DEVELOPMENT OF PREDICTIVE MODELS FOR THE COALESCENCE OF
FUSED DEPOSITION MODELING FIBERS

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ABSTRACT

Fused deposition modeling (FDM) is the prominent manufacturing method for fabricating end-use parts due to the ability to build complicated structures. In order to be used confidentially in the industry requires a thorough understanding of mechanical behavior of FDM parts under working conditions. The strength of FDM parts is negatively influenced by the insufficient bond strength achieved between fibers, the weakest links in the FDM parts are the weak inter-layer bonds and intra-layer bonds. The aim of this study is to create models that can accurately predict bond length and bond strength between fibers. Analytical equations describing the sintering processes and heat transfer between FDM fibers and surrounding environment are developed and presented. By comparing the predicted value to the actual bond length, the models are found to be moderately accurate. To validate the relation between bond length and bond strength and also determine the process parameters that affect the bond strength, design of experiments (DOE) and analysis of variance (ANOVA) were applied. The result showed that the extrusion temperature to be statistically significant. Further research is recommended to take in to account more factors that could affect the cooling and sintering process that will help improve the precision of predictive models.

1. Introduction

1.1 Additive Manufacturing

There are numerous methods for fabricating components. The conventional manufacturing method constructed parts by removing material away from a solid block of material. In opposite to that, an emerging technology has been explored and become more favorable in manufacturing industry which is additive manufacturing.

Additive manufacturing(AM) has much more advantages than conventional manufacturing method. The highlight benefit of AM is the ability to build complicated geometries without any extra tools at very short time. In fact, to construct an object with complicated structure, traditional manufacturing takes days to complete, it also requires at least three cutting tools and professional machine users. In addition to that, cutting tool will be wear after limited uses that require replacement. On the other hand, AM takes hours to complete the same task, works without tooling and require no professional training to operate the machine.

Additive manufacturing builds objects by adding layer upon layer of material until the object is completely built. This can be accomplished by various methods such as SLS, SLA, FDM. Selective Laser Sintering (SLS) uses a laser beam to heat and melt thermoplastic powder into a continuous bonding layer. SLA on the other hand build object in a pool of resin. A laser beam is directed into the pool of resin, the trajectory of the beam following the same cross-section pattern of the object. Different to the other methods, fused deposition modeling (FDM) extruded melted polymer filaments through a heated extrusion

nozzle onto the build platform, the deposited filaments formed layers that exact the same as cross-sectional of the desired object.

1.2 Fused Deposition Modeling

Among the variety of additive manufacturing method, FDM is superior to SLS and SLA. SLS requires a very complicated and expensive laser system to avoid oval projection on the polymer powder bed to produce acceptable precision products. SLA can only work with very limited types of material which are UV sensitive material. FDM has much simpler mechanism compare to the others. FDM also compatible with wide variety of material from plastic to metal or even biomaterial.

The input material for 3D printer is polymeric filament which is continuously fed into the printer using drive wheels. The filament then goes through liquifier which is a heating element. Liquifier is heated to the temperature higher than glass transition temperature of the filament causing the filament to melt. Molten then be extruded through extrusion nozzles onto a platform or other layers to generate desired objects. Once the filament has extruded through the nozzle, it has very small diameter so it is called the fiber. The nozzle is moved in horizontal directions along the x- and y-axis under control of the computer at a constant speed while printing. The moving speed of the nozzle is called printing speed. The platform moves in the vertical direction. The system is covered in a chamber/oven where the temperature is under control. One of the biggest obstacles for FDM technology to be used for high-end applications is that parts printed by FDM technology have weaker mechanical properties compare to parts produced by conventional techniques such as injection molding [1]. The reason for reducing in mechanical properties of FDM parts was put under investigation.

1.3 Weak Mechanical Properties of FDM part

Ahn et al. found that the manufacturing parameters that included extruded fiber geometry, the orientation of the fiber, the extrusion temperature had significant effects on tensile strength of part printed by FDM printer [1]. Reddy et al. found that road gap and chamber temperature had high impact on part strength [2]. Turner and Wang pointed out that thermal gradient inside the part while printing causes warping and internal stress that decrease the part strength [3] [4].

The weak strength of FDM parts is proved to be due to inadequate bond strength between fibers [5] [6] [7]. The weak inter- and intra-layer strength in the building direction often being the weakest and most critical link in the FDM parts. Figure 1.1 shows inter- and intra-layer bonding in FDM.

Figure 1. 1 Inter- and intra- layer bonding in FDM.

The bonding quality depends on the size of the neck form between the adjacent fibers and on the strength of bond that depends on molecular diffusion of the polymer chains across the interface. Molecular diffusion process at the interface between fibers while sintering is call healing process. The healing process takes place in five steps as shown in figure 1.2 [8] [9].

- (1) Surface rearrangement, (2) Surface approach: Intimate contact between the polymer surfaces is achieved after surface rearrangement and surface approach takes place.
- (3) Wetting: One surface then begins to wet the other leading to the increase of neck size between fibers. After completely wetted, the neck size between fibers will remain constant
- (4) Diffusion: Strength is then developed due to the diffusion of polymer chains across the interface. The mechanical properties of bonding reach the properties of the virgin material only when the polymer chains have penetrated across the interface to the equilibrium state.
- (5) Randomization: The polymer chains continue to diffuse and mix with the other body without any increase in the mechanical properties of the bond in randomization step [9]

Figure 1.2 Healing processes between fibers [10].

The main reason that the bond has lower strength than virgin material is the discontinuous nature of the healing process, initially, molten fiber comes into contact with one another but the temperature of both fibers drop rapidly under glass transient temperature cause them to solidify before the polymer chain to be diffused completely.

One solution to this problem is post-processing. Samples will be printed and put in the heating chamber for a specific duration. More details will be discussed in section 4.

1.4 Methods for Improving FDM Mechanical Properties

Much work has already been done in the area of minimizing the effects of the weak bond strength. Bellini et al. found the way to improve the strength of FDM parts by optimizing the orientation of the fiber to minimize load transfer through fiber bond [7]. Nikzad et al. successfully increased tensile strength and storage modulus of ABS parts by adding 30% in volume of copper and iron to ABS. That helps improve thermal conductivity between fibers thus increase diffusion of polymer chains across the contact interface [11]. The works of Zhong and Shofner proved that the addition of short fibers into printing material can help increase the part strength [12] [13]. In order to increase the sintering time between fibers, Partain locally heated the fibers surrounding extruding nozzle by using forced hot air, but the result showed no significant increase in bond strength [14].

While these works were successful optimizing manufacturing factors that can improve the strength of FDM parts, models capable of accurately predicting the bond length and bond strength are still missing.

2. Literature Review

2.1 Sintering Model Applied to FDM wetting

As mention previously, bond length is formed during the wetting process. Wetting was studied in FDM using sintering model. Frenkel built a sintering model by assuming work of surface tension equal to work done by viscous force. The model neglected other factors such as gravity [15]. Base on Frenkel work, Pokluda developed an equation for calculating the ratio of neck radius to initial radius of sphere polymer particle. Comparing ratio of predictive model to experimental data gave a great match [16]. Pokluda model was applied to FDM to predict the bond length between FDM fibers.

$$\frac{d\theta}{dt} = \frac{\Gamma}{a_o\eta} \frac{2^{-\frac{5}{3}}\cos\theta\sin\theta(2 - \cos\theta)^{1/3}}{(1 - \cos\theta)(1 + \cos\theta)^{1/3}} \quad (2.1)$$

Where Γ is the coefficient of the surface tension of printing material, η is the temperature-dependent viscosity, θ is the bond angle, a_o is the initial radius of cylindrical filament before sintering process happened.

Bonding length is then calculated

$$y = a_o\sin\theta \left(\frac{4}{[1 + \cos\theta]^2[2 - \cos\theta]} \right)^{1/3} \quad (2.2)$$

The limitation of Pokluda model was that the model built on an assumption of two spherical particles. That led to an inaccuracy prediction of bond length.

2.2 Heat Transfer Analysis across Fibers

Cooling process of fiber is very important for predicting bond length because viscosity η depends on the temperature of fibers. It also important for predicting bond strength because it affects wetting, diffusion process. Heat transfer process between FDM fiber and the ambient has been widely studied.

Li et al presented a heat transfer model in which FDM fiber is assumed to be semi-infinite rod length. Under that assumption, temperature varied along the length of the fiber, and temperature at cross section was uniform which means the temperature at the core of the fiber was the same as the temperature at the fiber surface. The model only applied to the bottom layer that adjacent to the platform. Heat transfer between fiber and ambient air is natural convection. Heat transfer between fiber and platform was assumed to be convection. As such, temperature profile varied significantly with convection coefficient and heat conduction between fiber and platform was underestimate [5].

Thomas et al developed a 2D heat transfer model which took into account the temperature gradient along the cross-section and the length of the fibers. They assumed the fiber has a rectangular geometry. With that assumption, heat transfer conduction will be overestimated. The heat transfer model showed that the temperature gradient between the core of the fiber and the surface will increase dramatically under the increase of convection coefficient. The model also pointed out the size of the fiber is an important factor that needs to be considered as from their model, 0.25mm diameter ABS molten fiber took 1.7s to reach glass transition temperature compared to 15s of 1mm diameter fiber [17].

Sun et al compared the heat transfer models developed by Thomas and Li to the actual experiment. Li's model was found to be underestimated conduction. As a result, the fiber cooled too slowly at the beginning then cooled too rapidly. On the other hand, Thomas's model was found to underestimate the convection. That led to the cooling happened too slowly and remain at high temperature for too long [6].

Costa et al used FEA to perform an analysis of the temperature contribution in FDM fiber. There were six types of heat transfer taken into account: convection with air, conduction with adjacent fibers, conduction with the platform, radiation with the ambient, radiation with adjacent filaments, convection with air pockets. Convection with air and conduction with platform were found to have the biggest effect on fiber cooling [18].

3. Methodology

3.1 Bonding Model

The strength of parts printed by FDM printer depends on the bond length and the strength of the bond between fibers. Therefore, a model that can predict bond length is developed.

3.1.1 Bonding Equation

By equating the work of surface tension to the viscous dissipation, a Newton sintering model proposed by Pokluda et al [16].

$$\frac{d\theta}{dt} = \frac{\Gamma}{a_o\eta} \frac{2^{-\frac{5}{3}}\cos\theta\sin\theta(2 - \cos\theta)^{1/3}}{(1 - \cos\theta)(1 + \cos\theta)^{1/3}} \quad (3.1)$$

In which, Γ is the coefficient of the surface tension of printing material.

η is the viscosity of printing material which will be calculated in the next section due to temperature dependent property.

θ is the bond angle,

r_o is the initial radius of filament before sintering process happened.

Figure 3. 1 Evolution of bonding between fibers

In this thesis, FDM filaments are elliptical so r_o is considered to be the equivalent radius of the ellipse fiber. Figure 3.1 presented shape evolution of two filaments bonding together.

For $\theta \rightarrow 0$. The following approximation can be made [16]:

$$\sin(\theta) = \theta \quad (3.2)$$

$$(1 - \cos \theta) = \theta^2 / 2 \quad (3.3)$$

$$\theta' = \frac{1}{2} \frac{\Gamma}{\eta r_o \theta} \quad (3.4)$$

With the initial condition $\theta(0) = \theta_0 = 0$, the solution for equation is

$$\theta(t) = \left(\frac{t\Gamma}{\eta r_o} \right)^{1/2} \quad (3.5)$$

The initial boundary condition is fixed at a time value slightly different than zero to overcome numerical instabilities when $\theta=0$.

The value of θ_0 is determined from equation 3.5

Bonding length is then calculated

$$y = r_o \sin \theta \left(\frac{4}{[1 + \cos \theta]^2 [2 - \cos \theta]} \right)^{1/3} \quad (3.6)$$

The aforementioned bonding equation is derived using the assumption that the two particles are spherical.

For better bonding prediction, a model was developed to predict bond length between 2 cylindrical fibers [19].

Bond length is defined as

$$y = r \sin \theta \quad (3.7)$$

Radius of the fiber after time t is obtained as

$$r = \frac{r_0 \sqrt{\pi}}{\sqrt{\pi - \theta + \sin \theta \cos \theta}} \quad (3.8)$$

Work of surface tension of the fiber is given as

$$W_s = -\Gamma \frac{dS}{dt} \quad (3.9)$$

Where S is the instantaneous cross-sectional area at time t

$$S = 2lr \sin \theta \quad (3.10)$$

The work of viscous force

$$W_v = 6\pi r_0^2 l \eta \frac{(\pi - \theta)^2 \sin^2 \theta}{[\pi - \theta + \sin \theta \cos \theta]^2} \dot{\theta}^2 \quad (3.11)$$

Assume that work of surface tension equal to work of viscous forces

$$\frac{d\theta}{dt} = \frac{\Gamma}{3\sqrt{\pi} r_0 \eta} \frac{[(\pi - \theta) \cos \theta + \sin \theta][\pi - \theta + \sin \theta \cos \theta]^{1/2}}{(\pi - \theta)^2 \sin^2 \theta} \quad (3.12)$$

Where, r_0 is initial radius of FDM fiber

For $\theta \rightarrow 0$. The following approximation can be made:

$$\sin(\theta) = \theta \quad (3.13)$$

$$(1 - \cos \theta) = \theta^2 / 2 \quad (3.14)$$

$$\theta' = \frac{1}{2} \frac{\Gamma}{\eta r_0 \theta} \quad (3.15)$$

With the initial condition $\theta(0) = \theta_0 = 0$, the solution for equation is

$$\theta(t) = \left(\frac{t\Gamma}{\eta r_0} \right)^{1/2} \quad (3.16)$$

The initial boundary condition is fixed at a time value slightly different than zero to overcome numerical instabilities when $\theta=0$.

Differential equation 3.12 was solved using 4th order Runge-Kutta method.

Bonding angle θ is calculated using equation 3.17

$$\theta_{i+1} = \theta_i + \frac{1}{6} \Delta t (k_1 + 2k_2 + 2k_3 + k_4) \quad (3.17)$$

In which Δt is time step. For this case, Δt is set equal to $2 \cdot dt$. dt is the time step that is used for the interval loop

$$t_{i+1} = t_i + \Delta t \quad (3.18)$$

$$k_1 = f(t_i, \theta_i) \quad (3.19)$$

$$k_2 = f\left(t_i + \frac{1}{2}\Delta t, \theta_i + \frac{1}{2}\Delta t k_1\right) \quad (3.20)$$

$$k_3 = f\left(t_i + \frac{1}{2}\Delta t, \theta_i + \frac{1}{2}\Delta t k_2\right) \quad (3.21)$$

$$k_1 = f(t_i + \Delta t, \theta_i + \Delta t k_3) \quad (3.22)$$

By solving equation 3.12, the bonding angle between fibers is obtained. Substitute the value of bonding angle to equation 3.7 we can calculate the bond length between fibers.

To improve the accuracy of the predictive model this research will include temperature dependent properties. To be specific, in equation 3.12 viscosity is a function of temperature. Next section will determine the value of viscosity according to the specific temperature of fibers.

3.1.2 Temperature-Dependent Viscosity

Polycarbonate is an amorphous material that has the viscosity varies with the temperature. Viscosity value is approximated as

$$\eta = \eta_r \exp(-0.01(T - T_r)) \quad (3.23)$$

Where, T is the absolute temperature

η_r is the viscosity at the reference temperature, T_r

Figure 3. 2 Viscosity versus temperature of PC

The temperature of fiber has to be determined in order to get the accurate viscosity. The next section will present a thermal model that provides the temperature of the fiber at the specific time.

3.2 Thermal model

Fiber geometry, temperature dependent material properties and convection heat transfer coefficient are factors that affect the heat transfer between fiber and the ambient. In order to get the temperature profile of fiber, these factors need to be determined.

3.2.1 Fiber Geometry

Under inspection of FDM fiber using scanning electron microscope (SEM). FDM fiber has an elliptical shape. Thus, area and perimeter of FDM fiber are calculated using formulas for the ellipse. These parameters are given by

$$A = \pi ab \quad (3.24)$$

$$P = \pi(a + b) \left(1 + \frac{3h}{10 + \sqrt{4 - 3h}} \right) \quad (3.25)$$

Where h is defined as

$$h = \frac{(a - b)^2}{(a + b)^2}$$

a is the major axis and b is the minor axis (as shown in Fig. 3.3). For this study, the values for these axes are 0.2 mm and 0.1 mm

Figure 3. 3 Graphical representation of the elliptical shape of a deposited fiber

3.2.2 Temperature profile of the fiber

Since the diameter of the fiber is fairly small compared to its length, the cooling process of the extruded fiber can be model using lumped system analysis [5]. Assume that

the temperature distribution at the cross-section of the fiber is uniform. Thus the cooling process of FDM fiber can be simplified into one-dimensional transient heat transfer model.

Figure 3. 4 Schematic of Deposition of FDM Fiber

Figure 3.4 is the schematic of the FDM extrusion process. In the FDM process, a typical road of FDM extrusion tip has a length of more than hundred times of fiber diameter. Therefore, the road can be assumed to be the semi-infinite line. When extruding, the head moves at a constant speed v along the x -axis. The reference coordinate has origin which is set at the tip of the extrusion nozzle.

A differential element of thickness dx is put under energy transfer analysis as follows:

The rate of heat transfer out the left face of the differential element:

$$\dot{E}_{out} = -kA \frac{\partial T}{\partial x} \quad (3.26)$$

The rate of heat transfer in the right face of the differential element:

$$\dot{E}_{in} = -kA \frac{\partial T}{\partial x} \Big|_{x+dx} = -A \left[k \frac{\partial T}{\partial x} + \frac{\partial \left(k \frac{\partial T}{\partial x} \right)}{\partial x} dx \right] \quad (3.27)$$

Convection heat transfer rate between the differential element and air:

$$\dot{Q}_{convection} = h(P - P_1)(T - T_{\infty})dx \quad (3.28)$$

Conduction heat transfer rate between differential element and foundation sheet:

$$\dot{Q}_{conduction} = -k_{Nylon}P_1(T - T_f)w dx \quad (3.29)$$

The rate of change of the energy content of the element:

$$\frac{\Delta E_{element}}{\partial t} = \rho CA \frac{\partial T}{\partial t} dx \quad (3.30)$$

Where: T is the averaged cross-section temperature, T_{∞} is the oven temperature, T_f is the foundation temperature, T_0 is the extrusion temperature, k is thermal conductivity of fiber, C is thermal heat capacity of fiber, ρ is density of the material, P is the perimeter of the fiber cross-section and P_1 is the cross-sectional contact length between the fiber and the foundation.

Energy balance on the element during small time interval can be expressed as:

$$\dot{E}_{in} - \dot{E}_{out} - \dot{Q}_{convection} - \dot{Q}_{conduction} = \frac{\Delta E_{element}}{\partial t} \quad (3.31)$$

$$-\frac{\partial \left(k \frac{\partial T}{\partial x} \right)}{\partial x} - h(P - P_1)(T - T_{\infty}) + k_{Nylon}P_1(T - T_f)w = \rho CA \frac{\partial T}{\partial t} \quad (3.32)$$

The origin moves at a velocity v , $x=vt$

Time dependence term $\partial T/\partial t$ can be transformed as:

$$\frac{\partial T}{\partial t} = \frac{\partial T}{\partial x} \frac{\partial x}{\partial t} = \frac{\partial T}{\partial x} v \quad (3.33)$$

Substitute into Equation 3.32,

$$-\frac{\partial \left(k \frac{\partial T}{\partial x} \right)}{\partial x} - h(P - P_1)(T - T_\infty) + k_{Nylon} P_1 (T - T_f) w = \rho C A \frac{\partial T}{\partial x} v \quad (3.34)$$

Assume that the temperature of the oven is equal to the temperature of the foundation.

The boundary conditions for non-homogeneous second order equation are:

$$T=T_o, \quad x = 0, \quad t \geq 0 \quad (3.35)$$

$$T=T_\infty, \quad x = \infty, \quad t > 0 \quad (3.36)$$

The solution for equation 3.34 with the boundary conditions in equations 3.35 and 3.36 is as follows:

$$T = T_\infty + (T_o - T_\infty) e^{-nvt} \quad (3.37)$$

Where, v is deposition velocity,

$$n = \frac{\sqrt{1 + 4\alpha\beta} - 1}{2\alpha} \quad (3.38)$$

$$\alpha = \frac{k}{\rho C v} \quad (3.39)$$

$$\beta = \frac{h(P - P_1) + k_{Nylon}P_1w}{\rho CA v} \quad (3.40)$$

Thermal conductivity k and heat capacity C of the fiber are not constant, they vary with the temperature of the fiber. The next sections will present methods to determine each factor in the cooling model.

3.2.3 Temperature Dependent Thermal Conductivity and Heat Capacity

Temperature dependent thermal conductivity and temperature dependent heat capacity are properties that have to gather by experimental work. The data was obtained from DatapointLabs [20] then put into matlab for fitting. A spline was created to fit the data points provided by DatapointLabs (as shown in Fig. 3.5-Fig. 3.6) (for raw data see Appendix A).

Figure 3. 5 Thermal conductivity versus temperature for PC

Figure 3. 6 Specific heat capacity versus temperature for PC

The interpolated material properties values at each time step can be obtained from the spline fit.

3.2.4 Convective Heat Transfer Coefficient

The FDM fiber printed horizontally on the foundation in a fully cover chamber. The chamber temperature was under control. Heat transfer between fiber and ambient air in the chamber is natural convection.

The convective heat transfer coefficient is

$$h = \frac{k \cdot Nu}{D} \quad (3.41)$$

in which, k is the thermal conductivity of air which is given by

$$k = 0.02624 \left(\frac{T_K}{300} \right)^{0.8646} \quad (3.42)$$

D is characteristic length of the fiber

$$D = 2\sqrt{ab} \quad (3.43)$$

a, b are major and minor axes of the elliptical fiber.

The fiber is assumed to be a horizontal cylinder experiencing natural convection. The value for Nusselt number is calculated using equation develop by Churchill [21].

$$Nu = \left\{ 0.60 + 0.387 \left[\frac{GrPr}{\left(1 + \left(\frac{0.559}{Pr} \right)^{9/16} \right)^{16/9}} \right]^{1/6} \right\}^2 \quad (3.44)$$

Where:

Pr is the Prandtl number that varies with temperature. For air, Prandtl number can be calculated as [22]

$$Pr = 0.68 + 4.69 \times 10^{-7} (T_{\infty} - 540)^2 \quad (3.45)$$

Gr is the Grashof number, which is defined as

$$Gr = \frac{g\beta(T_s - T_{\infty})D^3}{\nu^2} \quad (3.46)$$

Where,

T_s is the surface temperature of the fiber. From the previous assumption, temperature of the cross-section of the fiber is uniform which means $T_s = T$

g is gravitational constant

β is the volumetric thermal expansion coefficient. Volumetric thermal expansion coefficient of air is given by [22]

$$\beta = \frac{1}{T_\infty} \quad (3.47)$$

ν is the kinematic viscosity. By definition,

$$\nu = \frac{\mu}{\rho} \quad (3.48)$$

The dynamic viscosity μ is given by

$$\mu = \frac{1.458 \times 10^{-6} T_\infty^{1.5}}{T_\infty + 110.4} \quad (3.49)$$

At absolute pressure P (N/m^2) the density ρ is

$$\rho = \frac{P}{R_A T_\infty} \quad (3.50)$$

The specific gas constant is

$$R_A = 287.05 \text{ J/kgK} \quad (3.51)$$

Matlab was used to solve the cooling and bonding model (see Appendix B). With the help of matlab, properties of air and the polymer that depend on temperature were taken into account to obtain an accurate prediction for fiber temperature history and bond length.

4. Model Validation

Validating the model requires comparing predicted results with actual results.

4.1 Materials and Equipment for Printing the Sample

A Fortus 400mc from STRATASYS was used to print the samples. Fortus 400mc is capable of print multiple production-grade thermoplastics, such as ABS, PC, PPSF, ULTEM, and more. It has a print volume of 406×356×406 mm, and it can print at speeds up to 200mm/s with a layer resolution of 100-450μm. The printer has a controlled environmental chamber and a controlled temperature foundation.

The other 3D printing machine used to print the sample was Creatbot DX. Creatbot is capable of print ABS, PC and PLA. It has a print volume of 300×250×520 mm, and it can print at speeds up to 120mm/s with a layer resolution of 500μm. The printer does not have a controlled temperature chamber but it has a controlled temperature foundation. A 0.4mm diameter nozzle was used to printed sample.

All the sample were printed in Polycarbonate. Polycarbonate has glass transition temperature of 147⁰C, so it softens gradually above this point and flows above 155⁰C

4.2 Sample preparation

4.2.1 Sample for Image Analysis

To validate the cooling and bonding model, samples were built from Polycarbonate under various temperature conditions shown in table 4.1. There are two factors: extrusion temperature and oven temperature and 3 levels of temperature with the increment value of 5 degree.

Table 4. 1 Experimental matrix for image analysis

Experiment No.	Extrusion Temperature (K)	Oven Temperature (K)
1	543	373
2	543	378
3	543	383
4	548	373
5	548	378
6	548	383
7	553	373
8	553	378
9	553	383

Each sample is printed in the same dimension shown in Figure 4.1. The thickness of each sample was just a single fiber height. Printing speed was fixed at 30mm/s.

Figure 4. 1 Configuration for image analysis samples

4.2.2 Sample for Tensile Testing

Table 4. 2 Experimental matrix for tensile testing

Experiment No.	Extrusion Temperature (K)	Oven Temperature (K)
1	543	373
2	543	403
3	543	433
4	573	373
5	573	403
6	573	433
7	603	373
8	603	403
9	603	433

The printing temperature for nine samples was shown in table 4.2. The sample for tensile testing follows ASTM D638 standard. The dimension of the sample is shown in figure 4.2. The thickness of each sample was just a single fiber height.

Figure 4. 2 Dimension for tensile testing sample

The orientation of the fiber in the tensile testing sample is shown in figure 4.3.

The printing speed was fixed at 30mm/s.

Figure 4. 3 Orientation of fiber

4.2.3 Sample for Post-processing

Nine samples were printed at the printing temperature of 543K and oven temperature of 373K. Then individual sample was put in the heating chamber and heat at a specific temperature for a specific duration. The heating temperature and heating time for each sample is shown in table 4.3

Table 4. 3 Temperature and time setting for post-processing experiment

Experiment No.	Baking Temperature (K)	Baking Time (min)
1	423	5
2	423	15
3	423	25
4	473	5
5	473	15
6	473	25
7	523	5
8	523	15
9	523	25

After annealing, the chamber was left to cool down naturally. After the chamber temperature reached the room temperature. The sample was taken out for image analysis and tensile testing.

4.3 Tensile Testing

The tensile testing was done on an MTESTQuattro Material Testing System with MTESTQuattro software. Figure 4.4 shown MTESTQuattro Material Testing System.

Figure 4. 4 MTESTQuattro Material Testing System

The strain on MTESTQuattro Material Testing System is calculated from displacement and original length instead of measured directly with a strain gauge.

The testing procedure is as follows:

1. Turn on the MTESTQuattro Material Testing System and the computer.
2. Open MTESTQuattro software and load the Devlin tensile testing file in the procedure folder.

3. Control the grip to the starting position by turning the directional knob to “DN” and waiting until it reaches the measured length of the sample.

4. Load the specimen into the machine. Oriented the sample vertically at the center of the grip to avoid any shear or twisting while the tensile testing is in progress (see Fig. 4.5). Tighten the gripper by hand with the specimen properly oriented.

Figure 4. 5 Image of properly load samples

5. Set up the testing speed to 5.8 mm/min in the MTESTQuattro software. Zero all the value by pressing “0” on the interface window.

6. Start testing process by pressing the “play” button in the user interface window of MTESTQuattro software.

7. The testing is automatically. The machine was set up to stop when the sample was broken. After the sample was broken, export the data in image and excel format for later analysis. Figure 4.6 shows the exported data from the software.

Figure 4. 6 Stress versus position graph exported from the MTESTQuattro software

8. Remove the tensile specimen from the machine.

9. Repeat steps 3-11 for all specimens.

4.4 Image Analysis

The Quanta 600F ESEM shown in figure 4.7 was used for image analysis.

Figure 4. 7 The Quanta 600F ESEM system

Sample Preparation

1. Place samples on the holder stub using a double side sticky tape
2. Coating the sample with Platinum. The coating machine was EMS150T ES (as shown in figure 4.8). The thin of the coating is only 25nm.

Figure 4. 8 Samples in the coating chamber

3. Marks on the sample stub before put it in the microscope because it is very hard to recognize similar samples in the SEM.

Loading the sample into the SEM

1. To open the chamber door, click on the “vent” button to fill air in the chamber
2. Put the sample stub into the mounting holes and tighten the screws for the mounting holes (as shown in figure 4.9).

Figure 4. 9 Fixing the sample holder into the mounting hole of the SEM

3. Close the door and then press the EVAC button. Wait for about 3 minutes for the chamber to reach vacuum state.

Turning on the SEM

1. When vacuum reaches proper level, the green light will turn on. Set the acceleration voltage to 15 KV.
2. Click on the “image acquire” to record the image. Save the image as the .tif format. After all the images are obtained, they are processed with ImageJ.

The complete procedure for processing the images is:

1. Using ImageJ, open an FDM part image
2. Draw a line directly over the top of the image scale. Use the “Set Scale” option in ImageJ to assign the number of pixels in the line to the image scale

Figure 4. 10 Setting scale for the imagej

3. Draw a line over each bond to be measured for length. Measure the bond by combine button “Ctrl+M”

4. Export all measurements to Microsoft Excel for later analysis.

The observed bond lengths are compared to those predicted by the combination of the thermal and bonding models to validate the overall model accuracy.

5. Case Studies

5.1 Cooling and Bonding Result

Figure 5. 1 Predicted cooling at $T_0=543\text{K}$, $T_\infty=373\text{K}$

Figure 5. 2 Predicted bonding at $T_0 = 543\text{K}$, $T_\infty = 373\text{K}$

Figure 5. 3 Predicted cooling at $T_0 = 546\text{K}$, $T_\infty = 383\text{K}$

Figure 5. 4 Predicted bonding at $T_0 = 546\text{K}$, $T_\infty = 383\text{K}$

Figure 5. 5 Predicted cooling at $T_0 = 553\text{K}$, $T_\infty = 383\text{K}$

Figure 5. 6 Predicted cooling at $T_0 = 553\text{K}$, $T_\infty = 383\text{K}$

5.2 Observation of Bond Length Using SEM.

The bond lengths from the bonding model were obtained from matlab, the result will be put on table 5.1. By analyzing the SEM images, the actual bond length is gathered and compared to the predictive bond length to validate the accuracy of bonding models.

Image of a sample printed by FDM under SEM is showed in Fig 5.7

Figure 5. 7 Image of the mesostructure of FDM sample

The comparative analysis of predicted and realized bonding is seen in Table 5.1.

Table 5. 1 Comparison of predicted and actual bond lengths

Experiment No.	Predicted Bond Length (mm)	Observed Bond Length (mm)	% Error	% Error from the old model
1	0.2812	0.2428	15.81	99
2	0.2813	0.2521	11.58	41
3	0.2813	0.2550	10.31	40
4	0.2820	0.2612	7.96	36
5	0.2822	0.2612	7.96	36
6	0.2823	0.2613	8.03	36
7	0.2827	0.2620	7.9	36
8	0.2828	0.2619	7.98	36
9	0.2829	0.2630	7.56	35

Table 5.1 shows that the predicted bond length match well with the observed bond length with the maximum error 15.81%.

5.3 Miniature-tensile test

The results from tensile tests are summarized in table 5.2. The result showed the behavior of bond under tensile load.

Table 5. 2 Result of tensile tests conducted according to the L9 Taguchi matrix

Experiment No.	Maximum Tensile Stress (MPa)	Bond Length (mm)
1	40.11	0.2428
2	40.15	0.2521
3	40.23	0.2550
4	41.45	0.2612
5	41.57	0.2612
6	41.78	0.2613
7	42.01	0.2620
8	41.98	0.2619
9	41.87	0.2630

5.4 Miniature-tensile Test for Post-process Samples

The maximum tensile stresses obtained from the miniature tensile tests are shown in Table 5.3. As seen from the result, the post-processing has positive effects on tensile strength if it is heated under 200°C. The comparison of the maximum tensile stress of sample under different settings of post-process is shown in Table 5.3.

Table 5. 3 Maximum tensile stresses of post-processed specimens

Experiment No.	Maximum Tensile Stress (MPa)	Bond Length (mm)
Not Post-processed	40.11	0.2428
1	40.23	0.2437
2	41.32	0.2442
3	42.37	0.2450
4	42.51	0.2454
5	42.76	0.2454
6	42.87	0.2457
7	38.75	0.2701
8	38.68	0.2711
9	38.54	0.2723

Table 5.3 shows that annealing does increase the length of bonds between fibers, but in some cases, the bond strength decreased. The main reason and further discussion will be explained in the next section.

6. Discussion

6.1 Cooling and Bonding Models

By reviewing Table 5.1, the predictive bonding and cooling model shown an acceptable result compare to the observed bond length. The maximum error was 15.81% Contribution to the error of the cooling model are the missing of factors in the cooling equation. The missing factors are thermal radiation between fibers and the environment, heat conduction between fibers and contact resistances between fibers and foundation, fiber versus fiber. The error in the bonding model was also derived from the missing of gravitational force.

Base on the cooling and bonding model, bond length depend on the properties of printing material, temperature of oven and extrusion temperature. To determine the factor that has the most impact on bond length, a response plot was created (as shown in Figure 6.1).

Figure 6.1 Response plot showing the effect of fabrication parameters on bond length

The response plot shows a steep slope through three levels of extrusion temperature. With the increase of the extrusion temperature, the bond length increase dramatically. On the other hand, the bond length improved gradually when increasing the oven temperature. That means the extrusion temperature contribute the most to the increase of the bond length between fibers. In order to confirm that, analysis of variance was conducted. The result showed that extrusion temperature contributes over 92% to the bond length of the samples built from polycarbonate (oven temperature contributes only 8%). Extrusion temperature increased the duration that fiber will stay at the sintering temperature. Bellehumeur et al. proved that the neck growth is negligible at the temperature below sintering temperature [23]. The bonding model shown that increasing the extrusion temperature leads to an increase in the length of time at which the fiber maintained at the temperature above the sintering temperature.

6.2 Bond Strength Between FDM Fiber

According to bond strength model, final strength depends on the diffusion coefficient which is a material property, extrusion and oven temperature. In which, extrusion temperature is the most important factor. Due to the result shows in table 5.2, the higher the extrusion temperature the stronger the part is. The response plot shows a steep slope with extrusion temperature through 3 levels. With the increase of the extrusion temperature, the bond strength increases dramatically. On the other hand, the bond strength improved gradually when increasing the oven temperature. That means the extrusion temperature contribute the most to the increase of the bond length between fibers. In order to confirm that, analysis of variance was conducted. The result shows that extrusion temperature contributes over 84% to the bond strength of the samples built from polycarbonate (oven temperature contributes only 16%).

Figure 6. 2 Response plot showing the effect of fabrication parameters on part strength

With higher printing temperature, the strength of bonding increase. The reason for that is explained base on the healing process of fiber. The healing process is taking place in five steps: (1) Surface rearrangement, (2) surface approach, (3) wetting, (4) diffusion, (5) randomization as shown in figure 6.3 [2]

Figure 6. 3 Healing processes between fibers [10].

Intimate contact between the polymer surfaces is achieved after surface rearrangement and surface approach take place. One surface then begins to wet the other leading to the increase of neck size between fibers. After completely wetted, the neck size between fibers will remain constant. Strength is then developed due to the diffusion of polymer chains across the interface. The mechanical properties of bonding reach the properties of the virgin material only when the polymer chains have penetrated across the interface to the equilibrium state. The polymer chains continue to diffuse and mix with the other body without any increase in the mechanical properties of the bond in randomization step

Initially molten fiber comes into contact with one another but the temperature of both fibers drops rapidly under glass transient temperature cause them to solidify before completely fusing with the other. By increasing the extrusion temperature, the temperature

of fiber will be kept at high temperature for the longer time causing the diffusion to happen completely that lead to the improvement of bond strength.

6.3 Post-process of FDM Part

Annealing at the temperature below 250°C result in the increasing of both bond length and bond strength. While heating above 250°C tends to reduce the bond strength. PC is proved to experience thermal degradation at high temperature. Thermal degradation of PC is caused by chain scission of the isopropylidene linkage, and hydrolysis and decarboxylation of carbonate linkage [24] [25] [26] [27]. Hydrolysis is the scission of chemical functional groups by reaction with water.

In addition to that, pictures from SEM show that many micro-bubbles that formed between fibers. These bubbles apparently increase bond length between fibers but reduce the maximum load that bonding can withstand. PC tends to retain moisture from the air, during the post process at very high temperature micro-bubble was formed and prevented sintering process between fibers.

Post-process of FDM parts after printing showed a potential result in increasing the bond strength between fiber but thermal degradation and micro-bubbles need to be taken into account.

7. Conclusions

In this work, heat transfer process during the extrusion of FDM fiber on the platform was analyzed so the temperature profile of the extruded fiber was obtained. Based on polymer sintering model and heat transfer analysis, bond length between FDM fibers was predicted with the acceptable result. The manufacturing parameters that influence the bond formation in the FDM process are extrusion temperature, oven temperature and dimensions of the extruded fibers. According to the predictive model and experimental result, design of experiments and analysis of variance was conducted to determine the most important factor that affects bond formation. The results showed that extrusion temperature has more significant influence on the growth of bond than oven temperature does. The extruded fibers cannot be maintained at high temperature long enough for the completion of the diffusion process. As a result, the mechanical properties of the bond between fibers are lower than original material.

The tensile test showed the dependence of bond strength on extrusion temperature and oven temperature. In which, oven temperature play an important role in the increasing of bond strength. The strength of FDM part depends on the bond strength between fibers. Bond strength is a complicated function that depends on the diffusion coefficient of material, cross-section of the wetted area, degree of intimate contact between fibers.

The bond strength was calculated by equation built by Timothy, Wool [8] [9] [10].

$$\sigma = f_{wetting} \left[\sigma_o + (\sigma_{\infty} - \sigma_o) \left(\frac{D_{pre}}{D_{max}} \right)^{\frac{1}{4}} \right]$$

Where $f_{wetting}$ is a function that includes wetted area

$$f_{wetting} = \frac{bond\ width}{fiber\ width}$$

D_{max} is the diffusion amount before the bond strength has the same strength as the virgin material

D_{pre} is the total diffusion distance that polymer chains travel

$$D_{pre} = D_s * t$$

D_s is diffusion coefficient which is a temperature dependent property

σ_o Fracture stress due to wetting

σ_∞ is the fracture stress of the original material

The predictive model's result compares to the observed bond strength showed a limited prediction so in the future a better model is needed. By combining the models that built in this thesis and finite-element method, it is possible to achieve a process model that can successfully predict the overall strength of FDM parts. With these predictive models, engineers can be able to accurately predict the mechanical behavior of parts printed by FDM so that FDM can be a rapid manufacturing process that robust and reliable.

Finally, post-processing of FDM parts by annealing after printing was shown to have positive effects on bond strength if the heating temperature is lower than 250°C. Heating the part above 250°C showed detrimental effects on bond strength, despite the increased bond lengths achieved. Thermal degradation and micro-bubbles are the significant contributors to this phenomenon. In order to apply post-processing to the FDM part in the

manufacturing process a mathematical model that can describe the thermal degradation phenomenon is necessary.

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Appendices

Appendix A - Polycarbonate Properties Data Tables

Table A. 1 Temperature dependent thermal conductivity data

T (K)	K (W/m-K)
298	0.201
323	0.205
348	0.208
373	0.214
398	0.217
423	0.214
448	0.213
473	0.217
498	0.225
523	0.229
548	0.236
573	0.240

Table A. 2 Temperature dependent specific heat capacity data

T (K)	C _p (J/kg-K)
298	1315
323	1335
348	1354
373	1374
398	1404
423	1476
448	1486
473	1504
498	1521
523	1539
548	1545
573	1550

Appendix B - Matlab M-files

B.1. M-file: cp_k_polycarbonate.m

```
T_cp=[298 323 348 373 398 423 448 473 498 523 548 573];
cp_poly=[1315 1335 1354 1374 1404 1476 1486 1504 1521 1539 1545 1550];
xx=400:.1:600;
cp_spline=spline(T_cp,cp_poly,xx);
T_k=[298 323 348 373 398 423 448 473 498 523 548 573];
k_poly=[0.201 0.205 0.208 0.214 0.217 0.214 0.213 0.217 0.225 0.229 0.236
0.240];
k_spline=spline(T_k,k_poly,xx);
plot(xx,k_spline)
xlabel('Temperature (degree K)');
ylabel('Thermal Conductivity (W/m-K)');
figure
plot(xx,cp_spline)
xlabel('Temperature (degree K)');
ylabel('Specific Heat Capacity (J/kg-K)');
T_vis=550:10:600;
eta_r=252;
T_r=588;
eta_t=eta_r*exp(-0.035*(T-T_r));
figure
plot(T_vis,eta_t);
xlabel('Temperature (degree K)');
ylabel('Viscosity (Pa-s)');
```

B.2. M-file: cooling_model.m

```
function [T t]=cooling_model(To,Tinf,dt,tfinal)

%Specific Heat Capacity of PC J/kgK

T_cp=[298 323 348 373 398 423 448 473 498 523 548 573];

cp_poly=[1315 1335 1354 1374 1404 1476 1486 1504 1521 1539 1545 1550];

cp_spline=spline(T_cp,cp_poly);

%Thermal Conductivity of PC W/mK

T_k=[298 323 348 373 398 423 448 473 498 523 548 573];

k_poly=[0.201 0.205 0.208 0.214 0.217 0.214 0.213 0.217 0.225 0.229
0.236 0.240];

k_spline=spline(T_k,k_poly);

%Thermal Conductivity of Foundation sheet W/mK

k_Nylon=0.25;

%Air properties

T_air=Tinf;

k_air=0.02624*(T_air/300)^0.8646;

g=9.81;

beta_air=1/T_air;

Pr_air=0.68+(4.69*10^-7)*(T_air-540)^2;

Dyna_vis_air=(1.458*(10^-6)*(T_air^1.5))/(T_air+110.4);

Density_air=101325/(287.05*T_air);

Kine_vis_air=Dyna_vis_air/Density_air;

%Initial variables for iterative loop

T(1)=To;

T_new=To;

i=1;

v=0.03;

P1=0.214*10^-3;
```

```

w=10^-3;

%Geometry properties

a=.2*10^-3;

b=.1*10^-3;

h=(a-b)^2/(a+b)^2;

P=pi*(a+b)*(1+(3*h/(10+sqrt(4-3*h)))));

A=pi*a*b;

D=2*sqrt(a*b);

%Start iterative loop calculating time with temperature

for t=0:dt:tfinal

    error=1;

    while error > 1e-6

        T_old=T_new;

        k=ppval(k_spline,T_old);

        cp=ppval(cp_spline,T_old);

        rho=1200;

        Gr=g*beta_air*(T_old-Tinf)*(D)^3/(Kine_vis_air)^2;

Nu=(0.6+0.378*((Gr*Pr_air)/(1+(0.559/Pr_air)^(9/16))^(16/9))^(1/6))^2;

        h=k_air*Nu/(D);

        alpha=k/(rho*cp*v);

        beta=(h*(P-P1)+k_Nylon*P1*w)/(rho*cp*A*v);

        n=(sqrt(1+4*alpha*beta)-1)/(2*alpha);

        T_new=Tinf+(To-Tinf)*exp(-n*v*t);

        error=abs(T_new-T_old)/T_old;

    end

    T(i)=T_new;

    i=i+1;

end

t=0:dt:tfinal;

plot(t,T);

xlabel('Time (s)');

ylabel('Temperature (degrees K)');

end

```

B3. M-file: bonding_model_developed.m

```
function[bond,t_bond]=bonding_model_developed(To,Tinf,dt,tfinal)

[T t]=cooling_model(To,Tinf,dt,tfinal);

%Surface tension

gamma=.0342;

%Viscosity

eta_r=252;

T_r=588;

theta(1)=0;

t_bond(1)=0;

%Initial value

a=.2*10^-3;

b=.1*10^-3;

ao=sqrt(a*b);

eta_0=eta_r*exp(-0.01*(T(3)-T_r));

theta(2)=sqrt(2*(dt/10)*gamma/(eta_0*ao));

t_bond(2)=2*dt;

for j=4:2:(tfinal/dt-1)

    delta_t=2*dt;

    t_bond(j/2+1)=t_bond(j/2)+delta_t;

%k1 calculation at t_bond(i/2)

eta_1=eta_r*exp(-0.01*(T(j-1)-T_r));

theta_1=theta(j/2);

k1=(gamma/(3*ao*eta_1*sqrt(3.14)))*((3.14-theta_1)*cos(theta_1)+...

sin(theta_1))*((3.14-

theta_1+sin(theta_1)*(cos(theta_1)))^(1/2))/...

(((3.14-theta_1)^2)*((sin(theta_1))^2));
```

```

%k2 calculation

eta_2=eta_r*exp(-0.01*(T(j)-T_r));

theta_2=theta(j/2)+dt*k1;

k2=(gamma/(3*ao*eta_2*sqrt(3.14)))*((3.14-theta_2)*cos(theta_2)+...
sin(theta_2))*((3.14-
theta_2+sin(theta_2)*(cos(theta_2)))^(1/2))/...
(((3.14-theta_2)^2)*((sin(theta_2))^2));

%k3 calculation

eta_3=eta_2;

theta_3=theta(j/2)+dt*k2;

k3=(gamma/(3*ao*eta_3*sqrt(3.14)))*((3.14-theta_3)*cos(theta_3)+...
sin(theta_3))*((3.14-
theta_3+sin(theta_3)*(cos(theta_3)))^(1/2))/...
(((3.14-theta_3)^2)*((sin(theta_3))^2));

%k4 calculation

eta_4=eta_r*exp(-0.01*(T(j+1)-T_r));

theta_4=theta(j/2)+2*dt*k3;

k4=(gamma/(3*ao*eta_4*sqrt(3.14)))*((3.14-theta_4)*cos(theta_4)+...
sin(theta_4))*((3.14-
theta_4+sin(theta_4)*(cos(theta_4)))^(1/2))/...
(((3.14-theta_4)^2)*((sin(theta_4))^2));

%theta

theta(j/2+1)=theta(j/2)+(1/6)*delta_t*(k1+2*k2+2*k3+k4);

end

bond=ao*sin(theta);

```

```
figure  
  
plot(t_bond,bond);  
  
xlabel('Time (s)');  
  
ylabel('(Bond Length)/2 (m)');  
  
end
```