DEFORMATION AND DURABILITY STUDIES OF CONDUIT POLYMERIC MATERIALS

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DEFORMATION AND DURABILITY STUDIES OF CONDUIT POLYMERIC MATERIALS

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ABSTRACT

Polymeric materials are widely used in cable insulation, but the mechanical properties of insulation polymers are still not well studied and summarized, especially the fatigue behavior. This research aims to study the fatigue behavior of conduit polymer materials, viz nylon 6, polypropylene (PP) and calcium carbonate filled black colored polypropylene (PP (blk)) under different strain ranges and temperature conditions. Besides room temperature (25 °C) tests, low temperature (-40 °C) tests and high temperature (125 °C) for nylon 6, 65 °C for PP and PP (blk)) tests are also performed. All of the three materials are below their glass transition temperature in low temperature tests, so they are more brittle and fatigue life time is shorten obviously. High temperature gives polymers more viscosity, and fatigue life significantly extends. PP (blk) possesses the best fatigue behavior, followed be PP and nylon 6. Wei-Wong unified strain fatigue model was used to analysis fatigue data and predict fatigue life.

Thermal and mechanical analysis is conducted before fatigue tests. Thermal character, such as molecular degradation, glass transition and melting point, can be determined. Mechanical properties, such as ultimate tensile strength, elastic modulus and Poisson’s ratio were obtained from quasi static tensile tests. Scanning Electron Microscope (SEM) was employed to examine the failure structure.
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CHAPTER I

INTRODUCTION

1.1 Cable Insulation Materials

An important component to electrical devices is the insulation material. More than half a century ago, fibrous and solid insulants were widely used, oil-paper cable was commonly used, and natural fabrics were also used to insulate cable, such as cotton and jute fabrics, asbestos etc.

Polymers are increasing using as insulation materials in the last fifty years. Polymers always have long degradation life; good insulate behavior, and are flexible and cost effective. Polypropylene (PP) is one of the principle polymers, and is widely used in cable insulation. Nylon was firstly produced by DuPont on 1935. Nowadays nylon is one of the most commonly used polymers. Both of them are used as cable insulation materials in many areas, such as automotives, aircrafts and personal computers.

1.2 Test Methods of Insulation Polymers

From the view of industrial design, good design of cable insulation refers to long life time under loading [1]. Following the external loads, insulation will be ineffective
if it fails by mechanical deformation. So mechanical properties need to be well-understand, before insulation design. The general mechanical properties of polymers can be summarized as tensile properties, flexure properties, impact properties, compression properties, creep and stress-relaxation properties, hardness, tear resistance and abrasion resistance etc. In this study, tensile tests are performed to determine some of mechanical properties. In order to predict polymer insulation’s life time, we need to run cyclic load to polymers, and obtain raw data from laboratory. Then substitute these data to existing fatigue models to get fatigue life prediction results.

1.3 Tensile Tests

Tensile tests are one of the fundamental mechanical tests in material testing, and can provide the stress-strain relationship of polymers. During tensile tests, we need to assume there is no imperfection in specimen, and a homogeneous and isotropic materials state is assumed with respect to the specimen [2].

Engineering thermoplastics always can sustain large irreversible elongation after necking during tensile testing, as schematic in Figure 1.1. This phenomenon has been observed in kinds of thermoplastics, such polypropylene (PP) [3-5], polyethylene (PE) [6-8], polycarbonate (PC) [5,9,9].
True stress-strain behavior is one of the fundamental mechanical properties, which is a little different from engineering stress-strain behavior. The important information of yielding behavior, true rate of strain hardening, stress-induced transformation of crystalline phases can be obtained from true stress-strain curves [5,11,12]. The difference is the definitions of strain. Engineering strain is nominal strain, and true strain is the actual elongation. The equation below shows the relationship between true strain ($\varepsilon_t$) and engineering strain ($\varepsilon_{eng}$).

$$\varepsilon_t = \ln \left( 1 + \varepsilon_{eng} \right)$$

Elastic modulus is a material character, and is defined as the slope of stress-strain curve in elastic region. Specifically tensile elasticity can be named as Young’s modulus, which is defined as the ration of tensile stress to strain. When a specimen
was stretched, the ration of transverse strain to axial strain is named Poisson ratio, which is also an important character of structural materials. Elastic modulus and Poisson ration both can be calculated from stress-strain curve.

1.4 Fatigue Tests

Fatigue failure is a critical issue for engineering structural components, and a long fatigue life is preferred in engineering design. Almost 80% of plastic parts failure cases are fatigue failure [13], so that’s so important to understand fatigue properties and having better fatigue life design.

Assuming one may wish to predict the total fatigue life of some component, certain laboratory test results are needed. In fact, in order to simulate as well as possible actual component service conditions, this requires that specimen geometry need to be chosen; certain control variables, stress control or strain control, need to be established; various test conditions need to be chosen, such as test temperature, cyclic frequency, loading rate etc.

Key stress variables that contribute in different ways to the fatigue process under stress-controlled test conditions are shown in Figure 1.2.
In Figure 1.2, the definition is shown below:

\[ \sigma_{\text{max}}, \sigma_{\text{min}} = \text{maximum and minimum stresses}; \]
\[ \Delta \sigma = \text{stress range} = \sigma_{\text{max}} - \sigma_{\text{min}} \]
\[ \sigma_a = \text{stress amplitude} = \frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{2} \]
\[ \sigma_m = \text{mean stress} = \frac{\sigma_{\text{max}} + \sigma_{\text{min}}}{2} \]
\[ R = \text{stress ratio} = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} \]

In related fashion, for the case of strain controlled testing based on the variation of strain, the variables need to be changed to maximum and minimum strain, strain range, strain amplitude, mean strain and strain ratio.
1.5 Scope and Objective of Thesis

This study aims at static and dynamic mechanical properties of three polymer insulations, PP, PP (blk) and nylon 6. Quasi-static tensile tests were performed at room temperature to obtain true stress-strain relationship. Strain controlled fatigue tests are performed under various temperatures, in order to determine temperature effect on fatigue life. By using Wei-Wong unified strain model, fatigue life prediction is realized.
2.1 Introduction to Insulation Polymers

Cable insulation is required in various fields, such as auto and aircraft industry. Some commonly used insulation polymers will be discussed in this section.

2.1.1 Traditional Fibrous and Solid Insulants

More than fifty years ago, fibrous and solid insulants, such as cellulose paper, pressboard, vulcanised fiber, mica and bitumen etc, were the most wildly used in insulation [13]. The fibrous materials were used for their flexibility and simplicity of handling and shaping when the working temperature is not too high. And they also can provide moderate strength combined with lightness and low cost in resin bonded boards, tubes and bushings etc. Mica flake and mica paper products, as solid insulants, can be used for the insulation of high-voltage machines. But the temperatures can not be high. So water cooling or gaseous cooling is needed to avoid discharge.

2.1.2 Linear Synthetic Polymers

Polymers, in which each molecule consists of a chemical chain with few branches, always have a number of features in common. Generally speaking, linear
polymers have improved tensile strength, tear strength, low temperature toughness, 
softening temperature and impact strength. But fabrication, such as extrusion, 
molding etc, is made more difficult because of the high melt viscosity. Following is a 
introduction to several commonly used liner synthetic polymers.

Polyvinyl chloride (PVC) [13] is widely used as insulation for low and medium 
voltage unsheathed cables, and for jacketing cables of many kinds, because of its 
good adhesion and oil resistance. PVC is usually working under 90 °C. PVC has as 
good electric strength as most polymers, so the maximum temperature of use is often
limited by mechanical considerations, not electrical. The chemical configuration of 
PVC is shown below:

![Figure 2.1 Repeat unit of polyvinyl chloride](image)

Polyethylene (PE) [13] is classified into lots of different categories based on 
density and branching. Mechanical properties vary due to different branching, crystal 
structure and molecular weight. High-density polyethylene (HDPE), which is a cheap
thermoplastic made from petroleum. HDPE has low degree of branching, which gives it stronger intermolecular forces and tensile strength than lower-density polyethylene.

The chemical configuration of PE is shown below:

![Chemical configuration of PE](image)

Figure 2.2 Repeat unit of polyethylene

Polycarbonate (PC) [13] is a particular group of thermoplastic polymers. They are easily worked, moulded, and thermoformed. PC has pretty high impact resistance, temperature resistance, and is usually positioned between commodity plastics and engineering plastics. PC is quite commonly used in house wares as well as in industry. The applications of PC are various, such as drinking bottles, DVD discs, lighting lenses, computer components. The chemical configuration of PC is shown below:
2.1.3 Cross-Linked Synthetic Polymers

Linear polymers are formed from monomer molecules each of which reacts with two other molecules. But cross-linking requires a proportion of monomers, which are polyfunctional, can react with three or more other groups. In fact, the structural chemistry of cross-linked polymers can not be precisely described. And the technology of cross-linked polymers is more empirical than that of linear polymers.

 Linear polymers are usually thermoplastic, but cross-linked polymers, on the contrary, are taken to be thermosetting, so they decompose without melting at high temperatures, such as epoxy, which is a classical thermosetting resin.

The following sections give an introduction to the polymers which are studied in this research.

2.2 Nylon 6

Nylon is a generic designation for a family of synthetic polymers known generically as polyamides and first produced on February 28, 1935 by Wallace
Carothers at DuPont. Nylon 6 is one of the most commonly used polyamides. Nylon 6 is prepared from caprolactam, the reaction can be found in Figure 2.4. Unlike most other nylons, nylon 6 is not a condensation polymer, but instead is formed by ring-opening polymerization, the chemical configuration of nylon 6 is shown. This makes it a special case in the comparison between condensation and addition polymers. Nylon 6 possesses high tensile strength, high resistance to abrasion and chemicals such as acids, alkalis etc, and good oxygen index and flammability characteristics.

![Figure 2.4 Nylon 6 formation reaction](image)

2.3 Polypropylene (PP)

PP is isotactic and crystallisable, and the number-average molecular weight is usually in the range 80,000 to 500,000. The chemical configuration of PP is shown in Figure 2.5. PP is linear synthetic polymer, and has similar properties with HDPE, and PP has higher tensile strength, modulus and toughness. The electrical properties of PP are generally similar to those of PE.
Calcium carbonate (CaCO$_3$) is an important filler, in polymer matrix, with a particle size of about one micrometer [15]. In some case, CaCO$_3$ may be treated to improve interaction with thermoplastic. Different contents of CaCO$_3$ have different effect on polymers [16]. Low density of CaCO$_3$ decreases the viscosity of polymers, and when a high content of this filler is added, the CaCO$_3$ particles tend to agglomerate and thus, the polymers’ viscosity is enhanced. The PP (blk) tested has 30 wt% CaCO$_3$ inclusions. But whether the CaCO$_3$ particles enhance the PP (blk)’s viscosity, or other condition affects the viscosity, we are not clear based on current information.

2.4 Fatigue of Polymers

Fatigue failure always occurs after a certain number of similar load fluctuations have been experienced. Failure does not occur when the component is loaded initially
Fatigue is a form of failure that occurs in structures subjected to dynamic and fluctuating stresses.

Polymers are being utilized in the manufacture of loading-bearing structural components. In order to provide assurances that these parts will withstand the loading through their all service life, fatigue tests are demanded to give detailed and accurate life time prediction. The influence of important external or experimental variables, such as alternating stress amplitude, stress intensity factor range, frequency, mean stress, temperature, surface condition and environment and of significant material variables, such as polymer structure, viscoelastic characteristics, molecular weight, molecular weight distribution, rubber inclusions, fillers or diluents, etc should be carefully studied.

Though polymer fatigue is not a new topic, fatigue in polymers is not as well understood as in metals. Mechanism of polymer fatigue is similar as metal, but polymers are more sensitive to frequency of alternating loading than metals, because of their significant viscoelasticity and poor heat conductivity. Polymer fatigue failure always occurs either by thermal softening and melting due to hysteretic heating [17]. Since preexistent defects are sometimes encountered, it is often desirable to evaluate the fatigue resistance of a structure in terms of either crack initiation or subsequent crack propagation. So Fatigue crack propagation will be briefly discussed in this section.
2.5 Stress Controlled Fatigue Testing

Researchers have found the magnitude of the alternating stress has a strong bearing on the fatigue life of test specimen. For this reason, many fatigue data in plastics and metals compares cyclic life as a function of stress amplitude.

Stress load can be introduced either by rotational bending, reciprocal bending, reciprocal torsion, or by pulsating axial loads. Both the loading method and specimen shape has been adopted by ASTM standard. The specimen failure may take place by either of two modes. In one mode, the specimen may fail by the nucleation and growth of a crack across the gauge section. Then the fatigue life can be simply defined as the number of loading cycles before specimen separation. In the other mode, some specimens may become overhead before specimen separation. A point exists whereby the specimen is no longer capable of supporting the load introduced by testing machine within the deflection limits, so ASTM defined thermal fatigue failure life as the point when an apparent modulus decay 70% of the original modulus of the specimen [18].

The hysteretic energy generates during each loading cycle. Since this energy is largely dissipated in the form of heat, the test specimen will experience a temperature rise for every loading cycle.

A lot of attempts have been made to determine the heat generation and temperature increase quantificationally. Schmidt and Marlies found the rate of heat generation $H_F$ for stress controlled fatigue test can be given by [19]:

$$H_F \propto DF^2 / E_d$$
where \( D \) is the damping capacity of the material, \( F \) is force, and \( E_d \) is the dynamic modulus. When heating takes place during a test, the dynamic modulus is seen to decrease, and this decrease results in a greater degree of heat generation under constant load.

The energy dissipation rate also can be described by[20]:

\[
\dot{\mathcal{E}} = \pi f \Im \left( f, T \right) \sigma^2
\]

where \( f \) is the frequency, \( J'' \) is the loss compliance, and \( \sigma \) is the applied stress. This equation can be reduced to show the temperature rise rate \( \Delta \dot{T} \):

\[
\Delta \dot{T} = \pi f \Im \left( f, T \right) \sigma^2 / \rho c_p
\]

where \( \rho \) is the density, and \( c_p \) is the specific heat. The temperature rise based on this relationship is not absolutely correct, since this equation does not consider the energy loss to surrounding environment [21].

### 2.6 Strain Controlled Fatigue Testing

Strain controlled fatigue tests are thought to be useful in evaluating the cyclic life of components that contain blunt notches and other moderate stress concentrations. Most constant strain amplitude tests are conducted under fully reversed tension-compression loading conditions. From Figure 2.6, we see that cyclic hardening and cyclic softening may happen under reversed strains. For polymers, cyclic strain softening occurs in crystalline, amorphous and composite polymers. For metals, generally, initially hard materials will cyclically soften, while soft materials will cyclically harden.
The Coffin-Manson model is the most widely used model to portray metals’ cyclic strain fatigue life data, as shown below [22,23]:

\[ \Delta \varepsilon / 2 = CN_f^{-m} \]

where \( \Delta \varepsilon \) is the total strain, \( N_f \) is the number of cycles to failure, \( m \) and \( C \) are fatigue exponent and fatigue ductility coefficient respectively. Initially, they only considered the plastic strain affect the fatigue life. So Coffin and Manson defined \( \Delta \varepsilon \) as plastic strain. Later researcher found the sum of elastic and plastic strain amplitude may be better correlated to fatigue life. This has been mathematically modeled by Morrow, and presented as [24]:

\[ \frac{\Delta \varepsilon}{2} = \sigma' (2N_f)^b + \varepsilon' (2N_f)^c \]

where \( \Delta \varepsilon \) is the total strain amplitude, \( \sigma' \) is the regression intercept called the fatigue strength coefficient, \( E \) is the Young’s modulus, \( N_f \) is the number of cycles to failure, \( \varepsilon' \) is the regression intercept called the fatigue ductility coefficient, \( b \) is the...
Figure 2.6 Cyclic hardening and cyclic softening under reversed strains

Figure 2.7 Schematic of plastic strain and elastic strain effects, and mean stress effect
regression slope called fatigue strength exponent, and \( c \) is the regression slope called fatigue ductility exponent. He also account the mean stress as an influencing factor, the equation was modified to:

\[
\frac{\Delta \varepsilon}{2} = \frac{\sigma'_0 - \sigma_0}{E} (2N_i)^b + \varepsilon'_i (2N_i)^c
\]

where \( \sigma_0 \) is the mean stress. Morrow proposed that the mean stress only affects the elastic portion of the strain-life curve. The schematic of mean stress, plastic strain effect and elastic strain effect are shown in Figure 2.7. During high cycle large strain fatigue test, plastic strain is predominant. So for polymers, elastic portion can be neglected, so that means Morrow’s mean stress correction is ineffective for polymers.

Another well known mean stress correction model is Smith, Watson, and Topper’s (SWT) mean stress correction, shown as below [25]:

\[
\sigma_{\text{max}} \frac{\Delta \varepsilon}{2} = \frac{\sigma'_0}{E} (2N_i)^{2b} + \sigma'_i \varepsilon'_i (2N_i)^{b+c}
\]

where \( \sigma_{\text{max}} \) is the maximum stress. In their equation, \( \sigma_{\text{max}} \) is used to describe the mean stress effect, since in their assumption maximum stress has a relationship with mean stress, as shown in Figure 2.8. But for polymer materials, the initial mean stress can not reflect mean strain, because if a polymer positions in the necking region, stress does not change with strain increasing.

Recently Wong and coworkers derived a new unified strain fatigue model, which has considered not only the mean strain effect, but also amplitude, maximum strain and minimum strain effects, as shown below [26]:

\[
(\varepsilon_{\text{amp}} \times \varepsilon_m \times \varepsilon_{\text{max}} \times \varepsilon_{\text{min}})^2 = C N_i^{m}
\]
where $\varepsilon_{\text{amp}}$, $\varepsilon_m$, $\varepsilon_{\text{max}}$ and $\varepsilon_{\text{min}}$ are the absolute values of strain amplitude, mean strain, maximum and minimum strains, respectively. This semi-empirical unified model absorbs merits of each single strain model, and minimized their drawbacks. The unified strain model is a simple and useful method for fatigue life prediction.

Figure 2.8 A schematic of SWT mean stress correction
Figure 2.9 A schematic of relationship between $\frac{da}{dN}$ and $\Delta K$ in fatigue crack propagation.
2.7 Fatigue Crack Propagation (FCP)

Large number of structures and components in real life contain preexistent defects, and the defects may dominate the fatigue life as crack propagation, not the crack initiation during loading cycles. Notched specimen testing represents a realistic step toward laboratory test simulation of actual service conditions. Paris postulated that the stress intensity factor $K$ was the major controlling factor in the FCP process, and described $da/dN$ in terms of the stress intensity factor range $\Delta K$ with a relationship of the form [27]:

$$\frac{da}{dN} = A\Delta K^m$$

where $a$ is crack length, $N$ is cycles to failure, and $A$, $m$ is constants related to material variables, stress ration, temperature, frequency, environment etc. This equation can be express in another way:

$$\ln \frac{da}{dN} = m \ln \Delta K + \ln A$$

So a straight line relationship can be obtained, as schematic in Figure 2.9.

Paris Law is usually used to determine the cycles to failure during FCP processing theoretically [28]:

$$N_f = \frac{1}{A(Y \Delta \sigma \sqrt{\pi})^{m}} \int_{a_i}^{a_f} \frac{da}{a^{m/2}}$$

where $Y$ is a geometry correction factor that depends on both the crack and specimen sizes and geometries, $\Delta \sigma$ is the stress range, $a_i$ is the initial crack size, and $a_f$ is the final crack size at failure.
2.8 Case Study of Strain Controlled Fatigue Behavior

Stephens and coworkers [29] have done a strain-controlled fatigue study on a kind of polymer matrix composite. His study included strain-controlled fatigue behavior under constant and variable amplitude loading, and effects of mean stress/strain. SEM was used to examine the failed specimens revealed the degradation caused by cyclic loading. Fatigue life predictions using common strain-based models were made. An improved strain-based model was proposed by him.

Structural reaction injection molding (SRIM) is the process of injecting two low viscosity reactive liquids under high pressure into a closed mold containing a performed fiber reinforcement. SRIM polymer matrix composites were used in this study. The resin was Baydui STR 400, and the reinforcement was Owens-corning fiber glass M-8610 randomly oriented continuous strand fiber glass mat. All testing was performed using either an 89 kN or 100kN closed-loop electrohydraulic test. All tests were conducted at room temperature. The failure was defined as final fracture or a 50% drop in the maximum tensile load. Five different ratios of minimum to maximum strain, R=-2, -1.5, -1, 0 and 0.5. R= -1 is a fully reversed test, and others were used to investigate the effects of mean strains and stresses on the low cycle fatigue behavior. The variable amplitude fatigue tests were also performed in strain control at constant frequency for a given specimen.
2.8.1 Monotonic and Cyclic Stress-Strain Behavior

A typical monotonic tensile stress-strain curve is obtained, and shows in Figure 2.10. It is evident from Figure 2.10 that the plastic component of strain is very small, compared to the elastic component of strain in the monotonic response. Figure 2.11 shows the cyclic stress-strain response of the SRIM composite, the hysteresis loops were recorded periodically during the low cycle fatigue tests. At this strain amplitude ($\epsilon/2=0.017$), fracture occurred after 828 cycles. As shown in this figure, the material experienced a continuous decrease in tensile stiffness during cyclic loading. The cyclic degradation in stiffness observed in this composite causes an effect similar to cyclic softening in metals, in which the peak stresses decrease under strain controlled cycling. However, although the effects are similar, the mechanism of deformation and damage which cause the degradation or softening are vastly different. In the case of metals, the softening is generally associated with an increase in the plastic strain amplitude, which produces a widening of the hysteresis loops and a decrease in the peak stresses during cycling. In contrast, the change in the plastic strain amplitude during cycling of the composite was negligible. The degradation of mechanical properties found to occur in many fiber reinforced polymer composites is usually attributed to a variety of irreversible damage processes, including matrix cracking, fiber/matrix debonding, and fiber bucking and fracture.
Figure 2.10 Monotonic and half life cyclic stress-strain curves for R= -1.

Figure 2.11 Series of hysteresis loops for $\varepsilon/2=0.017$, R= -1.
2.8.2 Strain Controlled Fatigue Behavior

The conventional total strain life equation has been mentioned in Page 16, and shown as:

\[ \frac{\Delta \varepsilon}{2} = \frac{\sigma'}{E} (2N_r)^b + \varepsilon'_f (2N_r)^c \]

As discussed previously, the cyclic stress-strain response of this material was predominantly elastic. From Figure 2.12, we can see the elastic strain amplitudes were over an order of magnitude larger than the plastic strain amplitudes. Since the total and elastic strain amplitudes are nearly identical, the strain-life behavior can be accurately represented by plotting the total strain amplitude versus reversals to failure, reducing the conventional equation to a one parameter form:

\[ \frac{\Delta \varepsilon}{2} = B (2N_r)^d \]

From Figure 2.12, we can find this equation can be used with a high degree of confidence to model the strain-life behavior for the SRIM composite.
Figure 2.12 Fully reversed low cycle fatigue behavior, showing total, elastic, and plastic components of strain amplitude.
2.8.3 Mean Stress/Strain Effects and Models

All of the strain-life data for all the strain controlled low cycle fatigue tests shows in Figure 2.13. The tests performed at four strain ratios besides the fully reversed (R=-1) case, with a tensile mean strain, R=0 and 0.5, and two sets with a compressive mean strain, R=-1.5 and -2.

![Figure 2.13 Strain-life curves for all strain ratios](image)

It can be seen that although there was significant mean stress relaxation in the R=0 and 0.5 tests, the presence of tensile mean strains appeared to have a detrimental effect on the fatigue life. The presence of compressive mean strains, on the other hand, appeared to have very little effect on the fatigue life of this material as shown by the
nearly identical curves for the R= -1, -1.5 and -2. This may be in part due to the fact that the compressive mean stresses at half-life were fairly small when compared to the ultimate tensile strength. The preceding results demonstrate that, despite the fact that the mean stress may vary substantially during strain-controlled cycling, the effects of tensile mean strains must be accounted for when modeling the fatigue behavior of this material.

The two most common models used for mean stress/strain correction are Morrow mean stress model and the Smith, Watson and Topper (SWT) mean stress model, which has already been discussed in Chapter 2.6. In Figure 2.14, perfect correlation would be represented by data points lying on the solid diagonal line, and the dashed lines on either side of the diagonal represent error bands of a factor of 10.
Figure 2.14 Comparison of predicted lives by the Morrow and SWT models with experimentally obtained lives for the constant amplitude tests.

It is clear that neither of the mean stress models was successful in accurately predicting the fatigue lives of the constant amplitude specimens. For both models, many of the life predictions overestimated the experimental results by more than an order of magnitude larger than the experimental lives. The poor correlation between experimental and predicted lives using these models is mainly attributed to the transient nature of the cyclic stress-strain response of the composite. Both models
require as input the modulus of elasticity and the mean or maximum cyclic stress at any given strain level. Since these values are continually changing throughout the life of a test, significant errors could arise by simply selecting the half-life values.

It was found in this investigation, a new correction model can be used which is reported by Owen. The maximum strain (rather than the strain amplitude) is plotted versus the number of reversals to failure, and the R=0 and R=0.5 curves collapse onto the R=-1 curve. Based on these observations, a new strain based damage parameter was defined, called the effective strain amplitude, $\varepsilon_{ea}$, which is the maximum strain occurring in the cycles.

A power-law relation can be derived between $\varepsilon_{ea}$ and $2N_f$, was fit, resulting in the following expression:

$$\varepsilon_{ea} = C (2N_f)^m = (0.0343)(2N_f)^{-0.118}$$

This equation can be plotted in Figure2.15, and this plot appears to successfully correlate the effects of mean strains on the fatigue behavior of this material. By using this elective strain amplitude (ESA) method, the life to failure can be solved for directly, eliminating the need to employ the cyclic stress-strain curve.
Figure 2.15 Effective strain amplitude versus reversals to failure.

Figure 2.16 Comparison of predicted lives by the effective strain amplitude (ESA) model with experimentally obtained lives for the constant amplitude tests.
It should be noted, however, that a problem may arise through the use of this model when large tensile mean strains are coupled with very small strain amplitudes.

A cycle with this characteristic would be expected to cause relatively little damage due to the very small strain amplitude. However, due to the large maximum strain that would be present, this model would predict a high amount of damage for such a cycle, resulting in a very low estimate of the fatigue life. This drawback should be considered when using this model to make life predictions where this type of loading may occur to a significant degree.
CHAPTER III

EXPERIMENTAL PROCEDURES.

3.1 Materials and Preparation

The polymers used in this study are Nylon 6, Polypropylene (PP) and 20 wt% CaCO$_3$ modified polypropylene (PP-Blk).

Specimen preparation All of the three pellets materials, PP, PP (Blk) and nylon 6, were injection molded by Vandorn 55 injection-molding machine into ASTM 638D standard dog bone specimens. The dimensions of the standard specimen are shown in Figure 3.1. PP and PP (Blk) don’t need any drying process before injection molding, and nylon 6 was dried at 100 °C for 24 hours.

Injection molding is a commonly method to shape thermoplastics. This method is low-cost, high efficiency and easy manipulation. The schematic injection molding procedure shows in Figure 3.2. The polymer pellets are fed into the injection barrel by the hopper. The pellets are heated to a setting melt temperature in the barrel. Then a reciprocating screw injects the melting fluid into the mold. The mold is cooling down to a constant temperature which can allow the melting polymer to solidify. The mold plates are held together by hydraulic or mechanical force. And cooling method of the mold is always water cooling. Table 3.1 and Table 3.2 show the involved parameters in the injection molding process.
Figure 3.1 A schematic of the injection molded dog bone specimen. (The unit in the figure is in).
(a) Schematic process of injection molding

(b) Digital picture of the Van Dorn 55 injection molding machine

Figure 3.2 Injection molding machine and its mechanism.
Table 3.1 The parameters involved in the injection molding process for the nylon 6.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Temperature at the front of the barrel</td>
<td>500 °F</td>
</tr>
<tr>
<td>Temperature at middle of the barrel</td>
<td>500 °F</td>
</tr>
<tr>
<td>Temperature at rear of the barrel</td>
<td>500 °F</td>
</tr>
<tr>
<td>Temperature at the Nozzle</td>
<td>500 °F</td>
</tr>
<tr>
<td>Injection speed</td>
<td>2 in/sec</td>
</tr>
<tr>
<td>Max Pressure</td>
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</tr>
<tr>
<td>Holding pressure</td>
<td>400 psi</td>
</tr>
<tr>
<td>Holding Time</td>
<td>10 sec</td>
</tr>
<tr>
<td>Shot size</td>
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</tr>
<tr>
<td>Screw speed</td>
<td>120 rpm</td>
</tr>
<tr>
<td>Temperature in the Mold</td>
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<tr>
<td>Cooling Time</td>
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Table 3.2 Parameters involved in the injection molding process for the PP and PP (blk)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
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<tr>
<td>Temperature at the front of the barrel</td>
<td>400 °F</td>
</tr>
<tr>
<td>Temperature at middle of the barrel</td>
<td>400 °F</td>
</tr>
<tr>
<td>Temperature at rear of the barrel</td>
<td>400 °F</td>
</tr>
<tr>
<td>Temperature at the Nozzle</td>
<td>400 °F</td>
</tr>
<tr>
<td>Injection speed</td>
<td>2 in/sec</td>
</tr>
<tr>
<td>Max Pressure</td>
<td>2500 psi</td>
</tr>
<tr>
<td>Holding pressure</td>
<td>400 psi</td>
</tr>
<tr>
<td>Holding Time</td>
<td>10 sec</td>
</tr>
<tr>
<td>Shot size</td>
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<tr>
<td>Screw speed</td>
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</tr>
<tr>
<td>Temperature in the Mold</td>
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<tr>
<td>Cooling Time</td>
<td>35 sec</td>
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</table>
3.2 Thermogravimetric Analysis (TGA)

TGA was adopted to determine the thermal degradations of PP and Nylon 6. The TGA 2050 from TA Instruments was utilized for testing.

Both of the pellets and dog-bone specimen were tested separately. Testing sample has a weight less than 10 mg, and was suspended in an alumina pan from a sensitive arm which is enclosed by the furnace. Nitrogen was pumped for purging. The temperatures increased at a ramping rate of 30 °C/min from 25 °C to 500 °C (PP) or 600°C (Nylon 6). A relationship between sample weight and temperature can be determined by using this method.

Figure 3.3 Digital picture of the TA instruments TGA
3.3 Differential Scanning Calorimetry (DSC)

DSC is a thermo-analytical technique which measures the difference in the amount of heat to increase the temperature of a sample and reference. Then the heat flow difference can be plotted as a function of temperature. The phase transition of a simple can be reflected by heat flow changing in DSC plot. Both of the pellets and dog-bone specimen were tested separately, and weight less than 10 mg. Then temperature ranged from -50 °C to 300 °C for PP, and 0 °C to 300 °C for Nylon 6 with a ramping rate 10°C/min

Figure 3.4 Digital picture of the TA instruments DSC
3.4 Dynamic Mechanical Analysis (DMA)

DMA is a thermo-analytical technique used to study and characterize materials, where a small deformation is applied to a sample in a cyclic manner. This allows the materials response to stress, temperature, frequency and other values to be studied. It is most useful for observing the viscoelastic nature of polymers.

DMA works by applying a sinusoidal deformation to a sample of known geometry.

The sample can be subjected by a controlled stress or a controlled strain. For a known stress, the sample will then deform a certain amount. In DMA this is done sinusoidally. How much it deforms is related to its stiffness. DMA measures the stiffness and damping, these are reported as modulus and tan delta.

Complex modulus $E^*$ can be obtained from below equation:

$$E^* = E' + iE''$$

where

$E' = (\sigma_0 / \varepsilon_0) \cos \delta$

$E'' = (\sigma_0 / \varepsilon_0) \sin \delta$

$E'$ is the storage modulus, obtained from stress and strain relationship, and is a measure of the energy storable during the oscillation period. $E''$ is the complex or loss modulus which is a measure of energy dissipated during the oscillation period. $\sigma_0$ is the amplitude of the stress, and $\varepsilon_0$ is the amplitude of the strain. $\delta$ is the phase angle.
Loss factor tan δ, or called damping- the tangent of the phase angle, is given by the ratio of storage and loss modulus:

$$\tan \delta = \frac{E'}{E''}$$

A Perkin Elmer DMA was employed to perform the test in tensile mode at a frequency of 10 Hz. The temperatures are ranged from -40 °C to 120 °C with a ramping rate 4 °C/min. Liquid Nitrogen was supplied for cooling to -40 °C. Testing specimen were injection molded in the same condition with further testing dog-bone specimen, and were cut to rectangular bars with dimensions of 20mm(H)×10mm(W)×3.06mm(T).

Figure 3.5 Digital picture of the Perkin Elmer DMA
3.5 Tensile Tests

Uniaxial tensile tests are conducted in a quasi static Instron 5582 with a load cell of 10 kN at room temperature (25 °C). In order to determine the true stress-strain relationship, two mechanical contact extensometers are need. The longitudinal and transverse extensometers are used to measure the strains in longitudinal and transverse directions. The setup is shown in Figure 3.6. A set of specimens were loaded at selected loading rates, 20 to 100 mm/min for PP, 40 to 200 mm/min for PP (Blk), and 10 to 60 mm/min for nylon 6. And the true stress ($\sigma$) of each samples can be obtained from [30]:

$$\sigma = P / [W_0 T_0 (1-\varepsilon_z)^2]$$

where $P$ is the applied load, $W_0$ and $T_0$ are the original width and thickness of the specimen. $\varepsilon_z$ is the transverse strain, which can be directly obtained from the transverse extensometer.
Figure 3.6 Transverse and longitudinal extensometers are mounted on the centre of the specimen to measure the strains in the transverse and longitudinal directions.
With the high-precision extensometer employed, the elastic modulus and Poisson’s ration can be determined. Elastic modulus is the slope of true stress-strain curve in the liner range when the strain is relatively small. And Poisson’s ration is the ratio of longitudinal and transverse strain, and both of them can be directly obtained from the extensometers.

In order to determine other mechanical behavior which need to be obtained from engineering stress-strain curve, such as ultimate tensile strength, break strain, A set of specimens were testing at selected loading rates, 20 to 100 mm/min for PP, 40 to 200 mm/min for PP (Blk), and 10 to 60 mm/min for nylon 6. To compare the different mechanical characterization of PP and PP(blk), a set of five specimens of each were testing, and the comparison of PP and PP(blk ) will be discussed later.

3.6 Strain Controlled Fatigue Test

Fatigue tests were conducted under displacement control in tensile mode. An Instron 8500 servo-hydraulic testing machine was employed with a 100 kN load cell at a frequency of 5 Hz. The tests were performed at three different temperatures from each kind of specimen, for PP and PP(blk), -40 °C, 25 °C and 65 °C, for nylon 6 -40 °C, 25 °C and 125 °C. The displacement amplitudes varies from 0.5mm to 2.5mm, and 2.5 mm is the maximum cyclical displacement for this servo-hydraulic machine. At each temperature condition for each kind of specimen, at least three data points were obtained to plot a strain-life cycle curve.
Since the fatigue tests were conducted at both low and high temperature, the specimen need to be gripped up more tightly than performed in room temperature tests. A specific designed clamp was made to conduct well fixation. A threaded rod is machined to form D shaped holders to ensure a fully contact with specimen, and with four tighten screws the specimen can be well fixed in variable conditions.

A temperature chamber was used to control the temperature. The chamber can pump liquid nitrogen to cool down, and generate hot air to heat up. Temperature sensor was included in this chamber, and a settled temperature can be keeping during the test. Figure 3.7 shows the set up of the fatigue tests.

Figure 3.7 The fatigue test setup with sample mounted between the grips prior to testing.
3.7 Modeling of Fatigue Testing

The Wei-Wong unified strain model was used to do curve-fitting. Before Wei-Wong’s model, all of the strain controlled fatigue models are single parameter models, such as the classical Coffin-Manson model, which is widely used to predict strain fatigue life of metallic materials, as shown below:

\[ \frac{\Delta \epsilon}{2} = C N_t^{-m} \]

where \( \Delta \epsilon \) is the total strain, \( N_t \) is the number of cycles to failure, \( m \) and \( C \) are fatigue exponent and fatigue ductility coefficient respectively. This model only considered one strain parameter which will affect the number of life cycles. In fact, the changing of strain amplitude, mean strain, maximum and minimum strains affect the fatigue life, either. The Wei-Wong model integrated four strain effective parameters (strain amplitude, mean strain, maximum and minimum strains) in order to give a more accurate curve-fitting. The Wei-Wong model shows as below:

\[ \left( \epsilon_{amp} \times \epsilon_m \times \epsilon_{max} \times \epsilon_{min} \right)^{\frac{1}{4}} = C N_t^{-m} \]

where \( \epsilon_{amp} \), \( \epsilon_m \), \( \epsilon_{max} \) and \( \epsilon_{min} \) are the absolute values of strain amplitude, mean strain, maximum and minimum strains, respectively.

So if we have a log-log plot, there exists a liner relationship between unified strain \( \left( \epsilon_{amp} \times \epsilon_m \times \epsilon_{max} \times \epsilon_{min} \right)^{\frac{1}{4}} \) and the number of cycles to failure \( N_t \).
3.8 Factography Configuration

The technique of scanning electron microscopy (SEM) is the most widely used method of studying fracture surface morphology. SEM can still give good resolution and a high depth of field rather than optical microscopy even the specimen cross area is quite small. In this study, A SEM was utilized to obtain image of fracture surface after tensile tests and fatigue tests.

Figure 3.8 Digital picture of the Hitachi SEM
CHAPTER IV
RESULTS AND DISCUSSIONS

4.1 Molecular Degradation

TGA was done to determine whether the processing induce any molecular degradation in each kind of specimen or not. The relationship between mass loose of the sample and temperature increasing was plotted for each kind of specimen. Figure 4.1, 4.2 and 4.3 shows the comparison of TGA results before and after processing for nylon 6, PP and PP(blk) respectively. Thermal degradation temperature is defined as the temperature at 5% mass loss. The thermal stability results are summarized in Table 4.1. In the temperature range of 350-440 °C, nylon 6 has a mass loss of 95%. In case of PP, the major mass loss of 90% was observed at 400-480 °C and a mass loss of 65% PP (blk) was observed at 440-500 °C. Fig 4.4 shows a comparison of TGA curves of nylon 6, PP and PP(blk). From Figure 4.4, it can be inferred that PP has better thermal stability, followed PP(blk) and nylon 6 respectively.

Finally, The TGA plots of before and after processing show that there is a minimal and acceptable molecular degradation arising, since the melting injection molding conditions.
Figure 4.1 Thermogravimetric Analysis: Change in weight (%) of sample with rise in temperature (°C) for nylon 6 pellets before injection molding and nylon 6 samples after injection molding.
Figure 4.2 Thermogravimetric Analysis: Change in weight (%) of sample with rise in temperature (°C) for PP pellets before injection molding and PP samples after injection molding.
Figure 4. 3 Thermogravimetric Analysis: Change in weight (%) of sample with rise in temperature (°C) for PP (blk) pellets before injection molding and PP (blk) samples after injection molding.
4.2 Glass Transition Temperatures

Glass transition temperature ($T_g$) means where the polymer goes from a hard, glass like state to a rubber like state. Polymers can be determined by DSC and DMA. DSC defines the glass transition as a change in the heat capacity as the polymer matrix goes from the glass state to the rubber state. In the DSC the transition appears as a step transition and not a peak such as might be seen with a melting point. DMA shows $T_g$ as the peak of tan $\delta$, which is a more sensitive way to yield $T_g$, however some polymers are more amenable to DSC such as epoxies.

Figure 4.4 and 4.5 illustrate the variation of tan $\delta$ and storage modulus as a function of temperature of nylon 6, PP and PP(blk). The $T_g$ of nylon 6, PP and PP(blk), shown in Figure 4.5 the peak of tan $\delta$, are 49 °C, 3 °C and -11 °C respectively. The literature values of $T_g$ are 0 °C for PP and 47 °C for nylon 6. From Figure 4.5, we know that PP and nylon 6 exhibits superior storage modulus rather than PP (blk), and nylon 6 has a larger strange modulus during 0-47 °C than PP, but PP exhibits better storage modulus than nylon 6 below 0°C or above 47 °C.

4.3 Melting Point

Melting temperature is the temperature range at which the state of polymer changes from solid to liquid. Melting is an endothermic processing, and that results an endothermic peak in DSC curve. From Figure 4.4, we can find the endothermic peaks for each material obviously. And the melting point is 213 °C, 158 °C and 158 °C for nylon 6, PP and PP(blk) respectively. The integral of the peak is the amount of heat
which is needed to fulfill a melting process. PP has the best thermal capacity for melting, followed by nylon 6. PP (blk) has a similar melting point with PP, but since it has a high CaCO₃ content, the thermal capacity is poorer than PP.

All of the thermal character data can be found in Table 4.1.

Table 4.1 Thermal character data: glass transition temperature, melting point and degradation temperature for nylon 6, PP, PP (blk) respectively.

<table>
<thead>
<tr>
<th></th>
<th>Glass Transition Temperature (DSC)</th>
<th>Glass Transition Temperature (DMA)</th>
<th>Melting Point (DSC)</th>
<th>Degradation Temperature (TGA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nylon 6</td>
<td>35 °C</td>
<td>49 °C</td>
<td>213 °C</td>
<td>360 °C</td>
</tr>
<tr>
<td>PP</td>
<td>-2 °C</td>
<td>3 °C</td>
<td>158 °C</td>
<td>364 °C</td>
</tr>
<tr>
<td>PP (blk)</td>
<td>5 °C</td>
<td>-11 °C</td>
<td>150 °C</td>
<td>335 °C</td>
</tr>
</tbody>
</table>
Figure 4.4 Dynamic Mechanical Analysis: Variation of \( \tan \delta \) as a function of temperature.
Figure 4.5 Dynamic Mechanical Analysis: Variation of storage modulus as a function of temperature.
Figure 4.6 Differential Scanning Calorimetry: Variation of heat flows as a function of temperature.
4.4 Stress and Strain Relationships

The engineering stress-strain curves for each kind of materials were obtained by conducting tensile tests at selected loading rates, the figures can be found in APPENDIX A. The ultimate tensile strength is increased as a function of loading rate, as shown in Figure 4.7. Nylon 6 has larger ultimate tensile strength, and followed PP and PP (blk). PP(blk) exhibits best elongation character, and nylon 6 is the most ductile one.

For PP and PP (blk), five specimen of each were tested in the loading rate of 50 mm/min, Figure 4.8 shows the ultimate tensile strength for each is consistent for repeated tests, but PP possesses significant variation of break elongation. After engineering stress-strain testing, two extensometers were mounted, and tests were conducted again, the true stress-strain curves were obtained. Figure 4.9, 4.10 and 4.11 show true stress-strain relationships of nylon 6, PP, PP(blk).

The elastic modulus is determined by the slope of true stress-strain curve in the initial region. Different load rates hardly affect the elastic modulus. The average elastic modulus is 1.77 GPa, 0.98 GPa and 0.33 GPa for nylon 6, PP and PP (blk). The Poisson's ratios of nylon 6, PP and PP (blk) are 0.10, 0.18 and 0.19 respectively, which is obtained from the slope of transverse strain and longitudinal strain curves in the initial region. The elastic modulus data and Poisson ration data can be found in APPENDIX B.
Figure 4.7 The effect of loading rate on the ultimate tensile strength of nylon 6, PP and PP (blk).
Figure 4.8 Comparison of tensile responses of PP and PP (blk) at a loading rate of 50 mm/min.

(a) Engineering stress and strain relationship for PP at 50 mm/min loading rate

(b) Engineering stress and strain relationship for PP (blk) at 50 mm/min loading rate

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Figure 4.9 Variation of true stress with true strain for nylon 6 at different loading rates.
Figure 4.10 Variation of true stress with true stain for PP at different loading rates.
Figure 4.11 Variation of true stress with true stain for PP at different loading rates.
4.5 Strain Controlled Fatigue Test

Most of the fatigue data published in literatures is obtained from stress controlled test. Strain controlled fatigue data is as important as ones from stress controlled fatigue test for polymer insulation design. Since polymer can be cyclically loaded for several years or longer at small strain amplitude, we applied an initial mean strain to shorten polymers’ life time to an acceptable time scale. We set an arbitrary cut-off point at 2 million cycles for room temperature test and high temperature test. Due to the limitation of liquid nitrogen, the low temperature test is restricted to less than 40,000 cycles. The three different temperatures from each kind of specimen are PP and PP(blk), -40 °C, 25 °C and 65 °C, and nylon 6, -40 °C, 25 °C and 125 °C. Fig 4.12, 4.13 and 4.14 show plots of unified strains versus fatigue life predictions based on Wei-Wong model for nylon 6, PP and PP (blk) respectively. Table 4.2, 4.3 and 4.4 show all the fatigue testing data from different temperature tests of nylon 6, PP and PP (blk), and the fatigue exponents and ductility coefficients calculated from Wei-Wong’s model.
Figure 4.12 Nylon 6 fatigue life prediction based on Wei-Wong unified strain model.
Figure 4.13 PP fatigue life prediction based on Wei-Wong unified strain model.
Figure 4.14 PP (blk) fatigue life prediction based on Wei-Wong unified strain model.

\[
\left( \epsilon_{\text{amp}} \times \epsilon_{m} \times \epsilon_{\text{max}} \times \epsilon_{\text{min}} \right)^{\frac{1}{r}} = 291.7427 N_f^{0.05685}
\]

\[
\left( \epsilon_{\text{amp}} \times \epsilon_{m} \times \epsilon_{\text{max}} \times \epsilon_{\text{min}} \right)^{\frac{1}{r}} = 79.8601 N_f^{-0.1092}
\]
Table 4.2 Summary of nylon 6 fatigue testing data and materials constants.

<table>
<thead>
<tr>
<th></th>
<th>Amp (%)</th>
<th>Mean Strain (%)</th>
<th>Max Strain (%)</th>
<th>Min Strain (%)</th>
<th>R</th>
<th>Nf</th>
<th>M</th>
<th>C</th>
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</thead>
<tbody>
<tr>
<td>Nylon 6 (25 °C)</td>
<td>2.72</td>
<td>4.66</td>
<td>7.38</td>
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<td>0.26</td>
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<td>4.66</td>
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<td>10.1</td>
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<tr>
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<td></td>
<td>3.11</td>
<td>7.78</td>
<td>10.89</td>
<td>4.67</td>
<td>0.43</td>
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<td>248.57</td>
<td>233.85</td>
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<td></td>
<td>6</td>
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<td>0.97</td>
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Table 4.3 Summary of PP fatigue testing data and materials constants.

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<th>Amp (%)</th>
<th>Mean Strain (%)</th>
<th>Max Strain (%)</th>
<th>Min Strain (%)</th>
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<th>Nf</th>
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<tr>
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<td>0.82 67 723</td>
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<tr>
<td>PP (65°C)</td>
<td>2.36 14.15 16.51 11.79</td>
<td>0.71 2140 0</td>
<td>0.07 67 723</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP (0°C)</td>
<td>2.34 23.39 25.73 21.05</td>
<td>0.82 50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP (65°C)</td>
<td>4.48 268.85 273.33 264.37</td>
<td>0.97 1303 00</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP (0°C)</td>
<td>4.47 461.97 466.44 457.5</td>
<td>0.98 4572 00</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP (65°C)</td>
<td>4.48 468.33 472.81 463.85</td>
<td>0.98 3090 00</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

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Table 4.4 Summary of PP (blk) fatigue testing data and materials constants.

<table>
<thead>
<tr>
<th></th>
<th>Amp (%)</th>
<th>Mean Strain (%)</th>
<th>Max Strain (%)</th>
<th>Min Strain (%)</th>
<th>R</th>
<th>Nf</th>
<th>M</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP(BI)</td>
<td>7</td>
<td>445.7</td>
<td>452.7</td>
<td>438.7</td>
<td>0.9</td>
<td>137800</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>7</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP(BI)</td>
<td>7.01</td>
<td>543.5</td>
<td>550.5</td>
<td>536.5</td>
<td>0.9</td>
<td>115700</td>
<td>0.056</td>
<td>291.742</td>
</tr>
<tr>
<td>(25°C)</td>
<td></td>
<td>7</td>
<td>8</td>
<td>6</td>
<td>7</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>7.03</td>
<td>870.8</td>
<td>877.8</td>
<td>863.8</td>
<td>0.9</td>
<td>650</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.92</td>
<td>48.23</td>
<td>52.15</td>
<td>44.31</td>
<td>0.8</td>
<td>38900</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PP(BI)</td>
<td></td>
<td>6</td>
<td>9</td>
<td>3</td>
<td>8</td>
<td>650</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(−40°C)</td>
<td>3.96</td>
<td>137.4</td>
<td>141.4</td>
<td>133.5</td>
<td>0.9</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>5</td>
<td>3</td>
<td>4</td>
<td>65</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Compare to $T_g$ obtained from DMA are 49 °C, 3 °C and -11 °C for nylon 6, PP and PP (blk) respectively. All of the low temperature tests conducted under their glass transition, so only low stretch strain is allowed, and brittle materials is more sensitive to cyclical displacement load. Nylon 6 has significant temperature effect, and increasing temperature makes higher unified strain endurance and has longer fatigue life, so the fatigue life curve move to top left corner in the figure with temperature increase as expressed in Wei-Wong unified model. PP under low temperature and room temperature tests exhibit similar fatigue behavior, superposition of two fatigue curves was shown in Figure 4.13. But this superposition is just a theoretical phenomenon, PP in low temperature has low tensile strain endurance in real experiment, and can not reach the same unified strain level as in room temperature. PP is sensitive to high temperature, and fatigue life is significant increasing in high temperature test. In the same temperature range PP(blk) has the best fatigue behavior compared to nylon 6 and PP. During high temperature tests, PP (blk) exhibits rubber-like characters, and didn’t fail in two million cycles even the mean strain is 1200%, which is the maximum strain can be stretched to in our test machine.
4.6 Factographs of Fatigue Failure Specimens

The morphology of different materials’ cross section surfaces reveals the different failure mechanism. The SEM graph of cross section surfaces show in Figure 4.14, 4.15 and 4.16 for nylon 6, PP and PP(blk) respectively, and all of the specimens are obtained from room temperature failure. Nylon 6’s fracture surface has distinct beach marking, similar with aluminum fracture, and also observed in ductile polymers, such as polycarbonate, polysulfone and poly(methyl methacrylate)[31]. PP has a rough delaminated cross section. PP (blk) has a more uniform fracture cross section, and some CaCO₃ grains can be seen in this surface. More detailed discussion and advanced fractography research is beyond the scope of present fatigue behavior study.
Figure 4.15 SEM graph of nylon 6’s cross section by fatigue failure.
Figure 4.16 SEM graph of PP’s cross section by fatigue failure.
Figure 4.17 SEM graph of PP (blk)’s cross section by fatigue failure.
CHAPTER V

CONCLUSIONS

Three cable insulation materials are studied, viz nylon 6, PP and PP (blk). Their thermal behaviors were examined firstly. Glass transition, Melting point and degradation temperature were determined. Thermal dynamic tests give the relationships between modulus and temperature. Tensile tests were performed to determine true stress-strain relationship. The ultimate strengths are increasing with loading rate increase. Elastic modulus and Poisson ratio can be obtained from the true stress-strain curve, and they do not dependent to loading rate. Nylon 6 has the higher elastic modulus and the poorest Poisson ratio, PP is in the middle, and PP (blk) has lowest elastic modulus and highest Poisson ratio. Strain controlled fatigue tests were performed at three different temperatures. Nylon 6 exhibits significant temperature dependence and PP is sensitive to high temperature, not to low temperature. PP (blk) possesses the best fatigue behavior.
REFERENCES


Figure A.1 Nylon 6’s engineering stress-strain relationship at different loading rate.
Figure A.2 PP’s engineering stress-strain relationship at different loading rate.
Figure A.3 PP (blk)’s engineering stress-strain relationship at different loading rate.
Table A.1 Ultimate tensile strength data at different loading rate.

<table>
<thead>
<tr>
<th>Loading Rate (mm/min)</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>40</th>
<th>50</th>
<th>75</th>
<th>100</th>
<th>125</th>
<th>150</th>
<th>175</th>
<th>200</th>
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</thead>
<tbody>
<tr>
<td>Tensile Strength of Nylon 6 (MPa)</td>
<td>71.72</td>
<td>75.09</td>
<td>76.85</td>
<td>77.78</td>
<td>77.80</td>
<td>79.24</td>
<td>27.91</td>
<td>28.32</td>
<td>28.41</td>
<td>29.42</td>
<td>29.64</td>
<td>30.05</td>
<td>30.24</td>
<td>30.46</td>
</tr>
<tr>
<td>Tensile Strength of PP (MPa)</td>
<td>14.34</td>
<td>14.59</td>
<td>14.74</td>
<td>15.18</td>
<td>15.29</td>
<td>15.69</td>
<td>15.92</td>
<td>15.33</td>
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</table>
APPENDIX B
ELASTIC MODULUS AND POISSON’S RATIO DATA

Table B.1 Elastic modulus and Poisson’s ratio data at different loading rate.

<table>
<thead>
<tr>
<th>Loading Rate (mm/min)</th>
<th>Elastic Modulus</th>
<th>Poisson’s Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PP (MPa)</td>
<td>PP-blk (MPa)</td>
</tr>
<tr>
<td>5</td>
<td>118.5</td>
<td>106</td>
</tr>
<tr>
<td>10</td>
<td>118.9</td>
<td>100</td>
</tr>
<tr>
<td>15</td>
<td>122.4</td>
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<td>111</td>
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<td>25</td>
<td>132.3</td>
<td>108</td>
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<tr>
<td>30</td>
<td>147.2</td>
<td>111</td>
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<tr>
<td>40</td>
<td>167.2</td>
<td>107</td>
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<td>50</td>
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<td>108</td>
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<tr>
<td>60</td>
<td>-</td>
<td>100</td>
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</table>