2007

Development and Modeling of High Temperature Polymeric Heater

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DEVELOPMENT AND MODELING OF HIGH TEMPERATURE

POLYMERIC HEATER

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Bachelor of Science in Chemistry

City University of New York (York College)

May 2001

Submitted in partial fulfillment of requirements for the degree

MASTER OF SCIENCE IN CHEMICAL ENGINEERING

at the

CLEVELAND STATE UNIVERSITY

December 2007

ACKNOWLEDGEMENTS

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ABSTRACT

Polymers are generally known for their excellent insulative properties. The addition of carbonaceous fillers such as carbon black and graphite within a polymer matrix can impart electrical and thermal properties making them good conductors. The resulting composites can be used in applications requiring and/or ranging from electromagnetic and radio frequency interference (EMI/RFI) shielding, electrostatic discharge (ESD) and heaters/heating elements to which metals have been the materials of choice. The advantages of using such composites include cost reduction, part consolidation, chemical resistance, lighter weight, and ability to easily design into complex three dimensional shapes via injection molding.

For this work, various conductive thermoplastic composites were investigated as a metal (Ni-chrome heating element) alternative and/or substitute for use as heating elements through mechanisms of Joule heating. First, composites and test specimen were prepared via melt extrusion and injection molding respectively. Thereafter, electrical thermal and mechanical properties were characterized using both ASTM and non ASTM techniques. Results were then modeled using statistical software to determine correlations between formulations to responses and whether these results are desired and or meaningful.

Results from experiments indicated significant advantage in using semi-crystalline polymers as the base carrier due to the superior electrical properties at

equivalent filler loading compared to amorphous based composites, a criterion in joule heating. It was also determined that heating rate and maximum/plateau temperature was mainly a function of specimen resistance (formulation parameter) and voltage setting. Finally, the model obtained for plateau temperature was found to be significant. This indicated it is possible to develop polymeric type heaters with operating temperatures above 100ºC (current technology) and as high as 200ºC. Moreover, these composites would have self regulating properties other than positive temperature coefficient (PTC) type mechanism seen in olefin based polymeric heaters.

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CHAPTER I

INTRODUCTION

1.1. Introduction

In the last century we have witnessed rapid growth and advances made in material science and polymer engineering. In particular substantial contributions from polymers, (i.e.; polymers as metal, glass and wood substitutes) have led to cost reduction, improved performance, and increased durability from previous materials of choice (1). This evolution has led to the development of many other polymers, blends and composites as superior alternatives to prior material (1, 2). In order to understand the wide range use and benefits of these materials it is necessary to first review polymer fundamentals.

Polymers are long chain molecules which are made up by many covalently bound monomers and can be classified into two categories; thermoset and thermoplastic (3). The main differences between the two occur during heating. Thermosets undergo chemical change when heated while thermoplastics undergo physical change. Generally, thermosets can be cured once due to the chemical reaction/change while thermoplastics can be heated multiple times (4). Due to this effect, thermoplastics are more advantageous/favorable since they can be processed in stages (extrusion and molding). Thermoplastics also are classified into two categories; amorphous and semi-crystalline. The differences between the two are seen in their morphology, chemical, thermal and

mechanical properties as shown in Table 1.1. These attributes distinguish relevant

applications they can be incorporated into. In addition, to these classifications, many

thermoplastic polymers exist as shown in the plastic performance pyramid (Figure 1.1).

These can be divided in accordance to their use temperature and performance properties.

The top of the pyramid (PEEK) represents the highest continuous use temperature,

generally 240ºC and while the bottom (HDPE) has continuous use temperature of 100ºC.

Table 1.1: Chemical, Mechanical and Thermal Characteristics of Amorphous and Semi-Crystalline Polymers (1, 2)

Amorphous		Semi-Crystalline
Generally:	**Attributes:**	*Generally:*
Poor	**Chemical Resistance**	Good
Poor to Excellent	**Impact Resistance**	Moderate to Good
Low	**Shrinkage**	High
Low	**Warpage**	High
Moderate to Good	**Stiffness**	Good
Poor	**Barrier**	Good
Transparent or Translucent	**Optical Appearance**	Generally Opaque
Lower vs. Semi-Crystalline	**Heat Resistance**	Better vs. Amorphous
Lower vs. Semi-Crystalline	**Creep Resistance**	Better vs. Amorphous
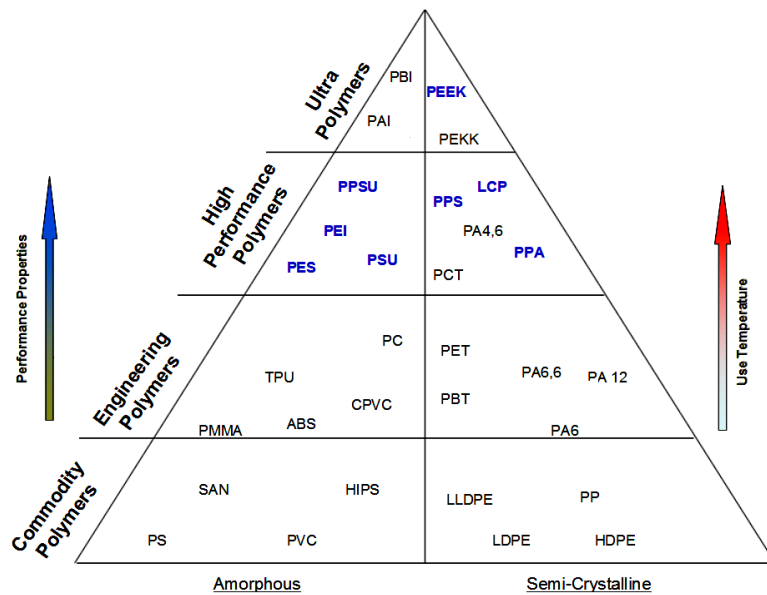
Figure 1.1: Plastics Performance Pyramid (Courtesy of PolyOne)

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With the wide property performance and temperature range, polymers can be used in many applications which metals, glass and wood has been the material of choice. However, for polymers to be used in electrical and or thermal conductive applications, it is necessary to incorporate some type of filler (carbon black, graphite and or metallic powders) such that it can become electrically and thermally conductive (5, 11). In order to understand this effect the electrical conductivity of various materials is first reviewed.

1.2. Electrical Conductivity of Various Materials

Electrical conductivity of any given material is dictated by how much current it can transmit (5). Depending on their inherent properties, such materials can also improve thermal conductivity. However, the reverse is not true; materials that impart thermal conductivity may not necessarily impart electrical conductivity (5). This is mainly dependent on the chemical and physical make up of material (polymers, metals and composites).

The electrical conductivity of materials can be measured using standardized techniques developed by American Standard Testing Techniques (ASTM) (6). Most commonly, the surface and volume resistivities are measured. Surface resistivity is determined by measuring surface resistance between two electrodes, while the volume resistivity measures bulk resistance between opposite faces. The unit of surface resistivity is reported in ohm/square while volume resistivity is reported as ohm-centimeter. Figure 1.2 contains electrical properties (volume resistivity) of various materials. Such materials can be broken down into three categories: polymers, polymer composites carbon black and metals which are discussed in subsequent section.
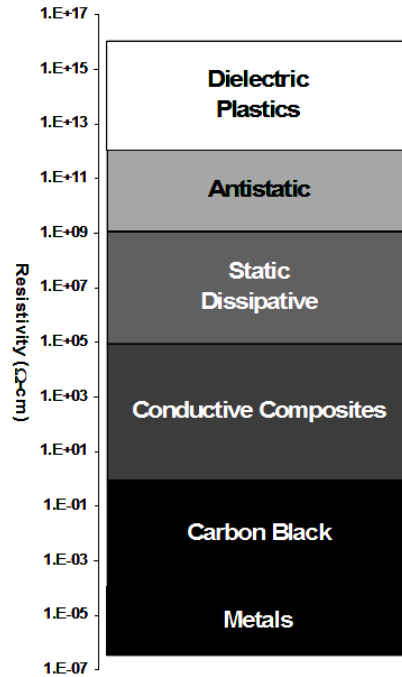
Figure 1.2: Electrical Properties of Various Materials and Composites
(Courtesy of Degussa)

1.2.1. Polymers

Polymers are generally known for their excellent insulation and are therefore considered as insulating materials. Typical these polymers have high volume resistivity ranging from $10^{12} - 10^{16}$ Ω-cm (Figure 1.2). However, specially synthesized polymers that are inherently conductive do exist. These polymers are referred to as inherently conductive polymers (ICP's). Among the classes of specially derived conductive polymers, polyaniline is of much interest worldwide due to its unique conduction mechanism and environmental stability (7). However at present state of art, these polymers are difficult to process at a large scale and more importantly lack thermal and physical stability needed in most applications.

1.2.2. Polymer Composites

The addition of carbonaceous fillers such as carbon black, graphite and or metal

powders/fibers within a polymer matrix can impart both electrical and thermal properties

making them good conductors (5, 8-20). The resulting composites can replace

applications in which metals have been the material of choice (21). The advantages of

using these composites include cost reduction, chemical resistance, lighter weight, and

ability to easily design into complex three dimensional shapes via injection molding (21).

In addition, these composites can be designed with varying properties depending on filler

shape, size, structure, purity and volume loading.

In order for polymer composites to be used in conductive applications they must impart

specific electrical properties namely surface resistivity as shown below (5, 11, 19):

- $10^5 - 10^9$ Ω/square for electrostatic discharge (ESD) applications

- $10^0 - 10^5$ Ω/square for moderately conductive applications

- 10^2 Ω/square and lower conductivity for shielding applications

These composites have found wide success and are used in many applications ranging

from electromagnetic and radio frequency interference (EMI/RFI) shielding, electronic

devices, and electrostatic painting.

1.2.3. Metals

Metals are excellent conductors of electricity and heat (5). Both electrical and

thermal conductivities are much higher than carbonaceous fillers ranging between $1x10^{-7}$

to $1x10^{-4}$ ohms centimeter (Figure 1.2). However due it its high cost, weight and

oxidation/corrosion characteristics, alternative materials such as polymer composites

have gained interest over the past 30 years and have become the materials of choice when

applicable(1, 2).

1.3. Resistive Heating

The discovery of resistive heating by James Prescott Joule in 1841 has provided for an efficient way of producing heat through conversion of electrical energy to thermal energy (25). It is a process by which electric current passes through a conductor thereby generating heat. From a molecular perspective, resistive heating occurs when electrons collide with atomic ions that make up the bulk of the conductor. These collisions result in the increase of kinetic energy manifesting into heat as byproduct (25). From a thermo-dynamical perspective, efficiency of resistive heating can be explained from the first law of thermodynamics which describes the conservation of energy. Essentially, electrical energy is transferred to thermal energy at nearly 99% since no work is done by the system. The benefits of this technology makes it suitable for number of applications such as electric heaters, toasters, coffee makers, electric stoves/blankets, thermistors and recently in self regulating polymeric heaters.

1.3.1. Resistive Heating with Polymers

The use of resistive heating polymers is not new, and has been well researched since the early 1970's (16). Applications utilizing this technology included for use in or as:

- *Curing of Thermosets*
  - Heat generated from electric current passing through wires embedded in material result in thermal curing of polymer.

- *Self Regulating Heaters*
  - Electrically conductive polymer composite is electrified resulting in heat generation. Often seen in heating blankets and shoe/glove heaters.

The latter example described works through principles of Positive Temperature

Coefficient (PTC) (8). Figure 1.3 illustrates example of operating curve for PTC type

materials. It shows that under normal operating conditions (Point 1), heat generated by

the device is in equilibrium to heat lost to environment thus device operates at constant

resistance and temperature. If electric current going through device is slightly increased,

temperature of device will increase as seen (Points 2 and 3).
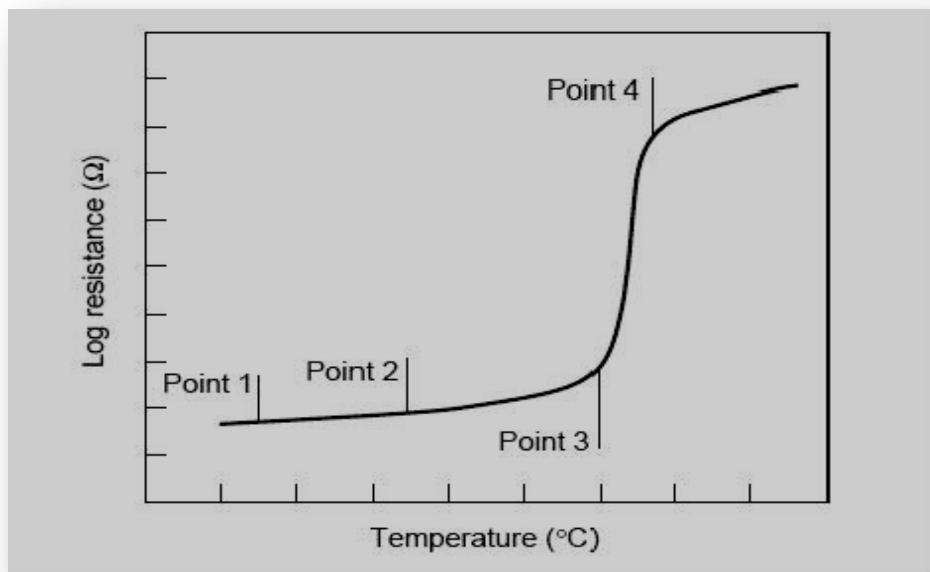
Figure 1.3: Electrical & Thermal Behavior of PTC Type Materials (8)

Any further increase in current beyond (Point 3) will cause composite to heat at a rate

faster than heat lost to the environment, eventually leading to a sharp increase in

resistance (Point 4). This is due to phase/density transition of composite as shown in

Figure 1.4. Here we see that as temperature of the composite increases polymer begins to

melt causing the bulk to expand in volume. This increase causes separation of

conductive particles thereby eliminating conductive pathway causing the composite to

sharply increase in resistance. At this point, electric current can no longer travel thereby

ceasing the resistive heating effect. As temperature of the composite begins to cool,

resistance decreases allowing again for current to flow and thereby generate heat. This

mechanism allows for self regulating properties required in order to maintain stability.
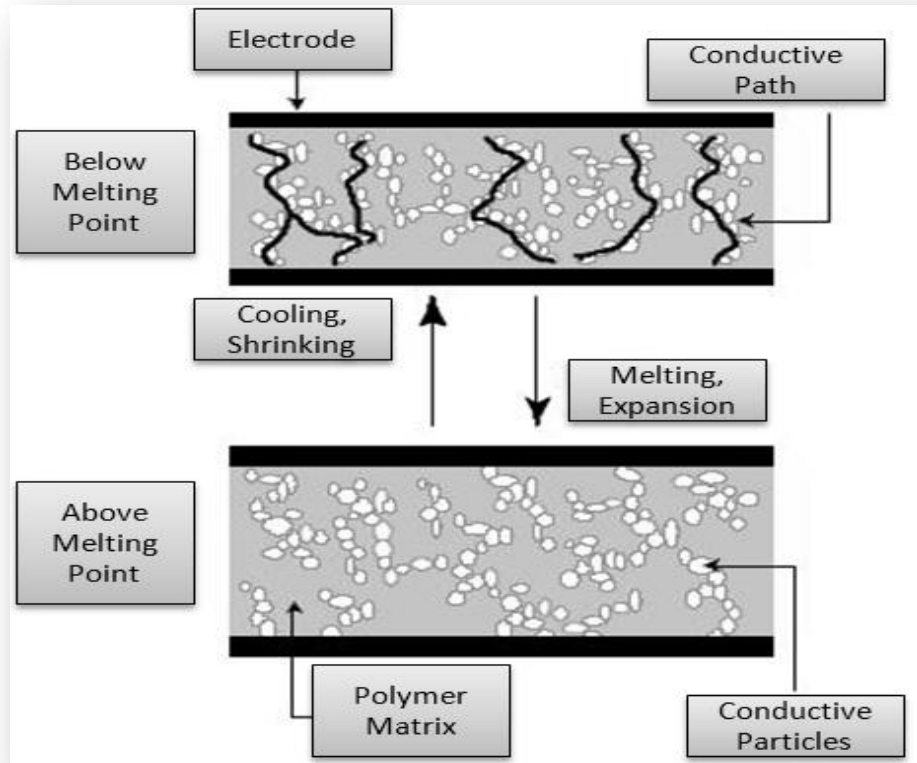
Figure 1.4: PTC Operating Mechanism (24)

Research articles discussing resistive heating of polymer composites are reviewed in

Table 1.2. Past accomplishments provide key understandings of success and limitations

from which to build future experiments/research. While these articles are briefly

discussed, key aspects of each journal are recognized.

Table 1.2: Earlier Studies of Resistive heating with Polymer Composites

Type	Description	Advantages	Disadvantages
Thermal Curing via Resistive Heating [45]	Heating through electric wires embedded in thermoset matrix results in curing of thermoset	Portable system, enables thermal curing to take place anywhere	Only used to cure thermoset, not used as heater
Self Regulating Polymeric Heaters [8, 10-11, 14, 17-18]	Heating through electrically conductive polymer composites	Controlled Heating, Efficient, Inherent Self Regulating Properties	Maximum Temperature well below 100°C, Limited to Olefin Based Polymers
Polymeric Thermistors [24]	Over-current Protection for use in/with electronics	Regulates Current Flow	Will only work with relative low electric current

1.4. Motivation

Using polymers as heaters have various advantages compared to the traditional Ni-chrome heating elements (metals) used in resistive heating today. The benefits include:

- *Cost* (Substantially lower than metals)

- *Lighter Weight* (Much lower in density than metals)

- *Part Consolidation* (Polymeric heaters will be designed as the end product thus entire product will heat, not the case for Ni-chrome heating elements which rely on heat transfer from element to end product). The entire polymeric end product can be considered as heating element.

- *Manufacturability* (Polymers can be easily molded into complex 3-D shapes with minimal waste)

- *Oxidation Free* (No rusting issues with polymers)

- *Chemical Resistance* (Most polymers have higher chemical resistance then metals)

- *Properties* (Electrical, thermal and mechanical properties can easily be tailored to meet specific needs, metals have limitations)

Past developments of polymeric heaters have shown promise but still have limitations. In particular, the operating temperatures often seen for these composites are well below 100 degrees Celsius since they work thorough principles of PTC. More importantly, this technology only works with olefin based polymers which have limited thermal, mechanical and chemical stability. Therefore, continuous research is needed in developing polymeric heaters which can operate with other polymer matrix/matrices at temperatures well above those seen in HDPE, whether through PTC or not.

1.5. Project Goals

The proposed research investigates the resistive heating of a carbonaceous filled thermoplastic as a viable polymeric heater. The objectives are to:

- *Determine suitable polymers as base carrier.* Both amorphous and crystalline polymers are investigated to determine effect of morphology on electrical thermal and mechanical properties.

- *Determine relationship between filler loading and electrical properties.* Measure resistance as a function of filler loading.

- *Establish heating rates.* Electrical current is applied to each sample; plateau temperature is then measured as a function of voltage and filler loading.

- *Determine relationship between electrical properties to plateau temperature.* Relationship is modeled using design of experiments.

- *Determine efficiency.* Measure heating efficiency via Calorimetry.

- *Determine heating repeatability (Cycle Study).* Measure plateau temperature as a function of heating cycles.

- *Determine if heating is uniform.* Thermal imaging camera is used to determine heat uniformity between electrodes.

CHAPTER II

BACKGROUND

2.1. Introduction

It has been previously stated that most polymers are generally known for their insulating properties. The addition of fillers within a polymer matrix such as carbon black, graphite and or metallic powders are known to impart electrical and or thermal conductivity. The advantages of these composites compared to metals (which have been the material of choice) are well known, they include; lighter weight, corrosion resistance, cost, ease manufacturing and properties can be tailored to desired needs. Since the demand for such composites are growing; EMI, RFI shielding, ESD, heat sinks and for our purpose (resistive heating) it is necessary to understand factors which influence composite conductivity.

In this chapter, we begin by reviewing and understanding the principles of percolation theory. Thereafter, we study the effects of raw materials (fillers and polymers), processing (extrusion and molding), and testing to electrical conductivity. Lastly, the physics of resistive heating and its self regulating mechanisms will be discussed.

2.2. Percolation Theory

The electrical conductivity of a polymer composite is generally dependent on the filler loading (5). By increasing the filler content within composite, filler particles begin

to form a continuous network thereby creating a path for electric current/electrons to travel (13). This formation of a continuous path and or network is based on the principles of percolation theory. Introduced in 1957 by Hammersley and Broadbent, percolation theory describes the connectedness of particulates within an object and provides technical basis for quantifying random distributions of these particulates (40). Figure 2.1, contains a percolation curve for an electrically conductive polymer composite. It shows the
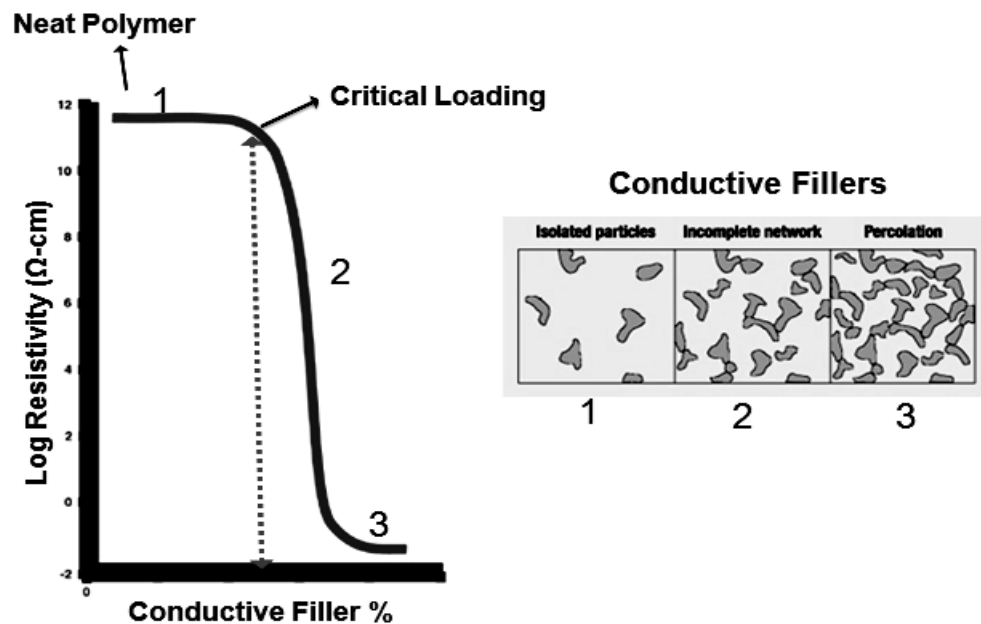
Figure 2.1 Percolation Curve (44)

volume resistivity of a composite as a function of (conductive) filler loading. As can be seen, unfilled (neat) polymers have volume resistivity of $1x10^{12}$ ohms-centimeter. With the addition of some conductive fillers (represented as 1), the volume resistivity remains unchanged. Here the conductive filler (carbon black, graphite and or metallic fibers/powder) act as conductors, however electrons are only free to flow within them and once they reach the end, they encounter the polymer matrix (insulator) which blocks the flow of electrons. At some filler loading (depending on properties), a percolation

threshold or critical loading is reached, meaning enough filler has been added thereby creating continuous networks which enables the electrons to travel. This region is represented as (2) on figure 2.1. At this point the system becomes very sensitive and responsive such that even a slight increase in filler can drop volume resistivity by a few orders of magnitude. When more filler is introduced, it becomes easier for the electrons to travel through the bulk due to higher continuous networks generated (represented as 3). After this region is reached, the conductivity levels off and any further addition of filler does not have any further benefits in reducing volume resistivity of composite.

2.3. Factors Effecting Electrical Properties

There are several factors that can have significant effect on the percolation curves. Most commonly, these are due to raw material selection (polymer, filler), processing methods (extrusion, molding) and testing (measurements made in the flow direction or normal to flow) methods (5, 11, 14, 36). In this section, we will review each of these effects from a literature perspective, data provided by our carbon black supplier and our own experiences.

2.3.1 Raw Materials

It is well understood that raw materials selection is of key importance in achieving desired electrical properties. In particular, the filler selection is critical since various grades exist, each consisting of different inherent electrical properties. For practicality of this thesis, we will focus on carbon black as the filler content. In order to categorize carbon black for use in electrically conductive application, it should meet following criteria (5, 36):

- Particle Size: *The lower the better, typically in the range of 15-75nm in radius (Provides more particles per volume thereby reducing the interparticle distance)*

- Surface Area: *High surface area, yields better electrical properties*

- Structure : *High structure/network increases the conductive path electrons can travel*

- Porosity: *Increased Porosity will yield more particles per unit weight than compact solid particles which helps reduce the interparticle distance*

- Volatility Content: *Low Volatility promotes tunneling effect which enables electrons to jump gaps which volatiles tend to inhibit*

Examples of some of these effects can be seen in Figure 2.2. This figure contains four various carbon backs (Printex XE2, L6, L and Vulcan XC-72), within a high density polyethylene (HDPE) matrix. Here we can clearly see that Printex XE2
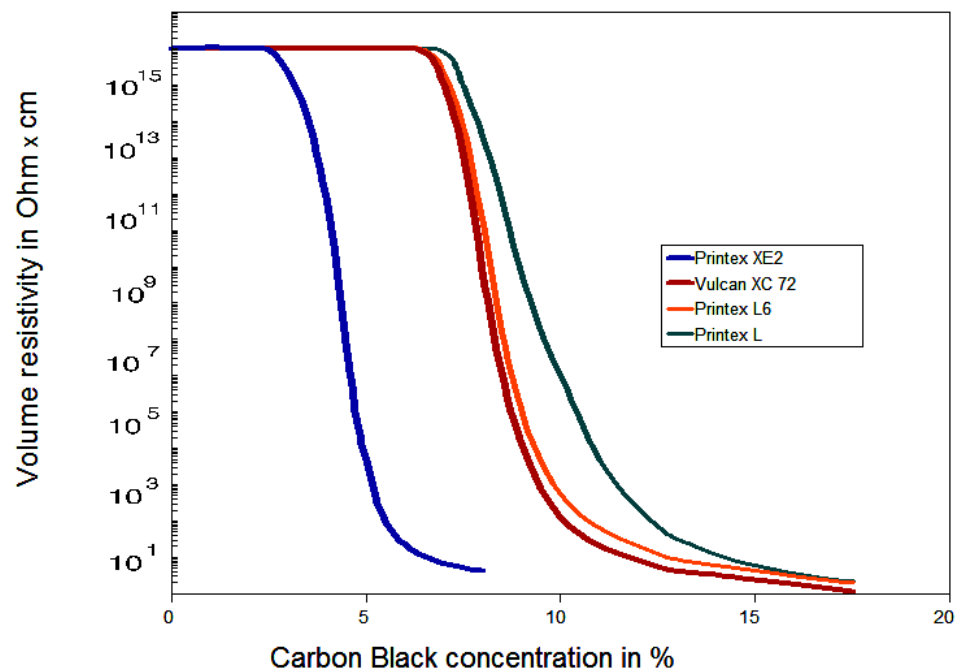
Figure 2.2: Percolation Curves of Various Carbon Blacks (Courtesy of Degussa)

(surface area of 912 m^2/g) has lowest critical loading necessary to achieve percolation threshold (3.5%); therefore it is the most electrically conductive black. This is followed by Vulcan XC 72, Printex L6 and Printex L which have surface area of 253, 250 and 150 m^2/g respectively.

Polymer choices are also important since its morphology can influence filler location and arrangement which can impact electrical properties. Studies have shown in amorphous polymers, conductive fillers are dispersed uniformly within the matrix while in semi-crystalline polymers the conductive fillers are ejected from the crystalline regions during crystallization and are concentrated in the amorphous region (5, 9, 36). This effect enables lower filler loading for semi-crystalline polymer to achieve percolation threshold due to the localized buildup of fillers (Figure 2.3).
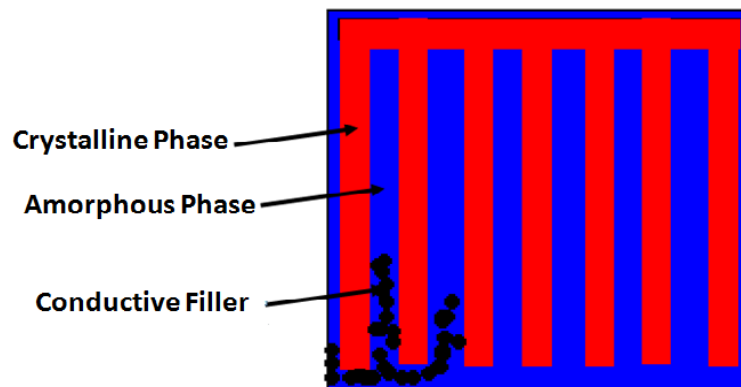
Figure 2.3: Morphology of Semi-Crystalline Polymer Composite
(Courtesy of Degussa)

2.3.2. Extrusion

One of the biggest challenges in preparing electrically conductive polymer composite is in processing, in particular during extrusion and injection molding. During extrusion, filler and polymer resin are introduced into a barrel through feed

hopper/section which is then conveyed by screw/screws until it exists through the die (Figure 2.4). The effect of Filler dispersion is generally a function of screw design, residence time, filler type and viscosity of polymer.
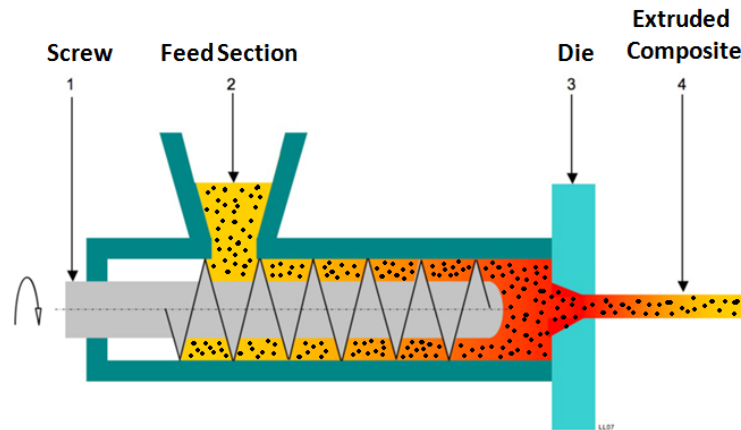
Figure 2.4: Schematic of Extruder

Screw designs affect shear forces generated during extrusion which in turn is responsible for mixing (filler dispersion). According to dispersion studies conducted by Dannenberg, conductivity of a composite resembles a probability curve; sharp increase in conductivity is seen during initial phase mixing, once critical mixing point is reached decline in conductivity takes place (43). This effect is due to the weak van der Waals forces of carbon black agglomerate to break down resulting into a decline of structure and network paths needed for electrons to travel (11).

Problems with feeding of raw materials during extrusion can also cause electrical properties of composite to deviate. Optimally, a separate feeder for each material is desired. This will eliminate any possible uneven feeding which can occur due to density, particle size differences of materials. In addition, gravimetric feeder is better suited for

such applications because unlike volumetric feeding which feeds a constant rate, it can

self adjust if feeding profile deviates.

2.3.3. Molding

The main problem encountered during injection molding has to do with filler

orientation. This is evident from data provided by Degussa (Figure 2.5, Table 2.1)
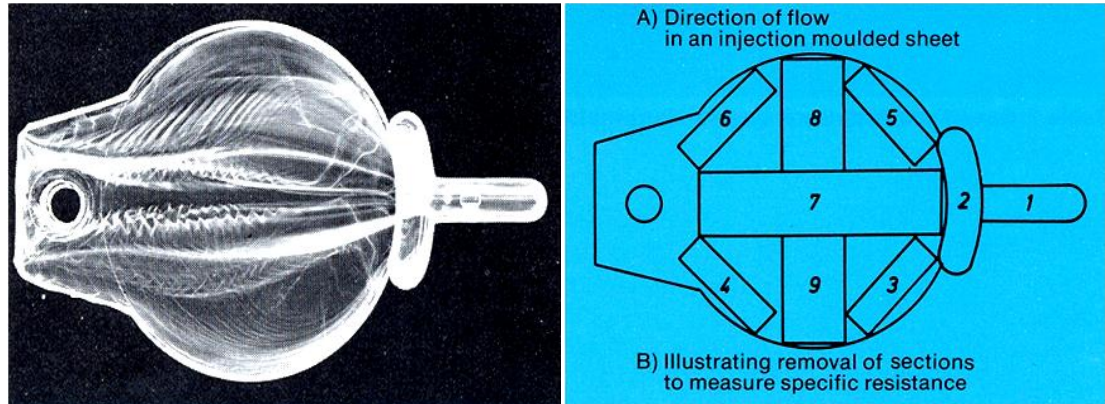
Figure 2.5: Flow/Orientation of Discs via Injection Molding
(Courtesy of Degussa)

In this figure, we can clearly see the effect of filler orientation during injection molding.

On the left picture, a non-uniform flow profile is shown which is common for injection

made from small nozzle/gate into a larger disc type cavity. Its corresponding electrical

properties (Table 2.5) show the highest resistance occurs at section one and two. Also,

conductivity is always higher when measured in the flow direction rather than across

(normal to flow) the specimen.

Table 2.1: Corresponding Electrical Properties
Of Sheet Shown in Figure 2.5

Conductivity of Injection Molded Sheet		
Part Number	**Specific Resistance [Ohm-cm]**	
	Lengthwise	**Across**
1	2.2×10^3	1×10^3
2	5.2×10^2	3.3×10^2
3	5.2×10^1	1.6×10^2
4	2.7×10^1	1.3×10^2

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Part Number	Specific Resistance [Ohm-cm]	
	Lengthwise	Across
5	4.9×10^1	1.4×10^1
6	2.9×10^1	8.2×10^1
7	2.7×10^1	3.9×10^1
8	2.9×10^1	5.3×10^1
9	3.1×10^1	6.9×10^1

Other molding parameters can also affect electrical properties. The most common has to do with mold surface temperature. Studies conducted by Bayer and co-workers show that higher mold temperatures yielded superior electrical conductivity at equal filler loadings (41, 42).

Studies conducted by Degussa illustrates that compression molded samples have superior electrical properties compared with injection molding. This comparison is shown from an SEM image (Figure 2.6). Picture on the left (compression molded) sample shows a high continuous networking of carbon black while picture to the right (injection molded) clearly lacks the networking type structure seen in compression molded samples resulting in lower electrical properties. This effect is believed due to the anisotropic distribution of fillers compared to the isotropic orientations seen in injection molding.
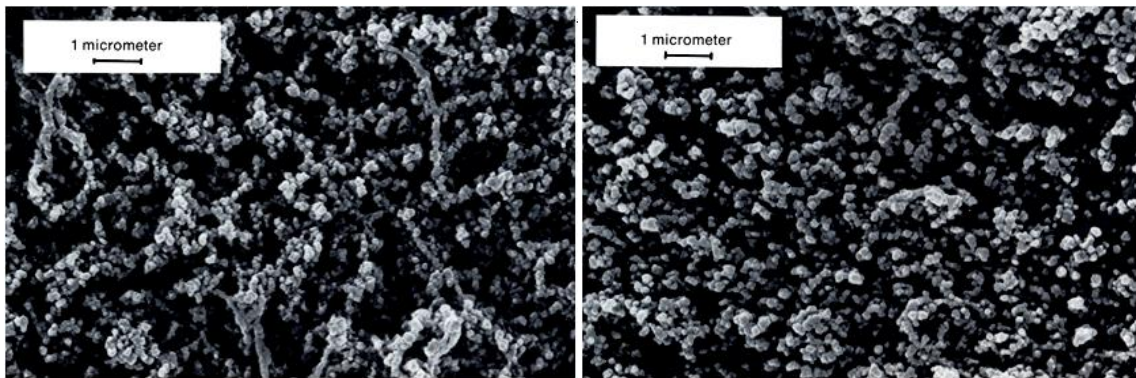
Figure 2.6: SEM Image of Compression Molded (left) Injection molded (right) Composite (Courtesy of Degussa)

2.3.4. Testing (Electrical Testing)

Our own experience has shown measurement techniques can also influence electrical conductivity. Generally, volume resistivity of a composite is measured using ASTM D257. This method measures electrical resistance normal to flow as shown on the left picture (Figure 2.7). However, when electrical properties are measured in the flow direction (right picture), significant reduction in volume resistivity (3-5 orders of magnitude) was found. We believe this be due to the filler alignment/orientation which enhances current/electrons to travel.
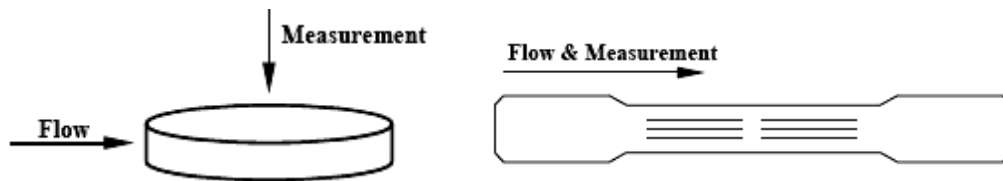
Figure 2.7: Electrical Conductivity Measurement Methods (a) right and (b) left

2.4. Physics of Resistive Heating

In order to understand how a self regulating polymeric heater operates, we must review:

1. The basic principles of resistive heating and its governing equations.

2. Energy Balance (self regulating properties)

As described in chapter 1, James Prescott Joule discovered when current travels across a resistive material, heat is generated. Specifically, he found that the rate of heat generation (Q) is proportional to the current (I) squared and directly proportional to resistance (R) as shown in (Eqn. 2.1).

$$Q = R\,I^2 \qquad\qquad\qquad (Eqn.\ 2.1)$$

As a result, we can control the thermal energy generated (Q) by adjusting the inherent resistance of material or current going through the conductor due to its relationship to ohms law (Eqn. 2.2) and power law (Eqn. 2.3).

$$V = IR \qquad \qquad \text{(Eqn. 2.2)}$$

$$P = VI \ \& \ P = R\,I^2 \qquad \qquad \text{(Eqn. 2.3)}$$

This is relatively easy to do for materials with constant resistance over temperature range 23-220 degrees Celsius (range dictated by our experiments) because you can control the current flow by simply adjusting the voltage (V). As a result we can predict the rate of heat generated by knowing the mass (m), specific heat (c_p) of sample and power supplied (RI^2) to conductor (Eqn. 2.4)

$$mc_p \frac{\Delta T}{\Delta t} = R\,I^2 \qquad \qquad \text{(Eqn. 2.4)}$$

With the understanding of how heat is generated, we must now focus on self regulating/equilibrium properties of polymeric heater. Figure 2.8 shows the overall scheme (energy balance) of polymeric heater. Considering the cylinder as the conductor, voltage supplied to the system will result in current flowing through the conductor in turn generating heat (Q), while heat lost to the environment (q_l) can be due to conduction, convection and radiation. In our experiments, the conduction term is negligible due to experimental setup/scheme shown in Figure 4.7 (essentially no contacts exist between heated composite to any other object). The convective term occurs due to air movement and can be described using Newton's law of cooling, while heat loss due to radiation can be modeled using Stefan-Boltzmann Law. Incorporating all mechanisms responsible for heat lost to the environment results in equation 2.5.
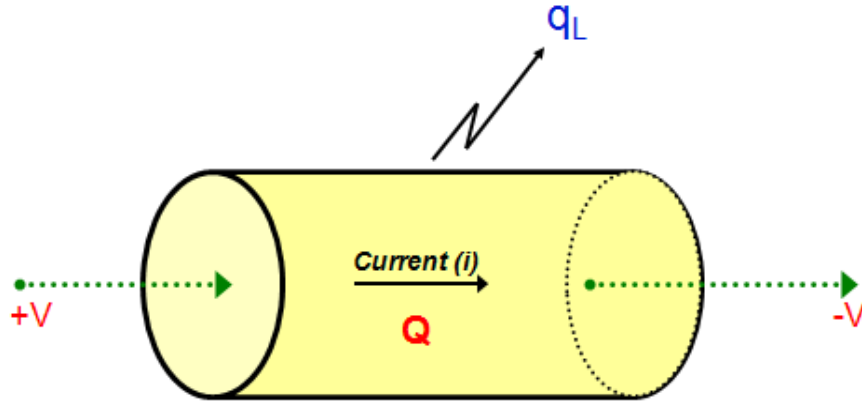
Figure 2.8: Energy Balance of Polymeric Heater

$$mc_p \frac{\Delta T}{\Delta t} = q_{g-} \left[q_{l1(Convection)} + q_{l2(Radiation)} \right] \qquad \text{(Eqn. 2.5)}$$

Here, (q_{l1}) is from Newton's law of cooling shown in (Eqn 2.6), where T_A is the ambient

temperature, T_F the final temperature, A the total surface area and U the overall heat

transfer coefficient.

$$q_{l1} = UA(T_F - T_A) \qquad \text{(Eqn. 2.6)}$$

While heat loss due to radiation (q_{l2}) is from Stefan-Boltzmann Law shown in (Eqn 2.7),

where ε is the emissivity of material, δ is Stefan-Boltzmann constant of 5.67 x $10^{-8} \frac{J}{s\,m^2 K^4}$

and A the total surface area.

$$q_{l2} = \varepsilon \delta A (T_F^4 - T_A^4) \qquad \text{(Eqn. 2.7)}$$

Combining both heat loss terms with equation 2.5 yields (Eqn 2.8).

$$mc_p \frac{\Delta T}{\Delta t} = Power - [UA(T_F - T_A) + \varepsilon \delta A(T_F^4 - T_A^4)] \qquad \text{(Eqn. 2.8)}$$

Once a plateau temperature and or steady state is achieved, equation 2.8 simplifies to

(Eqn. 2.9)

$$Power = [UA(T_F - T_A) + \varepsilon \delta A(T_F^4 - T_A^4)] \qquad \text{(Eqn. 2.9)}$$

CHAPTER III

PROJECT MATERIALS

3.1. Introduction

Raw materials chosen for this project were based on their properties (thermal and mechanical stability), processability, availability, effectiveness, and widespread use within industry. In total five thermoplastic polymers were investigated as base carriers/matrix, three crystalline polymers and two amorphous polymer. The crystalline polymers consisted of polyphenylene sulfide (PPS), high density polyethylene (HDPE) and nylon 6 (PA6) while amorphous polymers were acrylonitrile-butadiene-styrene (ABS) and polycarbonate (PC). These polymers are commonly used in electrical, electronic, automotive, packaging, appliance and many other applications. The two types of fillers: carbon black and graphite used in this project were chosen based on their ability to increase electrical and thermal conductivity. This chapter provides a brief background in addition to physical, electrical and thermal properties of each materials used for this project.

3.2. Polymer Matrices

3.2.1. Polyphenylene Sulfide (PPS)

The first thermoplastic used for this project was Chevron-Phillip's Ryton® PPS grade PR-26. This highly engineered semi-crystalline polymer is widely known for

its thermal and dimensional stability as well as chemical resistance, high modulus, and

dielectric properties (4, 28). Typical applications of PPS composites include automotive,

electrical and electronic devices which require high thermal and dimensional stability (4,

28). With its high thermal/dimensional stability and broad chemical resistance, PPS is

well suited for use in hostile chemical and thermal conditions. With these unique

attributes and high melting temperature, PPS has the characteristics necessary to achieve

and maintain high temperature heating through resistive/Joule heating. The chemical

structure of PPS is made up of alternating sulfur atoms and phenylene rings in a para

substitution, chemical structure is shown in Figure 3.1. Properties of Ryton® PPS PR-26

are shown in Table 3.1.
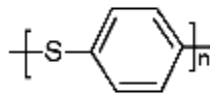
Figure 3.1: Chemical Structure of PPS

Table 3.1: Properties of Ryton® PPS PR-26 (4, 28)

Properties	ASTM Methods	Technical Data Sheet PR-26	Tested Data PR-26
Melt Flow Index 316 C°/5kg (g/10min)	D1238	750	-
Density (g/cc)	D792	1.36	1.34
Tensile Modulus (ksi)	D638	624	538
Tensile Strength at Break (ksi)	D638	11603	10638
Tensile Elongation at Break (%)	D638	1.6	2
Notched Izod Impact (ft-lb/in)	D256	0.3	0.3
Flexural Modulus (ksi)	D790	-	164
Heat Deflection Temperature @ 66psi (C°)	D648	115	113
Volume Resistivity (Ohm-cm)	D257	-	1×10^{14}
Particle Form/Type (Visual)	N/A	Powder	Powder
Melt Temperature (C°)	D3418	285	281
Glass Transition Temperature (C°)	D3418	90	93.7
Crystallinity (%)	D3418	-	60.1

3.2.2. High Density Polyethylene (HDPE)

The second thermoplastic used in this project was Solvay's and Dow's DMDA-8920 NT 7 HDPE. HDPE is a highly crystalline polymer most commonly used in milk jugs, plastic bags and cable insulations (29). Its benefits include ductility, chemical resistance, toughness, and dielectric properties (29). Much research has been conducted with this resin for use in conductive applications, thus open literature is available for data comparison. Examples of applications utilizing conductive HDPE composites are (8, 9, 11):

- *Electromagnetic interference (EMI)* Provides shielding which prevents interference thorough radiation to electronic devices

- *Electrostatic dissipative (ESD)* Prevents static charge accumulation

- *Self Regulating Heaters also known as thermistors* Works on the principles of resistive/Joule heating by Positive Temperature Control (PTC) mechanisms

The chemical structure and various properties of HDPE are shown in Figure 3.2 and Table 3.2 respectively.

$$\left[CH_2-CH_2\right]_n$$
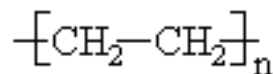
Figure 3.2: Chemical Structure of HDPE

Table 3.2: Properties of Solvay's HDPE[1] and Dow's DMDA-8920 NT 7 HDPE[2] (26-27, 29)

Properties	ASTM Methods	Technical Data Sheet[1]	Tested Data[1]	Technical Data Sheet[2]	Tested Data[2]
Melt Flow Index @ 190°C/2.16kg	D1238	18	18	20	20
Density (g/cc)	D792	0.95	0.95	0.95	0.95
Tensile Modulus (ksi)	D638	-	190	-	204
Tensile Strength at Break (psi)	D638	-	2832	2000	1832
Tensile Elongation at Break (%)	D638	-	230	250	238
Notched Izod Impact (ft-lb/in)	D256	-	0.7	-	0.7
Flexural Modulus (ksi)	D790	-	172	167	166
Heat Deflection Temperature @ 66psi	D648	-	71	72.8	66
Volume Resistivity (Ohm-cm)	D257	-	1×10^{14}	-	1×10^{14}
Particle Form/Type (Visual)	-	-	Powder	Pellet	Pellet
Melt Temperature (C°)	D3418	130	130	130	130.5
Glass Transition Temperature (C°)	D3418	-76	-	-76	-
Crystallinity (%)	D3418	-	-	-	64.7

3.2.3. Nylon 6 (PA6)

The third thermoplastic used in this project was BASF's Ultramid® B27-E01 Nylon 6. Nylon 6, a semi crystalline polymer is a widely used thermoplastic most commonly seen in fiber applications such as clothing and carpeting. It is also used as an engineering material for automotive and electronic applications. It has many benefits including fatigue resistance, low coefficient of friction, chemical resistance, good toughness and dielectric properties (30). Like HPDE, much research has been conducted using this resin/composite for heat sink, ESD and EMI applications (5, 21). The chemical structure and various properties of Nylon 6 are shown in Figure 3.3 and Table 3.3 respectively.

$$- \left[\overset{\overset{\textstyle H}{|}}{N} - \overset{\overset{\textstyle O}{||}}{C} - (CH_2)_5 \right] -$$
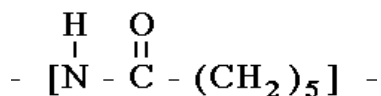
Figure 3.3: Chemical Structure of Nylon 6

Table 3.3 Properties of Ultramid B27-E01 Nylon 6 (26-27, 30)

Properties	ASTM Methods	Technical Data Sheet	Tested Data
Melt Flow Index @ (g/10min)	D1238	-	-
Density (g/cc)	D792	1.13	1.13
Tensile Modulus (ksi)	D638	435	414
Tensile Strength at Break (psi)	D638	-	6110
Tensile Elongation at Break (%)	D638	>50	80
Notched Izod Impact (ft-lb/in)	D256	1	1.1
Flexural Modulus (ksi)	D790	-	404
Heat Deflection Temperature @ 66psi (C°)	D648	103	101
Volume Resistivity (Ohm-cm)	D257	$1x10^{13}$	$1x10^{13}$
Particle Form/Type (Visual)	-	Pellets	Pellets
Melt Temperature (C°)	D3418	220	220
Glass Transition Temperature (C°)	D3418	60	-
Crystallinity (%)	D3418	-	55.4

3.2.4. Acrylonitrile Butadiene Styrene (ABS)

The fourth thermoplastic used in this project was Lanxess's Lustran® ABS 433. ABS is an amorphous polymer widely known for its tensile/impact strength, dimensional stability, surface hardness, dielectric characteristics, and heat, chemical, abrasion resistance (31). It has a unique structure which combines strength and rigidity of acrylonitrile and styrene with the toughness of butadiene rubber. These ratios can be varied to provide desired properties. ABS resins are translucent to opaque and are commonly used in applications ranging from automotive, appliances and protective/decorative devices. Examples include helmets, automotive instrument panel, interior components and home-security devices (26). The chemical structure and various properties of acrylonitrile-butadiene-styrene are shown in Figure 3.4 and Table 3.4 respectively.

$$CH_2{=}CH_2 \;+\; CH_2{=}CH_2 \;+\; -[CH_2{-}CH{=}CH{-}CH_2]_n$$

(styrene, with benzene ring)	C≡N acrylonitrile polybutadiene
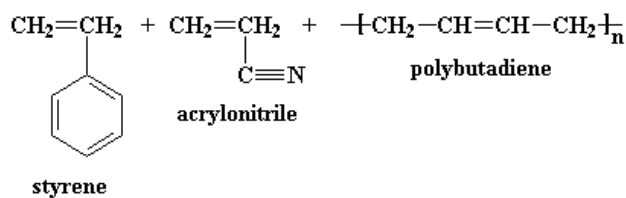
Figure 3.4: Chemical Structure of Acrylonitrile Butadiene Styrene

Table 3.4 Properties of Lustran® ABS 433 (26-27, 31)

Properties	ASTM Methods	Technical Data Sheet	Tested Data
Melt Flow Index @230 C°/3.8kg (g/10min)	D1238	5	8
Density (g/cc)	D792	1.05	1.05
Tensile Modulus (ksi)	D638	370	363
Tensile Strength at Break (psi)	D638	-	5160
Tensile Elongation at Break (%)	D638	30	21
Notched Izod Impact (ft-lb/in)	D256	7	7.6
Flexural Modulus (ksi)	D790	380	376
Heat Deflection Temperature @ 66psi (C°)	D648	92	90
Volume Resistivity (Ohm-cm)	D257	-	1×10^{15}
Particle Form/Type (Visual)	-	Pellets	Pellets
Melt Temperature (C°)	D3418	-	-
Glass Transition Temperature (C°)	D3418	-	-

3.2.5. Polycarbonate (PC)

The last thermoplastic used in this study was Bayer's Makrolon® 2658 Polycarbonate. Polycarbonate is an amorphous polymer widely known for its clarity, ductility, toughness and impact strength (32). It also has good dimensional and thermal stability but has limited chemical resistance and poor fatigue and wear properties (32). This thermoplastic is commonly seen in applications ranging from lenses, CD's, exterior automotive components, kitchenware and appliances (26). The chemical structure and various properties of Polycarbonate are shown in Figure 3.5 and Table 3.5 respectively.
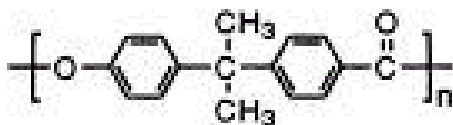
Figure 3.5: Chemical Structure of Polycarbonate

Table 3.5 Properties of Makrolon® 2658 Polycarbonate (32)

Properties	ASTM Methods	Technical Data Sheet	Tested Data
Melt Flow Index @ 300C° /1.2kg (g/10min)	D1238	12	12
Density (g/cc)	D792	1.2	1.2
Tensile Modulus (ksi)	D638	348	353
Tensile Strength at Break (ksi)	D638	-	10518
Tensile Elongation at Break (%)	D638	>50	136
Notched Izod Impact (ft-lb/in)	D256	-	15.6
Flexural Modulus (ksi)	D790	-	372
Heat Deflection Temperature @ 66psi (C°)	D648	137	135
Volume Resistivity (Ohm-cm)	D257	$1x10^{13}$	$1x10^{13}$
Particle Form/Type (Visual)	-	N/A	Pellets
Melt Temperature (C°)	D3418	290	-
Glass Transition Temperature (C°)	D3418	148	--

3.3. Fillers

3.3.1. Carbon Black

Carbon Black is widely used filler that has found widespread use in a number of applications. It can be used as or used to improve the following (5, 11):

- *Dimensional stability*

- *Physical properties*

- *Electrical properties*

- *Thermal properties*

- *UV stability*

- *Anti-oxidant*

- *Improve life span of polymer*

- *Pigment or Colorant*

It mainly consists of elemental carbon in the form of spherical/primary particles. Depending on the raw materials and processing techniques, van der Waals forces combine the primary particles to join into aggregates and or agglomerates that ranges around 10-300 nm in size (5). The particle size and extent of networking will then determine the inherent properties and type of application used in. For applications requiring good conductivity, carbon black must have high surface area and small particle size and be highly structured and networked (11). For this project Printex® XE2, a high surface area, highly structured and branched Carbon Black manufactured by Degussa was used. This filler was chosen due to its high electrical conductivity at relatively low loadings. The physical properties of Printex XE2 are given in Table 3.6.

Table 3.6: Properties of Printex XE2 (33-36)

Properties	Value
Particle Size	30nm
Particle Type/Form	Powder/Bead
Density (g/cc)	1.8
Electrical Resistivity (ohm-cm)	1×10^{-1}
Thermal Conductivity (W/m K)	40 -100
Ash Content (%)	1.0
CTAB Surface Area (m^2/g)	900
DBP Absorption (ml/g)	380

3.3.2. Graphite

Graphite is often used as an additive and or filler to improve lubrication, strength, and electrical and thermal conductivity within a polymer matrix (35). It consists of a carbon atom arranged in a hexagonal structure forming a planar orientation. Layers are stacked and held together by van der Waals forces. Numerous studies have shown when

fillers with high aspect ratio are used in combination with carbon black, increase in

electrical conductivity is seen by as much as two fold (10, 19). For this project, Graphite

from Superior Graphite manufacture, Formula FX™2939 was used. The physical

properties of FormulaFX™2939 are given in Table 3.7.

Table 3.7: Properties of Superior Graphite Formula™FX2939 (34-35)

Properties	Value
Particle Size	180-850μm
Particle Form/Type	Flake
Density (g/cc)	2.25
Electrical Resistivity (ohm-cm)	$2x10^{-2}$
Thermal Conductivity (W/m K)	40-400

CHAPTER IV

EXPERIMENTAL METHODS

4.1. Introduction

In order to start with the development of a polymeric heater, it is necessary to discuss techniques utilized in sample preparation and testing methods. Polymer composites were first prepared using a research scale extruder and injection molding machine. Several ASTM testing methods were then implemented to analyze polymer composite. These included a variety of tests for physical, electrical and thermal properties. In some cases, ASTM standards or equipment necessary to test samples were unavailable thus other techniques common throughout the field and literature were implemented for the remaining tests. This chapter will describe in detail the preparation and testing methods used to analyze various polymer composites. Discussions of the results from these tests are covered in chapter 5, 6 and modeled in chapter 7, while the complete processing and experimental data are provided in appendices. Figure 4.1 illustrates the flow of experimental methods.
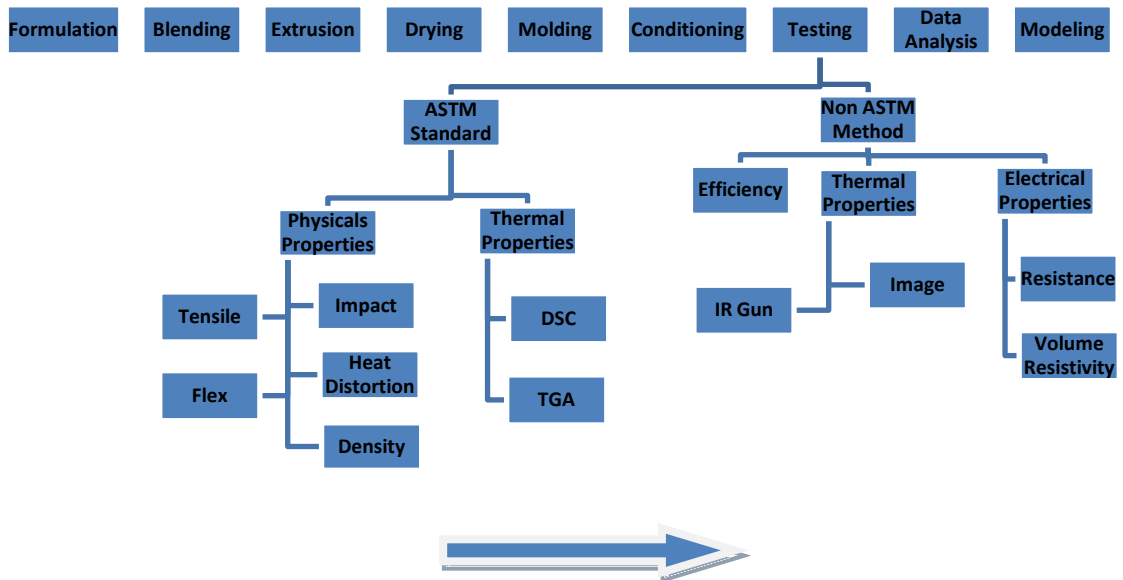
Figure 4.1: Flow of the Experimental Scheme

4.2. Test specimen preparation

4.2.1. Formulation

Initial formulations to determine necessary loading level of carbonaceous fillers to make polymer composite conductive were prepared based on literature review and prior experience. Subsequent formulation modifications were derived based on experimental results.

4.2.2. Blending

In order to maximize mixing and wet out, a Purnell Henschel model FM 10C was used for blending of powders (Figure 4.2). First, the base carrier/polymer matrix were added to the Henschel and mixed for one minute at 2500rpm. Thereafter, carbon black was added and mixed for two

Figure 4.2: Henschel

minutes at the same rpm. Lastly the graphite was added and mixed again for one minute at same the conditions.

4.2.3. Extrusion

Materials were extruded using a Thermo Electron Prism model 5401-2003 (Figure 4.3), a 16mm co-rotating, intermeshing twin screw extruder with a length/diameter ratio of 40 to 1 and 10 heating zones. Heating zones from the rear to nozzle were set to temperatures ranging between 200-315 degrees Celsius depending on the polymer.   A low shear mixing screw was chosen for optimal dispersion of fillers for highest possible conductivity while minimizing any possible degradation.   Configuration of the screw design can be seen Appendix C. A Kulurstrasse model 55-73 volumetric feeding system was used to introduce polymer blends to the extruder at zone one.   A complete list of formulations and processing conditions are given in Appendix A, B respectively. The extruded strands were then conveyed via conveyer belt for cooling and cut using Conair Jetro 304 pelletizer. Approximately 3-5 lbs of each formulation was produced.

Figure 4.3: Extruder Used for Compounding Composites

4.2.4. Drying

Pelletized samples were dried using a Stabil Therm Electric convection oven model ESP-400C-5 set to temperatures ranging from 90-120 degrees Celsius for 2-4 hours removing any possible surface moisture from pellets in preparation for injection molding.

4.2.5. Injection Molding

Test specimens were prepared using 33 Ton Cincinnati Milacron model VSX 33-2.27 and 120 Ton Van Dorn injection molding machines (Figure 4.4). Both presses utilized a general purpose mixing screw, having length to diameter ratio of 20 to 1. Testing bars were prepared using an ASTM and variable thickness plaque mold (Figure 4.4). The ASTM mold consisted of a standard type IV tensile bar, flexural and Izod bar while the plaque mold provided a 4" x 6" variable thickness plaque. First, test specimens composed of neat PPS, HDPE, Nylon 6, ABS and PC were molded to verify properties with technical data sheet in addition to having controls for our experiments. Approximately 12 ASTM bars of each test specimens were molded per formulations while only one specimen of the plaque sample was molded. Dimensions of the molded samples can be seen in Table 4.1. In between formulations, Dynpurge grade MX compound was purged through barrel to prevent any cross contamination and the first four injection shots were also disregarded.

Table 4.1: Dimensions of Molded Samples

Molded Samples	Dimensions (inches)		
	Length	width	Thickness
Tensile Bar	6.5	0.5	0.125
Flexural Bar	5	0.5	0.125
Izod Bar	2.5	0.5	0.125
Plaque	6	4	Variable
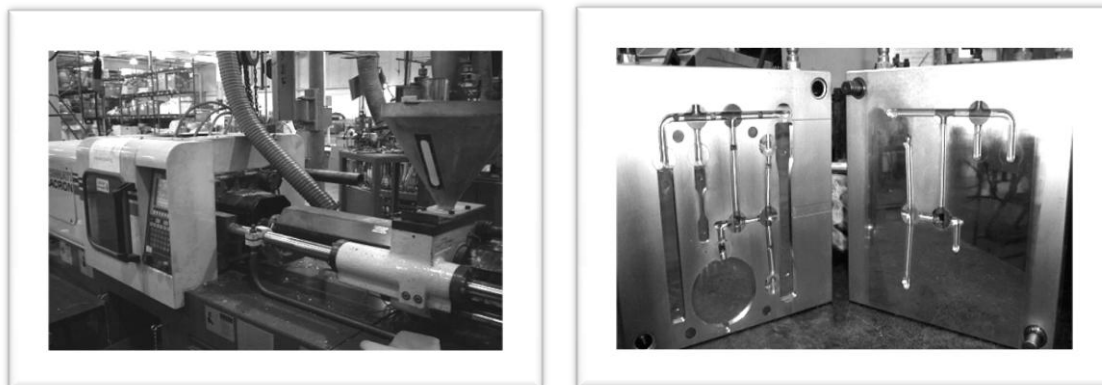
Figure 4.4: Pictures of Injection Molding Machine and ASTM Mold

Test specimens were labeled by, formulation number and injection order the specimen came out of mold. All the samples were labeled in the following form:

X-Z

All formulations designated with X denote formulation number while letter Z denotes bar sequence order. Upon molding, each formulation was inserted inside a conditioning chamber discussed in the next section 4.3. Injection molding conditions for all formulations can be found in Appendix D.

4.3. Sample Conditioning

Prior to testing, molded test specimens were conditioned to 23 ±2° Celsius and 50 ±5 % relative humidity for 40 hours in accordance to ASTM D618 standards. This ensures equilibrium conditions of the test specimen with respect to temperature and minimizes fluctuation of test results.

4.4. Testing

4.4.1. ASTM Methods (Thermal and Physical Properties)

Physical and Thermal Analytical properties were tested using standard ASTM methods common throughout the field. Table 4.2 illustrates testing type, ASTM number,

rate, and properties obtained per test.

Table 4.2: Testing Conditions and Methods

ASTM Method	Test Type	Properties Obtained	Rate/Conditions
D638	Tensile	Modulus, Elongation, Stress at Break	0.2 inches/min
D790	Flexural	Modulus	0.05 inches/min
D256	Notched Impact	Energy to Break	2 lb pendulum
D648	Heat Distortion Temperature (HDT)	Temperature	2°C/min
D792	Density	Composite Density	-
D3418	Differential Scanning Calorimetry (DSC)	Crystallinity, Melt and Glass Transition Temperature	10°C/min
D1131	Thermal Gravimetric Analysis (TGA)	Composition	10°C/min

Composite Density

As shown on Table 4.2, composite density was measured using ASTM D792, a
standard test that measures the composite density by water displacement. For this test,
the weight of an Izod bar was measured at two different conditions. First the dry weight
was measured using a Toledo Mettler AG204 scale and recorded; thereafter the same part
was suspended on a scale while completely submerged into a beaker of water at room
temperature yielding the submerged weight of composite. The density of the composite
can then be calculated using Equation 4.1

$$\left(\frac{Dry\ weight}{Dry\ weight - Submerged\ weight}\right) * Density\ of\ H_2O \qquad \text{(Eqn 4.1)}$$

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Measured values were then compared to theoretical densities according to Equation 4.2, which uses the densities and weight percent values for each composites.

$$\rho_c = \frac{1}{\sum_{i=1}^{n} \frac{w_i}{\rho_i}}$$
(Eqn 4.2)

where, ρ_c is the composite density and w_i and ρ_i are the weight fraction and inherent density of materials used.

4.4.2. Non ASTM Methods

4.4.2.1. Electrical Properties

A standard ASTM type IV tensile bar was used for measuring longitudinal resistance or resistance in the flow direction. A non ASTM technique was developed and implemented to quickly screen electrical properties. This involved

Figure 4.5: Tensile Bar with Two Brass Screws apart

drilling two holes measuring 2/10th of an inch in diameter and screwing/inserting two brass screws 4.5 inches apart onto the tensile bar. A Fluke 189 True RMS Multi-meter and Milli-To-2 meter was then used to measure resistance between the two electrodes/brass screws. For conductive samples with resistance below $1x10^9$ ohms, a Fluke 189 multi-meter was used while for non conductive samples with resistance above $1x10^9$ ohms, a Milli-T0-2 meter was used. A scheme of the testing setup to measure resistance is shown in Figure 4.6.

Figure 4.6: Measurement of Resistance using Fluke 189 Multi-meter

4.4.2.2. Thermal Properties

4.4.2.2.1. Resistive Heating Measurements via IR gun

A non ASTM method was again developed and implemented for measuring heat generation of polymer composite during resistive heating. This test was accomplished by using the same test specimen prepared in section 4.4.1. The outer tip of the tensile bar on both ends was raised and affixed to glass plate acting as insulation layer such that minimal electrical and or heat transfer can take place (Figure 4.7). Illustration of the testing set up is shown on Figure 4.8.

Glass Plate
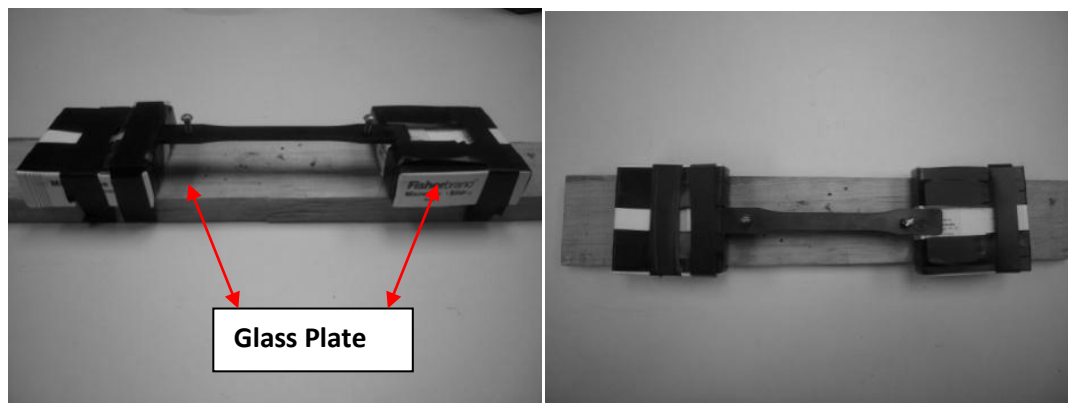
Figure 4.7 Tensile Bar setup for Electrification/Resistive Heating.

A Fluke 574 Closed Focus Infrared Thermometer was then mounted right above the tensile bar such that the temperature reading is within and at the center of the tensile bar. The emissivity setting on the IR Thermometer was adjusted to 0.95, recommended setting for plastics/polymers composites. Measurements were made using IR-graph version V2.3.16 data acquisition
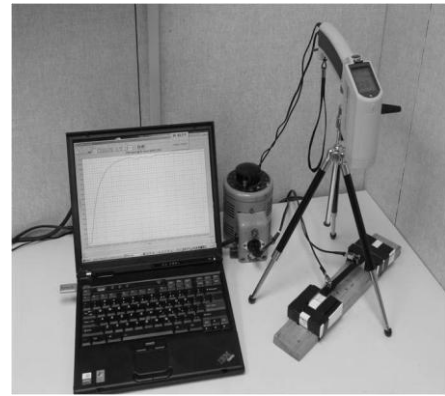
Figure 4.8: Resistive Heating Testing and Measurement Setup

software connected to the Fluke IR gun to which an average data point was collected every 15 seconds for 15 minutes, duration of the resistive heating. Both AC (Powerstat model S1138 Variac) and DC (Sorensen model XTS 120-5 Transformer) power sources were utilized during testing for data comparison.

4.4.2.2. Thermal Imaging

Thermo Imaging of the plaques during electrification/resistive heating was obtained using an Inframetrics Model 740 IR Imaging unit (Figure 4.9). Thermal Images were recorded upon initiation of electrical charge. Heating profile was captured to determine if heating is uniform between the two contacts. Testing samples were prepared by a two step molding process. First, a 1/16th inch thickness plaque sample was molded; thereafter two strips of conductive copper tape provided by 3M® was applied to the edges of plaque in parallel while being 4 inches apart. Lastly, the plaque containing

Figure 4.9: Inframetrics Model 740 IR Imaging unit

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the conductive strips was over-molded to a final thickness of $1/8^{th}$ in. This method

enabled a linear heating profile between contacts as opposed to the elliptical heating

profile seen when using brass screws as contacts. Picture of the plaque with embedded

copper is shown in Figure 4.10.
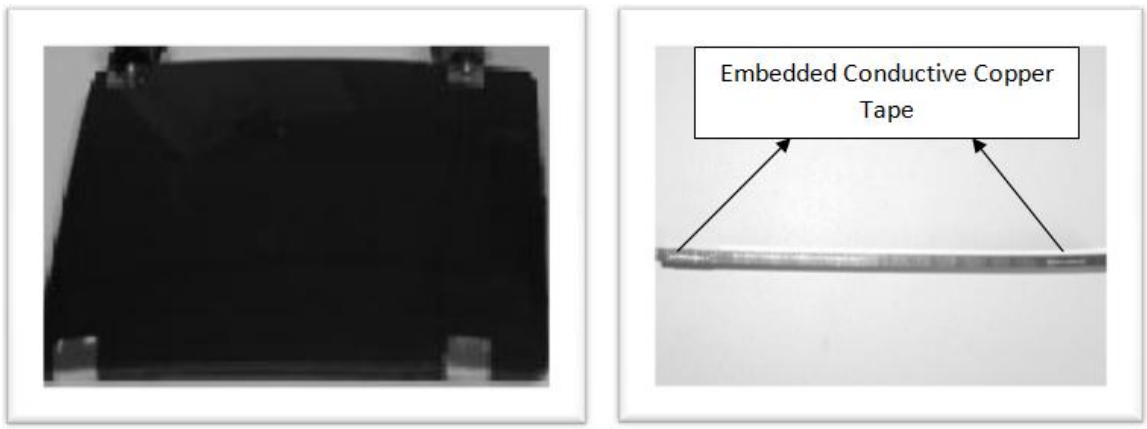
Figure 4.10: Plaques with Embedded Conductive Copper Tape

4.4.2.2.3. Efficiency (Heat Generation)

Resistive heating efficiency was measured
using the principles of calorimetry. The tensile bar
to be electrified was submerged under de-ionized
water within an insulative Styrofoam®. A picture
of the testing setup is shown in Figures 4.11 and
4.12. Brass screws and electrical wires attached
to the tensile bar were covered with silicon caulking to

Figure 4.11: Efficiency Testing Setup

eliminate electrolysis from occurring while in direct contact with water during

electrification. A Sorensen XTS 120-5 low variable direct current transformer model was

then used to apply a constant voltage to the tensile bar resulting to heat generation. The

temperature change of water was then measured using a Fluke 574 Closed Focus Infrared

Thermometer probe. The efficiency of the heat generating composite can then be calculated using Equations 4.4 and 4.5 from the principles of calorimetry.

$$P = VI = I^2R = mc_p \frac{\Delta T}{\Delta t} \qquad \text{(Eqn 4.4)}$$

$$\eta = \frac{P_{output}}{P_{input}} * 100\% \qquad \text{(Eqn 4.5)}$$

where P is Power, V, I and R denotes voltage, current and resistance respectively while, m the mass of water, c_p the specific heat of water, ΔT change in temperature and Δt change in time.

**Figure 4.12: Silicon Caulked Brass
Screws Submerged under water**

CHAPTER V

EXPERIMENTAL RESULTS I,

DETERMINATION OF BASE CARRIER

5.1. Introduction

The first step taken in the development of a polymeric heater was to determine a suitable polymer as base carrier. As discussed in section 3.1, three crystalline (HDPE, PA6 and PPS) and two amorphous polymers (ABS and PC) were investigated. The primary objective was to understand the effect of morphology (crystalline vs. amorphous polymers) as a function of electrical properties at a constant loading of carbon black. The secondary objective was to determine the effect of filler loading on physical and thermal properties. Once these effects are measured and analyzed, suitable polymer/polymers will be selected and used in the subsequent formulations in chapter 6.

5.2. Formulations

Polymers selected for use as base carrier are shown in Table 5.1. This table shows the experimental formulations for unfilled (neat) polymers (#1-5) and composites (#6-10). The inclusion of unfilled polymers serves as a control for gauging with CB filled systems in addition to comparison with technical data sheet provided by suppliers. All formulations shown in Table 5.1 were extruded and molded in accordance to processing guidelines provided by suppliers and are shown in Appendix B & D.

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Table 5.1: Formulation Table Consisting of Neat Polymers and Polymer Composites

Materials	Formulations									
	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
[1]HDPE	100					91				
Nylon 6		100					91			
PPS			100					91		
ABS				100					91	
PC					100					91
Carbon Black						9	9	9	9	9
Total (%)	100	100	100	100	100	100	100	100	100	100

[1] Dow's high density polyethylene in pellet form

5.3. Electrical & Thermal Properties

Electrical properties of unfilled polymers and composites were measured using techniques described in section 4.4.2, and are shown in Figure 5.1. Clearly, we can see the effect of carbon black in reducing resistance of all polymer composites compared with unfilled systems. In addition, it is evident that reduction of resistance is much higher in semi-crystalline polymers (decrease of 5-7 orders of magnitude) compared
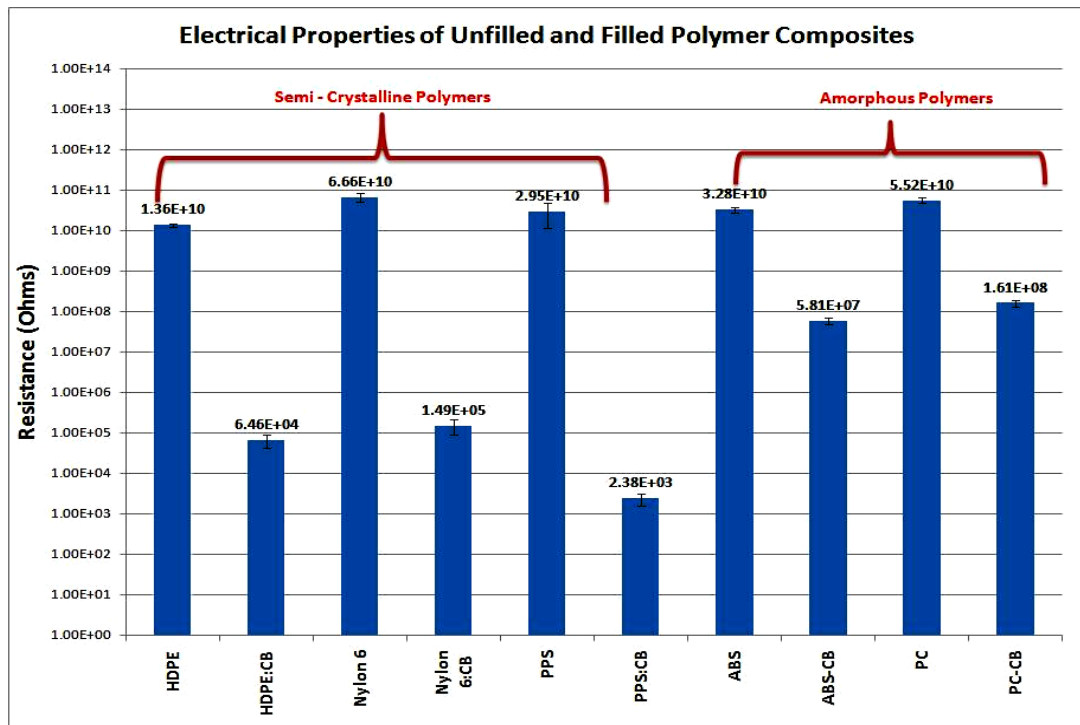
Figure 5.1: Electrical Properties of Unfilled and Filled Polymer Composites

to amorphous composites (decrease of 2-3 orders magnitude). This is in agreement with literature which describes favorable reduction of resistance for semi-crystalline polymers compared to amorphous polymers at same filler loading (5, 9, 36).

A similar correlation can also be made within semi-crystalline polymers by calculating their percent crystallinity using equation 6.1 and comparing them with corresponding electrical properties.

$$\alpha = \frac{\Delta H_f}{\Delta H_{f\ 100\%}} * 100\% \qquad \text{(Eqn. 6.1)}$$

Here, α is the percent crystallinity, ΔH_f is the heat of fusion (measured from area under melting peak curve of DSC) and $\Delta H_{f\ 100\%}$ is the theoretical heat of fusion calculated for 100% crystalline polymers (obtained from literature).

Table 5.2 shows percent crystallinity and corresponding electrical property (resistance) of HDPE, PPS and Nylon 6 and composites. From this table we can clearly see that the difference of crystallinity between unfilled polymer and composite is lowest in the PPS systems followed by the HDPE system and Nylon 6. Furthermore, its corresponding electrical property (resistance) is lowest for composites which in turn had minimal loss/difference in crystallinity between polymer and composite.

- HDPE (α= 64.7), HDPE-CB (α= 60.8): Difference of -3.9%
- Nylon 6 (α= 31.1), Nylon 6-CB (α= 38.8): Difference of +7.7%
- PPS (α= 60.1), PPS-CB (α= 59.5): Difference of -0.6%

This suggests that carbon black distribution is most concentrated and restricted within the amorphous region of PPS thereby yielding better electrical property (lower resistance) compared to other systems. Percent crystallinity calculations are shown in Appendix O using DSC curves of all polymers and composites from Appendix N.

Table 5.2: Electrical &Thermal Properties of Unfilled and CB Filled Polymers

Polymer Morphology	Materials	Differential Scanning Calorimetry (DSC)		Thermal Gravimetric Analysis (TGA)[2]	Electrical Resistance	Resistive Heating @ 70V(AC)[3]
		Melting Temperature	Crystallinity[1]			
		T_m (C°)	α (%)	(CB %)	(Ohms)	ΔT (C°)
Semi-Crystalline Polymers	HDPE	130.5	64.7	0.2	1.36×10^{10}	0
	HDPE-CB	131	60.8	7.3	6.46×10^{4}	63
	Nylon 6	220	31.1	0.3	6.66×10^{10}	0
	Nylon 6-CB	219	38.8	10.5	1.49×10^{5}	0
	PPS	281	60.1	-	2.95×10^{10}	0
	PPS-CB	285	59.5	-	2.38×10^{3}	76
Amorphous Polymers	ABS	-	-	0.7	3.28×10^{10}	0
	ABS-CB	-	-	11.8	5.81×10^{7}	0
	PC	-	-	-	5.52×10^{10}	0
	PC-CB	-	-	-	1.61×10^{8}	0

[1] Calculated using DSC & theoretical values, sample calculation provided in Appendix O,N
[2] TGA technique not possible for PPS and PC due to residual reaction/degradation during ash process
[3] Ambient temperature 24.5 degrees Celcius

Table 5.2 also shows Thermal Gravimetric Analysis (TGA) technique used to verify CB

loading (percent) in composite. Clearly, we can see discrepancy in CB content which

ranges between low (7.3%) and high (11.8%) even though all composites were supposed

to have 9% CB. However, this variation is not unexpected due to feeding techniques and

equipment used during extrusion. A picture of the feeder which illustrates the uneven

feeding of filler to polymer is shown in Figure 5.2.

Figure 5.2: CB Separation from Pellet Prior to Feeding to extruder
(Top View of Feed Hopper)

Here we can clearly see the buildup of filler at top half of feed hopper while the polymer pellets are concentrated on the bottom side of hopper. Due to this uneven build-up/mixing, feeding ratio of filler to polymer, fluctuates over time resulting in the variation of composition.

In order to verify CB composition for samples to which TGA was not possible (PPS, PC), an alternative testing method for composition analysis was implemented. This included the calculation of densities and comparison of measured to theoretical values by applying equation 4.2 to determine percent error (Table 5.3). Results show percent error of density to range -0.04 to 1.1 percent, verifying inconsistency in composition as seen in TGA.

Table 5.3: Comparison of Measured and Theoretical Density Polymer & Composites

Polymer Morphology	Materials	Measured Density	Theoretical Density	% Error
		$(g/cm)^3$	$(g/cm)^3$	(%)
Semi-Crystalline Polymers	HDPE	0.946	0.946	-
	HDPE-CB	0.989	0.988	0.03
	Nylon 6	1.130	1.130	-
	Nylon 6-CB	1.181	1.170	0.94
	PPS	1.337	1.337	-
	PPS-CB	1.384	1.369	1.10
Amorphous Polymers	ABS	1.049	1.049	-
	ABS-CB	1.095	1.090	0.46
	PC	1.196	1.196	-
	PC-CB	1.233	1.234	-0.04

5.4. Thermal Properties (Resistive Heating)

Thermal properties were measured using principles of resistive/Joule heating. Each CB filled composite were electrified and its heating effect measured using techniques described in section 4.4.2.1. Results from Table 5.2, show only two of the five composites; HDPE-CB (87.5ºC) and PPS-CB (100.5ºC) generated heat when charged with 70V (AC). Interestingly, PA6-CB composite (which was only half an order

of magnitude higher in resistance than HDPE-CB composite) did not heat, suggesting the minimum resistance needed to heat a tensile bar must be well below 1.49x 10^5 ohms. As expected, amorphous composites did not heat because their corresponding resistances were significantly higher than 1.49x 10^5 ohms. Heating rates of HDPE-CB and PPS-CB composites can be seen in Figure 5.3. Here we see HDPE-CB and PPS-CB samples heating to 87.5°C and 100.5°C respectively once steady state is achieved after approximately 6 minutes. Consequently, the PA6-CB composite which did not heat shows a constant line of 24.5°C with respect to time.
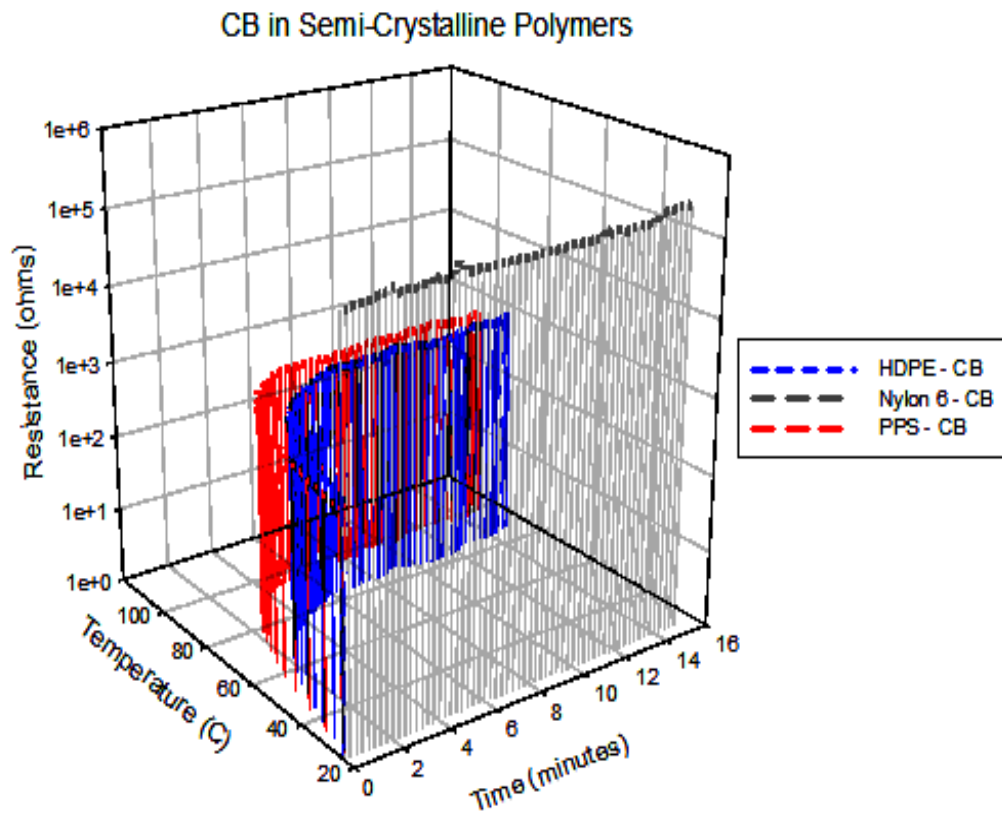
Figure 5.3: Heating rates of CB filled Semi-Crystalline Composites @70V (AC)

5.5. Mechanical Properties

Mechanical properties of unfilled polymers and CB filled composites were tested in accordance to ASTM standards discussed in chapter 4.4 and can be seen in Figure 5.4. Here, the properties of neat polymers tested are in close agreement to values provided by the polymer supplier seen in section 3.1. This is important because it verifies and validates our extrusion, molding and testing methods. As expected, the addition of carbon black shows significant impact on mechanical properties. It is clear that this addition can increase certain bulk properties while decreasing others.
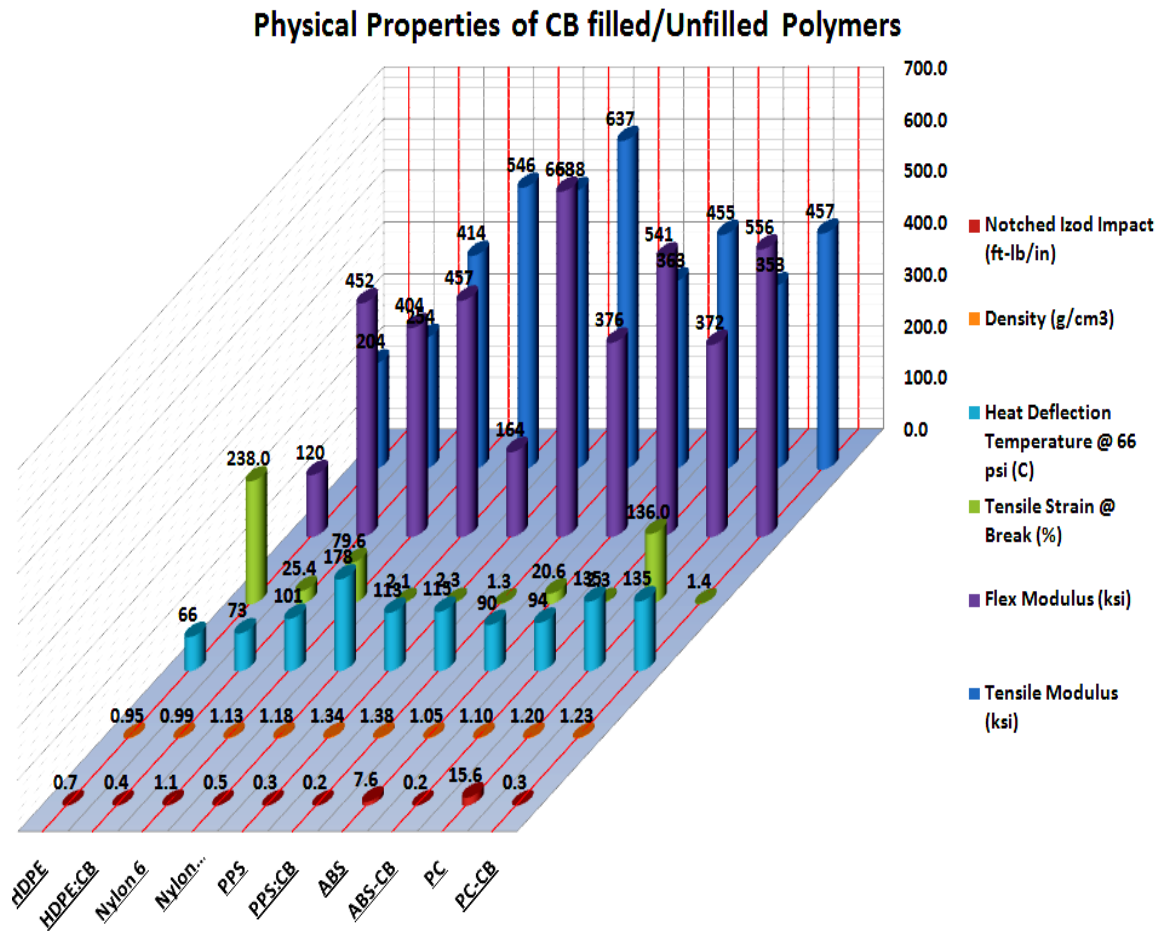
Figure 5.4: Physical Properties CB Filled Polymers

This effect is clearly shown in Table 5.4. The results are expressed in percent change between unfilled and CB filled polymers using equation 5.1. Positive values represent percent increase while negative values denote percent decrease for each test conducted.

Table 5.4: Influence of Carbon Black on Physical Properties

Test Type	Semi-Crystalline Polymers			Amorphous Polymers	
	HDPE	Nylon 6	PPS	ABS	PC
Izod Impact Strength	-41%	-54%	-29%	-97%	-98%
Tensile Modulus	24%	32%	18%	25%	29%
Tensile Strength at Break	359%	69%	-47%	43%	-48%
Tensile Elongation at Break	-89%	-97%	-43%	-89%	-99%
Flex Modulus	277%	13%	306%	44%	50%
Composite Density	4%	4%	3%	4%	3%
Heat Distortion Temperature	10%	75%	2%	5%	0.19%

$$\frac{CB\ filled\ Polymers_{(Bulk\ Properties)} - Unfilled\ Filled\ Polymers_{(Bulk\ Properties)}}{Unfilled\ Polymers_{(Bulk\ Properties)}} * 100\% \qquad \text{(Eqn.}$$

5.1)

Similar patterns/relationships can be observed for all CB filled composites to which increase is seen in Tensile Modulus and Flexural Modulus. In addition, minimal increase is seen in heat distortion temperature and density while significant reduction is seen in impact strength and tensile elongation properties.

5.6. Summary

From the results obtained in this section, several assumptions can be made which will guide in the preparation of subsequent formulations. The following are the key elements needed in developing a polymeric heater:

- According to the electrical properties measured, the use of semi-crystalline polymer is preferred since the reduction in resistance is orders of magnitude

higher then amorphous based polymers. Therefore, base carrier should consist of a semi-crystalline polymer.

- Determining percent crystallinity of polymer and composite can help guide selection of optimal semi-crystalline polymer.

- To check for sample composition, density comparisons between measured and theoretical values can be computed and compared when Thermal Gravimetric Analysis (TGA) is not possible.

- Both compositional analysis techniques (TGA & Density) show significant variations of filler loading suggesting that either feeding equipment and or techniques used to introduce materials into the extruder needs modification.

- For resistive heating to occur, the overall resistance of the tensile bar must be lower than 1.5×10^5 ohms as shown in Table 5.2.

- The resistance of the sample remains constant during heating. Therefore the plateau temperature achieved is due to energy balance rather than PTC type phenomenon.

- Mechanical properties can increase (Tensile/Flex Modulus, HDT and Density) and decrease (Impact, Elongation) with the addition of CB. This change is mainly dependent on filler type, loading percent and polymer matrix.

CHAPTER VI

EXPERIMENTAL RESULTS II,

DEVELOPMENT OF POLYMERIC HEATER

6.1. Introduction

Up to this point, the main objective was to determine and select a suitable

polymer matrix as the base carrier for developing a polymeric heater. From the

experimental results obtained in chapter 5, it became clear that this technology works

better in semi-crystalline polymers. This is not to say that this technology would be

limited to semi-crystalline polymers because by increasing filler (carbon black) loading it

is possible to achieve similar results for amorphous based systems. However this is not

optimal solution since increased CB loading can enhance process difficulty in addition to

unwanted effects on mechanical properties. Therefore, the use of semi-crystalline

polymers is preferred and was used such that the CB loading can be kept to a minimum.

In this chapter, suitable semi-crystalline polymer matrix/matrices were selected as

base carrier and its corresponding composite were prepared and tested. Electrical,

thermal and physical properties were then measured as a function of filler loading. In

addition the heating efficiency, uniformity and reproducibility/repeatability were

measured to determine whether this technology of developing a polymeric heater is

viable.

6.2. Base Carrier (Polymer Matrix)

Due to the unique thermal stability of PPS, processability of HDPE and experimental results from section 5 (resistive heating), a blend of PPS and HDPE was used as the polymer base carrier. The major polymer blend constituted PPS in order to maintain its thermal stability. While a minimum amount of HDPE were added to improve overall processability during the extrusion and molding. As for fillers, carbon black (CB) and graphite (GR) were used to increase electrical conductivity. Although graphite was not an ingredient used in section 5, plenty of work has been published suggesting when fillers with high aspect ratio are used in conjunction with CB, electrical conductivity to increase by two fold (10, 19). This occurs since the mixture of graphite-carbon black structure creates a better path for the current to cross resulting in a higher electrically conductive composite. In addition, graphite has a higher thermally conductivity compared with carbon black, therefore may improve heat transfer and heating rate. Finally, graphite's sheet like structure will help improve mechanical stability (23).

6.3. Formulations

Formulations prepared and tested are shown in Table 6.1. Included are formulations one through thirteen (ID#5 not included). As noted earlier, the majority of the polymer is made up of PPS (min-63% & max-76%) sum of both fillers make up (min-9% & max-18%). In addition a set of repeats, ID# 3 and 11 are included in order to test for repeatability and reproducibility.

Table 6.1: Formulation Table

Materials	Formulations											
	#1	#2	#3	#4	#6	#7	#8	#9	#10	#11	#12	#13
PPS	66	66	66	74.3	66	69	62.93	71	71	66	76	67
Carbon Black	4.2	7	9.8	4.7	4	5	9	9	0	9.8	7	9
Graphite	9.8	7	4.2	11.0	10	5	9	0	9	4.2	7	4
[1]HDPE	20	20	20	10.0	20	21	19.07	20	20	20	10	20
Total (%)	100	100	100	100	100	100	100	100	100	100	100	100

[1] Solvay high density polyethylene in powder form

6.4. Electrical Properties

Electrical properties were again measured using techniques described in chapter 4.4.2 (Tensile bar/brass screw technique). The resistance of each formulation shown in Table 6.1 was measured and their corresponding results are shown (Figure 6.1). Here we clearly see all formulations excluding ID#10 have resistance lower then $1x10^5$ ohms suggesting that upon electrification, samples will generate heat. This observation was first made from experimental results obtained in chapter 5.
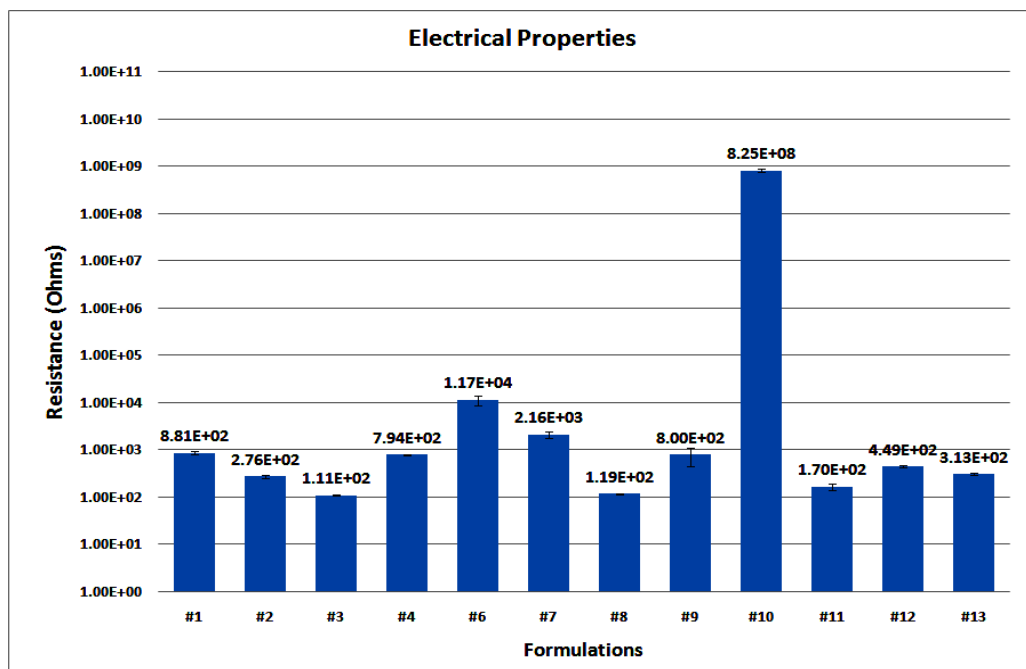
Figure 6.1 Electrical Resistance of PPS-HDPE Blend with CB & GR

Interestingly, two formulations tested contain single filler; sample 9 contains nine percent carbon black while sample 10 contains nine percent graphite. Their corresponding electrical resistance is in accordance with their inherent properties. Sample containing carbon black (#9) has a lower resistance since CB is more electrical conductive than graphite (#10) while graphite imparts stronger thermal conductivity.

6.5. Thermal Properties

Thermal properties were measured using same techniques implemented in section 5.2.2. A voltage regulator (Variac) was used to adjust the voltage desired for each test. For each formulation, four voltage setting; 15V, 30V and 70V and 100V (AC) were used to run each test. Figures 6.2 through 6.5 contain plots of heating rates for each formulation as a function of voltage for duration of fifteen minutes.
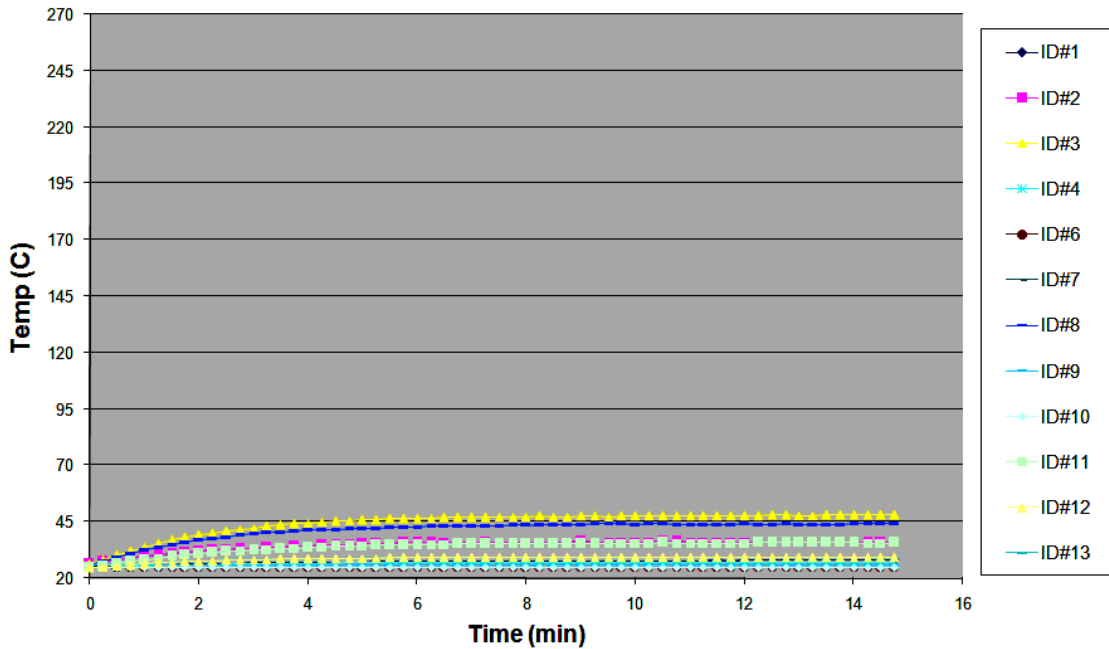
Figure 2Figure 6.2: Heat Generation at 15V Alternating Current (AC)

At 15V we can clearly see that the more conductive formulations begin to generate heat while others remain unchanged. Also, steady state is achieved in approximately 5-6 minutes. Finally we can conclude that formulation #3 had the highest heat generation reaching up to 48ºC.

Increasing the voltage setting to 30V yields higher temperature generation as can be seen in Figure 6.3. Here we can clearly see additional samples beginning to generate heat which did not at 15V. Again steady state is achieved at the 5-6 minute mark. However, formulations 6, 7 and 10 still fail to generate heat and remain at room temperature. Interestingly samples 3 and 8 heated beyond the boiling point heated

Figure 6.3: Heat Generation at 30V Alternating Current (AC)

beyond the boiling point of water at only 30V. Comparing formulation repeats of 3, 11 reveal that the plateau temperatures reached are not the same even though formulations are identical. Formulation 3 generated heat of up to133ºC while sample 3 only heated

107ºC, a difference of 26ºC. This behavior can be explained by comparing their corresponding resistance as illustrated earlier in Figure 6.3. Here we clearly see that resistance of sample 3 is lower than sample 11, and by as much as 55% thereby making sample 3 more conductive and more prone to heat generation.

As the voltage is increased to 70V, we again see similar behavior as shown in Figure 6.4. Heat generation increases while the most conductive formulations; 3, 8 and 11 begin to generate heat beyond the thermal stability of polymer matrices causing them to degrade. In addition, sample 10 still fails to generate heat and remain at room temperature while sample 6 has started heating.
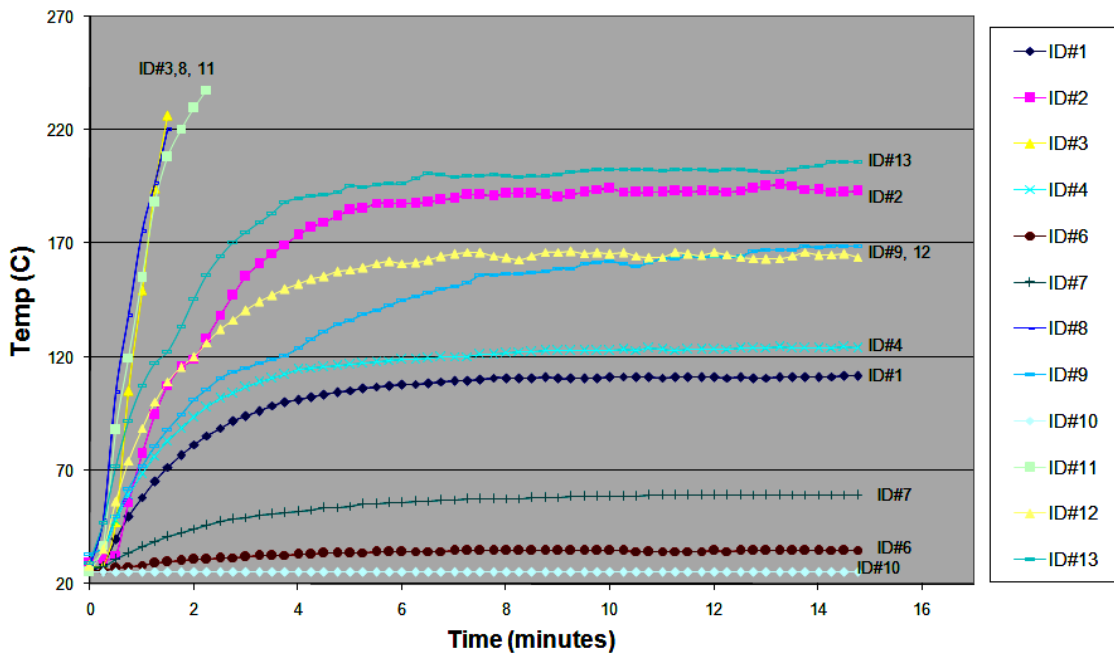
Figure 6.4: Heat Generation at 70V Alternating Current (AC)

Finally as the voltage is increased to 100V most if not all formulations begin to generate heat as can be seen in Figure 6.5. Once again certain formulations (ID#2, 9, 12, and 13) have heated beyond its thermal stability causing the part to degrade and fail while

formulation 10 remains at room temperature. From the plots shown in Figures 6.2

through 6.5 we can conclude that in order for any sample to generate heat it must allow

enough current to conduct through bulk of polymer composite. This is dependent on part

geometry, formulation and voltage applied to the composite.
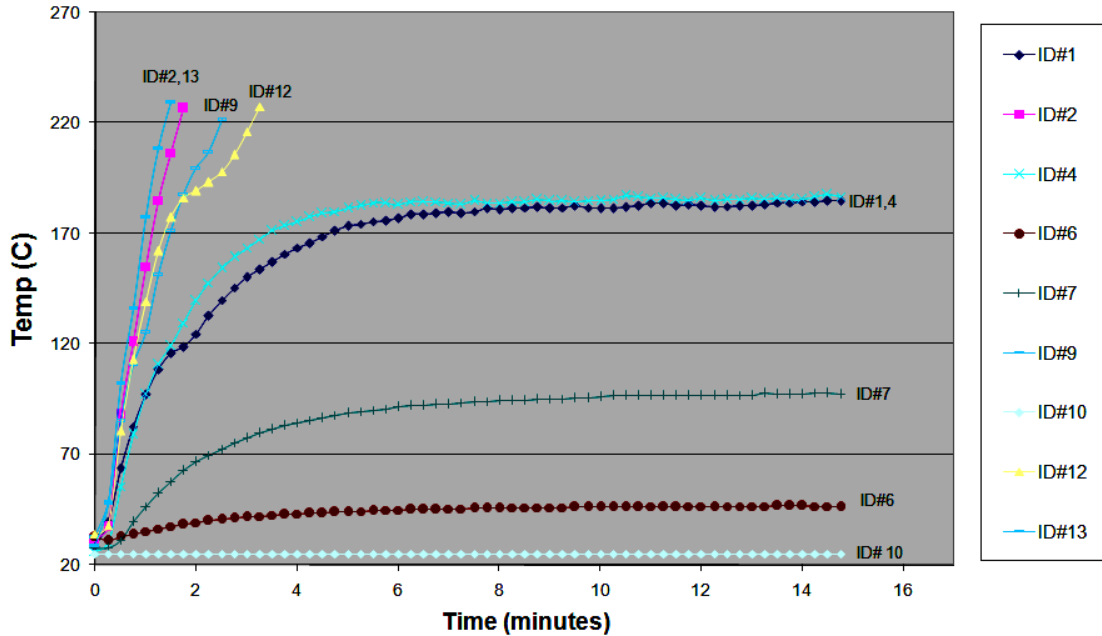
Figure 6.5: Heat Generation at 100V Alternating Current (AC)

Corresponding plateau temperatures achieved for all formulations at various

voltages are shown in Table 6.2. Here we can clearly see effects of voltage on plateau

temperature. As voltage increases, plateau temperature increases (excluding ID#10). In

addition, samples showing highest conductivity generated highest heating effect (plateau

temperature). This correlation is also evident from plateau temperature versus power plot

shown in Figure 6.6. Here the treadline indicates a linear model which is in accordance

with Joules law (Eqn 2.1) and energy balance (Eqn 2.8) once steady state is achieved.

Complete power calculations are shown in appendix Q.

Table 6.2: Plateau Temperatures at Various Voltage Settings

Voltage (AC)	Plateau Temperature (°C)											
	#1	#2	#3	#4	#6	#7	#8	#9	#10	#11	#12	#13
15V	29	38	52	30	26	26	51	27	25	44	34	37
30V	42	81	123	47	26	29	124	34	25	107	60	74
70V	114	>238	>254	121	34	58	>254	163	25	>238	169	222
100V	185			186	46	96		>255	25		>255	>254
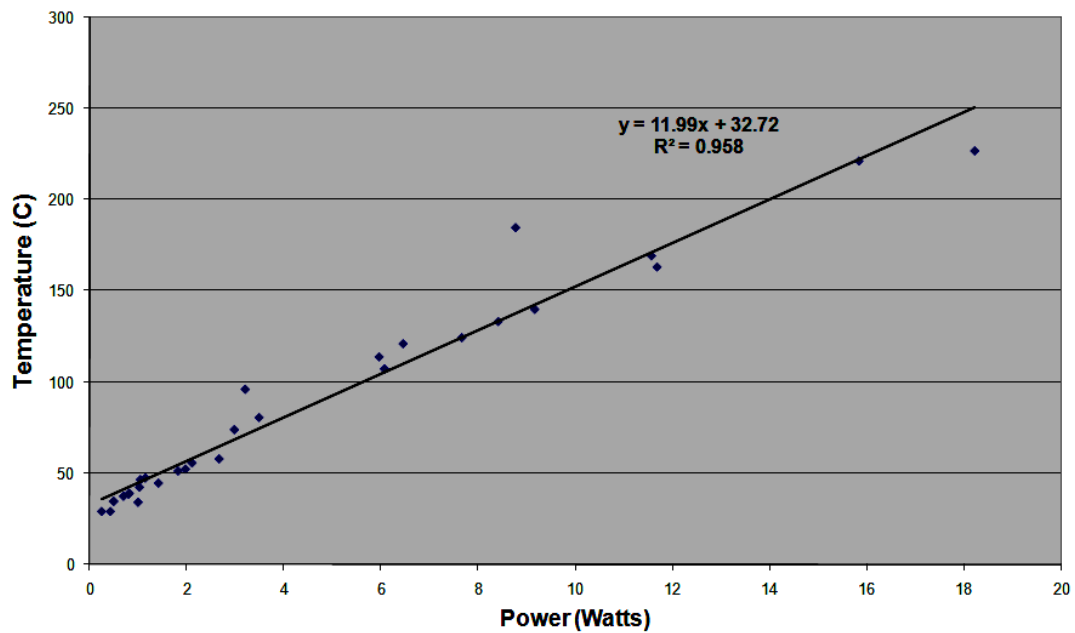
Figure 6.6: Plot of Plateau Temperature vs. Power

6.6. Repeatability

In order to determine whether heating effect is repeatable, specimen of the same formulations were heated for five cycles. Thereafter, plot was generated to determine if results overlapped. As can be seen from Figure 6.7, the heating rates of formulations 1, 2 and 3 are shown under testing conditions of 30 volts (AC). Clearly, we can see that the results overlap and plateau temperatures reached are nearly identical. The deviations between respective specimens are within 5 degrees Celsius.
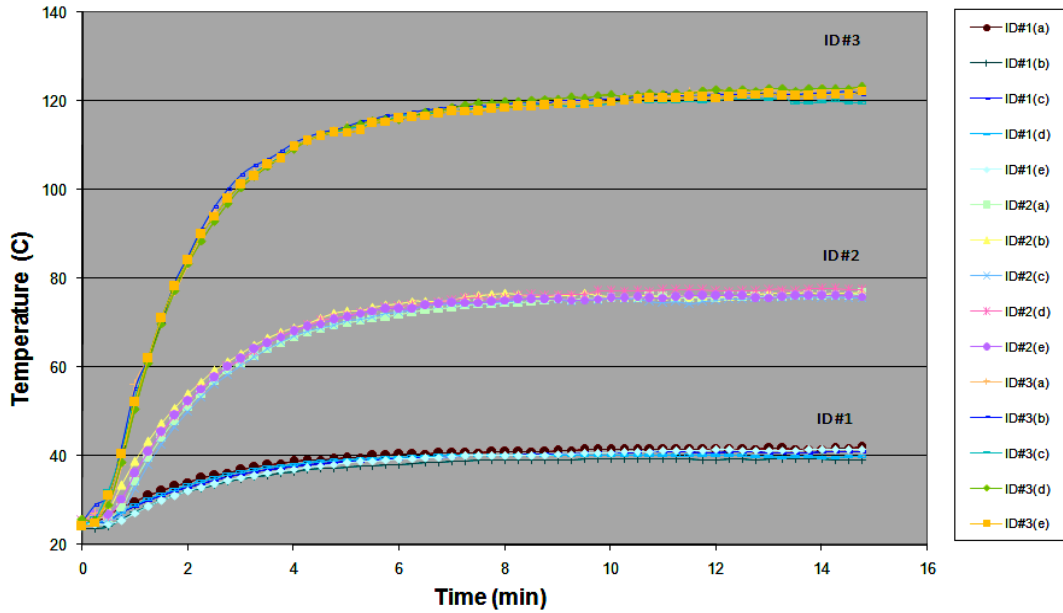
Figure 6.7: Repeatability under 30V (AC)

In addition, the effect of a heating/cooling cycle was investigated to determine whether electrical properties remain constant. This test was conducted due to literature work showing improved electrical properties upon sample heating (annealing). The technique of annealing is a common practice for certain polymers since it can enhance certain properties due to increase of crystallinity of the polymer.

Two formulations ID#1 and 13 were heated to150 degrees Celsius and cooled to 25 degrees Celsius for five cycles. Its corresponding resistance at pre-heat conditions was recorded for each cycle as can be seen in Figure 6.8. There is a slight decrease in resistance ranging from 3 to 8 percent after the first heating effect which. Thereafter, resistances of samples remain nearly constant. The slight decrease in resistance during first stage of heating may perhaps have to do with the increased crystallinity of the HDPE.
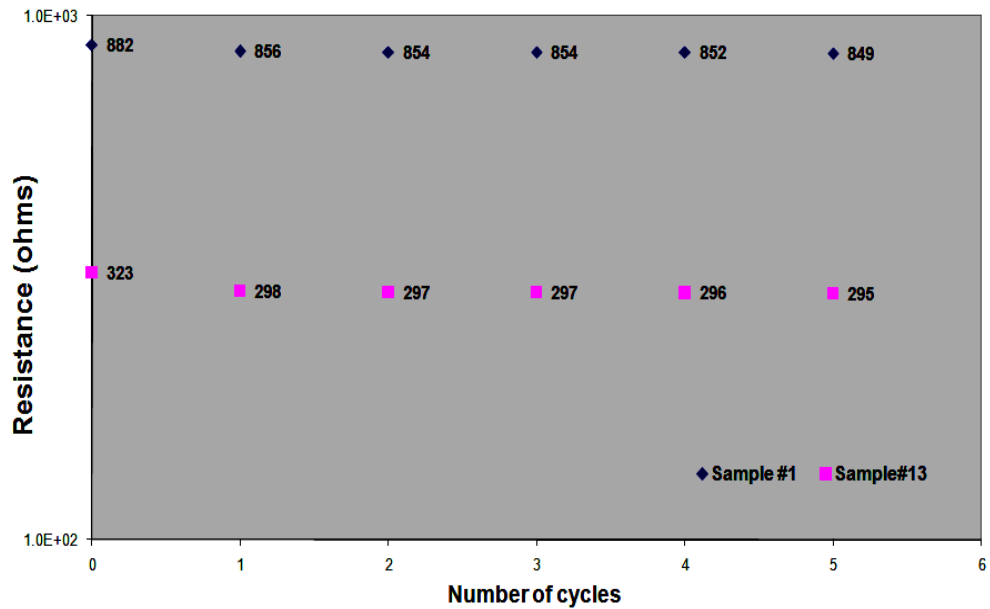
Figure 6.8: Cycle Study (Effect of Heating/Cooling on Resistance)

6.7. Composite Resistance during Resistive Heating

In order to determine the mechanism by which a polymeric heater operates (energy balance), we need to verify that the resistance remains constant during operation and that no other mechanisms such as Positive Temperature Control (PTC) exists. In order to measure the resistance of a sample during heating, an ammeter was connected in series such that current measurements could be made as heating took place. Figure 6.9 shows the effect of resistance to temperature for all formulations prepared excluding ID#10 which did not heat. From this plot, we can clearly see that the resistance remains constant with respect to temperature. Therefore we can conclude when plateau temperature/steady state is reached, it occurs due to the energy balance from heat lost to environment via convection.
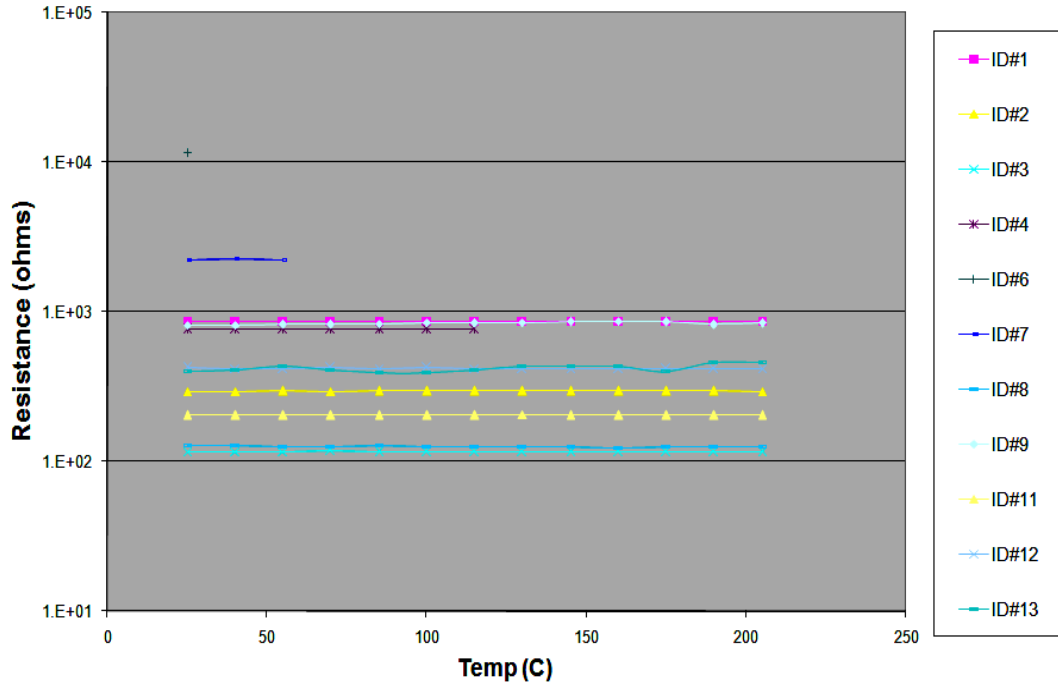
Figure 6.9: Effect of Temperature on Composite Resistance

6.8. Thermal Imaging

Heat uniformity during electrification was captured using a thermal imaging camera. A plaque measuring 4"x 6" x 0.125" was used to maximize an area of view. Here, two strips of conductive copper tape were embedded (over-molded) in parallel 4 inches apart. This was done to evenly distribute electric current across plaque (as opposed to the brass screw method). Further description on sample preparation and test method is discussed in section 4.4.2.2.

A thermal image of a electrified composite after 5 minutes is shown in Figure 6.10. Here we can see that the heat distribution for surface area of sixteen centimeters squared (4x4cm) to range between 50-70 degrees Celsius.
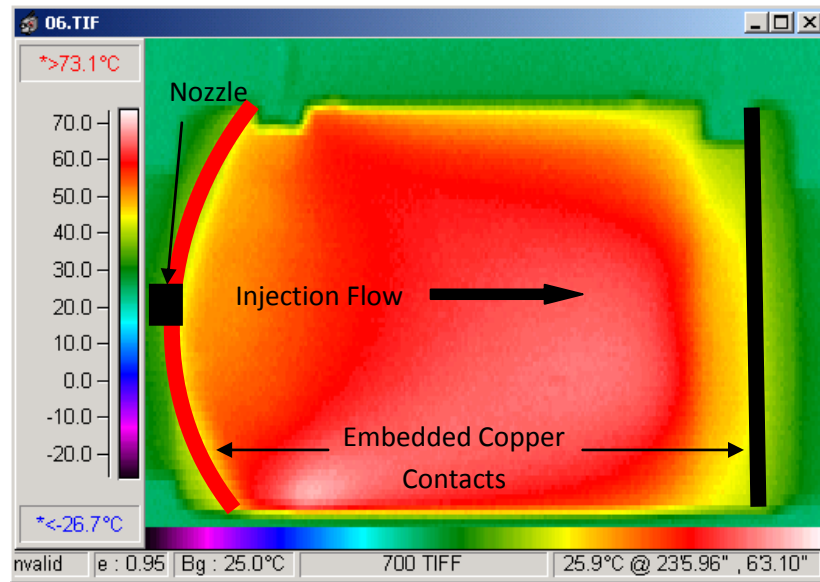
Figure 6.10: Thermal Image of Charged Plaque with Embedded Copper Tape

6.9. Efficiency

The purpose of this the study was to understand the heating efficiency of the composite during electrification and whether it resembles the 99% efficiency seen in common resistive heating applications. Testing techniques and equations used to calculate efficiency is described in chapter 4.5. Measured efficiency was computed to be in the range of 87-94% as shown in Table 6.3. The testing technique may have limited the overall heat transfer between the electrified composite and water due to the silicon caulking covering parts of the tensile bar thereby reducing the overall efficiency. However, we can conclude that the overall efficiency of the composite mimics that seen in common resistive heating elements (Ni-Chrome wires).

Table 6.3: Efficiency Data

Sample #3								
Specific heat of water = 4.184 J/g C								
Mass of water = 752.7 grams				Temperature				
Time	Volts	Current	Power In	Initial	Final	ΔT	Power Out	Efficiency
(sec)	(V)	(Amp)	(Watts)	(°C)	(°C)	(°C)	(Watts)	(%)
600	30	0.261	7.83	27	28.3	1.3	6.823	87
1020	30	0.258	7.74	27	29.2	2.2	6.793	88
1380	30	0.257	7.71	27	30	3	6.846	88
1740	30	0.256	7.68	27	31	4	7.241	94

6.10. Physical Properties

The mechanical properties of each composite (Formulations 1-13) were tested in accordance to ASTM standards as described in section 4.4. The results are shown in Figure 6.11 which contains Impact, Tensile and Flexural Modulus data. Here we can clearly see the influences of filler on properties. First, both Tensile and Flex Modulus has increased compared to the neat PPS tested earlier in chapter 5. Second, as expected tensile elongation properties have decreased for all samples while impact properties remained nearly constant compared with neat PPS. Comparing samples 9 and 10 reveal that graphite provides superior improvements to both tensile and flex modulus compared with carbon black at same loading.
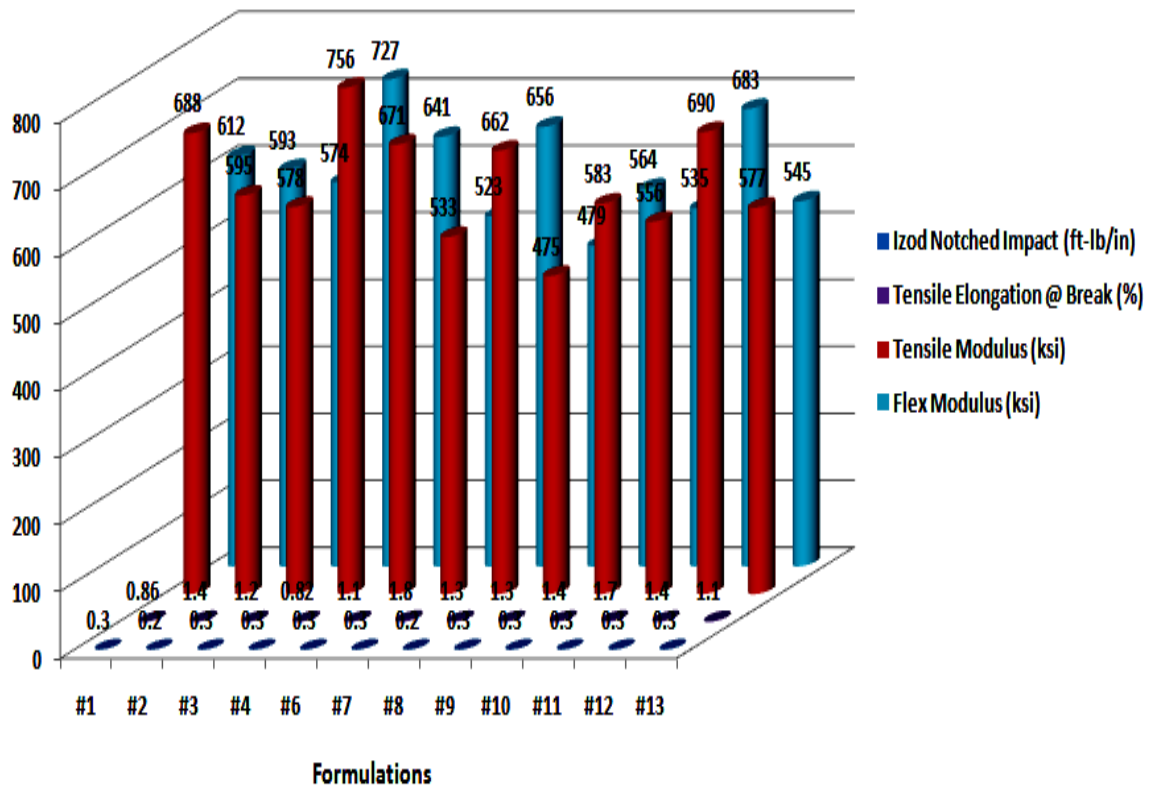
Figure 6.11: Mechanical Properties

The densities of the composite samples were measured using ASTM D792 and compared with theoretical values calculated using equation 4.2. Table 6.4 contains all the densities measured and theoretical densities calculated in addition to the percent error between the two. From this result we can clearly see a range of error between 0.94 to 2.7 percent error. Because the percent error values are negative, we can assume that the composites contain less carbon black ($\rho = 1.8$) or graphite ($\rho = 2.25$) or both. This suggests that our feeding technique during in extrusion maybe inadequate.

Table 6.4: Comparison between Measured and Theoretical Densities

Formulations	Measured Density (g/cm^3)	Theoretical Density (g/cm^3)	(%) Error
#1	1.295	1.260	**-2.735**
#2	1.290	1.276	**-1.102**
#3	1.285	1.273	**-0.939**
#4	1.358	1.339	**-1.409**
#6	1.296	1.269	**-2.068**
#7	1.269	1.243	**-2.043**
#8	1.312	1.294	**-1.363**
#9	1.262	1.242	**-1.577**
#10	1.278	1.243	**-2.741**
#11	1.285	1.273	**-0.939**
#12	1.344	1.327	**-1.252**
#13	1.282	1.261	**-1.602**

6.11. Summary

From the results obtained in this section, following conclusions can be made:

- It is possible to develop polymer composites that can deliver heat and via resistive heating with self regulating properties.

- Plateau temperature is determined by electrical property of composite (formulation) and amount of current that travels across specimen (determined by voltage). Too much power can cause sample to heat beyond its thermal and mechanical stability causing degradation (formulation dependent).

- Inconsistencies seen in reproducibility comparison between formulations 3 and 11. This may be due to inadequate feeding equipment/technique as verified from density calculations, or overall testing techniques using brass screws may have resulted to the incomplete contact of screw to composite.

- Linear relationship seen between plateau temperature and power (Figure 6.7) which is in agreement with Joules law equation 2.1.

- Repeatability and cycle study shows that the system does not exhibit hysterisis.

- Composite resistance remains constant at all temperatures 23-200 degrees Celsius, and does not exhibit PTC type behavior.

- Thermal imaging shows entire sample surface to heat during charge, however temperature ranges between 50-70 degrees Celsius.

- Heating effect appears to be efficient (87-94%) much like the common Ni-Chrome wires (99% efficiency)

- Deviations of measured and theoretical densities indicate that our feeding system maybe inadequate.

- Graphite did not significantly influence electrical (resistance) and or thermal (heating rate) conductivity as anticipated. However improvements were seen in mechanical properties.

CHAPTER VII

MODELING & ANALYSIS

7.1. Introduction

In order to model electrical (resistance), thermal (plateau temperature) and physical (Tensile and Flex) properties of a polymeric composite, the use of statistical design software was implemented. This statistical based process is a well established and thereby results in the valid and objective conclusion of model. It is a powerful tool used in many fields which helps (37, 39):

- Determines interaction of variables

- Determines variables which are influential to response

- Determines were to set influential variables in order to achieve favorable response

- Determines whether the desired response is even possible

- Efficient process which allows experimenter to reduce number of experiments

- Reduce time and cost to design/develop new products & processes

- Improve reliability and performance of products

- Evaluation of materials, design alternatives

In this chapter, Design-Expert® by Stat-Ease is used to model data collected from chapter 6. In particular, the electrical, thermal and physical properties will be modeled and analyzed to determine if correlation between formulations to responses

exists and whether these results are desired and or meaningful.

7.2. Approach to Modeling

A crossed D-Optimal design method was used to model the system. This method accounts for systems with both mixture design (PPS, HDPE, carbon black, and graphite) and process design (voltage) factors. In order to model effect, each of the mixture components, process factors and response were inputted accordingly. For the mixture design it is necessary to ensure that the sum of all components equal 100 percent (37-39). For our purposes, the mixture components are essentially formulations which all sum up to 100 percent. The factor process in our experiment consists of voltage adjustments made during resistive heating (15, 30, and 70 volts AC). The responses consist of plateau temperature, resistance, tensile and flex modulus. Appendix Q contains design parameters which includes mixture components, factors and responses used in the D-Optimal modeling process.

7.3. Analysis of Variance (ANOVA)

To determine whether and how the model fits, it is first necessary to review ANOVA (Table 7.1). By using Design-Expert® software, we will be able to analyze and determine whether model is significant or not. Corresponding ANOVA results of all analysis are shown in appendix R.

Table 7.1: Analysis of Variance (ANOVA) Key Terms (37)

Sum of Squares (SS)	Sum of the squared deviations from the mean due to the effect of the term
Degrees of Freedom (df)	Measures the number of independent pieces of information
Mean Square	The Sum of Squares (SS) / Degrees of Freedom (df)
F-Value	(Mean Square / Residual Mean Square); *The larger the F-Value the likely the variance contributed by model is significantly larger than random error.*
p- Value	*If Probability > 0.05 then the model has significant effect on the response.*
Standard Deviation (Std. Dev)	Square root of the Residual Mean Square, associated with experimental error.
Mean	Average value of a particular response
Coefficient of Variation (CV %)	(Std.Dev / Mean)*100; Error expressed as % of the mean
Predicted Residual Sum of Squares (PRESS)	Measures how well model fits each point. The smaller the PRESS statistic the better the model fits the data
R-Squared	Variation of data to model (SS $_{Model}$ / SS$_{Total}$)
Adjusted R-Squared	R-Squared adjusted for the number of parameters in the model relative to number of data in design.
Predicted R-Squared	How well the model predicts response value. *Should be within 0.2 of adjusted R-Squared to be in reasonable agreement.*
Adequate Precision	Measure of the range in predicted response relative to its associated error. *Basically it is the signal to noise ratio. Desired value is above 4*

Results 7.4

7.4.1. Thermal (Plateau Temperature)

Plateau temperature model generated by Design-Expert® software is shown in

Figure 7.1. As can be seen, the x-axis represents fillers (CB and GR), y-axis represents

voltage settings while polymer blend is held constant at PPS (76%) and HDPE (10%).

The contour lines represent plateau temperatures while the coloring scheme, red indicates

higher temperature and blue indicates lower temperature. From this model we can

determine that carbon black is more significant in yielding a higher temperature through

resistive heating as compared to graphite. In addition, as voltage is increased, plateau

temperature increases which is in accordance to principles of resistive/Joule heating and

Ohms law (Eqn 2.1-2.3). Finally, equation 7.1 contains a model equation determined by software. From this equation we can conclude that CB loading and voltage settings are the significant terms for designing polymeric type heaters. Finally, using this equation, we can predict plateau temperature of a given mixture and voltage.
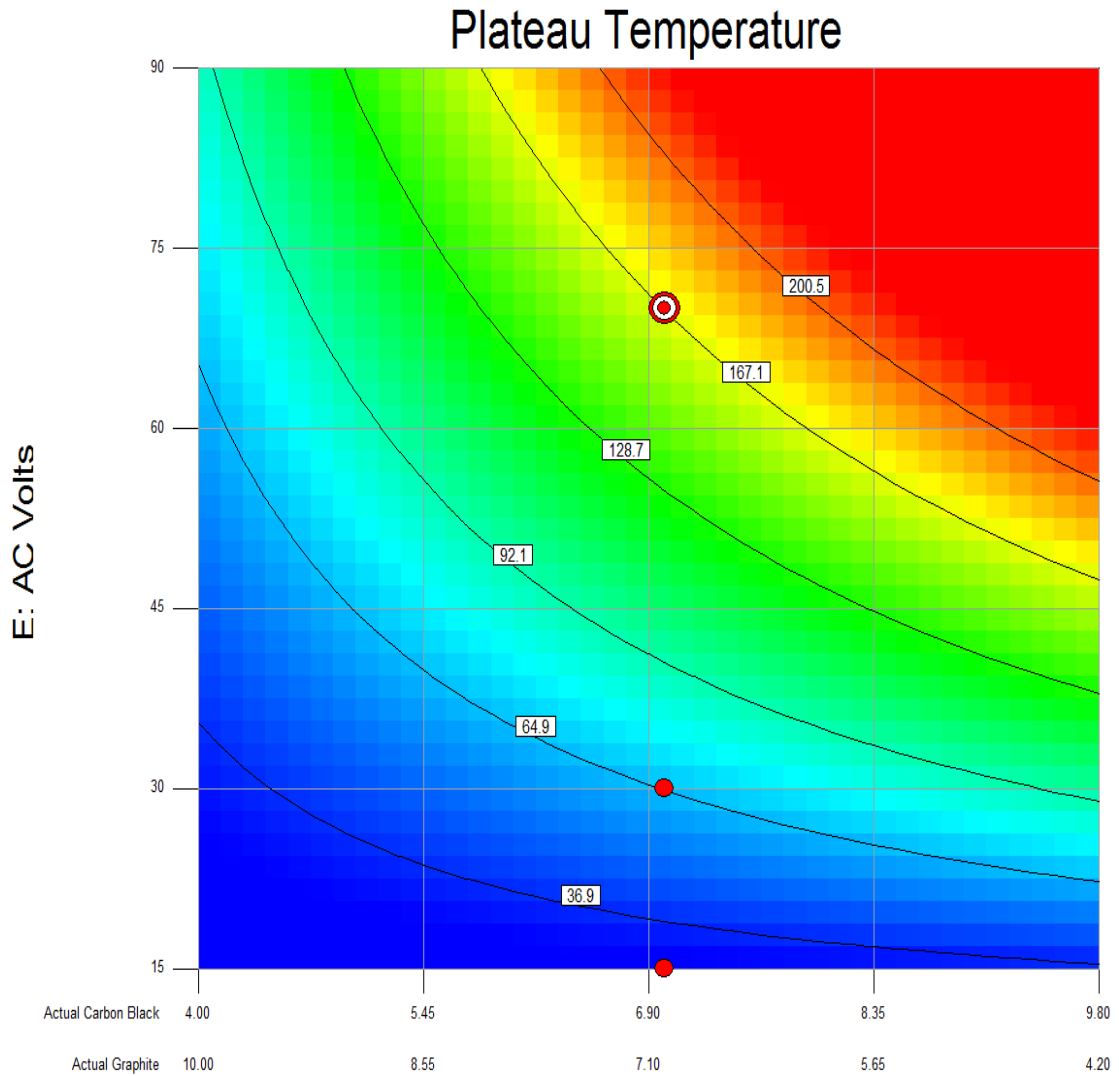
Figure 7.1: Modeling of Plateau Temperature at 76%PPS: 10%HDPE

Equation 7.1: Final Equation in Terms of Actual Components and Actual Factors:

Plateau Temperature (ºC) =

-0.16633	* PPS
-3.21333	* Carbon Black
+1.84721	* Graphite
+1.06247	* HDPE
-0.036923	* PPS * AC Volts
+0.68682	* Carbon Black * AC Volts
+0.14647	* Graphite * AC Volts
-0.046865	* HDPE * AC Volts

7.4.2. Electrical Resistance (Ohms)

A resistance model generated by Design-Expert® software is shown in Figure

7.2. This figure contains a ternary plot which shows three components of mixture; PPS,

CB and GR. The last component HDPE is held constant at 10 percent. The contour lines

represent electrical resistance values in inverse square root (due to transformation

function). From this figure and model equation shown (Eqn 7.2) we can clearly conclude

that CB is the most significant term in reducing composite resistance followed by

graphite. As expected both polymers (PPS and HDPE) have negative effect on reducing

composite resistance due to their insulative properties.

Equation 7.2: Final Equation in Terms of Actual Components and Actual Factors:

1/Sqrt[Resistance] (Ohms) =

-1.02141E-003	* PPS
+0.013821	* Carbon Black
+4.51318E-003	* Graphite
-4.12617E-004	* HDPE
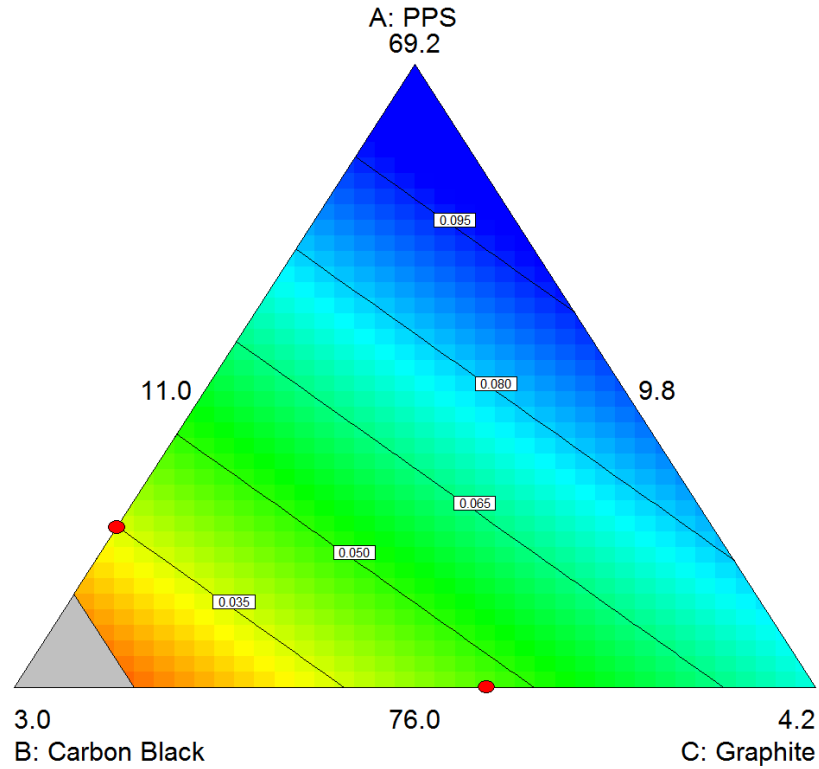
Figure 7.2: Modeling of Resistance with Inverse Square Root Transform

7.4.3 Physical Properties

7.4.3.1 Tensile Modulus

Tensile modulus model generated by Design-Expert® software is shown in Figure 7.3. This figure contains ternary plot which shows three components of mixture; PPS, CB and GR. The last component HDPE is held constant at 10 percent. The contour lines represent tensile modulus for any given mixture. From this figure and model equation shown (Eqn 7.3) we can clearly conclude that GR is the most significant term in increasing tensile modulus followed by CB and PPS content. HDPE resulted in the slight reduction of tensile modulus.
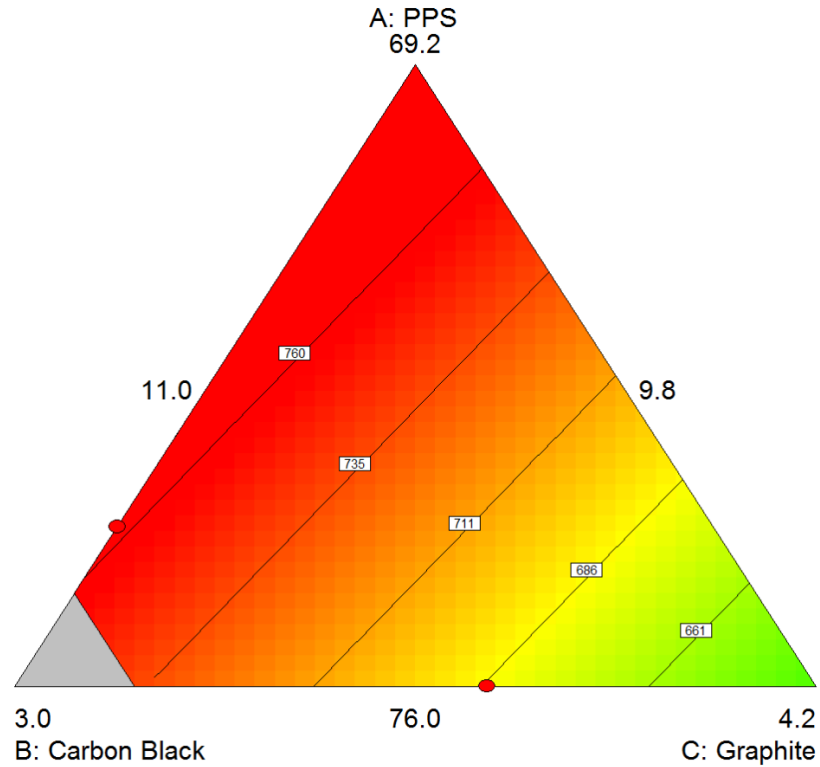
Figure 7.3: Modeling of Tensile Modulus

Equation 7.3: Final Equation in Terms of Actual Components and Actual Factors:

Tensile Mod =

+5.73767 * PPS
+10.11453 * Carbon Black
+27.35877 * Graphite
-1.33033 * HDPE

7.4.3.2. Flexural Modulus

Flexural modulus model generated by Design-Expert® software is shown in

Figure 7.4. This figure contains ternary plot with same mixture content as seen in tensile

modulus model. From this figure and model equation shown (Eqn 7.4) we can conclude

that model and coefficient s of significant variables (GR, CB and PPS) are almost

identical to those of tensile modulus. Again HDPE resulted in the slight reduction of

flexural modulus.
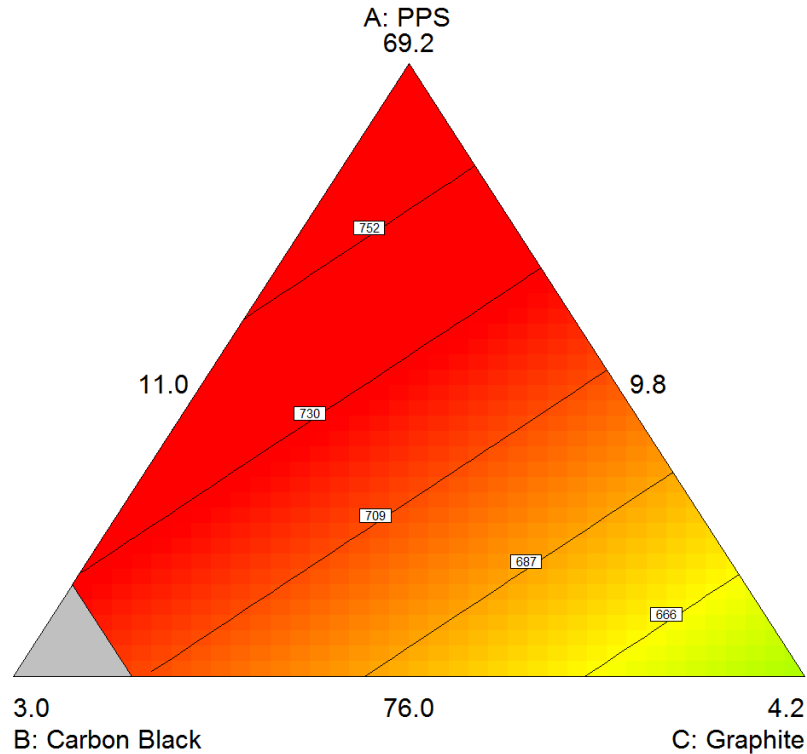
Figure 7.4: Modeling of Flexural Modulus

Equation 7.4: Final Equation in Terms of Actual Components and Actual Factors:

$$
\begin{aligned}
\text{Flex Modulus} = \\
+5.75348 \quad & * \text{ PPS} \\
+13.35340 \quad & * \text{ Carbon Black} \\
+24.72957 \quad & * \text{ Graphite} \\
-2.78943 \quad & * \text{ HDPE}
\end{aligned}
$$

7.6. Summary

Based on the modeling analysis performed using Design-Expert® we can say with confidence that models obtained are significant and therefore establish a clear correlation between formulations (inputs) to responses (outputs). In addition the following observations and remarks are made:

- Plateau temperature model showed significant interactions between mixture components (PPS, HDPE, carbon black and graphite) and process factor

(voltage). Based on the modeled equation, we can conclude that highest influence on plateau temperature is carbon black content and voltage applied.

- From the resistance model, we can clearly see the strongest component in reducing resistance is again carbon black.

- Both Tensile and Flexural Modulus showed similar behavior. In both cases, graphite had the highest influence followed by carbon black and PPS in improving stiffness and strength, while HDPE had a negative effect in both cases.

The models/results obtained from electrical (resistance) and physical (tensile and flex) are not only logical but in agreement with past studies/literature further suggesting that our models are valid. More importantly, analyzing the thermal (plateau temperature) model suggests that it is possible develop and model a polymeric heater

CHAPTER VIII

CONCLUSION & FUTURE RECOMMENDATION

8.1. Conclusion

The objective of this thesis was to develop a self regulating high temperature polymeric heater capable of heating to temperatures above those seen in current technology and as viable alternative and/or substitute to Ni-chrome heating elements. As a result of this work, several important conclusions were derived and are discussed in subsequent section.

- Significant advantage exists in using semi-crystalline polymers as base carrier due to their superior electrical properties at equivalent filler loading compared to amorphous based composites, a criterion in joule heating. This phenomenon is due to the conductive fillers being dispersed uniformly within the amorphous matrix while in semi-crystalline polymers the conductive fillers are ejected from the crystalline regions during crystallization and are concentrated in the amorphous region. This effect enables lower filler loading for semi-crystalline polymer to achieve percolation threshold due to the localized buildup of fillers.

- Determining percent crystallinity difference between polymer and composite can help guide in the selection of optimal polymer as base carrier.

- Heating rate and maximum/plateau temperature are merely a function of specimen resistance (formulation parameter) and voltage setting.

- The resistance of the sample remains constant during heating suggesting, that the self regulating effect is due to energy balance rather than PTC type phenomenon.

- Heating effect appears to be somewhat uniform; filler orientation seems to dictate flow orientation and thereby influencing heat uniformity.

- Heating effect is reproducible, no evidence of hysterisis upon heating cycle.

- The addition of fillers (carbon black and graphite) can increase certain mechanical properties such as tensile/flex modulus, heat distortion temperature and density while decreasing others impact and elongation.

- Inadequate feeding equipment/technique, verified from density calculations.

- DOE models of thermal (plateau temperature), electrical (resistance) and physical (tensile and flex) where found to be significant.

Finally, the experimental results and model indicates it is possible to develop polymeric type heaters that can operate above 100ºC (current technology) and as high as 200ºC. Moreover, these devices would have self regulating properties other than positive temperature coefficient (PTC) type mechanism seen in olefin based heaters.

8.2. Future Recommendation

Based on the experimental results obtained and observations seen, following suggestions are made for future recommendations.

Raw Materials

- Additional high temperature semi-crystalline polymers should be investigated as base carriers to determine if similar results as those seen in this thesis transpire.

- Incorporating polymer blends may also provide additional advantage with respect to electrical, mechanical and thermal stability in addition to possible reduction of percolation threshold.

- To minimize variation of composite resistivity during feeding/extrusion various grades of fillers (both low and high) could be blended to reduce response sensitivity.

- Other fillers (organic or inorganic) which can better improve thermal conductivity should be investigated to develop composites which can heat faster.

Process Equipment

- Effect of screw design during extrusion should be investigated to determine effect of shear/mixing on electrical properties. Essentially, this will help better understand how much work is needed to effectively disperse fillers without damaging the filler (carbon black) structure/network.

- Feeding individual components separately and concurrently through multiple feeders during extrusion could help reduce inconsistencies seen in composite density and TGA test. This can be further accomplished by using gravimetric feeders (mass flow rate self adjusts accordingly to minimize variations) as opposed to the volumetric feeders (constant mass flow rate).

Testing Technique

- Alternative testing method should be implemented to measure electrical and thermal properties. The current method of brass screw and tensile bar presents various contact problems between the screw and composite.

Additional Testing

- The life expectancy of the polymeric heater during operation should be

    investigated.

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APPENDICES

Appendix A: FORMULATIONS

Materials	Chapter 5: Experimental Formulations									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10a
Dow's HDPE	100					91				
Nylon 6		100					91			
PPS			100					91		
ABS				100					91	
PC					100					91
Carbon Black						9	9	9	9	9
Total (%)	100	100	100	100	100	100	100	100	100	100

Materials	Chapter 6: Experimental Formulations											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
PPS	66	66	66	74.3	66	69	62.93	71	71	66	76	67
Carbon Black	4.2	7	9.8	4.7	4	5	9	9	0	9.8	7	9
Graphite	9.8	7	4.2	11.0	10	5	9	0	9	4.2	7	4
Solvay's HDPE	20	20	20	10.0	20	21	19.07	20	20	20	10	20
Total (%)	100	100	100	100	100	100	100	100	100	100	100	100

Appendix B: EXTRUSION CONDITIONS

Samples	#1a		#2a		#3a		#4a	
	Set	Actual	Set	Actual	Set	Actual	Set	Actual
Zone 1 (C)	190	189	220	220	280	279	225	225
Zone 2 (C)	190	190	220	219	280	280	225	225
Zone 3 (C)	200	201	230	231	290	289	230	232
Zone 4 (C)	200	200	230	230	290	290	230	232
Zone 5 (C)	200	200	230	230	290	292	230	230
Zone 6 (C)	210	211	235	237	290	290	235	237
Zone 7 (C)	210	211	235	239	295	296	235	235
Zone 8 (C)	210	217	240	243	295	299	235	242
Zone 9 (C)	210	212	240	240	295	295	235	235
Die Temp(C)	205	205	245	245	295	295	240	242
RPM	500		500		500		500	
% Torque	64		73		42		75	
Feeder Setting	30		12		25		30	

Samples	#5a		#6a		#7a		#8a	
	Set	Set	Actual	Set	Set	Actual	Set	Actual
Zone 1 (C)	270	270	190	220	220	220	300	282
Zone 2 (C)	270	270	190	220	220	220	300	300
Zone 3 (C)	280	280	202	230	230	231	300	300
Zone 4 (C)	280	280	200	230	230	230	310	310
Zone 5 (C)	280	280	200	230	230	230	310	310
Zone 6 (C)	280	280	212	235	235	238	310	310
Zone 7 (C)	285	285	213	235	235	239	315	315
Zone 8 (C)	285	285	217	240	240	246	315	315
Zone 9 (C)	285	285	210	240	240	240	315	314
Die Temp(C)	285	285	206	245	245	245	315	317
RPM	500		500		500		500	
% Torque	77		76		89		64	
Feeder Setting	14		28		11		30	

Samples	#9a		#10a	
	Set	Actual	Set	Actual
Zone 1 (C)	225	225	270	269
Zone 2 (C)	225	225	270	270
Zone 3 (C)	230	229	280	279
Zone 4 (C)	230	231	280	283
Zone 5 (C)	230	230	280	281
Zone 6 (C)	235	236	280	281
Zone 7 (C)	235	236	285	289
Zone 8 (C)	235	237	285	285
Zone 9 (C)	235	238	285	288
Die Temp(C)	240	240	285	285
RPM	500		500	
% Torque	83		84	
Feeder Setting	27		10	

Appendix B1: EXTRUSION CONDITIONS

Samples	#1b		#2b		#3b		#4b	
	Set	**Actual**	**Set**	**Actual**	**Set**	**Set**	**Actual**	**Actual**
Zone 1 (C)	290	279	290	270	300	267	290	290
Zone 2 (C)	290	290	290	290	300	300	290	290
Zone 3 (C)	290	290	290	290	300	300	290	290
Zone 4 (C)	300	300	300	300	310	310	300	300
Zone 5 (C)	300	300	300	300	310	310	300	300
Zone 6 (C)	300	300	300	300	310	310	300	300
Zone 7 (C)	300	300	300	300	310	310	300	300
Zone 8 (C)	305	305	305	305	315	315	305	300
Zone 9 (C)	305	305	305	305	315	315	305	304
Die Temp(C)	305	307	305	305	315	315	305	304
RPM	500		500		500		500	
% Torque	46		61		61		55	
Feeder Setting	17		17		16		17	

Samples	#6b		#7b		#8b		#9b	
	Set	**Actual**	**Set**	**Actual**	**Set**	**Actual**	**Actual**	**Actual**
Zone 1 (C)	290	278	300	270	300	280	300	268
Zone 2 (C)	290	290	300	300	300	300	300	300
Zone 3 (C)	290	290	310	300	310	310	300	300
Zone 4 (C)	300	300	310	310	310	310	310	310
Zone 5 (C)	300	300	315	315	315	315	310	310
Zone 6 (C)	300	300	315	315	315	315	310	310
Zone 7 (C)	300	300	315	315	315	315	315	315
Zone 8 (C)	305	305	320	320	320	320	315	315
Zone 9 (C)	305	305	320	320	320	320	315	315
Die Temp(C)	305	305	320	320	320	320	315	315
RPM	500		500		500		500	
% Torque	62		61		51		62	
Feeder Setting	22		34		45		37	

Samples	#10b		#11b		#12b		#13b	
	Set	**Set**	**Actual**	**Actual**	**Set**	**Actual**	**Set**	**Actual**
Zone 1 (C)	300	263	300	268	300	267	300	274
Zone 2 (C)	300	300	300	300	300	300	300	300
Zone 3 (C)	300	300	300	300	300	300	300	300
Zone 4 (C)	310	310	310	310	310	310	310	310
Zone 5 (C)	310	310	310	310	310	310	310	310
Zone 6 (C)	310	310	310	310	310	310	310	310
Zone 7 (C)	315	315	315	315	315	315	315	315
Zone 8 (C)	315	315	315	315	315	315	315	315
Zone 9 (C)	315	315	315	315	315	315	315	315
Die Temp(C)	315	314	315	315	315	315	315	316
RPM	500		500		500		500	
% Torque	62		61		51		62	
Feeder Setting	22		34		45		37	

Appendix C: EXTRUSION SCREW DESIGN

- F1 to F30 – Denotes double Flighted conveying/feeding elements
- Ext 1.5 - Single Flighted conveying elements
- Remaining Elements denoted as A (Alternating), F (Forward) and R (Reverse) are kneading blocks,
  Corresponding numeric number represents degrees offset from previous element.
- Materials are feed into section F-2/3 and exist through Ext.1.5 (die)
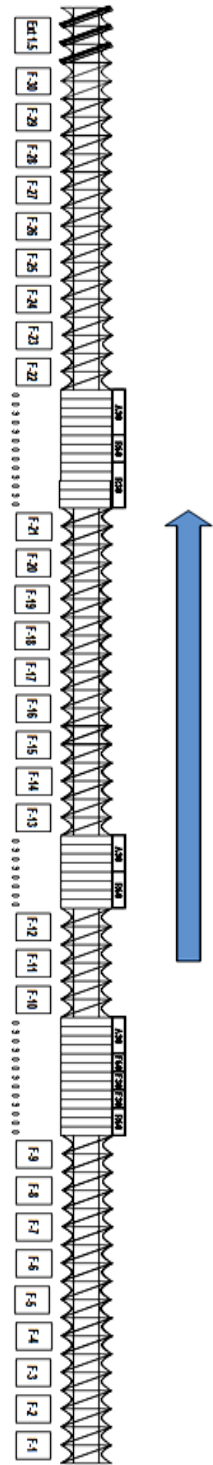
Appendix D: INJECTION MOLDING CONDITIONS

Formulations	#1a (HDPE)		#2a (Nylon 6)		#3a (PPS)	
	Set	**Actual**	**Set**	**Actual**	**Set**	**Actual**
Pre Mold (Drying)	-	-	4-6 hr/100ºC		4-6 hr/110ºC	
Temperatures:						
Nozzle (ºC)	220	219	255	257	315	311
Zone 1 (ºC)	215	216	250	252	310	307
Zone 2 (ºC)	210	209	245	245	305	305
Zone 3 (ºC)	200	200	240	239	300	299
Mold (ºC)	55	55	72	72	145	145
Speeds:						
Screw RPM	125	125	125	125	90	90
% Shot - Inj Velocity (in/sec)	100-1	100-1	100-1	100-1	100-1	100-1
% Shot - Inj Velocity (in/sec)	75-1	75-1	75-1	75-1	75-1	75-1
% Shot - Inj Velocity (in/sec)	50-1	50-1	50-1	50-1	50-1	50-1
% Shot - Inj Velocity (in/sec)	25-1	25-1	25-1	25-1	25-1	25-1
% Shot - Inj Velocity (in/sec)	0-1	0-1	0-1	0-1	0-1	0-1
Pressures:						
Pack/Hold (psi)	350	350	400	400	325	325
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	30	30	25	25	20	20
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.12		0.29		0.11
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8
Cut-Off (XFER) Pressure (psi)	650	650	575	575	410	410
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.2	0.2	0.4	0.4	0.35	0.35

*Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#4a (ABS)		#5a (PC)		#6a HDPE-CB	
	Set	Actual	Set	Set	Set	
Pre Mold (Drying)	4-6 hr/120ºC		4-6 hr/90ºC		-	
Temperatures:						
Nozzle (ºC)	250	252	300	300	225	226
Zone 1 (ºC)	245	244	295	295	220	221
Zone 2 (ºC)	240	240	290	290	215	213
Zone 3 (ºC)	235	235	285	285	205	204
Mold (ºC)	35	35	100	100	55	55
Speeds:						
Screw RPM	100	100	100	100	125	125
% Shot - Inj Velocity (in/sec)	100-2	100-2	100-2	100-2	100-1	100-1
% Shot - Inj Velocity (in/sec)	75-2	75-2	75-2	75-2	75-1	75-1
% Shot - Inj Velocity (in/sec)	50-2	50-2	50-2	50-2	50-1	50-1
% Shot - Inj Velocity (in/sec)	25-2	25-2	25-2	25-2	25-1	25-1
% Shot - Inj Velocity (in/sec)	0-2	0-2	0-2	0-2	0-1	0-1
Pressures:						
Pack/Hold (psi)	500	500	850	850	400	400
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	25	25	25	25	30	30
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.22		0.22		0.17
Cut-Off Position (in)	0.8	0.8	0.7	0.7	0.8	0.8
Cut-Off (XFER) Pressure (psi)	634	634	1120	1120	1000	728
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.2	0.2	0.3	0.3	0.2	0.2

*Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#7a Nylon-CB		#8a PPS-CB		#9a ABS-CB		#10a PC-CB	
	Set	Actual	Set	Actual	Set	Actual	Set	Actual
Pre Mold (Drying)	4-6 hr/100ºC		4-6 hr/110ºC		4-6 hr/90ºC		4-6 hr/120ºC	
Temperatures:								
Nozzle (ºC)	260	258	315	315	250	255	305	308
Zone 1 (ºC)	255	256	310	311	245	246	300	302
Zone 2 (ºC)	245	245	305	306	240	243	295	296
Zone 3 (ºC)	240	239	300	302	235	238	285	285
Mold (ºC)	72	72	145	145	35	35	100	100
Speeds:								
Screw RPM	125	125	90	90	100	100	100	100
% Shot - Inj Velocity (in/sec)	100-1	100-1	100-1	100-1	100-2	100-2	100-2	100-2
% Shot - Inj Velocity (in/sec)	75-1	75-1	75-1	75-1	75-2	75-2	75-2	75-2
% Shot - Inj Velocity (in/sec)	50-1	50-1	50-1	50-1	50-2	50-2	50-2	50-2
% Shot - Inj Velocity (in/sec)	25-1	25-1	25-1	25-1	25-2	25-2	25-2	25-2
% Shot - Inj Velocity (in/sec)	0-1	0-1	0-1	0-1	0-2	0-2	0-2	0-2
Pressures:								
Pack/Hold (psi)	450	450	350	350	650	650	950	950
Timers:								
Injection Hold Time (sec)	8	8	8	8	8	8	8	8
Cooling Time (sec)	25	25	20	20	25	25	25	25
Operation Settings:								
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.30		0.13		0.21		0.29
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8	0.7	0.7
Cut-Off (XFER) Pressure (psi)	1000	625	1000	475	1000	787	1000	1250
Cut-Off Time (sec)	10	10	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*	P*	P*
Decompression (in)	0.4	0.4	0.35	0.35	0.2	0.2	0.3	0.3

*Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#1b		#2b		#3b	
	Set	**Actual**	**Set**	**Actual**	**Set**	**Set**
Pre Mold (Drying)	4-6 hr/110ºC		4-6 hr/110ºC		4-6 hr/110ºC	
Temperatures:						
Nozzle (ºC)	315	311	315	316	315	315
Zone 1 (ºC)	310	310	310	310	310	310
Zone 2 (ºC)	305	305	305	305	305	305
Zone 3 (ºC)	300	299	300	300	300	297
Mold (ºC)	145	145	145	145	145	145
Speeds:						
Screw RPM	120	120	120	120	120	120
% Shot - Inj Velocity (in/sec)	100-2	100-2	100-2	100-2	100-2	100-2
% Shot - Inj Velocity (in/sec)	75-2	75-2	75-2	75-2	75-2	75-2
% Shot - Inj Velocity (in/sec)	50-2	50-2	50-2	50-2	50-2	50-2
% Shot - Inj Velocity (in/sec)	25-2	25-2	25-2	25-2	25-2	25-2
% Shot - Inj Velocity (in/sec)	0-2	0-2	0-2	0-2	0-2	0-2
Pressures:						
Pack/Hold (psi)	600	600	600	600	600	600
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	20	20	20	20	20	20
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.17		0.22		0.23
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8
Cut-Off (XFER) Pressure (psi)	1000	720	1000	733	1000	787
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.3	0.3	0.3	0.3	0.3	0.3

* Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#4b		#6b		#7b	
	Actual	**Actual**	**Set**	**Actual**	**Set**	**Actual**
Pre Mold (Drying)	4-6 hr/110ºC		4-6 hr/110ºC		4-6 hr/110ºC	
Temperatures:						
Nozzle (ºC)	315	317	315	318	315	315
Zone 1 (ºC)	310	310	310	310	310	310
Zone 2 (ºC)	305	305	305	305	305	305
Zone 3 (ºC)	300	301	300	300	300	303
Mold (ºC)	145	145	145	145	145	145
Speeds:						
Screw RPM	120	120	120	120	120	120
% Shot - Inj Velocity (in/sec)	100-2	100-2	100-2	100-2	100-2	100-2
% Shot - Inj Velocity (in/sec)	75-2	75-2	75-2	75-2	75-2	75-2
% Shot - Inj Velocity (in/sec)	50-2	50-2	50-2	50-2	50-2	50-2
% Shot - Inj Velocity (in/sec)	25-2	25-2	25-2	25-2	25-2	25-2
% Shot - Inj Velocity (in/sec)	0-2	0-2	0-2	0-2	0-2	0-2
Pressures:						
Pack/Hold (psi)	600	600	600	600	600	600
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	20	20	20	20	20	20
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.24		0.22		0.23
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8
Cut-Off (XFER) Pressure (psi)	1000	815	1000	812	1000	775
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.3	0.3	0.3	0.3	0.3	0.3

* Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#8b		#9b		#10b	
	Set	**Actual**	**Set**	**Actual**	**Set**	**Set**
Pre Mold (Drying)	4-6 hr/110ºC		4-6 hr/110ºC		4-6 hr/110ºC	
Temperatures:						
Nozzle (ºC)	315	316	315	315	315	318
Zone 1 (ºC)	310	310	310	310	310	310
Zone 2 (ºC)	305	305	305	307	305	305
Zone 3 (ºC)	300	300	300	300	300	303
Mold (ºC)	145	145	145	145	145	145
Speeds:						
Screw RPM	120	120	120	120	120	120
% Shot - Inj Velocity (in/sec)	100-2	100-2	100-2	100-2	100-2	100-2
% Shot - Inj Velocity (in/sec)	75-2	75-2	75-2	75-2	75-2	75-2
% Shot - Inj Velocity (in/sec)	50-2	50-2	50-2	50-2	50-2	50-2
% Shot - Inj Velocity (in/sec)	25-2	25-2	25-2	25-2	25-2	25-2
% Shot - Inj Velocity (in/sec)	0-2	0-2	0-2	0-2	0-2	0-2
Pressures:						
Pack/Hold (psi)	600	600	600	600	600	600
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	20	20	20	20	20	20
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		0.22		.24		.27
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8
Cut-Off (XFER) Pressure (psi)	1000	710	1000	695	1000	817
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.3	0.3	0.3	0.3	0.3	0.3

* Position

Appendix D: INJECTION MOLDING CONDITIONS CONT.

Formulations	#11b		#12b		#13b	
	Actual	**Actual**	**Set**	**Actual**	**Set**	**Actual**
Pre Mold (Drying)	4-6 hr/110ºC		4-6 hr/110ºC		4-6 hr/110ºC	
Temperatures:						
Nozzle (ºC)	315	317	315	315	315	315
Zone 1 (ºC)	310	310	310	310	310	310
Zone 2 (ºC)	305	306	305	305	305	305
Zone 3 (ºC)	300	299	300	300	300	302
Mold (ºC)	145	145	145	145	145	145
Speeds:						
Screw RPM	120	120	120	120	120	120
% Shot - Inj Velocity (in/sec)	100-2	100-2	100-2	100-2	100-2	100-2
% Shot - Inj Velocity (in/sec)	75-2	75-2	75-2	75-2	75-2	75-2
% Shot - Inj Velocity (in/sec)	50-2	50-2	50-2	50-2	50-2	50-2
% Shot - Inj Velocity (in/sec)	25-2	25-2	25-2	25-2	25-2	25-2
% Shot - Inj Velocity (in/sec)	0-2	0-2	0-2	0-2	0-2	0-2
Pressures:						
Pack/Hold (psi)	600	600	600	600	600	600
Timers:						
Injection Hold Time (sec)	8	8	8	8	8	8
Cooling Time (sec)	20	20	20	20	20	20
Operation Settings:						
Shot Size (in)	2.7	2.7	2.7	2.7	2.7	2.7
Cushion (in)		.19		.17		.18
Cut-Off Position (in)	0.8	0.8	0.8	0.8	0.8	0.8
Cut-Off (XFER) Pressure (psi)	1000	837	1000	844	1000	687
Cut-Off Time (sec)	10	10	10	10	10	10
Cut-Off Mode	P*	P*	P*	P*	P*	P*
Decompression (in)	0.3	0.3	0.3	0.3	0.3	0.3

* Position

Appendix E: TENSILE PROPERTIES

Tensile Properties	Tensile Modulus (ksi)									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10a
Avg	204	414	538	363	353	254	546	637	455	457
n	5	5	5	5	5	5	5	5	5	5
s	3	4	5	2	2	2	4	7	3	8

Tensile Properties	Tensile Modulus (ksi)											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
Avg	688	595	578	756	671	533	662	475	-		556	690
n	5	5	5	5	5	5	5	5	-	5	5	5
s	6	5	8	3	9	7	5	4	-	5	6	4

Tensile Properties	Tensile Strain at Break (%)									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10a
Avg	238	80	2	21	136	25	2	1	2	1
n	5	5	5	5	5	5	5	5	5	5
s	19	22	0.3	3	13	8	0.1	0.1	0.4	0.49

Tensile Properties	Tensile Strain at Break (%)											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
Avg	0.86	1.4	1.2	0.82	1.1	1.8	1.3	1.2	-	1.7	1.4	1.1
n	5	5	5	5	5	5	5	5	-	5	5	5
s	0.03	0.1	0.1	0.09	0.1	0.2	0.1	0.1	-	0.2	0.2	0.1

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix F: FLEXURAL PROPERTIES

Flexural Properties	Flexural Modulus (ksi)									
	#1a	**#2a**	**#3a**	**#4a**	**#5a**	**#6a**	**#7a**	**#8a**	**#9a**	**#10a**
Avg	120	404	164	376	372	452	457	668	541	556
n	5	5	5	5	5	5	5	5	5	5
s	0.8	3.78	1.05	2.15	2.68	1.05	1.5	3.63	1.07	3.28

Flexural Properties	Flexural Modulus (ksi)											
	#1b	**#2b**	**#3b**	**#4b**	**#6b**	**#7b**	**#8b**	**#9b**	**#10b**	**#11b**	**#12b**	**#13b**
Avg	612	593	574	727	641	523	656	479	-	535	683	545
n	5	5	5	5	5	5	5	5	-	5	5	5
s	4.25	4.54	3.84	3.71	2.85	3.91	4.53	1.75	-	3.77	4.44	2.55

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix G: IMPACT PROPERTIES

Impact Properties	Notched Izod Impact (ft-lb/in)									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10 a
Avg	0.7	1.1	0.3	7.6	15.6	0.4	0.5	0.2	0.2	0.3
n	5	5	5	5	5	5	5	5	5	5
s	0.04	0.05	0.05	0.56	0.24	0	0.04	0.05	0.04	0.13

Impact Properties	Notched Izod Impact (ft-lb/in)											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10 b	#11 b	#12 b	#13 b
Avg	0.3	0.2	0.3	0.3	0.3	0.3	0.2	0.3	-	0.3	0.3	0.3
n	5	5	5	5	5	5	5	5	-	5	5	5
s	0.04	0.03	0.03	0.04	0.04	0.05	0.04	0.04	-	0.03	0.04	0.05

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix H: COMPOSITE DENSITY

	Composite Density (g/cc)									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10a
Avg	0.9463	1.1304	1.3372	1.049	1.1965	0.9888	1.1805	1.38	1.0959	1.2331
n	2	2	2	2	2	2	2	2	2	2
s	0.0002	0.0008	0.0142	0.0001	0.00001	0.0008	0.0003	0.0008	0.0013	0.0001

	Composite Density (g/cc)											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
Avg	1.295	1.29	1.285	1.358	1.296	1.269	1.312	1.262	1.278	1.285	1.344	1.282
n	2	2	2	2	2	2	2	2	2	2	2	2
s	0.0004	0.0008	0.0006	0.0001	0.0007	0.0008	0.0008	0.0001	0.0003	0.0001	0.0008	0.0003

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix I: HEAT DEFLECTION TEMPERATURE

	Heat Deflection Temperature (^{o}C)									
	#1a	#2a	#3a	#4a	#5a	#6a	#7a	#8a	#9a	#10a
Avg	66	101	113	90	135	73	178	115	94	135
n	2	2	2	2	2	2	2	2	2	2
s	1.3	0.8	0	0.2	0.2	0.1	1	0.2	0.6	0.7

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix J: ELECTRICAL PROPERTIES

	Resistance (Ohms)									
	#1a	**#2a**	**#3a**	**#4a**	**#5a**	**#6a**	**#7a**	**#8a**	**#9a**	**#10a**
Avg	1.36E+10	6.66E+10	2.95E+10	3.28E+10	5.52E+10	6.46E+4	1.49E+5	2.38E+3	5.81E+7	1.61E+8
n	5	5	5	5	5	5	5	5	5	5
s	1.38E+9	1.53E+10	1.78E+10	5E+9	8.9E+9	2.26E+4	5.85E+4	8E+2	1.03E+7	3.12E+7

	Resistance (Ohms)											
	#1b	**#2b**	**#3b**	**#4b**	**#6b**	**#7b**	**#8b**	**#9b**	**#10b**	**#11b**	**#12b**	**#13b**
Avg	8.81E+2	2.76E+2	1.11E+2	7.94E+2	1.17E+4	2.16E+3	1.19E+3	8E+2	8.25E+8	1.7E+2	4.49E+2	3.13E+2
n	5	5	5	5	5	5	5	5	5	5	5	5
s	5.65E+1	1.61E+1	4.04E+0	3.63E+1	2.89E+3	3.29E+2	4.16E+0	3.39E+2	7.98E+7	2.68E+1	2.3E+1	1.19E+1

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix K: RESISTIVE HEATING (HEATING RATES)

	HDPE-CB			Nylon 6-CB			PPS-CB		
	ID#6a			ID#7a			ID#8a		
Time	Temp	Current	Resistance	Temp	Current	Resistance	Temp	Current	Resistance
(min)	(°C)	(A)	Ohms	(°C)	(A)	Ohms	(°C)	(A)	Ohms
0	24.2	6.1E-02	1.1E+03	23.6	4.3E-04	1.6E+05	24.3	8.8E-02	7.9E+02
0.25	25.8	6.2E-02	1.1E+03	23.8	4.3E-04	1.6E+05	26	8.9E-02	7.9E+02
0.5	32.1	6.2E-02	1.1E+03	23.7	4.1E-04	1.7E+05	35.3	8.9E-02	7.8E+02
0.75	38.5	6.2E-02	1.1E+03	23.8	4.1E-04	1.7E+05	44.1	9.0E-02	7.8E+02
1	44.2	6.2E-02	1.1E+03	23.8	4.0E-04	1.7E+05	52	9.0E-02	7.8E+02
1.25	49.6	6.2E-02	1.1E+03	23.8	4.0E-04	1.7E+05	58.9	9.0E-02	7.8E+02
1.5	54.1	6.3E-02	1.1E+03	23.9	3.7E-04	1.9E+05	64.6	9.1E-02	7.7E+02
1.75	58.2	6.3E-02	1.1E+03	23.7	3.6E-04	1.9E+05	69.7	9.0E-02	7.7E+02
2	61.9	6.3E-02	1.1E+03	23.7	3.2E-04	2.2E+05	73.9	9.0E-02	7.8E+02
2.25	64.7	6.3E-02	1.1E+03	23.7	3.9E-04	1.8E+05	77.8	9.1E-02	7.7E+02
2.5	67.2	6.3E-02	1.1E+03	23.8	3.9E-04	1.8E+05	80.6	9.1E-02	7.7E+02
2.75	69.3	6.3E-02	1.1E+03	23.9	3.8E-04	1.9E+05	83.2	9.0E-02	7.7E+02
3	71.2	6.3E-02	1.1E+03	23.9	3.9E-04	1.8E+05	85.6	9.0E-02	7.8E+02
3.25	73	6.3E-02	1.1E+03	23.9	3.8E-04	1.9E+05	87.9	9.0E-02	7.8E+02
3.5	74.5	6.3E-02	1.1E+03	24	3.8E-04	1.8E+05	89.8	9.1E-02	7.7E+02
3.75	75.6	6.3E-02	1.1E+03	24.1	3.3E-04	2.1E+05	90.8	9.1E-02	7.7E+02
4	77.1	6.3E-02	1.1E+03	24.1	3.8E-04	1.8E+05	92.2	9.0E-02	7.8E+02
4.25	78.3	6.3E-02	1.1E+03	24.2	3.9E-04	1.8E+05	93.5	9.0E-02	7.8E+02
4.5	79	6.3E-02	1.1E+03	24.2	3.6E-04	2.0E+05	94.4	8.9E-02	7.8E+02
4.75	79.7	6.3E-02	1.1E+03	24.3	2.6E-04	2.7E+05	95.3	8.9E-02	7.9E+02
5	81.1	6.3E-02	1.1E+03	24.3	3.4E-04	2.0E+05	95.8	8.9E-02	7.9E+02
5.25	81.8	6.3E-02	1.1E+03	24.3	3.7E-04	1.9E+05	95.9	8.9E-02	7.9E+02
5.5	82.1	6.3E-02	1.1E+03	24.3	3.6E-04	1.9E+05	96.1	8.9E-02	7.9E+02
5.75	83.5	6.4E-02	1.1E+03	24.4	3.7E-04	1.9E+05	96.6	8.9E-02	7.9E+02
6	84.2	6.4E-02	1.1E+03	24.3	3.7E-04	1.9E+05	96.7	8.9E-02	7.9E+02
6.25	84.4	6.4E-02	1.1E+03	24.3	3.6E-04	1.9E+05	96.5	8.8E-02	7.9E+02
6.5	84.3	6.4E-02	1.1E+03	24.3	3.6E-04	1.9E+05	96.6	8.8E-02	7.9E+02
6.75	83.8	6.4E-02	1.1E+03	24.2	3.7E-04	1.9E+05	96.5	8.8E-02	7.9E+02
7	83.8	6.4E-02	1.1E+03	24.2	3.7E-04	1.9E+05	96.4	8.8E-02	7.9E+02
7.25	83.9	6.4E-02	1.1E+03	24.2	3.6E-04	1.9E+05	95.9	8.9E-02	7.9E+02
7.5	84.6	6.4E-02	1.1E+03	24.2	3.8E-04	1.9E+05	96.1	8.9E-02	7.9E+02

Electrified at 70V (AC)

7.75	84.5	6.4E-02	1.1E+03	24.2	3.7E-04	1.9E+05	97.1	9.0E-02	7.8E+02
8	84.2	6.4E-02	1.1E+03	24.1	3.7E-04	1.9E+05	96.7	9.1E-02	7.7E+02
8.25	84.4	6.4E-02	1.1E+03	24.1	3.7E-04	1.9E+05	97	9.0E-02	7.8E+02
8.5	84.7	6.3E-02	1.1E+03	24.1	3.6E-04	1.9E+05	96.7	9.0E-02	7.8E+02
8.75	85.2	6.3E-02	1.1E+03	24.1	3.6E-04	1.9E+05	97.8	9.0E-02	7.8E+02
9	86	6.3E-02	1.1E+03	24.1	3.7E-04	1.9E+05	98.1	8.9E-02	7.8E+02
9.25	86.2	6.3E-02	1.1E+03	24.1	3.6E-04	1.9E+05	98.6	9.0E-02	7.8E+02
9.5	84.9	6.3E-02	1.1E+03	24.1	3.4E-04	2.1E+05	98.3	8.9E-02	7.9E+02
9.75	84.5	6.3E-02	1.1E+03	24.1	3.6E-04	1.9E+05	98.6	8.9E-02	7.9E+02
10	84.6	6.3E-02	1.1E+03	24	3.6E-04	1.9E+05	98.5	8.9E-02	7.9E+02
10.25	83.8	6.3E-02	1.1E+03	24	3.6E-04	1.9E+05	98.4	8.9E-02	7.8E+02
10.5	83.5	6.3E-02	1.1E+03	24	3.4E-04	2.0E+05	97	9.0E-02	7.8E+02
10.75	83.4	6.3E-02	1.1E+03	24	3.6E-04	2.0E+05	97.8	9.0E-02	7.8E+02
11	83.1	6.3E-02	1.1E+03	24	2.8E-04	2.5E+05	98.1	9.1E-02	7.7E+02
11.25	83.3	6.3E-02	1.1E+03	24.1	3.4E-04	2.0E+05	99.1	9.1E-02	7.7E+02
11.5	83.9	6.4E-02	1.1E+03	24.1	3.1E-04	2.2E+05	99	9.1E-02	7.7E+02
11.75	83.6	6.4E-02	1.1E+03	24.1	3.5E-04	2.0E+05	98.7	9.1E-02	7.7E+02
12	84.2	6.4E-02	1.1E+03	24.2	3.6E-04	2.0E+05	99.1	9.0E-02	7.7E+02
12.25	84.8	6.4E-02	1.1E+03	24.2	3.4E-04	2.1E+05	99.2	9.0E-02	7.8E+02
12.5	85.5	6.4E-02	1.1E+03	24.2	3.3E-04	2.1E+05	99.6	8.9E-02	7.8E+02
12.75	86.1	6.4E-02	1.1E+03	24.2	3.5E-04	2.0E+05	98.9	8.9E-02	7.8E+02
13	86.5	6.4E-02	1.1E+03	24.3	3.4E-04	2.0E+05	98.7	9.0E-02	7.8E+02
13.25	86.3	6.4E-02	1.1E+03	24.3	3.4E-04	2.0E+05	98.2	9.0E-02	7.8E+02
13.5	86.1	6.4E-02	1.1E+03	24.2	2.9E-04	2.4E+05	98	9.0E-02	7.8E+02
13.75	85.7	6.4E-02	1.1E+03	24.2	2.7E-04	2.6E+05	97.3	9.0E-02	7.8E+02
14	85.4	6.4E-02	1.1E+03	24.3	2.8E-04	2.5E+05	96.9	9.0E-02	7.8E+02
14.25	85.7	6.4E-02	1.1E+03	24.3	2.6E-04	2.7E+05	97.5	9.0E-02	7.8E+02
14.5	86.3	6.4E-02	1.1E+03	24.2	2.6E-04	2.7E+05	98.1	9.1E-02	7.7E+02
14.75	86.7	6.3E-02	1.1E+03	24.2	2.6E-04	2.7E+05	98.8	9.1E-02	7.7E+02
15	87.3	6.3E-02	1.1E+03	24.3	2.8E-04	2.5E+05	98.9	9.1E-02	7.7E+02

Appendix K: RESISTIVE HEATING (HEATING RATES) CONT.

	Electrified at 70V (AC)					
	ABS-CB			PC-CB		
	ID#9a			ID#10a		
Time	Temp	Current	Resistance	Temp	Current	Resistance
(min)	(°C)	(A)	Ohms	(°C)	(A)	Ohms
0	22.6	1.2E-07	6.0E+08	23.2	4.4E-07	1.6E+08
0.25	22.6	1.1E-07	6.4E+08	23.3	4.4E-07	1.6E+08
0.5	22.6	1.2E-07	6.0E+08	23.3	4.4E-07	1.6E+08
0.75	22.6	1.2E-07	5.9E+08	23.4	4.4E-07	1.6E+08
1	22.6	1.2E-07	5.8E+08	23.4	4.4E-07	1.6E+08
1.25	22.6	1.2E-07	6.0E+08	23.4	4.4E-07	1.6E+08
1.5	22.7	1.1E-07	6.2E+08	23.4	4.4E-07	1.6E+08
1.75	22.7	1.1E-07	6.2E+08	23.4	4.4E-07	1.6E+08
2	22.7	1.2E-07	5.9E+08	23.5	4.4E-07	1.6E+08
2.25	22.7	1.3E-07	5.5E+08	23.5	4.3E-07	1.6E+08
2.5	22.8	1.4E-07	5.0E+08	23.5	4.4E-07	1.6E+08
2.75	22.8	1.3E-07	5.5E+08	23.5	4.4E-07	1.6E+08
3	22.8	1.2E-07	5.9E+08	23.5	4.4E-07	1.6E+08
3.25	22.8	1.1E-07	6.2E+08	23.5	4.4E-07	1.6E+08
3.5	22.8	1.2E-07	5.7E+08	23.5	4.4E-07	1.6E+08
3.75	22.8	1.3E-07	5.6E+08	23.5	4.3E-07	1.6E+08
4	22.8	1.3E-07	5.5E+08	23.5	4.3E-07	1.6E+08
4.25	22.8	1.3E-07	5.4E+08	23.5	4.3E-07	1.6E+08
4.5	22.8	1.3E-07	5.6E+08	23.5	4.3E-07	1.6E+08
4.75	22.8	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
5	22.8	1.2E-07	5.6E+08	23.5	4.3E-07	1.6E+08
5.25	22.8	1.3E-07	5.5E+08	23.5	4.3E-07	1.6E+08
5.5	22.8	1.3E-07	5.5E+08	23.5	4.3E-07	1.6E+08
5.75	22.8	1.2E-07	5.7E+08	23.5	4.4E-07	1.6E+08
6	22.8	1.2E-07	5.7E+08	23.5	4.4E-07	1.6E+08
6.25	22.8	1.3E-07	5.5E+08	23.5	4.4E-07	1.6E+08
6.5	22.8	1.3E-07	5.4E+08	23.5	4.3E-07	1.6E+08
6.75	22.8	1.3E-07	5.4E+08	23.5	4.4E-07	1.6E+08
7	22.8	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
7.25	22.8	1.3E-07	5.4E+08	23.5	4.3E-07	1.6E+08
7.5	22.8	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08

7.75	22.8	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
8	22.8	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
8.25	22.7	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
8.5	22.7	1.3E-07	5.4E+08	23.5	4.3E-07	1.6E+08
8.75	22.6	1.3E-07	5.4E+08	23.5	4.4E-07	1.6E+08
9	22.6	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
9.25	22.6	1.3E-07	5.3E+08	23.5	4.3E-07	1.6E+08
9.5	22.6	1.4E-07	5.2E+08	23.5	4.3E-07	1.6E+08
9.75	22.6	1.3E-07	5.3E+08	23.5	4.4E-07	1.6E+08
10	22.5	1.3E-07	5.3E+08	23.5	4.4E-07	1.6E+08
10.25	22.6	1.3E-07	5.3E+08	23.4	4.4E-07	1.6E+08
10.5	22.6	1.3E-07	5.3E+08	23.4	4.4E-07	1.6E+08
10.75	22.5	1.3E-07	5.4E+08	23.4	4.4E-07	1.6E+08
11	22.6	1.3E-07	5.3E+08	23.4	4.4E-07	1.6E+08
11.25	22.6	1.3E-07	5.3E+08	23.4	4.4E-07	1.6E+08
11.5	22.6	1.3E-07	5.4E+08	23.5	4.4E-07	1.6E+08
11.75	22.6	1.4E-07	5.2E+08	23.5	4.4E-07	1.6E+08
12	22.6	1.3E-07	5.2E+08	23.5	4.4E-07	1.6E+08
12.25	22.6	1.4E-07	5.1E+08	23.5	4.4E-07	1.6E+08
12.5	22.6	1.4E-07	5.2E+08	23.5	4.4E-07	1.6E+08
12.75	22.6	1.4E-07	5.1E+08	23.5	4.4E-07	1.6E+08
13	22.6	1.3E-07	5.2E+08	23.5	4.4E-07	1.6E+08
13.25	22.6	1.3E-07	5.4E+08	23.5	4.4E-07	1.6E+08
13.5	22.6	1.3E-07	5.3E+08	23.5	4.4E-07	1.6E+08
13.75	22.6	1.4E-07	4.9E+08	23.5	4.4E-07	1.6E+08
14	22.6	1.4E-07	5.0E+08	23.5	4.4E-07	1.6E+08
14.25	22.6	1.3E-07	5.2E+08	23.5	4.4E-07	1.6E+08
14.5	22.6	1.4E-07	5.1E+08	23.5	4.4E-07	1.6E+08
14.75	22.6	1.3E-07	5.3E+08	23.5	4.4E-07	1.6E+08
15	22.6	1.4E-07	5.1E+08	23.5	4.4E-07	1.6E+08

Appendix K: RESISTIVE HEATING (HEATING RATES) CONT.

Time	Electrified at 15V (AC) Temperature (°C)											
(min)	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
0	26.6	24.9	26.3	26.5	25.6	25.4	25.7	25.7	25.6	25.9	26.3	25.2
0.25	26.7	25.6	26.4	26.7	25.6	25.4	25.9	25.8	25.6	26.3	26.1	25.4
0.5	26.9	26.5	28.8	27.2	25.6	25.4	28.2	26.2	25.6	28.2	26.8	26.3
0.75	27.3	27.3	31.4	27.6	25.6	25.4	30.7	26.4	25.6	30.1	27.6	27.4
1	27.6	27.9	33.9	27.9	25.6	25.5	33.1	26.6	25.6	31.7	28.2	28.3
1.25	27.8	28.4	36.1	28.3	25.6	25.6	35.2	26.8	25.6	33.3	28.8	29.2
1.5	28.0	28.8	38.1	28.6	25.6	25.7	37.1	26.9	25.6	34.8	29.3	29.9
1.75	28.2	29.2	39.7	28.8	25.6	25.8	38.7	27.0	25.6	36.0	29.8	30.6
2	28.3	29.4	41.1	29.1	25.6	25.8	40.1	27.0	25.6	37.1	30.2	31.2
2.25	28.5	29.6	42.4	29.2	25.6	25.9	41.4	27.1	25.6	37.9	30.6	31.8
2.5	28.7	30.1	43.5	29.3	25.6	25.9	42.6	27.1	25.6	38.7	31.1	32.4
2.75	28.8	30.5	44.7	29.5	25.6	26.1	43.6	27.1	25.6	39.4	31.4	33.0
3	28.9	30.8	45.7	29.6	25.6	26.1	44.6	27.2	25.6	40.2	31.6	33.3
3.25	29.1	31.2	46.6	29.8	25.6	26.1	45.4	27.2	25.6	40.7	31.9	33.6
3.5	29.2	31.6	47.0	29.9	25.6	26.1	46.1	27.2	25.6	41.2	32.2	33.8
3.75	29.3	31.9	47.4	29.9	25.6	26.1	46.8	27.2	25.6	41.6	32.4	34.1
4	29.4	32.4	48.0	30.1	25.6	26.2	47.4	27.2	25.6	42.0	32.6	34.4
4.25	29.6	32.6	48.5	30.1	25.6	26.2	47.9	27.2	25.6	42.3	32.9	34.7
4.5	29.6	32.8	48.9	30.3	25.6	26.2	48.2	27.2	25.6	42.6	33.1	34.8
4.75	29.6	33.4	49.3	30.3	25.6	26.2	48.5	27.2	25.6	42.9	33.2	34.9
5	29.7	33.9	49.8	30.3	25.6	26.3	48.9	27.2	25.6	42.8	33.3	35.2
5.25	29.8	34.4	50.0	30.4	25.6	26.3	49.3	27.2	25.6	43.1	33.4	35.4
5.5	29.8	34.8	50.2	30.3	25.6	26.3	49.6	27.2	25.6	43.4	33.4	35.7
5.75	29.9	35.2	50.6	30.3	25.6	26.3	49.8	27.2	25.6	43.5	33.6	35.9
6	29.9	35.4	50.7	30.3	25.6	26.3	49.8	27.2	25.6	43.7	33.6	35.9
6.25	29.9	35.8	50.9	30.3	25.6	26.3	50.1	27.2	25.6	43.8	33.6	36.0
6.5	29.9	36.1	51.3	30.2	25.6	26.3	50.1	27.2	25.6	44.0	33.7	36.2
6.75	29.9	36.3	51.4	30.2	25.6	26.3	50.3	27.2	25.6	44.2	33.7	36.2
7	30.1	36.6	51.4	30.2	25.6	26.3	50.4	27.2	25.6	44.1	33.8	36.4
7.25	30.0	36.8	51.4	30.2	25.6	26.3	50.6	27.2	25.6	44.2	33.8	36.6
7.5	30.1	37.1	51.5	30.1	25.6	26.3	50.8	27.2	25.6	44.2	33.8	36.4
7.75	30.1	37.2	51.6	30.2	25.6	26.3	50.9	27.2	25.6	44.2	33.9	36.3
8	30.1	37.2	51.8	30.1	25.6	26.3	51.0	27.2	25.6	44.1	34.0	36.4
8.25	30.1	37.3	51.8	30.1	25.6	26.3	51.2	27.2	25.6	44.0	34.0	36.4

8.5	30.1	37.4	51.7	30.1	25.6	26.3	51.3	27.2	25.6	44.2	34.1	36.5
8.75	30.1	37.4	51.6	30.1	25.6	26.3	51.3	27.2	25.6	44.0	34.1	36.6
9	30.2	37.6	51.6	30.1	25.6	26.3	51.3	27.2	25.6	43.9	34.2	36.7
9.25	30.2	37.7	51.5	30.2	25.6	26.3	51.3	27.2	25.6	43.9	34.2	36.7
9.5	30.2	37.8	51.6	30.2	25.6	26.4	51.3	27.2	25.6	43.9	34.2	36.7
9.75	30.2	37.9	51.7	30.2	25.6	26.3	51.2	27.2	25.6	44.1	34.2	36.7
10	30.2	38.1	51.6	30.3	25.6	26.4	51.3	27.2	25.6	44.1	33.9	36.7
10.25	30.2	38.1	51.6	30.2	25.6	26.3	51.2	27.2	25.6	44.0	34.0	36.7
10.5	30.2	38.1	51.8	30.2	25.6	26.3	51.3	27.3	25.6	44.1	34.0	36.7
10.75	30.2	38.2	51.9	30.2	25.6	26.3	51.4	27.3	25.6	44.3	34.1	36.8
11	30.2	38.3	51.9	30.2	25.6	26.3	51.4	27.3	25.6	44.2	34.1	36.9
11.25	30.2	38.3	51.9	30.2	25.6	26.3	51.4	27.3	25.6	44.2	34.2	37.0
11.5	30.2	38.3	51.8	30.1	25.6	26.3	51.4	27.3	25.6	44.1	34.2	36.9
11.75	30.1	38.4	52.1	30.1	25.6	26.3	51.3	27.3	25.6	44.2	34.2	36.9
12	30.1	38.3	52.0	30.1	25.6	26.4	51.2	27.3	25.6	44.3	34.2	37.1
12.25	30.1	38.4	52.0	29.9	25.6	26.3	51.2	27.2	25.6	44.1	34.2	37.2
12.5	29.9	38.4	51.9	29.9	25.6	26.4	51.3	27.2	25.6	44.2	34.3	37.3
12.75	29.9	38.4	51.9	30.0	25.6	26.3	51.3	27.2	25.6	44.1	34.3	37.3
13	29.9	38.3	51.9	30.1	25.6	26.4	51.3	27.2	25.6	44.1	34.3	37.3
13.25	29.8	38.4	51.9	30.1	25.6	26.3	51.3	27.2	25.6	44.2	34.3	37.3
13.5	29.8	38.4	51.8	30.1	25.6	26.3	51.2	27.2	25.6	44.1	34.3	37.3
13.75	29.8	38.4	51.9	30.0	25.6	26.3	51.1	27.2	25.6	44.2	34.3	37.3
14	29.7	38.4	52.1	29.9	25.6	26.3	51.1	27.2	25.6	44.2	34.3	37.4
14.25	29.6	38.2	52.2	29.9	25.6	26.3	50.9	27.1	25.6	44.2	34.3	37.4
14.5	29.4	38.3	52.3	30.1	25.6	26.3	51.1	27.1	25.6	44.2	34.3	37.4
14.75	29.3	38.4	52.3	30.1	25.6	26.3	51.1	27.1	25.6	44.1	34.3	37.4

Appendix K: RESISTIVE HEATING (HEATING RATES) CONT.

Time	Electrified at 30V (AC) Temperature (°C)											
(min)	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
0	26.3	26.2	26.7	25.9	26.1	25.9	26.3	25.1	25.6	25.8	26.1	26.1
0.25	27.2	28.0	33.6	27.0	26.1	25.9	26.1	25.3	25.6	26.2	27.1	26.9
0.5	28.8	34.2	47.8	29.3	26.1	26.2	26.3	26.1	25.6	33.9	30.8	32.2
0.75	30.3	39.9	59.8	31.3	26.1	26.6	36.3	26.9	25.6	42.8	34.2	37.3
1	31.5	45.1	70.0	33.1	26.1	26.9	49.1	27.7	25.6	50.6	37.2	41.6
1.25	32.6	49.6	78.8	34.7	26.1	27.2	60.0	28.4	25.6	57.2	39.9	45.3
1.5	33.6	53.3	86.0	36.2	26.1	27.5	69.1	28.9	25.6	62.9	42.1	48.5
1.75	34.4	56.7	92.2	37.5	26.1	27.8	76.8	29.5	25.6	68.1	44.1	51.3
2	35.3	59.5	97.3	38.6	26.1	27.9	83.6	30.1	25.6	72.5	45.8	54.0
2.25	35.9	62.1	101.6	39.4	26.1	28.2	89.2	30.4	25.6	76.4	47.3	56.5
2.5	36.7	64.6	105.4	40.2	26.1	28.4	94.1	30.7	25.6	79.7	48.7	58.3
2.75	37.2	66.7	108.4	40.8	26.1	28.5	98.4	30.9	25.6	82.6	49.9	60.1
3	37.7	68.4	110.8	41.5	26.1	28.6	101.7	31.2	25.6	85.4	51.2	61.6
3.25	38.1	69.6	112.8	42.1	26.1	28.7	105.2	31.5	25.6	87.6	52.4	62.9
3.5	38.5	71.0	114.4	42.4	26.1	28.8	107.6	31.7	25.6	89.4	53.5	64.3
3.75	38.8	72.6	115.8	42.7	26.1	28.8	109.4	31.9	25.6	91.2	54.3	65.5
4	39.1	73.5	116.7	43.1	26.1	28.9	111.7	32.1	25.6	93.4	54.9	66.3
4.25	39.3	74.3	117.5	43.4	26.1	28.9	114.1	32.3	25.6	94.3	55.4	67.2
4.5	39.4	74.8	118.2	43.7	26.1	28.9	115.7	32.4	25.6	95.8	55.8	67.7
4.75	39.7	75.3	119.0	44.1	26.1	29.0	116.6	32.5	25.6	96.8	56.4	68.2
5	39.8	75.9	119.6	44.5	26.1	29.0	117.0	32.7	25.6	97.6	56.9	68.7
5.25	40.0	76.8	120.3	44.9	26.1	29.1	118.1	32.7	25.6	98.8	57.2	69.1
5.5	40.1	77.3	121.0	45.0	26.1	29.1	118.5	32.8	25.6	99.3	57.7	69.4
5.75	40.2	77.7	121.2	45.1	26.1	29.0	118.9	32.9	25.6	99.4	58.2	69.7
6	40.2	78.1	121.7	45.2	26.1	29.1	119.2	32.9	25.6	100.1	58.3	70.2
6.25	40.6	78.6	122.3	45.6	26.1	29.1	119.8	33.0	25.6	100.6	58.4	70.3
6.5	40.6	78.6	122.9	45.6	26.1	29.0	120.2	33.0	25.6	101.4	58.6	70.9
6.75	40.6	78.9	123.6	45.7	26.1	29.0	120.3	33.0	25.6	102.6	59.1	70.9
7	40.7	79.2	124.4	45.8	26.1	29.0	121.0	33.1	25.6	102.4	59.1	71.4
7.25	40.7	79.6	125.3	45.9	26.1	29.1	121.3	33.2	25.6	102.4	59.2	71.4
7.5	40.8	79.7	126.5	45.8	26.1	29.1	121.7	33.2	25.6	103.2	59.3	71.4
7.75	40.8	79.8	127.0	46.1	26.1	29.1	121.4	33.2	25.6	103.6	59.4	71.8
8	40.8	80.2	127.6	46.1	26.1	29.1	121.2	33.3	25.6	103.8	59.4	71.8
8.25	40.9	80.0	127.7	46.2	26.1	29.1	121.6	33.4	25.6	104.4	59.4	72.2

8.5	40.9	80.1	128.3	46.2	26.1	29.0	121.7	33.4	25.6	104.9	59.6	72.4
8.75	41.0	80.3	128.8	46.3	26.1	29.0	121.8	33.4	25.6	104.9	59.7	72.7
9	41.1	80.6	129.0	46.4	26.1	29.0	121.8	33.6	25.6	105.6	60.0	72.7
9.25	41.2	80.6	129.1	46.6	26.1	28.9	122.1	33.6	25.6	105.1	60.1	72.9
9.5	41.2	80.7	129.7	46.6	26.1	28.9	122.5	33.6	25.6	105.4	60.0	72.9
9.75	41.2	80.8	130.3	46.6	26.1	28.8	122.8	33.6	25.6	105.3	60.0	72.6
10	41.2	80.7	130.7	46.7	26.1	28.8	123.2	33.8	25.6	105.8	60.1	72.8
10.25	41.3	80.7	130.9	46.9	26.1	28.8	123.3	33.8	25.6	105.8	60.1	72.7
10.5	41.2	81.1	130.8	46.9	26.1	28.8	123.1	33.8	25.6	106.1	60.1	72.8
10.75	41.1	81.1	130.8	46.8	26.1	28.8	123.6	33.7	25.6	106.6	60.0	72.6
11	41.1	80.9	131.0	47.0	26.1	28.9	123.9	33.7	25.6	106.3	59.9	72.7
11.25	41.2	80.7	131.4	47.0	26.1	28.9	124.2	33.8	25.6	106.7	60.1	72.8
11.5	41.2	80.7	132.1	47.0	26.1	28.8	124.4	33.8	25.6	106.8	60.0	72.9
11.75	41.4	80.8	132.0	46.9	26.1	28.9	124.3	33.7	25.6	107.2	60.2	73.2
12	41.6	80.7	131.9	46.9	26.1	28.9	122.9	33.7	25.6	107.6	59.9	73.4
12.25	41.4	80.9	132.2	47.1	26.1	28.9	122.1	33.7	25.6	106.9	60.0	73.3
12.5	41.4	80.5	132.4	46.9	26.1	28.9	122.3	33.6	25.6	107.0	60.2	73.6
12.75	41.6	80.6	132.4	47.0	26.1	28.9	122.2	33.6	25.6	106.8	60.2	73.6
13	41.4	80.6	132.4	46.9	26.1	28.8	123.1	33.6	25.6	106.6	60.3	73.8
13.25	41.5	80.6	132.9	46.8	26.1	28.8	123.1	33.7	25.6	107.0	60.3	73.8
13.5	41.6	80.6	133.5	46.9	26.1	28.8	124.3	33.7	25.6	107.4	60.3	73.9
13.75	41.7	80.7	133.1	47.1	26.1	28.8	124.1	33.7	25.6	107.5	60.2	74.1
14	41.7	80.6	133.4	47.0	26.1	28.8	124.6	33.8	25.6	107.3	60.3	73.8
14.25	41.7	80.5	132.6	47.1	26.1	28.8	124.9	34.0	25.6	106.9	60.1	74.1
14.5	41.8	80.6	132.6	47.2	26.1	28.8	124.7	34.0	25.6	106.9	60.0	74.2
14.75	41.8	80.7	133.4	47.1	26.1	28.9	124.7	34.0	25.6	107.4	60.1	74.0

Time (min)	Electrified at 70V (AC) Temperature (°C)											
	#1b	**#2b**	**#3b**	**#4b**	**#6b**	**#7b**	**#8b**	**#9b**	**#10b**	**#11b**	**#12b**	**#13b**
0	25.6	25.2	29	27.0	25.9	26.2	28	26.2	25	25.3	26.1	25.7
0.25	30.6	25.2	28	27.2	26.2	27.0	48	29.2	25	37.1	26.9	42.6
0.5	42.4	44.8	47	36.2	26.8	30.2	104	42.6	25	87.7	26.7	71.7
0.75	52.3	78.3	105	48.8	27.7	33.4	138	55.3	25	119.8	39.5	93.0
1	60.6	102.1	150	59.3	28.4	36.2	176	67.1	25	154.8	59.5	108.5
1.25	67.5	115.8	194	68.2	29.1	38.7	197	77.9	25	188.2	76.0	116.4
1.5	73.3	128.6	226	76.1	29.8	40.9	221	87.3	25	208.2	89.1	126.7
1.75	78.5	146.2		82.6	30.3	42.6		94.6	25	220.5	99.9	140.6
2	82.8	158.6		88.3	30.9	44.3		100.7	25	229.8	108.6	150.4
2.25	87.0	169.1		92.8	31.4	45.8		105.9	25	237.8	114.2	159.7
2.5	90.0	178.3		96.3	31.8	47.1		110.2	25		118.8	167.7
2.75	92.8	185.7		99.3	32.2	48.2		113.7	25		125.8	174.0
3	94.9	190.8		102.8	32.4	49.2		115.9	25		132.1	178.8
3.25	96.8	194.2		105.4	32.7	50.0		117.6	25		136.9	183.7
3.5	98.9	196.9		107.5	32.8	50.9		118.7	25		140.8	188.6
3.75	100.5	197.9		109.0	33.1	51.6		120.6	25		144.2	191.5
4	101.5	199.6		111.2	33.3	52.1		123.2	25		147.7	194.5
4.25	102.8	198.8		112.2	33.6	52.5		125.6	25		150.9	196.3
4.5	104.2	200.9		113.7	33.6	53.1		127.4	25		154.4	198.2
4.75	105.0	201.3		114.8	33.8	53.5		129.0	25		156.0	200.7
5	106.2	202.2		114.8	33.9	53.8		130.7	25		157.9	202.3
5.25	106.8	202.3		115.9	34.0	54.2		132.1	25		159.7	203.3
5.5	107.1	204.7		116.3	34.1	54.7		133.0	25		162.6	204.5
5.75	107.6	204.9		116.5	34.1	54.9		133.9	25		164.1	204.7
6	107.9	205.5		116.9	34.2	55.6		134.7	25		164.0	205.2
6.25	108.7	206.5		117.3	34.2	55.8		136.4	25		164.7	206.4
6.5	109.0	208.4		117.8	34.1	56.1		137.1	25		165.0	206.4
6.75	109.7	209.8		118.5	34.1	56.3		137.9	25		165.7	207.5
7	110.1	211.6		118.6	34.1	56.3		137.8	25		167.1	207.0
7.25	110.7	212.3		118.8	34.1	56.8		138.0	25		168.6	207.4
7.5	110.9	214.0		119.4	34.1	56.7		138.4	25		169.2	206.8
7.75	111.1	215.9		119.5	34.1	56.9		138.9	25		168.7	205.4
8	110.8	217.4		119.9	34.1	56.7		139.3	25		168.9	205.2
8.25	111.7	219.6		119.8	33.9	56.8		139.6	25		168.9	205.8

8.5	112.1	221.4		119.7	33.8	56.8		139.2	25		169.7	205.2
8.75	111.7	223.6		120.1	33.8	56.8		138.9	25		169.0	206.8
9	112.2	226.4		120.9	33.8	56.8		138.9	25		168.9	206.8
9.25	111.7	228.3		120.9	33.9	57.1		139.3	25		169.1	207.4
9.5	112.1	228.8		120.7	33.9	57.3		141.1	25		169.1	208.2
9.75	112.4	229.6		120.8	33.8	57.6		143.4	25		168.7	207.5
10	112.2	231.9		120.5	33.7	57.6		145.2	25		169.5	207.6
10.25	112.2	234.3		121.1	33.7	57.4		146.6	25		170.4	209.2
10.5	111.7	236.0		121.3	33.7	57.3		148.4	25		170.3	208.3
10.75	111.9	237.5		121.4	33.7	57.4		150.1	25		170.3	207.5
11	111.3			121.7	33.8	57.6		151.7	25		170.8	208.3
11.25	111.7			121.0	33.8	57.6		152.9	25		170.6	209.4
11.5	111.9			121.3	33.8	57.3		154.1	25		170.3	211.3
11.75	112.4			121.1	33.7	57.5		155.3	25		170.6	211.6
12	112.5			121.4	33.7	57.8		156.9	25		170.1	212.0
12.25	112.9			121.4	33.6	57.8		158.2	25		169.7	212.3
12.5	113.2			121.5	33.6	57.7		158.1	25		169.0	213.7
12.75	113.4			121.7	33.6	57.6		158.8	25		168.4	215.1
13	113.3			121.1	33.7	57.4		159.3	25		168.9	214.7
13.25	113.5			121.6	33.6	57.6		160.1	25		169.7	215.6
13.5	113.6			121.2	33.6	57.2		160.3	25		169.2	215.7
13.75	113.6			121.4	33.7	57.2		160.9	25		168.3	215.9
14	113.9			120.4	33.7	57.3		161.9	25		167.3	216.3
14.25	114.0			120.1	33.8	57.3		162.4	25		166.7	217.2
14.5	114.1			120.4	33.7	57.4		162.4	25		167.1	218.4
14.75	113.9			120.9	33.7	57.3		163.2	25		167.4	219.0

Appendix K: RESISTIVE HEATING (HEATING RATES) CONT.

Time (min)	Electrified at 100V (AC) Temperature (°C)											
	#1b	#2b	#3b	#4b	#6b	#7b	#8b	#9b	#10b	#11b	#12b	#13b
0	28.4	31.4		26.1	33	27		32.9	25		34.1	28.8
0.25	39.6	37		29.9	31	28		48.8	25		37.9	47.8
0.5	63.8	88		55.6	33	31		85.6	25		80.7	102.2
0.75	82.7	120.9		79.2	34	40		110.5	25		112.9	136.2
1	97.1	155.1		97.2	35	47		125.6	25		139.2	177.7
1.25	108.6	185		110.7	36	53		151.4	25		162.2	208.8
1.5	115.6	206.6		119	37	58		171.4	25		177.6	230
1.75	118.7	227		129.6	38	63		187.8	25		186.2	
2	124.6			139.4	39	66		199.7	25		189.2	
2.25	132.9			147.7	40	70		206.7	25		193.4	
2.5	139.8			154.4	41	73		221	25		198.1	
2.75	145.5			159.5	41	75			25		205.7	
3	150.3			163.6	42	78			25		216.2	
3.25	153.7			167.5	42	80			25		227	
3.5	157.2			171.1	43	81			25			
3.75	160.7			173.6	43	83			25			
4	163.6			175.1	43	84			25			
4.25	165.6			177.4	43	86			25			
4.5	168.6			179.2	44	87			25			
4.75	171.1			180	44	88			25			
5	173.5			181.5	44	89			25			
5.25	174.4			182.6	44	89			25			
5.5	175.4			184	45	90			25			
5.75	175.8			183.7	45	91			25			
6	177			183	45	91			25			
6.25	178.7			184.5	45	92			25			
6.5	178.6			184.4	45	92			25			
6.75	179.3			184.5	45	93			25			
7	179.6			184	45	93			25			
7.25	179.3			183.1	45	93			25			
7.5	180			184.6	46	94			25			
7.75	181.5			184	46	94			25			
8	180.9			184	46	94			25			
8.25	181.3			184.2	46	94			25			

8.5	181.7			184.2	46	94			25			
8.75	181.9			185.5	46	95			25			
9	181.3			184.7	46	95			25			
9.25	181.7			184.8	46	95			25			
9.5	182.5			184.4	46	95			25			
9.75	181.7			185	46	95			25			
10	181.5			184.7	46	96			25			
10.25	181.4			185.6	46	97			25			
10.5	182			186.9	46	96			25			
10.75	182.7			186.4	46	97			25			
11	183.5			186.2	46	96			25			
11.25	183.5			185.9	46	96			25			
11.5	182.7			185.4	46	97			25			
11.75	182.9			185.1	46	97			25			
12	182.4			186.1	46	97			25			
12.25	181.8			184.8	46	97			25			
12.5	182.2			185.5	46	97			25			
12.75	182.4			185.7	47	97			25			
13	182.6			185.9	46	97			25			
13.25	183			185.5	47	98			25			
13.5	183.9			186	47	97			25			
13.75	184			185.2	47	97			25			
14	184.5			185.2	47	97			25			
14.25	184.3			186.4	47	98			25			
14.5	184.6			187.5	46	98			25			
14.75	184.6			186.8	46	97			25			

Appendix L: RESISTIVE HEATING (REPEATABILITY)

Time	Electrified at 30V (AC) Temperature (°C)													
	ID#1b					ID#2b					ID#3b				
(min)	#a	#b	#c	#d	#e	#a	#b	#c	#d	#e	#a	#b	#c	#d	#e
0	25	24	25	25	25	25	25	25	25	24	25	25	24	26	24
0.25	25	24	25	25	25	26	26	25	26	27	27	29	26	26	25
0.5	27	24	26	26	25	26	28	25	26	27	29	31	32	29	31
0.75	28	26	27	27	25	29	33	27	30	30	38	42	41	38	40
1	30	27	29	29	27	34	39	33	36	36	56	55	53	50	52
1.25	31	29	30	30	29	40	43	38	41	41	62	62	62	61	62
1.5	32	30	31	31	30	44	47	43	45	45	71	70	71	70	71
1.75	33	31	32	33	31	48	51	47	49	49	77	79	78	77	78
2	34	32	33	34	32	51	54	50	52	52	83	85	84	83	84
2.25	35	33	34	34	33	54	57	53	55	55	90	91	90	89	90
2.5	36	34	35	35	34	57	59	56	58	58	95	96	94	93	94
2.75	36	34	35	36	34	59	61	58	60	60	99	100	98	97	98
3	37	35	36	36	35	61	63	60	62	62	101	103	101	101	101
3.25	37	35	36	37	36	63	65	62	64	64	104	105	104	103	103
3.5	38	36	37	37	36	64	67	64	65	65	106	107	106	105	106
3.75	38	36	37	38	37	65	68	66	67	67	108	109	108	107	107
4	39	37	38	38	37	67	69	67	68	68	109	111	110	109	110
4.25	39	37	38	39	37	68	70	68	69	69	111	112	111	111	111
4.5	39	37	38	39	38	69	71	69	71	70	112	113	112	112	112
4.75	39	37	39	39	38	70	72	70	71	71	114	114	113	113	113
5	40	37	39	39	38	70	73	71	72	71	114	114	114	114	113
5.25	40	38	39	39	39	71	73	71	72	72	115	115	114	115	114
5.5	40	38	39	39	39	71	73	71	73	73	115	116	115	116	115
5.75	40	38	39	40	39	71	74	72	73	73	116	117	116	116	115
6	40	38	39	40	39	72	74	72	74	73	116	117	117	116	116
6.25	40	38	40	40	39	72	75	73	74	73	117	117	117	117	116
6.5	40	39	40	40	40	73	75	74	74	74	117	118	117	117	117
6.75	41	39	40	40	40	73	75	74	75	74	118	118	118	118	117
7	41	39	40	40	40	73	75	74	75	75	118	119	118	118	118
7.25	41	39	40	40	40	74	76	74	76	75	118	119	118	119	118
7.5	41	39	40	40	40	74	76	75	76	75	118	119	119	120	118
7.75	41	39	40	40	40	74	76	75	75	75	119	119	119	120	118
8	41	39	40	40	40	75	77	75	76	75	119	119	119	120	119

8.25	41	39	40	40	40	75	76	75	76	75	119	120	119	120	119
8.5	41	39	40	40	40	75	76	76	76	75	119	120	119	120	119
8.75	41	39	40	40	40	75	76	75	76	75	119	119	119	121	119
9	41	39	40	40	41	75	76	75	76	75	120	120	119	120	119
9.25	41	39	41	40	41	75	76	75	77	75	120	120	119	121	119
9.5	42	39	41	40	41	76	77	75	77	75	120	120	119	121	119
9.75	42	39	41	40	41	75	76	75	77	75	120	120	119	121	120
10	41	39	41	40	41	76	76	76	77	76	121	120	120	121	120
10.25	41	39	41	40	41	76	76	75	77	76	121	121	120	121	120
10.5	41	39	41	40	41	76	76	75	77	76	121	121	120	122	120
10.75	42	39	41	40	41	76	76	75	77	76	121	121	120	122	121
11	41	39	41	40	41	76	76	75	77	76	121	122	120	121	121
11.25	42	39	41	40	41	76	76	75	77	76	121	121	120	122	121
11.5	41	39	41	40	41	76	76	74	77	76	122	121	120	122	121
11.75	41	39	41	40	41	76	76	75	77	76	122	121	120	122	121
12	41	39	41	40	41	76	76	75	77	76	122	121	120	123	121
12.25	41	39	41	40	41	76	76	75	77	76	123	121	120	122	121
12.5	41	39	41	40	41	76	76	75	77	76	122	122	121	123	121
12.75	42	39	41	40	41	76	76	76	77	76	122	122	121	122	122
13	42	39	41	40	41	76	77	76	77	76	122	122	120	123	122
13.25	42	39	41	40	41	76	77	76	78	76	122	122	121	123	121
13.5	42	39	41	40	41	76	77	76	77	76	122	121	120	122	121
13.75	42	39	41	40	41	76	77	76	78	76	122	122	120	123	121
14	42	39	41	40	41	76	77	76	78	76	123	122	120	123	121
14.25	42	39	41	40	41	76	77	76	78	76	123	122	120	123	122
14.5	42	39	41	40	41	76	77	76	78	76	123	122	120	123	122
14.75	42	39	41	40	41	77	77	76	77	76	123	122	120	123	122

Appendix M: RESISTIVE HEATING (CYCLE STUDY)

	Composite Resistance											
	ID#1b						ID#13b					
Cycles	0	1	2	3	4	5	0	1	2	3	4	5
Avg	882	856	854	854	852	849	323	298	297	297	296	295
s	3.8	4.2	4.6	5.2	3.8	4.8	2.2	3.2	2.9	2.8	2.8	3.1
n	2	2	2	2	2	2	2	2	2	2	2	2

n – Represents number of Specimen Tested
s – Represents Standard Deviation

Appendix N: DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Sample ID#1a (HDPE)

Sample: HDPE R21-20 XLW
Sample Size: 10.9350 mg
Method: 25 to 180°C at 10°C/min
Comment: PDI# 22150; clear,white pellets

DSC

File Path: C:\TA\Data\DSC\0716701.001
Operator: EMG; N2 50ml/min
Date of Experiment: 28-Sep-07
Instrument Type: 2920 DSC   V2.6A

0716701.001 HDPE R21-20 XLW
0716701.001-c HDPE R21-20 XLW
0716701.001-1 HDPE R21-20 XLW

Polymer Diagnostics Inc.

114.7°C

Cooling scan

117.3°C
170.82J/g

Second heating scan

122.4°C
189.63J/g

130.5°C

First heating scan

121.7°C
172.90J/g

131.5°C

Exo Up

Heat Flow (W/g)

Temperature (°C)

Universal V3.7A TA Instruments
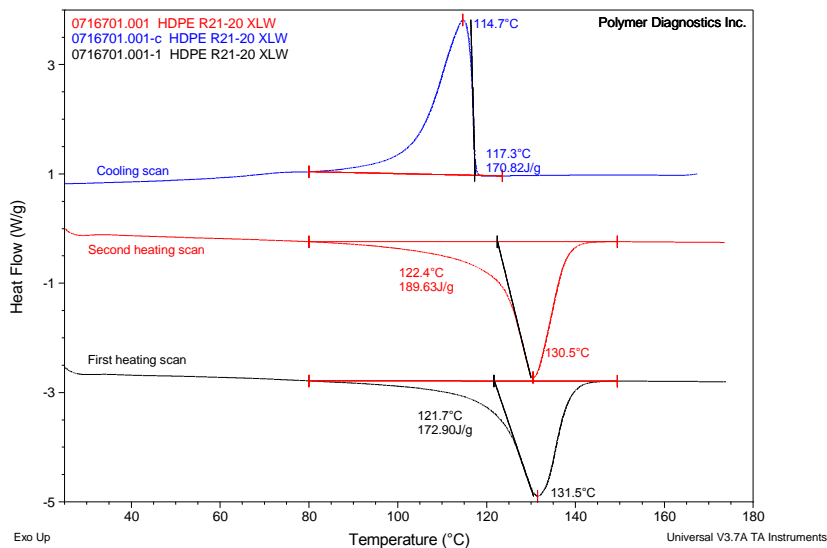
Sample ID#6a (HDPE-CB)

Sample: HDPE 9% XE2B
Sample Size: 8.84900 mg
Method: 25 to 180°C at 10°C/min
Comment: PDI# 22150; black bars

DSC

File Path: C:\TA\Data\DSC\0716702.001
Operator: EMG; N2 50ml/min
Date of Experiment: 28-Sep-07
Instrument Type: 2920 DSC   V2.6A

0716702.001 HDPE 9% XE2B
0716702.001-1 HDPE 9% XE2B
0716702.001-c HDPE 9% XE2B

Polymer Diagnostics Inc.

113.9°C

Cooling scan

117.3°C
151.17J/g

Second heating scan

121.9°C
162.32J/g

131.8°C

First heating scan

123.0°C
146.36J/g

129.5°C

Exo Up

Heat Flow (W/g)

Temperature (°C)

Universal V3.7A TA Instruments

Sample ID#2a (Nylon 6)

DSC

Sample ID#7a (Nylon 6-CB)

DSC

Sample ID#3a (PPS)

Sample: PPS
Sample Size: 8.60900 mg
Method: 25 to 320°C at 10°C/min
Comment: PDI# 22150, black pellets

DSC

File Path: C:\TA\Data\DSC\0716705.001
Operator: EMG; N2 50ml/min
Date of Experiment: 2-Oct-07
Instrument Type: 2920 DSC   V2.6A
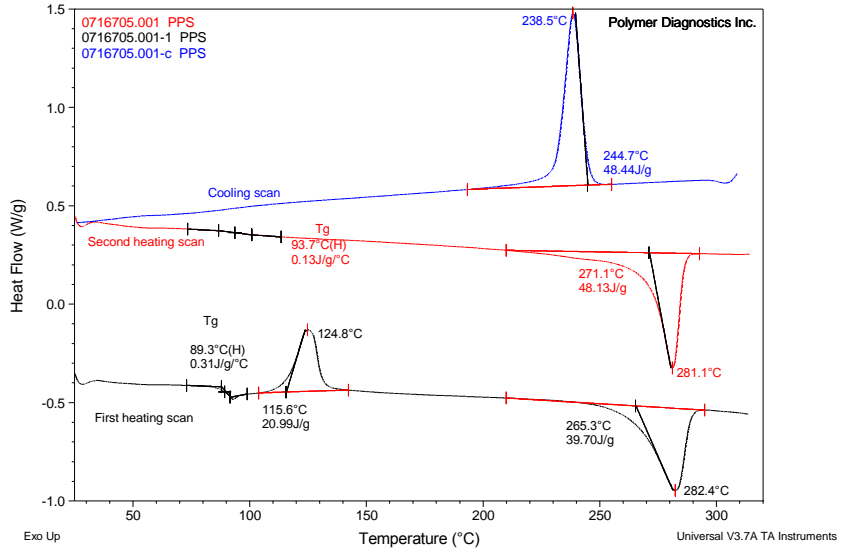
Sample ID#8a (PPS-CB)

Sample: PPS, 9% XE2B
Sample Size: 9.31200 mg
Method: 25 to 320°C at 10°C/min
Comment: PDI# 22150, black pellets

DSC

File Path: C:\TA\Data\DSC\0716706.001
Operator: EMG; N2 50ml/min
Date of Experiment: 3-Oct-07
Instrument Type: 2920 DSC   V2.6A

120
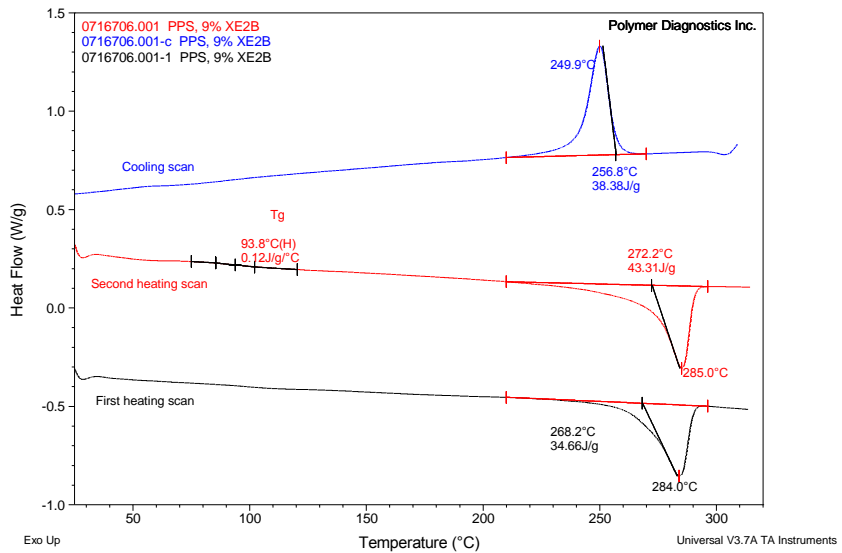
Appendix O: Calculation of Percent Crystallinity

Table O: Theoretical and Measured (DSC) Heat of Fusion of polymers and composites

	$^1\Delta H_f$ (j/g)	$^2\Delta H_{f\,100\%}$ (j/g)
HDPE	189.6	293
HDPE-CB	162.3	*266.6
Nylon 6	71.5	230
Nylon 6-CB	81.2	*209.3
PPS	48.1	80
PPS-CB	43.3	*72.8

1. Heat of fusion measured from second heat scan of DSC in Appendix N
2. Theoretical heat of fusion obtained from literature (45)
* Calculated from using theoretical value multiplied by 91% (actual polymer content)

Using equation 6.1, percent crystallinity can be calculated for all polymers and composites listed in Table O.

Sample Calculation of percent crystallinity for HDPE:

$$\alpha = \frac{189.6}{293} * 100\%$$

$$\alpha = 64.7\%$$

Appendix P: Thermal gravimetric Analysis (TGA)

Sample ID#1a (HDPE)

Sample: HDPE
Size: 7.4440 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150; white pellets

TGA

File: O:...\Thermal Analysis\TGA\0717703.01
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 24-Oct-07 12:05
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

299.7°C

99.7%
(7.4mg)

Gas Switch 600°C
Nitrogen to Air

Residue:
0.2%
(0.0mg)

0.2%
(0.0mg)

Universal V3.7A TA Instruments

Sample ID#6a (HDPE-CB)

Sample: HDPE 9% XE2B
Size: 7.1680 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150: black rubber piece

TGA

File: O:...\Thermal Analysis\TGA\0716702.03
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 15-Oct-07 11:47
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

91.9%
(6.6mg)

Gas Switch 600°C
Nitrogen to Air

Residue:
0.5%
(0.0mg)

7.3%
(0.5mg)

Universal V3.7A TA Instruments

122

Sample ID#2a (Nylon 6)

Sample: BASF PA6 B27 E01
Size: 5.7810 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150; white pellets

TGA

File: O:...\Thermal Analysis\TGA\0717702.01
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 24-Oct-07 09:43
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

1.2%
(0.1mg)

300.8°C

98.6%
(5.7mg)

Gas Switch 600°C
Nitrogen to Air

Residue:
0.4%
(0.0mg)

0.3%
(0.0mg)

Universal V3.7A TA Instruments

Sample ID#7a (Nylon 6-CB)

Sample: Nylon 6, 9% XE2B
Size: 13.6040 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150: black pellets

TGA

File: O:...\Thermal Analysis\TGA\0716704.03
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 15-Oct-07 14:40
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

299.7°C

1.2%
(0.2mg)

88.0%
(12.0mg)

Gas Switch 600°C
Nitrogen to Air

Residue:
0.4%
(0.1mg)

10.5%
(1.4mg)

Universal V3.7A TA Instruments

123

Sample ID#4a (ABS)

Sample: Lustran ABS 433
Size: 8.0170 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150; cream pellets

TGA

File: O:...\Thermal Analysis\TGA\0717701.01
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 23-Oct-07 12:39
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

200.2°C

98.8%
(7.9mg)

Gas Switch 600°C
Nitrogen to Air

Residue:
0.3%
(0.0mg)

0.7%
(0.1mg)

Universal V3.7A TA Instruments

Sample ID#9a (ABS-CB)

Sample: ABS 9% XE2B
Size: 12.5200 mg
Method: 30-600,gas-700 @10°C/min
Comment: PDI#22150: black pellets

TGA

File: O:...\Thermal Analysis\TGA\0716707.03
Operator: EMG; N2/Air(90ml/m;10bal)
Run Date: 16-Oct-07 10:17
Instrument: 2950 TGA HR V5.2B

Polymer Diagnostics Inc.

200.2°C

86.8%
(10.9mg)

Gas Switch 600°C
Nitrogen to Air

12.4%
(1.5mg)

Residue:
0.6%
(0.1mg)

Universal V3.7A TA Instruments

124

Appendix Q: DOE

C:\Users\Maz\Desktop\oct 15\DC HDS Fixed Finally.dx7 - Design-Expert 7.1.3

File Edit View Display Options Design Tools Help

Left panel tree:
- Notes for DC HDS Fixed Finally
  - Design (Actual x Actual)
    - Summary
    - Graph Columns
    - Evaluation
    - Constraints
  - Analysis
    - Plateau Temperature (Analyzed)
    - Resistance (Analyzed)
    - Tensile Mod (Analyzed)
    - Flex Mod (Analyzed)
  - Optimization
    - Numerical
    - Graphical
    - Point Prediction

Std	Run	Component 1 A:PPS %	Component 2 B:Carbon Black %	Component 3 C:Graphite %	Component 4 D:HDPE %	Factor 5 E:AC Volts Volts	Response 1 Plateau Tempe (C)	Response 2 Resistance ohms	Response 3 Tensile Mod KSI	Response 4 Flex Mod KSI
4	1	66.000	4.200	9.800	20.000	15.00	28.8889	937.500	688	612
29	2	66.000	7.000	7.000	20.000	15.00	38.3333	283.019	595	593
26	3	66.000	9.800	4.200	20.000	15.00	52.2222	114.504	578	574
31	4	74.250	4.725	11.025	10.000	15.00	30	833.333	756	727
8	5	66.000	4.000	10.000	20.000	15.00		15000.000	671	641
9	6	69.000	5.000	5.000	21.000	15.00		2500.000	533	523
32	7	62.930	9.000	9.000	19.070	15.00	51.1111	123.967	662	656
27	8	71.000	9.000	0.000	20.000	15.00	27.2222	1071.429	475	479
17	9	66.000	9.800	4.200	20.000	15.00	44.4444	159.574	556	535
10	10	76.000	7.000	7.000	10.000	15.00	34.4444	468.750	690	683
23	11	67.000	9.000	4.000	20.000	15.00	37.2222	326.087	577	545
25	12	66.000	4.200	9.800	20.000	30.00	42.2222	882.353	688	612
28	13	66.000	7.000	7.000	20.000	30.00	80.5556	258.621	595	593
19	14	66.000	9.800	4.200	20.000	30.00	133.333	107.143	578	574
20	15	74.250	4.725	11.025	10.000	30.00	47.2222	789.474	756	727
22	16	66.000	4.000	10.000	20.000	30.00		10000.000	671	641
21	17	69.000	5.000	5.000	21.000	30.00	28.8889	2142.857	533	523
2	18	62.930	9.000	9.000	19.070	30.00	124.444	117.647	662	656
13	19	71.000	9.000	0.000	20.000	30.00	33.8889	909.091	475	479
24	20	66.000	9.800	4.200	20.000	30.00	107.222	148.515	556	535
30	21	76.000	7.000	7.000	10.000	30.00	60	454.545	690	683
18	22	67.000	9.000	4.000	20.000	30.00	73.8889	303.030	577	545
3	23	66.000	4.200	9.800	20.000	70.00	113.889	823.529	688	612
5	24	66.000	7.000	7.000	20.000	70.00			595	593
14	25	66.000	9.800	4.200	20.000	70.00			578	574
16	26	74.250	4.725	11.025	10.000	70.00	121.111	760.870	756	727
12	27	66.000	4.000	10.000	20.000	70.00	34.4444	10000.000	671	641
6	28	69.000	5.000	5.000	21.000	70.00	57.7778	1842.105	533	523
1	29	62.930	9.000	9.000	19.070	70.00			662	656
7	30	71.000	9.000	0.000	20.000	70.00	163.333	429.448	475	479
11	31	66.000	9.800	4.200	20.000	70.00		200.000	556	535
33	32	76.000	7.000	7.000	10.000	70.00	169.444	424.242	690	683
15	33	67.000	9.000	4.000	20.000	70.00	221.667	309.735	577	545

Appendix R: ANOVA

The corresponding terms for components in mixture and process factors are shown in Table R.

Table R: Mixture and Process Factors

Mixture Components	
A	PPS
B	Carbon Black
C	Graphite
D	HDPE
Process Factors	
E	Voltage

ANOVA (Plateau Temperature)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	64657.79	7	9236.83	34.41	< 0.0001	**significant**
Linear Mixture	*6525.72*	*3*	*2175.24*	*8.10*	*0.0013*	
AE	*9416.72*	*1*	*9416.72*	*35.08*	*< 0.0001*	
BE	*5795.22*	*1*	*5795.22*	*21.59*	*0.0002*	
CE	*1881.78*	*1*	*1881.78*	*7.01*	*0.0164*	
DE	*13503.22*	*1*	*13503.22*	*50.30*	*< 0.0001*	
Residual	4832.48	18	268.47			
Lack of Fit	*4461.34*	*16*	*278.83*	*1.50*	*0.4723*	**not significant**
Pure Error	*371.14*	*2*	*185.57*			
Cor Total	69490.28	25				

Std. Dev.	16.39	R-Squared	0.9305
Mean	75.28	Adj R-Squared	0.9034
C.V. %	21.77	Pred R-Squared	0.8427
PRESS	10927.51	Adeq Precision	22.406

Component	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
A-PPS	223.09	1	19.07	183.02	263.15	7.53
B-Carbon Black	-270.89	1	36.10	-346.72	-195.05	4.51
C-Graphite	48.27	1	16.62	13.36	83.18	2.80
D-HDPE	208.71	1	17.02	172.95	244.48	2.55
AE	143.37	1	24.21	92.51	194.23	8.17
BE	-211.40	1	45.50	-306.99	-115.81	5.29
CE	53.48	1	20.20	11.04	95.91	2.86
DE	148.25	1	20.90	104.33	192.16	2.82

ANOVA (Resistance)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	0.017	3	5.689E-003	60.62	< 0.0001	**significant**
Linear Mixture	*0.017*	*3*	*5.689E-003*	*60.62*	*< 0.0001*	
Residual	2.440E-003	26	9.385E-005			
Lack of Fit	*2.232E-003*	*24*	*9.300E-005*	*0.89*	*0.6567*	*not significant*
Pure Error	*2.080E-004*	*2*	*1.040E-004*			
Cor Total	0.020	29				

Std. Dev.	9.687E-003	R-Squared	0.8749	
Mean	0.048	Adj R-Squared	0.8605	
C.V. %	20.05	Pred R-Squared	0.8393	
PRESS	3.135E-003	Adeq Precision	22.710	

Component	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
A-PPS	0.12	1	7.250E-003	0.10	0.13	3.73
B-Carbon Black	-0.15	1	0.015	-0.18	-0.12	2.86
C-Graphite	0.018	1	8.750E-003	4.801E-004	0.036	2.44
D-HDPE	0.11	1	8.409E-003	0.089	0.12	1.78

ANOVA (Tensile Modulus)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	2.005E+005	3	66848.74	277.82	< 0.0001	**significant**
Linear Mixture	*2.005E+005*	*3*	*66848.74*	*277.82*	*< 0.0001*	
Residual	6977.97	29	240.62			
Lack of Fit	*6251.97*	*26*	*240.46*	*0.99*	*0.5943*	*not significant*
Pure Error	*726.00*	*3*	*242.00*			
Cor Total	2.075E+005	32				

Std. Dev.	15.51	R-Squared	0.9664	
Mean	616.45	Adj R-Squared	0.9629	
C.V. %	2.52	Pred R-Squared	0.9583	
PRESS	8650.81	Adeq Precision	53.761	

Component	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
A-PPS	706.60	1	10.33	685.49	727.72	3.47
B-Carbon Black	628.59	1	22.47	582.63	674.55	2.60
C-Graphite	321.21	1	13.46	293.69	348.73	2.41
D-HDPE	832.59	1	13.12	805.76	859.42	1.71

ANOVA (Flexural Modulus)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	1.651E+005	3	55035.35	381.21	< 0.0001	**significant**
Linear Mixture	*1.651E+005*	*3*	*55035.35*	*381.21*	*< 0.0001*	
Residual	4186.68	29	144.37			
Lack of Fit	*1905.18*	*26*	*73.28*	*0.096*	*0.9999*	*not significant*
Pure Error	*2281.50*	*3*	*760.50*			
Cor Total	1.693E+005	32				

Std. Dev.	12.02	R-Squared	0.9753	
Mean	597.09	Adj R-Squared	0.9727	
C.V. %	2.01	Pred R-Squared	0.9692	
PRESS	5210.41	Adeq Precision	62.667	

Component	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
A-PPS	679.64	1	8.00	663.28	696.00	3.47
B-Carbon Black	544.17	1	17.41	508.57	579.77	2.60
C-Graphite	341.39	1	10.42	320.07	362.71	2.41
D-HDPE	831.91	1	10.16	811.13	852.70	1.71