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Thermoforming of Thin-Ply Composite Structures via In-Situ Heating

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THERMOFORMING OF THIN-PLY COMPOSITE STRUCTURES VIA
IN-SITU HEATING

by

BOJAN BIJELIC

A thesis submitted in partial fulfillment of the requirements
for the degree of Bachelor of Science
in the Department of Mechanical and Aerospace Engineering
in the College of Engineering and Computer Science
at the University of Central Florida
Orlando, Florida

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Thesis Chair: Kawai Kwok, Ph.D

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ABSTRACT

This thesis investigates thermoforming of thin-ply thermoplastic composites via in-situ heating for in-space manufacturing applications. The proposed composite concept is based on combining conductive carbon nanotube (CNT) films and high-temperature thermoplastic matrix. The CNT film is made of randomly aligned carbon nanotubes, which possesses outstanding electrical, thermal, and mechanical properties. When combined with polymer matrix, it becomes a multifunctional composite structure. The thermoplastic chosen is polyether ether ketone (PEEK), which is a semicrystalline high-performance thermoplastic that has exceptional physical and mechanical properties at high temperatures. The composite structure studied consists of a layer of CNT film sandwiched between two thin films of PEEK. The CNT acts as an in-situ conductive heater when a voltage difference is applied, and a mechanical reinforcement. The PEEK polymer impregnated with reinforcement fibers and CNT is capable of reforming by repeating the thermoforming process. The focus of this study is on developing and characterizing the manufacturing process suitable for in-space manufacturing, where CNT/PEEK can be treated as a composite prepreg capable of being reformed into different shapes on demand via thermoforming. The thermoforming of thin-ply composite structures is achieved solely via in-situ electrical heating.

To Katarina, the inspiration in my life.

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CHAPTER 1

INTRODUCTION

Thin-ply composites are rapidly gaining interest in the aerospace industry, because of the design possibilities that these structures can achieve. The main advantage of thin-ply composites is their flexibility and foldability. In addition, thin-ply composite laminates are capable of enduring high strains, and they are currently under investigation for deployable spacecraft structures [1]. Most of spacecraft structures are quite large. For example, solar sails, composite booms, and satellites antennas can have lengths up to 10 meters. In manufacturing, the size of molding equipment scales with size of composite structure. Therefore, the current composite manufacturing processes has a high footprint and high upfront development effort.

1.1 Thin-ply Composite Structures

Thin-ply composite structures are widely used in a variety of engineering structures. These structures are typically made of laminated layers of fibers pre-impregnated with thermoset or thermoplastic polymer [2]. The advantage of these structures is the optimization ability of their mechanical performance and the reduction of structural weight compared to conventional composite structures. Furthermore, the main benefit of using thinner layers in composite structures is the

ability to use large number of ply orientations to achieve a better design [3]. This is particularly important because it allows the use of multiple thin layers which can achieve optimal laminates of thin-ply composites.

Currently, the most common choice of material for ultra-thin composites is carbon fiber reinforced with thermoset polymers such as epoxy. The disadvantage of this technique are the microcracks caused by stress that occurs in the direction that is different from the direction of the fiber [4]. The material with great potential to overcome this issue is based on combination of the carbon fiber and thermoplastic polymer. The carbon fiber reinforced polyether ether ketone (CF-PEEK) will provide a good substitution for conventional material of choice such as carbon fiber with epoxy resin. The main advantage of using CF-PEEK is overcoming micro-cracking. Furthermore, the combination of these materials will provide strong fiber-matrix interface [4].

1.2 Carbon Nanotube / PEEK Thermoplastics

The CNT is a relatively new material that is rapidly gaining interest of researchers. The CNT provides a combination of mechanical, thermal, and electrical properties that are interesting for a variety of applications. The structure of CNT is formed by folding a single graphene layer to form a hollow tube composed of carbon atoms that are bonded together in a hexagonal mesh [5]. Hence, the CNT has outstanding mechanical properties. For example, the CNT is the stiffest and strongest fiber known, with Young's modulus as high as 1 TPa and tensile strengths up to 63 GPa [6]. The CNT has a much higher Young modulus compared to the stiffest conventional carbon fibers that

has Young's modulus of approximately 800 GPa [6]. Aside from mechanical properties, CNT has remarkable electrical and thermal properties as well. The CNT can be considered as electrical resistor, especially when CNT is combined with other polymers [7]. For example, most polymers can be treated as insulators since polymers have a range of electrical conductivity from 10^{-16} S/m to 10^{-12} S/m, while CNT has electrical conductivity around the 600 S/m [7]. Furthermore, CNT are excellent electrical conductor with current density up to 10^{11} A/m² [6]. In addition, CNT has a very high thermal conductivity. Due to the many outstanding properties of this material, CNT has wide application range.

PEEK is semicrystalline high-performance thermoplastic that has outstanding physical and mechanical properties at high temperatures. PEEK has exceptionally high melting and glass transition temperatures due to its c and its stiff backbone interconnected by ketone and ether groups [8]. Hence, PEEK thermoplastic polymer has wide commercial application in electronics, aeronautics and aerospace industries. Furthermore, PEEK thermoplastics have a growing interest in aerospace composite structures. Because of PEEK superior properties it has been widely used as the matrix for fiber-reinforced composites [9]. When PEEK polymer is used as a matrix in composite structures, the thermoplastic property of the PEEK allows for reusability of the material. For example, once the PEEK polymer is impregnated into fiber-reinforcement, it can be treated as a regular aerospace prepreg [4]. Thus, the combination of this materials is perfect for reforming and remolding applications.

1.3 Thermal Processing of Composite Structures

The conventional manufacturing process of composite structures is inefficient from the energy consumption and heat transfer aspect. The traditional curing techniques such as in-oven, consume a high amount energy during the curing process, yet just a portion of the energy is utilized to cure the composite structure. The energy losses are mainly through heat transfer and the geometry of the oven. Heating a large volume of the air in the oven results in inefficient consumption of the energy [10]. Therefore, the current composite manufacturing process is not suitable for in space manufacturing.

1.4 Objectives

The primary objective of this study is to develop a manufacturing process that is suitable for in space manufacturing. To achieve this, the study proposes to develop thin-ply composite structure using in-situ heating. The idea is to design a thin-ply composite prepreg using carbon nanopaper and PEEK (CNP-PEEK). The CNP-PEEK will be treated as aerospace composite preprag capable of being reformed by repeating thermoforming process. The CNP will be used as reinforcement fiber and integrated heating element in composite structures while PEEK thermoplastic will be a polymer matrix. In-situ thermal processing will be involved in the composite manufacturing process. The idea is to embed the CNP into thin-ply laminates as an in-situ heater. Hence, thermal processing of laminate will be achieved solely via electrical conductivity heating. Furthermore,

the advantage of using PEEK thermoplastic as a polymer matrix is the ability to reform by repeating thermoforming process while maintaining comparable mechanical properties as thermoset laminates.

CHAPTER 2

LITERATURE REVIEW

The structural applications of thermoplastic composites are increasing rapidly. Thermoplastic matrix composites possess distinct advantages compared to the thermoset matrix composites in terms of recyclability, high specific strength and specific stiffness, corrosion resistance, impact toughness, and flexibility in design [11, 12, 13, 14]. Composites are classified based on the type of polymer matrix as thermoset and thermoplastic. Thermosetting plastics and thermoplastics are both polymers, but they behave differently when exposed to heat. Thermoset plastics such as epoxy, phenolic, polyesters and vinyl ester resins are fully cross-linked [11]. This means that thermoset polymers retain their form and stay solid under heat once cured. On the contrary, thermoplastics such as polyamide (PA) polypropylene (PP), polyethylene (PE) poly phenylene sulphide (PPS), and poly(ether ether ketone) (PEEK) exhibit minimal cross-linking [11, 12]. Thus, thermoplastic polymers can be melted and formed repeatedly. Both of thermoset and thermoplastic polymers can be reinforced with a variety of reinforcements such as glass, carbon, or amid fibers [11].

The manufacturing processes of composites are broadly classified as pre-impregnation and post-impregnation methods. In pre-impregnation methods, fibers are wetted and impregnated by the polymer in one step where the composite is made either by melt or solution impregnation [11]. On the other hand, in the post-impregnation methods the polymer is in the dry form such as film,

filament or powder [11]. Another very important step in fabrication of thermoplastic composite is joining. Joining of thermoplastics composites is critical step in the process of manufacturing aerospace thermoplastic composite structures [13, 14]. In general, joining of thermoplastic composites can be categorized into mechanical fastening, adhesive bonding, solvent bonding, co-consolidation, and fusion bonding or welding [14]. The most promising fusion bonding techniques for thermoplastic composites are induction, resistance and ultrasonic welding. Fusion bonding is the principle of heating the polymer at the interface to a viscous state causing polymer chains to inter-diffuse, and cooling the polymer for joint consolidation [13, 14, 15].

2.1 Out of Oven Curing Process

The conventional composite manufacturing process has several limitations such as high energy consumption and geometric constrains [10, 16, 17]. The high energy consumption during composite manufacturing is related with composite curing process. The energy loss in conventional curing process is associated with convection heat transfer [10, 16]. For example, autoclaves and ovens transfer heat indirectly through gas medium (air) which leads to inefficient consumption of energy. The geometric constrains of autoclaves and ovens is another major limitation of conventional curing process [10, 17]. The manufacturing of large size composite parts requires sizing of ovens, which leads to inefficient consumption of energy due to larger volume of air that is needed to be heated during curing process. The capacity of autoclaves is limited by size. Furthermore, man-

ufacturing composites within autoclaves is accompanied by high acquisition and operation costs due to the necessity of a specialized heated pressure vessel [16].

Several studies focus on developing new composite curing processes that will overcome all of the limitations of conventional curing technique. The Out-of-Oven (OoO) curing process is technique that uses nanocomposites as conductive heaters. The advantaged of OoO curing process is that the laminates are cured directly due to conduction heat transfer [10, 17]. For example, by placing carbon nanotubes onto the surface of the laminate and by applying a voltage difference through CNT, the material is heated directly and the heat loss to surrounding is minimal [10]. This type of curing is more efficient in energy consumption because the heat is transferred directly from heating element to the laminate. Using this technique, studies show that the OoO curing process reduces energy consumption by two orders of magnitude compared to conventional curing process. Furthermore, laboratory testing shows that there are no significant differences in thermo-physical and mechanical properties between OoO and in-oven curing technique [16]. Since the integrated conductive heater directly and efficiently transfers heat via conduction, the study shows that an integrated microheater can be used to heat polymer matrix composites (PMC) regardless of the part size and shape [10].

In addition, by using a nanostructured resistive heater composed of the aligned nanotubes (A-CNT) during OoO curing process leads to significant energy reduction [10]. Furthermore, the A-CNT microheater film transfers approximately 90 % of input energy into the curing part regardless of the dimensions of the part, whereas the conventional oven technique transfers around 50 % of input energy and depends on the dimensions of the curing part and on the dimensions of the oven

[10]. The integrated microheater film directly transfers heat through the material structure via conduction. On the other hand, conventional oven technique transfers heat indirectly through gas medium (air). As a result, microheater film or OoO technique consumes less energy during the curing process. The study shows that the laminates cured using a conventional oven consumes 1 KW power compared to A-CNT heater that consumes less than 30 W of power [10]. Furthermore, the A-CNT microheater can operate at temperatures $> 500^{\circ}\text{C}$ in air and $> 1000^{\circ}\text{C}$ in nonoxidizing environments [10]. This makes A-CNT microheater a good candidate for thermal processing of high temperature polymers such as polyimides and polyetheretherketones.

2.2 In-Space Manufacturing Process

In-Space Manufacturing (ISM) process is a promising approach for construction next-generation space structures [18, 19]. The ability to manufacture structures in space instead of launching them from Earth will reduce logistic requirements of space missions. Both Low Earth Orbit (LEO) and beyond missions require significant logistic support from Earth. On the International Space Station (ISS), spare parts, tools and consumables are periodically shipped from the Earth into space [20]. Most importantly, the development of ISM and assembly capabilities is essential for future space exploration [21]. The ability to manufacture materials and parts in-space will extend human operations in space. So far, NASA and its partners developed several ISM processes which have been tested on the ISS [19]. Some of the ISM process developed so far are 3D Printing in Zero G, Additive Manufacturing, Vulcan Advanced Hybrid Manufacturing System etc. However, all

manufacturing processes require a significant amount energy which is another challenge in space missions.

2.3 Electric Power in Space

The primary source of power for space missions is solar energy. The solar panels are the only source of energy that enables satellites in orbit to be fed continuously [22]. The solar array converts solar energy into electricity on the space vehicle. The solar cells are made from purified chunks of silicon. These cells directly convert light to electricity using a process called photovoltaics [23].

The ISS is the largest spacecraft orbiting around Earth, and it is one of the most complex engineering systems ever developed [24]. The ISS electrical power system is the most critical system on station and it is crucial that it operates continuously. The electrical power system, consists of power generator, energy storage, power management, distribution equipment and various loads [24]. All electrical power on the ISS is collected by solar arrays. The structure of the power generation system consists of eight solar array wings with two solar blankets for each solar wing [24]. The total maximum power provided to ISS is approximately 128 kW [23, 24]. However, just about 75 % of the power is used to support the ISS systems. The major ISS loads are battery units, fans, and atmosphere controllers [24]. In addition, the large portion of electrical power is used for conducting research and experiments on the station.

CHAPTER 3 METHODOLOGY

3.1 Material Selection

3.1.1 Carbon Nanopaper

The CNP is thin sheet of film made from CNT fibers. The CNTs are fundamentally divided into two classes: single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). Both have aspect ratios from 100 - 10000, and thermal conductivities from 3000 - 6000 W/mK. The thermal conductivity of CNTs (3000 - 6000 W/mK) is 2 orders of magnitude higher than conventional carbon fibers [25].

CNPs are ideal for reinforcing fillers for polymer composites because their promising mechanical properties and excellent thermal and electrical properties [25, 26, 27]. CNP has an extremely high heat transfer rate which makes it an excellent material for Joule's heating applications [13]. A voltage difference applied to the CNP heats the CNP to high temperatures. The heat transfers from the CNP to the polymer matrix up to the melting temperatures by heat conduction and initiates the melting of polymer matrix. For these reasons, the CNP is selected as the conducive reinforcement filler for this study.

3.1.2 PEEK Polymer Matrix

PEEK is semicrystalline polymer that has drawn attention of many researchers mainly because of the remarkable characteristics of this polymer [28]. PEEK is a high performance thermoplastic with excellent mechanical and thermal properties [28, 29, 30, 31]. Furthermore, PEEK polymer has broad chemical resistance properties, and is one of the promising thermoplastic matrices for composite structures [29, 30]. It has a high melting and glass transition temperatures ($T_m = 340^\circ\text{C}$, $T_g = 143^\circ\text{C}$) [28]. Hence, PEEK has been used in vast amount structural and insulation engineering applications [28, 29]. The carbon-fiber (CF) is one of the most popular fibers used with PEEK matrix. The outstanding mechanical properties and low weight of CF makes this material a good choice for aerospace applications [29]. However, in past two decades the nanocomposites have drawn attention because of their industry applications [31]. Especially, CNT that have excellent mechanical, thermal and electrical properties is popular reinforcement fiber in nanocomposites structures [6, 25, 26, 27, 31]. The PEEK thermoplastic is promising candidate for the polymer matrix in nanocomposites [30]. In addition, by incorporating the CNT which is highly conductive material into insulating polymer, leads to remarkable increase in the electrical conductivity of composites [31].

3.2 Composite Fabrication

Manufacturing of aerospace composite structures is traditionally done by using autoclaves. This manufacturing technique enables high-quality reproducible parts [16]. However, manufacturing composites in autoclaves is accompanied by high operation cost due to specialized heated pressure vessel and energy consumption due to geometric constraints [10, 16, 17]. Furthermore, the capacity of autoclaves is limited by size and design of composite structures, and production rates depend on autoclave availability [16]. The next generation of composite manufacturing process needs to overcome the limitations of conventional composite manufacturing such as high energy consumption and large manufacturing equipment [16]. Currently, the focus in this area of research is to develop new composite manufacturing processes using nanocomposites as conductive heaters. This process is also known as OoO curing process or in-situ curing technique. Lee et al. show that carbon fiber reinforced plastic prepreg system is cured via single-side CNT network heater incorporated on the outer surface of laminate without using an autoclave [16]. Furthermore, the work shows that composites cured by CNT film heaters can achieve degree of cure that are equivalent or better than composites cured by an autoclave.

In addition, another study shows that using nonstructured resistive heater in composite curing process leads to significant energy reduction [16]. Furthermore, the study compares two curing techniques convectional and OoO curing process. The biggest disadvantage of the conventional curing process is inefficient energy consumption. The main reason why conventional curing process is inefficient in terms of energy consumption is because of convection heat transfer [16]. The

conventional oven technique transfers heat indirectly through gas medium (air). On the other hand, the integrated microheater film directly transfers heat through the material structure by conduction. As a result, microheater film or OoO technique consumes less energy during curing process. Furthermore, the energy required to cure the same size composite laminate is 10 times smaller using the OoO curing process compared to conventional curing process [16]. So, heating a large volume of the air in the oven results in inefficient consumption of energy.

3.3 Material Properties

In this research the main objective is to develop a manufacturing process using nanocomposite as an integrated heating element. Furthermore, the nanocomposite will also serve as the reinforced fiber filler in the composite structure while the composite matrix will be the thermoplastic polymer. The material selection for this research is cCNP with a high electrical conductivity and good thermal stability. A carbon nano paper mat (Nanotechlabs) is selected for this study with areal density of 60 gsm. The PEEK polymer film (AptivFilms) used has thickness of 0.250 mm. Tables 3.1 and 3.2 display material properties of CNP and PEEK thermoplastic polymer.

Table 3.1: *CNP Material Properties*

Measure	Value	Unit
Areal Density	60	gsm
Thickness	0.250	mm
Young's Modulus	2.206	MPa
Peak Load	4.92	N
Peak Stress	11.03	MPa
Failure Strain	0.85	%
Resistivity	0.150	$\Omega \cdot \text{cm}$
Electrical Conductivity	6.666	S/cm

Table 3.2: *PEEK Polymer Material Properties*

Measure	Value	Unit
Density	1300	kg/m^3
Melting Point	340-385	$^{\circ} \text{C}$
Thickness	0.250	mm
Young's Modulus	2.300	MPa
Resistivity	$4.0 \cdot 10^{16}$	$\Omega \cdot \text{cm}$
Specific Heat	1700	J/kgK
Latent Heat	$130 \cdot 10^3$	J/kg
Thermal Conductivity	0.25	W/mK
Electrical Conductivity	$9.4 \cdot 10^{-3}$	S/cm

3.4 Electro-Thermal-Mechanical Constitutive Model

Thin-ply composite structure is designed as shown in Figure 3.1. The CNP layer is sandwiched between two layers of PEEK polymer film of equal thickness. The voltage differences are applied on the electrically conductive CNP layer.

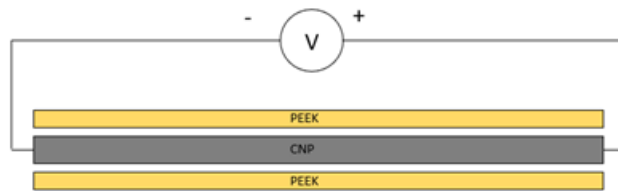


Figure 3.1: *Voltage Difference Applied on Thin-ply Composite*

By applying voltage differences onto CNP layer, the temperature rises via Joule heating. A fraction of this heat is transferred to the PEEK polymer matrix to bring the temperature above melting point of polymer. So, heat transfers from the CNP layer to PEEK polymer layer by heat conduction. The method of lumped mass heat transfer was used which assumes the temperature field in the CNP to be uniform and dependent on time only [7]. Furthermore, to prevent heat loss to the surrounding, the laminate is placed between the two layers of ceramic fiber thermal insulators (BXI Ceramic Fiber Thermal Insulation Board) as shown in Figure 3.2.

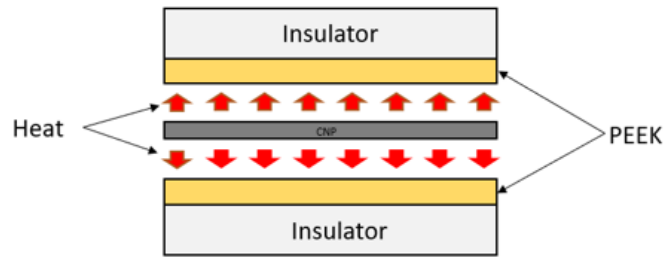


Figure 3.2: *Thin-ply Composite with Insulator*

In the manufacturing process, pressure is applied by two aluminum molds (6061) and the pressure is controlled by two clamps with bolts as shown in Figure 3.3. The goal is to apply voltage differences onto CNP to increase the temperature of CNP up to the melting point of PEEK polymer. As soon as PEEK melting point is achieved, pressure is applied to the thin-ply composite structure and the polymer diffuses into the CNP forming a composite structure.

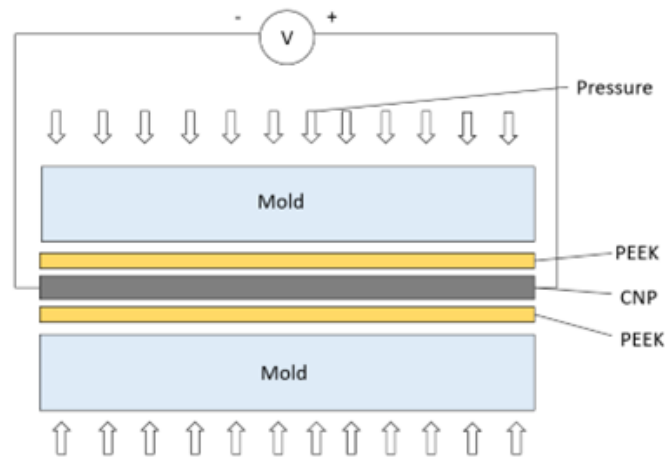


Figure 3.3: *Fabrication Process of Thin-ply Composite*

3.5 Electro-Thermal Model

In-situ heater or CNP has high electrical conductivity and extremely high heat transfer rate which makes it an excellent material for Joule's heating applications [27]. The electro-thermal model establishes the relation between voltage input and temperature fields in the CNP and the PEEK polymer film [7]. The CNP is considered as electrical resistor while the PEEK is assumed to be an electrical insulator. The following equations are derived by Ozdemir and Kwok [7]. The energy balance of CNP is expressed by following equation,

$$\dot{Q}_{gen} = \dot{Q}_{loss} + \dot{Q}_{stored} \quad (3.1)$$

where \dot{Q}_{gen} is the rate of heat generation in the CNP, \dot{Q}_{loss} is the rate of heat transferred to the PEEK polymer and surroundings, and \dot{Q}_{stored} is the rate of heat stored in CNP. The rate of heat generation is given by the Joule's first law,

$$\dot{Q}_{gen}(t) = \frac{V^2}{R_{CNP}} \quad (3.2)$$

where R_{CNP} is the electrical resistance of the CNP, and V is the voltage applied onto CNP. The CNP electrical resistance is related to its electrical conductivity γ_{CNP} as follows,

$$\gamma_{CNP} = \frac{L_{CNP}^2}{R_{CNP}V_{CNP}}, \quad (3.3)$$

where L_{CNP}^2 is the length of the CNP along the direction of current flow and V_{CNP} is the volume of the CNP. Since the CNP is thin and fully embedded into the PEEK polymer film, the heat loss due to radiation and convection are neglected. Finally, the heat rate of heat transferred into the PEEK polymer is shown below,

$$\dot{Q}_{loss}(t) = 2A_{int} \frac{T_{CNP}(t) - T_{int}(t)}{R_{int}^T} \quad (3.4)$$

where, A_{int} is the contact area between CNP and PEEK polymer, T_{CNP} is temperature of CNP, T_{int} is the temperature of PEEK polymer at the interface, and R_{int}^T is the thermal resistance at the CNP/PEEK interface.

3.6 Energy Consumption

3.6.1 Energy Consumption During Manufacturing Process

The manufacturing of thin-ply composite structure requires thermal processing in order to change the state of polymer from solid state to liquid state. The thermal processing can be split up into 3 stages as shown in Figure 3.4. Stage I is the initial heating stage, stage II is maintaining melting temperature of polymer, and stage III is the cooling stage.

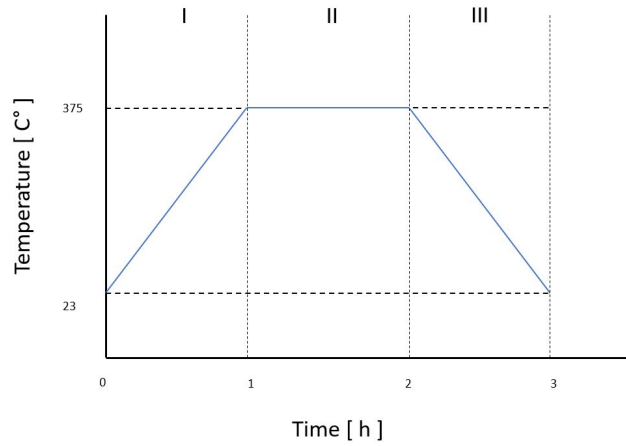


Figure 3.4: *Thermal Profile*

The procedure used to evaluate the energy consumption during the manufacturing process is described in this section. The amount of energy needed to melt the polymer is represented by the sum of latent heat, specific heat capacity of polymer, and energy stored into CNP. Since the CNP is thin and fully embedded into the PEEK polymer film, and due to fact that high thickness of insulator is used during manufacturing process, the heat losses due to radiation and convection are neglected.

$$Q_{melt} = Q_L + Q_{peek} + Q_{cnp} \quad (3.5)$$

Where, Q_L is latent heat of PEEK polymer and Q_{peek} is specific heat capacity of polymer.

$$Q_{melt} = m_p L + m_p c_p \Delta T + m_{cnp} c_{cnp} \Delta T \quad (3.6)$$

Where, m_p is mass of polymer, L is specific latent heat of a substance, c_p is specific heat of polymer, and ΔT is temperature difference during manufacturing process. The energy stored into CNP during the manufacturing process can be calculated by specific heat capacity of CNP. Where, m_{cnp} is mass of CNP, c_p is specific heat capacity of CNP, and the ΔT is temperature differences during manufacturing process. The value of specific heat capacity is $0.74 \frac{J}{gK}$ for the randomly oriented thin films [32].

3.6.2 Energy Consumption During Thermoforming of Composite Prepreg

The energy consumption during composite thermoforming experiment is an important aspect of this research as the idea of this study is to make thin ply prepreg material for In-Space Manufacturing. The assumption is that all manufacturing will be performed on Earth and sent into Space where the reforming of composite prepreg will be executed. The experiments show that the temperature required to change the shape of the composite is around glass transition temperature of polymer. This indicates that the amount of energy needs for reform will be smaller compared to manufacturing process of composite prepreg where is important to achieve melting temperature of polymer.

The energy needed for thermoforming of the composite prepreg is calculated by sum of energy stored in CNP and specific heat capacity of PEEK polymer.

$$Q_{SC} = Q_{peek} + Q_{cnp} \quad (3.7)$$

The experiment is conducted in a laboratory without use of any insulator, so the heat loss due to free convection is included in this calculation.

$$Q_{loss} = 2hAt(T_{inf} - T_s) \quad (3.8)$$

where, Q_{loss} is energy losses to environment, h is free convection heat transfer coefficient, t is total time of shape-changing process, ΔT is temperature differences during shape-changing process.

3.6.3 In Space Manufacturing Energy Consumption

The fiber-reinforced polymers in space requires heating of feedstock close to its melting point [18]. The following equations are derived by Bhundiya et al. The energy required to reach the melting temperature and the latent heat of melting, expressed as:

$$u_{melt} = C_p(T_m - T_0) + L_m \quad (3.9)$$

where C_p is the specific heat, T_m is the melting temperature, T_0 the ambient temperature, and L_m the latent heat. Assume the material is heated in space from a low ambient temperature $T_m \gg T_0$. The approximation of correlation $L_m \approx 0.4C_pT_m$ and an overall efficiency of 15 % for heat losses, the following equation approximate energy per kilogram.

$$u_M^* \approx 9.3C_pT_m \quad (3.10)$$

However, in order to calculate energy required for fiber-reinforced composites, the specific heats of the fiber and matrix are needed because the material is heterogeneous. The specific heat (C_{FRP}) is given by the rule of mixtures,

$$C_{FRP} = w_f C_f + (1 - w_f) C_m \quad (3.11)$$

where w_f is the weight fraction of the fiber, and C_f , C_m are the specific heats of the fiber and matrix. The processing energy differs between thermoplastic and thermoset matrices. Thermoplastics relies on heating the matrix beyond its glass transition temperature before it is cooled in the desired shape, while thermosets relies on a chemical curing reaction which causes permanent polymerization and cross-linking [18]. The following equation represents a thermoplastic matrix scenario, where estimate the energy required to heat the polymer matrix to its melting temperature, with a process efficiency of 15 % ,

$$u_{FRP-TP}^* \approx 9.3 C_{FRP} T_m \quad (3.12)$$

where, C_{FRP} is the specific heat of the composite, and T_m is the melting point of the thermoplastic matrix.

CHAPTER 4

THIN-PLY COMPOSITE MANUFACTURING PROCESS

4.1 Sample Manufacturing Process

The thin-ply composite is manufactured using CNP as the reinforced fiber and PEEK polymer film as the composite matrix. The laminate consists of one layer of CNP and two layers of PEEK film where each layer has a thickness of 0.25 mm. The CNP is sandwiched between two layers of PEEK film. The dimensions of the specimen are shown in Figure 4.1 where the size of CNP is 70 mm by 20 mm and the size of PEEK film is 60 mm by 25 mm. To apply voltage differences on the CNP two copper electrodes are attached on composite as shown in Figure 4.1.

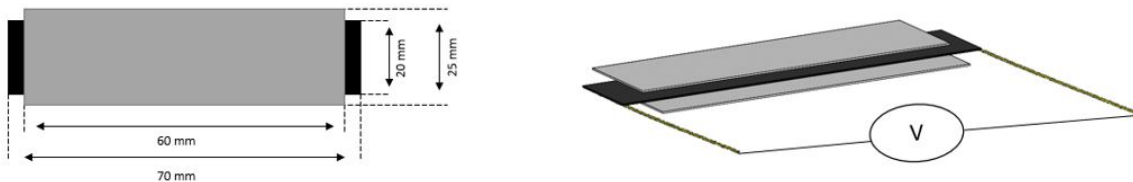


Figure 4.1: *Specimen Configuration*

The mold is created using two aluminum blocks (6061-t6) as shown in Figure 4.2. The dimensions of the mold are 300 mm by 150 mm with thickness of 75 mm. The purpose of the mold is to

generate the required pressure during the manufacturing process. Furthermore, the pressure is controlled with two screw clamps. In order to reduce heat loss during the fabrication process, the mold is insulated with a ceramic board (BXI Ceramic Fiber Thermal Insulation Board). The dimensions of insulator board are 200 mm by 150 mm with thickness of 20 mm.

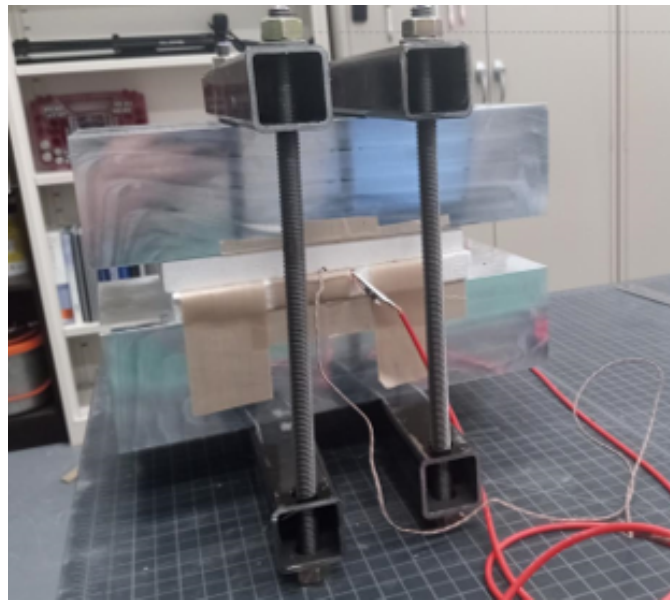


Figure 4.2: *Mold with Insulator and Screw Clamps*

The control system is built to control parameters and gather data during the manufacturing process. The block diagram of the control system is shown in Figure 4.3. The control system gathers data on the input power, the surface temperature of sample, the pressure applied on the sample, and the fabrication time. The voltage difference is applied on the composite sample by using laboratory DC power supply (GPS-3030DD). The surface temperature of sample is measured using thermocouple (Type-K) where the temperature is recorded every 1 second and can be monitored in real time. The load cell (TAS606) is used to record pressure applied during fabrication process.

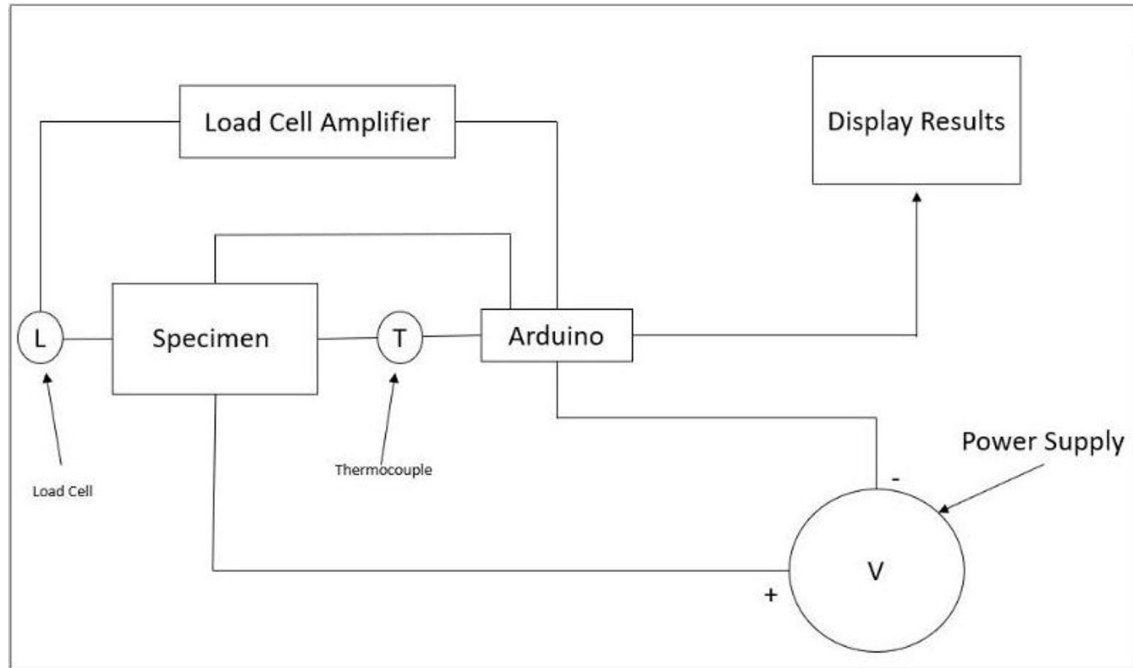


Figure 4.3: Control System for Fabrication Process

The manufacturing process is divided into three segments: 1) reaching melting point of PEEK film, 2) maintaining melting point temperature so that polymer diffuses into CNP, and 3) cooling the composite to room temperature. The time required to reach the melting point of the PEEK film mainly depends on power input. Reaching the melting temperature of PEEK requires less time when the input power is higher. Maintaining melting temperature is required until the polymer completely diffuses into the CNP. Maintaining melting temperature requires small power adjustments. Finally, the time required for cooling the composite before releasing the sample from the mold mainly depends from mold insulator.

The temperature distribution during fabrication process is presented in Figure 4.4. The total time required to manufacture the composite sample is 198 minutes, where the first 118 minutes are

needed to reach melting temperature (375°C) of polymer. The polymer is maintained in the melted state for the next 60 minutes at temperatures ranging between 375°C - 400°C . The time required to cool the sample to room temperature is 20 minutes. During the fabrications process, the power supply is set to an average voltage and current of 18 V and 1.8 A, respectively.

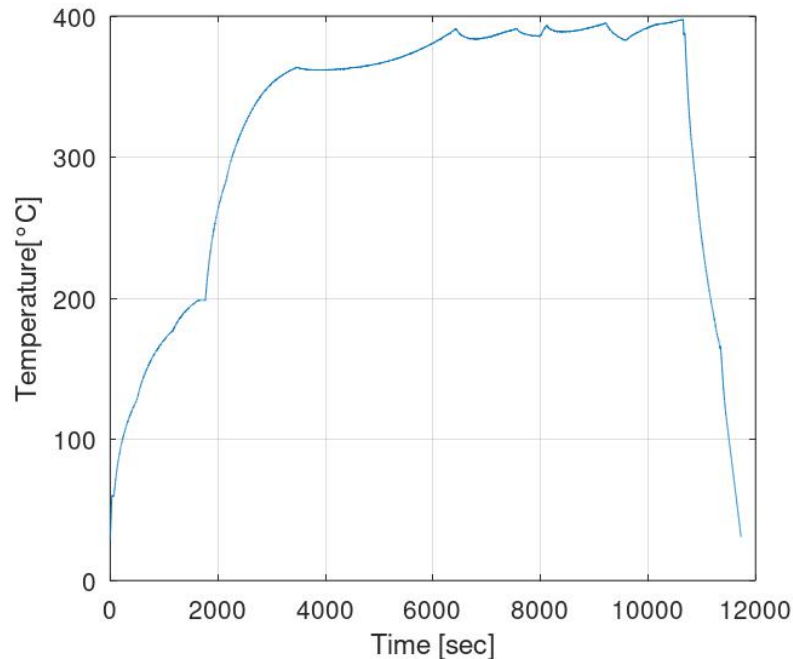


Figure 4.4: *Thermal Model During Fabrication Process*

After cooling the sample to room temperature, the specimen is released from the mold. Figure 4.5 displays the manufactured sample. Observing the sample surface, it may be concluded that the polymer melts and diffused into CNP. The average thickness of manufactured sample is 0.5 mm.



Figure 4.5: *Manufactured Specimen*

4.2 Thermofrming of Composite

Material shape changing is the process of changing an existing shape into another geometric shape. For this process, the idea is to repeat the thermoforming process on manufactured composite sample, and to apply pressure on it in order to change geometric shape. In this study, the shape changing is performed on thermoplastic composite (CNP/PEEK) sample previously manufactured. The thermoplastic composite matrix allows for repeated heating of composite without changing the mechanical properties of polymer matrix in composite sample. This approach allows for gradual change of the actual curvature of the material and increases reusability of thin-ply composites as is shown in Figure 4.6.

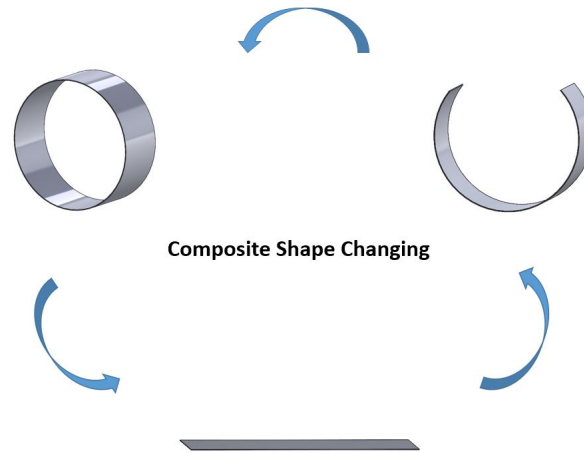


Figure 4.6: *Shape Changing*

Figure 4.7 shows the manufactured thin-ply composite sample used in the shape changing experiment. The composite sample has dimensions 85 mm by 20 mm with thickness of 0.5 mm. The electrodes are attached at the ends of the sample, and thin aluminum foil is used to prevent sticking of the sample onto the mold. The mold is used to form a simple geometric shape and it consists of the core and cavity mold. The thermocouple is placed on the surface of composite sample, and it is used to measure the surface temperature during the heating process. The flexible insulator is placed on the surface of the aluminum mold to reduce heat loss during the process. The scope of this experiment is to show the ability of shape changing of thin-ply thermoplastic composite from a flat sample to a simple arc. Future work, the focus will be on developing shape changing processes without molds which will be more suitable for In-Space Manufacturing applications and creating more complex shapes.

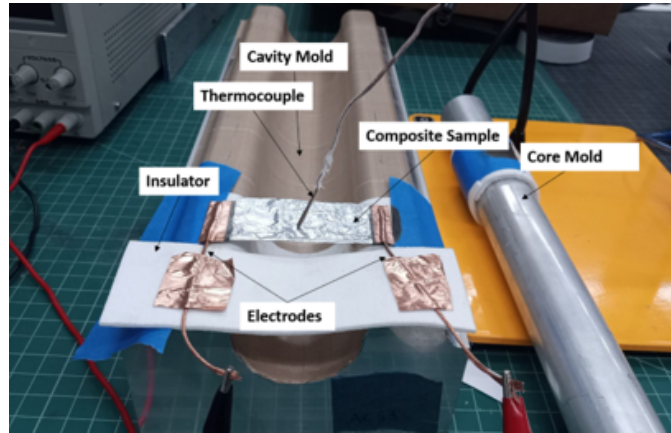


Figure 4.7: *Shape Changing Setup*

In the shape changing experiment, the composite is heated to glass transition temperature of polymer matrix (T_g) = 143° C by applying voltage difference onto composite sample. As soon the sample reaches the required temperature, the pressure is applied by the core mold. The sample stays pressed by molds until the surface temperature cools to room temperature.

The thermal model for reshaping composite structures is presented in Figure 4.8. The total time required for shape changing process is 14 minutes, where the first 3 minutes is the time required for the specimen reach glass transition temperature, and 11 minutes are needed for specimen to cool down to room temperature.

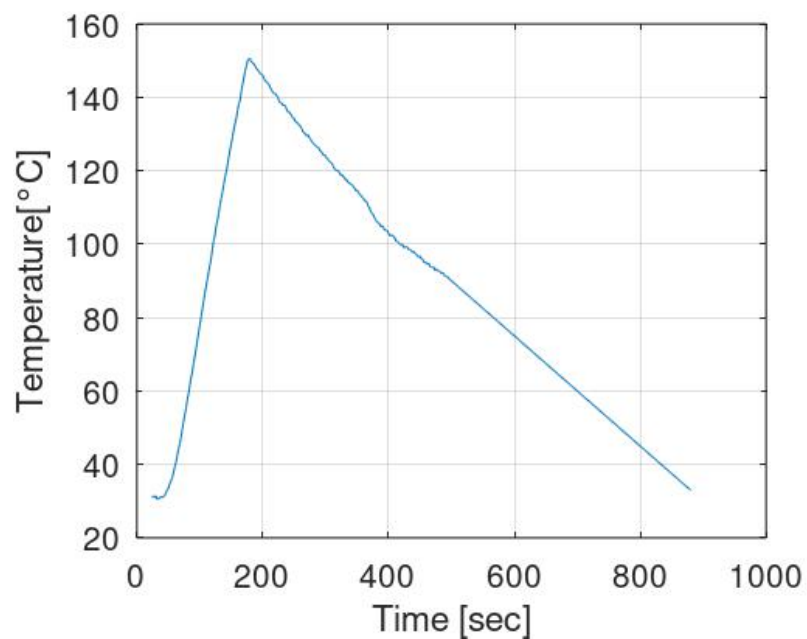


Figure 4.8: *Thermal Model During Shape Changing Process*

Figure 4.9 shows the sample after it is released from the molds. Observing the sample, it is concluded that repeating the thermoforming process successfully changed the state of polymer matrix which enables the material to change shape. The total time of this process is 14 minutes, and the applied voltage is 25 V.



Figure 4.9: *Thin-ply Composite Shape Changing*

The next step according to Figure 4.6 is joining thin-ply composite sample in order to create a closed section. In future work, the focus will be on developing techniques for joining thin-ply thermoplastic composites where the fusion bonding process will be investigated.

CHAPTER 5

EXPERIMENTATION AND DISCUSSION

5.1 Scanning Electron Microscopy

The Scanning Electron Microscope (SEM) is used to examine microscopic structures by focusing a beam of electrons that scans over the surface of a cross sections of the specimen. The Zeiss Ultra-55 SEM was used for this study. The Zeiss Ultra 55 microscope optimized for high resolution imaging. Like most modern SEMs, the Zess Ultra 55 is capable of operating at beam energies from 100 V to 30 kV. The idea of SEM imaging is to observe cross section of specimen and determine how well polymer matrix diffuses into reinforced fiber. Figure 5.1 displays cross-section of fabricated sample and shows that the polymer is completely diffuses into fiber. By observing the image of the sample cross-section, it is concluded that there is no sign of delamination of the composite or air pockets in the laminate, as opposed to Figure 5.2 where the middle part of the laminate is not diffused with polymer. The Figure 5.2 represents the cross-section of unsuccessful manufacturing sample. The sample is unsuccessful because of insufficient curing time. In other words, the polymer did not have enough time to diffuse through the whole cross section of fiber.

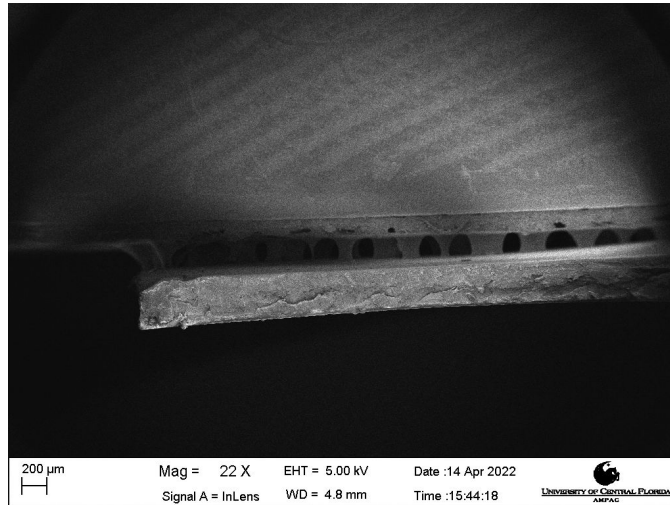


Figure 5.1: SEM Image of Sample Cross-Section, Magnification 22x

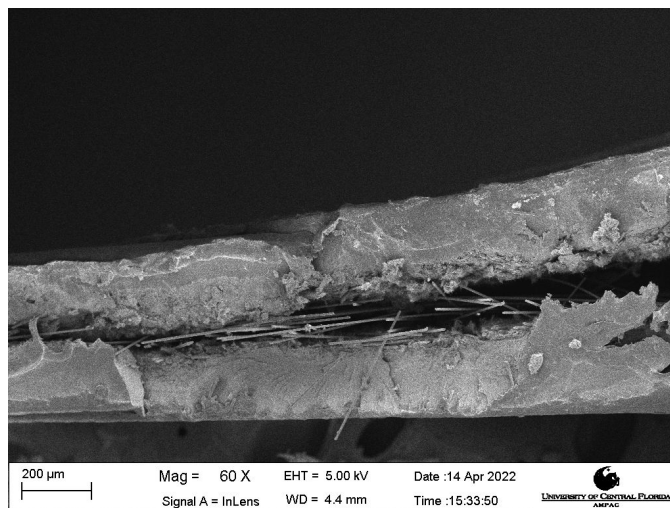


Figure 5.2: SEM Image of Sample Cross-Section, Magnification 60x

5.2 Energy Consumption During Composite Shape Changing

The calculation of energy required for changing state of polymer from solid state to liquid state is described by (3.6). The amount of energy needed to melt the polymer is represented as a sum of the latent heat and specific heat capacity of polymer. The values of specific heat capacity of PEEK polymer and specific latent heat of a substance of PEEK are obtained from the physical properties of PEEK polymer defined in [33]. The mass of the samples is measured using laboratory scale. The actual parameter values are listed in Table 5.2, and results are represented in Table 5.2. The amount of energy needs to melt the polymer during manufacturing process is 44.2 kJ.

Table 5.1: *Calculations Values*

Measure	Value	Unit
Mass of PEEK, m_p	57	g
Mass of CNP, m_{cnp}	23	g
Specific Heat Capacity of PEEK, C_p	1700	J/gK
Specific Heat Capacity of CNP, C_p	0.75	J/gK
Specific Latent Heat of a Substance, L	130	J/g

The value of specific heat capacity used in this calculation is obtained from the study of the specific heat and thermal conductivity of single-wall and multi-wall carbon nanotube [32]. The value of specific heat of CNP that is selected is for randomly oriented thin films of MWCNT tubes

which are best suited for this manufacturing process. The amount of energy stored in CNP during manufacturing process is 6.51 kJ.

Table 5.2: Results

Measure	Value	Unit
Energy Needed to Melt Polymer, Q_{melt}	50.71	kJ
Energy Stored in CNP, Q_{cnp}	6.51	kJ
Energy Needed to Shape-Changing, Q_{sc}	14.52	kJ
Energy Loss due Convection, Q_{loss}	5.32	kJ

The total energy required to heat the sample to glass transition temperature is calculated by using the following equation 3.7. The energy required for shape changing of the composite is calculated by sum of energy stored in CNP and specific heat capacity of PEEK polymer. Furthermore, the energy that leaves system due to free convection is estimated by the following equation 3.8. The average free convection heat transfer coefficient used in this calculation is $12.5 \text{ W/m}^2\text{K}$. The total amount energy needed to change shape of the composite is 19.8 kJ, where 5.3 kJ of energy leaves system to environment due to free convection heat loss. In future work, a similar experiment will be conducted where the shape changing will be performed in vacuum chamber. The advantage of this experiment will be reducing heat loss due to convection and performing shape changing in Space suitable environment.

The calculation shows that the amount of energy required for the manufacturing process is almost two times larger than the energy required for composite shape changing. According to Chapter 3,

the changing state of polymer from solid to liquid will required more energy compared to heating polymer to the softening point. Aside from the high melting point of polymer, the experiment shows that maintaining melting point of polymer for couple hours is necessary in order for polymer to completely diffuse into CNP. Since the manufacturing process requires melting of high temperature polymer and keeping the melting temperature during the diffusion process for some time, it is expected to consume more energy than composite shape changing. Most importantly, the experiments show that the heating polymer up to glass transition temperature is enough to soften the polymer for the composite shape changing process. Hence, the process of shape-changing requires less energy input than the actual manufacturing process. The idea of changing the geometric shape of material with small energy input is a promising solution for ISM.

CHAPTER 6

CONCLUSION

This work examines the use of conductive nanomaterial as integrated heaters during manufacturing process of thermoplastic composites. The idea of using carbon nano tubes as conductive heating element during thermoforming of thin-ply composite structures is driven by needs for developing ISM processes. The ability to perform ISM provides flexible and sustainable Space missions. The ability to manufacture structures in space instead of launching them from Earth will reduce logistic requirements of space missions by saving space and re-purposing the same material. Therefore, the focus of this study is developing manufacturing processes that will be suitable for Space manufacturing.

In manufacturing, the CNP is utilized as both fiber reinforcement and conductive heater while the composite matrix is thin PEEK film. The study shows that the CNP has the ability to transfer sufficient heat to melt high temperature polymers. Furthermore, the properties of thermoplastic polymers allows the repetition of thermoforming process of composite structures, in order to change geometric shapes of existing composite. Most importantly, the study shows that the energy required to change shape of composite structures is smaller than the energy required to melt polymer. Nowadays, the most research focuses on 3D printing of polymer or metals as a Space manufacturing process which also requires high energy consumption due to melting of materials

used in this process. However, the energy in Space is still in demand. Therefore, the idea of using composite pre-preg and forming the shapes by composite shape-chagrining process is a promising solution for ISM.

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