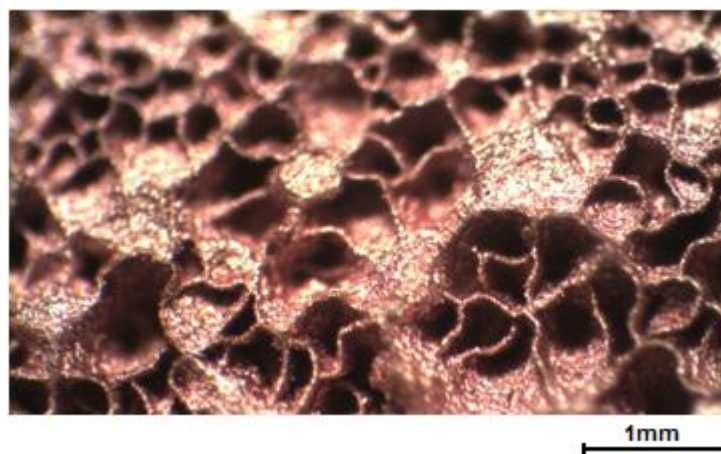


Figure 4.27: Surface of the pitted coating, sample LP03

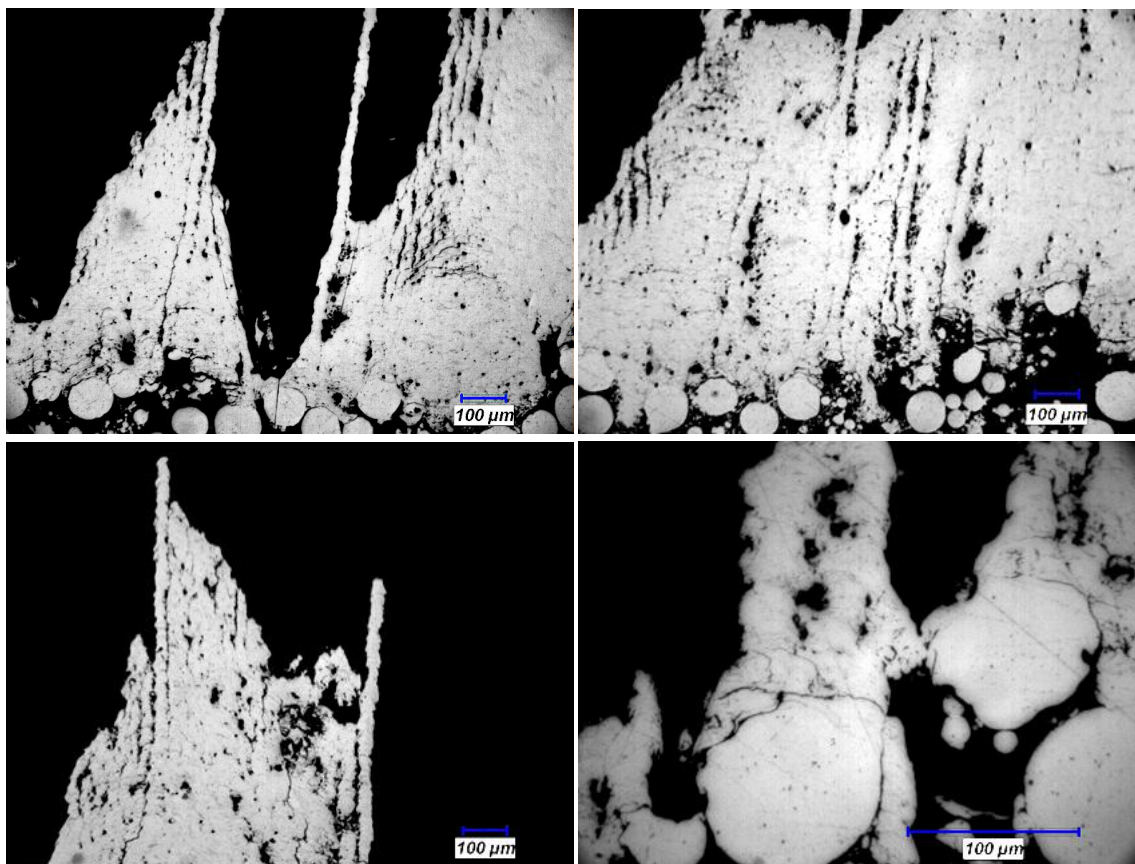


Figure 4.28: LP-CGDS coating cross-section, sample LP03

Seeing the results of LP-CGDS, the main question is why do coatings differ so much from those produced using PGDS? The major difference between the two processes is the method of accelerating the powder. For LP-CGDS, particles are continuously injected in a constant velocity flow whereas PGDS sprays in successive pulse. It is conjectured that in LP-CGDS, particle density in the flow is relatively low. When a particle hits the substrate it will either deposit or it will bounce off the substrate. For PGDS, particles in each pulse travel in aggregates, providing a high particle density. Some particles will deposit, whereas others will bounce off but they will still end up being trapped onto the substrate by the great number of incoming particles following them. This enabled filling of the gaps and producing coatings with a smooth surface. In this theory, troughs in a rough substrate are filled with particles travelling at a slower velocity, leading to higher porosity near the substrate-to-coating interface until the process completely fills the gaps. Such high porosity zones near the substrate-to-coating were observed in the PGDS process (Figure 4.15).

Nevertheless, further work with the LP-CGDS process may be warranted to confirm the above, and also because the process shows some promise of producing fully dense coatings using commercially available equipment (Figure 4.29).

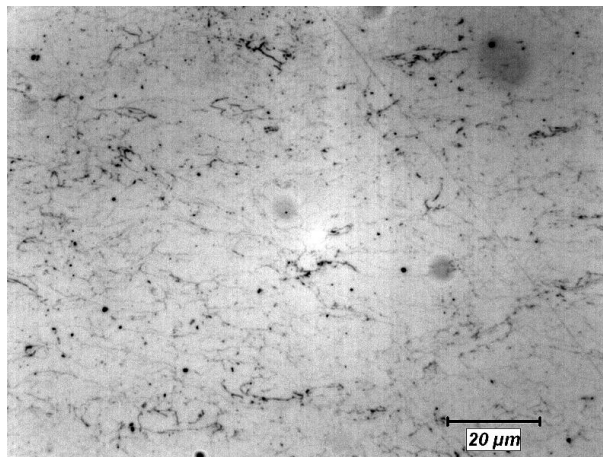


Figure 4.29: Fully consolidated coating regions, sample LP03 using LP-CGDS

4.2.8 Summary

The experimental programs discussed in the above sections provided valuable insights regarding the deposition of copper onto PMCs using cold spray processes. The main conclusion is that effective deposition of copper coatings on PMCs was possible due to the use of the co-cured layer. This layer was vital for preventing PMC erosion. However, this layer imposed limitations to the coating process and should be further investigated in future work. Roughness of the co-cured layer increased coating porosity at the substrate-to-coating interface and prevented proper deposition of a coating using LP-CGDS. Nevertheless, dense coatings were achievable with PGDS.

Another important milestone is that deposition of copper was achieved without heating the powder or the gas permitting low oxide content coatings which are desirable for good thermal and electrical conductivities.

Guidelines for coating deposition could be developed. It was found that coating density is promoted by high gas pressures and limited gas heating. Gas temperature must

be lower than the glass transition of the polymer matrix to mitigate the risk of erosion. Powder pre-heating is also recommended if it can be achieved in an inert environment preventing oxidation. Powder morphology also plays an important role which should be analysed further. Finally, for the powders tested, optimal coating conditions consist of a gas pressure higher than 3MPa and a gas heater temperature of 90°C for the out-of-autoclave CPMC substrates, a temperature slightly lower than the glass transition.

4.3 Coating machinability

As part of this research work, CPMCs needed machining and surface preparation to abide to certain test requirements. For example, precise dimensions were needed for short-span three point bending specimens. The specimens were cut, grinded, polished and milled to specifications. Among these processes, milling highlighted interesting elements of the behaviour of coatings that may have practical implications in manufacturing.

Before discussing coating machinability, a quick review of the machinability of pure copper is presented. The ASM speciality handbook of copper and copper alloys [139], quotes a pure copper machinability rating of 20%, suggesting that the material is difficult to machine, its surface finish after machining may be poor, and that it leads to lower cutting tool life. The overbearing factor is that copper is highly ductile. Upon machining, copper creates long chips that hinder heat removal. Also, pure copper has a tendency to weld itself to the cutter, impairing surface finish. Finally, it is reported that optimum cutting speed for pure copper is between 15-46m/min.

Based on machining recommendations for the milling machine used (Craftex Vertical Knee Mill), CPMCs were milled at 28m/min and 0.1mm of material was removed per pass, using a 12.7mm 4-flute HSS cutter. As seen in Figure 4.30, coatings could be machined. It should be noted that the depressions seen in the picture are not milling defects, but they correspond to thinner coated regions that were not milled.

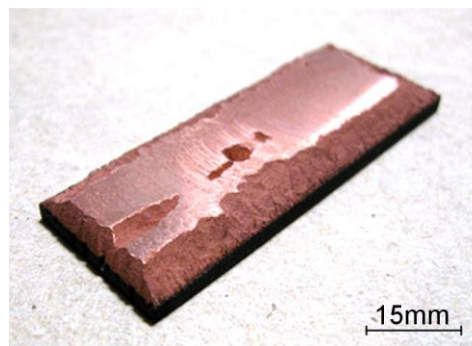


Figure 4.30: Coated specimen with a milled surface

Machinability of the coating proved to be fair but the behaviour differed greatly from that of pure copper. The greatest difference was that the chips were very short and tended to accumulate in the cutter. Furthermore, machining marks were left on the surface of the coating. Microscopic inspection of these milling strikes on Cu-105-3 coatings showed roughness that looked similar to copper particles (Figure 4.31). Hence, this likely resulted from poorly consolidated particles that were removed by shearing of particle interfaces during milling. From this, it can be deduced that denser coatings may provide a better surface finish. This was confirmed by milling SST-C5003 coatings (Figure 4.31), where no significant amounts of coating marks having a particle shape were observed. However, surface quality was hindered by coating defects.

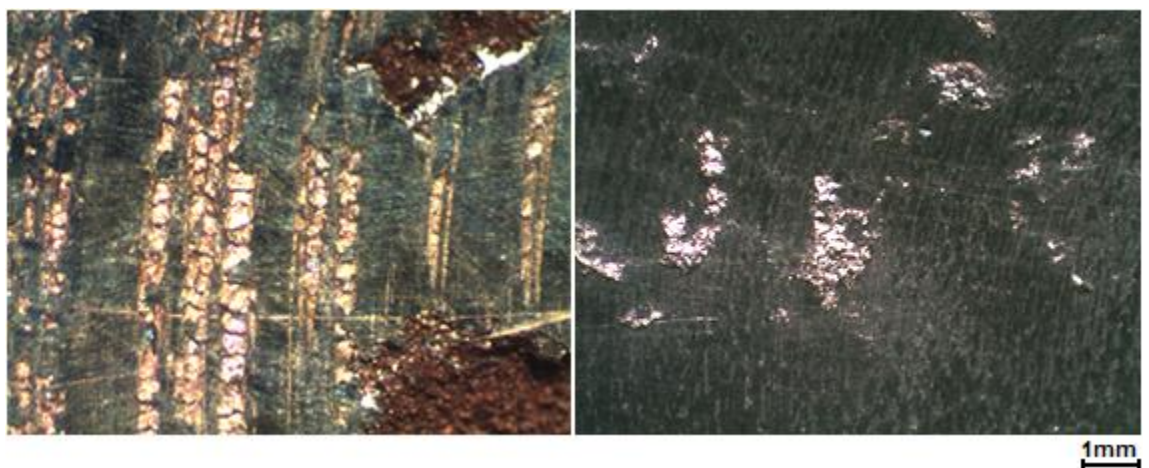


Figure 4.31: Milled coating surface of Cu-105-3 (left) and SST-C5003 (right)

As observed and discussed with University of Ottawa machinists Stan Weedmark and Michael Burns, life expectancy of the cutter appeared to be lower for copper coatings than for pure copper. This may be partly attributable to the short chips that accumulated on the cutter and hampered heat removal. Moreover, shorter life may also have been caused by greater hardness of the sprayed copper particles, resulting from the significant amount of plastic deformation as discussed in the next section. Thus, having hard particles striking the cutters likely promoted erosion of the blades.

As mentioned above, heat removal during milling was an issue. During one milling operation on a CPMC, only the PMC was constrained and heat accumulated in the material, resulting in a compressive failure of the PMC from thermal expansion (Figure 4.32). This partially delaminated the coating at the co-cured layer-to-coating interface, which is likely the weakest interface. From this it can be concluded that using a sufficient amount of cutting fluid is imperative when machining coatings.

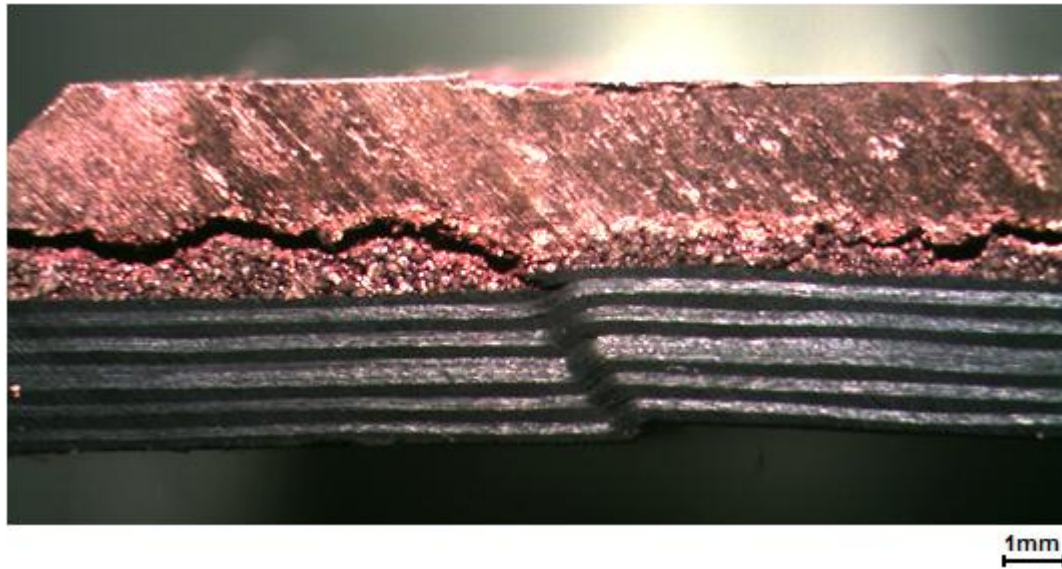


Figure 4.32: Compressive failure of the constrained PMC due to heat accumulation during milling

4.4 Experimental program “TR”: CPMC resistance to elevated temperatures

Temperature resistance of two CPMCs was investigated in experimental program “TR”. The coating on both samples was produced using the following parameters: $P_{\text{Gas}} = 3.5\text{MPa}$ and $T_{\text{Gas}} = 90^{\circ}\text{C}$. The first sample (TR01) was a milled Cu-105-3 CPMC having a width and length of 15mm, and thickness of approximately 4.5mm (Figure 4.33). The coating was deposited on substrate OOA-03 with PGDS system #2. The second sample (TR02) was a CPMC with a SST-C5003 coating deposited on substrate OOA-09 with PGDS system #3. The sample was 25mm wide, 43mm long and 5.5mm thick (Figure 4.33).

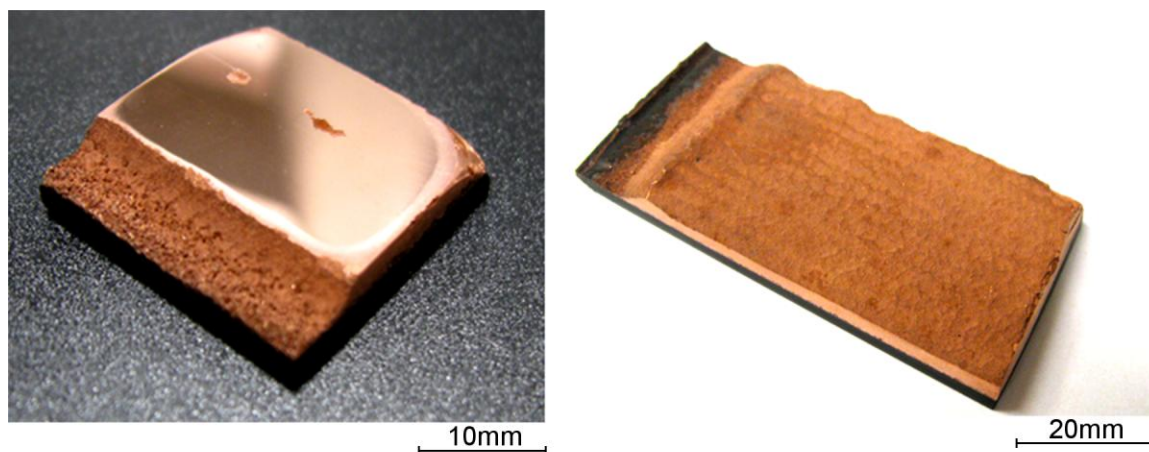


Figure 4.33: Samples TR01 (left) and TR02 (right) before thermal cycling

The samples were heated in 4 sequential cycles reaching maximum temperatures of 150°C , 200°C , 250°C and 250°C respectively (Figures 4.34-4.35). During testing both samples showed the same behaviour. After one hour at 150°C the copper surface started oxidising and there were no visual signs of coating spallation. Microscopic observations did not reveal any delamination or damage. After heating to 200°C for one hour the coatings further oxidised to a yellow colour. Again, there were no signs of coating spallation and microscopy of the substrate-to-coating interface did not show any damage. The sample was then heated up to 250°C once, again for one hour. The surface of the coating tarnished to a bronze colour, showing greater oxidation. There were no signs of delamination, but microscopic inspection showed few copper particles of the co-cured layer

being removed from the material. Another cycle at 250°C for one hour was performed to assess subsequent deterioration. The coating still did not show any sign of spallation but additional copper particles were forced out of the embedded layer (Figure 4.36). Damage was minimal but seemed to increase upon each 250°C heating cycle. Furthermore, on TR02 the damage was greater on the edge with the rougher cut, suggesting that the cutting process can weaken the material. Nevertheless, this experimental program showed that the CPMCs with the OOA substrates could resist temperatures of at least 250°C, which is higher than the cure temperature required for manufacturing SE-70 laminates.

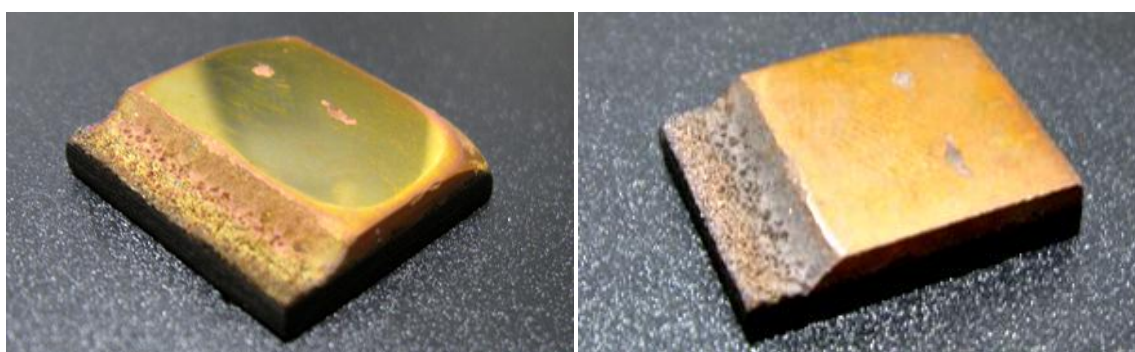


Figure 4.34: Sample TR01 after cycle at 200°C (left) and after 2nd cycle at 250°C (right)

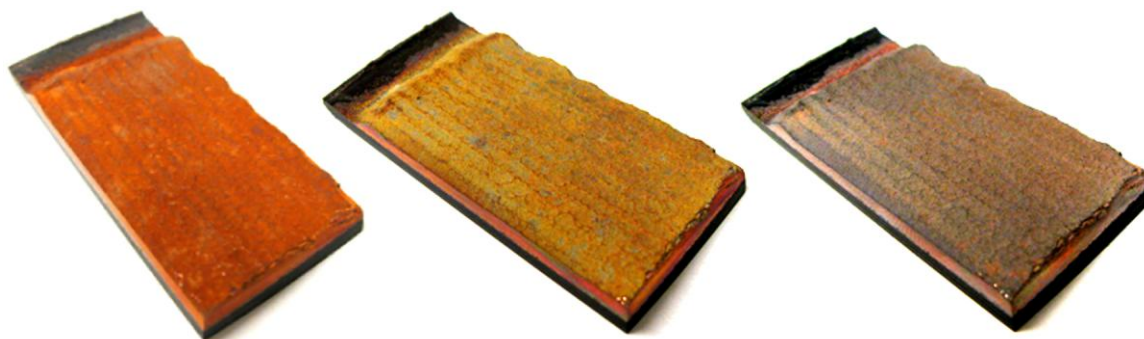


Figure 4.35: Sample TR02 after cycles at 150°C (left), at 200°C (centre) and 2nd cycle at 250°C (right)

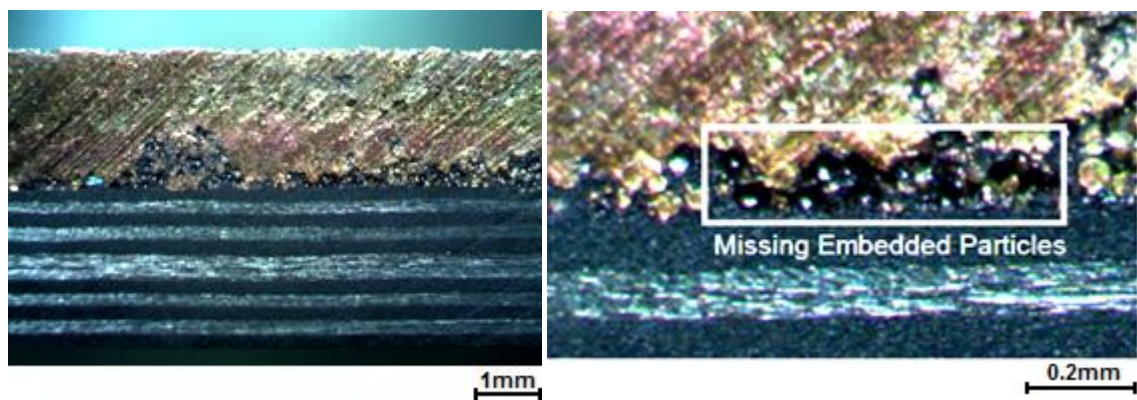


Figure 4.36: TR01 co-cured layer particles removed after heating at 250°C, lower magnification (left) and higher magnification (right)

4.5 Mechanical testing

4.5.1 Micro-hardness

Micro-hardness of CPMC copper coatings was measured. Samples were selected from experimental programs GP, GT, PH and LP. Measurements for PH18 were divided in two categories (porous or dense regions) due to the great difference in local porosity. Furthermore, measurements were taken on 4 off-cuts of SSB samples and regrouped as sample SSB00. Spray parameters for each sample are presented in Table 4.13.

Table 4.13: Samples used for micro-hardness measurements

Sample	System	Substrate	Powder	Gas	P _{Gas} (MPa)	T _{Gas} (°C)	T _p (°C)
GP09	PGDS #1	OOA-02	Cu-105-3	He	3.0	25	25
GP12	PGDS #1	OOA-02	Cu-105-3	He	3.5	25	25
GP15	PGDS #1	OOA-02	Cu-105-3	He	4.0	25	25
GT25	PGDS #1	OOA-02	Cu-105-3	He	4.0	100	25
PH18	PGDS #1	OOA-02	Cu-105-3	He	4.0	25	200
SSB00	PGDS #2	OOA-07	SST-C5003	He	3.5	90	25
LP03	LP-CGDS	OOA-05	SST-C5003	He	1.7	90	25

Micro-hardness results appear in Table 4.14. The GP samples show that hardness increases slightly with the gas pressure while its standard deviation decreases. This logically derives from the greater spray particle deformation imparted by an increase in gas pressure. Comparing GP15 and GT25 it can be seen that heating the gas to 90°C reduces hardness. Heating results in particle annealing and softening that promotes coating density as discussed in section 4.2.5. Similarly, annealing of the sprayed particles from powder pre-heating results in lower coating hardness. However, as shown by the porous coating region of PH18, pre-heating can produce an oxide layer on particles that increases hardness and hinders coating consolidation. It is also interesting to note that the SST-C5003 coating

on SSB00 is slightly harder than the Cu-105-3 coating on TG25 which was achieved with nearly the same spray parameters. This likely results from the higher coating consolidation of the prior. Finally, considering that the coating of LP03 was made at a lower gas pressure than SSB00, it can be deduced that LP-CGDS can offer similar coating hardness and consolidation capabilities than PGDS.

Table 4.14: Micro-hardness results

Sample	Hardness (HV _{0.1})			
	Average	Standard deviation	Maximum	Minimum
PG09	131	±20	159	98
PG12	136	±21	170	104
PG15	144	±14	162	121
TG25	127	±23	155	88
PH18 (Porous)	124	±14	150	101
PH18 (Dense)	97	±23	126	43
SSB00	135	±20	166	95
LP03	123	±15	144	87

4.5.2 Flexural modulus of SE-70 laminates

The flexural modulus of SE-70 as a function of temperature was measured using ASTM standard D780-02. Samples were made of a 12-ply crossply SE-70 laminate. A span of 75mm was used, providing a span-to-thickness ratio greater than 25. Testing was done at 25°C, 70°C and 120°C with three samples tested at each temperature. Flexural modulus E_B was calculated with the following equation [140]:

$$E_B = \frac{FL^3}{4bh^3\nu} \quad (4.1)$$

Dimensions and flexural modulus data for SE-70 laminates are presented in Table 4.17. At room temperature, an average flexural modulus of 70GPa was obtained. This is in agreement with the technical data sheet value of 68GPa [110]. At higher temperatures the flexural modulus decreased non-linearly. As expected, at 70°C the flexural modulus was similar to the value at room temperature, but it dropped to a value of 15GPa after heating past the glass transition temperature (Figure 4.37). The values are used at a later stage in this thesis as material layer properties for stress calculations in short-span bending strength of CPMCs.

Table 4.15: Dimensions and flexural modulus of SE-70 laminate

Temperature	Sample	Length (mm)	Width (mm)	Thickness (mm)	Flexural modulus (GPa)
25°C	SE-70-01	92.37	13.11	2.68	66.0
	SE-70-02	92.08	13.20	2.85	66.8
	SE-70-03	91.95	15.04	2.09	77.7
70°C	SE-70-04	91.50	13.51	2.89	59.2
	SE-70-05	92.11	13.46	2.74	63.9
	SE-70-06	91.30	14.14	2.19	57.2
120°C	SE-70-07	92.09	13.26	2.79	9.9
	SE-70-08	91.94	13.08	2.73	10.7
	SE-70-09	91.57	15.02	2.23	22.9
	SE-70-10	91.91	13.16	2.65	15.4

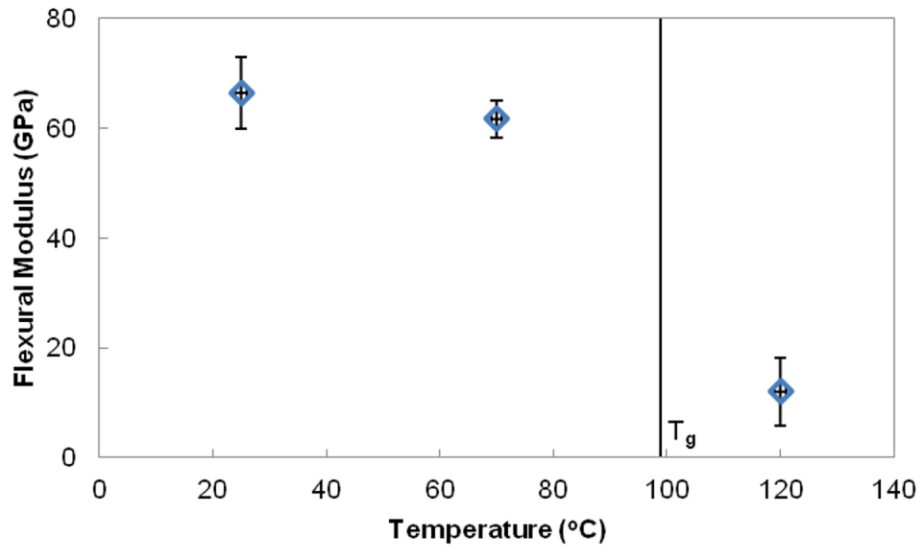


Figure 4.37: Variation of flexural modulus with temperature of crossply SE-70

4.5.3 Experimental program “SSB”: CPMCs short-span bending strength

Experimental program “SSB” evaluated the short-span bending strength and modes of failure of CPMCs at 25°C, 70°C and 120°C. Coatings were produced with $P_{\text{Gas}} = 3.5\text{MPa}$ and $T_{\text{Gas}} = 90^\circ\text{C}$. Descriptions of all the samples appear in Table 4.16 and

Table 4.17. It is important to note that most samples featured significant coating defects due to manual operation of the X-Y table in PGDS system #2 manual. Therefore, an automated system would likely lead to short-span bending strength values higher than what is presented. Two loading methods were used (Figure 4.38). Samples SSB01 to SSB08 were loaded on the coating, subjecting the coating to axial compression. Conversely, samples SSB09 to SSB21 were loaded on the opposite side, yielding axial tension in the coating.

Table 4.16: Description of short-span three-point bending samples SSB01 to SSB08

Sample	Substrate	Powder	Dimensions			Observations before bending
			Thickness (mm)	Width (mm)	Length (mm)	
SSB01	OOA-05	Cu-105-3	4.87	12.19	31.33	Cracks at interface
SSB02	OOA-05	Cu-105-3	4.93	11.63	40.57	Cracks at interface
SSB03	OOA-06	Cu-105-3	4.89	11.72	41.36	Coating defects
SSB04	OOA-07	SST-C5003	4.92	13.33	40.04	Good coating
SSB05	OOA-07	SST-C5003	5.04	12.75	43.52	Cracks at interface
SSB06	OOA-08	SST-C5003	5.01	12.15	43.90	Few cracks at interface
SSB07	OOA-08	SST-C5003	4.89	11.40	42.80	Good coating
SSB08	OOA-08	SST-C5003	5.12	12.12	42.73	Good coating

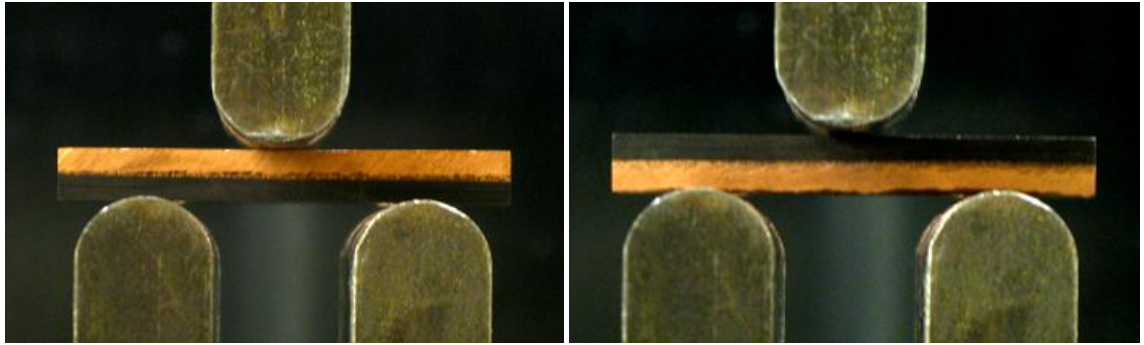


Figure 4.38: Coatings loaded in axial compression (left) and axial tension (right)

Table 4.17: Description of short-span three-point bending samples SSB09 to SSB21

Sample	Substrate	Powder	Dimensions			Observations before bending
			Thickness (mm)	Width (mm)	Length (mm)	
SSB09	OOA-05	Cu-105-3	5.06	11.76	36.28	Cracks at interface and coating defects
SSB10	OOA-05	Cu-105-3	4.93	12.12	32.76	Defects in the coating
SSB11	OOA-06	Cu-105-3	4.92	11.82	40.22	Cracks at interface and coating defects
SSB12	OOA-07	SST-C5003	4.90	13.16	42.23	Cracks at interface and coating defects
SSB13	OOA-07	SST-C5003	5.16	12.69	43.30	Cracks at interface and coating defects
SSB14	OOA-08	SST-C5003	5.07	12.41	43.03	Cracks at interface
SSB15	OOA-06	SST-C5003	5.06	12.48	41.41	A third of the coating already delaminated
SSB16	OOA-07	SST-C5003	5.21	13.37	43.06	Few coating defects
SSB17	OOA-08	SST-C5003	4.86	12.55	39.85	Good coating
SSB18	OOA-08	SST-C5003	4.96	12.73	45.04	Cracks at interface and coating defects
SSB19	OOA-05	SST-C5003	5.04	13.31	38.79	Good coating
SSB20	OOA-07	SST-C5003	4.65	13.25	44.64	Few coating defects
SSB21	OOA-08	SST-C5003	5.01	13.31	42.90	Few cracks at interface

4.5.3.1 Coating in axial compression

Loading the coating side always resulted in the failure of the CPMCs by coating delamination (Figure 4.39). At failure, a crack appears at the sample edges and it instantaneously propagates up to the sample centre along the substrate-to-coating interface. The crack then deviates to shear the coating through its thickness. It is interesting to note that the naturally ductile copper undergoes brittle failure. This is caused by the cold spray processes, which relies primarily on mechanical interlocking between particles to form a

coating. Since this type of bonding is weaker than pure metallic bonding, it is easier to shear the particle-to-particle interface than to deform the particles.

Microscopic inspection of fracture surfaces (Figure 4.40) shows co-cured layer particles and coating sections present on both the substrate and coating surfaces. Hence, the weakest region of the CPMCs under this loading case was the coating-to-co-cured layer interface.

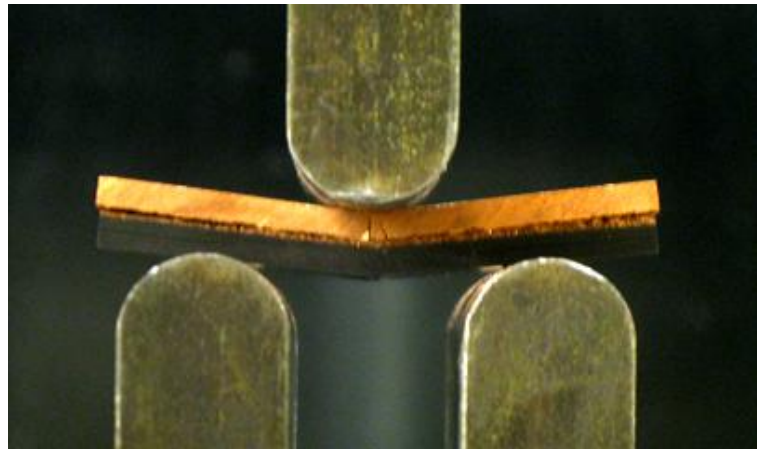


Figure 4.39: Typical failure due to coating delamination

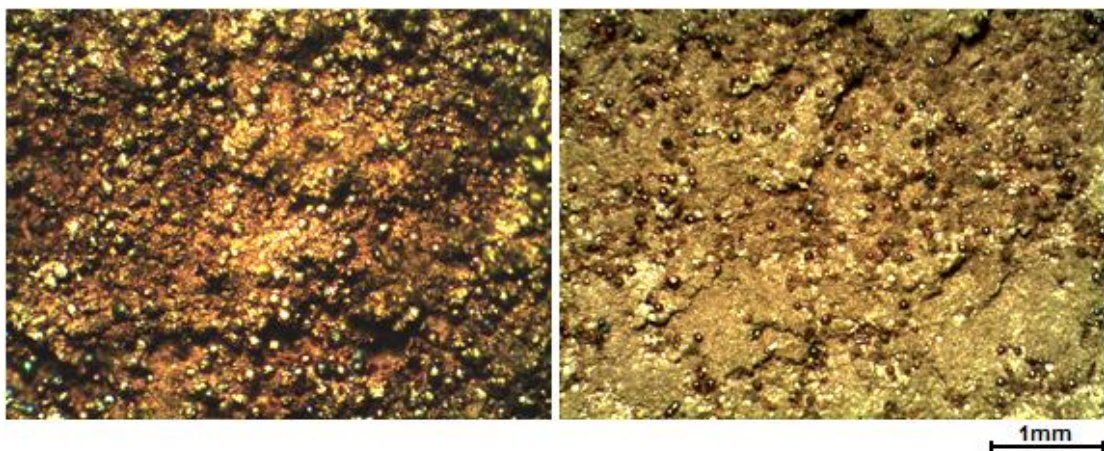


Figure 4.40: Fracture surface on co-cured layer side (left) and coating side (right)

Force-displacement curves for all samples appear in Appendix E. Measured forces at failure in CPMCs appear in Table 4.18. It can be seen that forces at failure are more

consistent with the Cu-105-3 than with the SST-C5003. Inconsistencies seen with the latter powder are likely attributable to non-deformed SST-C5003 dust particles at substrate-to-coating interface, as reported in section 4.2.1. This becomes more apparent when looking at failure forces at 70°C and 120°C, which are higher than some forces recorded at 25°C despite the reduction in PMC stiffness with increasing temperature.

Table 4.18: SSB forces at failure for coatings under axial compression

Sample	Powder	Temperature (°C)	Force at failure (N)
SSB01	Cu-105-3	25	1469
SSB02	Cu-105-3	25	1588
SSB03	Cu-105-3	25	1285
SSB04	SST-C5003	25	1511
SSB05	SST-C5003	25	596
SSB06	SST-C5003	25	717
SSB07	SST-C5003	70	1205
SSB08	SST-C5003	120	829

Through-thickness axial stresses at mid-span and shear stresses were evaluated from the forces at failure, sample dimensions and elastic properties of the materials. Calculations were made from Equations (3.18) and (3.19). The elastic moduli were assumed as 100GPa for the coating based on literature ([81, 141]) and as 7GPa for the co-cured layer based on the Mori-Tanaka model, Equation (2.18), with a particle volume fraction of 55%. Finally, the elastic modulus of the PMC layer was taken from experimental data presented in section 4.5.2. Typical curves for the through-thickness axial and shear stresses are presented in Figure 4.41, with the remainder of the curves for other samples appearing in Appendix F. At room temperature and at 70°C the neutral axis passed through or near the co-cured layer. Coupled with a low elastic modulus, the co-cured layer was mainly affected by shear stresses and therefore the synergetic effect with the axial stresses was neglected in the calculations. At 120°C the stiffness of the PMC layer decreases significantly which moved the neutral axis of the SSB08 sample into the coating, leaving the co-cured layer subjected to both axial and shear stresses. Calculations also showed that shear stresses at all temperatures were nearly constant through the entire co-cured layer. Conversely, the

coating and PMC layers were subjected to maximum axial stresses at the outer surfaces of the CPMCs where shear stresses are negligible. It is important to note that the actual stress field in the CPMCs likely differs from calculated stresses obtained through the short-span bending theory. Since the effects of local compression of the loading nose and material interfaces are neglected, the beam theory potentially underestimates the stress at failure. Nevertheless, it can be used as a comparison tool between similar samples.

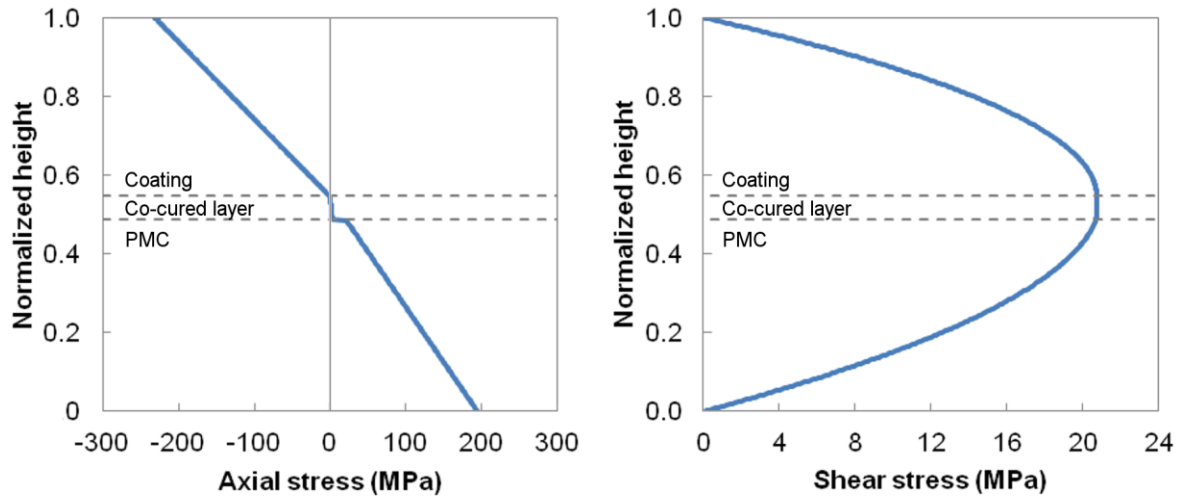


Figure 4.41: Mid-span axial (left) and shear (right) stress distributions in SSB02 at failure

Maximum axial stresses in the coating and PMC layers, as well as the maximum shear stress in the co-cured layer, are presented in Table 4.19. Results show that the shear stress required for breaking the coating-to-co-cured layer interface is approximately 18MPa for Cu-105-3 at room temperature. For SST-C5003 it is impossible to assess an average shear stress value due to large data scatter caused by the powder. Moreover, the variation of shear stress at failure could not be correlated to the temperature due to limited data and lack of a general trend. Nevertheless, it is seen that both Cu-105-3 and SST-C5003 coatings were able to sustain at least 180MPa in compression, irrespective of the tested temperature.

Table 4.19: Maximum axial and shear stresses at failure for coatings under axial compression

Sample	Powder	Temperature (°C)	σ_{Coating} (MPa)	σ_{PMC} (MPa)	$\tau_{\text{Co-cured}}$ (MPa)
SSB01	Cu-105-3	25	-209	175	18.5
SSB02	Cu-105-3	25	-231	194	20.7
SSB03	Cu-105-3	25	-190	158	16.6
SSB04	SST-C5003	25	-194	163	17.3
SSB05	SST-C5003	25	-76	64	6.9
SSB06	SST-C5003	25	-97	81	8.8
SSB07	SST-C5003	70	-189	148	16.1
SSB08	SST-C5003	120	-182	62	10.3 ^a

^a Maximum shear stress of SSB08 at the co-cured layer was calculated from Cartesian stresses due to the presence of axial and shear

stresses, using: $\tau_{\text{max}} = \sqrt{\sigma_x/2^2 + \tau_{xy}^2}$

4.5.3.2 Coating in axial tension

Loading the PMC side resulted in CMPC failure by coating fracture (Figure 4.42). At failure, a crack initiated on the outer coating surface at mid-span and propagated instantaneously in the thickness of the CMPC until stopped by the less brittle co-cured layer. At that point, the coating remained attached to the CMPC. As seen on Figure 4.43, upon applying a considerably greater amount of force delamination of the coating occurred. This case is a very good demonstration of the brittle nature of the coating as fracture preceded delamination, and it also shows the change in behaviour of the coating subjected to tension and compression.

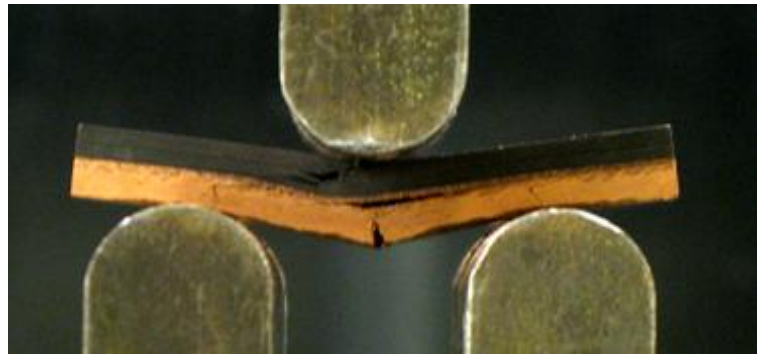


Figure 4.42: Failure initiating at coating surface

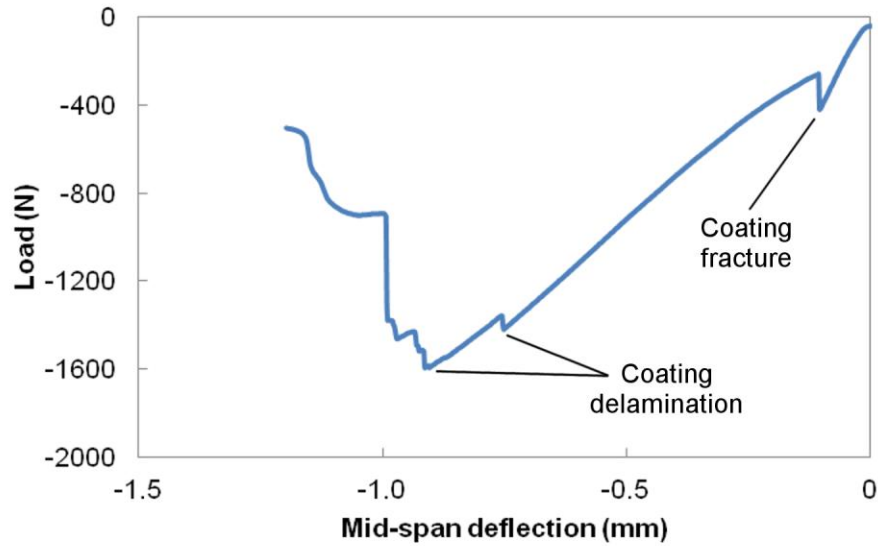


Figure 4.43: Load-deflection curve for SSB09

Force-displacement curves for all samples appear in Appendix E. Measured forces at failure for CPMCs appear in Table 4.20. The values have low variability for each combination of powder and temperature with the exception of tests conducted at 120°C. Unlike the previous set of tests (SSB01-SSB08), results indicate that Cu-105-3 samples exhibited a much lower resistance to failure than SST-C5003 samples. Comparing the modes of failure for both powders, it can be deduced that Cu-105-3 provides greater coating adhesion with the co-cured layer while the SST-C5003 yields better coating cohesion strength. It is also seen that force at failure decreases with an increase in temperature.

Table 4.20: SSB forces at failure for coatings under axial tension

Sample	Powder	Temperature (°C)	Force at failure (N)
SSB09	Cu-105-3	25	418
SSB10	Cu-105-3	25	444
SSB11	Cu-105-3	25	404
SSB12	SST-C5003	25	779
SSB13	SST-C5003	25	746
SSB14	SST-C5003	25	754
SSB15	SST-C5003	70	212 ^a
SSB16	SST-C5003	70	690
SSB17	SST-C5003	70	727
SSB18	SST-C5003	70	735
SSB19	SST-C5003	120	507
SSB20	SST-C5003	120	421
SSB21	SST-C5003	120	384

^aLow force at failure caused by major coating defects

Mid-span CMPC stresses at failure are presented in Table 4.21. Again the shifting of the neutral axis away from the co-cured layer at 120°C was accounted for in the calculations of maximum shear stresses. Estimated results show that Cu-105-3 coatings failed under a tensile stress of 59MPa while SST-C5003 coatings failed at 96MPa. The lower cohesion of Cu-105-3 was likely attributable to its greater porosity in coatings. The coatings also showed tensile strengths inferior to their compression strengths. The estimated value for SST-C5003 is less than half the strength of annealed copper at room temperature (209MPa) [121], again proving that particle-to-particle interlocking from mechanical deformation is weaker than metallic bonding. Potentially, this interlocking could benefit from copper diffusion between particles. The low thermal resistance of the SE-70 used in this study prevented copper annealing, but this may work with other composites. The results also show that even if the force at failure was reduced with increasing temperature, there was no drop in coating strength within the temperature range tested. The reduction in force at failure was thus caused by the PMC epoxy going into a rubbery state, reducing the stiffness of the CMPCs.

Table 4.21: Maximum axial and shear stresses at failure for coatings under axial tension

Sample	Powder	Temperature (°C)	σ_{Coating} (MPa)	σ_{PMC} (MPa)	$\tau_{\text{Co-cured}}$ (MPa)
SSB09	Cu-105-3	25	57	-48	5.3
SSB10	Cu-105-3	25	61	-52	5.5
SSB11	Cu-105-3	25	59	-49	5.2
SSB12	SST-C5003	25	101	-85	9.1
SSB13	SST-C5003	25	91	-76	8.5
SSB14	SST-C5003	25	97	-82	9.0
SSB15	SST-C5003	70	28	-22	2.5
SSB16	SST-C5003	70	81	-64	7.3
SSB17	SST-C5003	70	105	-82	8.9
SSB18	SST-C5003	70	101	-78	8.7
SSB19	SST-C5003	120	105	-36	5.4 ^a
SSB20	SST-C5003	120	102	-37	5.5 ^a
SSB21	SST-C5003	120	80	-28	4.2 ^a

^a Maximum shear stress at the co-cured layer was calculated from Cartesian stresses due to the presence of axial and shear stresses,

using: $\tau_{\text{max}} = \sqrt{\sigma_x/2^2 + \tau_{xy}^2}$

4.5.4 Experimental program “LCF”: low frequency cycling of CPMCs

Low frequency cycling was performed on CPMCs during experimental program “LCF”. Samples are described in Table 4.22. Cyclic loading was done on the PMC outer surface of CMPCs to generate axial tensile stresses in coatings. This loading case was selected because it showed the lowest load resistance during the “SSB” program. Load amplitude was controlled during cycling and its value was selected based on the average static loads at failure reported in the “SSB” program. Coating stresses induced by the loads were also calculated for comparison.

Table 4.22: Description of low frequency cycling samples

Sample	Substrate	Powder	Dimensions			Observations before bending
			Thickness (mm)	Width (mm)	Length (mm)	
LFC01	OOA-08	SST-C5003	4.87	11.49	42.05	Few cracks at interface
LFC02	OOA-07	SST-C5003	5.21	13.37	43.06	Few cracks at interface
LFC03	OOA-08	SST-C5003	4.74	12.90	42.29	Coating defects and cracks at interface
LFC04	OOA-08	SST-C5003	4.95	11.97	45.13	Coating defects and cracks at interface
LFC05	OOA-07	SST-C5003	5.05	13.03	43.62	Few coating defects
LFC06	OOA-08	SST-C5003	4.98	12.14	41.65	Few coating defects
LFC07	OOA-08	SST-C5003	4.76	12.49	42.06	Coating defects and cracks at interface
LFC08	OOA-07	SST-C5003	4.88	13.22	44.35	Few cracks at interface

Test conditions and results are presented in Table 4.23, whereas all cycling curves appear in Appendix G. At room temperature and at 70°C, samples could be cycled 100 times at 60% of their maximum static force at failure without any visible damage. Further loading resulted in the failure of LFC04 at 488N during the first cycle. Surprisingly, from theoretical calculation, this is equivalent to a 71MPa coating stress, which is significantly lower than the maximum stress at failure observed in the “SSB” program and was likely caused by coating defects. At 120°C, two tests were performed at 60% of the CMPC maximum static force at failure. Unlike at lower temperatures, sample failure was observed and results showed that LFC07 was cracked before cycling began while LFC06 failed after 66 cycles. However, another test was performed at the same temperature but at a higher loading of 70% of the maximum static load at failure. Cycling results for LFC08 sample showed that it could withstand 100 cycles without visible damage. This shows that CMPCs

are greatly affected by coating defects. In this case, it was conjectured that defects resulted mainly from the lack of an automated X-Y table for the coating process of PGDS system #2. From the results, reducing defects may likely yield CMPCs having better mechanical properties than what is reported in this work.

Table 4.23: LCF samples testing conditions and results

Sample	Temperature (°C)	Cycling force (N)	Coating stress (MPa)	Force ratio	Stress ratio	# of cycles	Observations after cycling
LFC01	25	470	71	0.62	0.74	100	No visible damage
LFC02	70	405	48	0.56	0.5	100	No visible damage
LFC03	70	445	66	0.62	0.69	100	No visible damage
LFC04	70	580	85	0.81	0.89	0	Coating fracture at 488N
LFC05	120	200	42	0.46	0.44	100	No visible damage
LFC06	120	260	60	0.60	0.63	66	Coating fracture at 260N after 66 cycles
LFC07	120	260	64	0.60	0.67	0	Coating was already fractured
LFC08	120	300	67	0.69	0.70	100	No visible damage

Samples subjected to various cycling conditions displayed the characteristic fatigue behaviour of PMCs [142], wherein the maximum deflection increased with each successive cycle (Figure 4.44). The rate at which deflection increases is reminiscent of a creep curve. Initially, in a primary stage the rate was high but decreased with each cycle. In a secondary stage, the rate stabilised and deflection increased linearly with each cycle. In a tertiary stage the rate increased rapidly with each cycle until sample failure occurred, which always resulted from coating fracture.

A deflection rate was calculated in the linear secondary stage of deformation using the following equation:

$$\text{Deflection rate} = \frac{\nu_1 - \nu_0}{\Delta \text{Cycle}} \quad (4.2)$$

where ν is the maximum deflection at a given cycle and ΔCycle is the number of cycles required for the change in deflection.

Deflection rates for the relevant samples are presented in Table 4.24. For LFC01 at room temperature, the deflection rate was 1.4×10^{-5} mm/cycle. At 70°C, LFC02 showed a similar deflection rate for a comparable loading based on the ratio of maximum static force at failure, at around 60%. However, LFC03 which was subjected to slightly higher loads showed an absence of increase in deflection for the secondary stage. This phenomenon for LFC03 is not well understood. It is conjectured that it may be due to a balance between crack growth and strain hardening in the coating, and to a better coating quality. At 120°C, LFC05 showed the same deflection rate as LFC01 at room temperature. However, its loading force based on the maximum static force at failure was much lower at 46%. Increasing the applied load resulted in an increase of deflection rates, which in the case of LFC05 and LFC06 were much higher than LFC01 at room temperature. Since the increase in deflection rate happened at 120°C, it can be conjectured that the weakening was mostly caused by the epoxy in the PMC which was in its rubbery state.

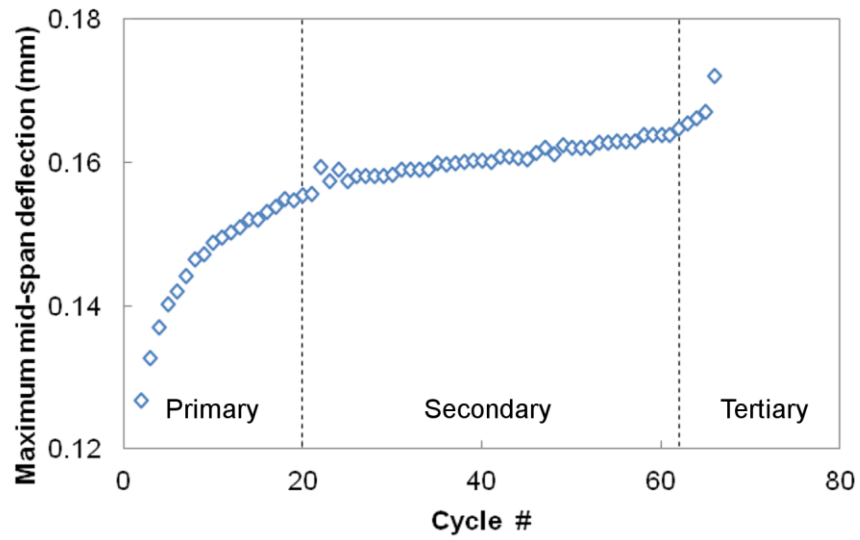


Figure 4.44: Typical mid-span deflection cyclic curve for LFC samples

Table 4.24: Deflection rate of LFC samples

Sample	Temperature (°C)	Cycling force (N)	Deflection rate (mm/cycle)
LFC01	25	470	1.4×10^{-4}
LFC02	70	405	1.3×10^{-4}
LFC03	70	445	0
LFC05	120	200	1.4×10^{-4}
LFC06	120	260	1.7×10^{-4}
LFC08	120	300	3.3×10^{-4}

4.6 Conclusions

Original contributions were made by developing a new method for applying copper coatings on PMCs and characterising the new material system. Copper coatings were applied onto PMCs featuring a co-cured layer of copper particles and were produced using the PGDS process. Various spraying parameters/conditions were tested for improving coating density. The parameters and their effect on coating density are presented in Table 4.25. Coating density was optimised for the Cu 105-3 powder and under the conditions tested, the best spraying parameters were a gas pressure of 3MPa-4MPa and a gas heater temperature of 90°C for the out-of-autoclave CPMC substrates.

Table 4.25: Effect of varying the spraying conditions on the coating density

Spraying condition	Coating density
Lowering displacement rate	Higher
Increasing gas pressure	Higher
Increasing gas temperature	Higher, but risk of erosion at high temperatures
Heating powder	Lower as there is oxidation
Smooth substrate surface finish	Higher compared to a coating made on a rough surface

Larger CPMCs were produced using the optimal spray parameters and two spray powders (Cu 105-3 and SST-C5003) for thermal and mechanical testing. The main results are presented in Table 4.26. An important conclusion was that the particle-to-particle bonding of the spray particles greatly affected the mechanical behaviour of the copper coating in CPMCs. Greater mechanical interlocking of the particles resulted in lower coating porosity, but it also improved the tensile strength of the coatings significantly. Moreover, since mechanical interlocking was achieved through plastic deformation, strain hardening of the spray particles increased the hardness of the coatings compared to that of bulk copper. Results also showed that CPMCs can resist at least a limited number of cycles at 250°C without any delamination, showing potential for IHCT applications. Additionally, CPMCs devoid of major defects could resist low frequency cycling at more than 60% of the maximum static tensile stress at failure for at least 100 cycles up to 120°C, further supporting their potential for IHCT applications.

Table 4.26: Summary of the results for the thermal and mechanical testing

Test	Observations
Machining	Short chips due to weaker spray particle-to-particle interface
Micro-hardness	Great plastic deformation of spray particles provides higher hardness than bulk copper
Resistance to elevated temperatures	No coating delamination observed after 2 cycles at 250°C
Short-span three-point bending	Failure of CPMC is governed by the coating and coating-to-co-cured layer interface
	Coating fracture is brittle
	A greater coating density improves tensile strength of coatings
	Dust left by the sprayed SST-C5003 can reduce greatly the shear strength of the CPMC.
Low frequency cycling	Low defect CPMCs can resist a stress of more than 60% of the maximal static stress for 100 cycles up to at least 120°C

Chapter 5

Integrally heated tooling prototype

Following the characterisation of the coated PMCs (CPMCs), work was undertaken towards the fabrication of larger CPMC parts. The contribution presented in this chapter is the fabrication of a prototype for an integrally heated composite tooling made out of CPMCs (IHCT-CPMC). Improved in-plane thermal conductivity provided by the copper coating showed great potential for IHCT-CPMC to be used in out-of-autoclave PMC manufacturing.

This chapter covers the manufacturing and inspection of the IHCT-CPMC, and it investigates the temperature distribution/uniformity at the PMC tool surface under various heating cycles. The IHCT-CPMC is also compared with three similar tooling plates (Figure 5.1): 1) a 4.0mm thick IHCT made of PMC devoid of copper (IHCT-PMC), 2) an IHCT made of a 4.0mm thick PMC structure and a 1.6mm thick copper sheet cladding bonded with silicone as developed by NRC-IAR (IHCT-NRC #1), and 3) a modified version of the IHCT-NRC consisting of a 4.0mm thick PMC structure with a co-cured 1.6mm thick copper sheet cladding (IHCT-NRC #2).

It is important to note that despite the comparison made between the copper coating and copper bonded sheet, the aim of the coating is to improve in-plane thermal conductivity and not to obtain the same conductivity value as that of bulk copper. A value similar to bulk copper is not required as the NRC-IAR successfully built and tested IHCTs made with aluminium in the past.

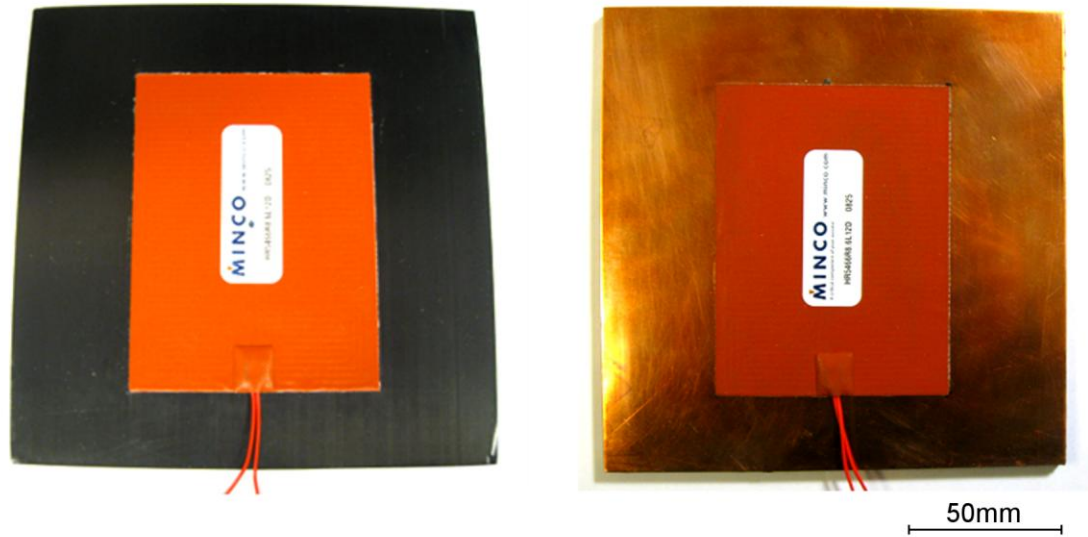


Figure 5.1: IHCT-PMC (left) and IHCT-NRC #1 (right) used for comparison

5.1 IHCT-CPMC manufacturing

The CPMC substrate for the IHCT-CPMC consisted of a 175mm by 175mm 20-ply SE-70 laminate with a co-cured layer of copper particles on its surface (Figure 5.2). A SST-C5003 copper coating was deposited using PGDS system #3 with spray parameters presented in Table 5.1. Unlike previous experiments, forced powder feeding was used instead of natural power feeding due to the greater holding capacity of the forced feeder which reduced the number of refills required.



Figure 5.2: Substrate for the IHCT-CPMC

Table 5.1: Spray parameters used for fabrication of the IHCT-CPMC

Parameter	Value
Gas pressure	3MPa
Gas temperature	90°C
Displacement rate	10mm/s (first half) 15mm/s (second half)
Standoff distance	7mm
Spacing between spray passes	2mm
Pulse rate	10Hz
Powder pre-heating	None
Feeder gas flow rate	10ml/s

The coating on the IHCT-CPMC (Figure 5.3) was achieved in a single spray pass. The coating process had to be interrupted three times for refilling the powder feeder and repositioning the clamps holding the IHCT-CPMC. After the 2nd refilling, the displacement rate was increased from 10mm/s to 15mm/s as the coating appeared too thick for the desired application. Overall, the coating process was accomplished without any problem

aside from occasional inconsistencies in the powder feeding. The resulting IHCT-CPMC (Figure 5.4) showed much less surface defects than the SSB specimens, attributable to the automated control of the X-Y table of PGDS system #3.

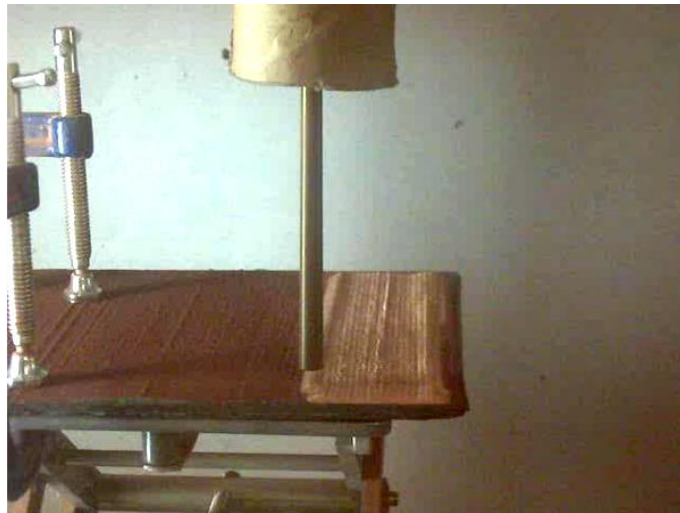


Figure 5.3: Picture of the coating process for the IHCT-CPMC

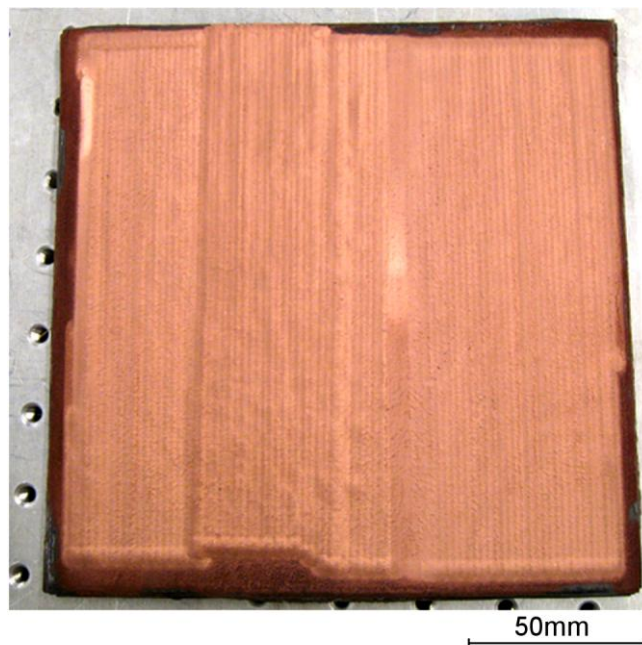


Figure 5.4: IHCT-CPMC after the coating process

The IHCT-CPMC was cut down to dimensions of 150mm by 150mm using a bandsaw. The coating surface was grinded slightly using SiC paper to obtain a flatter surface. A 75mm by 100mm region in the centre of the coating surface was milled using a 12.7mm 4-flute HSS cutter, for providing an adequate surface for bonding the heating element to the IHCT-CPMC. A MINCO HR5466R8.6L12D flexible heater was bonded onto the coating using MINCO PSA #12 adhesive film and cured in an oven at 150°C for 30min. The completed IHCT-CPMC is depicted in Figure 5.5 with labelled edges.

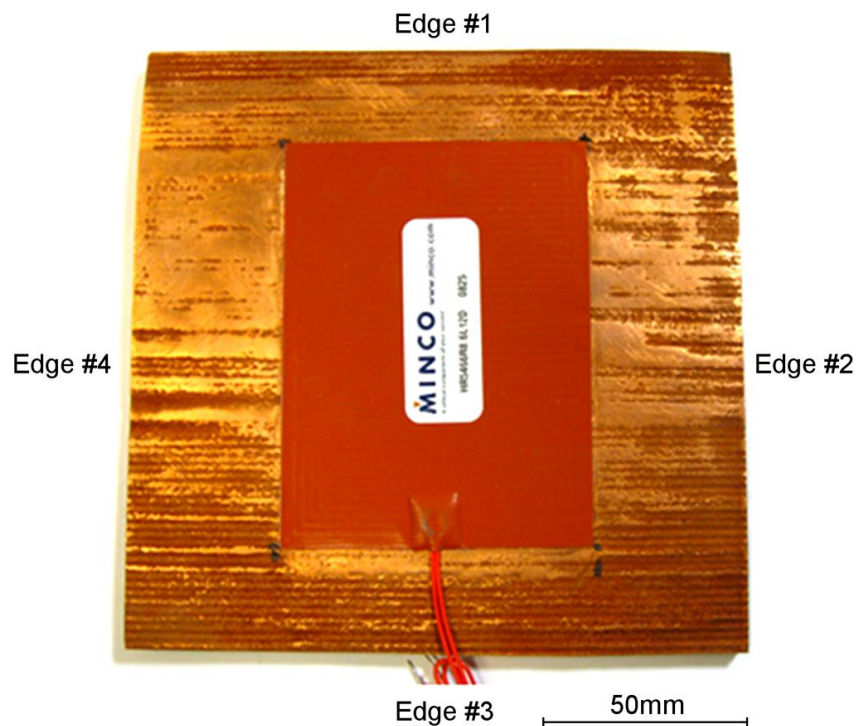


Figure 5.5: Coating side of the completed IHCT-CPMC

5.2 IHCT-CPMC inspection

5.2.1 Visible defects

No major defects were observed on the coating following the surface grinding and milling operations. Compared to the SSB samples, fewer spray defects were seen on the milled surface. Inspection of the cut edges of the IHCT-CPMC revealed minimal macroscopic coating defects. Generally, the coating appeared to be in intimate contact with the co-cured layer. However, there was slight coating delamination at the co-cured layer interface, on the corner uniting edges #3 and #4 (Figure 5.6). This was likely caused by SST-C5003 dust particles left on the co-cured layer and may have affected other internal regions of the IHCT-CPMC.



Figure 5.6: Coating delamination in one corner of the IHCT-CPMC

5.2.2 Thickness variation

Powder feeding inconsistencies during spraying and the different displacement rates used led to variations in coating thickness. Variation was quantified by taking thickness measurements along the edges of the IHCT-CPMC using a calliper ($\pm 0.01\text{mm}$). Results presented in Figure 5.7 show that along a spray line, i.e. edges #1 and #3, the coating thickness was relatively constant. However, it decreased non-linearly during spraying. The total CPMC thickness went from the initial 6.8mm of edge #1 to the 6.0mm of edge #3. As seen from edges #2 and #4, thinning occurred mid-way during spray, which concords with the change in displacement rate. Aside the change of displacement rate, the biggest cause of thickness variation was the conical shape of the spray lines, which left a rippled surface.

Such minor issues may be mitigated by reducing both the spacing between spray lines and the powder feed rate for spraying multiple thinner layers.

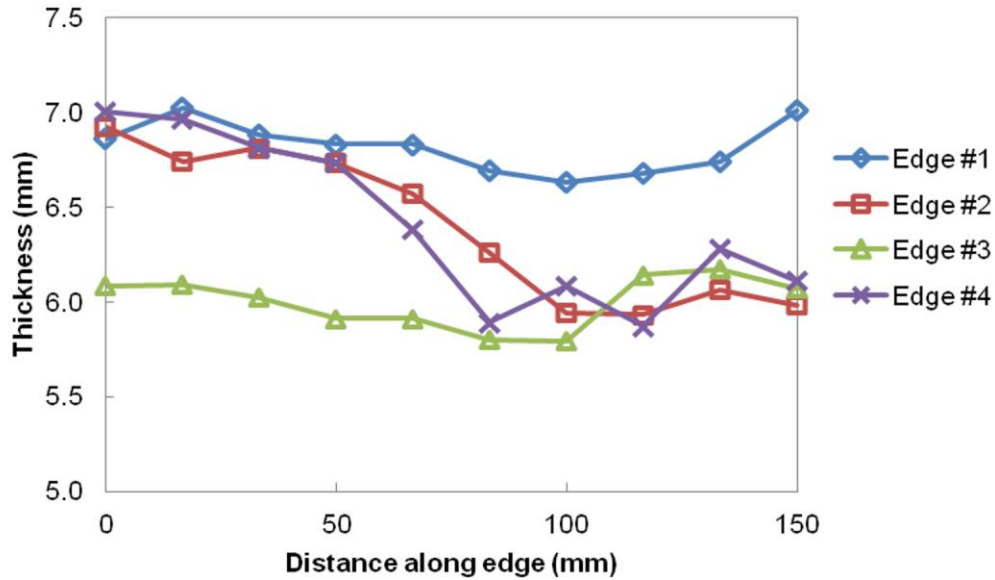


Figure 5.7: Thickness at the edges of the IHCT-CPMC

The thickness range of the coating was estimated from optical microscopy measurements of the PMC and co-cured layers. Measurements were made from the cut edges of the IHCT-CPMC. The thickness of the PMC and co-cured layers were measured at $4.1\text{mm} \pm 0.2\text{mm}$ and $0.32\text{mm} \pm 0.07\text{mm}$, respectively. It follows that the coating varied from 2.4mm at edge #1 to 1.6mm at edge #3. Hence, half of the coating had the same thickness as the copper sheet of the IHCT-NRC #1 and #2, while the other half was slightly thicker.

5.2.3 Curvature

Both IHCT-PMC and IHCT-NRC #1 remained flat after manufacturing. However, the IHCT-NRC #2 and IHCT-CPMC bowed during fabrication. The bow of these two IHCTs featured the copper layer on the concave side. For NRC-IHCT #2, bowing was caused by the co-curing operation at elevated temperature. Upon cooling the copper cladding contracted and bent the plate due to its much high coefficient of thermal expansion. Conversely, IHCT-CPMC bowed because of residual compressive stresses at

the surface of the coating, which were induced by the shot peening effect of the spraying process.

Curvature was estimated for each edge of the IHCTs. The curvature was calculated from the geometrical representation of the curved beam in Figure 5.8. Using Pythagorean theorem, the curvature R is equal to:

$$R = \sqrt{\left(\frac{L}{2}\right)^2 + (R - \Delta h)^2} \quad (4.3)$$

where L is half of the edge length and Δh is the deflection caused by the residual stresses. Isolating the curvature in the equation yields:

$$R = \frac{\Delta h}{2} + \frac{L^2}{8\Delta h} \quad (4.4)$$

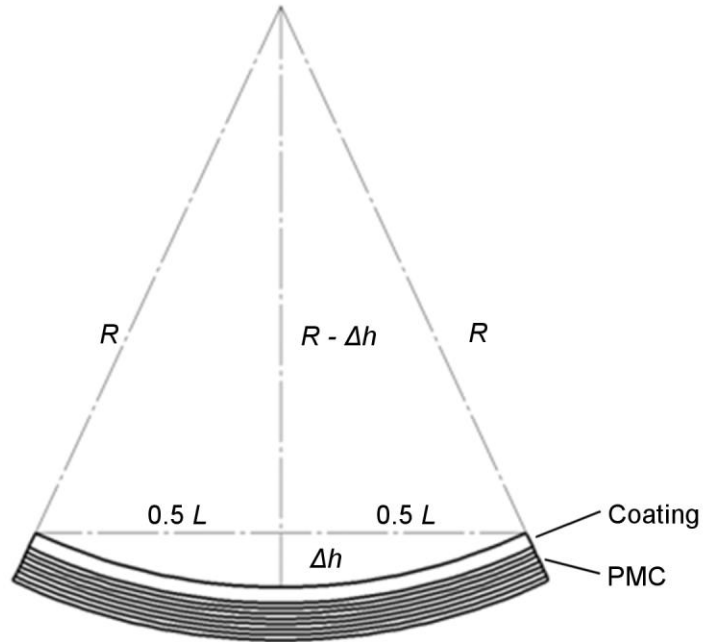


Figure 5.8: CPMC curved due to the coating process

Since the copper side of the IHCT-CPMC presented an uneven surface, deflection measurements were taken on the PMC side. The corners of the plates were elevated and their height was compared to that of the centre of the edges, providing a deflection value Δh (Figure 5.9).

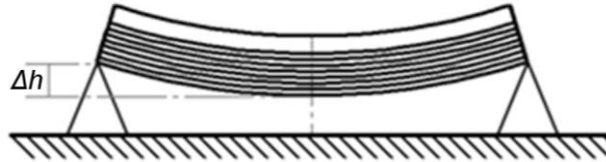


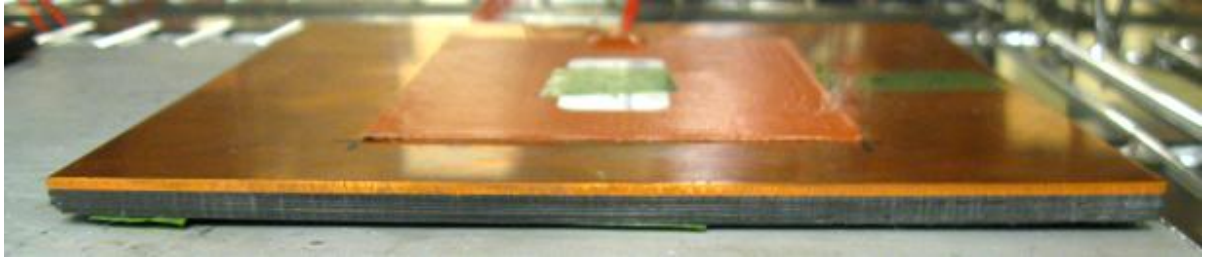
Figure 5.9: Deflection measurement of IHCTs

Results for curvature of the edges of the IHCTs are presented in Table 5.2. Similar curvature variability was found in both IHCTs. No correlations between curvature of the IHCTs and PMC laminate orientation or coating thickness were found. Nevertheless, IHCT-CPMC showed less deflection than IHCT-NRC #2, even though the sprayed copper coating was generally thicker than the copper sheet. Hence, with a higher curvature radius, the copper coating induced less deflection in the IHCT than the co-cured copper sheet.

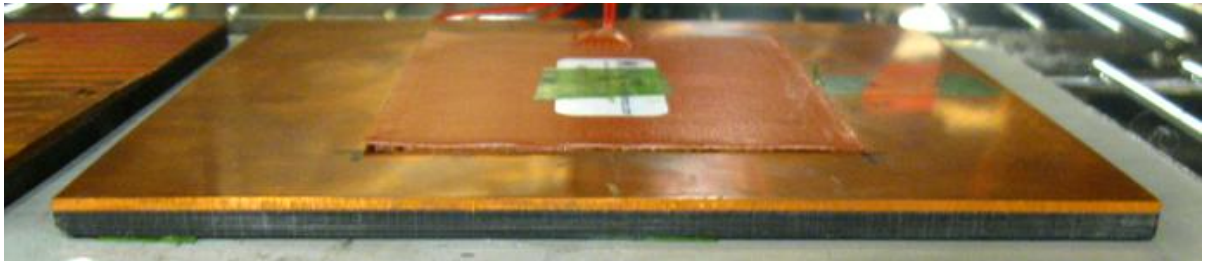
Table 5.2: Curvature of the IHCT-CPMC and IHCT-NRC #2

IHCT	Edge	Deflection (mm)	Curvature (m)
IHCT-CPMC	#1	0.45	6.4
	#2	0.40	7.4
	#3	0.57	5.2
	#4	0.48	6.0
IHCT-NRC #2	#1	0.94	3.1
	#2	0.70	4.2
	#3	0.86	3.4
	#4	0.81	3.6

In theory, heating these curved IHCTs should relieve stress, and reduce deflection. Characterisation of such recovery was made by heating the IHCTs to 120°C and looking at IHCT deflection. It should be noted that a basic visual qualitative assessment was performed due to the lack of proper measuring equipment for hot environments. For IHCT-NRC #2 (Figure 5.10), nearly all deflection was removed. The slight deflection remaining was likely attributable to edge trimming of the IHCT that altered residual stresses. The near absence of deflection was foreseeable as the IHCT was manufactured at that same temperature of 120°C. Heating back to the bonding temperature between the copper sheet and the PMC would eliminate the effect of thermal expansion. Thus, if an IHCT-NRC #2 is to be manufactured, co-curing must be done at same temperature as the intended usage temperature of the tooling. Heating IHCT-CPMC showed similar results. The temperature relieved most of the residual stresses (Figure 5.11). Slightly lower deflection was observed compared to IHCT-NRC #2 under the described spray conditions. Different spraying parameters may alter deflection and its variation with temperature. Nevertheless, for the above IHCTs, usage temperature should be 120°C.

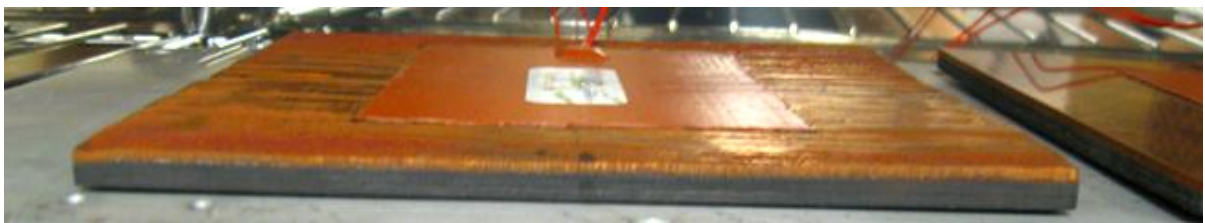


Room temperature



Temperature of 120°C

Figure 5.10: IHCT-NRC #2 deformation at room temperature and at 120°C



Room temperature



Temperature of 120°C

Figure 5.11: IHCT-CPMC deformation at room temperature and at 120°C

5.2.4 Coating inspection

Quality of the IHCT-CPMC coating was investigated through optical microscopy performed on cut edges. Inspection revealed that macroscopic defects were reduced using the automated displacement system, compared to the manual process used for fabricating SSB samples. However, the coating had a lower density than previous SST-C5003 coatings made with PGDS system #2. Consolidation was not homogeneous. As shown in Figure 5.12, lamellae of fully dense copper coating were intercalated with porous lamellae that were parallel to the surface of the coating. The number of strata appeared to correlate with the number of PGDS pulses. From the thickness of the lamellae, it was conjectured that each pulse produced a coating approximately 100 μ m thick featuring superimposed dense and porous zones.

The non-homogeneity in coating consolidation likely resulted from the powder injection system. The automated powder feeding used in PGDS system #3 coupled with the high feeding rates used in fabricating IHCT-CPMC potentially lead to a continuous flow of powder in the PGDS barrel. Since gas in the barrel is accelerated by a shockwave, the powder is accelerated either by a compressed or an expanded gas flow, section 2.2.1.4. It was conjectured that powder in the compressed gas flow is heated and becomes more malleable, resulting in dense coating lamellae. On the other hand, the powder in the expanded gas flow is cooled, giving the porous coating lamellae. Interestingly, a similar effect was not observed with PGDS systems #1 and #2. Gravity-based feeding used in those systems provided non-constant feeding based on the pressure of the barrel, ensuring that the powder was always fed in the same type of flow (compressed or expanded). Nevertheless, this occurrence of non-homogenous coating density confirms the need for further investigation of powder injection mechanism in PGDS.

Coating micrographs also showed that the dense lamellae were thicker than their porous counterparts. Since the valve opening time is shorter than its closed time, the dense lamellae were likely caused by the accumulations of powder in the barrel between pulses. Upon valve opening the accumulated powder was accelerated by the initial flow of compressed gas.

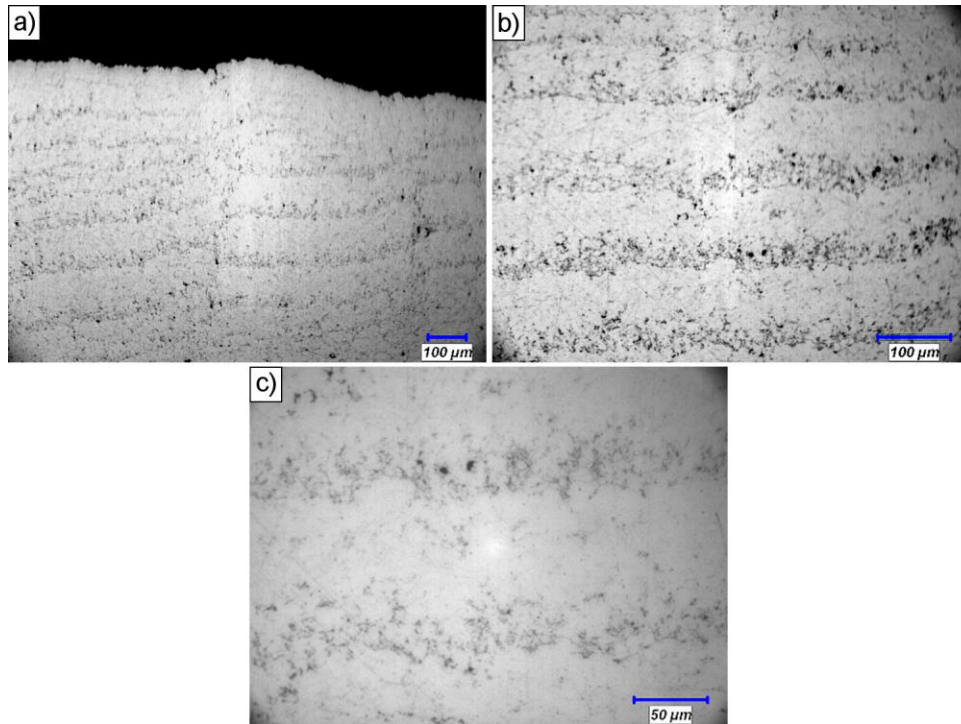


Figure 5.12: Micrographs of the IHCT-CPMC coating at magnifications of 100X (upper left), 200X (upper right) and 400X (bottom)

On a different note, the edges of the IHCT-CPMC were also analysed because they featured regions with coatings deposited on zones of bare PMC devoid of a co-cured layer. Microscopy confirmed that the coating was actually deposited on dust particles on the surface of the PMC (Figure 5.13). The apparent adhesion of such coating regions was attributable to cohesion with the adjacent coating regions attached to the co-cured layer. Even if this bares no structural integrity, it is nonetheless interesting to see that PGDS can form a coating above an unstable surface like a dust particle bed.

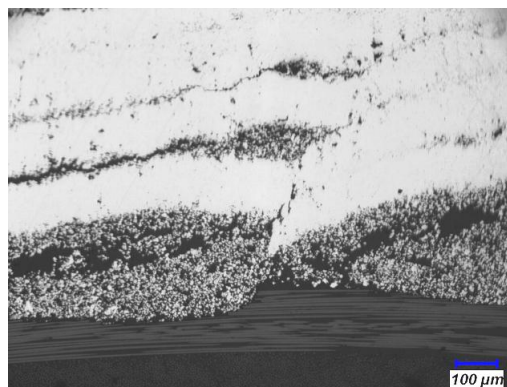


Figure 5.13: Copper coating above dust particles at the edge of the PMC

5.3 Temperature distribution

This section presents the thermal behaviour of the IHCT-CPMC. First, a comparison is made between the IHCT-CPMC and the other IHCTs related to temperature distribution when subjected to a constant heat flow. Second, an analysis about the thermal response of the IHCT-CPMC subjected to a temperature profile mimicking cure is presented.

5.3.1 IHCTs subjected to a constant heating power

The temperature distribution capability of the IHCT-CPMC was compared to the three other IHCTs in heating experiments performed under constant power. The fully insulated IHCTs were heated for 800s, using a single 15W surface heater. Temperature across the inner PMC tool face was measured using 8 thermocouples with locations as shown in Figure 5.14. Thermocouple TC1 was a control thermocouple (CTC) located 12mm from the heater, thermocouple TC2 was an over-heat thermocouple (OHTC) located directly on the heater to prevent heater overheating, and thermocouples TC3 to TC8 measured the temperature across the inner tool face. Three repeats were made per IHCT. Between each repeat the IHCTs were cooled back to ambient temperature. Thermocouple data recorded during testing are presented in Appendix H. After heating for 800s, insulation was removed and an infrared thermal image of the inner tool face was taken for each IHCT.

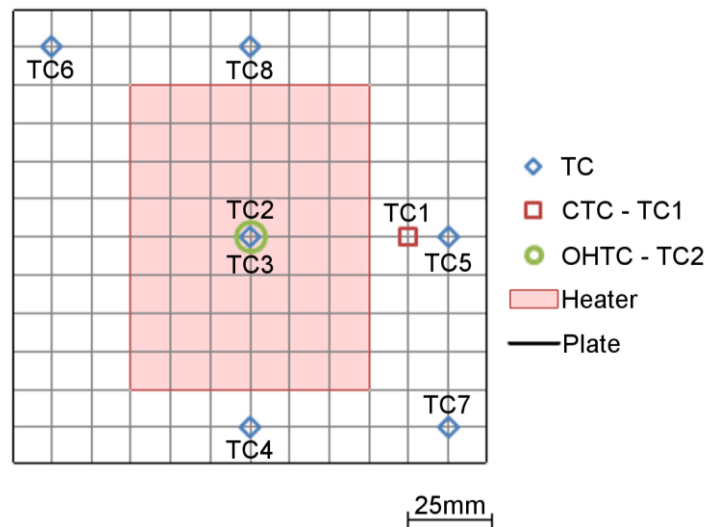


Figure 5.14: Thermocouple locations on the IHCTs

5.3.1.1 IHCT-CPMC

Temperature evolution for the warmest and coolest thermocouples located on the inner tool face of IHCT-CPMC is depicted in Figure 5.15. The warmest thermocouple was TC3 due to its proximity to the centre of the heater, while the coolest were TC6 and TC7 depending on the trial. Results showed that the inner tool face under the heater was heated quickly at the beginning of the experiment while heating of corners lagged due to thermal diffusivity of the materials. After a certain time, the temperature at both thermocouples increased linearly with time. This linear progression relates to uniform heat accumulation in the IHCT due to insulation that prevented most heat from escaping. The temperature increase rate, calculated at $2.3^{\circ}\text{C}/\text{min}$, was associated to the volumetric heat capacity (ρC_p) of the entire IHCT. The greater this value, the greater is the energy needed for heating. At the end of the trial, the centre of the inner tool face was at 60.4°C which was 8.6°C higher than at the corners. The temperature difference across the thickness was 3.1°C at the centre of the IHCT.

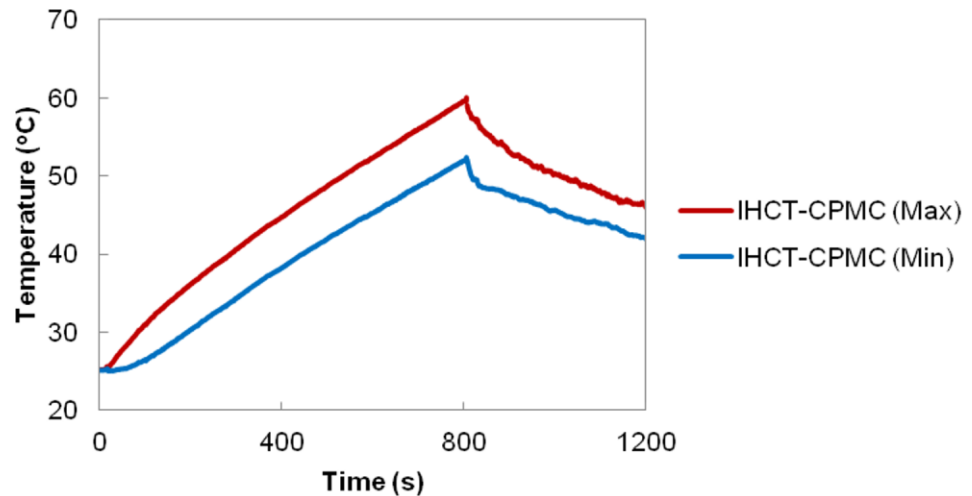


Figure 5.15: Temperature of the warmest and coolest thermocouples at the inner tool face of the IHCT-CPMC

Infrared thermal imaging (Figure 5.16) showed that surface temperature of the IHCT-CPMC was relatively uniform even though heat accumulated under the heater. It was seen that temperature was lower at the corner intersecting edges #3 and #4. This can be related

with the delaminated coating zone that was observed upon inspection. Hence, proper coating adhesion is imperative for heat transfer. Reassuringly, from the thermograph, no other low temperature zones were observed, suggesting consistent coating adhesion inside the IHCT. Since damage was limited to the edges, it is possible that delamination was caused by the cutting operation.

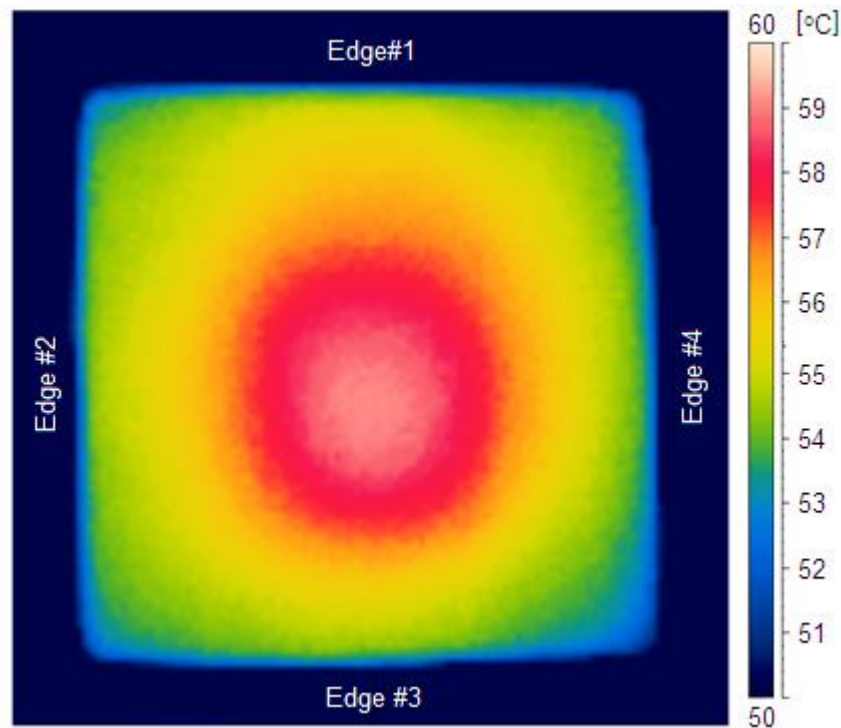


Figure 5.16: Infrared thermal image of the inner tool face of the IHCT-CPMC after 800s of heating

5.3.1.2 IHCT-PMC

Evolution of maximum and minimum temperatures measured on the inner tool face of the IHCT-PMC is presented in Figure 5.17. The temperatures were compared with measurements made on the IHCT-CPMC. Results showed that the temperature difference across the inner tool face of the IHCT-PMC was much higher and reached 72.0°C after 800s of heating. This experiment confirmed the need to improve thermal conductivity of bare PMCs for a tooling application, and it demonstrated the significant thermal conductivity improvement brought by copper coatings.

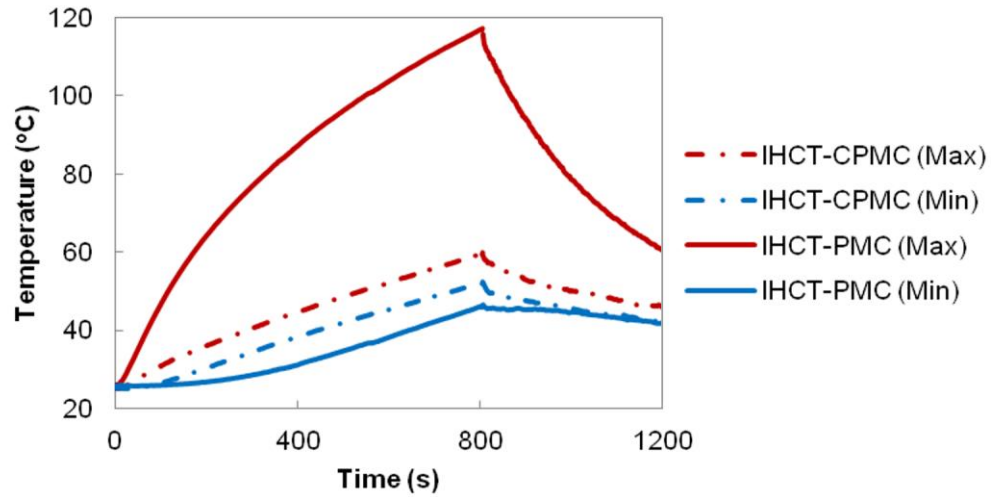


Figure 5.17: Temperature of the warmest and coolest thermocouples at the inner tool face of the IHCT-CPMC and IHCT-PMC

Infrared thermal imaging (Figure 5.18) confirmed that temperature varied significantly across the entire inner tool face of the IHCT-PMC. The temperature profile was uniform in all directions, suggesting a marginal amount of defects in the thermally isotropic IHCT.

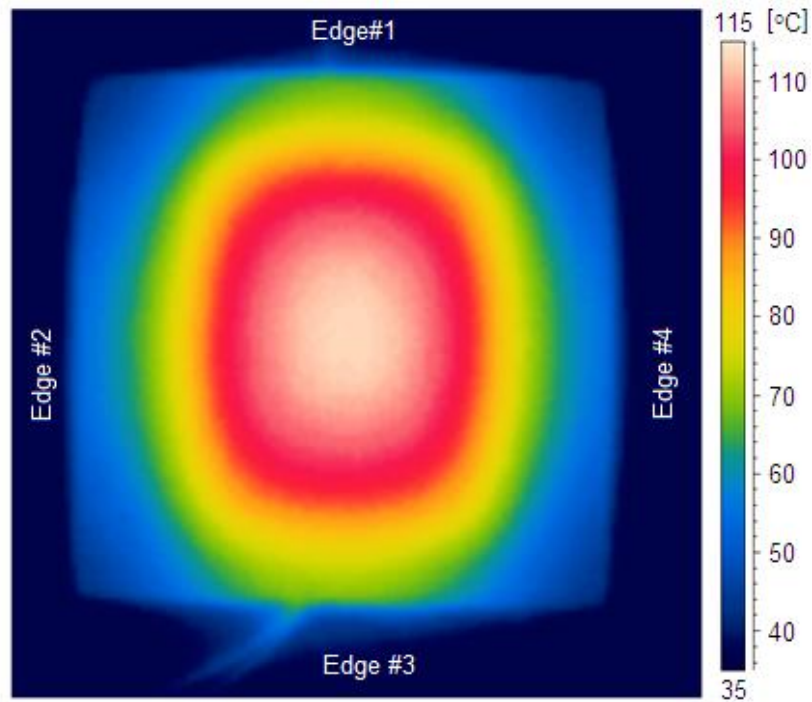


Figure 5.18: Infrared thermal image of the inner tool face of the IHCT-PMC after 800s of heating

5.3.1.3 IHCT-NRC #1

Temperature evolution across the IHCT-NRC #1 and IHCT-CPMC is compared in Figure 5.19. The difference in temperature across the inner tool face of the IHCT-NRC #1 was 5.5°C, 40% lower than for the IHCT-CPMC. This suggests that the copper coating was less thermally conductive than bulk copper, in all likelihood the result of thermal resistance at the particle-to-particle boundaries in sprayed coatings, as reported by Stoltenhoff et al. [63]. However, it is important to note that the silicone used for bonding the copper sheet to the PMC may have aided temperature distribution. Insulation provided by the silicone would have directed heat to flow in the copper sheet before crossing the silicone layer in the thickness. As a consequence, the temperature difference across the thickness was 4.7°C at the centre of the IHCT-NRC #1, which was higher than what was observed on the IHCT-CPMC.

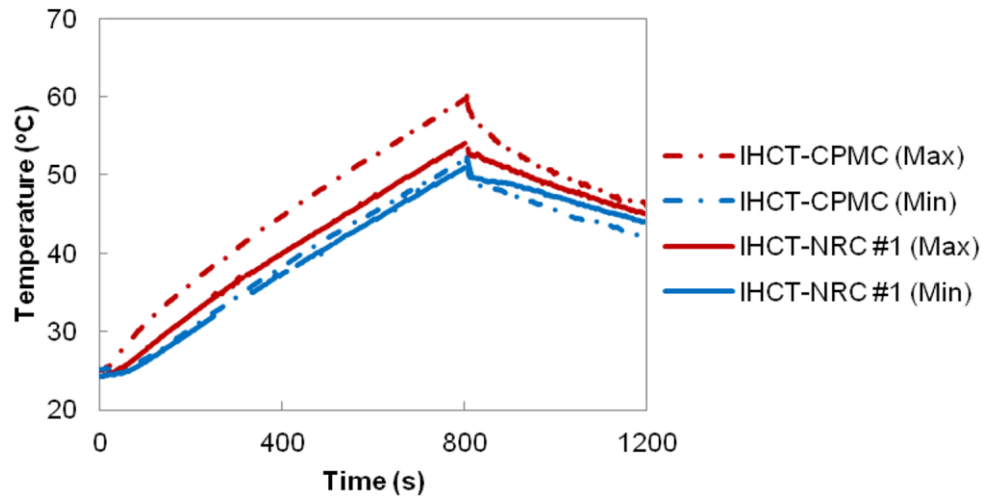


Figure 5.19: Temperature of the warmest and coolest thermocouples at the inner tool face of the IHCT-CPMC and IHCT-NRC #1

Infrared thermal imaging (Figure 5.20) showed that the temperatures across the IHCT-NRC #1 inner tool face were more uniform than for the IHCT-CPMC. However, the temperature distribution was less symmetric. The corner intersecting edges #1 and #4 was warmer than the three other corners. Removing the copper sheet revealed that silicone was lacking at the corner intersecting edges #3 and #4 which had the same effect as coating delamination for IHCT-CPMC. Furthermore, uncured silicone was found at the centre of

the IHCT which correlated to the warmest region and thus offered better heat transfer than cured silicone. Figure 5.21 shows the inside of the IHCT having cured silicone on its edges and where centre has been cleaned off of uncured silicone before taking the picture. It shows that this NRC-IHCT also present defects that affected heat flow negatively.

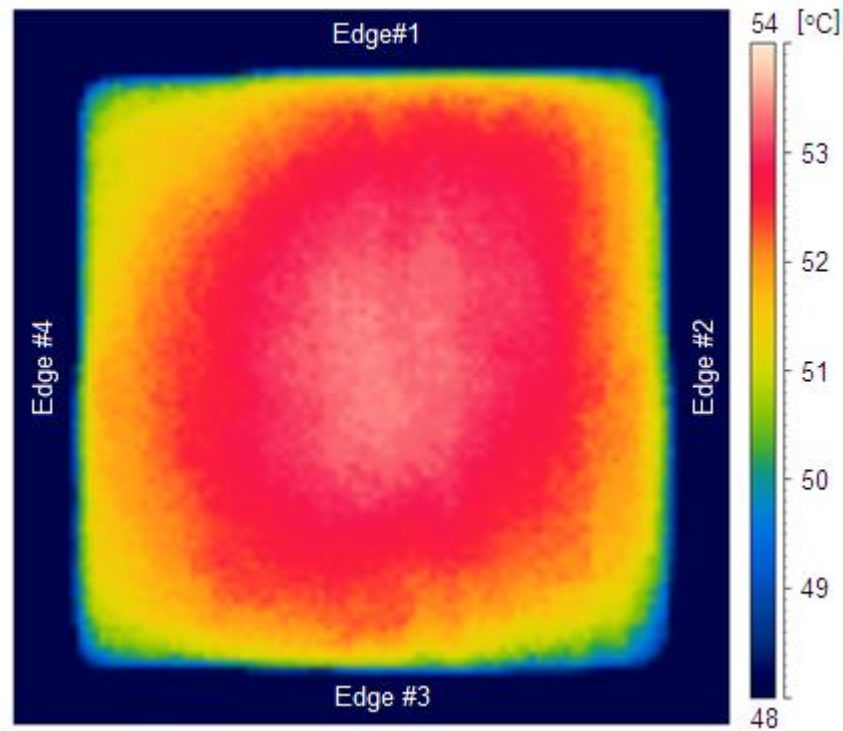


Figure 5.20: Infrared thermal image of the inner tool face of the IHCT-NRC #1 after 800s of heating



Figure 5.21: PMC plate with cured silicone (left) and copper sheet with cured silicone (right) of the IHCT-NRC #1 after disassembly where the uncured silicone was cleaned before taking pictures

5.3.1.4 IHCT-NRC #2

Temperature evolution across IHCT-NRC #2 and IHCT-CPMC are compared in Figure 5.22. The temperature difference of 5.0°C across the inner tool face of IHCT-NRC #2 was comparable to that observed in IHCT-NRC #1. Considering that IHCT-NRC #2 was only composed of a PMC laminate with a co-cured copper sheet on its outer surface, this confirmed definitively that bulk copper was more conductive than the copper coating.

Infrared thermal imaging (Figure 5.23) showed that temperature across the inner tool face of IHCT-NRC #2 was more uniform than for IHCT-CPMC and IHCT-NRC #1. Co-curing mitigated bonding defects between the PMC and copper, resulting in a symmetric temperature profile across the inner tool face of the IHCT.

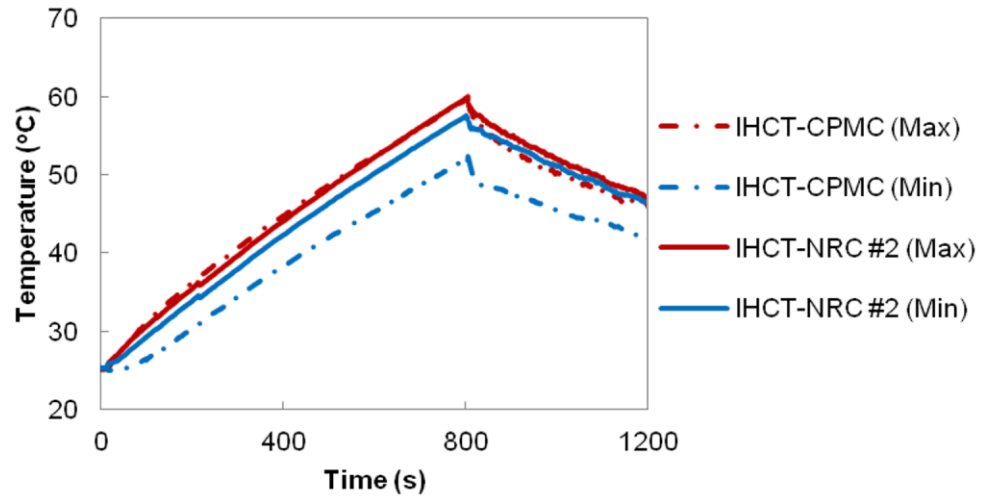


Figure 5.22: Temperature of the warmest and coolest thermocouples at the inner tool face of the IHCT-CPMC and IHCT-NRC #2

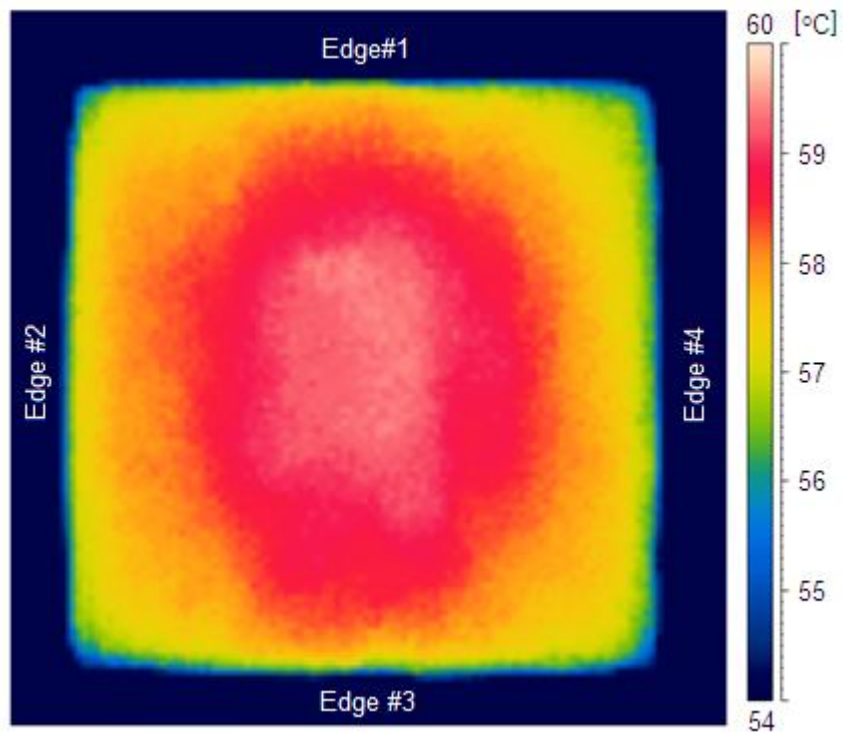


Figure 5.23: Infrared thermal image of the inner tool face of the IHCT-NRC #2 after 800s of heating

5.3.1.5 Comparison of IHCTs

A statistical analysis of temperatures collected with the infrared thermal images was made. Results are presented in Table 5.3. The temperature differences obtained across the inner tool face of the IHCTs were slightly greater than those measured from the thermocouples. This was expected since thermographs enabled measurements closer to the IHCT edges. Infrared results confirmed that the coating was significantly more conductive than bare PMC. However, the IHCT-NRC #1 and #2 offered the most uniform temperatures, with a standard deviation of temperature across the inner tool face of 0.9°C. IHCT-NRC #1 offered slightly lower temperature variation across the inner tool face than IHCT-NRC #2, which was likely related to its insulating silicone layer that promoted heat flow within the copper sheet ahead of heat flow crossing to the PMC.

The results also showed that the average inner tool face temperatures in the IHCT-PMC was the greatest after 800s heating under 15W, proving that it required the least energy to heat. This is logical considering that no additional material is present for distributing heat. However, the lower energy consumption cannot compensate for the severe lack of temperature uniformity. The other IHCTs, IHCT-CPMC, IHCT-NRC #1 and IHCT-NRC #2 showed similar average inner tool face temperatures and heating rates, but IHCT-NRC #2 was the most energy efficient. It provided the quickest heating rate due to the absence of an intermediate layer between the copper sheet and PMC laminate. The least energy efficient was IHCT-NRC #1 because of its thick silicone layer. The energy efficiency of the IHCT-CPMC was between that of the two IHCT-NRCs. This is interesting considering that its copper coating was thicker than their copper sheet and that it also featured a co-cured layer that accumulated thermal energy. From this, it can be deduced that the co-cured layer has a marginal impact on the energy storage in the IHCT-CPMC.

Table 5.3: Temperature distribution across the inner tool face after 800s of heating

IHCT	Temperature (°C)				
	Average	Standard deviation	Maximum	Minimum	Difference
CMPC	55.7	1.6	59.2	50.0	9.2
PMC	77.4	20.1	113.7	40.9	72.8
NRC #1	52.5	0.9	54.1	47.9	6.2
NRC #2	58.0	0.9	59.5	51.9	7.6

5.3.2 Simulating manufacturing conditions

In a final set of experiments for this chapter, the IHCT-CPMC was subjected to a temperature profile simulating tooling conditions required for manufacturing SE-70 prepreg PMCs. The IHCT was fully insulated on both faces and inner tool face temperatures were measured from thermocouples located as per the diagram of Figure 5.14. The IHCT-CPMC was heated at a rate of 2°C/min up to a prescribed dwell temperature, which was held for 30min; afterwards the inner tool face insulation was removed to let the IHCT cool to room temperature. Two dwell temperatures were tested: 70°C and 120°C, which are the lower and upper manufacturing limits for SE-70 PMCs respectively. Thermocouple TC1 controlled the on/off controller of the heater.

5.3.2.1 Lower dwell temperature

Two different single run experiments were performed for the lower dwell temperature. Testing was performed with a heater supplying 15W and 20W of power. Thermocouple readings generated during both experiments are presented in Figures 5.24-5.25. In both experiments it was seen that supplied power was sufficient to heat the IHCT-CPMC to the desired 70°C. The controller had to consistently turn on or off the heater, creating temperature spikes on the IHCT. The magnitude of these temperature spikes varied significantly for different thermocouples; it was attenuated by the distance from the

heater as heat first accumulated under the heater before flowing outwards in the IHCT. The delay resulting from the heat flow was estimated; in both experiments, 20s lapsed between temperature spikes at the heater and at the centre of the inner tool face of the IHCT. An additional 65s lapsed before a temperature spike was seen at the inner tool face extremities (TC6 and TC7). Similarities between the experiments were attributed to the comparable dwell conditions that yielded similar temperature gradients across the IHCT.

Comparing the two experiments showed that the number of heater activation cycles, and therefore temperature spikes, was lower under 15W due to slower heating. This was mostly visible upon heating where 15W barely sufficed to achieve 2°C/min. However, the lower power output resulted in inferior temperature rises in the heater due to greater heat dissipation. As such, during dwell the maximum temperature measured at the heater was 74.7°C in the 15W experiment compared to 76.2°C in the 20W experiment, while the magnitudes of the respective spikes were 7.8°C and 6.5°C. Surprisingly, this difference in temperature spike magnitude was not seen on the inner tool face of the IHCT. The magnitude of these spikes at the centre of the inner tool face was 3.8°C in both experiments while they were 1.8°C and 1.6°C at the inner tool face edges in the 15W and 20W experiments respectively. Hence, at this stage, no correlation could be made between the heater power and the temperature spikes at the inner tool face.

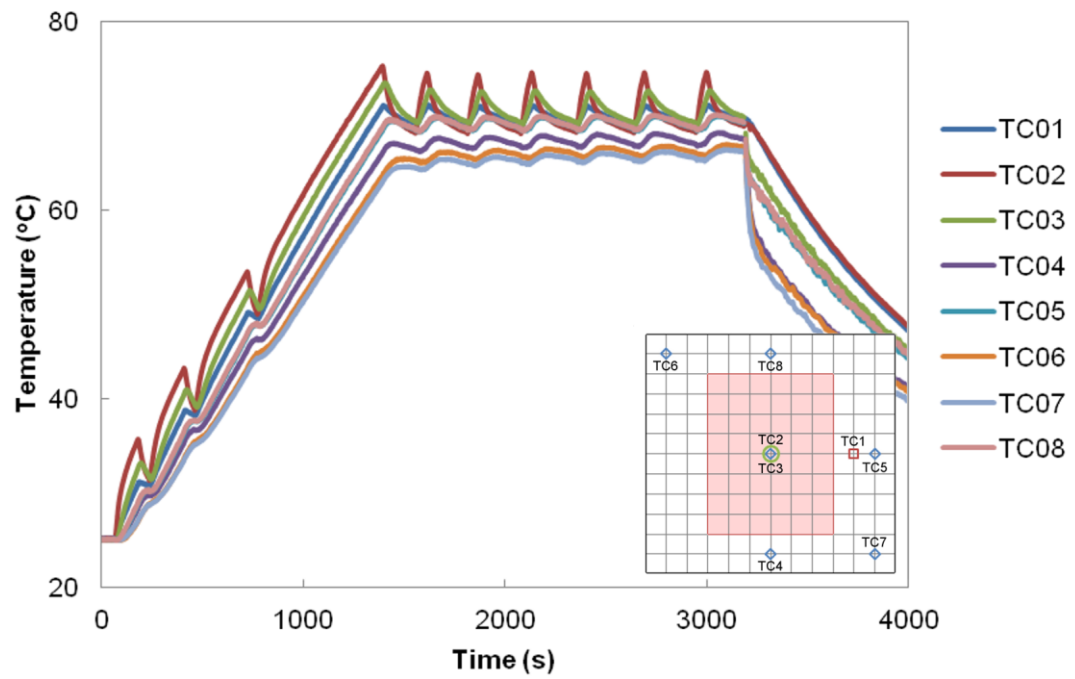


Figure 5.24: Temperatures across the IHCT-CPMC in the 70°C dwell temperature, 15W experiment

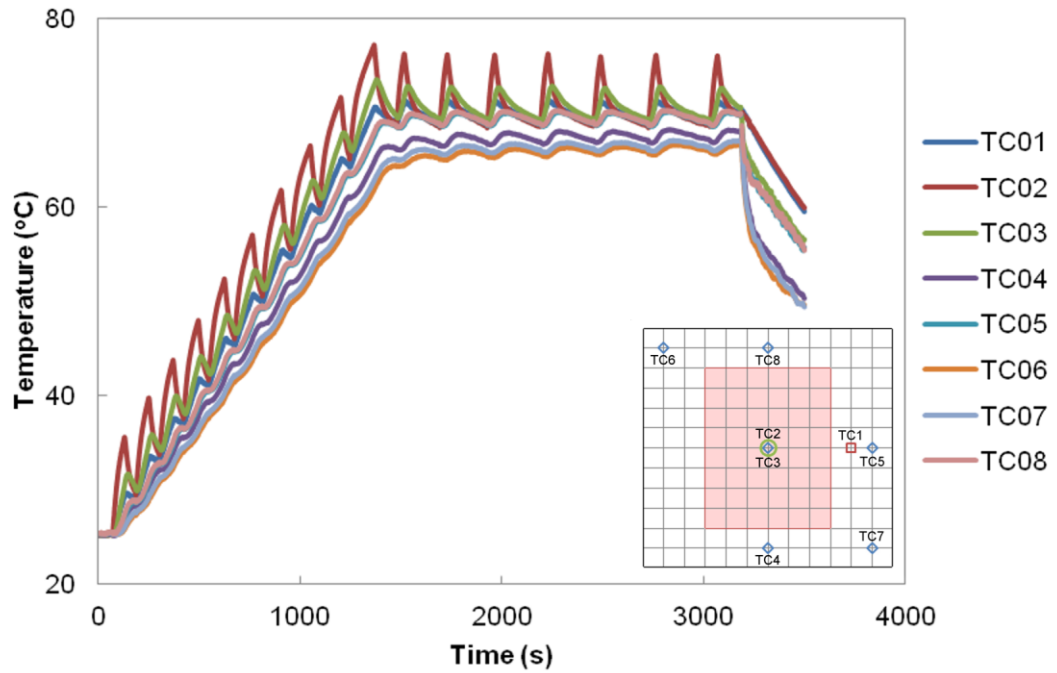


Figure 5.25: Temperatures across the IHCT-CPMC in the 70°C dwell temperature, 20W experiment

Maximum temperature differences (ΔT) across the inner tool face of the IHCT were calculated at any instant for both experiments (Figure 5.26). These ΔT values were obtained from the temperature difference between the warm centre (TC3) and cool extremity (TC6 and TC7). Upon heating, it was seen that ΔT increased with time and that it was larger in the 20W experiment. The lower heat flux of the 15W experiment was better suited to the power requirement of the heating ramp, which lead to fewer temperature fluctuations. During dwell both experiments showed similar ΔT . This was likely caused by the marginal difference in heating power between experiments, in light of very low power required for maintaining the dwell temperature in an insulated system. The required power for the dwell was estimated at 2.8W from the duty cycle, which is the percent of time that the heater was activated. The duty cycles were 18.3% and 14.5% for the 15W and 20W experiments respectively. During dwell, the magnitude of ΔT varied between 7.8°C and 3.5°C, and the average ΔT reduced with dwell time, improving temperature uniformity.

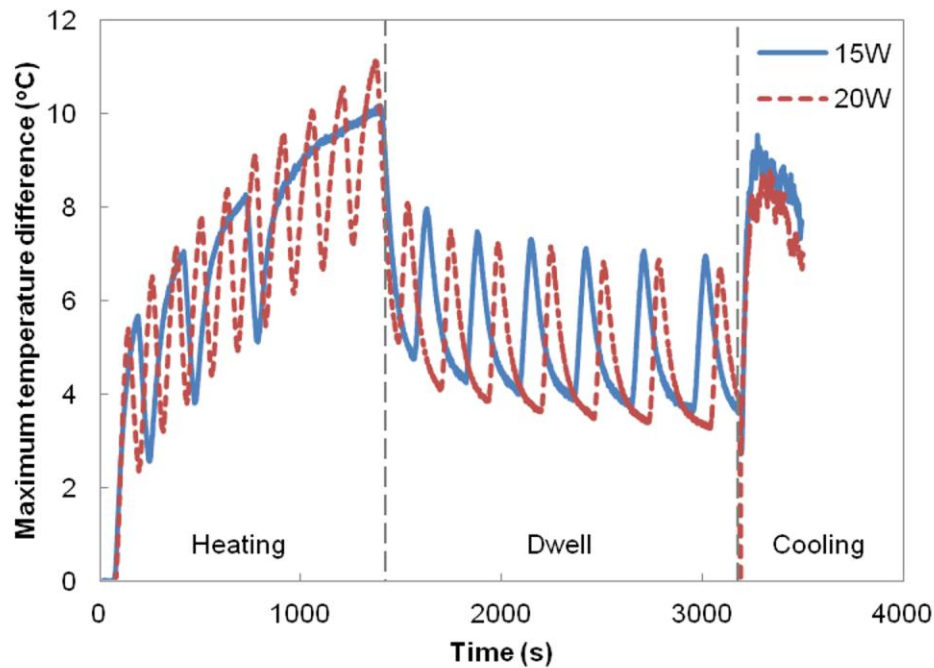


Figure 5.26: Difference between the maximum and minimum temperatures at the inner tool face of the IHCT-CPMC during experiments heated with 15W and 20W

5.3.2.2 Higher dwell temperature

A single run experiment was performed at a dwell temperature of 120°C. Testing was performed with a heater supplied with 20W. Thermocouple readings are presented in Figure 5.27. Heater power was sufficient to enable a heating ramp of 2°C/min up to 120°C, but at higher temperatures the heater was nearly always activated. The heater duty cycle, i.e. percent of time that the heater was activated, was evaluated at 32.1% during dwell, suggesting heat losses of 6.4W. The maximum and minimum ΔT throughout the IHCT-CPMC at dwell were measured at 11.1°C and 6.2°C respectively. This is approximately 3°C higher than ΔT values for the experiments at 70°C. Hence, ΔT appears to be related to the heat losses. Conversely, the magnitude of the temperature spikes was 3.5°C in the centre of the inner tool face whereas it was 2.1°C at the extremities, similar to the 70°C experiments. Thus it appears that the temperature spikes are mostly governed by the heater control system and by the physical properties of the IHCT-CPMC.

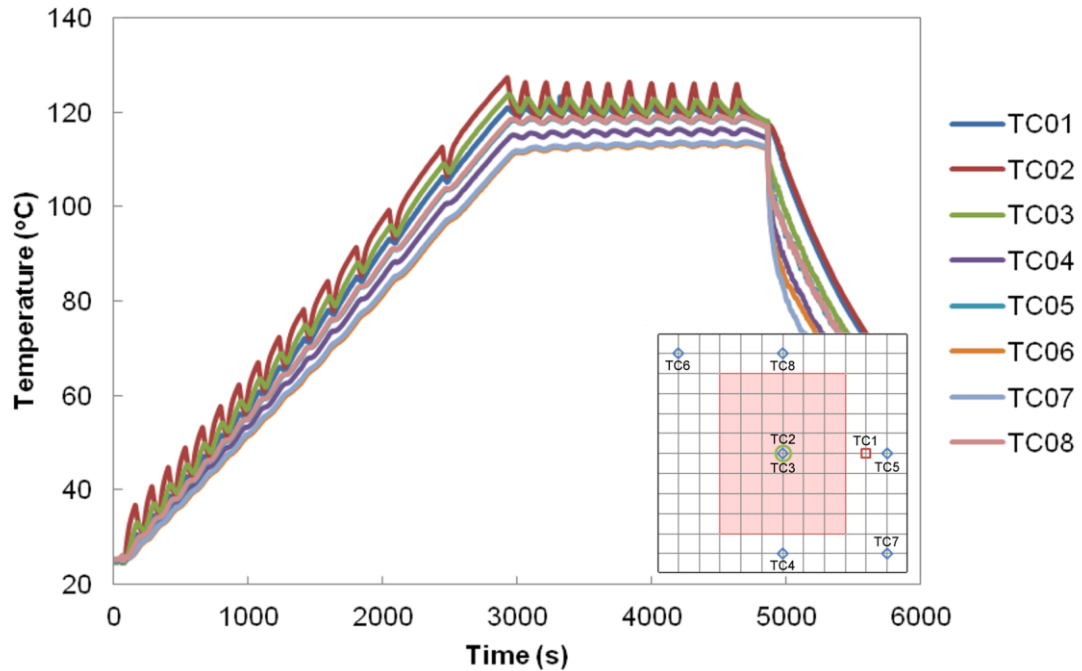


Figure 5.27: Temperatures across the IHCT-CPMC in 120°C dwell temperature, 20W experiment

5.4 Conclusions

The contribution of this chapter lies in the successful fabrication of an IHCT prototype made with CPMC that significantly improved in-plane thermal conductivity compared to bare PMC.

Despite highly successful trials, it may be pointed out that the IHCT-CPMC prototype featured a SST-C5003 coating that had defects, including coating zones of higher porosity and partial coating delamination at the edges of the IHCT. These defects were caused by imperfect spraying conditions and by the nature of the copper powder used. The spraying process also induced residual stresses in the coating, bending the CPMC. These residual coating stresses were alleviated when the tooling plate was heated to its application temperature of 120°C, but no correlations between the coating properties and stress relieving temperatures were made. In further work, these coating defects and residual stresses may potentially be mitigated. Nonetheless, the coating provided significant in-plane thermal conductivity improvement to the CPMC over bare PMC, although not exactly as much as bulk copper due to imperfect particle-to-particle interfaces in the coating.

The prototype was compared to three other IHCTs of different types. Comparison of the IHCTs (Table 5.4) showed that both IHCT-NRCs provided slightly better temperature uniformity during heating than the IHCT-CPMC. However, the main advantage for the IHCT-CPMC resides in its potential for easily applying a conductive layer on curved surfaces and changing the thickness of the conductive layer locally. It is also important to remember that the goal of the coating is only to offer a thermally conductive layer, which it does, and not to reach the same conductivity value as that of bulk copper.

Heating experiments also showed that temperature distribution across IHCTs is greatly affected by the heating system and its control mechanism, which will be the focus of the next chapter.

Table 5.4: Comparative summary of the tested IHCTs

Characteristic	Tool			
	IHCT-PMC	IHCT-CPMC	IHCT-NRC #1	IHCT-NRC #2
Thermally conductive layer	None	Copper coating	Copper sheet bonded with silicone	Co-cured copper sheet
Temperature uniformity	Very low	Good	Highest	Highest
Residual stresses	Low	Highest, but mitigated through heating	Low	Highest, but mitigated through heating
Conductive layer formability	N/A	Potentially similar formability for flat and curved geometries	Costly forming for highly curved geometries	Costly forming for highly curved geometries

Chapter 6

Heat transfer simulations of IHCTs

6.1 Introduction

Optimisation of IHCTs was pursued using heat transfer simulations. Simulation work was divided into several programs. The first two programs, HT-01 and HT-02, contributed to the validation of the heat transfer simulations based on experimental IHCT results.

Afterwards, programs HT-03 to HT-06 contributed to the identification of parameters that affect temperature uniformity across IHCTs, leading to the development of guidelines for designing IHCTs with more uniform cure temperature.

In program HT-03, a parametric study using simulations was performed for identifying the factors affecting surface temperatures across IHCTs. From the results of HT-03, programs HT-04 and HT-05 focused on the thickness of conductive layer. Finally, the effect of localised heat generation was investigated in program HT-06 by comparing the temperature uniformity across a single heater IHCT and multi-heater IHCT.

6.2 Program HT-01: uniform thickness IHCT

Program HT-01 sought to validate transient simulation results with those of a physical experiment. This was achieved for investigating the viability of simulations for predicting the behaviour of IHCTs. A flat IHCT-NRC featuring a uniform thickness copper sheet bonded on a PMC using high temperature silicone was used for validation. Temperatures across its surfaces were measured using thermocouples and an infrared camera, similarly to what is described in section 5.3.

6.2.1 Physical description

The flat IHCT used for validation of simulations in HT-01 was provided by NRC-IAR and first utilised by Maksoud [24]; it appears in Figure 6.1. It was composed of a 6.0mm thick, 300mm by 300mm AS4/3501-6 PMC laminate structure manufactured in autoclave. A 1.05mm thick copper sheet was bonded to its outer surface using high temperature silicone. Heat was generated by a MINCO HR6600R576L24A flexible heater supplied with 30W of power. During testing the IHCT was entirely insulated with thermal blankets for mitigating convective heat losses. Testing consisted of a 34min heating phase followed by a cooling phase, in which insulation was always present.

Temperatures across the IHCT were measured using 12 thermocouples. Similarly to experimentation reported in section 5.3, thermocouple TC1 was used as a heater control thermocouple positioned on the outer copper face close to the heater. Thermocouple TC2 was placed directly on the heater. The remaining thermocouples (TC3-TC12) were placed across the inner tool face of the IHCT as depicted in Figure 6.2. It should be noted that the data acquisition channel for TC4 stopped working during testing, preventing readings at that location.



Figure 6.1: IHCT inner tool face (left) and outer face (right), HT-01

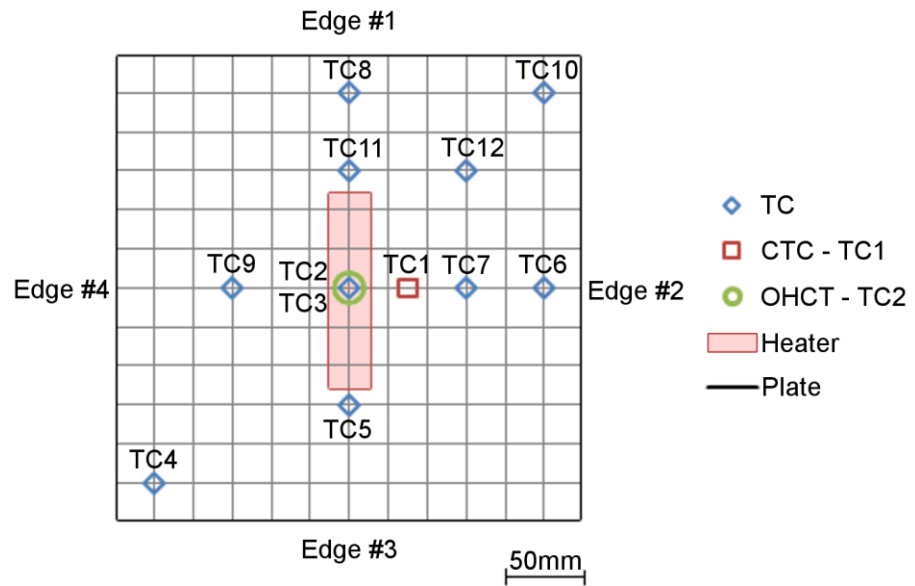


Figure 6.2: Thermocouple locations on the inner tool face of the IHCT, HT-01

6.2.2 Model description

Two models were used for transient simulations in HT-01. The models are respectively named HT-01-1 and HT-01-2 and differ only in terms of material properties. The models followed the actual geometry of the physical IHCT as closely as possible. The greatest difference between the simulations and experiment was related to the locations of the thermocouples, which were at most 5mm away from actual locations on the IHCT.

Both a coarse mesh having 1748 hexahedron elements and a refined mesh containing 13984 hexahedron elements were compared; refinement was achieved by doubling the number of elements along each axis. The characteristics of the meshes are presented in Table 6.1. A depiction of the coarse mesh appears in Figure 6.3.

Table 6.1: HT-01 model characteristics

Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 6mm
- Copper sheet	300mm x 300mm x 1.05mm
- Heater	25mm x 125mm x 1.5mm
Element type	Hexahedron
Element length	12.5mm (6.25mm)
Element thickness	
- PMC	3mm (1.5mm)
- Copper	1.05mm (0.525mm)
- Heater	1.5mm (0.75mm)
Number of elements	1748 (13984)
Thermal resistance at interfaces	
- Heater/copper	$0.0028 \text{ m}^2 \cdot \text{K/W}$
- Copper/PMC	$0.0110 \text{ m}^2 \cdot \text{K/W}$
Heater power	30W
Global convection coefficient	$1 \text{ W/m}^2 \cdot \text{K}$
Transient time steps	2s

Note: Values for refined mesh appear in brackets

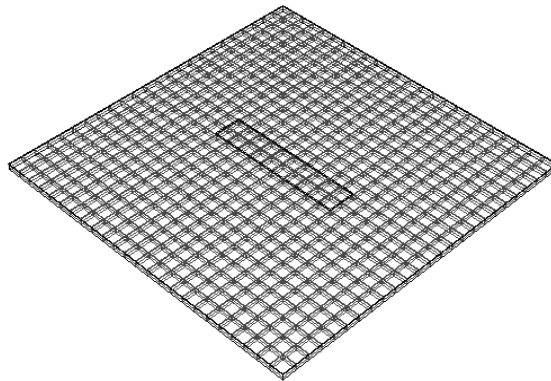


Figure 6.3: Coarse mesh, HT-01

Material properties for both models appear in Table 6.2, and were taken from Chapter 3. HT-01-1 uses material properties found in literature whereas HT-01-2 modified the PMC properties for matching the experimental results. Properties for the flexible heater made of silicone rubber were assumed to be similar to those of the high temperature silicone adhesive due to the lack of data. Furthermore, thermal resistance values between the various material layers were unknown and had to be approximated. The values were obtained by matching the temperature drops between the heater and the copper plate (TC2 and TC1) and between the copper and the PMC surface. The latter was calculated from the temperature difference between the heater (TC2) and centre of the PMC (TC3), and was separated from the heater-to-copper resistance. Thermal resistance was taken as $0.0028\text{m}^2\cdot\text{K}/\text{W}$ between the heater and the copper sheet, and $0.0110\text{m}^2\cdot\text{K}/\text{W}$ between the copper sheet and the PMC.

Table 6.2: Material properties used for simulations, HT-01

Material	Density (g/cm^3)	Specific heat capacity ($\text{J}/\text{kg}\cdot\text{K}$)	Thermal conductivity ($\text{W}/\text{m}\cdot\text{K}$)
Copper	8940	386 at 20°C 400 at 150°C	401 at 20°C 392 at 150°C
PMC (AS4/3501-6) -HT-01-1	1590	808 at 20°C 1138 at 120°C	3.78 (in-plane) 0.74 (transverse)
PMC (AS4/3501-6) -HT-01-2	1590	808 at 20°C 1500 at 120°C	3.78 (in-plane) 0.74 (transverse)
Silicone and heater	1470	1465	0.18

Note: Values were taken from Chapter 3

The models also had to take into account heat losses through insulation. Heat losses were modelled as convective losses to ambient air at 25°C because this is the simplest method available in FLUENT for relating the heat flux to the temperature gradient between the IHCT and environment. A single convective coefficient was used for the entire outer surfaces of the IHCT, including the edges. This may not be perfectly representative as some areas may exhibit stronger losses. The convective coefficient was estimated using the cooling phase temperature readings to eliminate the effect of heat generation. A convective

coefficient of $1\text{W/m}^2\cdot\text{K}$ was obtained by curve fitting simulations with the experimental temperatures.

6.2.3 Results

For the experiments, repeatability of thermocouple measurements was assessed by performing the same experiment twice, on two different days. Results presented in Appendix I show good temperature agreement between the two repeats. Additionally, comparison of simulation results obtained with the coarse and refined meshes appears in Appendix J. Temperature analysis shows minimal differences in results obtained from the meshes. At most, a 1°C difference was observed in the heater. Following such results, simulations of all HT programs were conducted using coarse meshes to reduce computational time.

In HT-01-1, thermocouple measurements are compared with simulation values in Figures 6.4-6.5. It should be noted that thermocouple measurements presented were from the first repeat. Experimental and simulated results showed comparable trends in temperature profile, confirming that temperature decreases with increasing distance from the heater. Temperature profiles of simulations and experiments during the heating phase matched nearly perfectly for thermocouples TC2, TC8 and TC11, but differed for other thermocouples. After 2000s, the difference in temperature between the simulation and the thermocouples was on average 4.0°C .

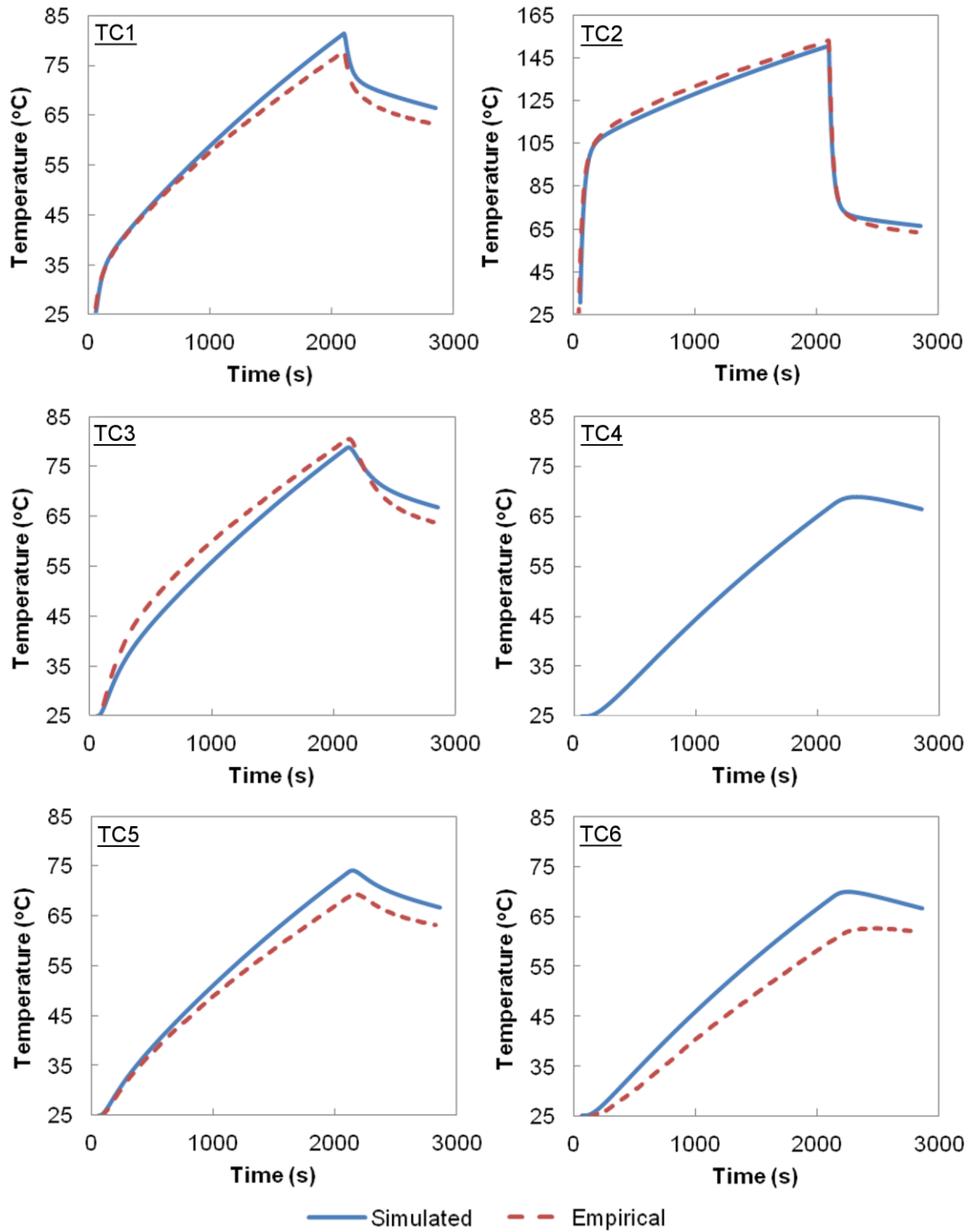


Figure 6.4: Comparison between simulated and experimental temperatures at thermocouples TC1-TC6, HT-01-1

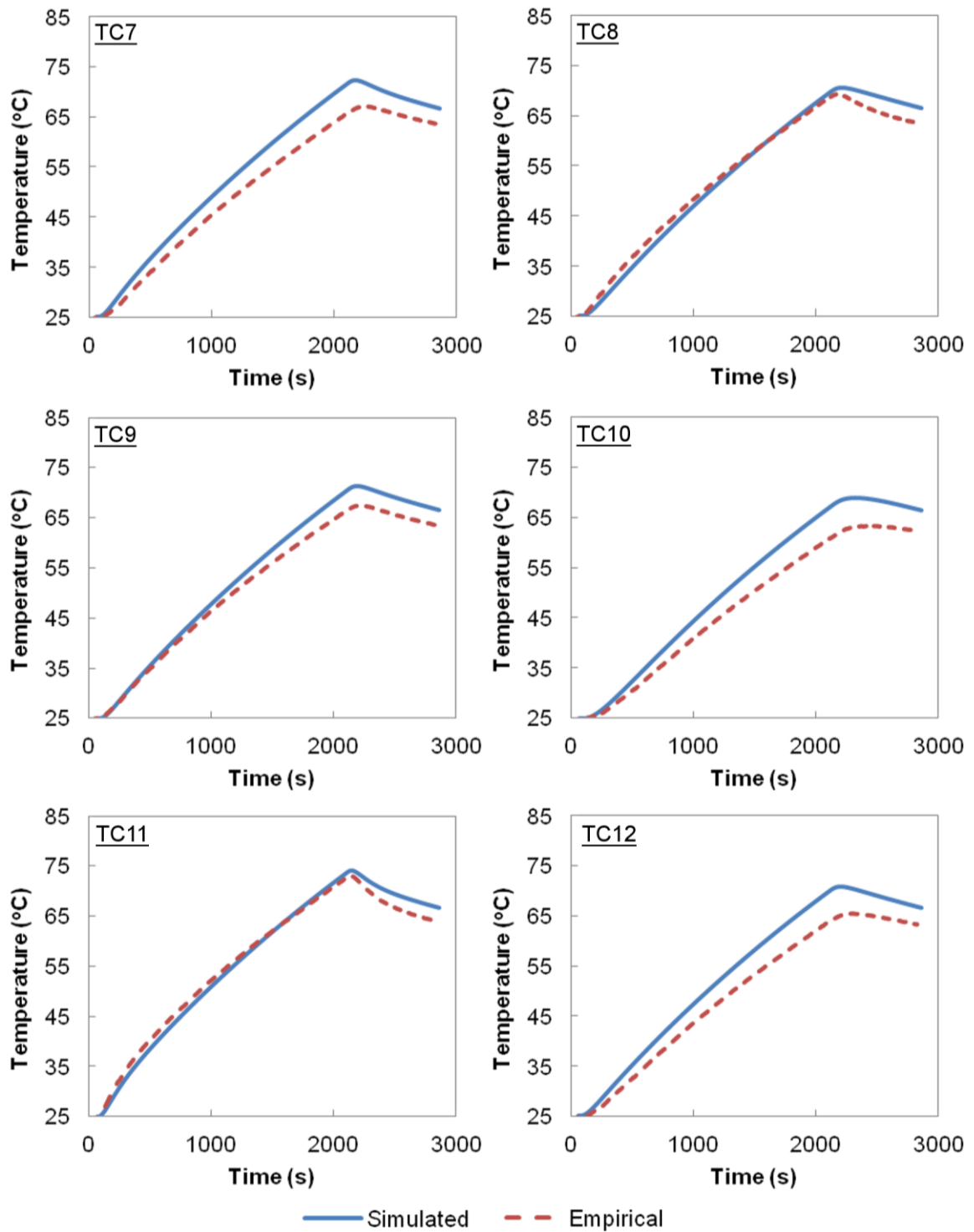


Figure 6.5: Comparison between simulated and experimental temperatures at thermocouples TC7-TC12, HT-01-1

Data from thermocouple TC3, which was located under the heater, revealed that the thermocouple saw higher temperatures than predicted by the simulations during the heating phase. This suggested that there was likely less through-thickness thermal resistance than what was used for the simulations. However, the chosen contact resistance was necessary for matching as closely as possible the temperature values of the other thermocouples. Furthermore, as shown by experimental results for the cooling phase, thermocouple TC3 saw more heat dissipation than predicted. This was caused by either greater localised heat losses or greater heat distribution in the IHCT. The prior is unlikely as insulation blankets cover both sides of the IHCT. The edges of the IHCT should therefore be subjected to greater heat losses than the centre of the IHCT where TC03 lies. Hence, heat was likely redistributed more effectively inside the plate, suggesting a possibly greater in-plane thermal conductivity for the PMC than what was used in simulations.

Analysis of the cooling phase showed that discrepancies in heat dissipation between the simulated and experimental results are affected by location. Based on Figure 6.2, thermocouples TC5 and TC9 near edges # 3 and #4 showed heat dissipation similar to simulation predictions. Thermocouples TC6, TC7, TC10 and TC12 near edge #2 and near the corner of edges #1 and #2, dissipated less energy than predicted. Thermocouples TC8 and TC11 near edge #1 dissipated more energy than predicted. A similar trend was observed looking at temperature differences between experimental and simulated results at the end of the heating phase. After 2000s, temperature differences were greatest, at 6.5°C, for thermocouples near edge #2 and near the corner of edges #1 and #2; conversely they were the lowest at 0.9°C for the thermocouples near edge #1. Temperature differences for the thermocouples near edges #3 and #4 were in-between, at 4.2°C. Therefore, part of the discrepancies is due to localised effects. This was confirmed by infrared imaging of the IHCT, where it was seen that the temperature profile was not symmetric throughout the inner IHCT surface (Figure 6.6). Inspection of the IHCT edges revealed debonding between the copper sheet and PMC. Slight debonding was observed at the corner of edges #1 and #2 and corner of edges #3 and #4, while greater debonding was observed in the corner of edges #2 and #3. Hence, as reported in Chapter 5, it is important to limit defects for maximising temperature uniformity.

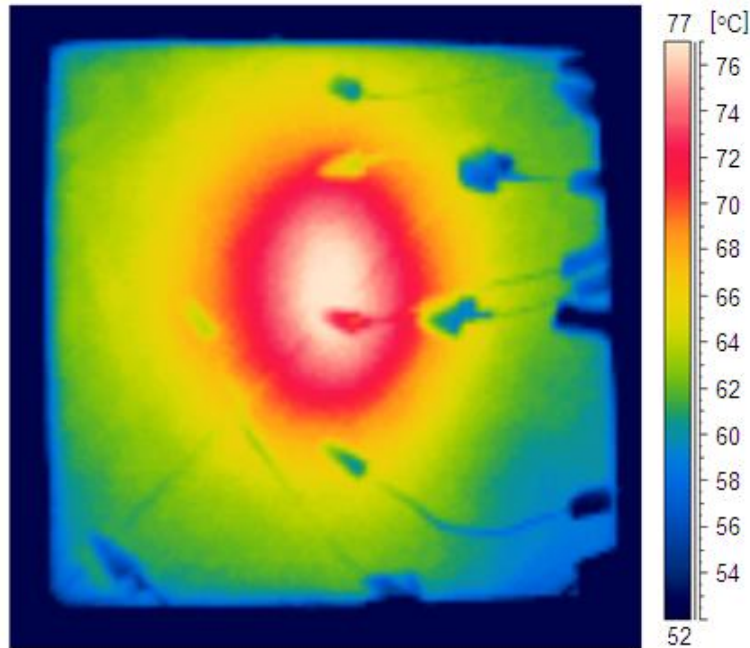


Figure 6.6: Infrared thermal image of IHCT, HT-01

Temporal analysis of temperatures revealed another parameter that significantly affected discrepancies between experimental and simulation. Looking at the heating rate, it was observed that for nearly all thermocouples, temperature differences increased with both time and temperature compared to simulation values. This suggested that at higher temperatures the IHCT could store more energy per degree Celsius than what was simulated. Furthermore, DSC results in section 3.4.5 showed that the specific heat capacity of epoxy can vary significantly from sample to sample.

In HT-01-2, a different estimate for the specific heat capacity of the PMC was tried. Instead of using a linear progression from 808J/kg·K at 20°C to 1138J/kg·K at 120°C, the specific heat capacity was approximated by a linear progression from 808 J/kg·K at 20°C to 1500J/kg·K at 120°C. A comparison of thermocouple temperatures with the modified specific heat capacity values is presented in Figures 6.7-6.8. Results showed that in most cases, the HT-01-2 simulation predicted temperatures closer to thermocouple measurements than in HT-01-1. As such, after 2000s the simulated temperatures were on average 2.7°C off thermocouple values. From the lower discrepancies between experimental and simulated values and considering that IHCT manufacturing defects were

neglected in simulations, it can be concluded that heat transfer simulations can predict or at least identify trends for heat transfer in simple IHCTs.

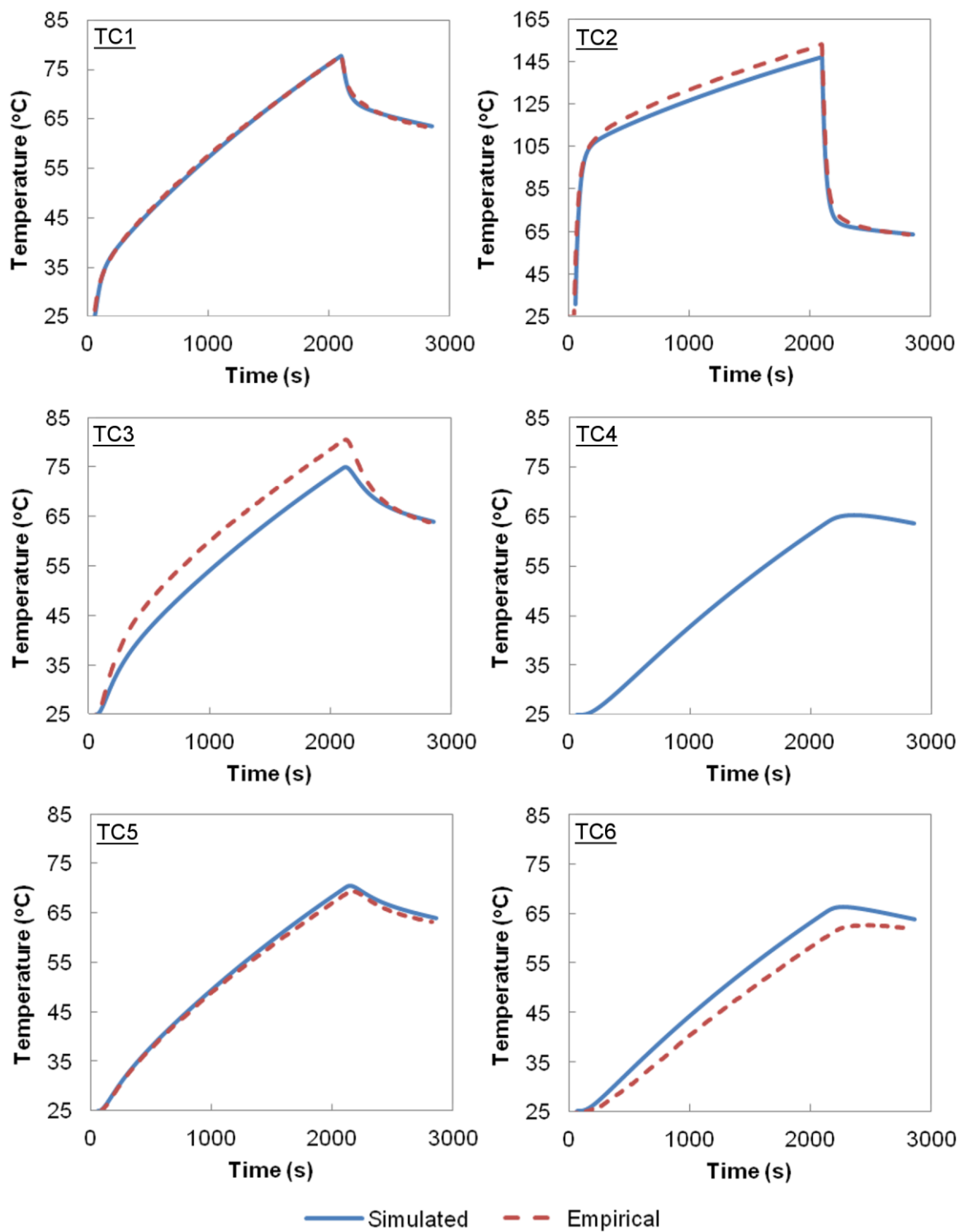


Figure 6.7: Comparison between simulated and experimental temperatures at thermocouples TC1-TC6, HT-01-2

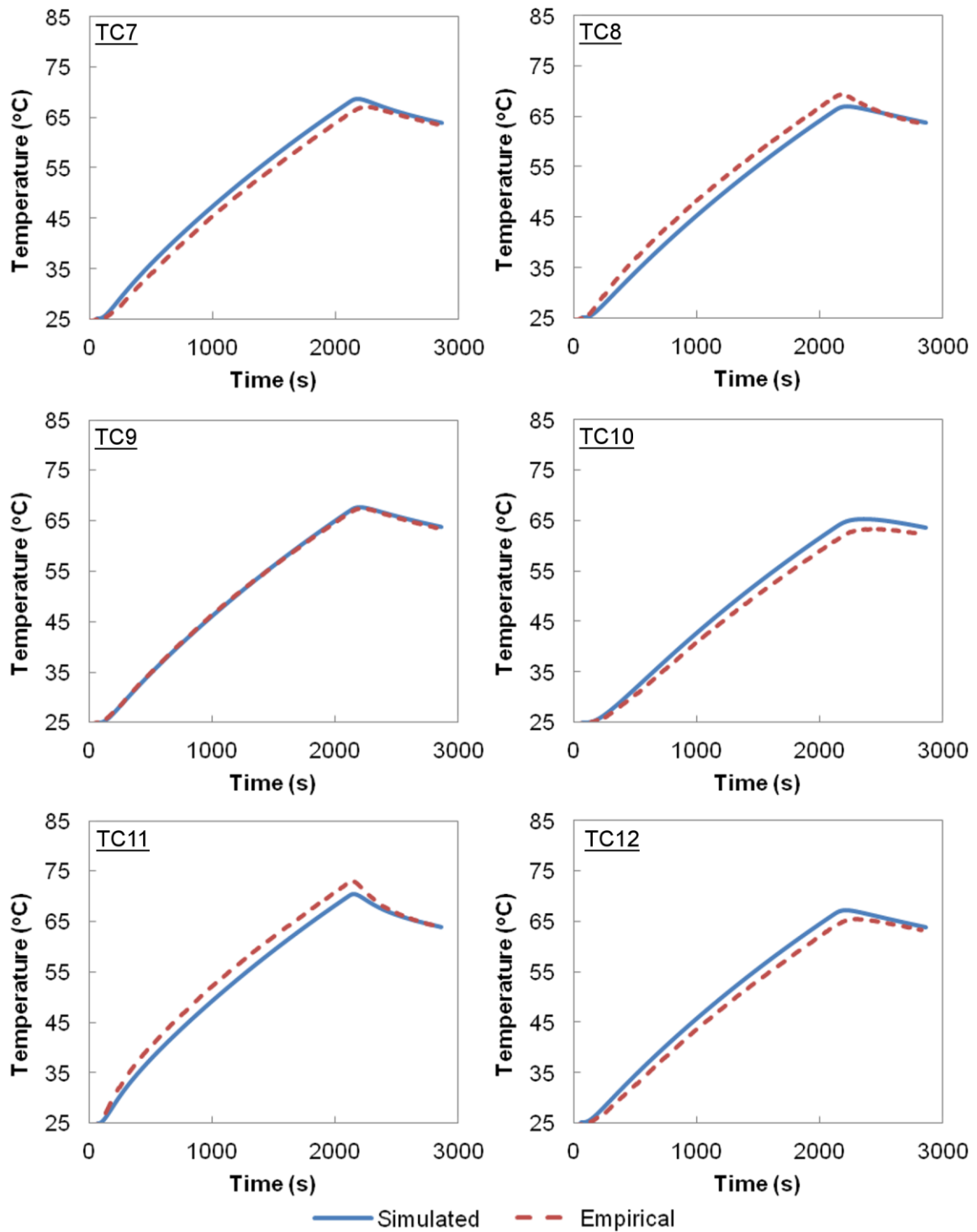


Figure 6.8: Comparison between simulated and experimental temperatures at thermocouples TC7-TC12, HT-01-2

6.3 Program HT-02: non-uniform thickness IHCT

Program HT-02 aimed at validating transient simulations results of higher complexity geometries compared to program HT-01. Experimentation and simulation was carried out using a flat IHCT-NRC featuring a non-uniform thickness copper layer that was co-cured with the PMC. Similarly to what was done in program HT-01, experimental temperatures across the IHCT surfaces were measured using thermocouples and an infrared camera.

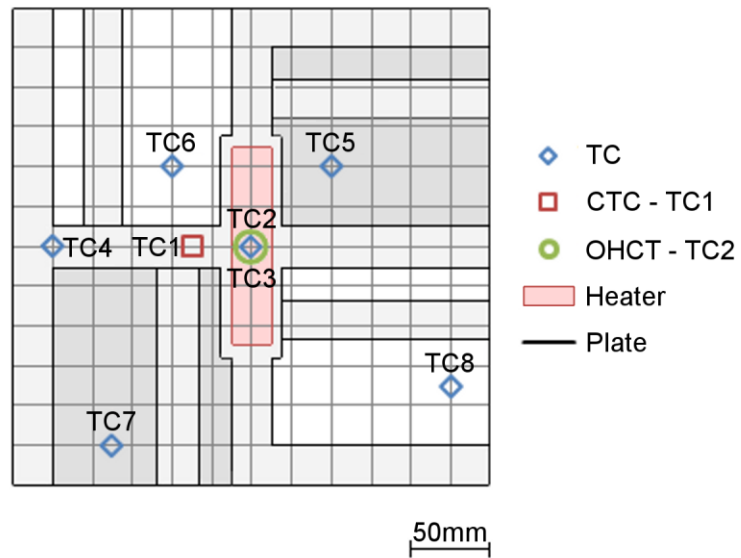
6.3.1 Physical description

The IHCT (Figure 6.9) used for validating complex geometry simulations in program HT-02 featured a 4.0mm thick 300mm by 300mm SE-70 PMC structure. A copper layer (called pattern) machined to non-uniform thickness was co-cured on its surface using a layer of SA-70 film. The copper pattern featured sections with various thicknesses and tapered fins. The geometry of the copper pattern is described in the following section. Heat was generated by a MINCO HR6600R576L24A flexible heater supplied with 22.5W of power. During testing, the IHCT was entirely insulated using an insulation blanket for mitigating convective heat losses. Testing consisted of a 50min heating phase followed by a cooling phase.

Temperatures across the IHCT were measured using 8 thermocouples. Similarly to HT-01, thermocouple TC1 was used as a heater control thermocouple positioned close to the heater on the outer copper face, while thermocouple TC2 was placed directly on the heater. The remaining thermocouples (TC3-TC8) were placed distributed across the inner tool face of the IHCT as depicted in Figure 6.10. An infrared image of the inner IHCT surface temperature was also taken during heating.



Figure 6.9: IHCT inner tool face (left) and outer face (right), HT-02



□: Thinnest copper pattern regions

□: Thicker copper pattern regions

□: Regions not covered by copper pattern

Figure 6.10: Thermocouple locations on the inner tool face of the IHCT, HT-02

6.3.2 Model description

Three models were used for simulations in HT-02. They were named HT-02-1, HT-02-2 and HT-02-3, and differed only in terms of material properties. Their mesh represented as closely as possible the actual geometry of the physical IHCT. The model is represented in Figure 6.11. The modelled and physical copper patterns featured sections of different thicknesses with the thinnest regions being 0.75mm and thickest regions being 2.50mm. The copper pattern also featured two types of stepped fins. The smaller fins went in thickness from 1.00mm to 2.50mm while the bigger fins went in thickness from 1.5mm to 2.50mm. Due to mesh size limitations, the simulated stepped fins featured fewer steps than the physical ones. The smaller fins featured three steps of 0.50mm, while the bigger fins featured four steps of 0.25mm.

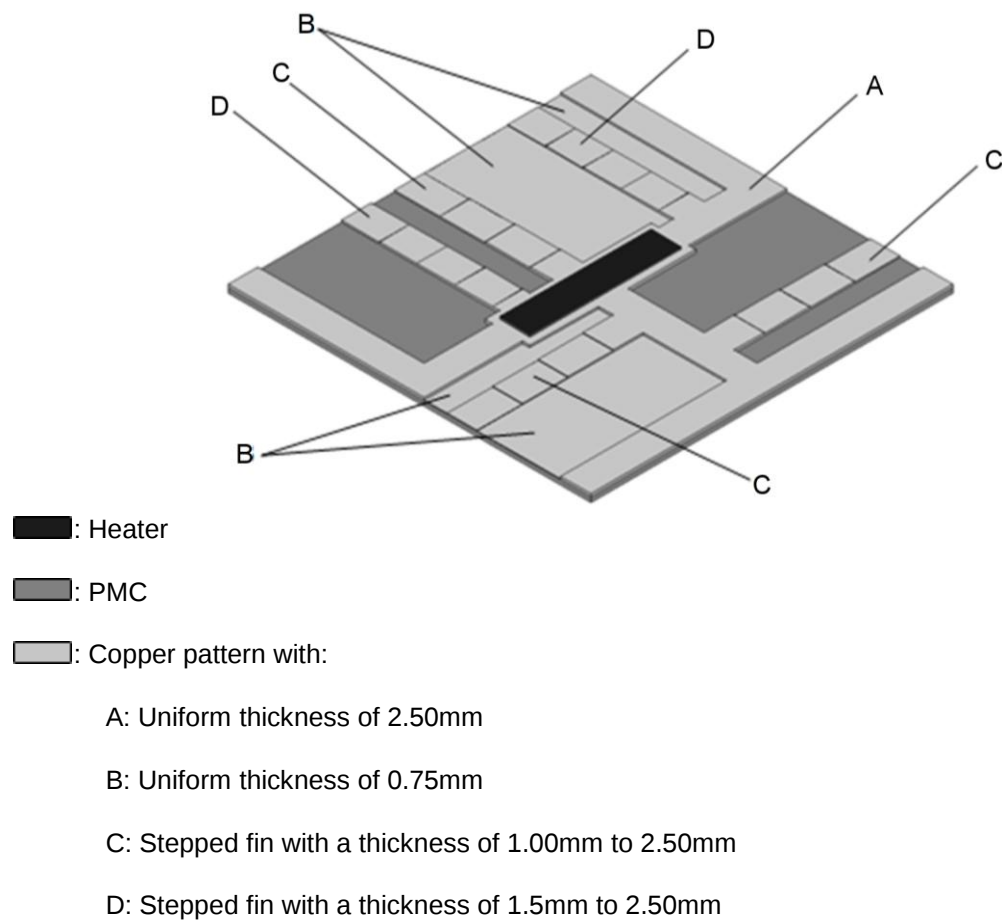


Figure 6.11: Representation of the computational model, HT-02

Characteristics of the model are presented in Table 6.1. The mesh featured cell lengths comparable to those of the HT-01 refined mesh. Such fine mesh was required for capturing all the fine details of the copper pattern. The thermal resistance between the heater and copper was kept identical to that in HT-01, but no thermal resistance was modelled between the copper and PMC due to the intimate bonding resulting from co-curing. A uniform convection coefficient of $1.5\text{W/m}^2\cdot\text{K}$ was applied to all external boundaries with a free stream temperature of 25°C . The convection coefficient was obtained by curve fitting the cooling phase of the simulation with thermocouple temperatures as in HT-01.

Table 6.3: HT-02 model characteristics

Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 4mm
- Copper sheet	300mm x 300mm x 0 to 2.5mm
- Heater	25mm x 125mm x 2mm
Element type	Hexahedron
Element length	6.25mm
Element thickness	
- PMC	2mm
- Copper	0.25mm
- Heater	2mm
Number of elements	16258
Thermal resistance at interfaces	
- Heater/copper	
-HT-02-01 and HT-02-02	$0.0028\text{m}^2\cdot\text{K/W}$
-HT-02-03	No thermal resistance
- Copper/PMC	No thermal resistance
Heater power	
-HT-02-01 and HT-02-02	22.5W
-HT-02-03	20W
Global convection coefficient	$1.5\text{W/m}^2\cdot\text{K}$
Transient time steps	2s

Material properties used in simulations appear in Table 6.4. The copper and heater properties were kept identical to the ones in HT-01 while the values of the PMC were selected based on the analysis of SE-70 in sections 3.4.3 and 3.4.5. The specific heat capacities of the of HT-02-01 and HT-02-02 were based on the predictions developed from DSC cycle #1 and DSC cycle #4 for SE-70, respectively. HT-02-01 used cycle #1 which was obtained after the first heating cycle following PMC cure, and had the highest specific heat capacity value. HT-02-02 followed cycle #4 which was obtained from the last DSC cycle. Finally, HT-02-03 used the same material properties as HT-02-01 but featured lower heater power (20W) and no contact resistance between the heater and copper.

Table 6.4: Material properties used for simulations, HT-02

Material	Density (g/cm ³)	Specific heat capacity (J/kg·K)	Thermal conductivity (W/m·K)
Copper	8940	386 at 20°C 400 at 150°C	401 at 20°C 392 at 150 °C
PMC HT-02-1 (SE-70 from DSC cycle #1)	1530	930 at 20°C 1830 at 120°C	2.32 in-plane 0.51 transverse
PMC HT-02-2 (SE-70 from DSC cycle #4)	1530	551 at 20°C 1401 at 120°C	2.32 in-plane 0.51 transverse
PMC HT-02-3 (SE-70 from DSC cycle #1)	1530	930 at 20°C 1830 at 120°C	2.32 in-plane 0.51 transverse
Silicone and heater	1470	1465	0.18

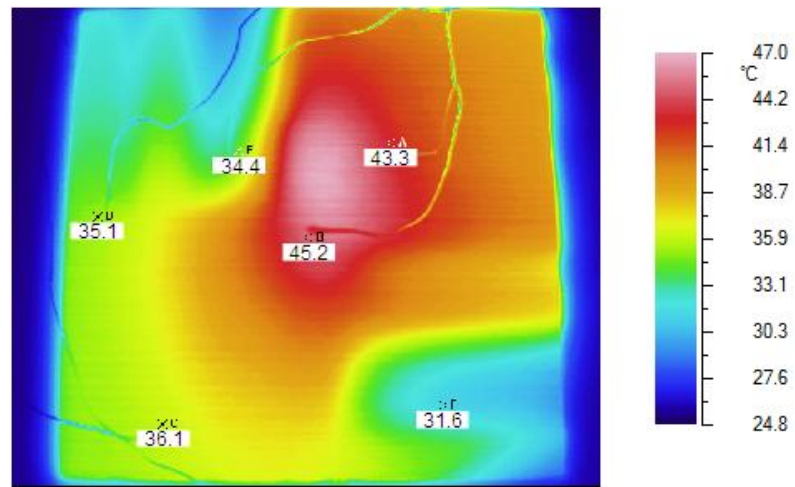
6.3.3 Results

An infrared thermal image of the inner tool face of the IHCT was taken during the heating phase of the experiment (Figure 6.12). It was seen that the temperature profile was non-symmetric, as expected. Temperatures were higher under the heater, but they were also greater under thicker regions of the copper pattern. Hence, these results confirmed the possibility of orienting heat using a pattern having different thicknesses.

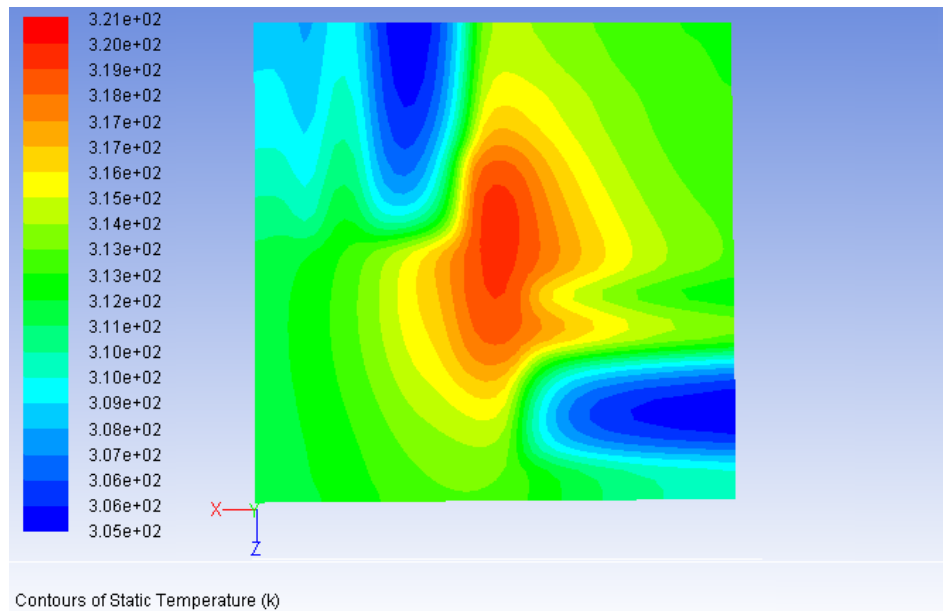
Simulations showed the same behaviour as the experiment. A comparison between the experimental thermal infrared image and the simulation inner tool face temperatures values appear presented in Figure 6.12. The simulation picture that is presented was taken at approximately 1000s for HT-02-01 when the inner tool face temperatures under the heater matched those of the infrared thermal image. Qualitative comparison of both the experimental and simulation images showed similar temperature profiles. The greatest difference was observed on the edges, where the experimental IHCT was subjected to greater cooling. Nonetheless, the temperature range predicted at the inner tool face from the simulation was 305K to 320K (32°C to 47°C), which was very close to the 29°C to 47°C range obtained from the infrared image.

Quantitative comparison of thermocouple temperatures with simulated values for both HT-02-01 and HT-02-02 appear in Figures 6.13-6.14. Results showed that both simulations predicted higher temperatures than those measured and that the temperatures were relatively far from actual measurements. Greater discrepancy was observed with HT-02-02 than with HT-02-01. The maximum and minimum temperature differences with the inner tool face thermocouples after 50min were 11.4°C and 7.0°C for HT-02-02, and 5.4°C and 0°C for HT-02-01.

It follows from the results that the actual specific heat capacity of the PMC was closer to the HT-02-01 values based DSC cycle #1 in section 3.4.5. This was somewhat expected because measurements on the IHCT were made in the same conditions as cycle #1, which follows cure, without any other high temperature heating that would have affected cure or vitrification. From these results, it would be interesting to see if the specific heat capacity of the IHCT PMC would change in subsequent heating and what range of temperatures are required to effectively alter the specific heat capacity.



Infrared image of HT-02



FLUENT simulation for HT-02

Figure 6.12: Inner tool face IHCT temperature profile for HT-02 based on experimental thermal infrared measurements (°C) (above) and on simulation (K) (below)

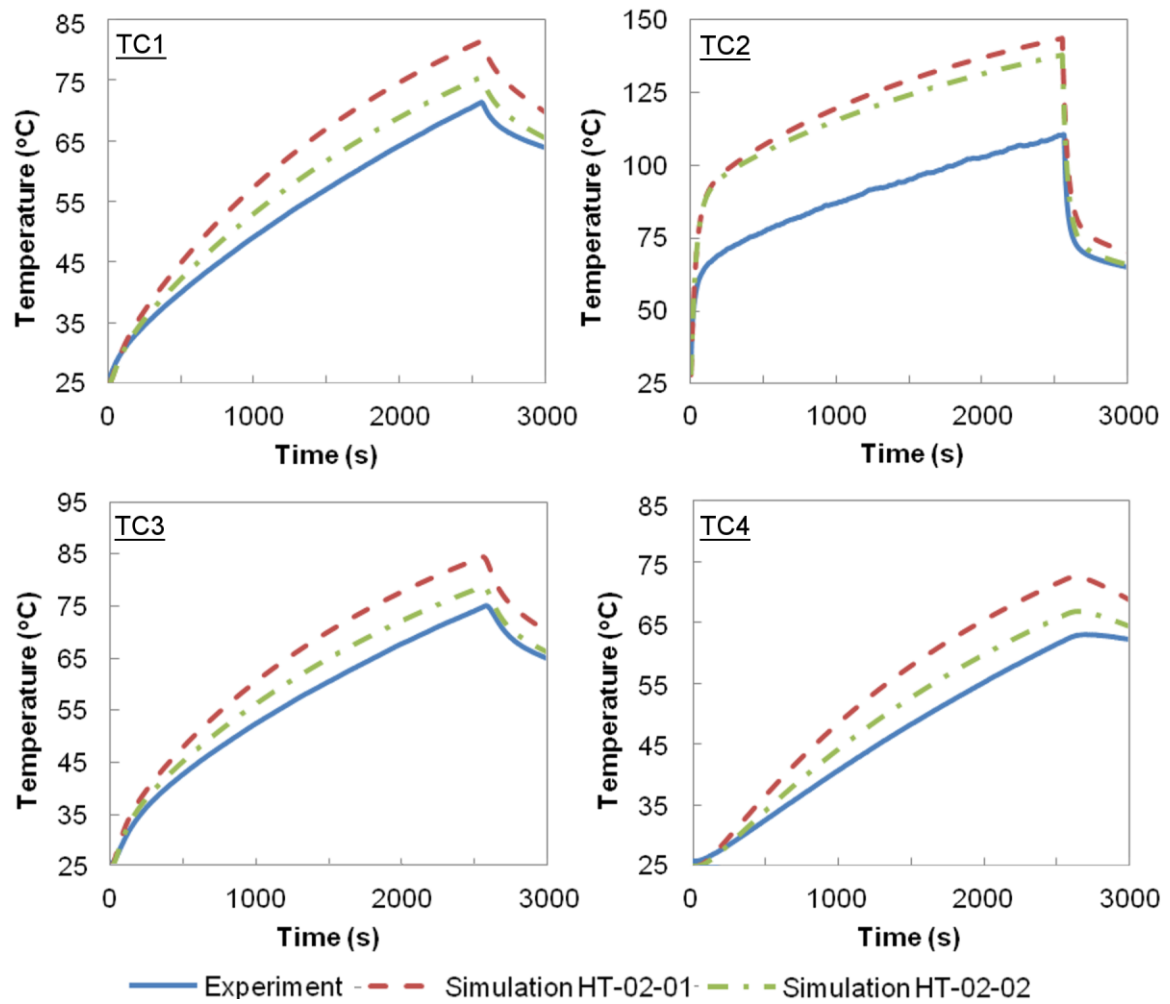


Figure 6.13: Comparison between HT-02-01, HT-02-02 and experimental temperatures at thermocouples TC1-TC4

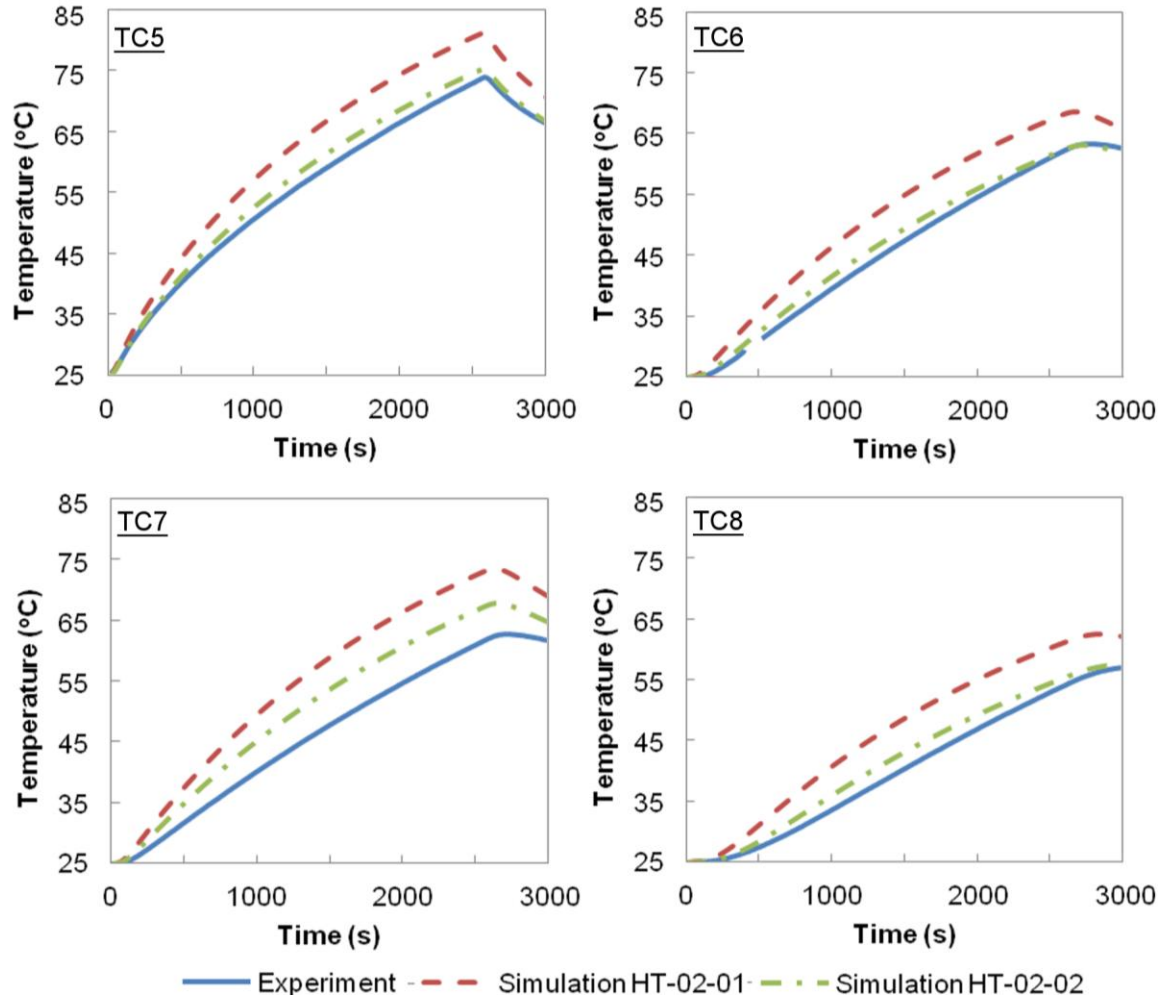


Figure 6.14: Comparison between HT-02-01, HT-02-02 and experimental temperatures at thermocouples TC5-TC8

Seeing the discrepancies between simulations and measurements, it was decided to see what was required in terms of simulation values to match thermocouple measurements. Based on HT-02-01 and assuming that the specific heat capacity values were accurate, it was conjectured that the heater generated less heat than what was believed. Additionally, it was observed that the simulated contact resistance between the heater and the copper was too high as heat accumulated in the heater.

Results for HT-02-03 with a lower heater power of 20W and no contact resistance between the heater and the copper appear in Figures 6.15-6.16. The modifications enabled matching the surface temperatures of the IHCT with much greater accuracy. The maximum

and minimum differences with the inner tool face thermocouples after 50min dropped to 3.3°C and 0.1°C respectively. The greatest discrepancy occurred at higher temperatures which suggested that the actual specific heat capacity in that range may be slightly lower than what was selected for HT-02-01 and HT-02-03.

From HT-02-3, it was concluded that simulations can predict with relatively good accuracy the temperatures across IHCTs. However, there are many uncertainties regarding material properties, boundary conditions and heat generation that may cause deviation between simulations and experimental measurements. These uncertainties can nonetheless be addressed through corrective measures such as curve fitting with experimental results if an exact solution is required.

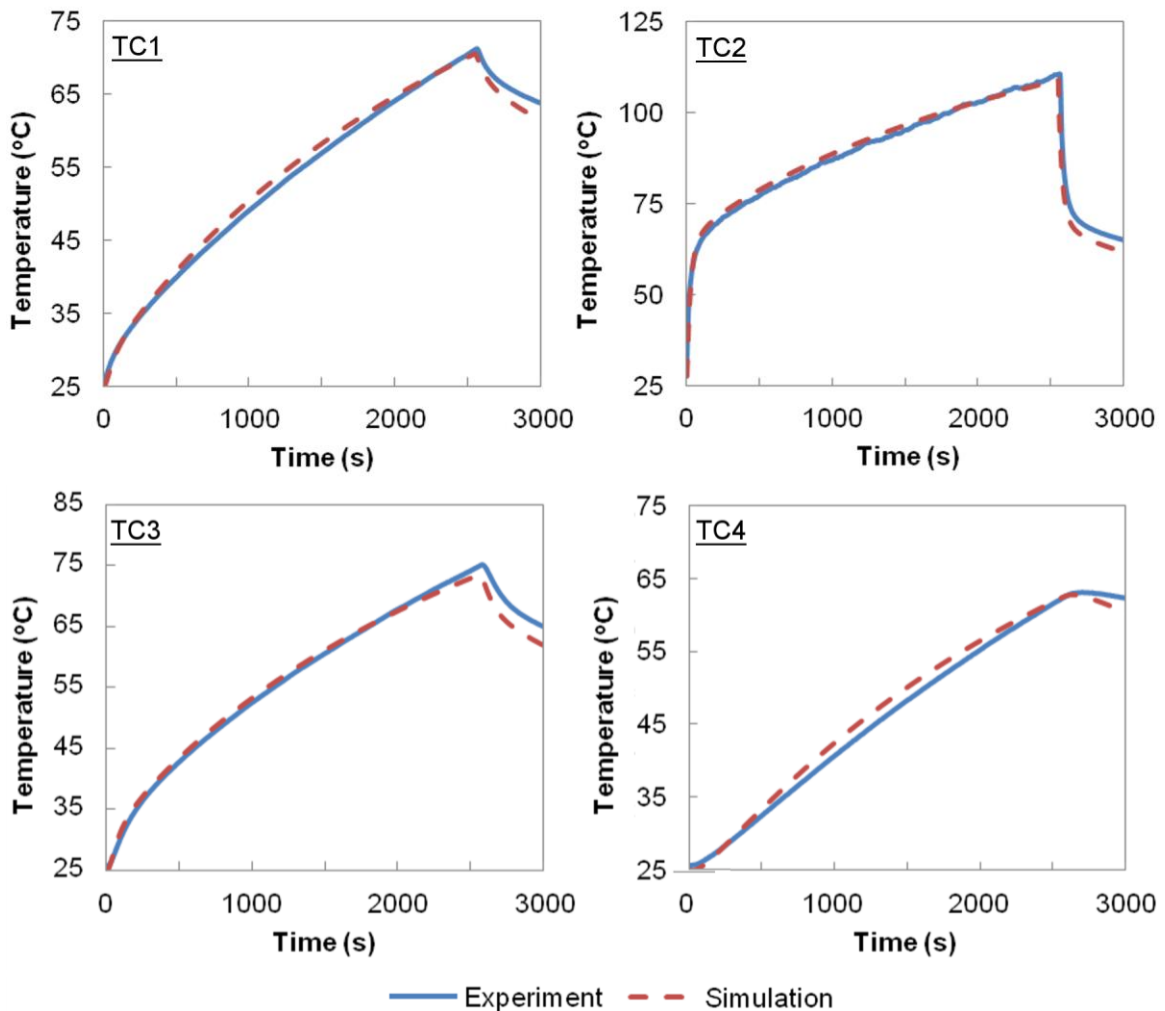


Figure 6.15: Comparison between HT-02-03 and experimental temperatures at thermocouples TC1-TC4

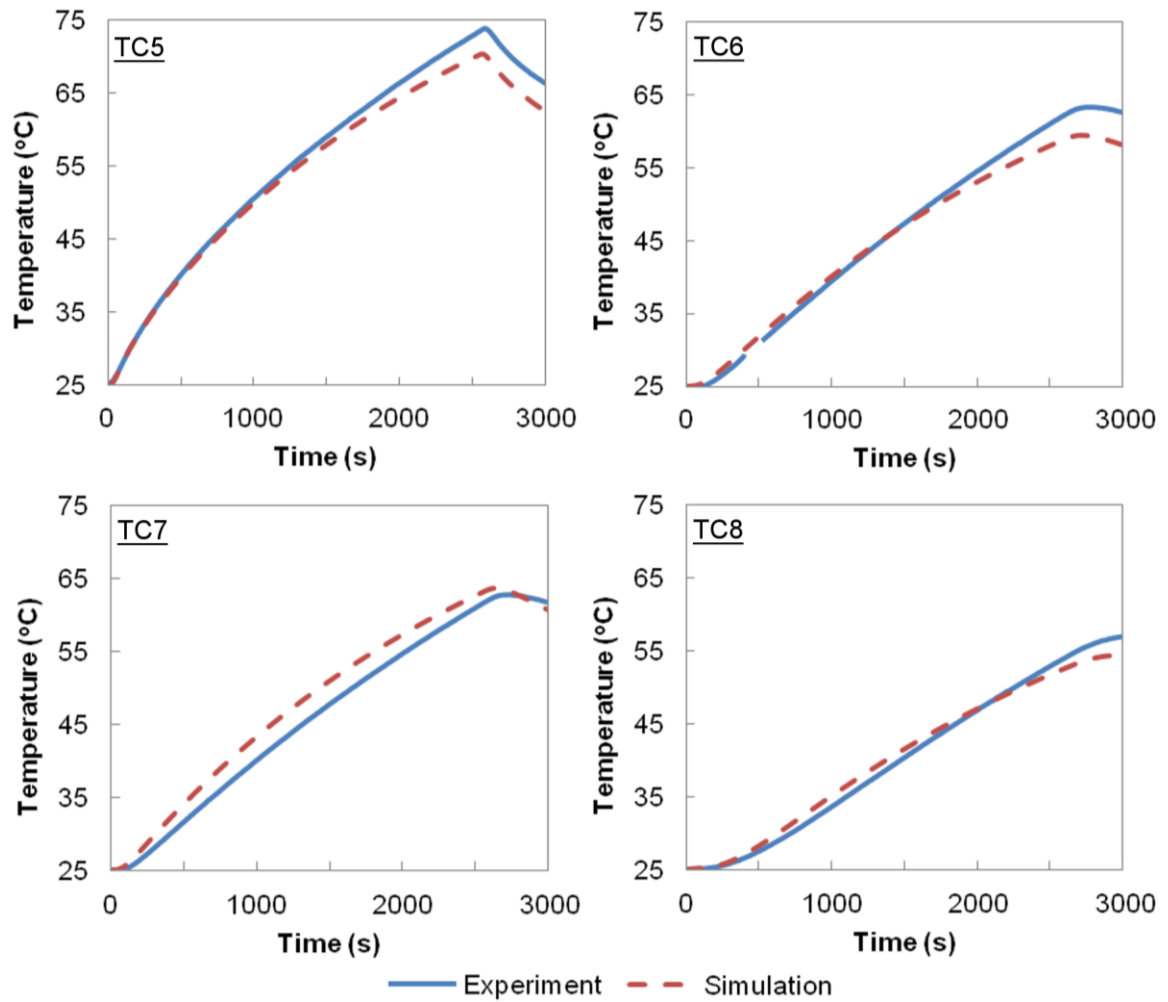


Figure 6.16: Comparison between HT-02-03 and experimental temperatures at thermocouples TC5-TC8

6.4 Program HT-03: parametric study of IHCTs

Transient simulations for program HT-03 sought to identify parameters affecting temperature uniformity at the inner tool face of IHCTs. Computational models of IHCTs having different physical properties and boundary conditions were produced and compared. The selection of parameters was based on the results of previous work performed at the University of Ottawa by Houde [23], Maksoud [24], and Brilliant [25], and on experimental observations made during the project. In total, four parameters were selected:

- A. Copper thickness;
- B. PMC thickness;
- C. Convection coefficient; and
- D. Location of the heater control thermocouple.

A simulation plan was devised following a Plackett-Burman approach. For simplicity a 2-level design was used, where a minimum and a maximum value were attributed to each parameter (Table 6.5).

In the case of the copper layer, it was decided to vary the thickness instead of investigating the effect of its presence due to the great increase in thermal conductivity brought by the copper, as seen in Chapter 5. Copper thickness was varied from 0.75mm to 2.00mm. The lower bound value was chosen to mimic the thin copper of IHCT-NRC while the higher value was chosen as an upper practical limit for such application.

Thickness values for the PMC were 3mm and 6mm respectively. Selection was based on prior IHCT-NRC tools [23]. The objective was to gauge the effect of the transverse thermal resistance on the temperature uniformity.

Convection coefficient values were $5\text{W/m}^2\cdot\text{K}$ and $15\text{W/m}^2\cdot\text{K}$ respectively. The range encompassed natural convection and light forced convection; its centres around $10\text{W/m}^2\cdot\text{K}$ as estimated for an IHCT in ambient air by Maksoud [24]. This parameter was used for gauging the potential of adding insulation.

Lastly, Chapter 5 showed temperature spikes in IHCTs resulting from heater controllers, which are detrimental to temperature uniformity. Thus, reduction of the spikes was investigated by altering the location of the heater control thermocouple. The control thermocouple was placed either near or far from the heater (Figure 6.17). In the near position, the control thermocouple was located 50mm away from the heater while in the far position it was located near the corner of the IHCT.

Table 6.5: Parameters and values for the design of experiments of HT-03

Parameter	Value	
	Minimum	Maximum
A. Copper thickness [mm]	0.75	2.00
B. Laminate thickness [mm]	3.00	6.00
C. Heat transfer coefficient [$\text{W}/(\text{m}^2\cdot\text{K})$]	5	15
D. Control thermocouple location with respect to the heater	Close	Far

Testing all combinations of parameters in a full factorial design would have required 16 distinct experiments. As this would have been time intensive, an 8-run fractional factorial design was used. The 8-run design was obtained by ignoring combined interaction of parameters ABCD as described in Appendix K. The choice of ignoring this interaction was made because of its low probability of having a strong influence over simulation results. The fractional factorial design generated contrasts (i.e. parameters and their interactions) with confounding factors. However, the effect of individual parameters was only confounded with 3-parameter interactions that have low chances of influencing the results (Appendix K). The plan of simulations is presented in Table 6.6.

Table 6.6: Plan of simulations of HT-03

Experiment	Copper thickness (mm)	PMC thickness (mm)	Convection coefficient ($\text{W/m}^2\cdot\text{K}$)	Controller proximity
HT-03-1	0.75	3.00	5	Close
HT-03-2	0.75	3.00	15	Far
HT-03-3	0.75	6.00	5	Far
HT-03-4	0.75	6.00	15	Close
HT-03-5	2.00	3.00	5	Far
HT-03-6	2.00	3.00	15	Close
HT-03-7	2.00	6.00	5	Close
HT-03-8	2.00	6.00	15	Far

6.4.1 Model description

Simulation models represented a 300mm x 300mm CPMC-IHCT (Figure 6.17) with different PMC and copper thicknesses in different simulations. A 200 μm thick co-cured layer was added between the PMC and copper layers as a thin wall thickness, in all simulations. Heat was generated by a flexible heater placed at the centre of the copper layer. The heater was 100mm wide, 100mm long and 2mm thick. It provided 350W of power. Since these simulations were used only for comparison purposes, no thermal resistance was included between the heater and the copper. The computational model geometry and mesh characteristics appear in Table 6.7. The imposed temperature profile for the IHCT was representative of the cure for SE-70 material; it consisted of heating at a rate of 2°C/min up to 122°C, keeping that temperature for 30min and cooling at a rate of 5°C/min.

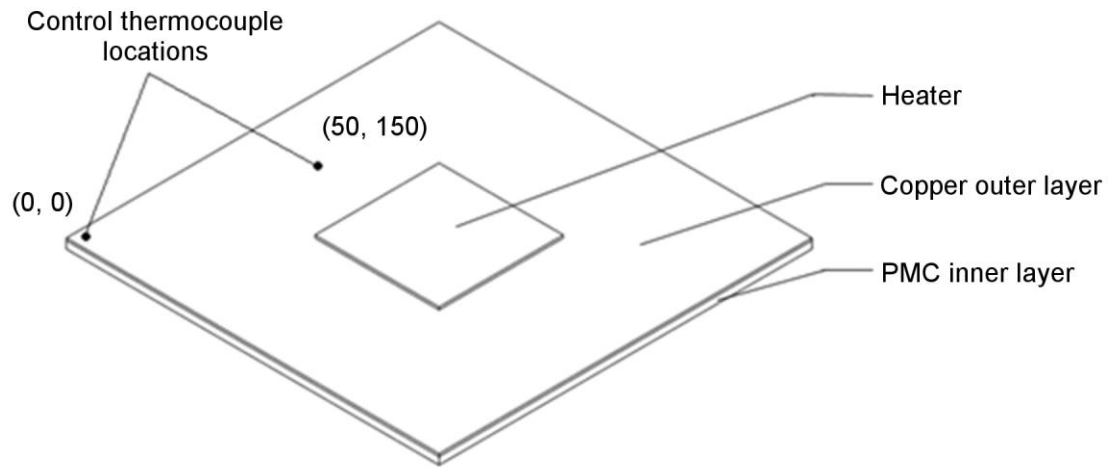


Figure 6.17: HT-03 model

Table 6.7: HT-03 model characteristics	
Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 3mm to 6mm
- Copper sheet	300mm x 300mm x 0.75mm to 2mm
- Heater	100mm x 100mm x 2mm
Element type	Hexahedron
Element length	12.5mm
Element thickness	
- PMC	3mm
- Copper	0.75mm to 2mm
- Heater	2mm
Number of elements	1216
Interfaces	
- Heater/copper	None
Copper/PMC	0.2mm of co-cured layer
Heater power	350W
Global convection coefficient	5W/m ² ·K to 15W/m ² ·K
Transient time steps	10s

Material properties are presented in Table 6.8. For simplicity and as the simulations were only used for comparison, properties were made independent of temperature. Furthermore, properties were not related to those of any specific material used in this work because simulations were performed prior to any material characterisation. Properties of the copper coating were based on the bulk properties of pure copper (section 2.2.2). The co-cured layer was assumed to be composed of 60% spherical copper particles embedded in an epoxy matrix, a similar value to what was measured experimentally in section 4.1.3; the thermal conductivity was obtained from the Lewis-Nielsen model in section 2.3.2.1. Properties for the PMC were approximated from literature values for a 60% fibre volume fraction, section 3.1.1.

Table 6.8: Material properties used for simulations, HT-03

Layer	Density (kg/m ³)	Specific heat capacity (J/kg·K)	Thermal conductivity (W/ m·K)
Copper coating	8940	381	387.6
Co-cured layer	6000	650	6.0
PMC	1600	795	5 ⊥ 0.4
Heater	1470	1465	0.18

All external boundaries for the models were set as uniform forced convection with a free stream temperature of 27°C (300K), characterised by a convection coefficient that is specific to each simulation either on or off providing. The heater controller was simulated using a FLUENT user define function. Heating was activated when the control thermocouple temperature fell 1°C below the target temperature, and it was deactivated when the temperature reached 1°C above the target temperature. The heater was shut down when the temperature of the heater reached a critical temperature of 200°C to simulate a protective system preventing overheating of the heater.

At each time step during the simulations the average temperature and the standard deviation across the inner and outer surfaces of the IHCT were monitored, as well as the control thermocouple temperature.

6.4.2 Results

Temperature evolution at the IHCT inner tool face and control thermocouple appears in Figures 6.18-6.19. Even though the control thermocouple temperature followed the desired temperature profile for all simulations aside HT-03-2, the average temperature at the IHCT inner tool face differed by being either greater or lower, depending on the experiment. In the future, this can be remedied by tailoring the imposed temperature profile for obtaining the desired average temperature across the inner IHCT surface.

In the case of HT-03-2, the heater became too hot and had to be turned off, preventing the control thermocouple from reaching its target temperature. Heat accumulation in the heater likely resulted from the combined effect of having a thin layer of copper that limited temperature distribution, and a control thermocouple far away from the heater that was cooled by convection.

Results also showed that the amplitude of temperature fluctuations was significantly reduced when the control thermocouple was positioned closer to the heater rather than at the corner of the IHCT. From this, a standalone simulation was performed where the control thermocouple was positioned in closer proximity to heater (~5mm), but it could not be simulated properly because the reaction time of the control thermocouple was too fast for the 10s simulation time steps. In reality, closer proximity would work but may be undesirable due to the high frequency at which the heater is activated and deactivated, leading to greater controller wear.

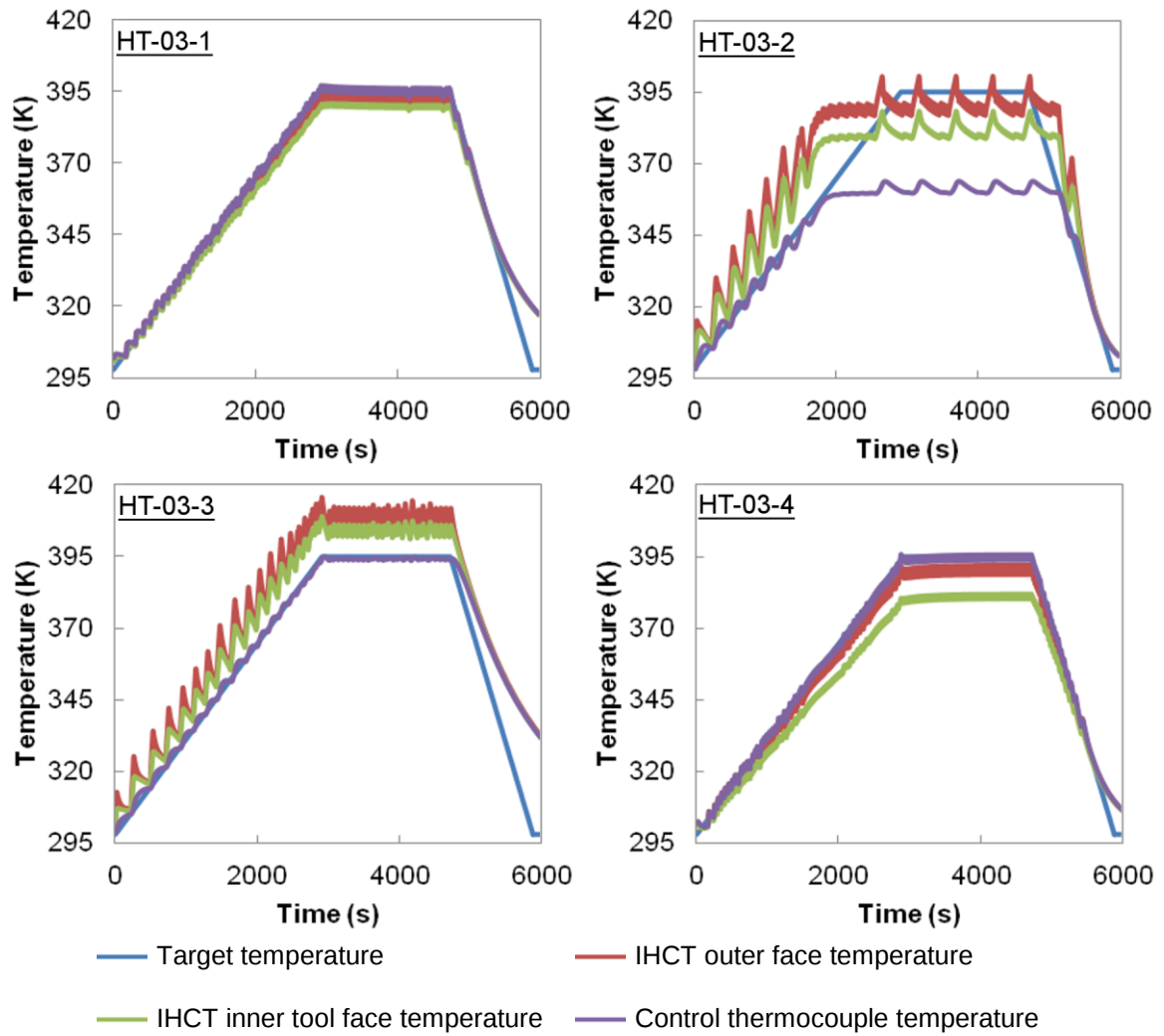


Figure 6.18: Temperature evolution for HT-03-1 to HT-03-4

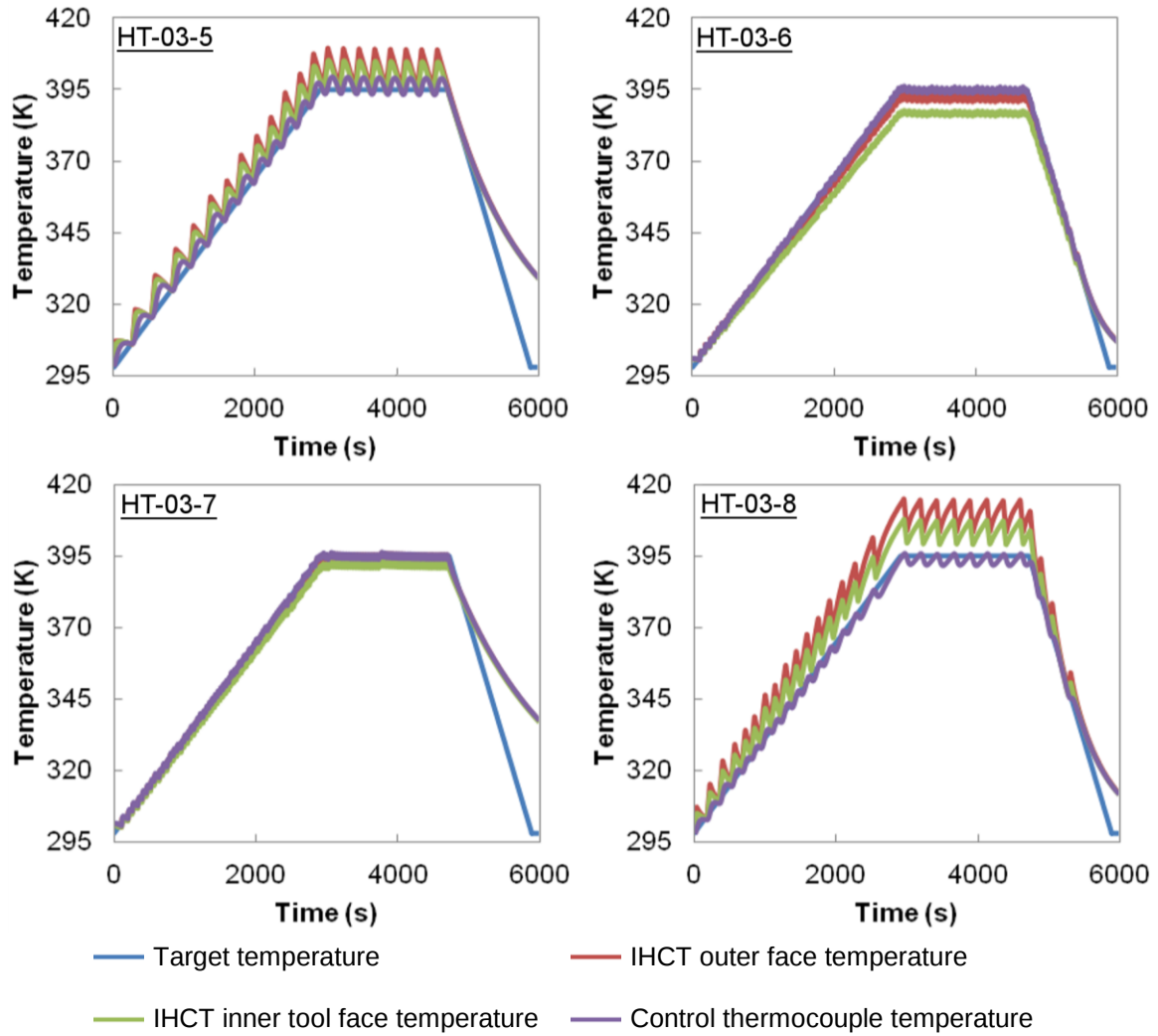


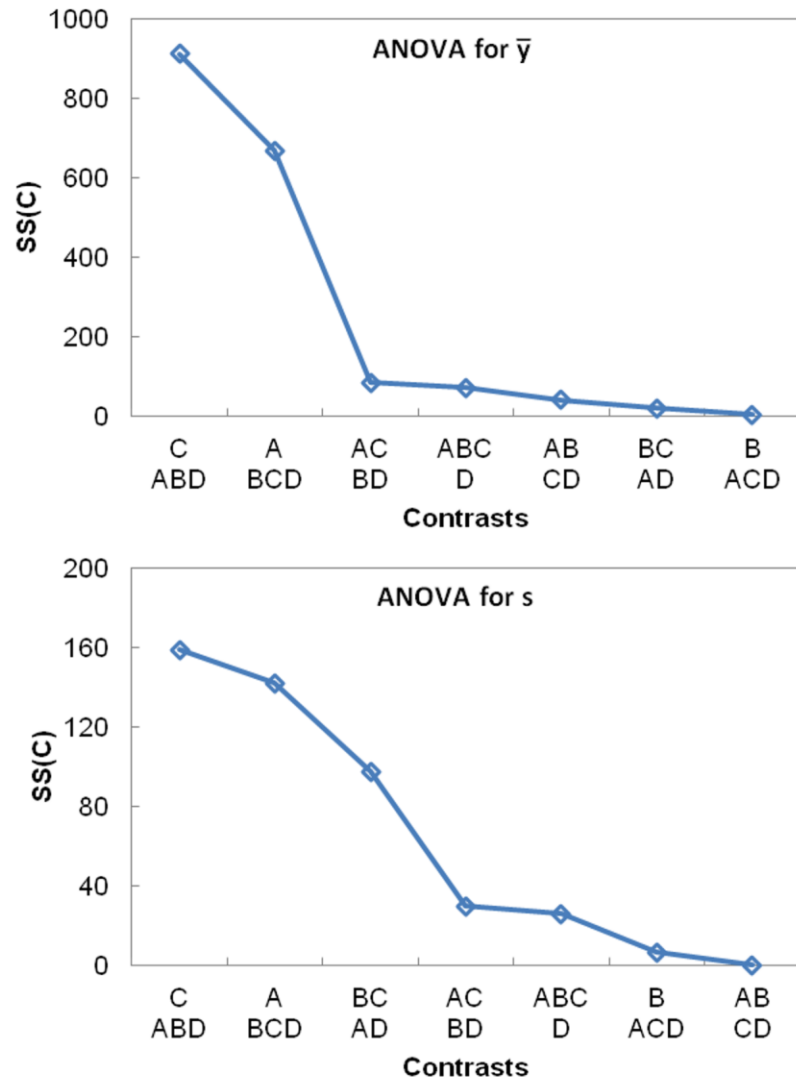
Figure 6.19: Temperature evolution for HT-03-5 to HT-03-8

Further analysis of the results was achieved by performing an analysis of variance (ANOVA) for finding parameters having the strongest influence over temperature uniformity across the inner tool face of IHCTs. The analysis investigated the variations of the standard deviation of the surface temperatures at the inner tool face for each time step. Two responses were analysed: the average $\bar{y}$ value of the standard deviation of surface temperatures for all time steps, and the standard deviation s of the standard deviation of surface temperatures at each time step. The latter response was used because it is tied to the frequency and amplitude of temperature spikes. Results appear in Table 6.9.

Table 6.9: ANOVA responses for the standard deviation of the inner tool face temperatures, HT-03

Experiment	Responses	
	$\bar{y}$ (°C)	s (°C)
HT-03-1	7.29	4.06
HT-03-2	20.42	9.64
HT-03-3	8.38	4.54
HT-03-4	14.95	7.99
HT-03-5	3.65	3.61
HT-03-6	7.90	4.28
HT-03-7	3.69	1.75
HT-03-8	9.96	4.67

Using the ANOVA responses, scree charts of the contrasts (i.e. parameters and their interactions) were developed following the Taguchi approach [143] and are depicted in Figure 6.20. The influence of the contrasts was evaluated using the sum of square of a contrast $SS(C)$, which is presented in Appendix K. In the analysis, the 3-factor interactions included in the contrasts were ignored due to the low probability of them having a significant effect on contrasts. Scree plot results of the two ANOVA showed that temperature uniformity was mostly affected by copper thickness and convection coefficient. The results also indicated that there was no important synergy between any parameters aside for interactions of factors AD (copper thickness and control thermocouple location) or BC (PMC thickness and convection coefficient) for response “s”. It is conjectured that the interaction arose from parameters BC. The effect of interaction BC was explained by the PMC acting as heat storage which varying capacity based on its thickness. Having more material for storing heat can slow the cooling caused by convection, thus minimising temperature spikes.



A: Copper thickness B: PMC thickness
C: Convection coefficient D: Control thermocouple location

Figure 6.20: Scree plots of the ANOVA responses, HT-03

The two main parameters A and C, copper thickness and convection coefficient, were compared using response plots for “ $\bar{y}$ ” and “ s ” (Figure 6.21). These plots compared the responses of each simulation based on the selected state of the parameters. For a two parameter analysis, the plots were made by drawing a square where each corner represents a possible combination of parameters with either a minimum or maximum state value. The

responses of each simulation were then classified in their respective corners based on the parameter combination.

Particular interest was given to lower response values as they correlate to the lowest standard deviation of the inner tool face temperatures. Results for “ $\bar{y}$ ” and “ s ” suggested that for maximising temperature uniformity, the IHCT design should be based on A+ and C-, high copper thickness and low convection coefficient.

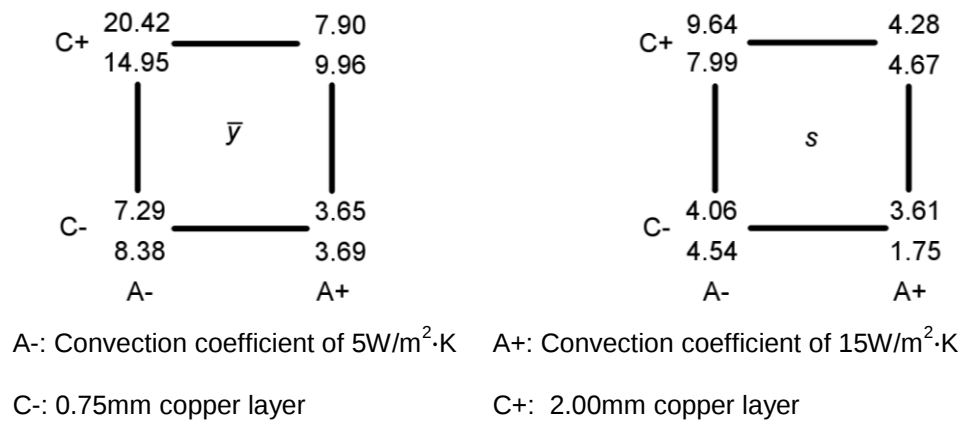


Figure 6.21: Response plots for parameters A and C, HT-03

In the end, results from HT-03 provided insight about parameters influencing temperature uniformity across IHCTs. It was found that heat losses from IHCTs had to be minimised, showing the importance of using insulation blankets. Another important parameter was the thickness of the copper layer. This parameter is further investigated in HT-04 and HT-05 due to the possibilities offered by the kinetic spray processes, such as varying copper thicknesses locally over a large area.

6.5 Experimental program HT-04: variable copper thickness

Based on results of HT-02 and HT-03, program HT-04 investigated the effect of channelling heat in IHCTs through copper layers of variable thickness. The premise was that kinetic spray processes can deposit copper patterns on a PMC as needed, to channel heat towards cooler regions (e.g. IHCT edges).

For this program, multiple IHCT copper patterns were compared through simulations. Comparison was made possible by using a constant copper volume. Marginal mass variations of up to 2.4% were observed between the models, due to geometrical limitations. Ultimately, the volumes of the copper patterns were equivalent to a copper sheet with a uniform thickness of 1mm that would cover the entire surface of an IHCT.

The models were classified in four distinct categories (Figure 6.23). The first category, labelled *Plain*, used a uniform copper thickness. It served as a benchmark to which other models were compared. The second category, labelled *Crater*, featured geometries with incremental physical steps, having a layer of copper thinnest at the centre of the IHCT and thickest at its edges. The premise of this category was that the copper added on the edges would store heat and mitigate the convective cooling of the edges. The third category, labelled *Hill*, featured a layer of copper thickest at the centre of the IHCT and thinnest at its edges. Three *Hill* models were made with increasing copper thickness at centre of the IHCT while keeping the copper volume constant. The rationale behind *Hill* models was that added copper would reduce thermal resistivity around the heater, so that more heat may be removed from the surrounding of the heater and be transferred in-plane. The final category, labelled *Cross*, featured a more complex copper. The pattern had thicker edges and centre, which were connected together by channels which resembled a cross.

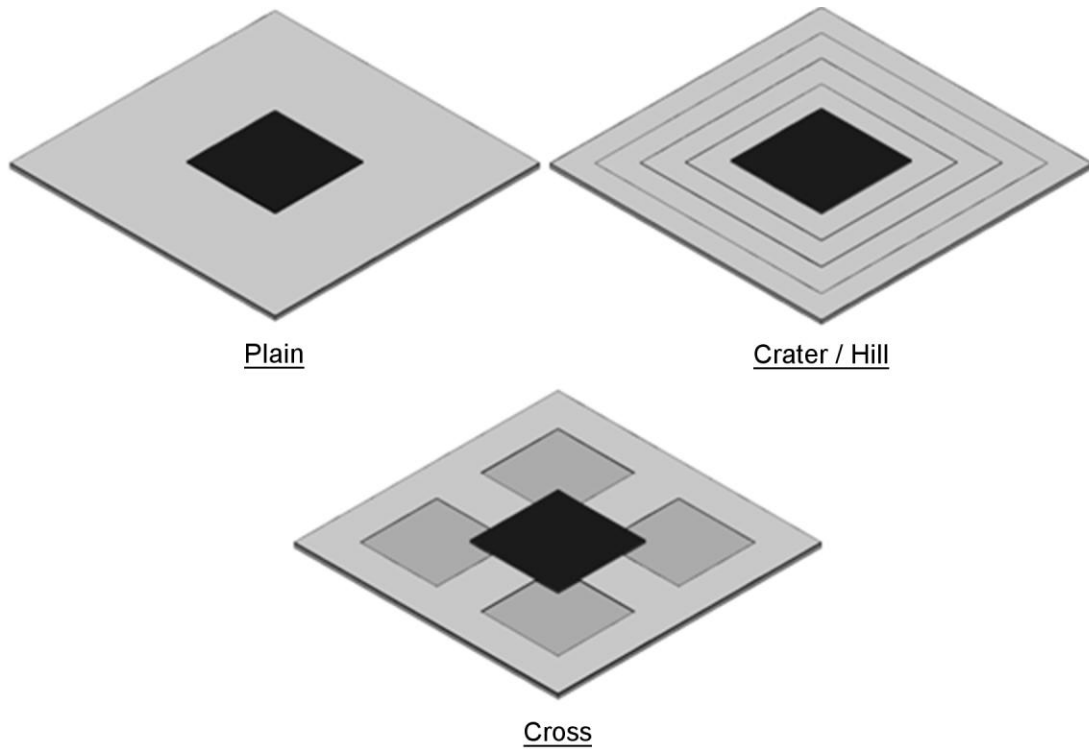


Figure 6.22: Representations of model categories, HT-04

6.5.1 Model description

Simulation models represented a 300mm by 300mm IHCT featuring a 4mm thick PMC layer. Heat was generated by a 100mm by 100mm heater located at the centre of the copper layer, producing a 75W of power. The simulations were performed under steady-state conditions to minimise computational time. Transient simulations were not deemed valuable as the effect of the heater controller was not the focus of HT-04.

The geometry of the different copper patterns is represented in Figures 6.23-6.24. In the figures, a number is given to each region of the copper pattern. The copper thicknesses associated with the regions appear in Table 6.10.

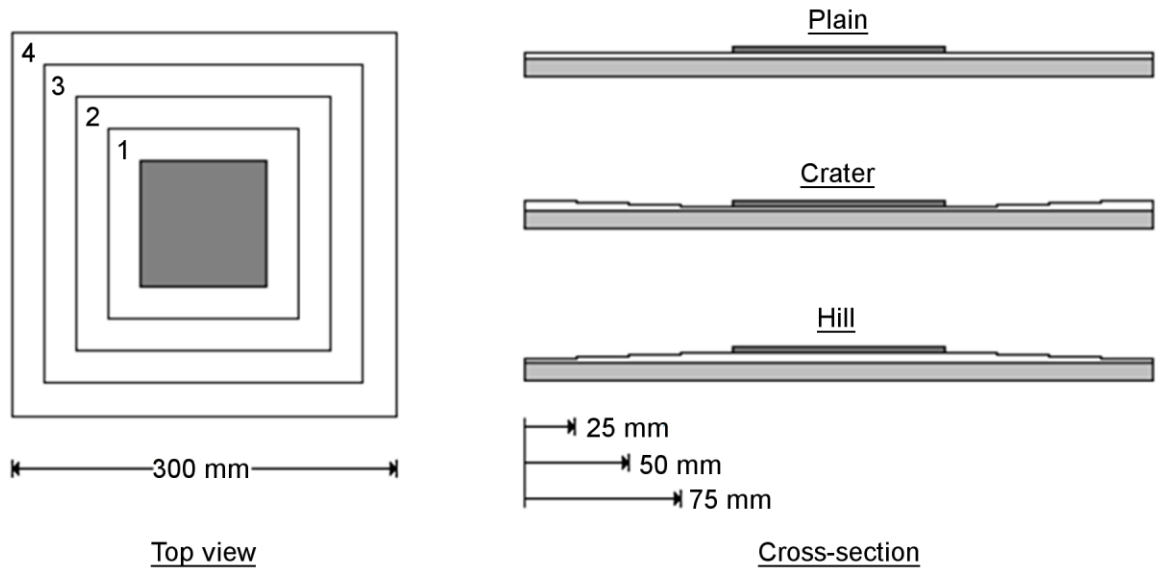


Figure 6.23: Diagram for the plain, crater and hill IHCTs, HT-04

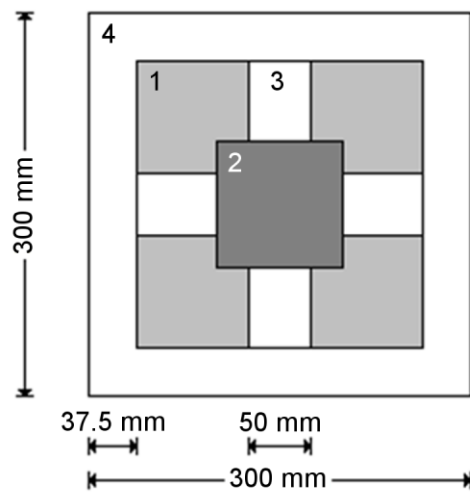


Figure 6.24: Diagram for the cross model IHCT, HT-04

Table 6.10: Geometry and volume of the copper patterns, HT-04

Region	Copper pattern thicknesses (mm)					
	Plain	Crater	Hill-1	Hill-2	Hill-3	Cross
1	1.0	0.5	1.3	1.6	2.1	0.50
2	1.0	0.8	1.1	1.3	1.4	1.23
3	1.0	1.1	0.9	0.8	0.6	1.23
4	1.0	1.5	0.8	0.5	0.2	1.23
Volume difference (%)	Reference	+1.4	+0.8	+0.6	+0.8	+2.4

Note: (+) sign indicates that the pattern occupies a greater volume than the reference.

Characteristics of the models appear in Table 6.11. A uniform convection coefficient of $5\text{W/m}^2\cdot\text{K}$ was applied to all external boundaries. Furthermore, since these models were only used as comparative tools, no contact resistance was modelled between layers, and no co-cured layer was modelled. The material properties used are the same as in HT-03 (Table 6.8). Validation of the steady-state analysis was achieved through a transient analysis using the same user defined function as in HT-03 to control the temperatures of models *Plain*, *Hill-2* and *Cross*.

Table 6.11: HT-04 model characteristics

Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 4mm
- Copper sheet	300mm x 300mm x variable
- Heater	100mm x 100mm x 2mm
Element type	Hexahedron
Element length	12.5mm
Element thickness	
- PMC	2mm
- Copper	4 cell thick for <i>Crater</i> and <i>Hill</i> 2 cell thick for <i>Plain</i> and <i>Cross</i>
- Heater	2mm
Number of elements	
- Plain	2368
- Crater	2720
- Hill-1	2592
- Hill-2	2592
- Hill-3	2592
- Cross	2188
Interfaces	
- Heater/copper	None
- Copper/PMC	None
Heater power	75W
Convection coefficient	5W/m ² ·K
Transient time steps ^a	10s

^a For validation of models *Plain*, *Hill-2* and *Cross*

6.5.2 Results

Steady-state temperature profiles of the simulations obtained at the inner tool face were analysed. The images, appearing in Figure 6.25, show that most patterns lead to radial heat flow. This was expected as thickness steps of the copper patterns were generally concentric to the heater. The only exception was observed using the *Cross* pattern that channelled heat towards the thicker copper sections. However, this heat channelling was not sufficient for adequately heating the edges.

Statistical analysis of temperatures at the inner tool face, appearing in Table 6.12, revealed modest differences between the copper patterns in terms of average surface temperature. However, a greater impact of the patterns was observed in terms of maximum and minimum surface temperatures. The maximum temperature increased when there was less copper around the heater (*Crater* and *Cross*) due to the larger thermal resistance, which prevented heat from leaving the area. This was even observed despite heat channels in the case of the *Cross* model. Conversely, a drop in maximum temperature was observed with thicker conductive layers around the heater (*Hill-2* and *Hill-3*). Furthermore, all patterns except *Hill-1* reduced the minimum surface temperature compared to the *Plain* model. This was either due to a lack of copper at the edges (*Hill-2* and *Hill-3*) or because heat could not be extracted from the centre of the IHCT (*Crater* and *Cross*).

Table 6.12: Analysis of the inner tool face temperatures, HT-04

Model	Inner tool face surface temperatures (K)			
	Average	Standard deviation	Maximum	Minimum
Plain	379.2	± 7.3 (8.4)	397.4	370.0
Crater	378.1	± 11.1	406.9	367.3
Hill-1	383.6	± 6.9	399.9	373.5
Hill-2	380.6	± 6.6 (7.2)	395.4	369.7
Hill-3	380.1	± 7.4	394.3	364.8
Cross	377.8	± 9.2 (10.9)	399.5	366.8

Note: Brackets used for transient results

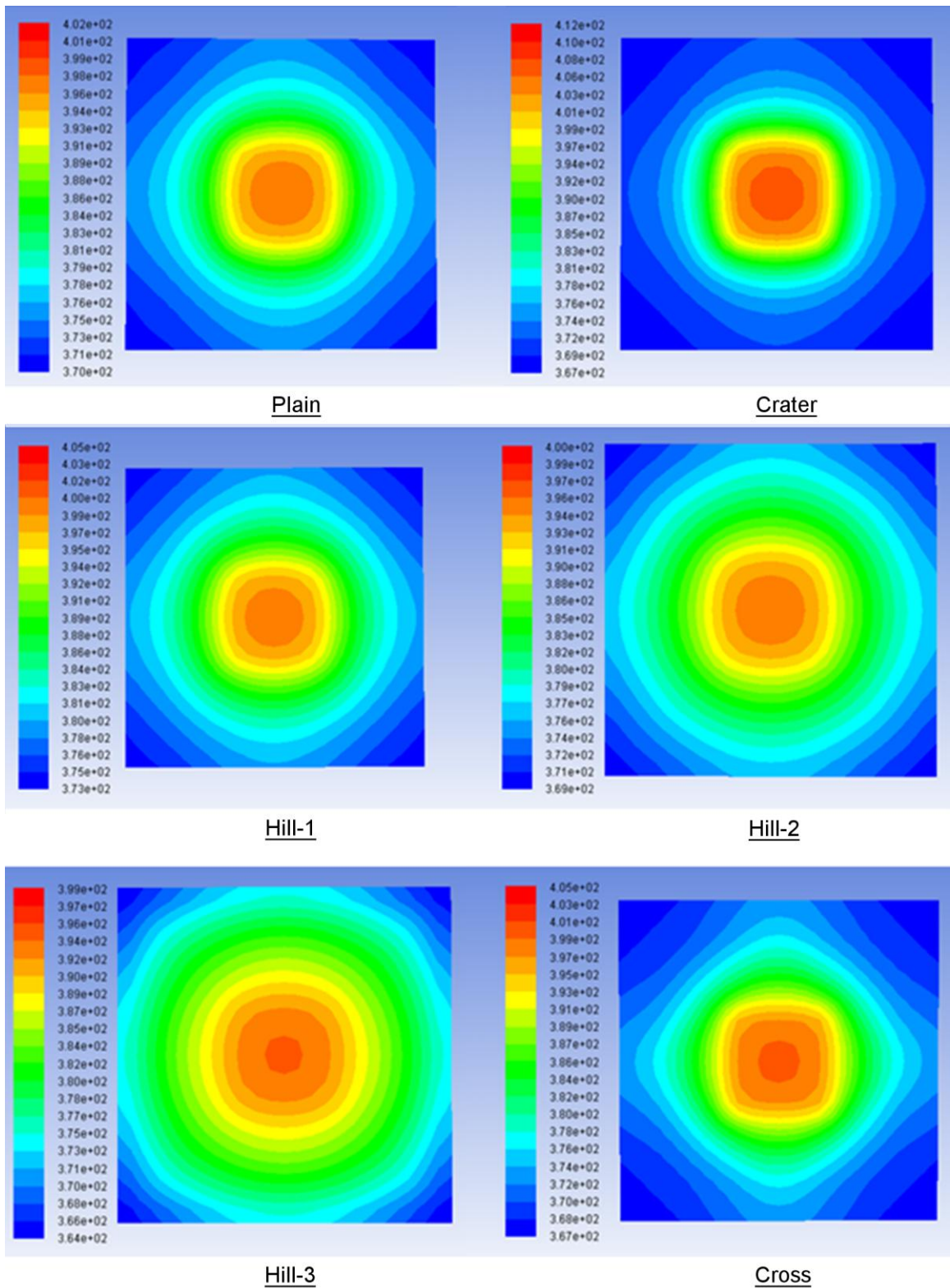


Figure 6.25: Temperature distribution (K) across the inner tool face of IHCTs, HT-04

Analysis of the standard deviation of surface temperatures showed that temperature uniformity was adversely affected by reduced thermal conduction around the heater (*Crater* and *Cross*) compared to a case of a copper layer having a uniform thickness. Additionally, marginal improvements were obtained by having a thicker copper layer near the heater (*Hill-1* and *Hill-2*). However, temperature uniformity was hindered when too much copper was removed from the edges and placed at the centre of the IHCT, such as with *Hill-3*. Nevertheless, a 10% increase in temperature uniformity was achieved by *Hill-2*, suggesting that a thicker copper layer should be deposited in the vicinity of heaters when using kinetic spray processes.

It is interesting to note that the transient analysis of selected experiments showed the same trend as steady-state results. The major difference was that greater standard deviations of surface temperatures were observed due the temperature spikes induced by the on/off control system of the heater. Nevertheless, this confirmed that a steady-state analysis may be used for evaluating conduction in a copper pattern, which is desirable for reducing computational time.

From program HT-04 it was concluded that marginal improvements can be achieved for temperature uniformity of IHCTs by using copper layers of variable thickness. Nevertheless, copper patterns may still be useful for channelling heat flow for cases that require non-uniform temperatures.

6.6 Program HT-05: investigation of copper thickness

Program HT-05 further investigated the effect of copper layer thickness on temperature uniformity in IHCTs. Unlike HT-04, this program sought to analyse the effect of altering the global copper mass used in IHCTs. Copper layers of various thicknesses were tested and compared by analysing the standard deviation of inner tool face temperatures. Simulations were performed under steady-state conditions for minimising computational time.

6.6.1 Model description

Simulation models were based on the *Plain* model of program HT-04. They consisted of a 300mm by 300mm IHCT featuring a 4mm thick PMC layer. The thickness of the copper layer was varied from 0mm to 10mm. Heat was generated by a 100mm by 100mm heater that produced 75W of power. The characteristics of the models were similar to those of the *Plain* model, and appear in Table 6.13. Material properties used are the same as in programs HT-03 and HT-04 which appear in Table 6.8.

Table 6.13: HT-05 model characteristics

Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 4mm
- Copper sheet	300mm x 300mm x 0mm to 10mm
- Heater	100mm x 100mm x 2mm
Element type	Hexahedron
Element length	12.5mm
Element thickness	
- PMC	2mm
- Copper	1 cell thick for every mm
- Heater	2mm
Number of elements	
- For 2mm copper layer	2368
Interfaces	
- Heater/copper	None
- Copper/PMC	None
Heater power	75W
Convection coefficient	5W/m ² ·K

6.6.2 Results

Standard deviations of inner tool face temperatures are presented for each copper layer thickness in Figure 6.26. It should be noted that a standard deviation value for a 0mm copper layer was omitted from the figure because its value (121K) was more than one order of magnitude greater than the second highest value which was 7.3K for the 1mm copper layer. This further confirmed the great advantage of using a copper layer in IHCTs.

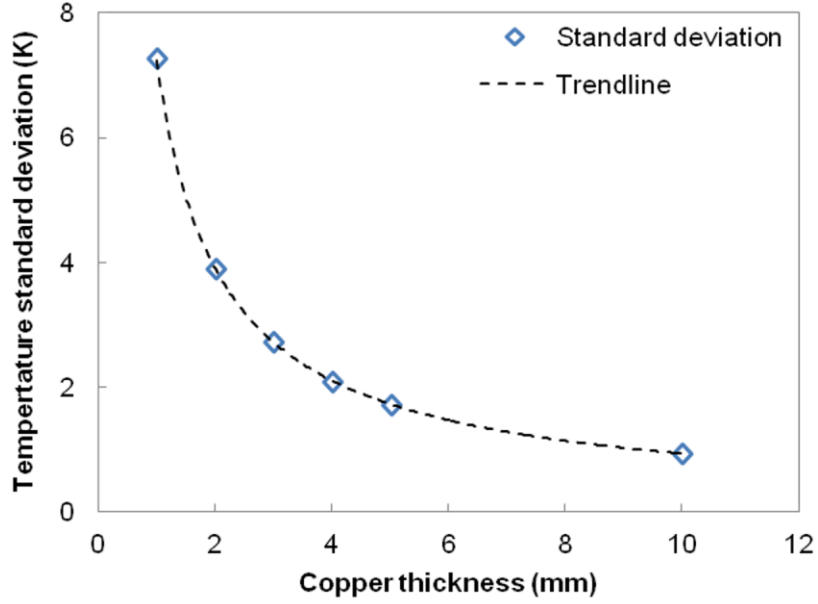


Figure 6.26: Effect of copper thickness on standard deviation of inner tool face temperatures, HT-05

Increasing the copper layer thickness beyond 1mm resulted in greater temperature uniformity but provided diminishing returns as improvements reduced in significance as the copper layer became thicker. The trend of the reduction of standard deviation of surface temperatures followed a power function law of the form:

$$\bar{y} = a h_{Cu} + b^c \quad (4.5)$$

where $\bar{y}$ is the standard deviation of surface temperature, h_{Cu} is the copper thickness in mm, and a , b and c are constants. For this particular set of simulations, the constants a , b and c were respectively evaluated at 7.61, 0.0511 and 0.93, using curve fitting options in Excel. It is interesting to note that constant b denotes the offset of the curve caused by the PMC layer.

From the model and results it was observed that standard deviation of surface temperatures nearly halved (reduction of 46%) every time the copper layer doubled in thickness. Interestingly, this improvement in temperature uniformity appeared to be directly related to thermal resistance. From Equation (3.5) ($R = l/kA$) it can be seen that

thermal resistance doubles each time the heat flux area doubles, which is equivalent to doubling the copper thickness. Hence, in an optimal scenario the standard deviation of surface temperatures should be halved by doubling the copper thickness. It is conjectured that the small discrepancy between simulations and theory were caused by the presence of the PMC layer. However, most of the difference may likely have come from the fact that the heater was not a singularity, as it covered approximately 11% of the IHCT, which distributed heat more effectively.

Finally, it was deduced from the results that obtaining a reasonable standard deviation of surface temperature ($\sim 2\text{K}$) from an industrial standpoint would require a 4mm thick layer of pure copper. Considering that kinetic sprayed copper is less conductive than bulk copper, an even thicker copper layer would be required. This would result in impractical copper thicknesses, suggesting that other means are required for achieving the desired temperature uniformity.

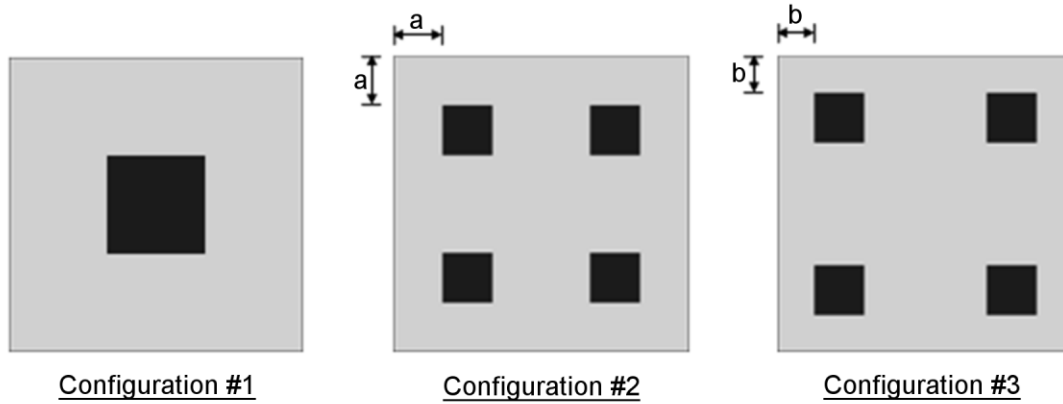
6.7 Experimental program HT-06: Multiple heaters

In all previous programs, a single heater was positioned in the middle of the IHCT. This resulted in uneven IHCT temperatures, even when using thick copper layers. Program HT-06 was performed for assessing the effect of de-localising heat generation. It consisted in comparing the standard deviation of the inner tool face temperatures when using one and four heaters having the same total heat output. Simulations were again performed under steady-state conditions for minimising computational time.

6.7.1 Model description

Simulation models were based on the *Plain* model of program HT-04. They represented a 300mm by 300mm IHCT featuring a 4mm thick PMC layer. Two different copper layer thicknesses were tried, 1mm and 2mm. Heat was generated either by a 100mm by 100mm heater that produced 75W, or by four 50mm by 50mm heaters producing 18.75W each. All heaters had a thickness of 2mm. Three distinct heater configurations were tested and appear in Figure 6.27. Single heater experiments used configuration #1, wherein the heater was positioned in the middle of the IHCT. Additionally, two other heater configurations were tried in multiple heater simulations. Configuration #2 had the heaters positioned in the middle of each quadrant of the IHCTs, while the configuration #3 had the heaters slightly closer to the IHCT edges for mitigating the convective losses at the edges.

Characteristics of the simulation models were similar to the *Plain* model and appear in Table 6.14. Material properties used are the same as those in programs HT-03, HT-04 and HT-05 which appear in Table 6.8.



Note: $a = 50\text{mm}$ and $b = 37.5\text{mm}$

Figure 6.27: Heater configurations for HT-06

Table 6.14: HT-06 model characteristics

Property	Characteristic
Domain dimensions	
- PMC	300mm x 300mm x 4mm
- Copper sheet	300mm x 300mm x 1mm and 2mm
- Heater	100mm x 100mm x 2mm
Element type	Hexahedron
Element length	12.5mm
Element thickness	
- PMC	2mm
- Copper	1 cell thick for every mm
- Heater	2mm
Number of elements	
- For the 1mm thick copper	1792
- For the 2mm thick copper	2368
Interfaces	
- Heater/copper	None
- Copper/PMC	None
Heater power	75W
Convection coefficient	$5\text{W/m}^2\cdot\text{K}$

6.7.2 Results

Temperature profiles for simulations using 1mm thick copper layer are presented in Figure 6.28. Results show that using multiple heaters reduced the overall surface temperature range and that temperature peaks under the heaters were much lower compared to the single heater experiment. Configuration #2 resulted in more uniform temperatures at the centre of the IHCT at the expense of greater cooling along the edges. Conversely, configuration #3 mitigated the heat losses at the edges but resulted in a cooler IHCT centre.

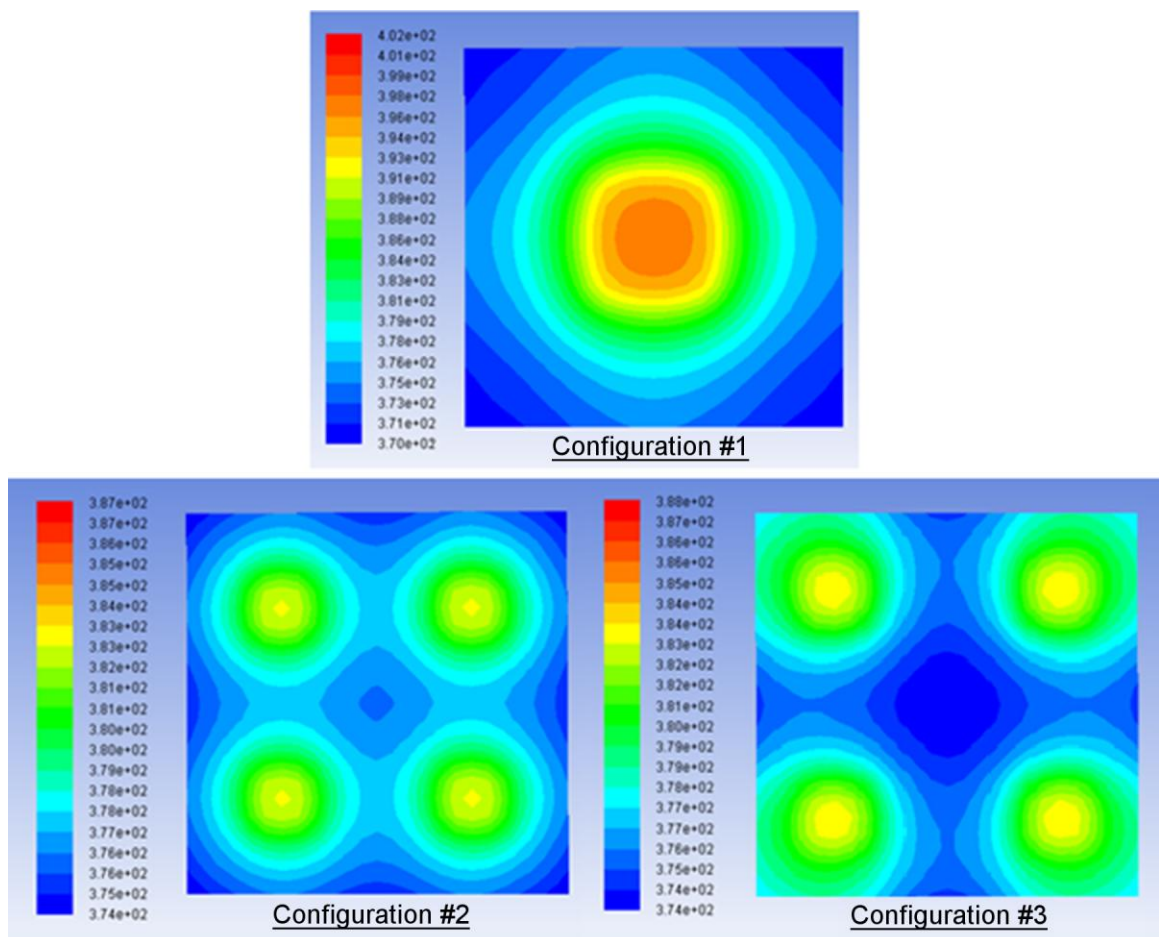


Figure 6.28: Inner tool face temperature profiles (K) for HT-06 models using a 1mm copper layer

Analysis of standard deviation of the inner tool face temperatures, appearing in Table 6.15, revealed substantial improvement in temperature uniformity brought by multiple heaters. For all simulations the standard deviation reduced by 3.6 times when using configuration #2 instead of configuration #1, which was nearly twice the improvement that could be achieved by increasing copper thickness from 1mm to 2mm.

Results also showed that configuration #2 was better than configuration #3. This indicated that there are little convective losses from the edges in thin IHCTs, and that heaters should be distributed uniformly across the IHCT outer face. However, further analysis should be performed on thicker IHCTs and IHCTs with flanges, to assess the effect of edge cooling.

Table 6.15: Standard deviation of the inner tool face temperatures, HT-06

Copper layer thickness	Standard deviation of the inner tool face temperatures (°C)		
	Configuration #1	Configuration #2	Configuration #3
1 mm	±7.27	±2.00	±2.56
2 mm	±3.90	±1.09	±1.34

In the end, a very satisfactory 1.09°C standard deviation of the inner tool face temperatures in terms of industrial practice was achieved by using multiple heaters with a 2mm copper layer. In the future, greater effort should be put into increasing heater coverage area of IHCTs for achieving better temperature uniformity.

6.8 Conclusions

Programs HT-01 and HT-02 showed that heat transfer simulations can be used for predicting trends regarding temperature distributions in IHCTs. The following programs HT-03 to HT-06 contributed to the identification of important factors affecting temperature distributions across the inner tool face in IHCTs. These parameters included the thermal insulation, thickness of the conductive copper layer and distribution of the heaters (Table 6.16). Such knowledge can be used later as guidelines for improving temperature distribution for achieving uniform and consistent PMC cure conditions. It was also observed that even though heat flow can be directed using variable thickness copper patterns, only marginal improvements can be achieved with such patterns in terms of temperature uniformity, compared to a copper layer of uniform thickness.

Table 6.16: IHCT design considerations

Design considerations	Guidelines
Improving tool surface temperature uniformity	Lower heat losses and convection Increase tool in-plane thermal conductivity Distribute the heat source
Conductive layer geometry	Conductive material around the heater is mandatory for lowering heat accumulation Uniform thickness layer provides a simple yet effective method for distributing heat in a tool

Chapter 7

Conclusion

The goal of the thesis consisted in investigating the feasibility of applying copper coatings on PMCs using pulsed gas-dynamic spraying (PGDS) and assessing the viability of these coated PMCs (CPMCs) in integrally heated composite tooling (IHCT-CPMC) applications. Several milestones were sought and achieved from this thesis work, including:

- Producing CPMCs by depositing copper coatings on PMCs using PGDS;
- Characterising the physical, mechanical and thermal behaviours of CPMCs;
- Manufacturing an IHCT-CPCM prototype;
- Comparing an IHCT-CPMC with other types of IHCTs; and
- Identifying influential IHCT design parameters from heat transfer simulations made using FLUENT.

7.1 Production of CPMCs

The results presented herein showed that PGDS can be used effectively for building copper coatings onto carbon fibre/epoxy PMCs. The method featured the use of a layer of copper particles embedded in the epoxy resin and co-cured onto PMCs, for preventing spray-induced erosion and promoting coating adhesion. Success was achieved, much beyond earlier achievements where the method was used for applying zinc coatings on PMCs as developed by Robitaille et al. [22]. The fact that copper coatings could be produced, which are more difficult to form using kinetic spraying compared to zinc coatings [18], hints towards the possibility of depositing other materials for different applications using this basic method. This could potentially spark investigations towards erosion-resistant coatings for gas turbines, which motivated early work on thermal spray coatings for PMCs [14–17, 61, 62, 64, 101].

7.2 Characterisation of CPCMs

Processing parameters were varied, aiming at improving coating density towards bulk-like properties [66]. This was achieved by increasing carrier gas pressure and temperature. Ideal values of these parameters for the copper powders and PMC materials tested were 3MPa to 4MPa and 90°C respectively, which enabled satisfactory adhesion while preventing damage to the substrate. Based on previous CGDS results [59, 65–67] it is not expected that these PGDS spraying parameters will induce any oxidation due to the fast nature of the process. This constitutes a critical advantage over thermal spray processes as better thermal conductivity of the copper coating should result. However, PGDS currently stands as the only kinetic spraying process that can produce such copper coatings on PMCs. Work on CGDS showed that particles had to travel in clusters to be deposited efficiently on substrates featuring a high surface roughness like that of the co-cured layer, as enabled by PGDS. A similar trend was shown by Assaad et al. when they tried to form coatings on wire mesh [96]. The roughness of the mesh prevented the formation of any coating when CGDS was used, but coatings were realised using PGDS.

Characterisation of the CPMCs through short-span three-point beam bending showed that the coatings were brittle, which is expected of kinetic spray coatings due to particle-to-particle mechanical interlocking being weaker than metallic bonding. It was shown that CPMCs were mostly prone to failure through tensile stresses. Tensile stress at failure was dependent on the spray powder, as reported in [69]. A dendritic copper powder (SST-C5003) formed coatings with a greater cohesion strength of 96MPa compared to 59MPa for a water-atomised powder (Cu 105-3) having an irregular shape. The performance of the dendritic powder resulted from more intimate particle-to-particle contact, reflected by the lower coating porosity. These values are lower than what was reported for copper coatings made with HVOF (140-151MPa), wire arc (93-102MPa) and CGDS using helium (453-472MPa), but better than those obtained with CGDS using nitrogen (26-46MPa) [60]. A reason for the much lower performance of PGDS coatings compared to those made with CGDS using helium, other than spray defects, was the low temperature resistance of the PMCs that prevented the carrier gas from being heated beyond 100°C, limiting gas velocity. Hence, it is conjectured that stronger copper coatings could likely be obtained using higher temperature-resistant polymers in the PMCs.

Moreover, the nature of the spray powder also affected the shear strength of the coating-to-co-cured layer interface. In contrary to the tensile strength results, the water-atomised powder showed greater shear strength (18MPa) than the dendritic powder (7-17MPa). The reason was that during impact of dendritic particles with a substrate, particles partly shattered into dust that remained in the coatings without being systematically deformed. Hence, future work may focus on finding an ideal spray powder that could offer tensile strengths similar to those of the dendritic powder, and shear strengths at the coating-to-co-cured layer similar to those of the water-atomised powder. Nevertheless, shear strength was slightly above what was reported for zinc on PMC using PGDS [22], which was already greater than the shear strength of zinc on PMCs deposited using plasma and wire arc [16].

Characterisation of thermal resistance showed that both powders produced CPMCs that could resist temperatures up to 250°C in preliminary testing, which is of central

importance for IHCTs-CPMC. This was likely attributed to the co-cured layer that helped bridge the coefficient of thermal expansion between the coating and PMC.

Characterisation of mechanical cycling at an elevated temperature of 120°C showed that the integrity of the CPMC was mostly dependent on the initial amount of defects. Nonetheless, in the best case no fracture was seen when subjecting a CPMC to 69% of its maximum static stress for 100 cycles. Thus it can be concluded that CPMC tools can be envisaged confidently at least for IHCT applications involving smaller production runs.

7.3 Characterisation of IHCT-CPMCs

Following CPMC characterisation, an IHCT prototype made out of CPMCs was fabricated. The IHCT-CPMC was compared with an IHCT consisting of a bare PMC plate (IHCT-PMC) and with two IHCTs consisting of PMCs with a copper sheet cladding for improving in-plane thermal conductivity (IHCT-NRC). Temperature distributions across the inner tool face of the IHCTs were compared when they were heated by a 15W heater placed on their outer face. Results showed that the IHCT-CPMC significantly improved the in-plane thermal conductivity compared to the IHCT-PMC; the standard deviation of the inner tool face temperatures after 800s of heating was reduced to 1.6°C with the IHCT-CPMC from the 20.1°C of the IHCT-PMC. Coating defects and limited metallic bonding did lead to lower temperature uniformity compared to the IHCT-NRC, where the standard deviation of inner tool face temperatures was 0.9°C after 800s of heating. Nevertheless, the lower thermal conductivity of the coating is compensated by the ease of applying it on the curved surfaces of complex tools compared to the metal forming required to ensure intimate contact between a copper sheet and a curved PMC.

The thermal conductivity of the copper coating was not measured in this work. However, simulations in Chapter 6 revealed that for a case of point heating, the standard deviation σ_{std} of inner tool face temperatures is approximately proportional to the thermal resistance R of copper:

$$\sigma_{\text{std}} \sim R_{th} = \frac{l}{kbh} \quad (7.1)$$

where l is the length, k is the thermal conductivity, b is the width and h is the thickness of the copper coating or plate. Assuming that heat emanated from a point location, a crude estimate of the upper values of the coating in-plane thermal conductivity can be obtained by comparing the standard deviations of surface temperatures for the IHCT-CPMC and IHCT-NRC, as presented in Chapter 5. From Equation (7.1) only the standard deviation, thermal conductivity and thickness varied between the IHCTs. Assuming a linear dependency on these parameters, taking the ratio of standard deviations of the IHCTs yields:

$$\frac{\sigma_{\text{std, IHCT-CPMC}}}{\sigma_{\text{std, IHCT-NRC}}} \approx \frac{R_{th, \text{IHCT-CPMC}}}{R_{th, \text{IHCT-NRC}}} = \frac{kh_{\text{IHCT-NRC}}}{kh_{\text{IHCT-CPMC}}} \quad (7.2)$$

where the thermal conductivity of the coating can be isolated:

$$k \approx \frac{kt\sigma_{\text{std, IHCT-NRC}}}{t\sigma_{\text{std, IHCT-CPMC}}} \quad (7.3)$$

Values that were used in the equations were the standard deviation of the inner tool face temperatures (0.9°C for IHCT-NRC and 1.6°C for IHCT-CPMC), copper thickness (1.6mm for IHCT-NRC and 2mm IHCT-CPMC) and a thermal conductivity of copper (386W/m·K for IHCT-NRC). Solving the equation yielded an upper bound estimate of 174W/m·K for the in-plane thermal conductivity of the copper coating. This is equal to 45% of the thermal conductivity of bulk copper and falls in the range of 120-210W/m·K expected of a CGDS copper coating [97]. In reality the thermal conductivity value would be slightly lower due to the fact that the heater was not a point heat source. Nevertheless, it still offers a comparative value which suggests that the coating is better at conduction than thermal spray coatings [63], a desirable characteristic for an IHCT application.

7.4 Influential parameters for IHCTs

Simulations were also used for investigating parameters that influenced the inner tool face temperature distribution in IHCTs. A parametric study was undertaken for assessing the effect of copper thickness, PMC thickness, convection coefficient and location of the heater control thermocouple. It was shown that the most important parameters for distributing heat evenly were a low convection coefficient and a thick copper layer. For the prior, a practical solution can be found in covering IHCTs with insulation blankets to mitigate heat losses; for the latter a thicker copper sheet/coating may be applied. In the case of copper coatings there is also the possibility of varying the coating thickness locally to steer the heat flow. Unfortunately, simulations showed that heat flow can indeed be directed by copper heat paths but no real improvements could be made regarding temperature uniformity across the inner tool face of the IHCT by reorganising a fixed volume of copper. Hence, an optimal temperature distribution was obtained through a copper layer of uniform thickness. Simulations also showed that distributing heat by using numerous sources located evenly across the IHCTs constitutes a more effective solution for minimising temperature variations across the inner tool face of IHCTs. It follows that the copper coatings should be used for preventing hot spots between closely packed flexible surface heaters and not as a mean of distributing heat across large surfaces.

7.5 Future work

The work presented in this thesis showed the feasibility of fabricating copper CPMCs and confirmed their potential as an IHCT material. Future work can be done towards minimising coating defects by investigating different spray powders, altering the spray parameters, or by changing the co-cured layer. The latter is particularly interesting considering that surface roughness played such a great role when spraying PMCs with CGDS. Also, it is important to develop a co-cured layer that can be applied easily on curved surfaces; this is not the case with current practice as reported in the thesis. A solution may be found by producing epoxy films with copper particles already embedded, or possibly by applying a liquid resin loaded with copper particles.

Regarding the coatings, it would be interesting to measure the thermal conductivity of the PGDS copper coatings and observe how it really compares to that of coatings obtained with other spray processes. Copper coatings on PMCs also open the possibility of spraying other types of materials for other applications such as erosion-resistant coatings as mentioned above.

Finally it was shown that for IHCTs, distributing the heat sources was the most important factor towards temperature uniformity. It follows that another approach to IHCT-CPMCs should be investigated. Instead of relying on uniform thickness copper coatings heated through flexible heaters, it may be more desirable to use sprayed coatings as electrical resistances for generating heat. One could imagine spraying a path of resistive metal following say a serpentine shaped pathway (Figure 7.1) and passing an electrical current in the coating for achieving temperature uniformity. This would have the advantage of using less metal and spray gas, and it would eliminate the need for flexible heaters. Additionally, electrically resistant coatings may be applied onto curved surfaces, which is not possible with flexible heaters due to bending limitations. Finally, the heat flux could also be controlled by changing the configuration and spacing between the spray line, and by changing the width of the coating. Therefore, this opens up several new avenues for IHCTs and PGDS coatings.

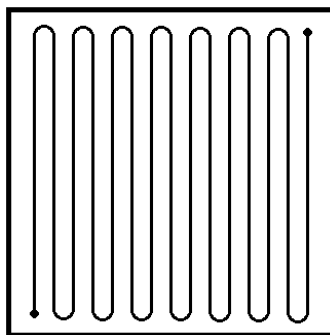


Figure 7.1: Diagram of an electrical resistive pathway sprayed by PGDS

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Appendices

Appendix A: Oven analysis

A convection oven (Carbolite PF60) with controlled temperature cycling was purchased for manufacturing the various PMC parts in the project. Several characteristics of the oven were tested, such as the heating rate ($5^{\circ}\text{C}/\text{min}$ vs $2^{\circ}\text{C}/\text{min}$), the effect of opening the cooling vent, and the error between the oven thermocouple readings and actual temperature. For comparison, all tests were performed following the same temperature cycle. This consisted in heating the oven to 100°C based on the target heating rate, holding that temperature for 5min, and setting a cooling rate of $5^{\circ}\text{C}/\text{min}$. Four tests were performed:

- Heating rate of $2^{\circ}\text{C}/\text{min}$ with cooling vent closed;
- Heating rate of $5^{\circ}\text{C}/\text{min}$ with cooling vent closed;
- Heating rate of $2^{\circ}\text{C}/\text{min}$ with cooling vent opened; and
- Heating rate of $5^{\circ}\text{C}/\text{min}$ with cooling vent opened.

Standalone temperature measurements were made with K-type thermocouples using a REED ST-9610B thermometer. Finally, based on the results, corrective measures were made to improve the oven.

Results

The desired oven temperature (set point), the temperature value of the oven thermocouple and the temperature of a thermocouple placed in the middle of the oven were compared for all temperature cycles. It can be seen from the results (Figure A.1) that the oven thermocouple measurements closely followed the target temperature during the heating and dwell phases. However, they were delayed by 1 minute during heating as compared to the target temperature, meaning that the temperature was lagging by 5°C for the quicker rate and by 2°C for the slower. Surprisingly, measurements from the oven thermocouple did not convey the actual temperature inside the oven chamber as showed by the standalone thermocouple measurements. The chamber never reached the target temperature, not even after the 5min dwell, for either heating rate.

Opening the cooling vent did have a significant effect for the higher heating rate but was marginal for the lower heating rate. Results indicate that at a target rate of 5°C/min was never reached; the maximum heating rate achievable was 4.0°C/min with the closed vent, but only 2.4°C/min with the vent opened. Minimal impact of the vent on the 2°C/min heating rate was explained by the slower heating rate than the maximum that was achieved with the opened vent which was 2.4°C/min. Moreover, the vent did help slightly in reducing the chamber temperature during the cooling phase, but a cooling rate of 5°C/min was never reached. Hence, a proper cooling system should be added to the oven for achieving precise control over the cooling rate.

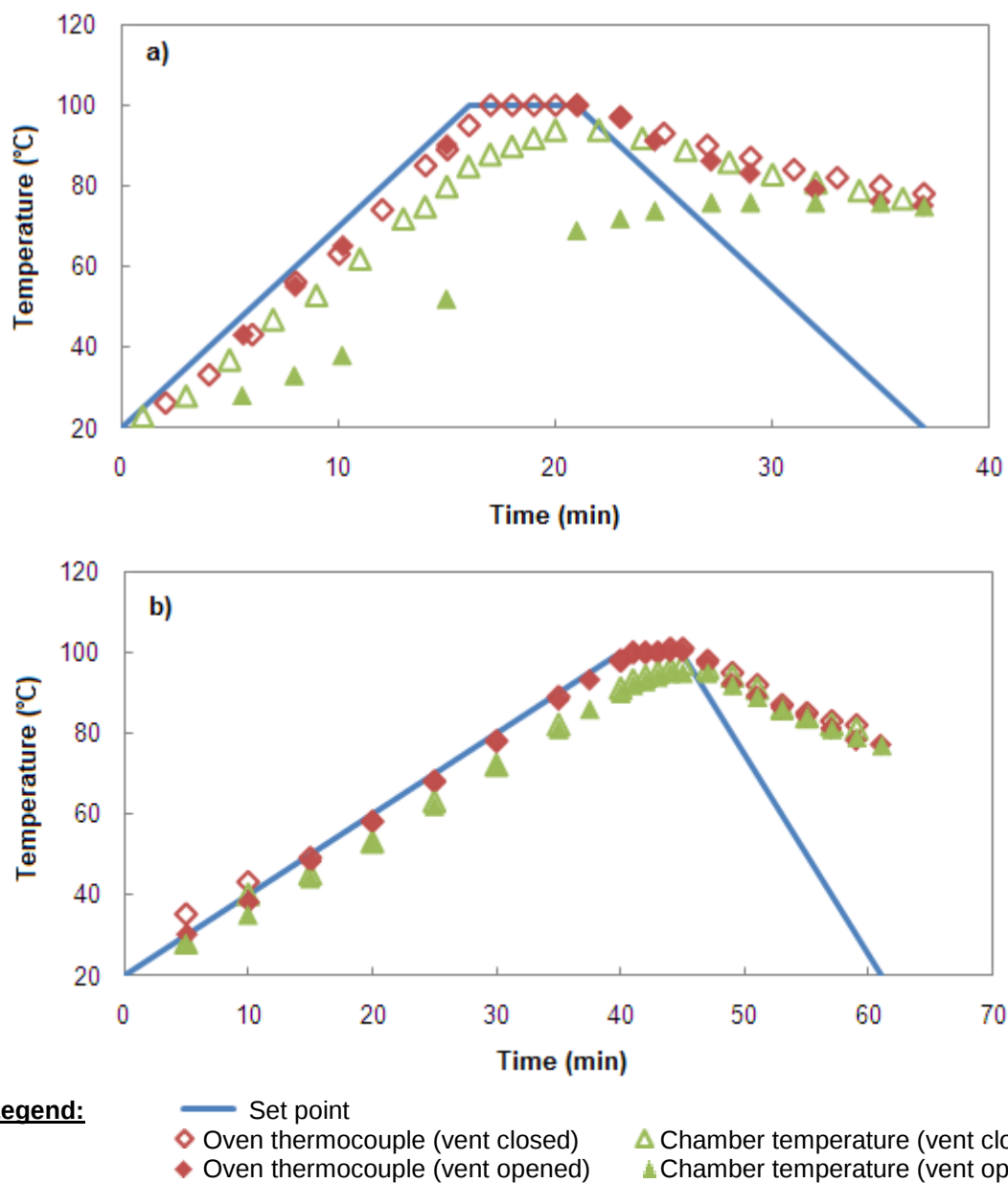


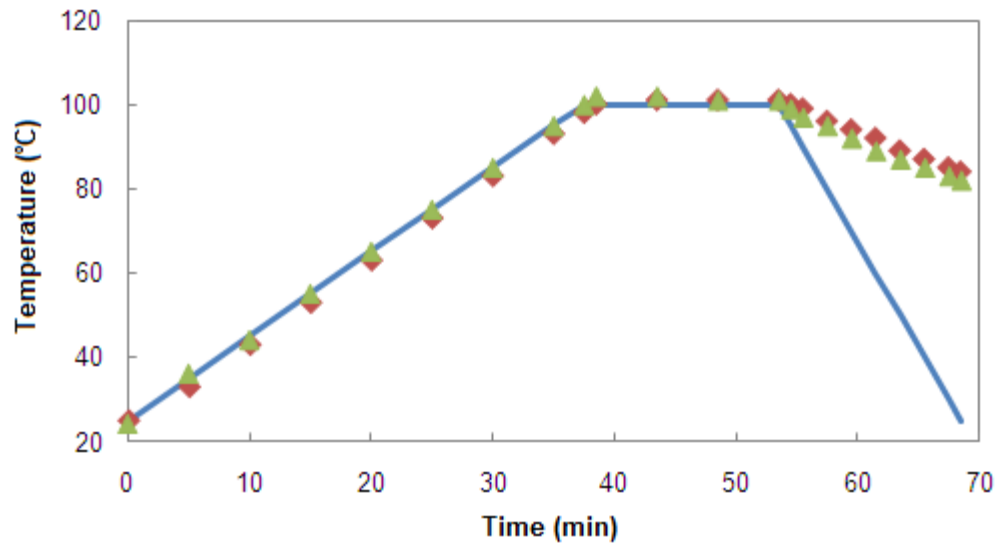
Figure A.1: Oven temperatures when heating rates of a) 5°C/min and b) 2°C/min are used

After testing, the oven was opened to understand the reason behind the inability of the oven in reaching the target temperature. It was found that the oven thermocouple was placed next to the heating element to hasten control response. However, convection was not sufficiently strong for achieving quick temperature uniformity, causing a bias in the oven thermocouple due to the local temperature increase from the heating element. Hence, corrective measures were taken.

Corrective measures

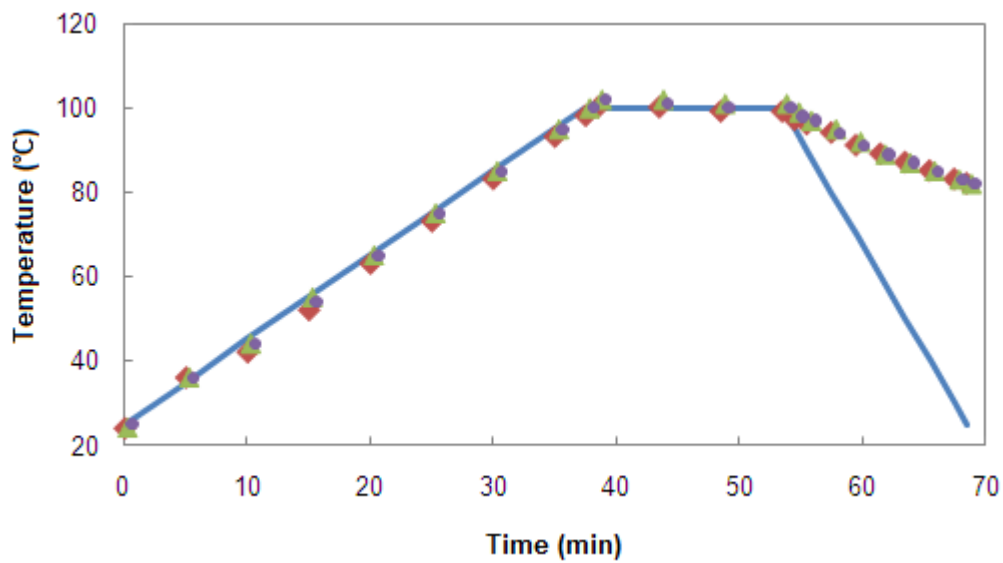
The issue of control of the oven was mitigated by relocating the thermocouple. Instead of being positioned next to the heating element, the thermocouple was moved inside the chamber, as far as the length of the wire permitted it, which is near the middle of the bottom of the oven chamber. A test was performed for targeting a 2°C/min heating rate with the cooling vent closed. The results are presented in Figure A.2 where it can be seen that both the oven thermocouple and the standalone thermocouple recorded similar temperatures. More importantly, the chamber easily reached the target temperature. As before, there was still a 1 minute delay between the target temperatures and oven temperature reading, but this can be accounted for in the dwell phase. The biggest limiting factor was a 1-2°C overshoot above the target temperature when reaching dwell, due to the oven thermocouple being farther to the heater which yielded a slower control response. Nevertheless, this is marginal compared to the gain in the chamber temperature accuracy.

Additionally, during the same test the oven was checked for temperature uniformity by comparing temperature measurements from 3 thermocouples that were positioned at different heights in the oven chamber. A thermocouple was placed 10cm from the top, another at 10cm from the bottom, and the last one in the middle. Measured temperatures appear in Figure A.3. The difference in temperature between the thermocouple lies between 1°C and 2°C, with the middle thermocouple being the warmest, and the top thermocouple being the coldest. A possible explanation is that the middle one is closer to the forced air flow from the heater whereas the bottom thermocouple is near the return vent of the chamber, leaving the top thermocouple slightly cooler.



Legend: — Set point ◆ Oven thermocouple ▲ Chamber temperature

Figure A.2: Oven temperatures with relocated oven thermocouple



Legend: — Set Point ◆ Top ▲ Middle ● Bottom

Figure A.3: Oven temperatures at different heights in the oven chamber

Appendix B: Classical bending theory for multilayered materials

The classical beam theory used in developing bending equations for multilayered materials was based on [144].

In three-point bending, a bending moment is generated by a force acting at mid-span of the specimen. Flexion of the specimen causes compression and tension. The average compressive and tensile stresses negate each other, resulting in the absence of axial forces.

$$\sum F_x = \int \sigma_x dA = 0 \quad (\text{A.1})$$

where the local stresses are equal to the modulus of elasticity of a material E multiplied by the axial strain ε_x :

$$\sigma_x = \varepsilon_x E \quad (\text{A.2})$$

In the classical bending theory, it is assumed that the material layers are bonded together and that everything remains in the elastic domain, implying absence of slippage. This means axial strain continuity at the material interface. Furthermore, it is assumed that the axial strain varies linearly across the thickness of the beam. In such case, axial strain is proportional to the axis curvature κ and to the distance y from the neutral axis of the beam:

$$\varepsilon_x = -\kappa y \quad (\text{A.3})$$

For convention of signs, a negative multiplier is used in the above equation. From Equations A.2 and A.3, Equation A.1 can be rearranged as:

$$\int \sigma_x dA = \int \varepsilon_x E dA = -\kappa \int y E dA = 0 \quad (\text{A.4})$$

In pure bending, the curvature is constant and can thus be eliminated from the equation, yielding:

$$\int y E dA = 0 \quad (\text{A.5})$$

Since the modulus of elasticity is constant within each distinct layer of material, the integration can be addressed separately for each layer, giving:

$$\int y E dA = \sum_{i=1}^n E_i \int y dA_i = 0 \quad (\text{A.6})$$

where the subscript i represents a specific material layer. In this last equation the integral can be recognised as the first moment of area, which by definition is:

$$\int y dA_i = \bar{y} A_i = y_i - y_n A_i \quad (\text{A.7})$$

where $\bar{y}$ is the distance from an axis y_n to the centroid y_i of an area A . If the integral equates to zero, it gives the location of a neutral axis. Rewriting Equation A.6 gives:

$$\sum_{i=1}^n E_i y_i - y_n A_i = 0 \quad (\text{A.8})$$

Finally, an expression for the neutral axis is obtained:

$$y_n = \frac{\sum_{i=1}^n E_i y_i A_i}{\sum_{i=1}^n E_i A_i} \quad (\text{A.9})$$

The next step for calculating the axial stresses in a composite beam is to evaluate the curvature (κ). This is achieved by looking at the externally applied and internally resisting moments that are at equilibrium. The bending moment M is obtained from the bending diagram, while the resisting moment inside the beam is caused by the compressive and tensile stresses.

$$M = - \int_A \sigma_x y dA \quad (\text{A.10})$$

Substituting Equations A.2 and A.3 gives:

$$M = \int_A \kappa y E y dA = \kappa \int_A E y^2 dA \quad (\text{A.11})$$

Similarly to the neutral axis equation, the modulus can be pulled out of the integral for each distinct material layer.

$$M = \kappa \sum_{i=1}^n E_i \int_A y^2 dA_i \quad (\text{A.12})$$

The remaining integral corresponds to the second moment of area I along the bending axis:

$$I = \int_A y^2 dA \quad (\text{A.13})$$

For a rectangular beam, or for uniform layers like laminates and coatings, the second moment of area is:

$$I = \frac{bh^3}{12} + Ad^2 \quad (\text{A.14})$$

where the first term is the second moment of area of the section upon its own centroid, with beam width b and thickness h . The latter term is an adjustment term for the parallel axis theorem required for any offset d of the centroid of a section area A to the neutral axis.

It follows that Equation A.12 can be rewritten as:

$$M = \kappa \sum_{i=1}^n E_i I_i \quad (\text{A.15})$$

where $\sum_{i=1}^n E_i I_i$ is the structural rigidity of the beam. The equation can be rearranged with curvature as the dependent variable, yielding:

$$\kappa = \frac{M}{\sum_{i=1}^n E_i I_i} \quad (\text{A.16})$$

Finally, from Equations A.2, A.3 and A.16, a general expression for the internal stresses of a composite beam is obtained:

$$\sigma_x = -\frac{M}{\sum_{i=1}^n E_i I_i} y E_i \quad (\text{A.17})$$

For CPMCs, the weakest region is presumably the coating-to-co-cured layer interface. Depending on the actual media and their thickness, the weak interface may be near the neutral axis where bending stresses vanish. However, there will be shear stresses that can induce failure. If an external force is applied on the beam, an internal resisting force is generated that changes the bending moment of the beam. By definition the relation of the variation of bending moment is the integral of the shear force V times a certain beam length dx .

$$dM = Vdx \quad (A.18)$$

This change in bending moment produces different bending stresses across a beam element of length dx , resulting in shear flow.

The shear flow is obtained by taking the difference in forces acting on a beam section. The force for a single side of an element is simply the sum of the stresses multiplied by the area:

$$F = \int_A \sigma_x dA \quad (A.19)$$

From Equation A.2, the force equation for a composite beam is rearranged into:

$$F = -\frac{M}{\sum_{i=1}^n E_i I_i} \int_A y E_i dA \quad (A.20)$$

Taking the difference in axial force across a beam element dx gives the shear per beam length:

$$\frac{dF}{dx} = -\frac{dM}{dx} \frac{\int_A y E_i dA}{\sum_{i=1}^n E_i I_i} \quad (A.21)$$

From Equation A.18, the bending moment can be replaced with the shear:

$$\frac{dF}{dx} = -V \frac{\int_A y E_i dA}{\sum_{i=1}^n E_i I_i} \quad (\text{A.22})$$

Considering that the shear stresses act in a plane defined by the length of the beam and bending axis, the sheared area is simply the width b of the beam times the beam length dx :

$$\tau_{xy} = \frac{1}{b} \frac{dF}{dx} = -\frac{V}{b} \frac{\int_A y E_i dA}{\sum_{i=1}^n E_i I_i} \quad (\text{A.23})$$

For rectangular beams like those used in three-point bending, the width can be brought out of the integral, which simplifies the equation to:

$$\tau_{xy} = \frac{1}{b} \frac{dF}{dx} = -\frac{Vb}{b} \frac{\int E_i y dy}{\sum_{i=1}^n E_i I_i} = -V \frac{\sum_{i=1}^n \left[\int E_i y dy \right]_i}{\sum_{i=1}^n E_i I_i} \quad (\text{A.24})$$

The maximum shear stress is found at the neutral axis and is calculated by solving the previous equation from the surface of the composite beam, up to the neutral axis.

Appendix C: Short-span bending calculation program

The C++ program that was written to calculate the bending stresses and deflection is as follow:

```

/*****
/* Simon Baril-Gosselin 4281100
/* Department of Mechanical Engineering
/* University of Ottawa
/*
/* Bending_Calculations_sbg.c
/* Last Modified: January 2012
/*
/* The program calculates the bending stresses and shear
/* stresses in three -point bending at the mid-span location
*****/

#include <iostream>
#include <fstream>
#include <sstream>
#include <math.h>
using namespace std;

/*Declaration of Variables*/
#define F -500 //Specify the applied Force [N]
#define L 0.025 //Specify the span [m]
#define b 0.01212 //Specify the width [m]
#define step 0.00001 //Increments for integration [m]
#define LIN 3 //Number of Material Layers
int i;
double j;

double a1 = 0, a2 = 0; //Variables
double d = 0; //Deflection [mm]
double ds = 0; //Shear deflection [mm]
double db = 0; //Bending deflection [mm]
double EI = 0; //Flexural Rigidity, Sum of the Modulus x I [GPa-mm^4]
double kappa = 0; //Curvature
double M = 0; //Bending moment
double ProductTau = 0; //Product of shear stress [MPa^2]
double Sigma = 0; //Stress [MPa]
double Tau = 0; //Shear Stress [MPa]
double Thick = 0; //Total Thickness [m]
double TotalTau = 0; //Sum of ProductTau for each layer
double V = F/2.0; //Shear force [N]
double yneutral = 0; //Neutral Axis [m]

double E [LIN] = { 65.5, 10, 100}; //Modulus of Elasticity of materials (from bottom)[GPa] {
65.5, 10, 100}
double G [LIN] = { 5, 3.6, 46.8}; //Shear Modulus of the materials from teh bottom - Coated {
5, 3.6, 46.8} Al {26,26,26}
double y[LIN]; //Centroids from the bottom

```

```

double t[LIN] = {0.0024, 0.0003, 0.0022}; //Thickness of material layers from bottom [m] {0.0024,
0.0003, 0.0023}
double I[LIN]; //Second moment of Area [mm4]

int main()
{
    ofstream Info("Bending_Results.txt");

    /*Writing test info to file*/
    Info<<"Number of Layers:\t"<<LIN<<"\n";
    Info<<"Force:\t"<<F<<"\tN\n";
    Info<<"Increments:\t"<<step<<"\tm\n";

    for (i=0; i<LIN;i++){
        Info<<"E"<<i+1<<":\t"<<E[i]<<"\tGPa\n";
    }
    Info<<"\nSpan:\t"<<L<<"\tm\n";
    Info<<"Width:\t"<<b<<"\tm\n";
    Info<<"\n";
    for (i=0; i<LIN;i++){
        Info<<"Thickness"<<i+1<<":\t"<<t[i]<<"\tm\n";
    }

    /*Total Thickness*/
    for(i=0; i<LIN;i++){
        Thick = Thick + t[i];
    }

    cout<<"Total Thickness: \t\t"<<Thick<<" m\n\n";
    Info<<"Total Thickness: \t"<<Thick<<"\tm\n\n";

    /*Bending Moment*/
    M = (F*L)/4.0;

    cout<<"Shear Force: \t\t\t"<<V<<" N\n";
    cout<<"Bending Moment: \t\t"<<M<<" N-m\n\n";
    Info<<"Shear Force: \t"<<V<<"\tN\n";
    Info<<"Bending Moment: \t"<<M<<"\tN-m\n\n";

    /*Neutral Axis*/
    y[0] = t[0]/2.0;
    for(i=1; i<LIN;i++){
        y[i] = y[i-1] + ((t[i] + t[i-1])/2.0);
    }
    for(i=0; i<LIN;i++){
        cout<<"Centroid y"<<i+1<<" from the bottom is: \t"<<y[i]<<" m\n";;
        Info<<"Centroid y"<<i+1<<" from the bottom is: \t"<<y[i]<<"\tm\n";;
    }

    for (i=0; i<LIN; i++){
        a1 = a1 + (E[i] * y[i] * b * t[i]);
        a2 = a2 + (E[i] * b * t[i]);
    }

```

```

}
yneutral = a1/a2;

cout<<"\nNeutral Axis (from bottom):\t\t"<<yneutral<<" m\n\n";
Info<<"\nNeutral Axis (from bottom):\t"<<yneutral<<"\tmm\n\n";

/*Second Moment of Area*/
for(i=0; i<LIN; i++) {
    I[i] = b*((pow(t[i],3)/12.0) + (t[i] * (pow(y[i]-yneutral,2))))* 1000*1000*1000*1000;
    cout<<"Second moment of area I"<<i+1<<": \t\t"<<I[i]<<" mm^4\n";
    Info<<"Second moment of area I"<<i+1<<": \t"<<I[i]<<"\tmm^4\n";
}

/*Flexural Rigidity EI*/
for(i=0; i<LIN; i++) {
    EI=EI+(E[i]*I[i]);
}
cout<<"EI: \t\t\t\t\t"<<EI<<" GPa-mm^4\n\n";
Info<<"EI: \t"<<EI<<"\tGPa-mm^4\n\n";

/*Bending Stress*/
ofstream Output("Bending_Stress.txt");
Sigma = M * (0-yneutral) * E[0]/EI*1000*1000; //Sigma Min
cout<<"Tensile Stress:\t\t\t\t"<<Sigma<<" MPa\n";
cout<<"Tensile Strain:\t\t\t\t"<<Sigma/(E[0]*1000)<<" mm/mm\n\n";
Info<<"Tensile Stress:\t"<<Sigma<<"\tMPa\n";
Info<<"Tensile Strain:\t"<<Sigma/(E[0]*1000)<<"\tmm/mm\n\n";

a1 = 0;
for(i=0; i<LIN; i++) {
    for(j= (a1); j<= (((t[i]/2.0) + y[i])/step);j++){
        Sigma = M * ((j*step)-yneutral) * E[i]/EI*1000*1000;
        //cout<<"Sigma:\t"<<j<<"\t"<<Sigma<<"\n";
        Output<<j*step<<"\t"<<Sigma<<"\n";
    }
    a1 = (((t[i]/2.0) + y[i])/step);
}

Sigma = M * ((Thick)-yneutral) * E[LIN-1]/EI*1000*1000; //Sigma Max
cout<<"Compressive Stress:\t\t\t\t"<<Sigma<<" MPa\n";
cout<<"Compressive Strain:\t\t\t\t"<<Sigma/(E[2]*1000)<<" mm/mm\n\n";
Info<<"Compressive Stress:\t"<<Sigma<<"\tMPa\n";
Info<<"Compressive Strain:\t"<<Sigma/(E[2]*1000)<<"\tmm/mm\n\n";

Output<<Thick<<"\t"<<Sigma<<"\n";
Output.close();

/*Shear Stress*/
a1=0;
ofstream Output2("Shear_Stress.txt");
for(i=0; i<LIN; i++) {
    ProductTau = 0;
    for(j= (a1); j<= (((t[i]/2.0) + y[i])/step);j++){
        Tau = Tau-(V * ((j*step)-yneutral)*step * E[i]/EI*1000*1000);
    }
}

```

```

        //cout<<"Sigma:\t"<<j<<"\t"<<Sigma<<"\n";
        Output2<<j*step<<"\t"<<Tau<<"\n";
        ProductTau = ProductTau + (Tau*Tau*step*1000.0*b*1000.0)/(1000.0*G[i]*2.0);
    }
    if (i<2){
        cout<<"Shear stress at "<<i+1<<"th interface is:\t"<<Tau<<" MPa\n";
        Info<<"Shear stress at "<<i+1<<"th interface is:\t"<<Tau<<"\tMPa\n";
    }
    TotalTau = TotalTau + ProductTau;
    a1 = (((t[i]/2.0) + y[i])/step);
}

Tau = Tau-(V * ((Thick)-yneutral)*step * E[LIN-1]/EI*1000);
//cout<<"Tau Compression:\t"<<Thick<<"\t"<<Tau<<"\n";
Output2<<Thick<<"\t"<<Tau<<"\n\n";
Output2.close();

/*Deflection*/

/*Bending Deflection*/
db = F*pow(L*1000,3)/(48000*EI);
cout<<"\nBending Deflection:\t\t\t"<<db<<" mm\n";
Info<<"\nBending Deflection:\t"<<db<<"\tmm\n";

/*Shear Deflection*/
ds = TotalTau*L*1000*2/F;
cout<<"\nShear Deflection:\t\t\t"<<ds<<" mm\n";
Info<<"\nShear Deflection:\t"<<ds<<"\tmm\n";

/*Total Deflection*/
d = db+ds;
cout<<"\nTotal Deflection:\t\t\t"<<d<<" mm\n";
Info<<"\nTotal Deflection:\t"<<d<<"\tmm\n";

/*Kappa*/
kappa = -M/EI;
cout<<"\nKappa: \t\t\t\t\t"<<kappa<<" mm^-1\n";
cout<<"Ratio: \t\t\t\t\t"<<pow(b*1000,2)*kappa/(Thick*1000)<<"\n";

Info.close();

system("PAUSE");
return (0);

```

Appendix D: Heater controller UDF for FLUENT

```
/******  
/* Simon Baril-Gosselin */  
/* Department of Mechanical Engineering */  
/* University of Ottawa */  
/* */  
/* Heater_UDF_Pretial Series */  
/* Last Modified: April 2010 */  
/* */  
/* This program controls a user defined function in simulation software */  
/* Fluent. It simulates the control of a heater based on a ON/OFF */  
/* process for ramp and dwell profiles from the feedback of thermocouples. */  
/* CTC Control Thermocouple */  
/* OHTC Over Heat Thermocouple */  
/******  
  
#include <udf.h>  
#include <mem.h>  
  
//Declaration of the Global Variables  
static Thread *CTC_Thread; //Pointer for the thread of the Thermocouple CTC  
static Thread *OHTC_Thread; //Pointer for the thread of the Thermocouple OHTC  
static face_t CTC_Face; //Pointer for the face ID of CTC  
static face_t OHTC_Face; //Pointer for the face ID of OHTC  
  
real TC_T0, TC_T1; //Temporal events  
real TC_Temp0, TC_Temp1; //Actual temperature of the TC  
real source; //Return variable of the heat source  
real Target_T0, Target_T1; //Targeted temperatures of the mould  
real CTC_Face_ID; //ID of the closest face to the CTC  
real OHTC_Face_ID; //ID of the closest face to the OHTC  
int State; //State of the heater (ON/OFF) (1/0)  
  
/*Function to initialize the system*/  
DEFINE_INIT(Acquire_CTC_Location,d)  
{  
  
//Declaration of the Local Variables  
#define CTC_x 0.13 //Coordinate of the CTC on the x-axis  
#define CTC_y 0.0 //Coordinate of the CTC on the y-axis  
#define CTC_z 0.15 //Coordinate of the CTC on the z-axis  
#define OHTC_x 0.15 //Coordinate of the OHTC on the x-axis  
#define OHTC_y 0.002 //Coordinate of the OHTC on the y-axis  
#define OHTC_z 0.15 //Coordinate of the OHTC on the z-axis  
#define CTC_ID 6 //Thread ID of the surface on which the CTC lies  
#define OHTC_ID 1 //Thread ID of the surface on which the OHTC lies  
  
int i; //Counter  
real Coordinates[ND_ND]; //Table of coordinates for the faces (x,y,z)  
real Min_Distance=256.0; //Minimum distance between the CTC and a face (Initialization at  
256.0m)  
real Delta_x; //Distance in the x-axis between the CTC and a face  
real Delta_y; //Distance in the y-axis between the CTC and a face  
real Delta_z; //Distance in the z-axis between the CTC and a face
```

```

real Distance;           //Absolute distance between the CTR and a face
real Coord_x;           //Coordinates in the x-axis of the face closest to the thermocouple
real Coord_y;           //Coordinates in the y-axis of the face closest to the thermocouple
real Coord_z;           //Coordinates in the z-axis of the face closest to the thermocouple
real Zone_ID;           //Thread ID of the surface on which the CTC lies
real Pos_x;             //Thermocouple Location on the x-axis
real Pos_y;             //Thermocouple Location on the y-axis
real Pos_z;             //Thermocouple Location on the z-axis

//Initialization of the Thread
CTC_Thread = Lookup_Thread(d,CTC_ID);    //Finds the proper information for the thread of
CTC
OHTC_Thread = Lookup_Thread(d,OHTC_ID);  //Finds the proper information for the thread
of CTC

//Finding the closest element to the CTC
begin_f_loop(CTC_Face,CTC_Thread){      //Loops over faces in a face thread
    F_CENTROID (Coordinates,CTC_Face,CTC_Thread);

    Delta_x = Coordinates[0]-CTC_x;
    Delta_y = Coordinates[1]-CTC_y;
    Delta_z = Coordinates[2]-CTC_z;

    Delta_x = pow(Delta_x,2.0);          //Squared value of Delta_x
    Delta_y = pow(Delta_y,2.0);          //Squared value of Delta_y
    Delta_z = pow(Delta_z,2.0);          //Squared value of Delta_z

    Distance = pow(Delta_x + Delta_y + Delta_z,0.5);

    if (Distance < Min_Distance){
        Min_Distance = Distance;
        CTC_Face_ID = CTC_Face;
    }
    //Message("Coordinates of CTC: %g/%g/%g \t Distance: %g \n", Coordinates[0],
Coordinates[1], Coordinates[2], Distance);

}
end_f_loop(CTC_Face,CTC_Thread);
CTC_Face = CTC_Face_ID;

Distance = 256;           //Re-initialize value
Min_Distance=256;        //Re-initialize value

//Finding the closest element to the OHTC
begin_f_loop(OHTC_Face,OHTC_Thread){    //Loops over faces in a face thread
    F_CENTROID (Coordinates,OHTC_Face,OHTC_Thread);

    Delta_x = Coordinates[0]-OHTC_x;
    Delta_y = Coordinates[1]-OHTC_y;
    Delta_z = Coordinates[2]-OHTC_z;

    Delta_x = pow(Delta_x,2.0);          //Squared value of Delta_x
    Delta_y = pow(Delta_y,2.0);          //Squared value of Delta_y
    Delta_z = pow(Delta_z,2.0);          //Squared value of Delta_z

    Distance = pow(Delta_x + Delta_y + Delta_z,0.5);

```

```

        if (Distance < Min_Distance){
            Min_Distance = Distance;
            OHTC_Face_ID = OHTC_Face;
        }
        //Message("Coordinates of CTC: %g/%g/%g \t Distance: %g \n", Coordinates[0],
Coordinates[1], Coordinates[2], Distance);

    }
    end_f_loop(OHTC_Face,OHTC_Thread);
    OHTC_Face = OHTC_Face_ID;

//Verification of the coordinates

F_CENTROID (Coordinates,CTC_Face,CTC_Thread);
Message("Coordinates of CTC: %g/%g/%g", Coordinates[0], Coordinates[1], Coordinates[2]);

F_CENTROID (Coordinates,OHTC_Face,OHTC_Thread);
Message("Coordinates of CTC: %g/%g/%g", Coordinates[0], Coordinates[1], Coordinates[2]);

}

/*Procedure to control the operation of the heater*/
DEFINE_SOURCE(heater_source_udf,cell,thread,dS,index)
{

//Declaration of the local variables
#define a1 0.0333          //Heating rate [K/s] for the profile A of the cure cycle
#define a2 0.0            //Heating rate [K/s] for the profile B of the cure cycle
#define a3 -0.0833        //Heating rate [K/s] for the profile C of the cure cycle
#define b1 298.0          //Initial temperature [K] for the profile A of the cure cycle
#define b2 395.0          //Initial temperature [K] for the profile B of the cure cycle
#define b3 787.5          //Temperature constant [K] for the profile C of the cure cycle
#define Power_Heater 56000000.0 //Heat density of the heater (Wm-3)
#define Time_A 2910.0     //Time [s] of the heating ramp of the cure cycle (~+1K/min to 373K)
#define Time_B 4710.0     //Time [s] of the steady state of the cure cycle (2hrs)
#define Time_C 5874.0     //Time [s] of the cooling ramp of the cure cycle (~-5K/min to 298K)
#define Threshold_High 1.0 //Upper boundary of the threshold (K)
#define Threshold_Low 1.0  //Lower boundary of the threshold (K)
#define Max_Heater_Temp 473 //Maximum operating temperature of the heater

//Initialization of the variables
TC_T0 = PREVIOUS_TIME;
TC_T1 = CURRENT_TIME;

//Evaluation of the targeted values
if(TC_T1 <= Time_A){                //Heating profile 1
    //Target_T0 = (a1*CTC_T0)+b1;
    Target_T1 = (a1*TC_T1)+b1;
}

else if( TC_T1 > Time_A && TC_T1 <= Time_B){    //Heating profile 2
    //Target_T0 = (a2*CTC_T0)+b2;
    Target_T1 = (a2*TC_T1)+b2;
}

```

```

else if( TC_T1 > Time_B && TC_T1 <= Time_C){    //Heating profile 3
    //Target_T0 = (a3*CTC_T0)+b3;
    Target_T1 = (a3*TC_T1)+b3;
}

else{                                           //Elsewise
    //Target_T0 = b1;
    Target_T1 = b1;
}

//Finding the actual temperature of the CTC

    //CTC_Temp0 = F_T_M1(CTC_Face,CTC_Thread);    //Temperature at the previous
time
    TC_Temp1 = F_T(CTC_Face,CTC_Thread);          //Temperature at the current time

//Determining the activity of the heater from the CTC
if(TC_T1 <= 10)    //Heater always on at the beginning
    State = 1;
else{
    if(TC_Temp1 > (Target_T1 + Threshold_High))
        State = 0;    //Above the threshold
    else if(TC_Temp1 < (Target_T1 - Threshold_Low))
        State = 1;    //Above the threshold
}

//Preveting the temperature overload of the heater
TC_Temp1 = F_T(OHTC_Face,OHTC_Thread);          //Temperature at the current time

if(TC_Temp1 > Max_Heater_Temp)    //Ensuring that the temperature is below limit of
the heater
    State = 0;

//Heater power
source = State * Power_Heater;

return source;

}
DEFINE_EXECUTE_AT_END(Print_values)
/*Runs at the end of every time step. Lines 1, 2, 3 and 5 creates an output file to print various useful
variables for debugging.
Line 4 assigns the temperature of CTC at the current time step to variable CTC_T0 for use at the
next time step.*/
{
    FILE *fout=fopen("Trial9_output.txt", "w");
    fprintf(fout, "Time %gs %g %g %d\n", TC_T1, Target_T1, TC_Temp1, State);
    fclose(fout);
    //CTC_T0=CTC_T1;
    Message("Time %g %g %g %d\n", TC_T1, Target_T1, TC_Temp1, State);
}

```

Appendix E: Force-displacement curves for short-span bending

Coatings in axial compression

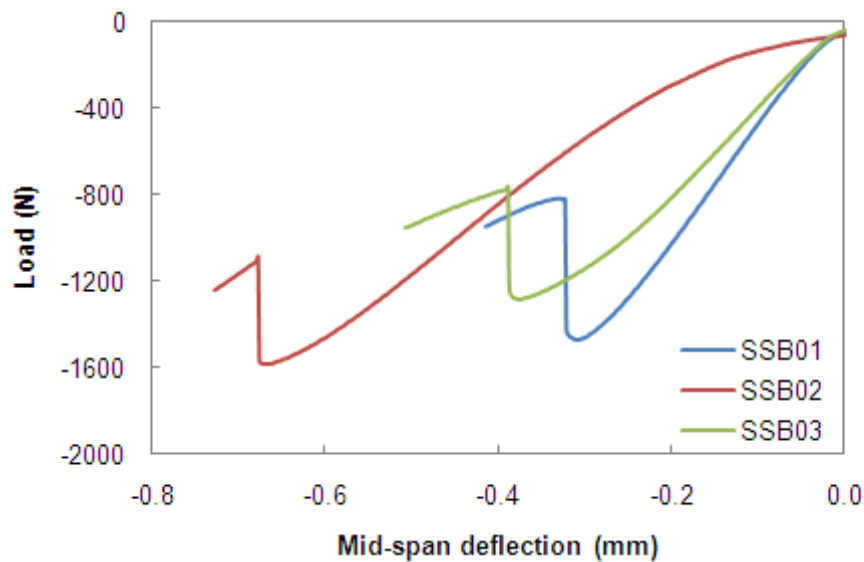


Figure A.4: Force-displacement curves of Cu-105-3 at 25°C for SSB01, SSB02 and SSB03

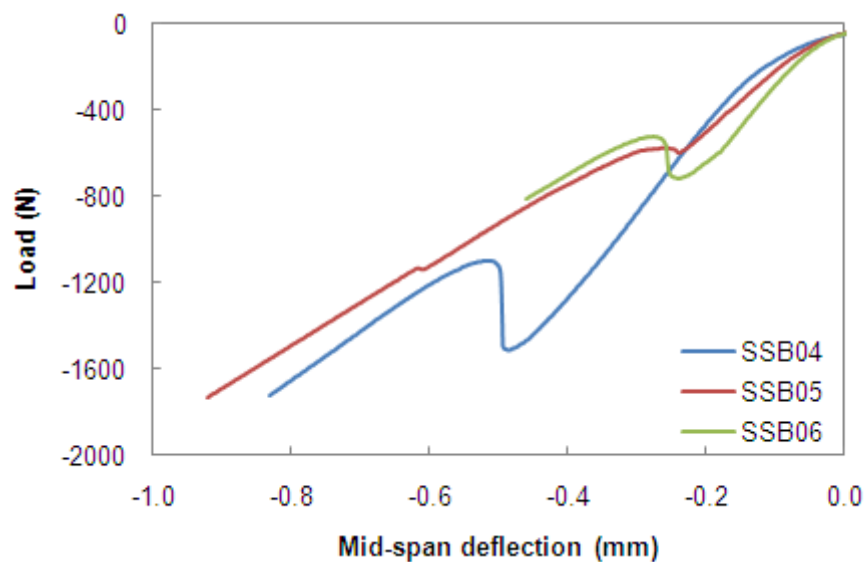


Figure A.5: Force-displacement curves of SST-C5003 at 25°C for SSB04, SSB05 and SSB06

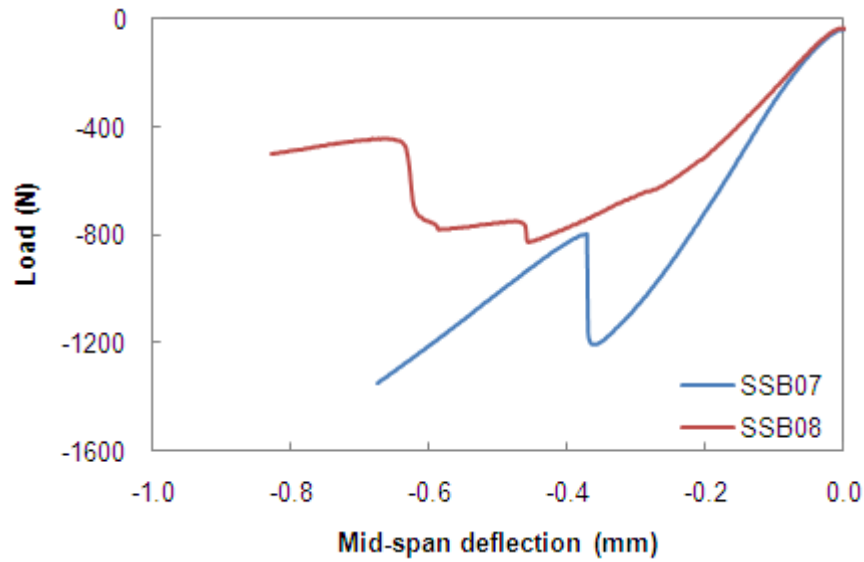


Figure A.6: Force-displacement curves of SST-C5003 at 70°C and 120°C for SSB07 and SSB08

Coating in axial tension

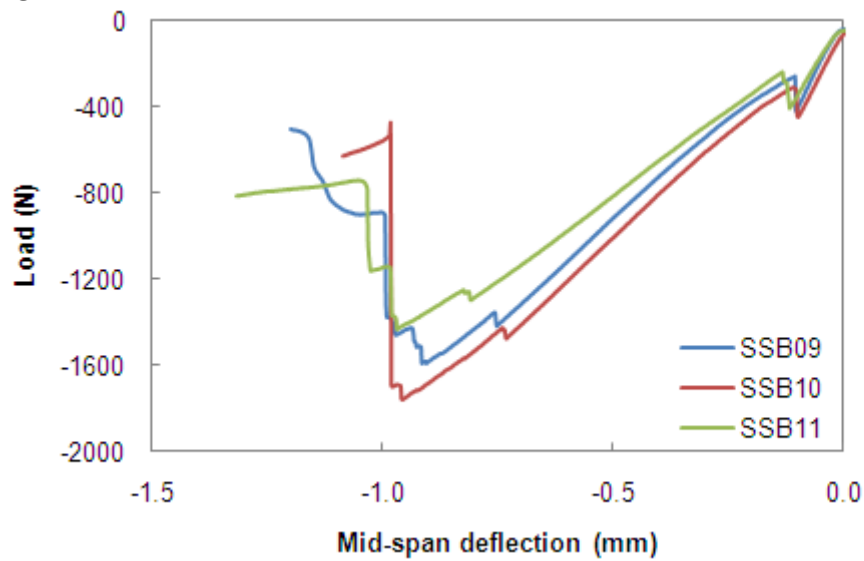


Figure A.7: Force-displacement curves of Cu-105-3 at 25°C for SSB09, SSB10 and SSB11

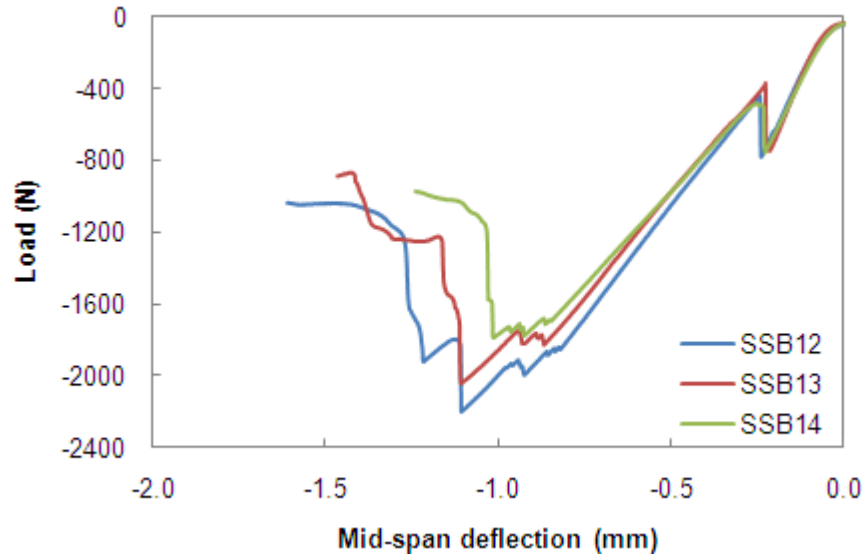


Figure A.8: Force-displacement curves of SST-C5003 at 25°C for SSB12, SSB13 and SSB14

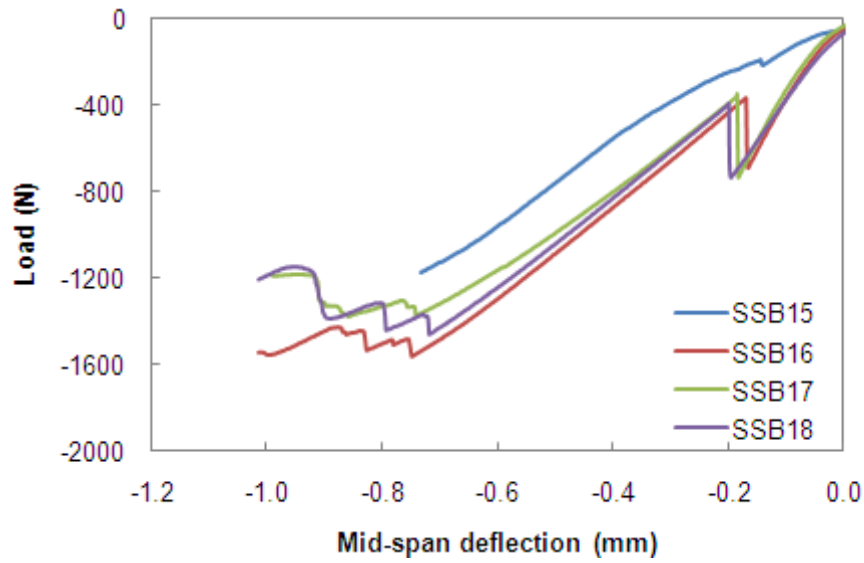


Figure A.9: Force-displacement curves of SST-C5003 at 70°C for SSB15, SSB16, SSB17 and SSB18

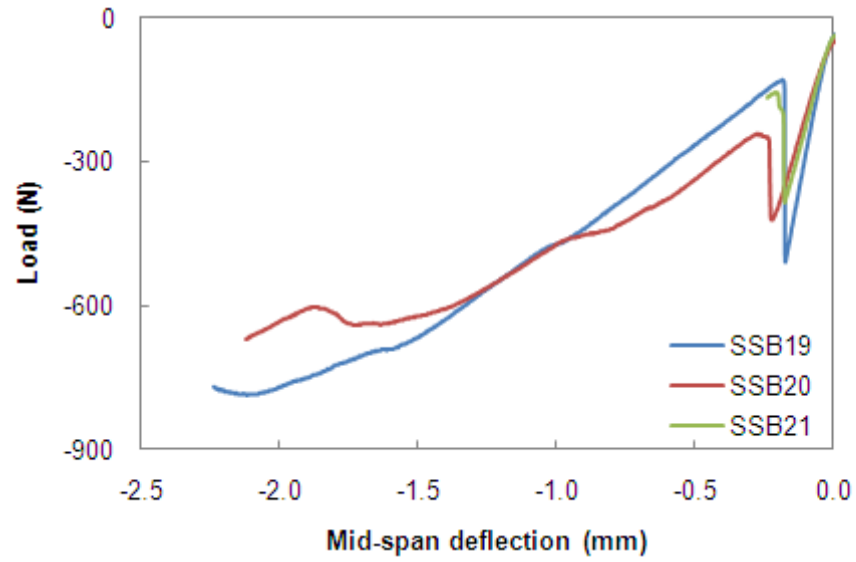


Figure A.10: Force-displacement curves of SST-C5003 at 120°C for SSB19, SSB20 and SSB21

Appendix F: Axial and shear stresses in CPMCs

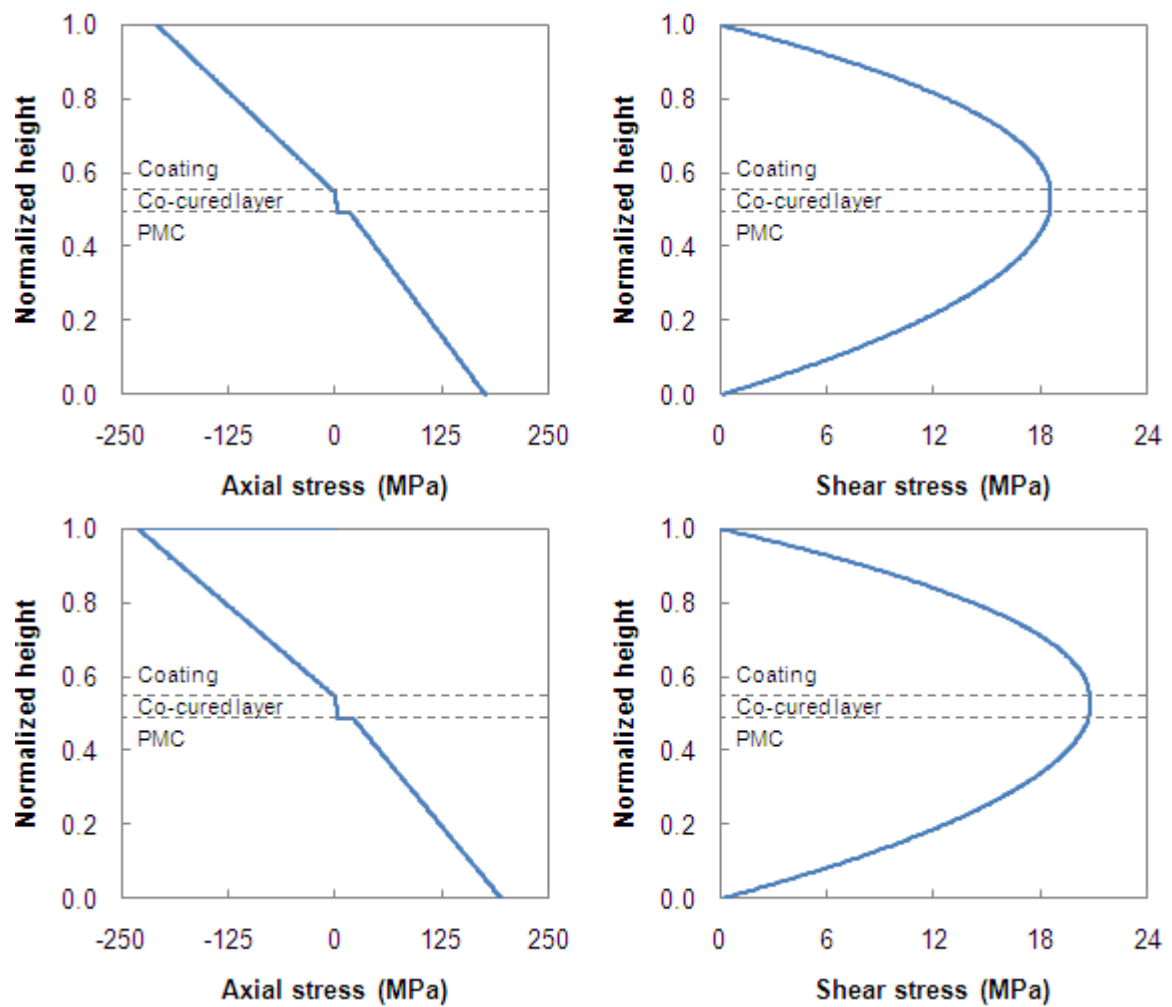


Figure A.11: Axial and shear stress curves at failure for SSB01 (top) and SSB02 (bottom)

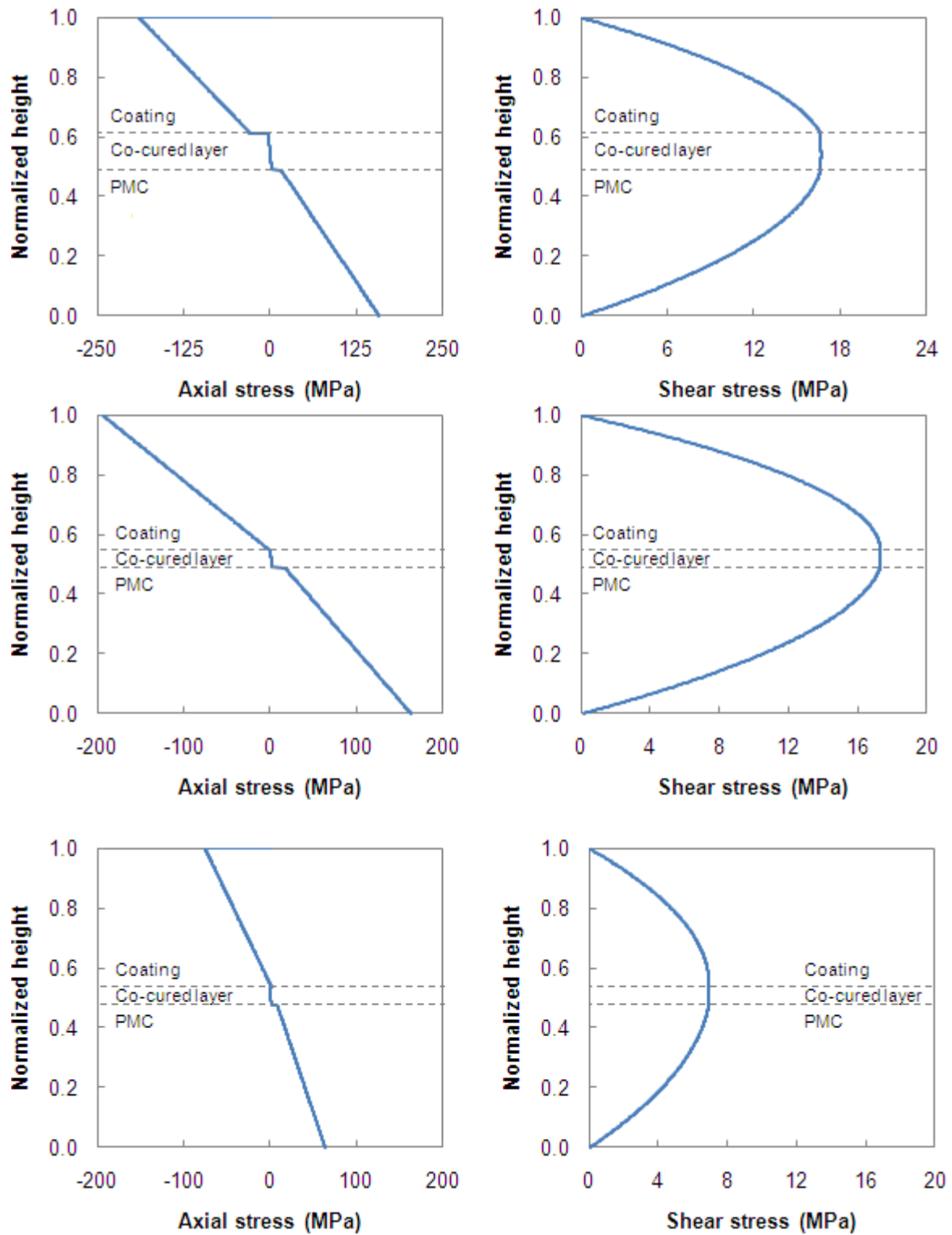


Figure A.12: Axial and shear stress curves at failure for SSB03 (top), SSB04 (middle) and SSB05 (bottom)

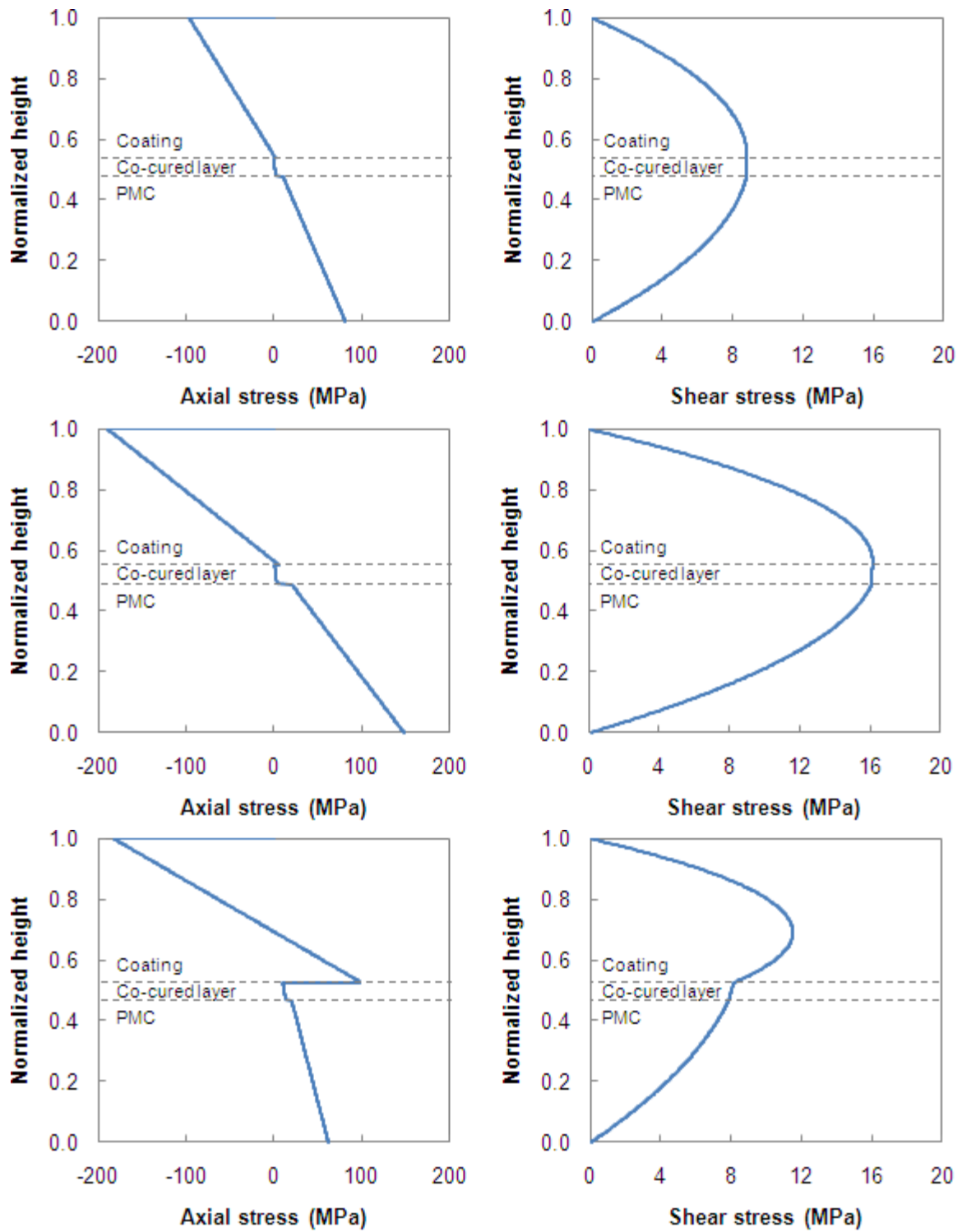


Figure A.13: Axial and shear stress curves at failure for SSB06 (top), SSB07 (middle) and SSB08 (bottom)

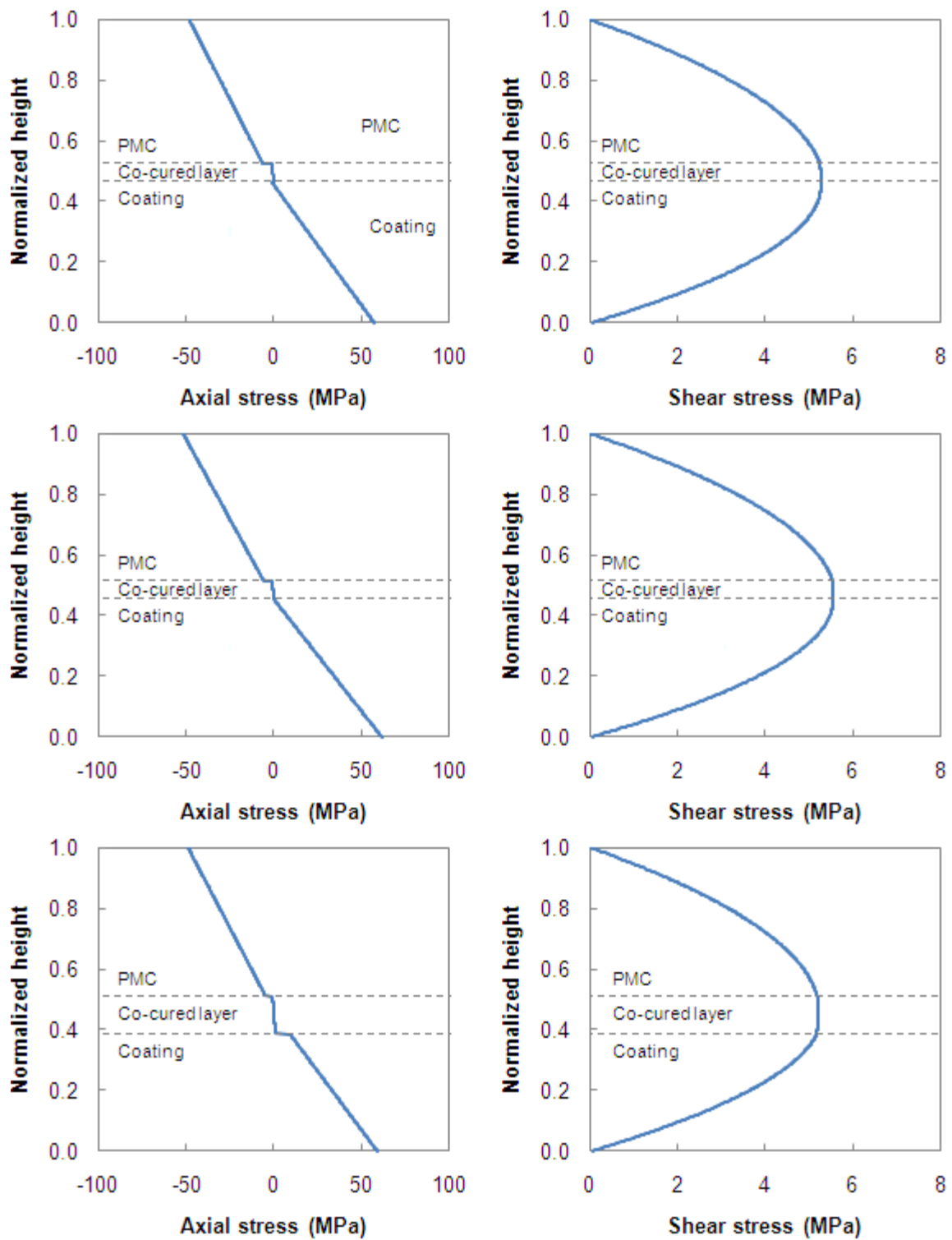


Figure A.14: Axial and shear stress curves at failure for SSB09 (top), SSB10 (middle) and SSB11 (bottom)

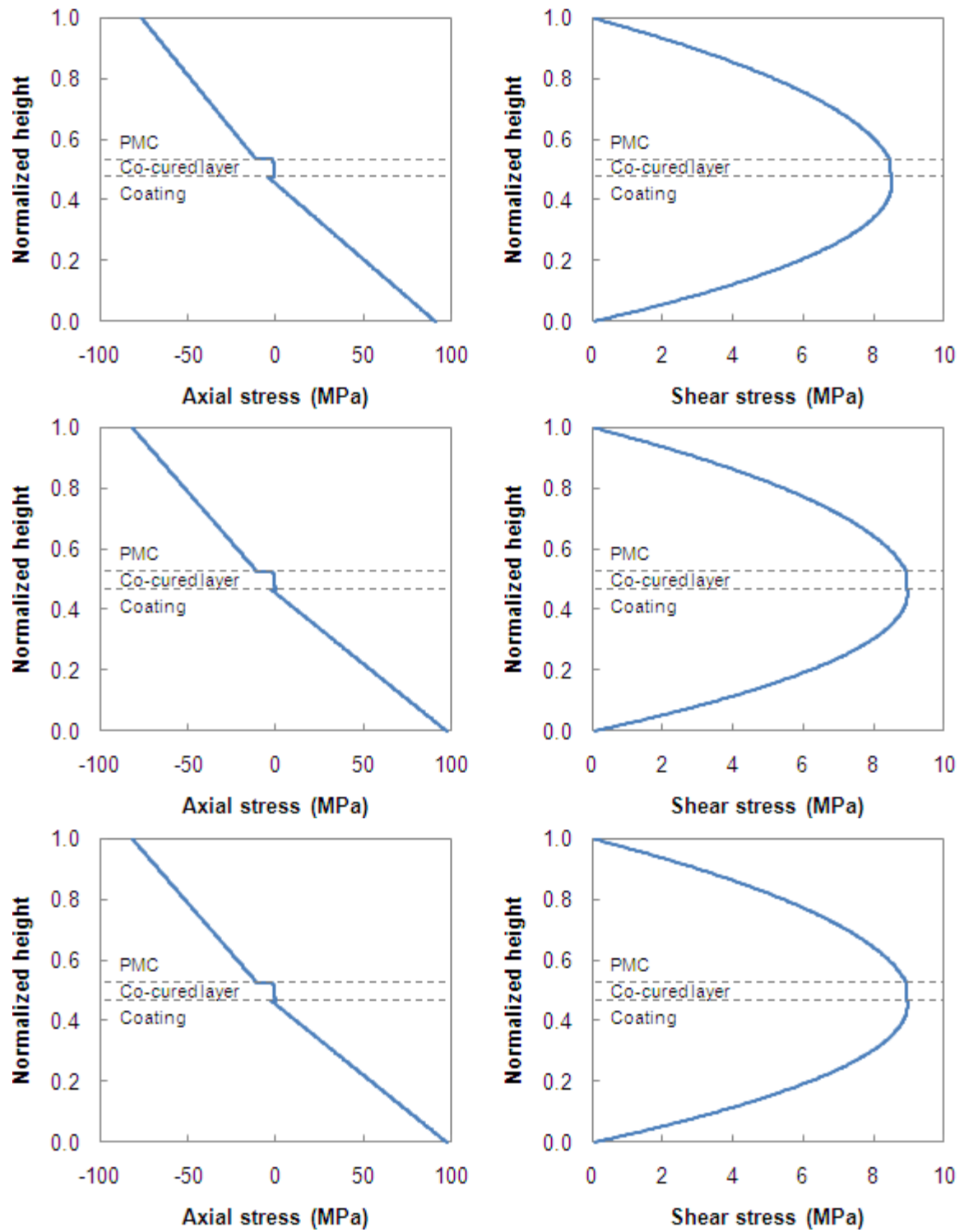


Figure A.15: Axial and shear stress curves at failure for SSB12 (top), SSB13 (middle) and SSB14 (bottom)

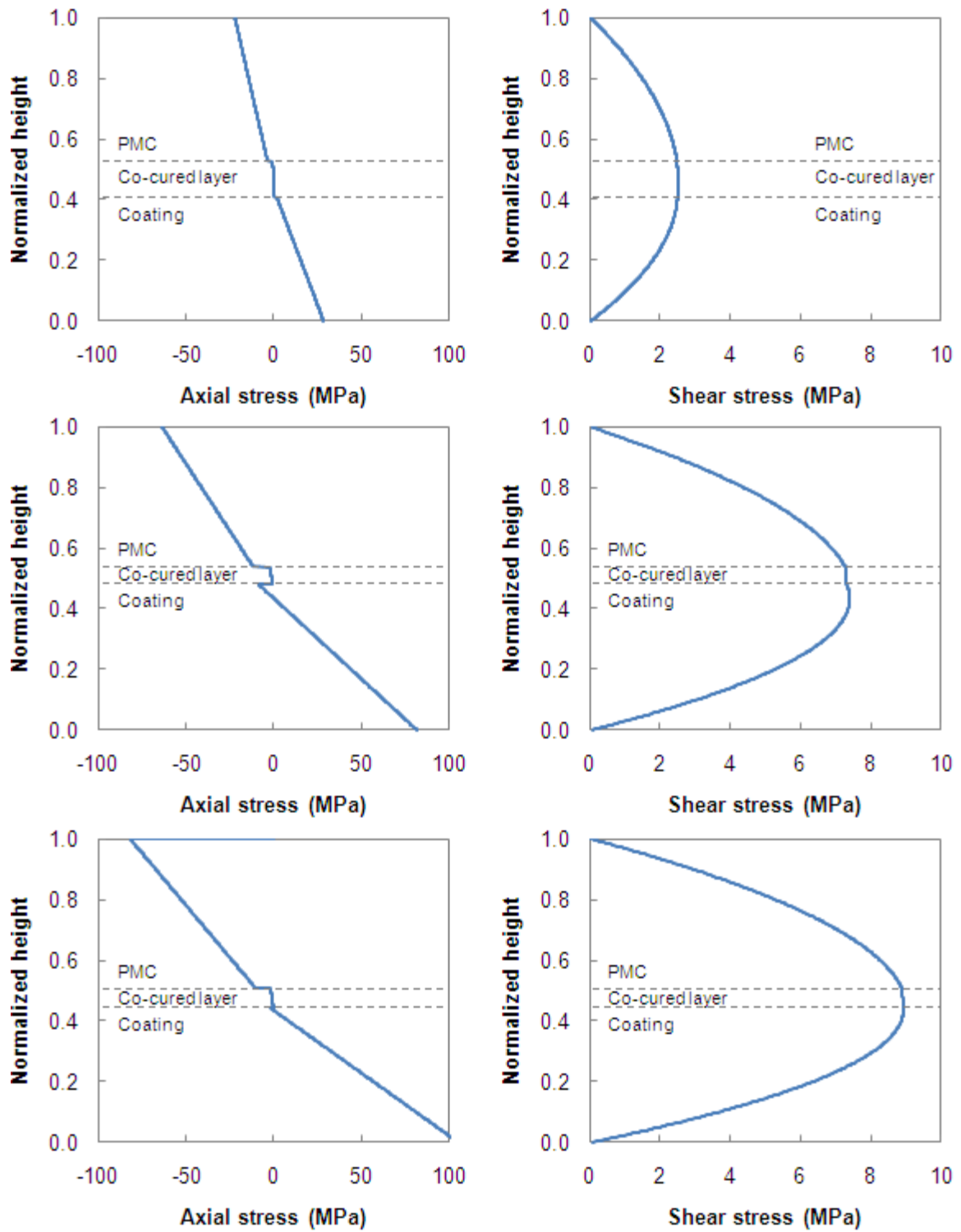


Figure A.16: Axial and shear stress curves at failure for SSB15 (top), SSB16 (middle) and SSB17 (bottom)

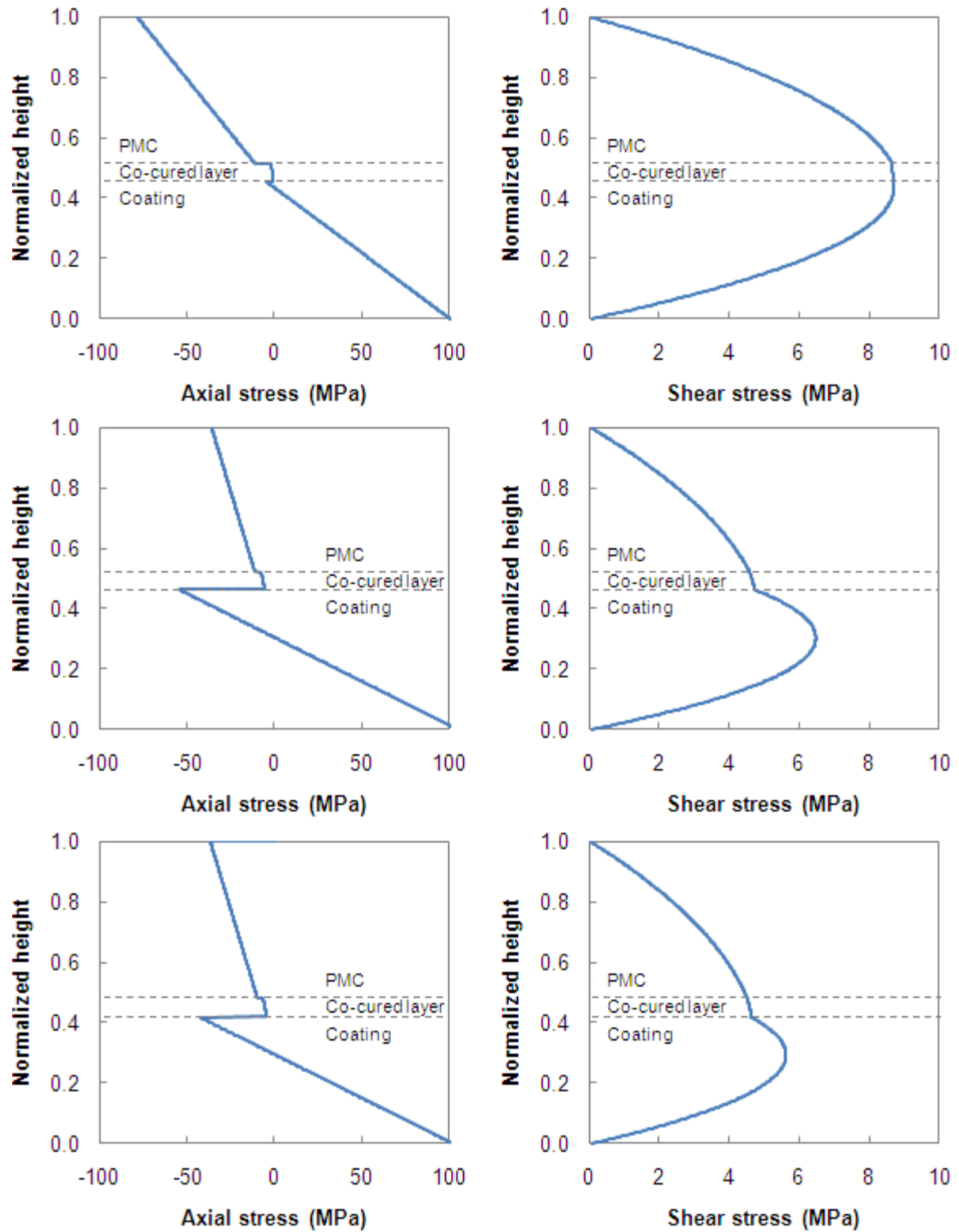


Figure A.17: Axial and shear stress curves at failure for SSB18 (top), SSB19 (middle) and SSB20 (bottom)

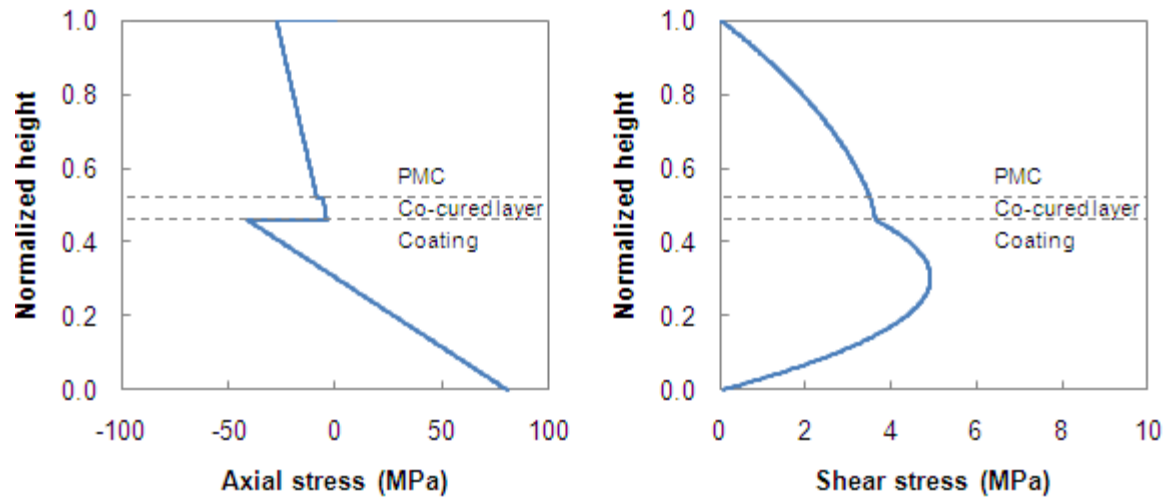


Figure A.18: Axial and shear stress curves at failure for SSB21

Appendix G: Low frequency cycling curves

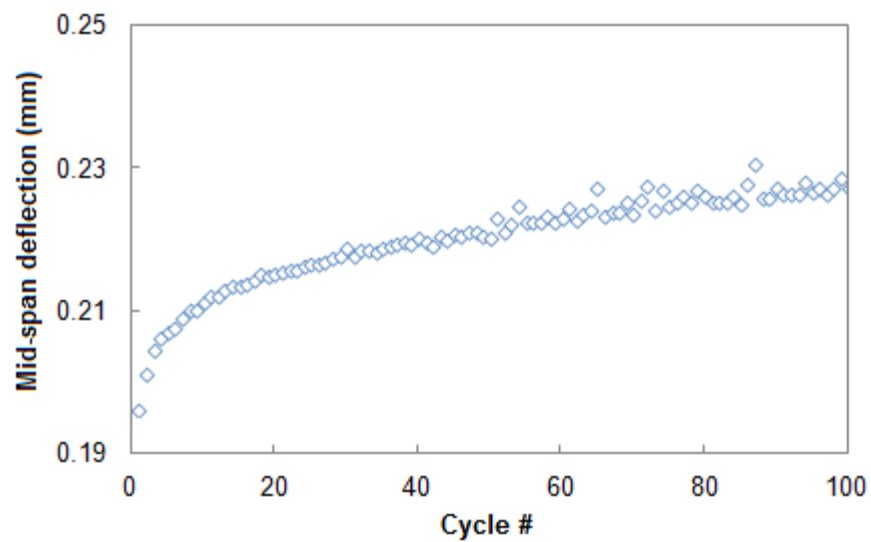


Figure A.19: Mid-span deflection of LFC01 during cycling

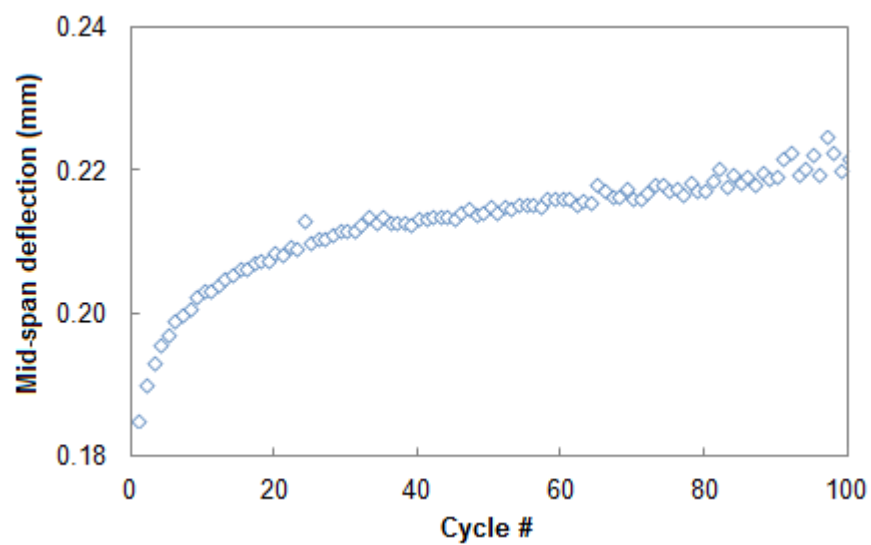


Figure A.20: Mid-span deflection of LFC02 during cycling

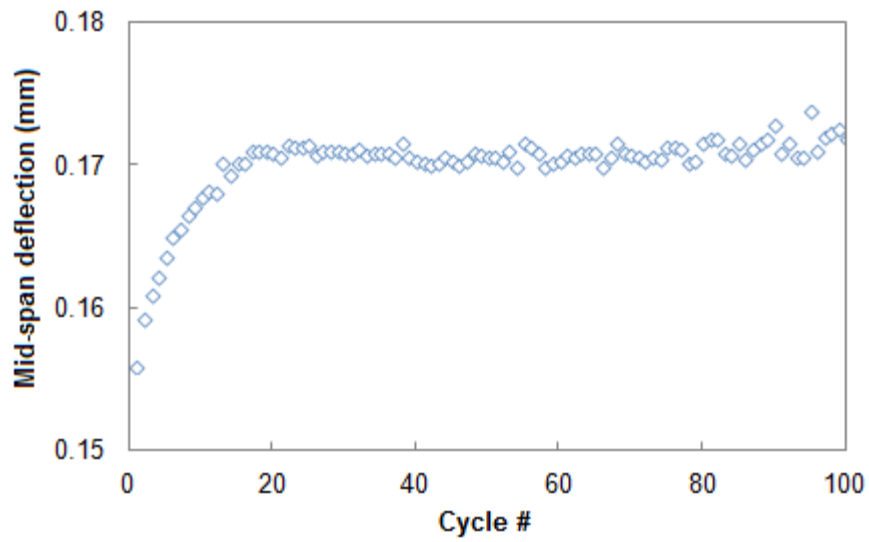


Figure A.21: Mid-span deflection of LFC03 during cycling

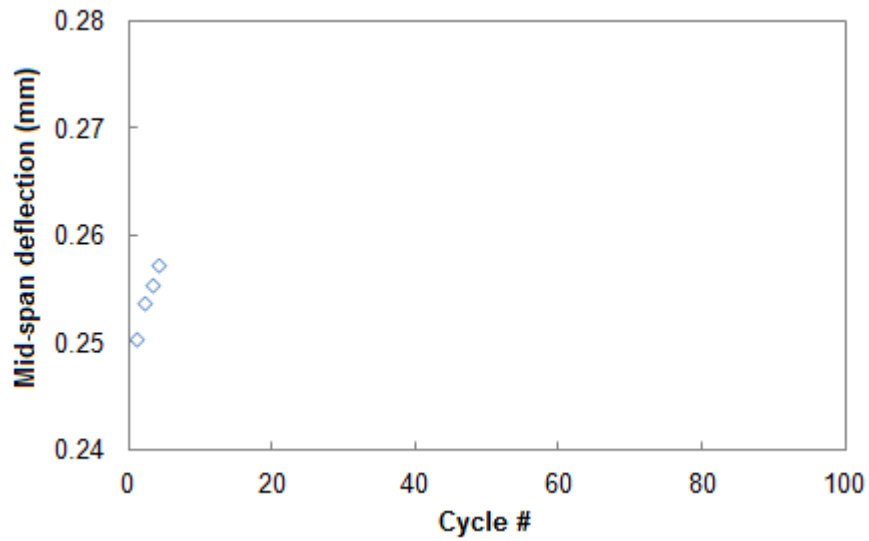


Figure A.22: Mid-span deflection of LFC04 during cycling

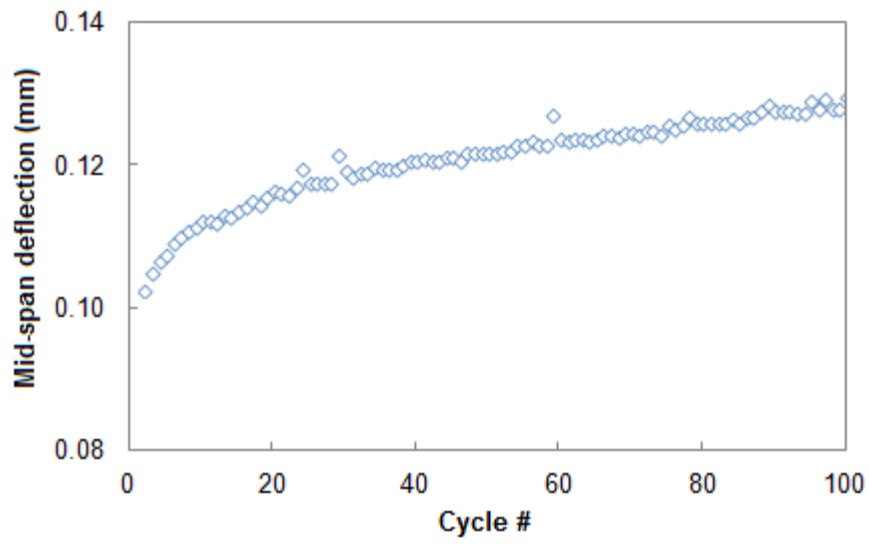


Figure A.23: Mid-span deflection of LFC05 during cycling

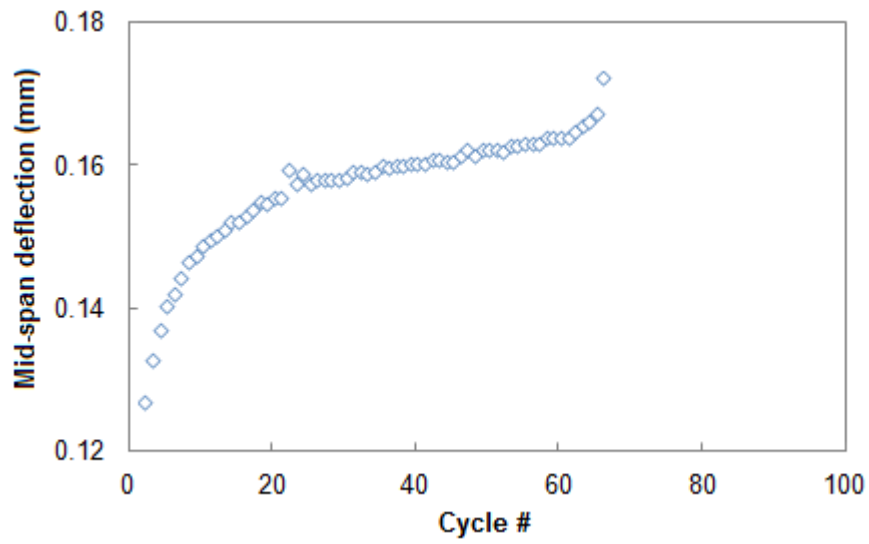


Figure A.24: Mid-span deflection of LFC06 during cycling

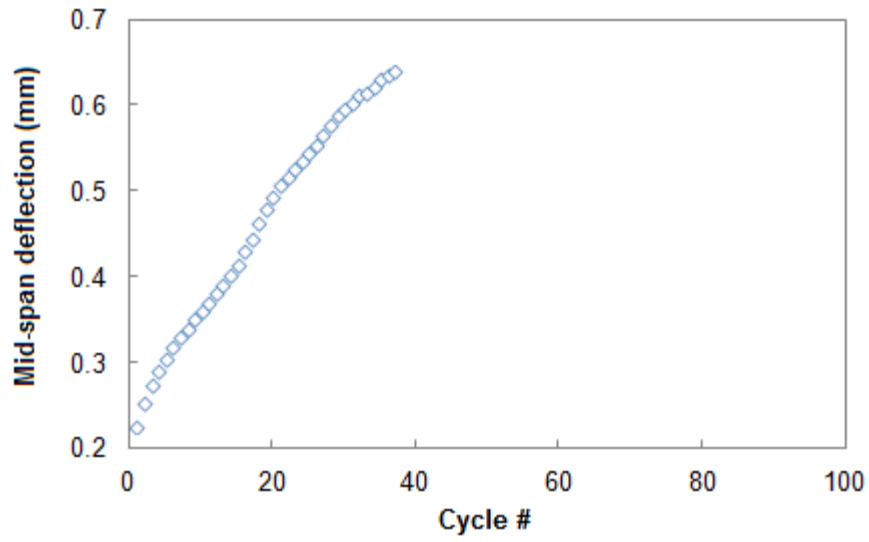


Figure A.25: Mid-span deflection of LFC07 during cycling

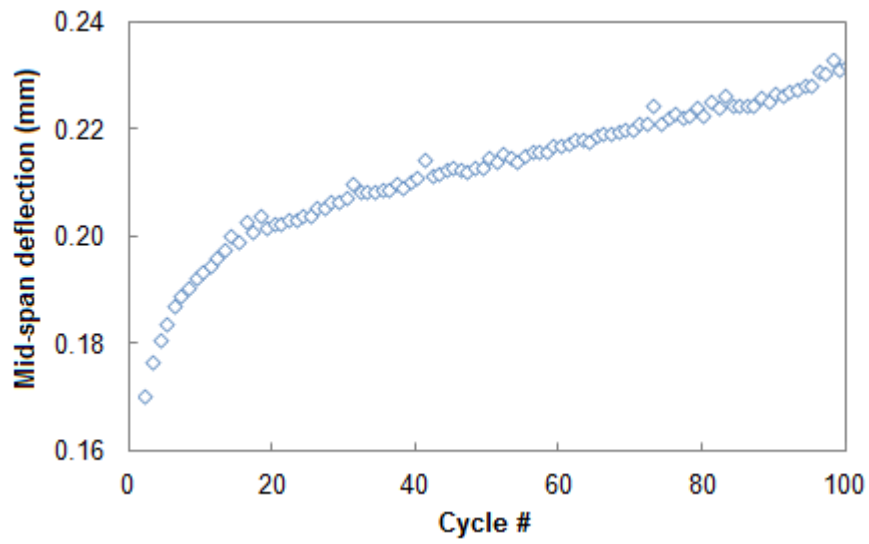


Figure A.26: Mid-span deflection of LFC08 during cycling

Appendix H: Temperature distribution across IHCT-CPMC

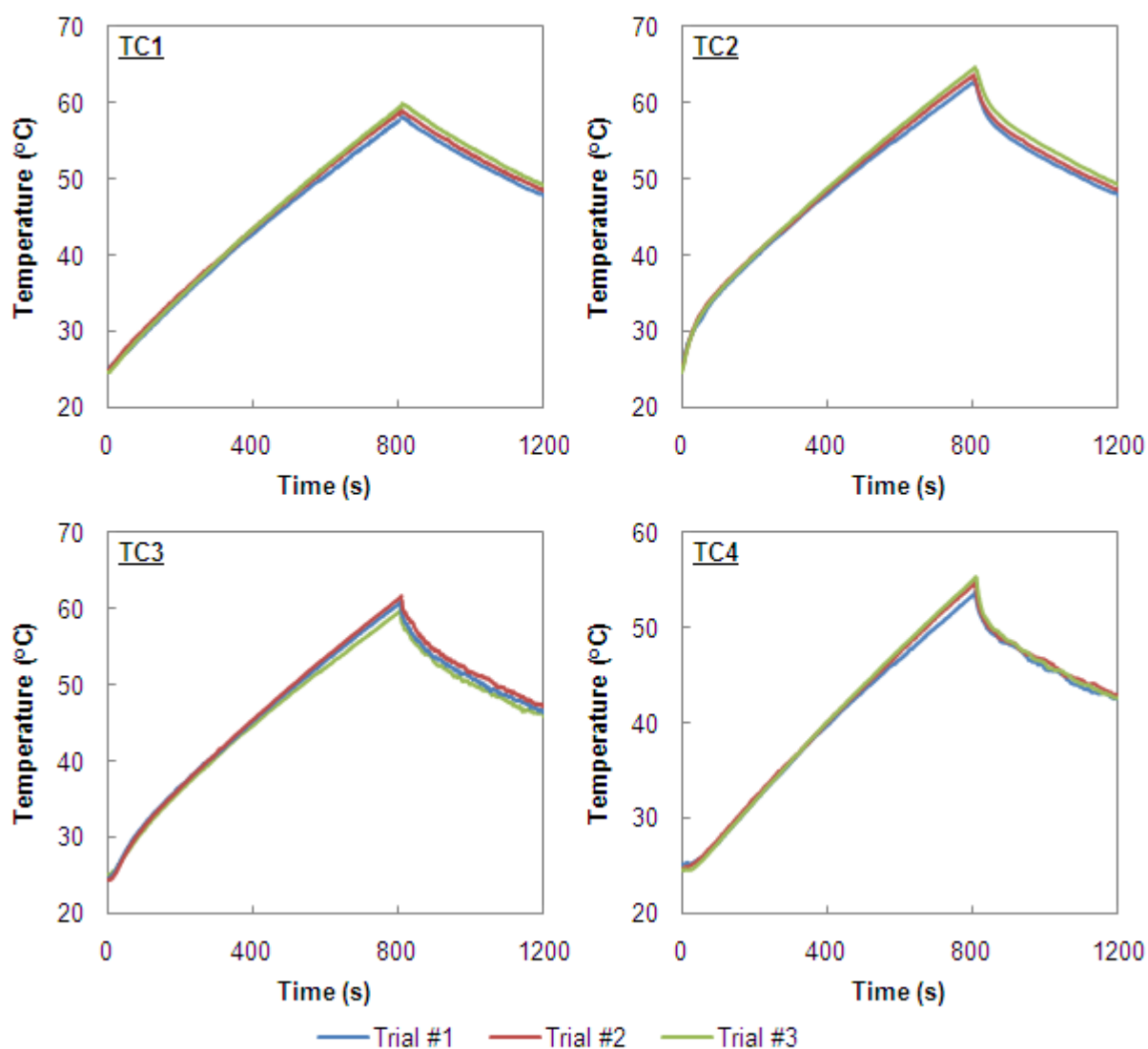


Figure A.27: Temperatures of thermocouples TC1-TC4 for IHCT-CPMC heated with constant power

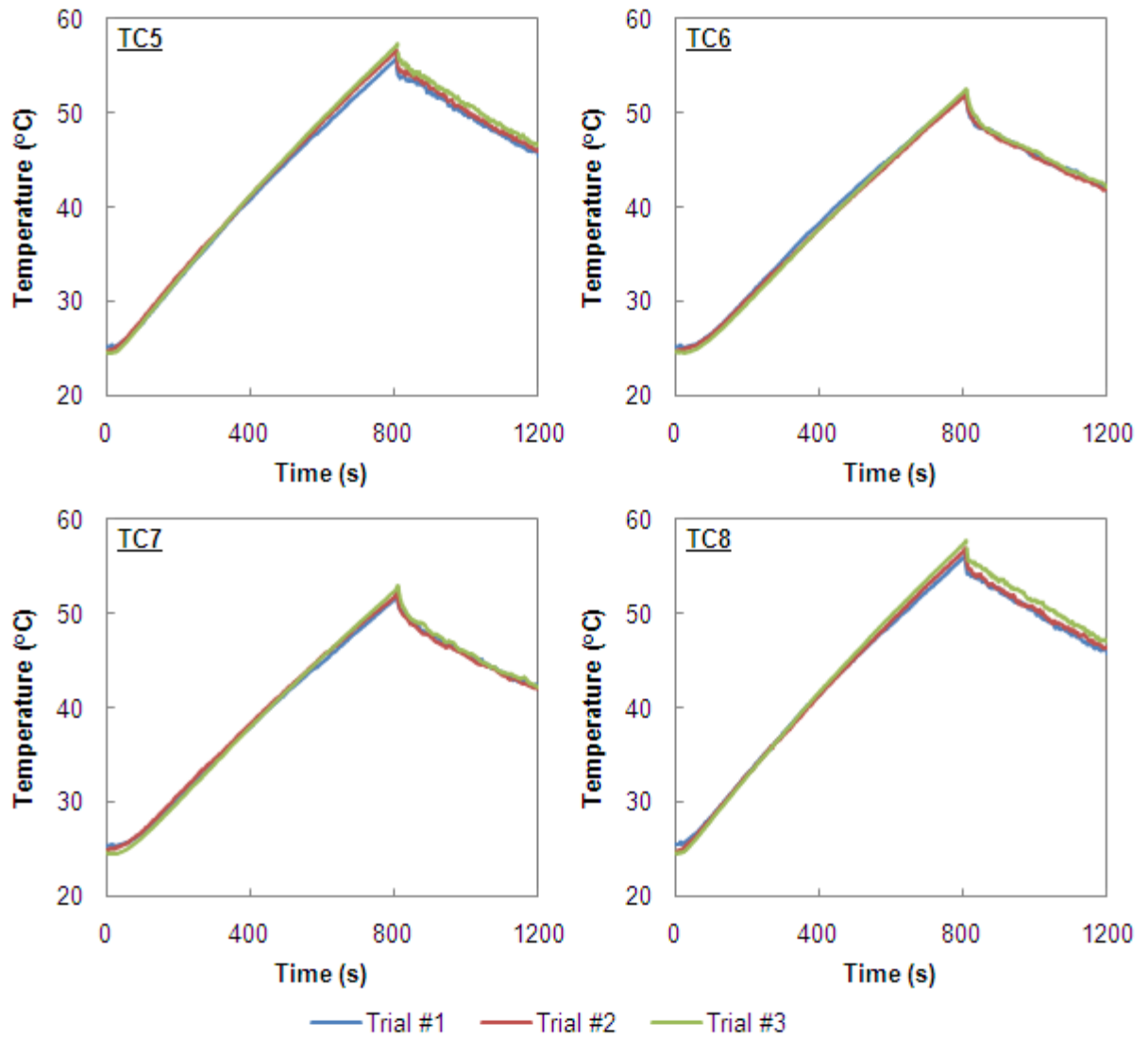


Figure A.28: Temperatures of thermocouples TC5-TC8 for IHCT-CPMC heated with constant power

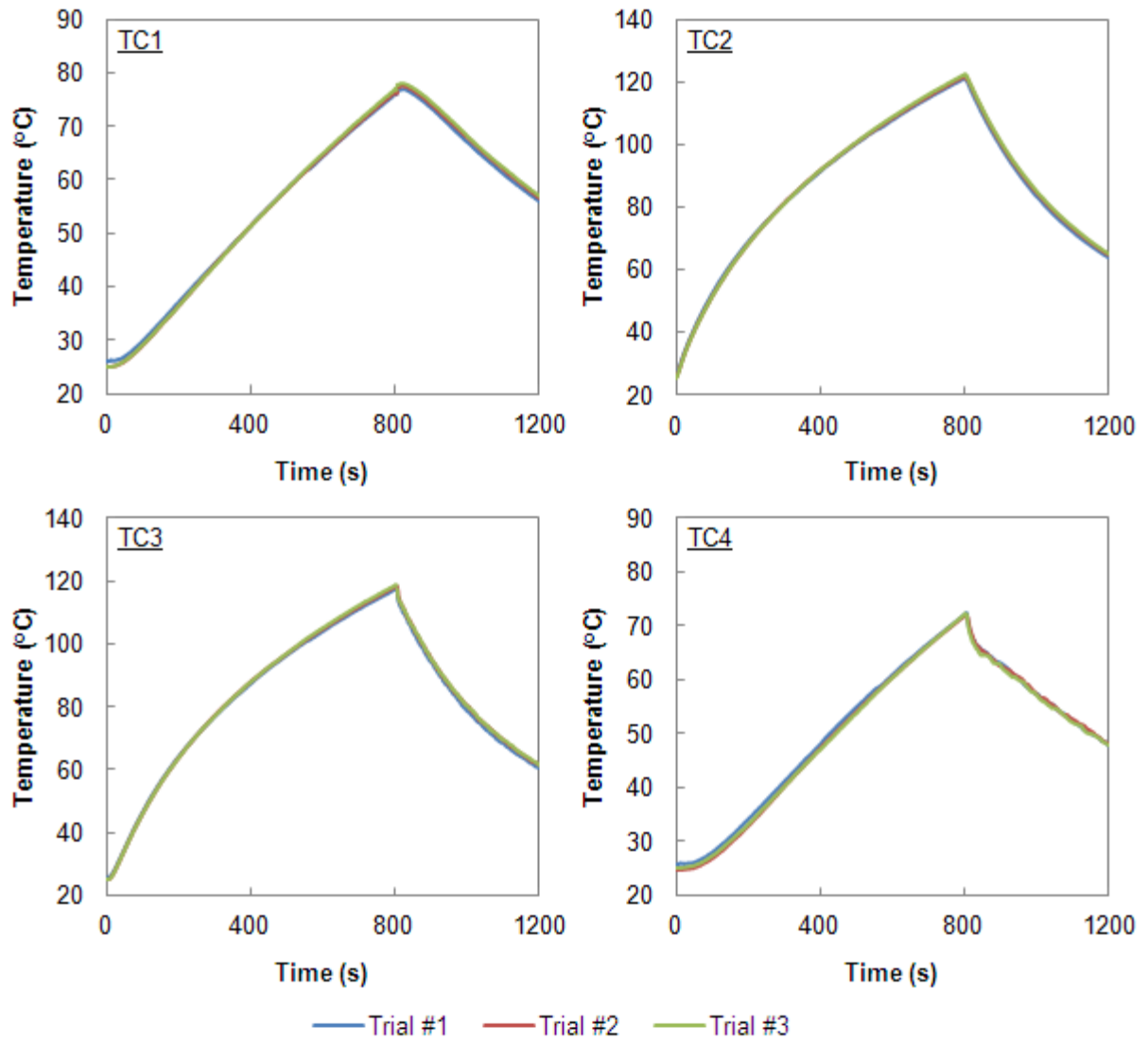


Figure A.29: Temperatures of thermocouples TC1-TC4 for IHCT-PMC heated with constant power

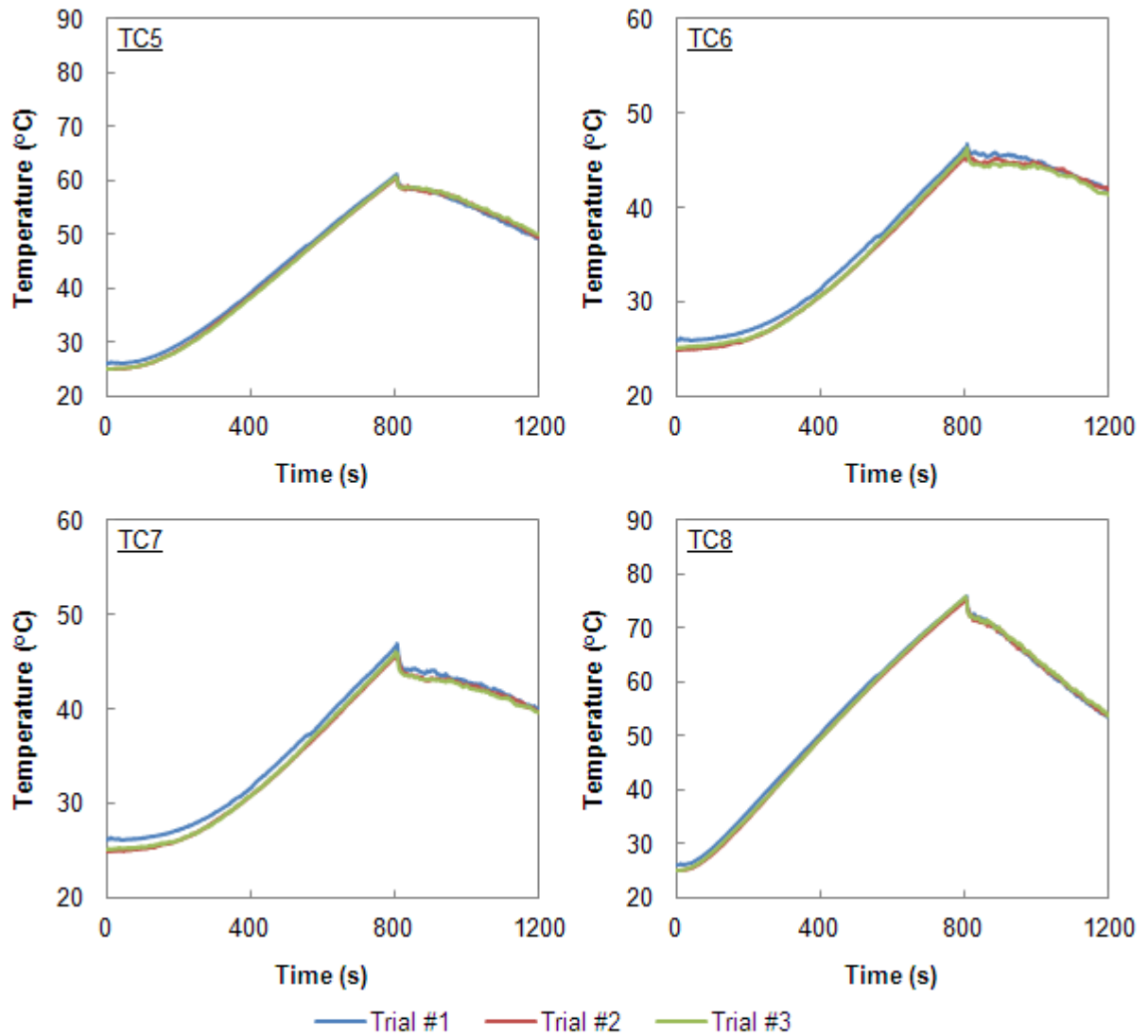


Figure A.30: Temperatures of thermocouples TC5-TC8 for IHCT-PMC heated with constant power

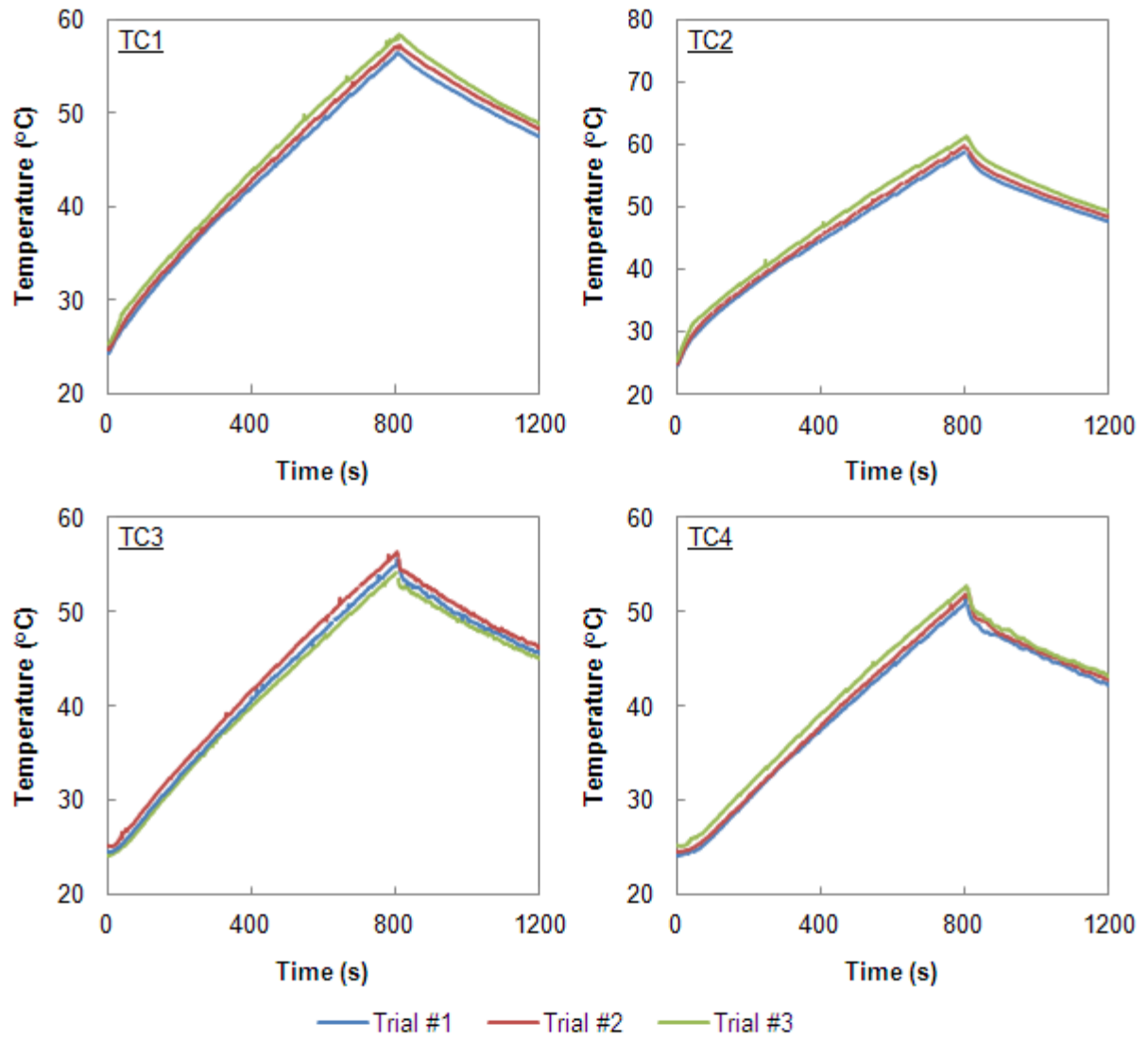


Figure A.31: Temperatures of thermocouples TC1-TC4 for IHCT-NRC #1 heated with constant power

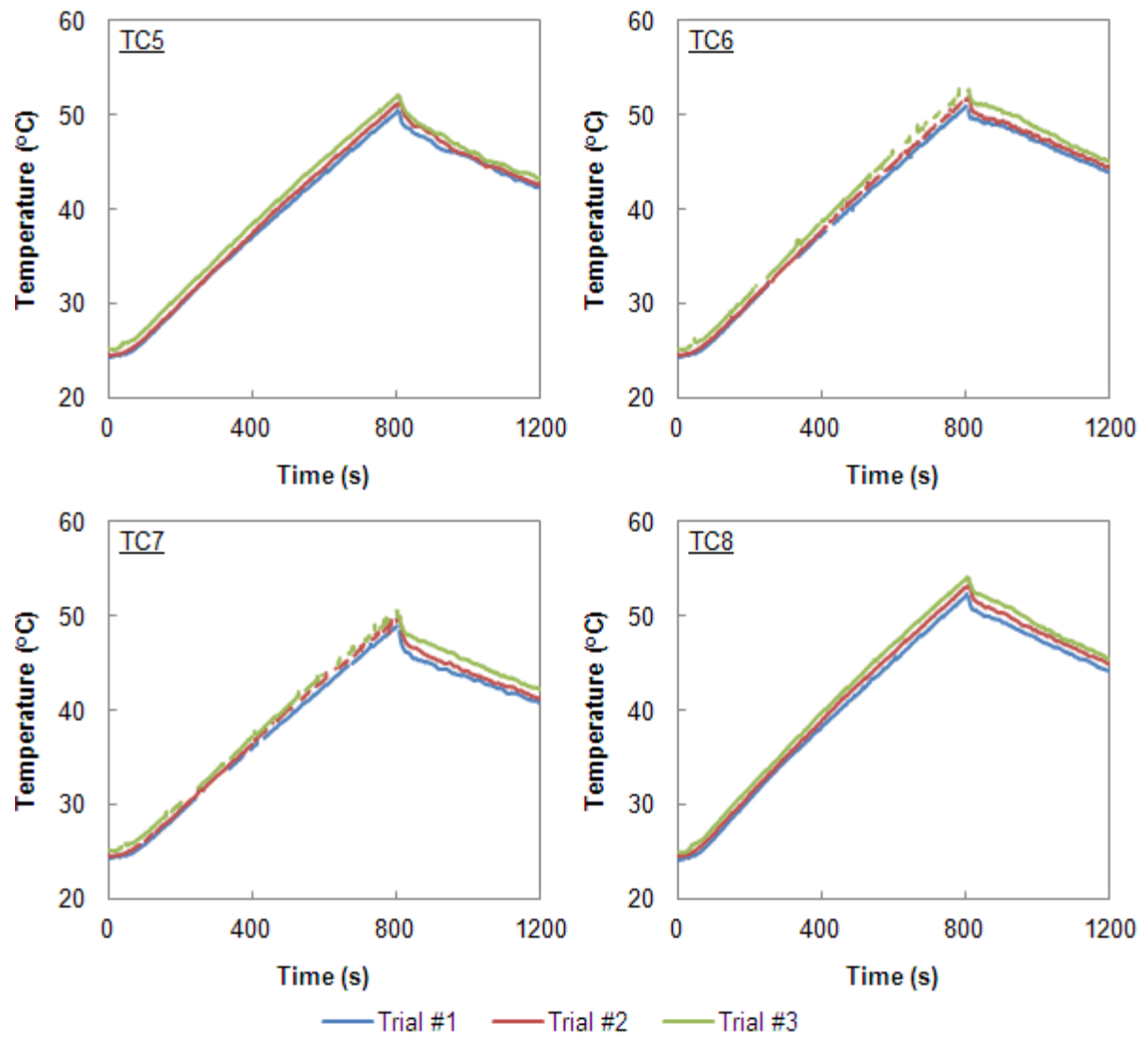


Figure A.32: Temperatures of thermocouples TC5-TC8 for IHCT-NRC #1 heated with constant power

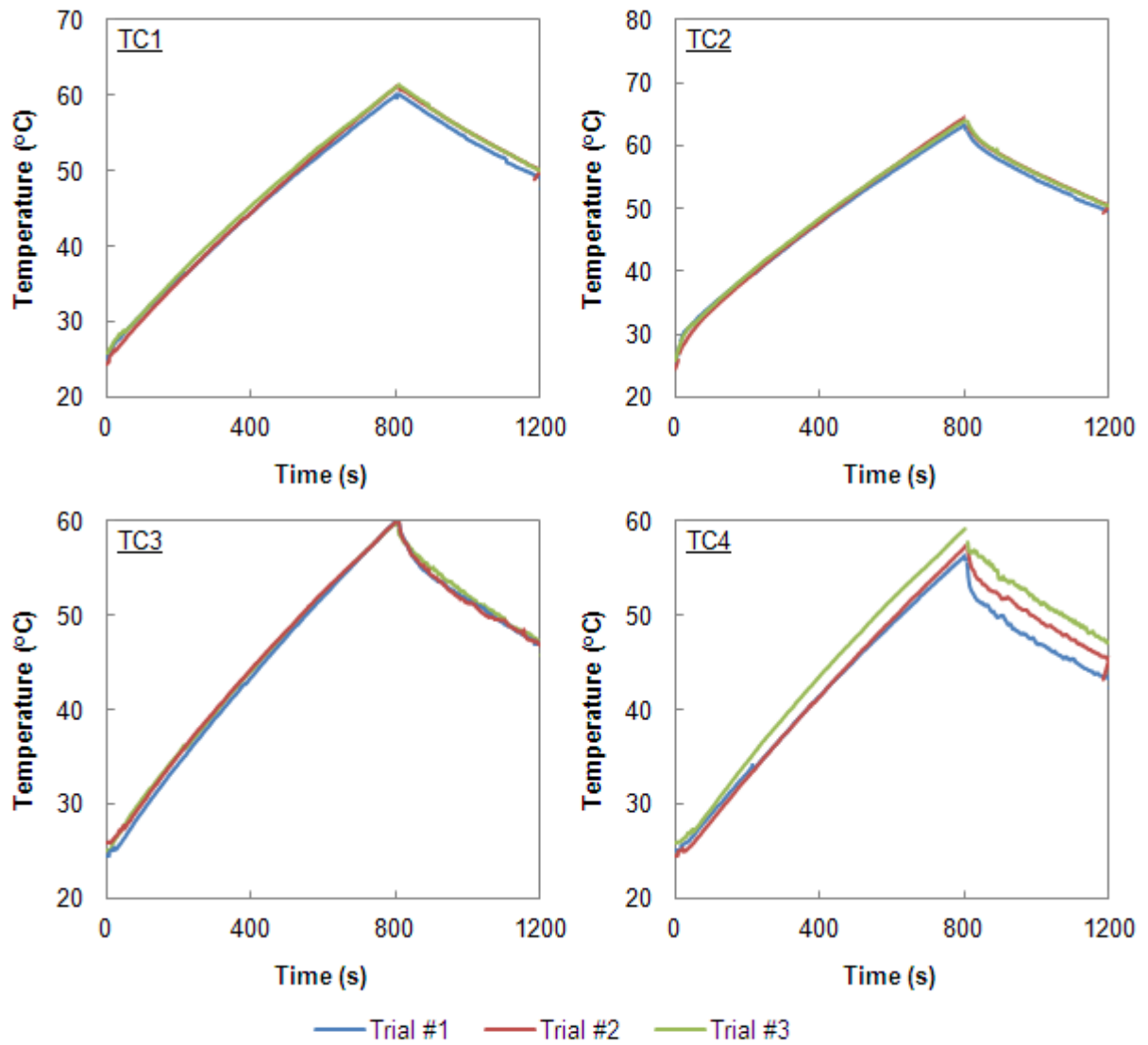
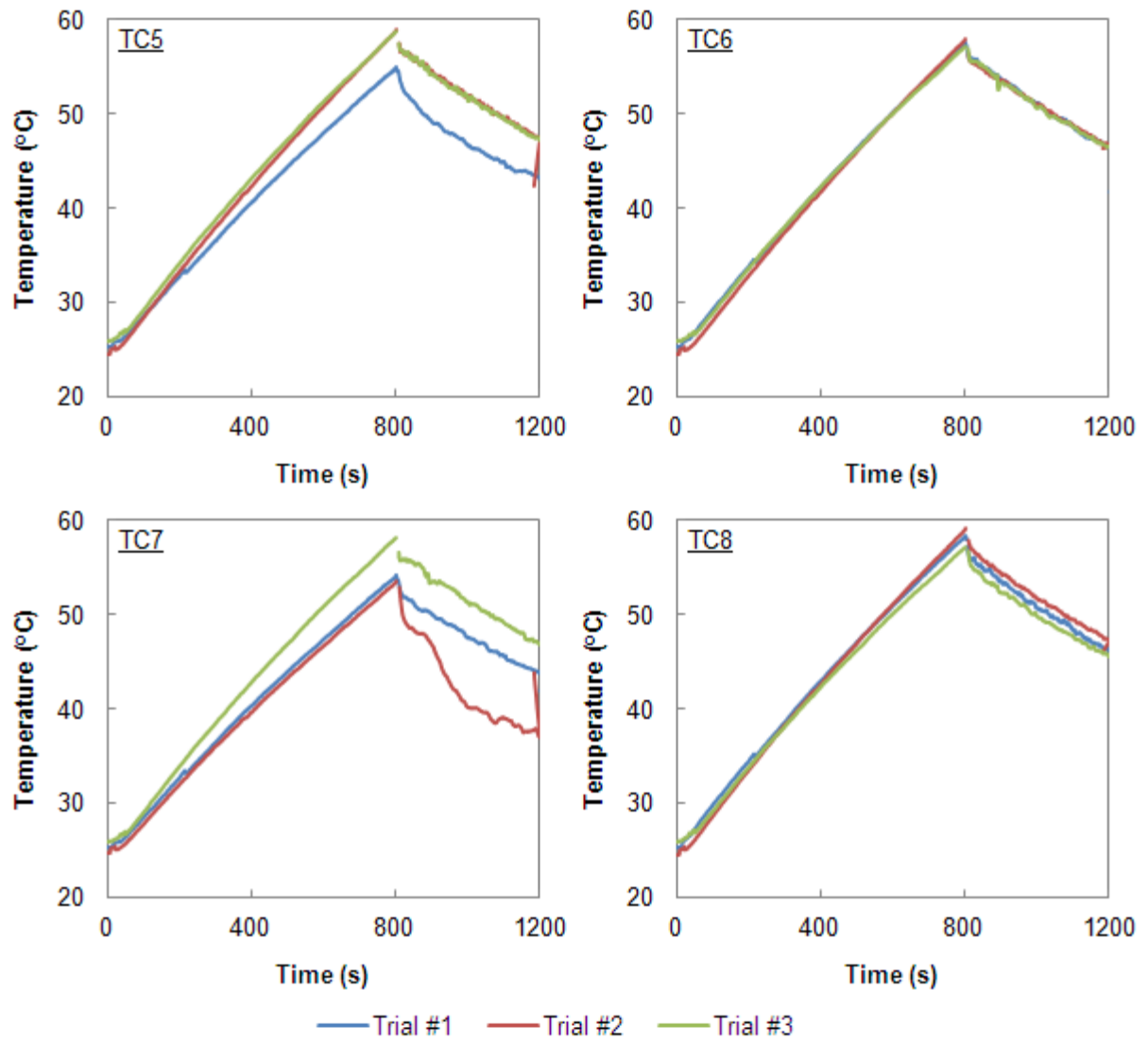


Figure A.33: Temperatures of thermocouples TC1-TC4 for IHCT-NRC #2 heated with constant power



Note: Discrepancies for TC5 and TC7 were caused by debonding of the thermocouple

Figure A.34: Temperatures of thermocouples TC5-TC8 for IHCT-NRC #2 heated with constant power

Appendix I: Temperature repeatability of HT-01

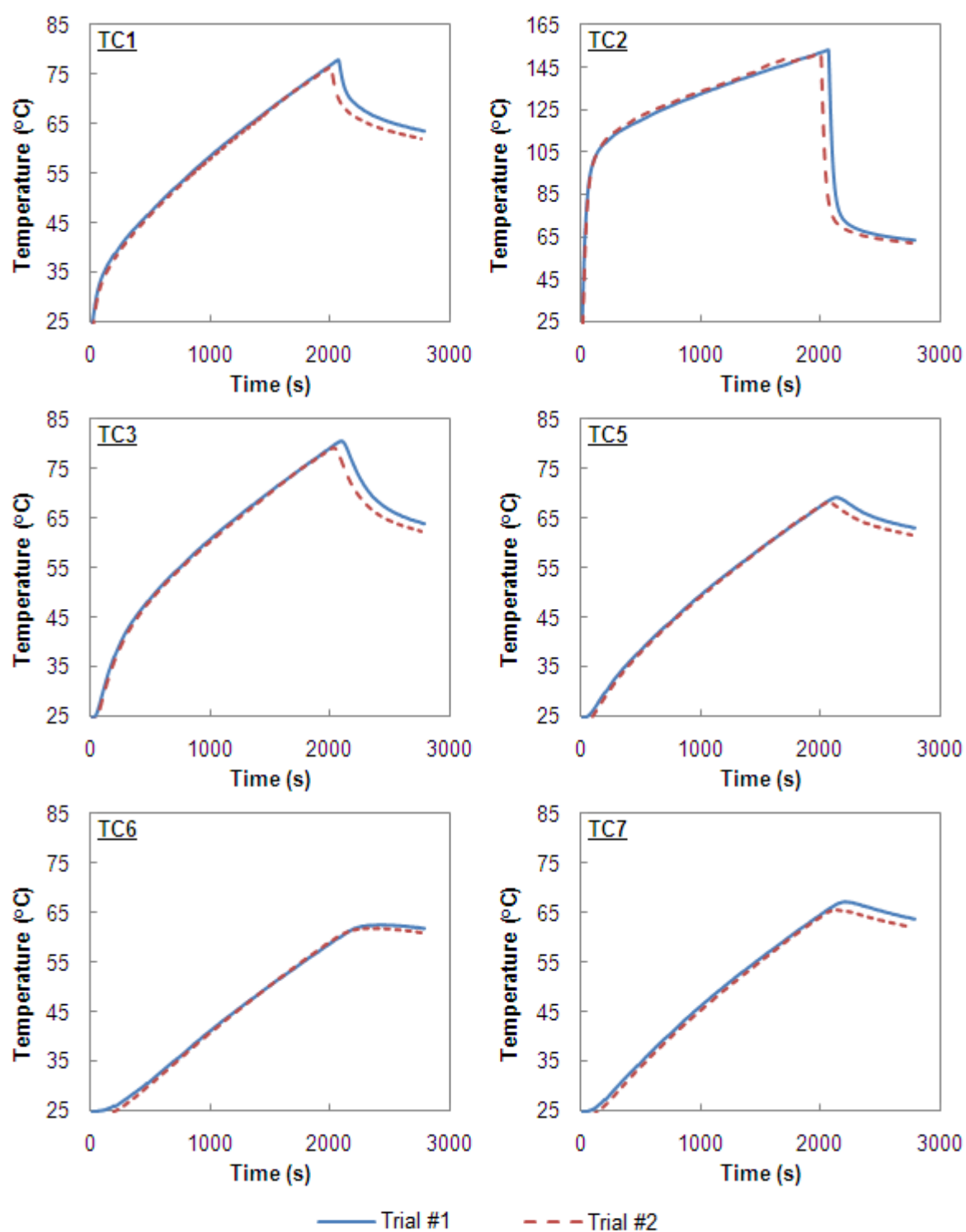


Figure A.35: Repeatability trials for thermocouples TC1-TC7, HT-01

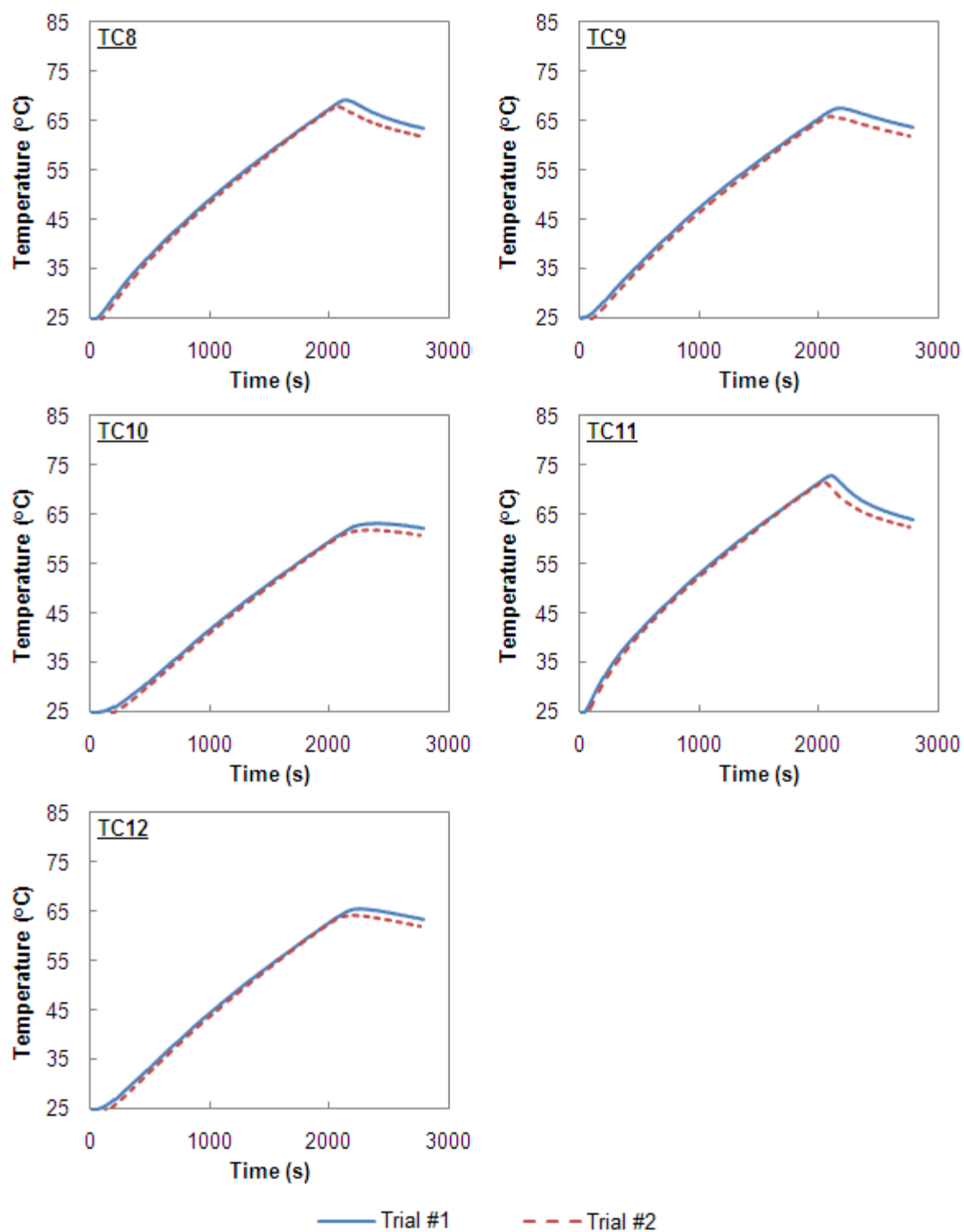


Figure A.36: Repeatability trials for thermocouples TC8-TC12, HT-01

Appendix J: Effect of mesh refinement for HT-01

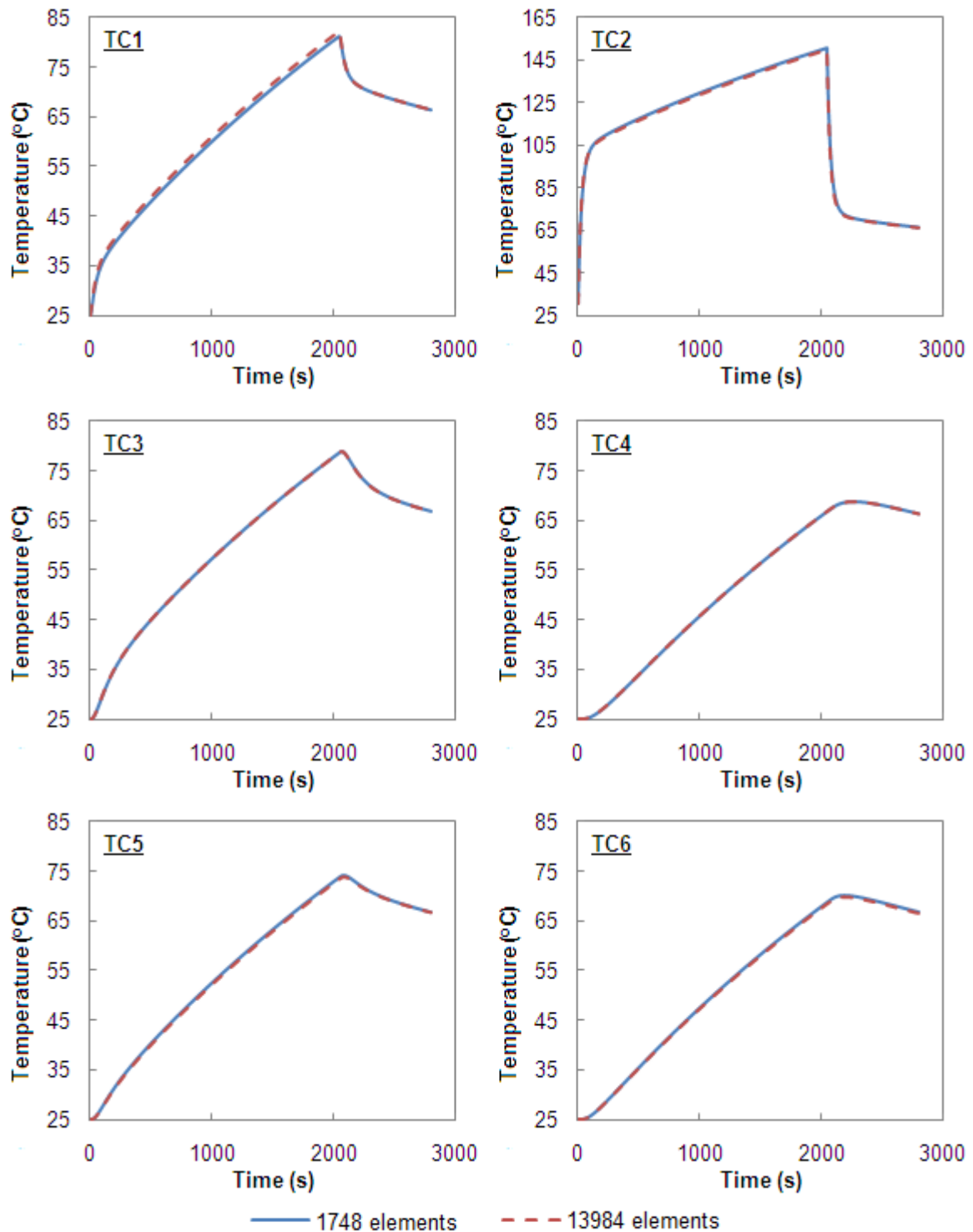


Figure A.37: Temperature comparison between coarse and refined meshes for thermocouple TC1 to TC6, HT-01

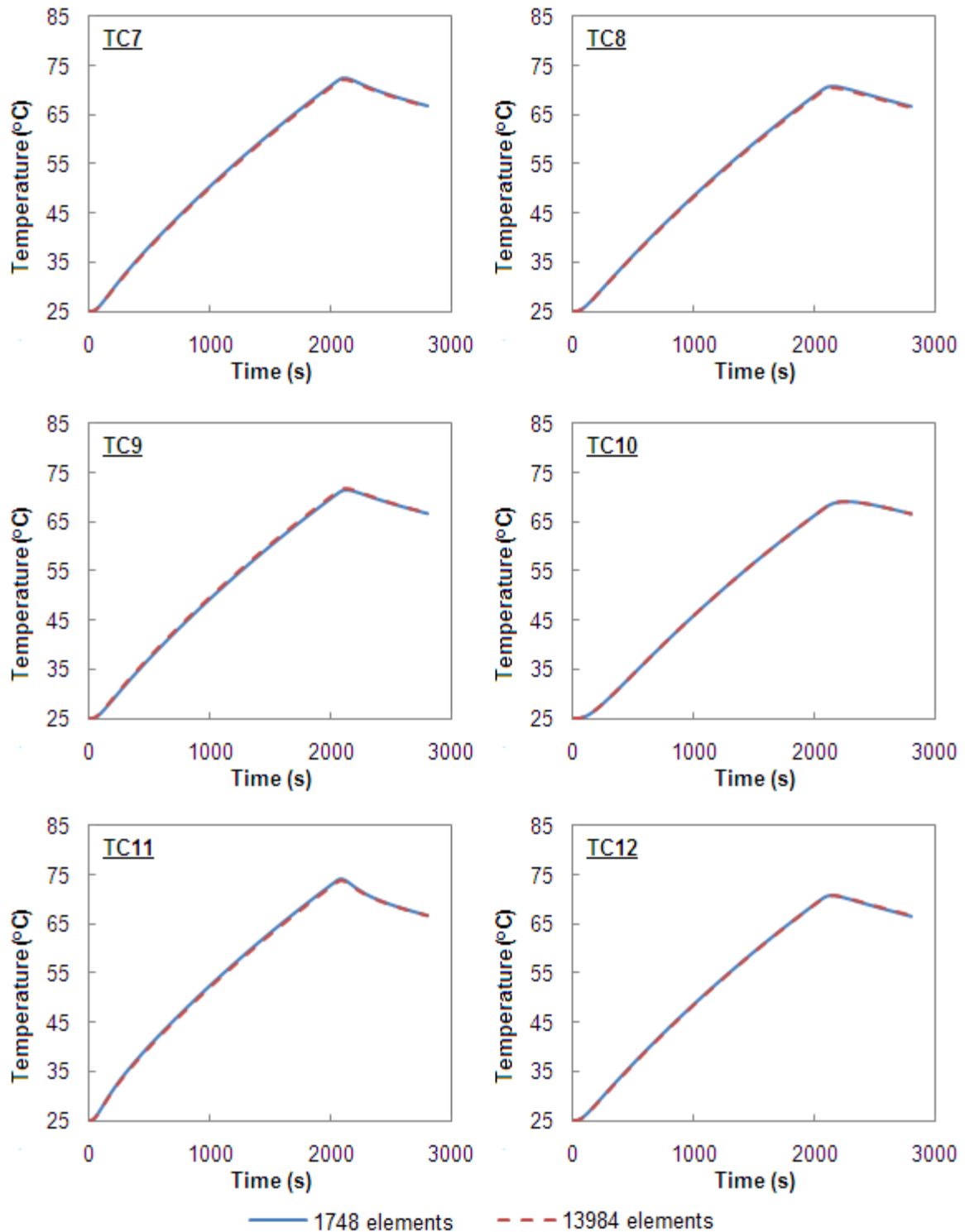


Figure A.38: Temperature comparison between coarse and refined meshes for thermocouple TC7 to TC12, HT-01

Appendix K: Factorial designs for HT-03

The full factorial design used in HT-03 is presented in Table A.1, while the 8-run fractional factorial design appears in Table A.2. Parameters were given a state, +1 represented the maximum value of the parameter, while -1 represented the minimum value. The fractional factorial design was obtained by retaining all positive values of interaction ABCD. This reduction led to confounding parameters and interaction that were grouped in contrasts (Table A.2).

Table A.1: Parameters and interactions of the full factorial design, HT-03

Experiment	Parameters and interactions													
#	A	B	AB	C	AC	BC	ABC	D	AD	BD	CD	ABD	BCD	ABCD
1	-1	-1	1	-1	1	1	-1	-1	1	1	1	-1	-1	1
2	-1	-1	1	-1	1	1	-1	1	-1	-1	-1	1	1	-1
3	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	-1
4	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	1
5	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	1	-1
6	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	-1	1
7	-1	1	-1	1	-1	1	-1	-1	1	-1	-1	1	-1	1
8	-1	1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1
9	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	-1	-1
10	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	1	1
11	1	-1	-1	1	1	-1	-1	-1	-1	1	-1	1	1	1
12	1	-1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	-1
13	1	1	1	-1	-1	-1	-1	-1	-1	-1	1	-1	1	1
14	1	1	1	-1	-1	-1	-1	1	1	1	-1	1	-1	-1
15	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1
16	1	1	1	1	1	1	1	1	1	1	1	1	1	1

Table A.2: Contrasts and contrast states of confounding parameters and interactions of the 8-run factorial design, HT-03

Simulation	Retained experiment #	Contrasts							
		A	B	AB	C	AC	BC	D	
		BCD	ACD	CD	ABD	BD	AD	ABC	
HT-03-1	1	-1	-1	1	-1	1	1	-1	
HT-03-2	4	-1	-1	1	1	-1	-1	1	
HT-03-3	6	-1	1	-1	-1	1	-1	1	
HT-03-4	7	-1	1	-1	1	-1	1	-1	
HT-03-5	10	1	-1	-1	-1	-1	1	1	
HT-03-6	11	1	-1	-1	1	1	-1	-1	
HT-03-7	13	1	1	1	-1	-1	-1	-1	
HT-03-8	16	1	1	1	1	1	1	1	

Scree plots of the contrasts were built from the response of each experiment. The importance value attributed to each contrast was obtained by multiplying the response of an experiment by its contrast state (+1 or -1) and taking the sum of the squared values of all experiments:

$$\text{Sum of squares for a contrast} = SS_C = \sum \left[\text{Response} \cdot \text{Contrast state}^2 \right] \quad (\text{A.25})$$