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INVESTIGATION OF MICROWAVE WELDING OF THERMOPLASTICS USING INTRINSICALLY CONDUCTIVE POLYANILINE

DISSERTATION

Presented in Partial Fulfillment of the Requirements for the Degree Doctor of Philosophy in the Graduate School of The Ohio State University

By

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ABSTRACT

Intrinsically conducting polymer, such as polyaniline, absorbs microwave energy which offers the opportunity to develop new joining techniques. The main objective of this work is to develop a novel joining technique which combines microwave energy and intrinsically conducting polymers. The effect of welding parameters such as heating time, pressuring method, and polyaniline concentration on joint strength were studied and an equivalent circuit heating model was also constructed to predict the power absorption of the conducting composites. The finite element method was also used to calculated the internal heat generation rate during welding process. Multi-mode microwave welding of HDPE was successfully demonstrated by placing the conducting composite at joint interface. It was found that increasing the gasket thickness results in higher joint strength using constant pressuring method. Also, increasing the polyaniline concentration in the gasket results in higher joint strength. It was also found that increasing the welding pressure results in higher joint strength. However, the constant pressuring method squeezes out the heating composite in the middle of the heating stage; thus, less heating occurs. The maximum joint strength was 86% of the HDPE strength for both 50%, 1mm thick gasket and 60%, 0.5mm thick gasket. A modified multi-mode microwave welding
method, post heating pressure, was also demonstrated to improve the joint quality. The maximum joint strength was equal to the bulk strength of HDPE. It was found that post heating pressure method not only increases the maximum joint strength but also reduces the heating time to reach a specific joint strength. The FEM calculation of the heat generation rate shows that 60%, 0.5mm gasket provides faster heating rates and higher temperatures than the 50%, 1mm which reveals that gasket heating ability depends on the gasket composition and not the amount of the conducting powder in the gasket. Single mode microwave welding of HDPE was also successful and it reduced the heating time from 80 seconds in multi-mode to 15 seconds in single mode due to a higher power source and better energy transfer efficiency. The maximum heat generation rate using 1800 watts and 60%, 0.5mm PANI composite was $6.5 \times 10^8$ (w/m$^3$) which was estimated from FEM calculations. An equivalent circuit model representing the single mode microwave heating of conducting composite was constructed to predict the initial power absorption (initial heat generation rate). It is in very good agreement with experimental temperature measurements. In addition, the circuit model provides design guideline for future development in conducting composites. Single mode microwave welding of nylon 6/6 was also demonstrated using polyaniline/nylon12 composite films. The maximum joint strength was 97% of the nylon 6/6 strength in 10 seconds of heating time.
DEDICATED TO MY PARENTS
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CHAPTER 1

INTRODUCTION

The use of plastics and their composites in structure applications is rapidly growing due to the many advantages that these materials offer. However, the use of these materials is often limited by the ability to quickly produce high quality joints with good repeatability and predictable properties. Therefore, it is critical to develop a new and faster joining technique. Intrinsically conducting polymer, such as polyaniline, offer this opportunity in developing new joining technologies. Microwave energy has been used in drying and heating of materials for many years. The high penetration ability of the microwave energy provides the fast heating and short processing cycle in industrial applications. Therefore, the main objective of this work is to develop a novel joining technique which combines the microwave energy and intrinsically conducting polymers. Chapter 1 gives a brief introduction on polymers and on existing joining techniques. Chapter 2 describes the use of conducting polymers in joining. Chapter 3 discusses the feasibility study on conductive composite heating. Chapter 4 shows the multi-mode microwave welding of thermoplastics. Chapter 5 discusses the equivalent circuit model for
microwave heating. Chapter 6 shows the single mode microwave welding of high density polyethylene (HDPE) and nylon 6/6.

1.1 Joining of Plastics

A polymer is a structure, containing many groups with identical chemical units, linked together, which exhibits specific thermal, mechanical, electrical, and flow properties. The natural form of polymers such as deoxyribonucleic acid (DNA), ribonucleic acid (RNA), and proteins in the human body serve very important functions in human life. Artificial polymers such as Nylon, Polyvinyl chloride (PVC), and Epoxy are made by the synthesis of chemical compounds which play a significant role in modern society.

Today, people refer to artificial polymers as plastics which are divided into three categories: elastomers, thermosets and thermoplastics [1]. Elastomers are either crosslinked polymers or polymers with a large degree of entanglements. They exhibit an enormous extension and recovery under tensile testing. The word rubber is usually used, and the glass transition temperature(Tg) is defined as Tg + 75°C ≤ room temperature, T_{room} [2]. Since most of the elastomers are crosslinked materials, they cannot be reheated and deformed repeatedly. Thermosets are rigid highly crosslinked polymers, where the long molecules are linked together through the primary chemical bonds. Therefore, they cannot be deformed at elevated temperature. Hence, continuous processing is not possible due to the chemical decomposition of its network structure. Epoxy is one example of a
Thermoplastics are linear or branched polymers that can be deformed and shaped into new geometries upon the application of heat and pressure. This shape modification process is repeatable and also reversible.

Thermoplastics contain two groups namely, amorphous and crystalline. An amorphous material has a randomly arranged structure. The temperature for deformation is defined by the glass transition temperature. When the polymer is heated to Tg, the polymer chains start to rotate and move which increase their mobility for processing. In many cases, amorphous polymers are transparent. Also, the modulus drops dramatically at the Tg enabling in easy deformation. Materials such as polycarbonate (PC), Polyester glyol (PETG), and polymethy methacrylate (PMMA) are examples of amorphous thermoplastics. Crystalline materials have their molecules orderly arranged and thus are usually opaque. However, a pure crystalline material is difficult to obtain. Thus, a thermoplastic material containing 50% to 90% crystallinity is defined as a semicrystalline polymer [2]. For a semi-crystalline polymer, the softening temperature is defined as the melting temperature, Tm, where the ordered polymer chains become randomly distributed and the modulus drops rapidly. Some of this crystalline structure may be recovered upon cooling but some remain in an amorphous state. These materials, such as polyethylene (PE), nylon, polypropylene (PP), can be processed at temperatures above Tm under pressure. The detailed descriptions of these materials mechanical, thermal, and flow properties can be found in the references [1,2,3,4]. In general, plastics are easy to shape in the molten stage. Compression molding, injection molding, extrusion can produce very
complex geometry; however, when insertion of a second part is required or the product is too large and complicated in geometry, joining of plastics is needed to manufacture the product.

Joining is a process which combines two separated parts together to perform a particular function. Techniques for joining of thermoplastics and their composites include mechanical joining, adhesive bonding, and welding. Mechanical joining can be separated into fastening, press fitting, and snap fitting. However, fasteners are an expensive multi-step process, including hole drilling and screw insertion and fastening. Also it introduces stress concentrations at the joint. Press fitting and snap fitting are loose joints that cannot transfer large loading. Although adhesive bonding results in a more uniform load transfer, it introduces a second phase material at joint interface and it requires surface preparation and long curing time, resulting in high cost. Welding needs shorter times than adhesive bonding and can transfer large loads uniformly; therefore, it is considered as the best candidate in joining engineering thermoplastics.

The welding processes generally can be divided into five stages, (1) Material preparation (2) Heating and heat transfer, (3) Pressing and squeeze flow, (4) Intermolecular diffusion, and (5) Cooling and solidification. First, the materials under welding must be formed into the desired geometry and have the surfaces free of contamination for joining. Heating provides energy to raise the sample temperature which softens the parts and creates a molten layer for joining. Depending on the heating method applied, this step requires anywhere from seconds to minute to create the appropriate
molten layer for joining; usually this step is the dominating factor regarding to welding cycle. Following the application of heat is the pressing and squeeze flow stage which deforms the surface asperities and provides intimate contact between the weldments. The molten layers are squeezed out to remove entrapped air and unfavorable materials such as mold release. The squeeze flow process is relatively short, on the order of a few seconds but pressure is usually applied until the termination of the process. Intermolecular diffusion happened in a very short time and it causes chain entanglements which builds up the joint strength. Cooling and solidification determine the final microstructure and level of residual stresses and distortions.

Welding techniques can be divided into three categories based on the heating method applied, External (thermal) heating methods, Internal (friction) heating methods, and Electromagnetic heating methods. External heating methods include hot gas welding, extrusion welding and hot plate welding. Hot gas welding uses compressed air (or inert gas) heated by a heating coil, flowing into the joint interface thus melting the parts and filler material. Due to convection heat transfer, this is a slow and low efficiency process, but with low cost and light equipment. Extrusion welding is similar to hot gas welding, but the filler material is melted first and then injected into the joint interface. It is faster than hot gas welding. This method is suitable for automation when welding large structures. Hot plate welding is a widely used joining method. It uses a heated plate contacting the materials to be welded. Molten layers are created through heat conduction. Once the molten layers are generated, the heated plate is removed and the samples are pressed
together. When joining two different materials, two heated plates with different temperature settings or non-contact heating method with different gaps (between sample and heated plate) can be used to achieve the joint [5]. It is a slow process on the order of 30 seconds for small parts and 30 minutes for large parts, but it provides good joint quality.

Internal heating methods include spin welding, vibration welding and ultrasonic welding. Spin welding rotates the sample under pressure and uses surface friction to generate the heat. The process is limited in joint geometry to circular parts, but it is fast and it can provide good joint quality. This is a low cost process; in most cases, a drilling machine or a lathe can be modified to form a spin welder. In fact, the air intake resonators in automobiles have been successfully welded by spin welding [6]. Vibration welding uses surface friction by linear motion or orbital motion to generate the heat and it is a rapid process. Most machines operate at 120 to 240 Hz with less than 5 mm amplitude of vibration [7]. It can join a car bumper that is 1.5 meter long at a rate of 70 parts/hour [8], but the equipment is expensive and limited in joining flat geometry samples. The welding cycle is short, usually less than 10 seconds. Ultrasonic welding, the most popular welding process, uses mechanical vibration at 15KHz, 20KHz or 40KHz. The amplitude of vibration is in the range from 15 to 60 μm. The mechanical energy is converted into heat due to intermolecular friction and surface asperities at the joint interface. An energy director is usually used in ultrasonic welding which creates a high concentration of vibration energy resulting in a fast heating rate. This is a very fast process and it is
especially good for small joint areas. It can also join advanced thermoplastic composites [9] and the use of ultrasonic vibration to mold the ultrahigh molecule weight polyethylene was also reported [10].

Electromagnetic heating methods include implant resistance welding, induction welding, radio frequency welding, microwave welding and infrared welding. Implant resistance welding uses a DC or AC power source which passes a current through a metallic resistive element and due to Joule’s Law, heat is generated. Because of the insertion of the resistive material, thermomechanical mismatch is introduced into the joint that may reduce the joint strength or causes corrosion problems and stress concentrations [11]. However, the process does provide a simple way for welding a complex joint.

Induction welding uses an AC power supply in the KHz to MHz range which generates an alternating magnetic field and thus causes an induced voltage inside the resistive material which generates eddy current. The process depends on the resistance of the material at the joint interface. For most polymers no heat can be generated; but this process can be used in the food packaging industry where an aluminum foil is introduced into the container [12]. In some applications, ferromagnetic material was inserted into the joint to generate the heat. This is a fast process and it can handle complex joints. Also it can be used to disassemble the joint by re-heating the ferromagnetic material but it is an expensive process.

Radio frequency welding uses an alternating electromagnetic field between two parallel plates that causes polar molecules to rotate and align with the electric field. Heat is
generated by internal molecular friction. Most of the applications are in sealing of films. The process is fast but is limited to specific materials which have a highly polar molecules, such as PVC, ABS, and nylon. By placing an electromagnetic absorber at the joint interface, such as a conducting polyaniline composite, radio frequency welding can be used in joining non-polar materials [13] such as HDPE.

Microwave welding is similar to radio frequency welding but with higher operating frequency. Microwave is usually generated by magnetron tube which operates at 2.45 GHz. The concept of microwave welding is by placing a heating element at joint interface which absorbs the microwave radiation and generates the heat. However, there are only few publications regarding microwave welding.

Infrared welding uses radiation energy from an infrared tube. Heating occurs due to the material absorption of the radiation energy. Most polymers exhibit a strong absorption at wavelength of 3.2 to 3.6 micrometers because of the carbon-hydrogen bonds. Therefore, some polymers have very fast heating rates under infrared radiation, and thus a short welding cycle can be achieved [14]. In contrast, some polymers are transparent; thus, through transmission welding is possible through joint design [15]. In most cases, a dark colored polymer has a strong absorption. The process is fast, efficient and low cost; however, the joint geometry is sometimes restricted by the source geometry.

All of the welding techniques previously mentioned are suitable for making small joints except for vibration welding. However, vibration welding is usually limited to joining flat and simple geometry. It is highly desirable to produce large and complex joints
in automotive, aerospace and infrastructure industries; besides, in some cases, it is required to produce a joint that can be separated later for recycling purposes. Therefore, it is necessary to develop a new joining technique that can satisfy these requirements.

1.2 Applications of Microwave Energy

The science of magnetism was founded in the thirteenth century; however, electricity began four hundred years later. It was not until 1873 when James Clerk Maxwell (1831-1879) published his Maxwell’s Equations that electromagnetic waves were defined. However, it was 15 years later, when Heirrich Hertz, a physics professor at Karlsruhe, Germany, made a series of experiments which proved the Maxwell’s theory of electromagnetic waves. With the invention of the telephone by Bell and Gray, intensive study on the transmission line was carried out. Oliver Heaviside made significant contributions on simplifying Maxwell’s theory, and provided its application to transmission line theory. In 1897, Lord Rayleigh proved mathematically that a wave can propagate in the waveguide for both circular and rectangular sections. He also developed the theory of the modes of propagation transverse electric, TE, transverse magnetic, TM, and the cut-off frequency. The experimental work was finished by George. C. Southworth at AT&T in New York through a water filled cooper pipe in 1932. The developments of modern microwave applications were started during World War II due to military requirements. The magnetron was developed in Great Britain as a high power microwave source which engaged the development of the modern radar system. The first microwave oven was built in America in January 1947 by Raytheon. The MIT Radiation Laboratory
also contributed to the theories and the applications. The first industrial application was in
drying of potato chips. Today, wide ranges of industrial microwave applications are
utilized.

Microwave frequency falls in a range from 300 MHz to 300 GHz that is between
the far infrared and FM broadcast radio band in the electromagnetic spectrum as shown in
figure 1 [16]. Microwaves are electromagnetic waves that contain time varying electric
and magnetic fields. Microwave heating of materials is based on the material's nature,
while some absorb the magnetic field and some absorb the electric field or both. Modern
microwaves are operated at 915 MHz or at 2450 MHz (USA) by the restrictions of
Federal Communication Committee (FCC). The major applications are in drying, thawing,
cooking, and heating. Of course, radar and telecommunication are another field of
applications using microwaves.

The industrial applications in drying include paper, printing, leather, textile, wood,
plywood, ceramics, rubber and plastic industries. In the paper industry, the microwave is
not only used in evaporation of water but also in drying the coating and glue on the paper.
A 100-kW klystron operating at 2.45 GHz was installed for paper drying [17]. The major
advantage of using microwave is that it raises the bulk material temperature volumetrically
instead of conduction from the surface such as conventional oven heating. It is especially
Wavelength (m) | Frequency (Hz)
---|---
10^{-15} | 10^{24}
1 nm | 10^{21}
1 pm | 10^{18}
1 μm | 10^{15}
1 mm | 10^{12}
1 m | 1 GHz
1 km | 1 MHz
10^6 m | 1 kHz
1 Hz

γ-rays
X-rays
Ultraviolet
Visible light
Infrared
Millimetre wave
Microwave
UHF
VHF
Short wave
Medium wave
Long wave

Radiotherapy
Imaging
Photolithography
Optoelectronics
Radar
Communications
TV
Radio
Navigation

Figure 1: Electromagnetic spectrum [16]
useful for low thermal conductivity materials. Some special applications includes sterilization [18], and molding of reinforced plastics [17]. In addition, curing of rubber was also reported [19] which showed that microwave curing is more economic than conventional curing methods. Manufacturing of a submarine structure using PEEK-carbon composite was reported to have significant heating results through microwave applicator design [20]. Microwave can also be used in sintering ceramics and welding of ceramic parts [21,22,23]. Furthermore, morphology in thermoplastic modified epoxy can be controlled by using microwave energy [24].

In food industries, the microwave was used in cooking of poultry, bacon, and meat loaf. Cooking of vegetable products were also reported to have significantly improvement in quality of grains and beans. Thawing of frozen foods by microwave is also used in food industries due to the excellent penetration of microwave energy. The major advantage in microwave thawing is time savings compared to conventional thawing methods. Drying of foods such as pasta, onion and tomato paste were reported faster and more beneficial than using conventional methods [17]. Some special applications exist such as, soil treatment, in which microwave energy is used to destroy unfavorable weeds [25], and wine making, in which microwave energy is used to increase the grape temperature in fermentation [17]. Even more, opening of oysters was also attempted using microwave energy [26]. The domestic microwave applications include home cooking, baking, heating and thawing. One
advanced application is a newly developed microwave clothes dryer which was reported to generate remarkable energy savings [27].

Most of the applications mentioned above are based on the dielectric losses of the materials presented in the structure. This is based on the ability of the charges inside the material to align with the incoming alternating electric field and incapacity of polarization to follow the reversal field. In general, there are four types of polarization, namely electronic, atomic, dipolar and interfacial (Maxwell-Wanger) polar [28]. Electronic polarization comes from the displacement of electrons around the nuclei. Atomic polarization is similar to electronic polarization but with unequal charge distribution in the molecule. Dipolar polarization is caused by the presence of permanent dipoles in the materials. Maxwell-Wagner polarization occurs at interfaces in a multi-phase system. The term “loss” is usually used to describe the heating ability of a material under high frequency radiation. In addition to these losses, a material with a finite conductivity (σ) also experiences Joule losses, thus an effective loss factor (\(\varepsilon_{\text{eff}}^\ast\)) that includes all losses is used. When conductive losses of the material are high compared to dielectric losses, then dielectric losses can be ignored, especially at low frequency. The measure of the losses is effective loss tangent (\(\tan \delta_{\text{eff}}\)) [28]

\[
\tan \delta_{\text{eff}} = \frac{\varepsilon_{\text{eff}}^\ast}{\varepsilon^\ast} \quad \text{(Eq. 1-1)}
\]

where \(\varepsilon^\ast\) is the real part of the dielectric constant, and the effective loss factor is

\[
\varepsilon_{\text{eff}}^\ast(\omega) = \varepsilon^\ast_d(\omega) + \varepsilon^\ast_a(\omega) + \varepsilon^\ast_{\text{MW}}(\omega) + \frac{\sigma}{\varepsilon_0 \omega} = \varepsilon^\ast(\omega) + \frac{\sigma}{\varepsilon_0 \omega} \quad \text{(Eq. 1-2)}
\]
where the subscript d, e, a, MW, represents dipolar, electronic, atomic, and Maxwell-Wagner, respectively. The \( \omega \) is \( 2\pi \) times the frequency, \( \varepsilon_0 \) is the permittivity of free space, and \( \sigma \) is conductivity.

The material's heating ability depends on this effective loss tangent, the higher the loss tangent the higher the heat dissipation. However, the loss tangent is always a function of temperature. It changes with temperature thus increasing the difficulty in heating prediction. In some cases, it is not only a function of temperature but also a function of time of material exposure to the elevated temperature. Therefore, the heating calculations can become very complicated. A general and a useful formula to calculated the average power dissipation (\( P_{ave} \)) in the material under high frequency radiation is [28]

\[
P_{ave} = \omega \varepsilon_0 \varepsilon_{eff} E_{rms}^2 V \quad \text{(Eq. 1-3)}
\]

where \( \omega \) is \( 2\pi f \) (frequency), and \( \varepsilon_0 \) is the permittivity of free space, \( E_{rms} \) is the root mean square value of the electric field strength inside the material, and \( V \) is the volume. This expression includes electric losses only. If a material shows a magnetic loss then the total losses will be:

\[
P_{ave} = \omega \varepsilon_0 \varepsilon_{eff} E_{rms}^2 V + \omega \mu_0 \mu_{eff} H_{rms}^2 V \quad \text{(Eq. 1-4)}
\]

where \( \mu_0 \) is the permeability of free space, \( \mu_{eff} \) is the effective magnetic loss factor, \( H_{rms} \) is the magnetic field strength inside the material. Once the material properties become temperature dependent, then the temperature prediction becomes extremely difficult.
1.3 Literature Review

As mentioned previously, joining of thermoplastics implies the need to raise the sample temperature above $T_g$ for amorphous materials and $T_m$ for semi-crystalline materials. The use of microwave energy is possible if the material can absorb the electromagnetic wave and increase its temperature. For a volumetrically heated material under microwave radiation, the peak temperature always occurs at the center of the parts. It is difficult to control such a system to have peak temperature at the joint interface only. Furthermore, some materials, such as high density polyethylene (HDPE) and polypropylene (PP), are microwave transparent which means they will not absorb microwave energy and thus cannot raise their temperature. Therefore, a microwave sensitive material (absorber) can be used to generate the heat. The concept of using microwave energy in joining of thermoplastics implies the placement of an absorber at the joint interface. Through heat conduction, the molten layers can be created for joining.

Varadan et. el. [29] uses a conducting polymer and chiral microinclusions as microwave absorbers placed at the joint interface to absorb the microwave energy. This chiral material can be implanted into the polymer structure, such as ABS, or can be made as an adhesive film and placed at the joint interface. They also demonstrated the use of a taper microwave applicator to change the characteristic impedance of the waveguide to match the load impedance and have more efficient power transfer. Microwave joining of SiC and alumina ceramics were also reported. Nevertheless, the effect of welding
parameters, such as welding time, pressure, microwave power, and concentration of conductive polymers, on joint strength are not mentioned.

Varadan et. el. [30] made a modification on attaching more polar groups in ABS structure which modified the loss factor at the room temperature resulting in faster heating rate at room temperature. This produces more heating at low temperature and increase the evaporation rate of the solvent that increases the joint strength and reduces the welding time. However, the effect of the welding parameters was not discussed.

P. Kathigamanathan [31] used an intrinsically conducting polymer (ICP), polyaniline, in the form of powder or tape placed at the joint interface for microwave welding. He also showed that conducting polypyrrole can also be used in microwave welding, and it provided better joint strength than polyaniline powders. With either polyethylene or polycarbonate, he was able to achieve the lap joint strengths as high as 19±2 MPa using polypyrrole. However, the effect of welding parameters on joint strength was not studied.

1.4 Objectives

As described in section 1.3, electromagnetic energy can be used in joining of thermoplastics. In addition, intrinsically conducting polymers can also be used in joining of thermoplastics. However, the research in using ICP and microwave welding is limited. Therefore, the main objective of this study is to develop a systematic study on microwave welding of thermoplastics using ICP. The effect of welding parameters on joint strength
will be studied on both multi-mode and single mode microwave systems. These parameters include heating time, pressuring methods, welding pressure, concentration of ICP in heating composite, and microwave power. In addition, the equivalent circuit model will be constructed to estimate the initial heat generation rate of the ICP composites. Furthermore, feasibility study on electromagnetic heating, such as resistance heating, induction heating and radio frequency heating, will be explored to evaluate the possibility of those welding methods using ICP. The final goals are to understand the microwave welding using ICP and ICP for future development in large scale welding.
CHAPTER 2

CONDUCTING POLYANILINE IN JOINING

2.1 Introduction

During the past 20 years, a new class of electrically conductive polymers, such as polythiophene, polypyrrole and polyaniline, have been intensively studied. These materials have a unique combination of mechanical and electrical properties making them very useful for welding. Furthermore, they provide an opportunity for developing new joining techniques for assembling large structures. In this study, intrinsically conductive polyaniline was chosen because it is relatively inexpensive, it is easy to synthesize and process, and it is stable at room temperature [32]. Until recently, most applications of polyaniline have focused on its insulator-to-metal transition properties, such as for rechargeable batteries, integrated circuits [33], electrolytic capacitors, sensors and color-changing windows [34]. It can also be used for electromagnetic shielding [35]. For plastic joining, intrinsically conductive polyaniline was successfully used in radio frequency welding [36].
Like in semiconductors, polyaniline conducts electricity through doping, that creates partially filled bands through which free moving electrons conduct electricity. The electrical properties of polyaniline are a function of frequency, temperature, morphology and doping level. For example, microwave conductivity is higher than DC conductivity at low doping levels [37]. In the temperature range of 600K to 3000K, the electrical conductivity increases with increasing temperature [38]. Increasing the doping level increases the conductivity, and changing the type of dopant used changes the conductivity [39]. The molecular structure or morphology of polyaniline can also affect its conductivity and loss tangent. Stretched films or fibers that have a higher level of crystallinity have a higher electrical conductivity than unstretched films and powders. The electrical conductivity of polyaniline can be varied from that of an insulator (10^-10 s/cm) to that of a conductor (10^2 s/cm), depending on the processing techniques.

2.2 Polyaniline

Polyaniline has been known as a material for more than 150 years; however, its application was restricted to the textile industry as a dye. It was not until 20 years ago when researchers found out that some polymers can increase their conductivity by 10 orders of magnitude through a doping process, that other applications were developed. For example, polyacetylene can increase its conductivity from ~ 10^-10 to 10^5 s/cm when doped with iodine [40,41]. Polyaniline is also one of the conducting polymers changing its
conductivity dramatically upon doping by acid. Doping is a chemical process that adds or removes electrons in the structure. Doping of polyaniline is different in that the total number of electrons remains constant on the polymer backbone but the number of protons is increased; thus, protonation is used in doping polyaniline. Before doping, polyaniline has three different forms, the fully oxidized pernigraniline base (PEB), the fully reduced leucoemeraldine (LEB), and the half oxidized emeraldine base (EB) as shown in figure 2 [42]. The conducting form of polyaniline is defined as emeraldine salt (ES). Polyaniline contains benzenoid rings, quinoid rings, and nitrogen atoms. The nitrogen has five valence electrons that occupy in \( sp^2p_z \) orbital and there are two types of bonding among them. First, the amine nitrogen, -NH-, has a lone pair in the \( p_z \) orbital that is perpendicular to nitrogen plane. Second, an imine nitrogen, -N=, with a pair of electrons in a \( \sigma \) lobe in nitrogen plane and one electron in \( p_z \) orbital. When doped with HCl, a \( \sigma \) bond was formed by proton (H\(^+\)) and nitrogen atom from the imine site, and the counter ion C\(^-\) will be around the proton to neutralize the charge. This proton is the key for de-localization. After the protonation, internal redox reactions occur that change the quinoid ring to benzenoid followed by polaron separation. The transformation from non-conducting to conducting form is shown in figure 3 [43]. The alternative way of doping EB to ES is by changing the counter ion [44], such as when using camphorsulfonicacid as a dopant. An alternative way of doping is that the counter ion was covalently bonded to the backbone using sulphuric acid which provides better heat resistance but lower conductivity [45].
Figure 2: Three oxidation states of polyaniline, Leucoemeraldine base (a), Emeraldine base (b), and Pernigraniline base (c) [42]
Figure 3: Transformation of polyaniline from non-conducting to conducting; Emeraldine base (a), σ bonds at imine sites, A bipolaron form, and Polaron separation (d) [43]
There are thousands of papers regarding the chemical, physical, and electrical properties of polyaniline. However, HCl doped polyaniline was the first conducting polymer reported in its family. Thus, HCl doped polyaniline will be the primary material utilized in this study.

2.3 Synthesis Procedure for Polyaniline

There are two major methods to synthesize the conducting form of polyaniline, chemical oxidation and electrochemical oxidation. Chemical syntheses will be used due to simplicity. The polymerization of aniline \((C_6H_5)NH_2\), is carried out by the oxidation agent, ammonium peroxydisulfate \((NH_4)_2S_2O_8\), in hydrochloric acid. The following is the procedure for making conducting polyaniline [46]:

1) Dissolve 11.5 gram, 0.0504 mole of ammonium peroxydisulfate in 200 cc. 1M HCl at 1°C in a 400 cc beaker

2) Dissolve 20 cc. 0.219 mole of aniline solution in 300 cc. 1M HCl at 1°C in a 750 cc. Erlenmeyer flask with magnetic stirring bar in the ice bath.

3) The ammonium peroxydisulfate solution is gradually added into the aniline solution using a small pipette. This procedure takes 45 minutes to 1 hour to finish. The solution is stirred while adding ammonium peroxydisulfate. After 3-5 minutes, the solution changes its color from blue-green tint and becomes blue-green with a coppery glint. The precipitates begin to form at this time.

4) The above solution is kept stirring for a total of 1 to 2 hours. The temperature is kept below 5°C.
5) After stirring, the precipitation is collected in a Bucher funnel (7.5 cm in diameter) using a water aspirator. The precipitation "cake" is washed with 600 cc. 1M HCl. During washing, the HCl level in Bucher funnel must remain at the top surface of the cake to prevent cracking of the cake. The purpose of washing is to remove un-reacted chemicals.

6) After washing, leave the cake in the funnel and continue aspirating for 10 to 15 minutes. This will remove the HCl between the precipitation particles. At this stage the cake has (Cl/N) = 42%. It is not the highest conductivity level at this stage; further stirring is needed to convert the 42% cake to 50%, i.e., fully doped.

7) Remove above precipitation cake and transfer the cake into a flask and add 500cc. 1M HCl for another 15 hours stirring at room temperature. Usually, 4 to 5 grams of conducting polyaniline can be obtained.

8) After 15 hours stirring, repeat steps 5 and 6. The cake is now the fully doped conducting polyaniline.

9) After a second washing, the cake is transferred to a vacuum oven for drying to remove the residual HCl between conducting particles. The temperature for drying should be kept below 50°C to keep from overheating the cake which will reduce the conductivity of the polyaniline. The length of the vacuuming time reduces with increasing oven temperature. For example, at room temperature, the vacuuming time is 48 hours; at 50°C, it only takes 5 hours.
10) The final step is to grind the conducting polyaniline into a fine powder using a mortar and pestle. After finishing this step, the conducting polyaniline (PANI) is ready for use.

At this stage the HCl doped polyaniline is obtained. Furthermore, the doping acid can be modified to acquire different material properties other dopants include H$_2$SO$_4$ and HSO$_3^−$. The doping process cannot be performed as previously described. In general, HCl doped polyaniline (ES) has to be obtained first, than de-dope ES to EB. Using EB then choose the favorable acid to dope EB into the desired conducting polyaniline. To de-dope the conducting polyaniline to EB a 28%, NH$_4$OH solution is used. Therefore, replacing the HCl with NH$_4$OH in step (7) to step (10), EB can then be obtained for further processing.

2.4 Conducting Heating Element

The concept of using conducting polyaniline in welding is by placing polyaniline or a polyaniline composite at the joint interface under electromagnetic radiation. Of course, this composite can have as little as 5 to 100% PANI concentration. Due to the fact that polyaniline cannot be melted for processing, it is not easy in handling the powder during welding. Therefore, a composite has to be made for handling purposes. One criterion for the polymeric matrix is that it cannot absorb electromagnetic energy. In such a way, the effect of polyaniline under electromagnetic radiation can be studied. Thus, HDPE was chosen for the matrix material. Unfortunately, this composite is not commercially available in the market; thus, making the composite heating element needs to be studied. There are
several ways to fabricate the composite in the laboratory, such as compression molding, ultrasonic molding, and microwave molding.

2.4.1 Compression Molding

The simplicity of compression molding makes it attractive for fabricating the composite gasket. A stainless steel cylindrical mold with 1.25” in diameter is used for molding. The amount of powder in the mold determines the heating element (gasket) thickness. By calculating the volume of heating composite, the amount of powder can be estimated. For example, a total of 0.4 grams of HDPE powder in the mold results in 0.45mm thick gasket, 0.8 grams of powder results in 0.9mm thick gasket. Figure 4 shows the experimental setup for compression molding. The temperature of the press was controlled by 4 individual heaters, two heaters were installed in the bottom plate and the other two were installed in the top plate. A one inch thick aluminum block was placed between the mold and the bottom plate which gave a more uniform temperature distribution in the mold. A thermocouple was placed beneath the mold to monitor the temperature. The final molding time and temperature were determined by a series of experimental trials that provides superior molding quality with the lowest molding temperature. Figure 5 shows the molding temperature history during heating and cooling. The compression force was controlled at 1500 lb. at beginning, due to thermal expansion and by keeping the gaps between the plates constant, the force increased to 2200 lb. at the
Figure 4: Schematic representation of compression molding system
Figure 5: Temperature history during compression molding
end of heating. By the time when the polymer melts and gets squeezed, the pressure drops back to about 800 lb. After determining the molding parameters, the conducting powder was ground and mixed with HDPE powder using a mortar and pestle. The concentration of polyaniline in the conducting gasket can be changed from as low as 5% to as high as 60% by weight. A 70% PANI loading was also attempted; however, due to low matrix concentration the gasket was very brittle. After molding, the gasket was sliced into 6.35 by 6.35 mm squares for heating studies. Due to the presence of hydrochloride acid in polyaniline, corrosion occurs on the mold that results in damaging the mold and increases the difficulty in removing the gasket from the mold. Therefore, light polishing was required and mold release was also applied every 4 to 6 moldings.

2.4.2 Ultrasonic Molding

Ultrasonic molding has been successfully used in molding of UHMWPE [47]. Therefore, it is possible to mold the HDPE + PANI composite. This prototype ultrasonic molder offers 32000 N force on the sample using a combination of pneumatic and hydraulic 2.5” bore diameter OHMA cylinder. A precision die set with four precision guide pins was used to hold the ultrasonic converter, booster, and horn that provides good contact between horn and sample. A circular horn with the same diameter (1.25”) as compression molding was used. It was found that large clearance between horn and mold cavity reduced the molding quality. Therefore, the mold was 0.025mm largerer than the horn to prevent too much squeeze of the molten material. An LVDT was installed to
monitor the collapse during molding. Figure 6 shows the photograph of the ultrasonic molder. Conventional ultrasonic welders trigger the ultrasonic vibration at the time when the horn makes contact with the sample. In the prototype molder, the pneumatic system was separated from the ultrasonic system. The horn came down making contact with the powder at a very large pressure which ensures that the powder was compacted together, and then the ultrasonic vibration was activated. The molding time is short depending on the booster used, for example, 0.8 gram HDPE with the 1:2.5 booster takes 2 seconds, but with 1:1.5 booster it takes 5 seconds.

The results of the ultrasonic molding were not satisfactory because the molding quality was not consistent enough and the sample was not flexible enough. For a thicker sample, such as 1mm thick, the variation was smaller, but for a thin sample such as 0.5mm thick, the heating was not uniformed. In most cases, the sample surfaces which contacted with the horn and the bottom plate attained higher temperatures than the middle sections. Even with longer molding time, the HDPE power at the middle section was still not melted completely. Another drawback of ultrasonic molding is that the temperature distribution along the radius direction was not uniform either. The temperature was higher at the edge of the sample; however, the center of the sample was still below the melting temperature.
Figure 6: Photograph of prototype ultrasonic molding machine
2.4.3 Microwave Molding

As mentioned before, the microwave absorption depends on the effective loss tangent which includes the conductivity term; hence, a conducting polymer has the ability to absorb the microwave. By that reason, it is possible to use microwave energy as a heating source to mold the conducting composite. Due to the small skin depth of metal at microwave frequency, no microwave can penetrate into the stainless steel mold and heat up the polyaniline. A Teflon mold was made which has the same dimension as the mold used in the compression molding. A total of 0.4 gram composite with 50% and 60% PANI loading was used. Pressure was applied by wrapping the rubber bands on the mold. A domestic microwave oven (Toshiba, 600Watts) with rotation table was used as heating source. The heating time was on the order of 10 to 20 seconds.

The results of the microwave molding were similar to ultrasonic molding. Inconsistency was always the problem, the high temperature regularly occurred at the edge of the gasket for a short heating time. The temperature distribution through thickness direction did not exhibit temperature differences like ultrasonic molding. Due to the fact that the mold was rotating inside the microwave, pressure was difficult to apply and the temperature of the sample was difficult to measure. Therefore, the quality of the composite was hard to control. Besides, raise the temperature during molding reduces the conductivity of PANI powder which results in reducing the heating ability of the composite during welding.
From the above three molding methods, compression molding was selected as the gasket making method for welding studies. Although it is slow and time consuming, it does provide the best quality and consistency.
CHAPTER 3

FEASIBILITY STUDY ON CONDUCTIVE ELEMENT HEATING

3.1 Introduction

Once the gaskets were made by compression molding, the next step was to determine which electromagnetic heating method will provide the best heating. The electromagnetic heating methods that were studied included resistance heating, induction heating, radio frequency heating and microwave heating. The methodology in this study was to evaluate the adiabatic heating of the conducting element.

3.2 Resistance Heating

Resistance heating is a simple but efficient method. The heat generation follows Joule's Law that is $P = I^2R$, where $P$ is the power generation in Watts, $I$ is the current in amp., and $R$ is the resistance of the heating element in ohms ($\Omega$). The power supply used in resistance heating was a STACO AC variable transformer. The voltage could be varied from 0 to 140 volts. Direct electrode contact method was used to heat the composite.
Two multi-meters; one in parallel connection and one in series connection to the composite; and one digital thermometer were used to measure, voltage, current, and temperature, respectively, as shown in figure 7. A small voltage was applied at the beginning of the heating, then higher voltage was applied. The heating parameters such as voltage, current, temperatures and heating time were recorded manually.

Resistance heating of a 10mm in diameter and 1.5 mm thick dry pressed pure polyaniline was studied first. The $\text{H}_2\text{SO}_4$ doped polyaniline sample could be heated to 100°C in 15 seconds by using 5 volts DC input. However, the resistance of the sample changed from 10 ohms to 140 ohms after heating. The temperature could not be raised above 100°C because of the increase in resistance. This resistance changes mainly because of the increase in contact resistance between the electrode and the sample and the reduction in conductivity of the sample. The increase in contact resistance results from localized burning under the electrodes. The reduction in conductivity results from loss of dopant. Therefore, the power dissipation in the sample was not enough to raise the temperature to acceptable welding conditions. When applying 110 volts AC 60 Hz to the sample, it exploded and burned immediately. After the explosion the resistance decreased to 3 ohms, which may caused by carbonization.

Heating of a $\text{HSO}_3^-$ doped pure polyaniline disk (10 mm in diameter, 1.5mm thick) was also studied by using 20 volts AC, 60 Hz. The disk temperature could be raised to 80°C in 23 seconds. The temperature was measured by attaching the thermocouple to the sample surface. It is anticipated that a higher voltage input will reduce the heating time.
Figure 7: Experimental set-up for resistance heating of conducting composite
and reach the same temperature. Therefore, by increasing the AC power to 30 volts, the sample reached 80°C in 8 seconds and the process was repeatable. With further increases in the voltage, the sample exploded and burned. The above studies show that the maximum current density which polyaniline can accommodate is limited. Increasing the current does not provide an advantage in heating. Similar results were found with HCl doped polyaniline disks.

Resistance heating of 0.5mm thick, 35mm long, 60% polyaniline concentration and 40% HDPE composites was also studied. The experimental setup was the same as described above. A typical temperature-voltage vs. time diagram is shown in figure 8 in which an AC 12.55 volts supply was used. When heating begins, the temperature increase as time increases. After 100 seconds of heating, the resistance increases i.e. conductivity decreases, the gasket was kept at the same temperature. Figure 9 shows the change in temperature, resistance, and voltage with time using a 0.5mm thick 60% HCl doped polyaniline composite. Raising the voltage at the point when the resistance begins increasing results in a rapid drop in the resistance as new conducting paths are established in the composite. Using this approach the sample can be heated to a temperature of 100°C at which point the resistance becomes too high to produce additional heating. Based on this, it appears that a ramp voltage input would provide fast heating. Some localized melting and fusion between the composite and the backing HDPE plate did occur.
Figure 8: Typical voltage and temperature curves during resistance heating of conducting composite
Resistance Heating of 60% 0.45mm PANI Composite

Figure 9: Typical resistance, temperature and voltage curves during resistance heating of conducting composite
Experiments using resistance welding of HDPE/PANI composite bars (53.2 x 5.32 x 3.16 mm) were performed with modest success. The samples were held by the plastic sample holder, an AC power source (100 volts) was used to generate the current flow. The constant pressure was applied during whole welding process. The averaged joint strength was 82% of the molded composite strength for 50% PANI composite as shown in figure 10.

3.3 Induction Heating

The equipment used in induction heating was a 9 to 110 KHz, ENI model EGR 1600B variable frequency power generator and EIB-3A with MT2 induction heating transformer with 0.25" outside diameter copper tube heating coil. The maximum power output was 1600 watts. Figure 11 shows the experimental setup for the ENI induction system. The heating composite was placed under the coil. For high frequency induction, a Lepel 4 MHz and 5000 Watts generator with a pancake coil was used at The Edison Welding Institute.

The results of induction heating of HCl doped and H₂SO₄ doped 100% polyaniline disk were successful at 4MHz. The temperature rise was high enough to melt the low density polyethylene film which surrounded the sample in 60 seconds. At low frequencies, it was not possible to heat polyaniline even for very long heating time. This is probably due to the high electrical resistance of the sample. Heating of the 60% PANI composites
Figure 10: Results of resistance welding of PANI/HDPE composite
Figure 11: Photograph of the induction heating machine
was not possible at neither 4 MHz nor 9 KHz - 110 KHz, due to the high resistance of the composites.

3.4 Radio Frequency Heating

The radio frequency machine operates at 27.12 MHz with 2000 watts of power and is manufactured by Callana (model 20SB). The major heating principle for the polymeric material is the rotation of the polar molecules along with the alternating electric fields. Table 1 shows the weldability of some polymers [48]. However, the HCl doped polyaniline and HDPE contain no polar groups in their structure; at first it seems that the PANI composite cannot be heated using radio frequency welder. As mentioned in section 1.2, the heating ability depends on the effective loss tangent of the material and by reviewing equation (1.1) and (1.2), it can be seen that the loss tangent includes the conductivity term. Due to the fact that the PANI has a finite conductivity; therefore, it can be heated using radio frequency. For a low conductivity material, like PANI composite the conductivity increases with increasing frequency. Therefore, increasing the heating frequency to 27.12 MHz may result in higher heat generation in the composite as compared to induction heating. Figure 12 shows the experimental set-up for radio frequency heating. The top electrode was subjected to an alternating voltage at 1500 volt rms while the bottom electrode was grounded. The heating composite was placed between
<table>
<thead>
<tr>
<th>Material</th>
<th>Sealability</th>
<th>Material</th>
<th>Sealability</th>
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</thead>
<tbody>
<tr>
<td>ABS</td>
<td>G</td>
<td>Polycarbonate</td>
<td>P</td>
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<tr>
<td>Acetal (Delrin)</td>
<td>P</td>
<td>Polystyrene</td>
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<tr>
<td>Acrylics</td>
<td>F</td>
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<td>G</td>
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<tr>
<td>Cellulose Acetate</td>
<td>G</td>
<td>Polyvinyl Chloride</td>
<td></td>
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<tr>
<td>Cellulose Acetate Butyrate</td>
<td>G</td>
<td>Flexible, Clear</td>
<td>E</td>
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<td>E</td>
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<td>Cellulose Triacetate</td>
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<td>Semi-rigid</td>
<td>G</td>
</tr>
<tr>
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<td>Rigid</td>
<td>F</td>
</tr>
<tr>
<td>Nylon</td>
<td>G-P</td>
<td>With glass scrim</td>
<td>E</td>
</tr>
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<td>Coated paper and cloth</td>
<td>E</td>
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<tr>
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<td>Adhesive emulsions</td>
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<td>Rubber</td>
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<td>Saran (Polyvinylidene Chloride)</td>
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<tr>
<td>Polypropylene</td>
<td>NO</td>
<td>Silicon</td>
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<tr>
<td></td>
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<td>Teflon (Tetrafluoroethylene)</td>
<td>NO</td>
</tr>
</tbody>
</table>

Code for Sealability: E = Excellent; G = Good; F = Fair.

Table 1: Sealing ability of materials at radio frequency [48]
Figure 12: Schematic representation of radio frequency heating experiments with sample in vertical orientation
the electrodes. The separation of the electrodes determines the electric field strength during heating. As illustrated in equation (1.3), the power generation is proportional to the square of the electric field inside the material. Therefore, decreasing the distance between the electrodes results in increasing the electric field and the power generation in the material. It is noted that $E_{ms}$ in equation (1.3) represents the electric field strength inside the material; however, increasing the electric field strength between the electrodes results in increasing the $E_{mu}$. It is found that if the distance between sample and electrode is too small, arcing occurs. Therefore, a 12.7 mm (0.5") gap between the electrodes was set for adiabatic heating of the 6.35 x 6.35 mm PANI composite. A fluoroptic thermometer with fiber optical probe was used to measure the temperature. The probe not only measures the temperature during heating but also serves as a sample holder. The temperature data was collected by a personal computer through RS232. There are two possibilities in placing the sample between the electrodes. One in vertical orientation as shown in figure 12 and another in horizontal orientation. The effect of orientation on heating and the effect of PANI concentration in the composite were evaluated.

Figure 13 shows the effect of orientation on heating. The samples used in this study were 60% PANI composites which had the dimension 6.35 x 6.35 x 0.5 mm. The sample used in radio frequency heating were all with these dimensions. It was found that the vertically orientated sample had a faster heating rate than the horizontally orientated sample which means that the sample must be placed in parallel with the electric field.
Figure 13: Effect of conducting composite orientation on radio frequency heating
direction. This can be explained by using the boundary conditions of electromagnetic waves in which the normal displacement current is continuous and tangential electric field is continuous. For vertical aligned sample, the electric field strength is higher than the horizontal aligned sample due to the conducting composites having low conductivity and high dielectric constant. Therefore, the vertical orientation will be used in the following studies. This result was in good agreement with Faisst’s work [13]. Figure 14 shows the effect of electrode separation on heating; the samples used in this study were 60% PANI composite. It is clear that the smaller the electrode separation the higher the heat generation because of the higher electric field strength existing between the electrodes. Therefore, it is advantageous to reduce the electrode separation during welding.

Figure 15 shows the effect of the PANI concentration on heating. The sample used were 25%, 30%, 40% and 50% PANI loading composites. The electrodes separation was 12.7 mm. It was found that a higher concentration composite does not provide the higher temperatures. The best heating sample was 40% PANI loading sample which raised its temperature to almost 250°C in 30 seconds. The 50% PANI loaded sample exhibited the same heating pattern as 30% PANI loaded sample. This result is similar to Faisst’s work; however, in his work, 40% and 50% gasket had same heating rate and temperatures and 60% gasket resulted in lower temperature. The reason is that the electric field strength inside the material depends on the dielectric constant and the conductivity of the material. For example, for metal which has a very high conductivity the internal electric field is close
Figure 14: Effect of electrodes separation on radio frequency heating
Figure 15: Effect of polyaniline concentration on radio frequency heating
to zero when exposed to a radio frequency field. As a matter of fact, aluminum foil was placed inside the machine and resulted in no temperature changes after 30 seconds of heating. Therefore, a higher PANI loading in the composite results in a lower electric field strength inside; hence, lower temperature. On the other hand, a low PANI loading composite has a very low conductivity which also results in low temperature since the heat generation rate is \( \dot{Q} = \sigma E_{\text{rms}}^2 \) (in watts/m\(^3\)). Therefore, there exists an optimal PANI concentration in the composite for radio frequency heating.

3.5 Microwave Heating

The microwave heating principle is similar to the radio frequency heating except it occurs at a much higher frequency. Equation (1.4) can be used to calculate the power absorption. The microwave used in this study was a domestic microwave oven, Toshiba ERS-5740B with a rotation table and digital timer, which delivers 600 watts of power. Microwave heating was first tested by making a sandwich using a 60% PANI composite and polycarbonate plates. The sample was placed at the center of the rotation table and after 60 seconds of heating, the polycarbonate plates were melted and formed the joint. However, the joint strength was low due to having no squeeze flow. Therefore, more detailed studies were performed.

Due to the fact that the microwave oven is a multi-mode cavity, the field patterns are too complicated to analyze mathematically; however, finding the hot spot for microwave heating was needed. There are several ways to find the hot spot inside the
microwave oven. The simplest one is by placing a paper and watching the color change after heating. The hot spots exhibit a brown color due to over heating the paper. The second way to examine the field pattern is by putting many small water blocks inside the cavity, after heating the water blocks, temperature measurement can be used to determine the hot spot location. However, these methods are only good for determine the location of the hot spot without the field directions. As demonstrated in section (3.4), the heat generation of the PANI composite was affected by the orientation of the sample relative to the electric field direction. Therefore, direct heating of the composite is the best way to determine the heating ability of the PANI composite. By using Luxtron fluoroptic thermometer and a temperature probe as sample holder, the location and orientation of the hot spot was determined. A quarter inch hole was drilled on the cavity wall to insert the temperature probe into the microwave oven. The probe cannot reach all places inside the cavity due to the flexibility of the probe; thus, only the places where potential high electric fields might exist were tested such as the center of the cavity, and the surrounding area of microwave outlet. The low electric field locations were ignored such as cavity walls, corners of the cavity. It was found that the area located under the microwave source has the best heating efficiency. Figure 16 shows the best heating location in a multi-mode microwave oven. Therefore, the composite heating studies were done this location. The composites used in multi-mode microwave heating were all PANI and HDPE powder composites of 6.35 x 6.35 x 0.45 mm, unless otherwise noted.
Figure 16: Schematic representation of the hot spot in a multi-mode microwave oven
Figure 17 shows the adiabatic heating results for a 50% 0.45mm thick PANI composite. The sample temperature rise exceeds 280°C in 30 seconds which is enough to melt most polymers. The drop in temperature after the peak temperature should be associated with the changes in conductivity of the composite at elevated temperatures. Figure 18 shows the heating result of a 0.9mm thick 50% PANI composite. The peak temperature is a little lower than the 0.45mm gasket but the drop after the peak temperature is smaller to the 0.45mm sample. The temperature remains constant at 220°C after 180 seconds of heating. Figure 19 shows the heating of a 60% 0.45mm thick PANI composite. As expected, it reaches a higher temperature than the 50% PANI composites and also retains the temperature at 220°C. Therefore, both 50% and 60% PANI composites are suitable in microwave welding of thermoplastics. It was found that there existed no optimal PANI concentration and higher PANI loading gave better heating. When the PANI composite was subjected to a cyclic microwave radiation, it followed the re-heating even after a 5 minute period as shown in figure 20. This provides the opportunity for applications where heating is needed repeatedly, such as assembly and disassembly of the joint.

The results of the multi-mode microwave heating of PANI composite were very promising. It is the best among the methods studied; therefore, the follow-on work focused on microwave welding using PANI composites.
Figure 17: Adiabatic heating result using 50% PANI, 0.45mm thick composite using multi-mode microwave oven
Figure 18: Adiabatic heating result using 50% PANI, 0.9mm thick composite using multi-mode microwave oven
Figure 19: Adiabatic heating result using 60% PANI, 0.45mm thick composite using multi-mode microwave oven
Figure 20: Temperature histories of 50% PANI, 0.45mm thick composite with cyclic microwave radiation using multi-mode microwave oven.
CHAPTER 4

MULTI-MODE MICROWAVE WELDING

4.1 Introduction

The most widely used microwave oven is a multi-mode microwave cavity, an example of such a device is the domestic microwave oven. The dimensions, operating frequency, and dielectric constant of the material inside the cavity determine the number of modes. Due to FCC restrictions, the operating frequency was fixed at 2.45 GHz. Therefore, the dimensions of the cavity determines the number of modes in the empty cavity. When the cavity is partially filled with some irregularly shaped dielectric material, the number of modes and wave pattern becomes very complicated and difficult to analyzed. Therefore, field analysis will be excluded in this study.

A Toshiba domestic microwave oven (ES-5740B) was used in multi-mode microwave welding. The microwave power source was a magnetron tube. A magnetron is basically a microwave vacuum resonator, one example is given in figure 21[49]. It contains a hollow cylindrical anode with a cathode which was heated at the center of the structure. The slots between the anode and the cathode perform as the resonant cavities
Figure 21: Schematic representation of a typical magnetron cavity [49]
which are separated by vanes. A constant voltage drop is applied between anode and cathode to create an attraction force for the electrons released from the heated cathode. A magnetic field is also applied parallel to the cathode axis. Due to the Lorentz's Law, \( F = q (v \times B) \), where \( F \) is the force, \( q \) is the charge, \( v \) is velocity, and \( B \) is magnetic field strength, a circular force around the cathode was created on the moving electrons. The electron cloud is formed by these moving electrons. The length of the vane is a quarter wave length. During resonance, the electric field strength at vane walls is zero; however, by transmission line theory, the open tips will have a maximum electric field strength. Due to the \( \pi \) mode resonance, the adjacent cavity has a phase difference of 180\(^\circ\); thus, a strong electric field occur between the vanes. These fields are altered respect to time between vanes as shown in figure 21 that look likes an induced microwave field. Therefore, electrons clouds are decelerated by the opposite direction to the electric field and they fall to the anodes. On the other hand, if the field is the same as the electron motion direction, electrons are accelerated toward the cathode until they decelerate and give up their energy to the induced microwave field [50]. The microwave energy can be coupled out by coaxial cable or by waveguide. These microwaves were guided in a banded waveguide to the microwave chamber (280 x 280 x 216mm).

Once the waves are introduced into the oven cavity; they propagate in every directions and interfere with each other. The field is too complex to analyze; however, the design principle is to have the field as uniform as possible. Because the heating composite was made by HDPE and PANI, welding of HDPE bars will be studied first.
4.2 Welding of HDPE - Constant Pressure

4.2.1 Experimental Preparation

The parameters which affect the final joint strength are PANI doping level, %PANI in the gasket, welding time, welding pressure and gasket thickness. The PANI doping level affects the intrinsic conductivity and loss tangent of the polyaniline powder which affects the properties of the composite gasket. The effect of doping level was excluded in this study. The effect of process parameters such as welding time and welding pressure on joint strength were studied. Moreover, material parameters such as the effects of gasket thickness and percentage of polyaniline in the gasket on joint strength were also studied.

Two 50.8mm long and 6.35mm by 6.35mm cross section HDPE bars were butt joined. The gasket was placed at the joint interface and the parts were placed in a special fixture and they were wrapped with rubber bands to apply the pressure as shown in figure 22. The fixture was then placed at the center of the rotation table in the microwave oven for welding. The weight of the thinner gasket (0.5mm in thickness) was 0.023±0.002g. The thicker gasket (1mm thickness) had twice the weight 0.046±0.002g. The welding time was digitally controlled. The effects of welding time, gasket thickness and welding pressure on joint strength were studied with the 50%PANI gasket. The 60%PANI gasket was used to study the effect of %PANI on joint strength.

The tensile strength of the polymeric composite gasket and HDPE powder must be determined before welding. This way the weak link can be found. Figure 23 shows the
Figure 22: Photograph of the sample-fixture assembly for a multi-mode microwave welding - constant pressure
Figure 23: Strength of compression molded PANI/HDPE composite
tensile strength of the composite for various weight percentages of polyaniline. The samples were made by compression molding in a 53.33mm x 5.33mm x 3mm rectangular mold under the condition shown in Figure 5 with a pressure of 18.76±3.13 MPa. The samples weighed 1g before molding. Tensile tests were performed after molding in an Instron model 4201 machine. The rectangular parts were machined into the ASTM D638-90 dumbbell shape (except the overall dimensions of these sample were smaller than the standard ASTM D638-90 samples, see figure 24) in order to insure failure in the gage region. Parts molded from pure HDPE powder had an average strength of 22.60±0.62 MPa. The strength of 50% and 60% PANI composites was 12.34±1.41 MPa and 10.71±1.23 MPa respectively. At first glance it appears that the maximum joint strength cannot exceed the strength of the composite. However, in reality the joint strength exceeded the composite strength for reasons which will be discussed shortly.

4.2.2 Results and Discussion

Figure 25 shows the effect of welding time on joint strength. The gasket used here was 50%PANI with 0.5mm thickness and a welding pressure of 0.30 MPa was applied during heating and cooling. As shown in Figure 25, longer welding times resulted in stronger joints. The average strength of 50%PAN gasket was only 12.34 MPa which was close to the joint strength made at 120 seconds. For welding times longer than 120 seconds, all the samples had better joint strength than the strength of the gasket. This is because the molten gasket breaks under pressure and squeezes out from the interface.
Figure 24: Photograph of a reduced ASTM-D638 sample Left: compression molded composite, Right: microwave welding sample
50%-0.5mm PANI Composite, 0.3MPa joint Pressure

![Graph showing the effect of welding time using 50% PANI, 0.5mm thick composite with 0.3 MPa joining pressure using multi-mode, constant pressure.]

*Figure 25: Effect of welding time using 50% PANI, 0.5mm thick composite with 0.3 MPa joining pressure using multi-mode, constant pressure*
resulting in direct contact and intermolecular diffusion between the two HDPE bars. These HDPE bars are stronger than the composite gaskets which results in higher joint strength.

The effect of percentage of PANI on joint strength is shown in Figure 26. In this case, increasing the PANI concentration improves the joint strength. The 60% PANI gasket contains more PANI powder than 50% PANI gasket of the same thickness which results in different electrical properties. Therefore with the 60% PANI gasket more heat was generated at the interface. This results in a larger molten layer and creates more flow. Therefore, it results in faster heating and squeezes out more of the gasket from the interface. As mentioned before more squeeze out of the gasket results in stronger joints.

Increasing the gasket thickness increases the PANI content in the gasket which results in more heating using constant pressuring method. Figure 27 shows the effect of gasket thickness on joining strength. Both samples had the same 50%PANI in the gasket and the same welding pressure. As shown in Figure 27, the parts with 1mm thick gaskets had stronger joints than the ones with 0.5mm thick gasket. Since more heat generation occurs in the 1mm gasket under constant pressure, it increases the thickness of the molten layer resulting in greater flow. This transverse flow can drive more gasket out of the interface and increase the contact area between the HDPE bars. Figure 28 shows enlarged photographs of welds with an intact gasket (a) and with a squeezed out gasket (b).
Figure 26: Effect of PANI concentration on joint strength using 0.5mm thick gasket with 0.3MPa joint pressure using multi-mode and constant pressuring method
Figure 27: Effect of gasket thickness on joint strength using 50% PANI, gasket with 0.3MPa joint pressure using multi-mode and constant pressuring method.
Figure 28: Photograph of microwave welded joints, intact gasket (a), and squeezed out gasket (b).
The effect of welding pressure on joining strength is shown in Figure 29. Both samples were 50% PANI and 0.5 mm in thickness. Figure 29 shows that higher pressure results in higher joint strength and it also shortens the welding time needed to reach a certain strength. The welding pressure was estimated from the force required to pull the rubber bands in the tensile testing machine to the same elongation as experienced in welding. In reality, the rubber bands will absorb some of the microwave energy and become softer which would reduce the applied pressure. Therefore, the welding pressure is probably lower than indicated here.

4.2.3 Summary

A novel technique for joining high density polyethylene using microwaves was developed. Welding of HDPE was done by placing these gaskets at the joint interface. Longer welding times resulted in stronger joints. The higher the PANI loading in the gasket, the faster and stronger the welds became. The joint strength using 60% PANI gasket with a weld time of 120 seconds and pressure of 0.3 MPa was 19.43±0.77 MPa, which was 86% of the strength of the molded HDPE bar. For 1 mm thick gasket with 50% PANI, the joint strength for a weld time of 80 seconds and a pressure of 0.3 MPa was 19.42±0.47 MPa which was also 86% of the strength of the molded HDPE bar. Doubling the pressure increases the joint strength. Pressure will be an important parameter for future study. To achieve the maximum joint strength, one should squeeze
Figure 29: Effect of welding pressure on joint strength using 50% PANI, 0.5mm thick gasket using multi-mode and constant pressuring method.
out most of the gasket from the interface. Based on this work, microwave welding of plastics using conductive polymers appears to have a promising future. The next step is to modify the welding technique to achieve better joint quality.

4.3 Welding of HDPE - Post Heating Pressure

4.3.1 Experimental Aperture

The experiments made in previous section indicted that welding of HDPE using conducting composites is possible. It also reveals that increasing the joining pressure improves joint strength. However, it is difficult to apply pressure on a rotating subject; therefore, a new design is required. As discussed in section 3.5, the best heating location was near the microwave power source. Therefore, a new fixture which raise the sample was designed and built and the method of pressure application was also modified to improve the joint quality. Figure 30 shows a drawing of the newly designed fixture which holds the original sample holder and lifts the sample to the microwave outlet. Pressure method was applied by using an air cylinder outside the microwave oven as shown in figure 31. The pressure was activated manually at the end of welding. An extension bar was used between the sample and air cylinder to transfer the pressure. An LVDT was also installed below the air cylinder to monitor the displacement during pressure stage. A Metrabyte DAS-20 data acquisition system was used to collect the displacement data with 200 points/seconds sampling rate. The data acquisition program is listed in Appendix A.
Figure 30: Schematic representation of a new fixture for multi-mode microwave welding.
Figure 31: Photograph of an air cylinder and an LVDT for multi-mode microwave welding
4.3.2 Heat Generation Rate

4.3.2.1 Initial Heat Generation Rate - PANI Composite

Since the sample was raised to 25mm below the microwave power source in the oven, adiabatic heating tests were used to evaluate the new fixture design. Figure 32 shows the temperature rise rate for both 50% and 60% PANI gaskets. It was found that both gaskets had the same initial heating rates. However, as shown in Figure 32, as the heating time increases, the temperature rise rate (heat generation rate) and the absolute temperature for the 60% PANI exceeds that of 50% PANI. Therefore, 60% PANI gaskets were used for most of the welding experiments.

Figure 32 shows that thermal runaway is not a problem for these gaskets because at elevated temperatures, the temperature rise rate (heat generation rate) decreases with increasing temperature. This decrease in heat generation rate is probably due to a reduction in the electrical conductivity of PANI at elevated temperatures. Figure 33 shows that in subsequent heating of the same 60% PANI gasket the temperature and temperature rise rate in the gasket are substantially lower than they were during the first heating. This irreversible loss in electromagnetic absorption is probably due to permanent chemical changes which cause an irreversible loss of conductivity in PANI at high temperatures [51]. Further experiments were carried out to estimate the initial power dissipation inside the gasket during heating.
Figure 32: Adiabatic heating results of 50%, and 60% 0.5mm thick composites using multi-mode new fixture
Figure 33: Re-heating curves for 60%, 0.5mm thick composite
To determine the initial power dissipation in the gasket, it is necessary to
determine the electric field distribution in the material. Assuming the electric field in the
gasket to be harmonic and of constant magnitude (gasket is small compared to the
wavelength and skin depth), the average power dissipated in the gasket is [28]

\[ P_{\text{avg}} = \sigma E_{\text{rms}}^2 V \]  

(Eq. 4-1)

where \( P_{\text{avg}} \) is the average power dissipated, \( E_{\text{rms}} \) is the root mean square value of the
electric field strength in the gasket, and \( V \) is the volume of the gasket. It is important to
note that the electric field strength in the gasket is related to the conductivity of the
gasket. Therefore, increasing the conductivity of the gasket may not necessarily increase
the power dissipated. For example, for a perfect conductor the microwaves are reflected
and the internal electric field strength is zero resulting in no heating. Unfortunately,
determining the internal field strength in the gasket is very complicated, since its
introduction into the microwave cavity alters the field. Therefore, the field strength and
the internal heat generation rate in the gasket were estimated using calorimetry or
adiabatic heating of the gasket while measuring the temperature rise. Therefore by

\[ \rho C_p \frac{\partial T}{\partial t} = \dot{Q} = \frac{P}{V} = \sigma E_{\text{rms}}^2 \]  

(Eq. 4-2)

Rearranging above equation, \( E_{\text{rms}} \) can be obtained:

\[ E_{\text{rms}} = \sqrt{\frac{\rho C_p \frac{dT}{dt}}{\sigma}} \]  

(Eq. 4-3)

where \( \rho \) is the density, \( C_p \) is the specific heat, \( \sigma \) is conductivity, \( T \) is temperature, and \( t \) is
time. This way the heating effectiveness of each type of gasket could be evaluated.
Using equation (4-3) requires knowledge of material properties such as density, specific heat and composite conductivity. Density of pure HDPE and PANI were obtained by forming a disk and measuring the weight and the volume of the disk. By using weight $W = \rho \cdot V$, density can be obtained. Following the rule of mixture, the density of the composite was obtained. It was found that HDPE has a density of 0.9 g/cm$^3$ and the density of pure PANI was 1.04 g/cm$^3$. Therefore, the density of a 50% PANI composite is 0.95 g/cm$^3$ and the density of 60% PANI composite is 0.96 g/cm$^3$. While the density is a function of temperature, the calculation of initial heat generation was performed at low temperature range (i.e., near the room temperature). Therefore, it is assumed that the density is a constant. This assumption is also applied to specific heat and conductivity.

The next step is to determine the specific heat of the material. Therefore, Differential Scanning Calorimeter (DSC - Thermal Analysis 2000, Du Pont Instrument) was used to measure the specific heat. Three runs were needed to calculate the heat capacity, i.e., base line (empty container), standard (sapphire), and sample (PANI). A small amount of the sample (~ 4 mg) was placed in an aluminum capsule which was hermetically sealed. The heating rate was 10°C/minute. The heat flow vs. temperature curve was obtained to calculate the specific heat of PANI. Figure 34 shows the specific heat of pure PANI-HCl powder and HDPE as function of temperature. It was found that the peak $Cp$ of HDPE corresponds to the $T_m$ at 132°C which is in very good agreement with published data. The value used in this calculation was the $Cp$ at 60°C, i.e., 2749 (KJ/g°C) for HDPE and 2274 (KJ/g°C) for PANI. Therefore, using the rule of mixture,
Specific Heat Measurement

Figure 34: Specific heat of HDPE and pure PANI-HCl measured from DSC
the value for 50% PANI composite was 2511.5 (KJ/g°C) and for 60% composite was 2464 (KJ/g°C).

The complex permittivity of the gasket was measured at room temperature using a transmission line technique. A HP 8753C network analyzer was used for the measurement. The annular sample with inside diameter of 3mm and outside diameter of 7mm and length of 7mm was precisely compression molded to fill the cross section of a coaxial line. The s-parameter was measured, s11 and s21, to calculate the complex permittivity. The measurement was done in the frequency range of 0.2 to 6 GHz. Figure 35 shows the results of the measurement as function of frequency at room temperature. It is found that at 2.4 GHz the conductivity was 8 s/m for 60% PANI composite and 3.27 s/m for 50% PANI composite. The loss tangent at 2.4 GHz for 60% and 50% PANI composite was 1.23 and 0.69, respectively.

The heating rate (dT/dt) can be obtained by direct temperature measurement. The temperature was measured by using a Luxtron 755 fluoroptic thermometer. The heating rate is the slope of the temperature versus time curve. Therefore, linear regression was used to estimate the slope. The samples used in this study included 50% PANI 0.5mm thick and 60% PANI 0.5mm thick.

Table 2 shows the estimated electric field strength in the gasket at 60°C. It was found that higher conductivity results in lower electric field in the material. This is reasonable because the electric field is reflected more from a higher conductivity surface.
Figure 35: Microwave conductivity from 0 to 6 GHz at room temperature
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Field strength(V/m) 60%-PANI-0.5mm</th>
<th>Field Strength(V/m) 50%PANI-0.5mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2270</td>
<td>4230</td>
</tr>
<tr>
<td>2</td>
<td>2860</td>
<td>3330</td>
</tr>
<tr>
<td>3</td>
<td>2320</td>
<td>4350</td>
</tr>
<tr>
<td>4</td>
<td>2670</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>2530±245</td>
<td>3970±455</td>
</tr>
<tr>
<td>Averaged Initial heat generation rate</td>
<td>$5.12 \times 10^7$ (W/m$^3$)</td>
<td>$5.15 \times 10^7$ (W/m$^3$)</td>
</tr>
</tbody>
</table>

Table 2: Estimated electric field strength in gasket at 60°C from adiabatic heating
The initial heat generation rate can be calculated from equation (4-2). The average initial heat generation rate for 50% 0.5mm thick PANI composite was $5.15 \times 10^7$ w/m$^3$ and for 60% 0.5mm thick PANI composite it was $5.12 \times 10^7$ w/m$^3$.

4.3.2.2 Heat Generation Rate - During Welding

In order to have better understanding of the heating process during welding, the heat generation rate has to be determined through the whole welding process. The method used in previous section provides a simple but effective way to determine the heat generation. Therefore, the concept will be extended to the welding case.

An alternative way to find internal heat generation rate is through the finite element method (FEM) with temperature measurement. Consider the one-dimensional heat transfer equation

$$\rho C_p \frac{\partial T}{\partial t} = \lambda \frac{\partial^2 T}{\partial x^2} + Q$$

(Eq. 4-4)

Where $\lambda$ is thermal conductivity. By placing any number of $\dot{Q}$ into FEM analysis (ANSYS in this case), one can obtain a temperature history for a point of interest. The $\dot{Q}$ of the gasket can be determined by matching the FEM temperature history with experimentally measured temperature history for the same position. A Luxtron 755 multi-channel
fluoroptic thermometer was used to measure the temperature rise during microwave welding. Figure 36 shows typical temperature histories for 60\%PANI-0.5mm and 50\%PANI-1mm gaskets in microwave welding. The fluoroptic probe was inserted between the gasket and the HDPE bar to obtain a direct temperature reading. As discussed previously and shown in Figure 36, the 60\%PANI gasket experienced faster heating and higher temperatures when exposed to an electromagnetic field. It is noted that the higher the temperature, the lower the conductive \cite{52}; therefore, thermal runaway is not the issue here. Heat generation rate in microwave welding for different gaskets was then estimated by using a one-dimensional finite element heat conduction model (ignore convection losses at surrounding). This heat generation rate can be found by changing the value of the heat generation rate with respect to time in the FEM analysis to obtain a temperature history at a specific point which matches the experimental measurement at the same point. This requires a lot of trials to have the correct temperature output from the FEM. The input file for the FEM calculation is listed in Appendix B. Figure 37 shows the experimental and computer simulated temperature histories and the estimated heat generation rate of the 50\%PANI-1mm gasket. The density and thermal conductivity in the FEM analysis are set to be constant, and the measured Cp as a function temperature was used. The maximum heat generation rate in the 50\%PANI gasket is $7.7 \times 10^7$ Watt/m$^3$. The decrease in the heat generation rate for elevated temperatures is caused by the changes in the electrical properties of the composite gasket. This change is caused by elimination of the HCl on amino group and by chlorinating of the aromatic ring in the
Figure 36: Temperature histories at the interface between the HDPE bar and conducting composite using multi-mode
Figure 37: Experimental and FEM predicted internal heat generation rate during welding for 50% PANI, 1mm thick gasket
polyaniline [51]. Figure 38 shows the experimental and computer simulated temperature history and estimated heat generation rate for the 60% PANI-0.5mm gasket, for which the maximum heat generation rate is $1.96 \times 10^8$ Watt/m$^3$. Because of differences in the electrical properties of 50% PANI-1mm and 60% PANI-0.5mm gaskets, the 60% PANI gasket can absorb more microwave energy. Note that the 50% PANI-1mm thick gasket contains more polyaniline powder than the 60% PANI-0.5mm gasket, but the latter can still have a higher temperature rise rate and attain higher temperatures, indicating that the heat generation per unit volume is determined by the weight percentage of polyaniline in the gasket (e.g. intrinsic electrical properties of the gasket) and not just by the total amount of polyaniline powder in the gasket. There are many advantages to have the estimates for the heat generation rate in the process, such as prediction of molten layer thickness, determination of the optimal PANI-HCl content in the gasket and the thickness of the gasket; moreover, it can be used to determine the heating time for different materials, those parameters are all related to the final joint strength.

Figure 39 shows the FEM predicted temperature distribution along the sample for 60 seconds of heating time, the plot is based on half of the sample due to symmetry. This figure provides the information on the molten layer thickness during welding. If the melting temperature of HPDE is 135°C, then the molten layer thickness is 1mm (0.04") using 50% 1mm thick PANI composite and the molten layer thickness is 1.78mm (0.07") for using 60%, 0.5mm composite. This enable the designer to manipulate the conducting composite in microwave welding and to achieve the desired heating pattern.
Figure 38: Experimental and FEM predicted Internal heat generation rate during multi-mode microwave welding for 60% PANI, 0.5mm thick gasket
Figure 39: FEM predicted temperature profile along the HDPE using 60%, 0.5mm thick gasket using multi-mode microwave oven
4.3.3 Welding Results and Discussion

During the initial microwave welding tests, the applied pressure on the samples was kept constant throughout the heating and cooling of the parts, resulting in equal heating and welding pressures. Figure 40 shows that for heating and welding pressures (constant pressure) of 0.31 MPa, even long heating times could not improve the average weld strength beyond about 16 MPa, which is only 65% of the bulk strength of HDPE. Therefore two new pressuring methods were evaluated in welding: step pressuring, and post heating pressuring. Step pressuring applied a small pressure during heating which ensures the good contact between samples and the heating gasket. A higher pressure was activated manually right after the heating and throughout the cooling process. Post heating pressure applied no pressure during heating, a high pressure was applied at the end of heating and throughout the cooling process. In order to understand the gasket deformation during welding, two magnifiers were placed in the microwave oven to enlarge the interface. The deformation of the interface was recorded using a video camera outside the oven through a 1 cm hole for three different pressuring methods. Viewing the heating process showed that under constant pressuring heating conditions (heating pressure of 0.31 MPa) the gasket gets squeezed out of the interface as the HDPE in the gasket heats up and melts. As the gasket squeezes out, less conductive material remains at the interface resulting in reduced heating of the interface and in a smaller molten layer in the
Figure 40: Effect of constant pressure and post heating pressure (0.3MPa) on joint strength using 60% PANI, 0.5mm thick gasket
parts. Step pressuring does not apply large pressure on the gasket during heating; however, due to thermal expansion, the gasket got squeezed out also. Therefore, to minimize the squeeze out of the gasket, the parts were brought in contact with the gasket, but no pressure was applied during the heating. Figure 40 shows that for the case of no heating pressure and a welding pressure of 0.31 MPa, the weld strength increases with increasing heating time and it approaches the bulk strength of HDPE (24.43±0.31MPa). Therefore, in all subsequent welding tests, no heating pressure was applied.

During microwave welding, increasing the heating time results in the development of a thicker molten layer in the parts which improves the joint strength. The larger molten layer, provides more time for intimate contact and diffusion to occur prior to resolidification. Also, the thicker molten layer enables complete squeeze out of the gasket during the welding stage so that the mechanically weak gasket does not limit the strength of the joint. Figure 41 shows the effect of heating time on weld strength for two welding pressures. In both cases, the weld strength increases with increasing heating time and it approaches the strength of the bulk HDPE. Figure 41 also shows that increasing the welding pressure increases the weld strength for all heating times. The higher welding pressure results in more rapid and complete squeeze out of the mechanically weak gasket and it enables the parts to achieve intimate contact at the interface faster. Figure 42 shows that for a constant heating time of 60 seconds, increasing the welding pressure substantially improves the weld strength, and for a weld pressure of 0.9 MPa the weld strength is equal to the bulk strength of HDPE. However, as was observed in hot plate
Figure 41: Effect of heating time on joint strength for two welding pressure using 60%PANi, 0.5mm thick composite
Figure 42: Effect of post heating pressure on joint strength
welding, it is expected that for high welding pressures, the weld strength will decrease because most of the molten polymer would be squeezed out prior to intimate contact and diffusion occurring. Figure 43 shows the effect of heating time on joint strength for 50% PANI-1mm gasket. The post-heating pressure used here was 0.62 MPa. The maximum joint strength was 23.75±1.07 MPa, which is 97% of the HDPE strength. These results indicate the possibility of both the 50% PANI-1mm and 60% PANI-0.5mm gaskets resulting in joints that are as strong as the bulk material when optimal pressures are used.

As mentioned before, the displacement (reduction in sample length) was also measured during pressuring. Figure 44 shows a typical displacement curve as function of time for 50% 1mm PANI composite after 80 seconds of heating and at 0.6 MPa welding pressure. It was found that the displacement increased dramatically in the beginning of the squeezing. This represents the large molten layer that was produced during heating and the interface is more like "liquid". At the end of squeezing, the remaining molten polymer acts more like "solid" material. Further study is needed to understand the squeeze flow phenomena in microwave welding. Figure 45 shows the relation between joint strength and displacement in microwave welding which uses 50%, 1mm thick PANI composite. The data points include the different heating time but same pressure. It was found that higher the displacement results in better joint strength. This may give an index to correlate the displacement to the joint quality. However, more efforts are required to quantify the relation between displacement and joint strength.
Figure 43: Effect of heating time on joint strength for 50% PANI, 1mm gasket
Figure 44: Displacement during squeeze flow for 50% PANI, 1mm thick gasket
Figure 45: Relation between displacement and joint strength for 50% PANI, 1mm gasket
4.4 Welding of Thermoplastics

Welding of other thermoplastics materials such as PP, PC, PETG and Nylon was also attempted. Figure 46 shows the maximum joint strength for these polymers using 60% 0.5mm PANI + 40% HDPE gasket. The joint strength achieved are quite high especially considering that the gasket used had HDPE and not the polymer being joined. There is also considerable latitude in the processing conditions to optimize the joint strength. For example, it is noted that nylon contains polar groups that will absorb the microwave energy and hence increase its temperature. The polyaniline gasket therefore acts like a catalyst to speed up the nylon in reaching its melting temperature. However, the low viscosity of nylon above its melting temperature makes the process more difficult to control, as does the swelling of nylon above its melting temperature. The gasket should be replaced by a polyaniline and nylon blend to achieve higher joint strength. A detail study of this case is discussed in chapter 6.
Figure 46: Maximum joint strength for PP, PC, PETG and Nylon using HDPE conducting composite
CHAPTER 5

MODELING OF MICROWAVE HEATING
OF CONDUCTING ELEMENT

5.1 Introduction

The heating element in microwave welding performs a key role in determining the welding time, and hence affecting the final joint strength. Too much or too little heating results in low joint strength compared with base material strength. Therefore, prediction of heat generation in microwave heating of the conductive composite is important. When a heating element is subjected to microwave radiation, heat is generated by the absorption of the microwave energy in the element. Currently, there are several ways to predict the heat generation, such as the finite element method (FEM), finite difference time domain (FDTD), experimental temperature measurements, and the transmission line method. Although FEM and FDTD methods produce the most accurate models, they require significant effort to develop. In addition, these methods need information on material properties such as electrical conductivity, complex permittivity and permeability which are
often difficult to measure as function of temperature. The experimental temperature measurement method which was discussed in the previous chapter also needs thermal properties. In this chapter, a transmission line method is proposed, and equivalent circuit models are constructed to predict the heating ability of the conductive composites. Even though transmission line theory is approximate (based on one dimensional approximation), the heating system setup is ideally suited for the application of this method. The use of transmission line theory in conjunction with scattering parameters allows us to calculate the power absorbed by the heating element. The results of the prediction are compared to experimental temperature measurements on the same sample to verify the accuracy of the model.

5.2 Transmission Line Theory

A transmission line is a device which transfers electromagnetic energy from one point to another. Typical transmission line structures are pair of wires, parallel plates, coaxial cables, and waveguides. A traditional AC circuit model of the transmission line is inaccurate if the line lengths are comparable to the wave length. The transmission line, on the other hand, allows for the voltage and circuit to vary along the length of the line. Thus, it can produce a good approximate model for wave propagation in the waveguide. Discontinuities inside the waveguide can be modeled with discrete circuit elements. Because of the simplicity of the transmission line theory, it is advantageous to use it to solve the microwave waveguide heating system.
Let us begin by reviewing basic transmission theory. First, consider a uniform transmission line which can be modeled by the circuit elements as shown in figure 47. The \( R' \) is series resistance per unit length representing the dimension and conductivity of the metallic conductor. \( G' \) is shunt conductance per unit length representing the loss tangent of insulating material between the conductors. \( L' \) is inductance per unit length representing the magnetic flux on the conductor. \( C' \) is shunt capacitance per unit length representing the charge on the conductors. Applying Kirchhoff's voltage and current law to the line results in the following expression [53]

\[
V = (R'\Delta z)I + (L'\Delta z)\frac{\partial I}{\partial t} + (V + \Delta V)
\]

\[
I = (G'\Delta z)(V + \Delta V) + (C'\Delta z)\frac{\partial V}{\partial t}(V + \Delta V) + (I + \Delta I)
\]

(Eq. 5-1)

If \( \Delta z \to 0 \) then \( V + \Delta V \to V \)

\[
- \frac{\partial V}{\partial z} = R'I + L'\frac{\partial I}{\partial t}
\]

\[
- \frac{\partial I}{\partial z} = G'V + C'\frac{\partial V}{\partial t}
\]

\( V \) and \( I \) represent the time varying voltage and current at the input. When equation (5.1) is subjected to a sinusoidal source the above equation becomes

\[
- \frac{dV}{dz} = (R' + j\omega L')I = Z'I
\]

\[
- \frac{dI}{dz} = (G' + j\omega C')I = YV
\]

(Eq. 5-2)

\( Z' = R' + j\omega L' \) is series impedance/length

\( Y' = G' + j\omega C' \) is shunt admittance/length
By Kirchhoff's voltage and current laws to the line section yields

Figure 47: Circuit representation of a uniform transmission line
Equations (5.2) are the time harmonic transmission line equations. Differentiating the first equation in (5.2) with respect to $Z$ and substituting $-YV'$ for $dI/dz$ into it, yields

\[
\frac{d^2 V}{dz^2} = Z'Y' V \quad \text{and} \quad \frac{d^2 I}{dz^2} = Z'Y'I \quad \text{Eq. 5-3}
\]

These are the second order differential equations. The phasor solutions are

\[
V = V_0^+ e^{-\gamma z} + V_0^- e^{\gamma z} = V^+ + V^-
\]

\[
I = I_0^+ e^{-\gamma z} - I_0^- e^{\gamma z} = I^+ - I^-
\]  \quad \text{Eq. 5-4}

$V^+$ represents the forward voltage wave traveling in $+z$ direction and $V^-$ represents the reflected wave traveling in $-z$ direction. $V_0^+$, $V_0^-$ are the values of $V^+$ and $V^-$ at $z = 0$, and $\gamma$ is the propagation constant and is defined as

\[
\gamma = \sqrt{Z'Y'} = \sqrt{(R' + j\omega L')(G' + j\omega C')} = \alpha + j\beta \quad \text{Eq. 5-5}
\]

$\alpha$ is the attenuation constant (Np/length) and $\beta$ is the phase constant (rad/length).

The characteristic impedance $Z_0$ is defined as:

\[
Z_0 = \sqrt{\frac{Z'}{Y'}} = \frac{V^+}{I^+} = \frac{V^-}{I^-} \quad \text{Eq. 5-6}
\]

The average power flow on the line is defined as:

\[
P = \text{Re} (V*I^*) = VI \cos(\theta) \quad \text{Eq. 5-7}
\]

The $*$ represents the complex conjugate and $\theta$ is the power factor angle. $V$ and $I$ are in rms value. When this transmission line is terminated with a load with impedance $Z_L$ and $Z_L \neq Z_0$ as shown in figure 48(a), the net power flow on the line will also follow equation (5.7). By use of $V^* = I^*Z_0$ and $V^* = \Gamma V^*$ equation (5.4) can be rewritten as
(a) A transmission line terminated by a load \( Z_L \)

(b) Equivalent circuit

Figure 48: Transmission line terminated by a load \( Z_L \) before impedance transmission (a) and after impedance transmission (b)
\( V = V^*(1 + \Gamma) \) and \( I = \frac{V^*}{Z_0}(1 - \Gamma) \)  

(Eq. 5-8)

where \( \Gamma \) is the reflection coefficient that is defined as reflected voltage divided by forward voltage at the point \( z \), and it can be written as

\[
\Gamma = \frac{V^-}{V^+} = \frac{1}{\Gamma^+}
\]

(Eq. 5-9)

Substituting equation (5.8) into equation (5.7), the solution of the net power flow on the line is obtained as

\[
P = \text{Re}[(1 - |\Gamma|^2 + \Gamma - \Gamma^*) \frac{(V^*)^2}{Z_0^*}] \]

(Eq. 5-10)

For a low loss line, \( Z_0 \) can be approximated by a real number and the above equation reduces to

\[
P = P^* - P^- \quad \text{and} \quad P_{in} = P^*_n \frac{1 - (\Gamma_n)^2}{Z_0^*} \]

(Eq. 5-11)

where \( P^* \) is the forward power and \( P^- \) is the reflected power. \( P^*_n \) represents the forward power at the input terminal. Thus, the net power is the difference between the forward and reflected power. Because \( Z_0 \) is real; \( V^* \) and \( I^* \) are in phase. Thus \( P^* \) can be expressed as

\[
P^* = \frac{(V^*^2)}{Z_0} = \frac{(V_{0^*})^2}{Z_0} e^{-2\gamma x} \quad \text{and at input} \quad P_{in}^* = \frac{(V_{0^*})^2}{Z_0} \]

(Eq. 5-12)

By applying Kirchhoff's voltage law at the input end, \( V_{0^*} \), as shown in figure 48(b), can be solved as
\[
V_0^* = \frac{V_G Z_0}{(Z_G + Z_0)(1 - \Gamma_G \Gamma_{in})} = \frac{V_G Z_0}{(Z_G + Z_0)(1 - \Gamma_G \Gamma_L e^{-2\gamma_2})}
\] 
(Eq. 5-13)

\[\Gamma_G = \frac{Z_G - Z_0}{Z_G + Z_0}
\] 
(Eq. 5-14)

\(Z_G\) is the impedance of the generator, and \(V_G\) is the open circuit voltage of the generator.

The input impedance \((Z_{in})\) in figure 48(b) is

\[Z_{in} = \frac{Z_0}{Z_0 + Z_L \tanh(\gamma d)}
\] 
(Eq. 5-15)

which represents the equivalent impedance of all the circuit to the right of point \(z = 0\).

Therefore, by knowing \(V_0^*\), the net power can be calculated. In order to develop the general form for the net power, let us substitute \(V_0^*\) from equation (5.13) into equation (5.12). Thus, the forward power is

\[P^* = \frac{V_0}{4 Z_0} \left| 1 - \Gamma_G \right|^2 e^{-2\gamma_2}
\] 
(Eq. 5-16)

At the input \((z = 0)\), the forward power is

\[P_{in}^* = \frac{V_0}{4 Z_0} \left| 1 - \Gamma_G \right|^2
\] 
(Eq. 5-17)

Equation (5.17) can be expressed in terms of the available power \((P_a)\), where

\[P_a = \frac{V_0^2}{4 R_G}
\] 
(Eq. 5-18)

and \(R_G\) is real part of \(Z_G\). Thus, equation (5.17) can be expressed as
By substituting equation (5.19) into equation (5.11), we can write the net input power as

\[ P_{in} = P_A \frac{1 - |\Gamma_G|^2 (1 - |\Gamma_m|^2)}{|1 - \Gamma_G \Gamma_m|^2} \]  

(Eq. 5-20)

The net power absorbed by the load is

\[ P_L = P_A \frac{(1 - |\Gamma_G|^2 (1 - |\Gamma_m|^2) e^{-2\gamma_1}}{|1 - \Gamma_G \Gamma_m e^{-2\gamma_1}|^2} \]  

(Eq. 5-21)

The difference between \( P_L \) and \( P_{in} \) is the power absorbed by transmission lines.

Based on the above theory, an equivalent circuit model can be used to calculate the microwave heating of the conductive composite.

5.3 Construction of Equivalent Circuit

The initial heat generation rate of conductive composites under single mode microwave radiation is discussed in this section. In order to predict the heating, the equivalent circuit representing the microwave adiabatic heating experiment must be constructed. The development of this circuit requires knowledge of the parameters such as the characteristic impedance of the waveguide, the transmission line lengths, the impedance of the various components and the impedance of the heating element. Some of these parameters are well defined and can be calculated, such as characteristic impedance.
of the line; others must be found through experimental measurements, such as the heating element impedance. Hence, a series of experiments is necessary to construct the circuit.

5.3.1 Experimental Setup

As discussed in the previous section, the impedance of the conductive composite must be determined at the operating frequency in the S band waveguide. Thus, a HP 8753C network analyzer system was used to measure the impedance. A diagram of the network analyzer is shown in figure 49. The system contains the network analyzer and an S-parameter test set which has an APC-7 connector output. Microwave heating was also performed in the S band waveguide. In order to couple the microwave signal to the waveguide, two types of adapters are needed. The first one is a APC-7 to N type female which is an air-filled section (HP 11525A). The second one is an N type male coaxial to waveguide adapter (HP S281A). This adapter contains two coaxial lines, one which is air-filled and one which is dielectric (Rexolite) filled, and with a transition section from coaxial to waveguide. This transition is located at the broad side of the waveguide that separates the waveguide into two parallel connected transmission lines. One of the transmission line is shorted by the waveguide housing, and one is open and it can be connected to any waveguide for measurement. Figure 50 [54] shows a detailed drawing of the HP S281A adapter. All of these coaxial lines and waveguides need to be considered in the equivalent circuit model for determining the heating element impedance.
Figure 49: Diagram of HP 8753C network system
Figure 50: Detailed drawing of HP S281A adapter [54]
For a WR 284 S band waveguide, the characteristic impedance \( Z_0 \) is defined as \( V'/I' \), where \( V' \) is a voltage between conductors, and \( I' \) is the current in the propagation direction for the traveling wave. The voltage between the conductors is a function of waveguide width; therefore, there is no unique definition for \( Z_0 \). A modified power-voltage definition is used for all calculations [53]

\[
Z_0 = 377 \frac{b}{a} \sqrt{\frac{\mu_r}{\varepsilon_r}} \frac{\lambda_g}{\lambda} \quad \text{(Eq. 5-22)}
\]

where \( \lambda_g \) is the wave length in waveguide and \( \lambda \) is the wave length in free space, \( a \) is the width of the waveguide and \( b \) is the height of the waveguide. For the APC-7 line, the characteristic impedance \( Z_{01} \) is 50 \( \Omega \), while the characteristic impedance of the air filled coaxial line is also 50 \( \Omega \). For Rexolite filled coaxial line, its characteristic impedance \( Z_{02} \) can be calculated from

\[
Z_{02} = 60 \sqrt{\frac{\mu_r}{\varepsilon_r}} \ln \frac{b}{a} \quad \text{(Eq. 5-23)}
\]

where \( a \) is the radius of the inner conductor and \( b \) is the radius of the outer conductor. In this case \( Z_{02} \) is also 50\( \Omega \). The physical dimensions of the coaxial lines and waveguides are also needed to construct the circuit model. These dimensions can be found from caliper measurements. The detailed dimensions are listed in Appendix C.

5.3.2 Experimental Techniques
The construction of the equivalent circuit must account for all the components in microwave system. As discussed in section 5.2, the length of each transmission line has to be known for the construction of the equivalent circuit. These lengths can be obtained by direct measurement, with the exception of the transition section from coaxial to waveguide as shown by Ztrans in figure 50. This transition is difficult to use existing formula to find the associated parameters, i.e., resistance, capacitance or inductance. Two series of experiments are employed to solve for this parameter. Figure 51 shows the first experiment with the shorted circuit on the HP S281A waveguide. Figure 52 shows the second experiment which is the same as figure 51 but with a 610mm (24") waveguide connected to an HP S281A adapter and shorted at the end. Before experiments can be performed, the network system must be calibrated. An APC-7 type calibration kit (HP 85031B-7mm) was used to calibrated the system at the Zina position in figure 51(a). There are three calibration impedance the short, open and 50Ω load. The output from the network analyzer contains the reflection coefficient, phase angle, SWR, Smith chart, and impedance. These quantities are all linked together mathematically; thus, by using any one of them, the input impedance can be obtained. For convenience, direct impedance reading from the network analyzer is used.

The concept of this measurement is to obtain the input impedance as shown in figure 51(b); therefore, S_{11} is measured. The first measurement is shown in figure 51 where the reading from the network analyzer is Zina which represents all the impedance to the right of the calibration point. In order to find the Ztrans in figure 51(b), Zin12a and Zin4a must
Figure 51: Schematic representation of impedance measurement using network analyzer and HP S281A with short (a), and equivalent circuits (b)
(a) Experimental Setup

(b) Equivalent Circuit in Parallel

(c) Equivalent Circuit in Series

Figure 52: Schematic representation of impedance measurement using network analyzer and HP S281A with 24" waveguide with short (a), and equivalent circuit with parallel transition (b) and equivalent circuit with series transition (c)
be determined. Therefore, by use of impedance transformation (equation 5.15) the load impedance, \( Z_{L1} \) and \( Z_{L2} \), can be transferred to \( Z_{in1a} \) and \( Z_{in2a} \), respectively. In addition, since these two impedance are connected in parallel, \( Z_{in12a} \) can be obtained, as follows:

\[
\frac{1}{Z_{in12a}} = \frac{1}{Z_{in1a}} + \frac{1}{Z_{in2a}}
\]

(Eq. 5-24)

\( Z_{in12a} \) represents the impedance to the right of plane A-A. Furthermore, the transformation from \( Z_{in1a} \) (read from the network analyzer) to \( Z_{in3a} \), and then from \( Z_{in3a} \) to \( Z_{in4a} \) is also possible by rearranging equation (5.15). The new equations become

\[
Z_{in3a} = Z_{01} \frac{Z_{in1a} - Z_{01} \tanh(\gamma_1 \text{dc1})}{Z_{01} - Z_{in1a} \tanh(\gamma_1 \text{dc1})}
\]

and

\[
Z_{in4a} = Z_{01} \frac{Z_{in3a} - Z_{02} \tanh(\gamma_2 \text{dc2})}{Z_{02} - Z_{in3a} \tanh(\gamma_2 \text{dc2})}
\]

(Eq. 5-25)

\( Z_{in4a} \) represents the impedance to the left side of plane B-B. By doing so, the transition from the coaxial line to the waveguide can be calculated. Two solutions (\( Z_s \) or \( Z_p \)), one in parallel (\( Z_p \)) with the transmission line and one in series (\( Z_s \)) with the transmission line are obtained. However, only one solution is the correct answer. Therefore, a second experiment is needed to determine which is the correct equivalent circuit. Figure 52 shows a similar experiment but with an additional 610mm (24") line. The ideal behind the second experimental set-up is that the transition impedance is not influenced by the transmission line length. Since, after the first experiment, it is not known if the transition impedance is a
series or a parallel component, by changing the transmission line length the transition impedance can be uniquely determined.

The second measurement is performed in the same manner as the first experiment, measuring the input impedance (Zin) as shown in figure 52(a). Since two solutions exist from the first experiment, two equivalent circuits models can be constructed as shown in figure 52(b) and figure 52(c). The input impedance can also be calculated by using impedance transformation; thus, two input impedance can be obtained, Zinpa as parallel case and Zindcl as series case. However, only one of the impedance Zinpa (parallel case) or Zindcl (series case) will match to the measured impedance Zin. Both calculations will be discussed to verify the solution.

First, let us assume that the transition section is in parallel with the transmission line; therefore, Zp will be used in the calculation as shown in figure 52(b). The calculation starts from transferring ZL1 to Zdw3 and ZL2 to Zdw2 by adding them together using equation (5-24) Zindw23 can be obtained. If the transition section is in parallel to the transmission line, equation (5-24) can be used such that

\[
\frac{1}{Zin_{4pa}} = \frac{1}{Zp} + \frac{1}{Zindw23} \quad \text{(Eq. 5-26)}
\]

The input impedance (for parallel case) can be obtained by transferring Zin_{4pa} two more times using equation(5-15) from Zin_{4pa} to Zin_3pa and from Zin_3pa to Zinpa. This result will be compared to the measured impedance (Zin). Next it is assumed the transition impedance is in series with the transmission line as shown in figure 52(C). The calculation starts from transferring ZL1 and ZL2 to Zindw23. If the transition is in series to the
transmission line then $Z_{in4s}$ equals to the summation of the $Z_s$ and $Z_{indw23}$. The input impedance (for series case) can be obtained by transferring $Z_{in4s}$ two more times using equation (5-15) from $Z_{in4s}$ to $Z_{indc2}$ and from $Z_{indc2}$ to $Z_{indc1}$. It is found that only the series connected impedance ($Z_s$) satisfies both the shorted adapter and the 24” waveguide experiments. Therefore, the transition from coaxial line to waveguide should be modeled as a series component. This value will change with measured frequency. An example of the detailed calculation is attached in appendix C.

The next step is to obtain the heating element impedance ($Z_{g}$). In microwave heating, the element is placed at a quarter wavelength ($\lambda_g$) from the shorted end, and at the centerline of the waveguide. The $\lambda_g$ is the wave length in the waveguide which can be calculated by [55]

$$\lambda_g = \frac{\lambda_0}{\sqrt{1 - \frac{\lambda_0}{\lambda_c}}}$$  \hspace{1cm} (Eq. 5-27)

where $\lambda_0$ is the wave length in free space and $\lambda_c$ is the cut-off wave length. For a TE$_{10}$ mode and WR 284 air-filled waveguide operating at 2.45GHz, $\lambda_0$ is 122.45mm and the $\lambda_c$ is twice the width of the waveguide, which is 144 mm. Therefore, the heating element was placed at quarter $\lambda_g$ as shown in figure 53(a). Figure 53(b) shows the equivalent circuit used to calculate the heating element impedance. The input impedance ($Z_{in}$) is measured using network analyzer. Six subsequent impedance transformations were performed starting from $Z_{in}$ and finally obtaining $Z_{in2}$. Since the waveguide is shorted $Z_L = 0$, and by doing one impedance transformation, $Z_{in1}$ can be determined with ease.
Figure 53: Schematic representation of impedance measurement using network analyzer and HP S281A with 24" waveguide with sample (a), and equivalent circuit (b)
The heating element impedance can then be deduced from \( Z_{\text{in2}} \) to \( Z_{\text{in1}} \). Once more, two cases are presented, a parallel and a series connection. However, it is found that the series connection has a negative resistance which is physically impressible. Thus, the heating element impedance is a parallel connection. Of course, the heating element can be placed at any place along the waveguide which results in a different impedance reading. By using the same method, the respective impedance can be calculated. A detailed calculation was attached in Appendix C.

5.3.3 Construction of Microwave Heating Circuit

Once the impedance of the heating element is determined, an equivalent circuit model can be constructed to predict the heating. Figure 54(a) shows the microwave heating setup and figure 54(b) shows the equivalent circuit for microwave heating. Equation (5.21) can be used to calculate the power absorption in the heating elements. However, to use equation (5.21) one needs the value of \( \Gamma_G \), the reflection coefficient from the generator. Due to the presence of a 3-port circulator in the microwave system, there will be no reflection from the generator; thus, \( \Gamma_G = 0 \). Equation (5.21) is then reduced to

\[
P_L = P_a \left(1 - |\Gamma_L|^2\right) e^{-2\alpha l} \tag{Eq. 5-28}
\]

where \( P_a \) is the power delivered to waveguide.

An alternative way to calculated the power absorbed by the load is to use

\[
P_L = Re \left( V_L \* I_L^* \right) \tag{Eq. 5-29}
\]
Figure 54: Schematic representation of microwave heating of conducting composites (a), and equivalent circuit (b)
Use of this formula requires knowledge of the forward voltage at the input end which can be obtained from equation (5-13). Since $\Gamma_G = 0$, equation (5-13) reduces to

$$V_0^* = \frac{V_G Z_0}{(Z_G + Z_0)} \quad \text{(Eq. 5-30)}$$

With a carefully designed microwave system, the generator is always matched to the waveguide impedance, which is the case here. Thus, since $Z_G = Z_0$, equation (5-30) reduces to

$$V_0^* = \frac{1}{2} V_G \quad \text{(Eq. 5-31)}$$

Depending on the input microwave power, $V_G$ can be found by using equation (5-18), where $R_G = Z_G = Z_0$. The voltage at the load ($V_L$) in equation (5-29) can be found by combining equations (5-4) and (5-9) where

$$V_L = V_0^* e^{-\gamma l (1 + \Gamma_L)} \quad \text{(Eq. 5-32)}$$

and $I_L$ can be found from the impedance definition, $I_L = (V_L/Z_L)$. By substituting these results into equation (5-29) power absorption at the load can be calculated. Detailed calculations are shown in Appendix C for both methods.

5.4 Results of the Measurement

Table 3 shows the results of impedance measurements for a group of heating composites of different concentrations, thickness, shape, material and orientations. The results of the heating element impedance show that the heating elements can be modeled
<table>
<thead>
<tr>
<th>sample No.</th>
<th>Description</th>
<th>2.45GHz Re(Zg)</th>
<th>2.45GHz Im(Zg)</th>
<th>Power (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>103</td>
<td>PANI10% + HDPE90% = 6.86x7.24x0.47mm</td>
<td>536</td>
<td>-31640</td>
<td>0.071</td>
</tr>
<tr>
<td>152</td>
<td>PANI15% + HDPE85% = 7.06x7.07x0.47mm</td>
<td>863</td>
<td>-29430</td>
<td>0.132</td>
</tr>
<tr>
<td>202</td>
<td>PANI20% + HDPE80% = 6.93x7.03x0.45mm</td>
<td>1941</td>
<td>-23340</td>
<td>0.468</td>
</tr>
<tr>
<td>251</td>
<td>PANI25% + HDPE75% = 6.86x7.17x0.49mm</td>
<td>1627</td>
<td>-22140</td>
<td>0.437</td>
</tr>
<tr>
<td>303</td>
<td>PANI30% + HDPE70% = 6.64x7.53x0.5mm</td>
<td>2449</td>
<td>-17100</td>
<td>1.081</td>
</tr>
<tr>
<td>4012</td>
<td>PANI40% + HDPE60% = 6.94x7.20x0.43mm</td>
<td>4105</td>
<td>-13050</td>
<td>3.382</td>
</tr>
<tr>
<td>5014</td>
<td>PANI50% + HDPE50% = 6.83x7.12x0.41mm</td>
<td>3282</td>
<td>-9598</td>
<td>4.14</td>
</tr>
<tr>
<td>604</td>
<td>PANI60% + HDPE40% = 6.84x7.1x0.41mm</td>
<td>2463</td>
<td>-8299</td>
<td>4.255</td>
</tr>
<tr>
<td>503</td>
<td>PANI50% + HDPE50% = 6.81x7.08x0.9mm</td>
<td>1226</td>
<td>-7926</td>
<td>2.489</td>
</tr>
<tr>
<td>6011</td>
<td>PANI60% + HDPE40% = 6.66x6.92x0.69mm</td>
<td>1552</td>
<td>-8479</td>
<td>2.729</td>
</tr>
<tr>
<td>AL 1</td>
<td>Al foil = 7.53x9.17x0.01mm</td>
<td>15.51</td>
<td>-4313</td>
<td>0.11</td>
</tr>
<tr>
<td>V501</td>
<td>Versicon 50% + HDPE 50% = 7.15x7.7x0.42mm</td>
<td>76.13</td>
<td>-5543</td>
<td>0.327</td>
</tr>
<tr>
<td>40%-long</td>
<td>PANI40%+HDPE60% = 22.9x7.15x0.44mm</td>
<td>1416</td>
<td>-3198</td>
<td>14.127</td>
</tr>
<tr>
<td>40%-wide</td>
<td>PANI40%+HDPE60% = 7.15x22.9x0.44mm</td>
<td>1403</td>
<td>-5483</td>
<td>5.632</td>
</tr>
<tr>
<td>50%-long</td>
<td>PANI50%+HDPE50% = 15.75x7.25x0.5mm</td>
<td>1321</td>
<td>-1919</td>
<td>27.245</td>
</tr>
<tr>
<td>50%-wide</td>
<td>PANI50%+HDPE50% = 7.25x15.75x0.5mm</td>
<td>1346</td>
<td>-4508</td>
<td>7.716</td>
</tr>
<tr>
<td>ilm-1-2.34</td>
<td>PANI+HCSA+Nylon12 = 6.9x7.32x0.04mm</td>
<td>19980</td>
<td>-32980</td>
<td>1.962</td>
</tr>
<tr>
<td>ilm-2-100</td>
<td>PANI+HCSA+Nylon12 = 6.4x9.7x0.01mm</td>
<td>61.78</td>
<td>-3059</td>
<td>0.95</td>
</tr>
<tr>
<td>Nylon 6/6</td>
<td>Nylon 6/6 film = 10.29x11.33x0.5mm</td>
<td>756</td>
<td>-25500</td>
<td>0.154</td>
</tr>
</tbody>
</table>

Table 3: Result of the impedance measurement and power absorption
as a resistor (real part of impedance) and a capacitor (negative imaginary part of impedance) connected in series. The value of the imaginary part of the impedance is determined by the size of the conducting particle, the distance between the conducting particles, and the gap between heating element and the waveguide. Figure 55 shows a plot of impedance as a function of the polyaniline loading. It reveals that increasing the polyaniline concentration reduces the imaginary part of impedance and changes the real part of impedance. One possible reason for the decrease in imaginary part is because the capacitance becomes larger at higher concentrations due to the smaller distance between conducting particles. The effect of orientation on impedance is also shown in Table 3. For this case two orientations were considered. In the “long” case, the sample’s longer dimensions is parallel to the electric field, while in the “wide” case, the longer dimension is perpendicular to the electric field. The 40% and 50% PANI elements were used to study the orientation effect, sample 40%-long vs. 40%-wide and 50%-long vs. 50%-wide. Each pair of the samples shows very similar real parts but different imaginary parts of impedance, mainly because the long samples had shorter distance between sample and waveguide resulting in larger capacitance. Therefore, both of the long samples have smaller imaginary part compare to wide samples. The effect of sample thickness is also shown in Table 3, sample 503 vs. 5014 and 604 vs. 6011, which indicates that the thicker samples have smaller real parts. This is due to two samples having the same conductivity, i. e., same polyaniline loading, with the same length but different thickness, then the resistance, Re(Z), follows the equation:
Figure 55: Measured gasket impedance as a function of polyaniline concentration
\[ R = \frac{V}{I} = \frac{\int E \cdot dI}{\int J_s \cdot ds} = \frac{1}{\sigma} \int E \cdot ds \]  

(Eq. 5-33)

Therefore, a thicker sample has a larger surface area (ds), which results in smaller resistance. Of course, the thickness must be smaller than the skin depth which is the case for polyaniline composites. (For pure polyaniline HCl doped powder (\(\sigma \approx 100 \text{ s/m}\)) the skin depth is approximately 1 mm at 2.45 GHz).

As discussed in the section 5.3, the heating element was placed at a quarter wavelength (\(\lambda_g\)) from the shorted end during the measurements. It was found that the gap between the heating element and the bottom of the waveguide causes a change in impedance measurements which changes the power absorption. The gap causes a capacitive effect on the transmission line and decreasing the gap produces a larger capacitance which results in a smaller imaginary part of the impedance. From transmission line theory, it was expected that a smaller imaginary part in the impedance should produce higher power absorption. It is also found that increasing the sample dimension in parallel with the electric field direction also increases the power absorption as shown in Table 3. For example, see sample 40%-long and 4012 and sample 50%-long and 5014. The reason for that can be explained by using the Smith chart. Figure 56 shows the data points for 4012, 40%-long, 5014, and 50%-long on the smith chart. From the transmission theory, the maximum power absorption occurs when the data points are located at the center of
Figure 56: Normalized impedance shown in Smith chart
the chart. If the data points are not at the center of the chart, the one which is closer to the center results in the highest power absorption. The distance between the point to the center represents the magnitude of the reflection coefficient ($|\Gamma_c|$). From equation (5-29), the power absorption is proportional to the $(1-|\Gamma_c|^2)$, therefore, smaller the reflection coefficient results in higher power absorption. The 50%-long sample is the one closest to the center; thus, it absorbs more power than the other samples. Therefore, approximately a 6.5mm high and 6.5mm wide sample with a 10 mm gap distance is used throughout the measurement which is the same as in the adiabatic heating cases. Figure 57 shows the predicted power absorption as a function of polyaniline concentration in HDPE composites at different frequencies where the input power equals 100 watts. It is clear that a higher polyaniline concentration in the element gives a higher power absorption. The power absorption increased dramatically from 30% to 50% and remained the same at 60%. The reason is that increasing the polyaniline loading in the element reduces the imaginary part of the impedance and the smaller the impedance the higher power absorption. Figure 58 shows the power absorption calculation as a function of impedance. It shows that an increase in the imaginary part of the impedance can dramatically reduce the power absorption when the real part of the impedance is small. The maximum absorption occurs in the small impedance region which equals the characteristic impedance ($Z_0$) of the waveguide. This means that the load must match the line impedance in order to have the highest power transfer.
Figure 57: Predicted power absorption as a function of polyaniline concentration in different frequencies (input power is 100 Watts)
Figure 58: Theoretical calculation of power absorption as a function of impedance, and the input power is set to be 100 watts.
Therefore, the heating element impedance can be controlled through the conducting polymer concentration, thickness, and dimensions to satisfy the proper impedance condition as a function of frequency. Figure 59 shows the effect of heating element thickness on internal heat generation rate using circuit model. It is found that increasing the element thickness reduces the heat generation rate. If the heating is not at quarter wave length from the short, the model is still valid for predicting the power absorption. Figure 60 shows the initial internal heat generation rate as a function of sample location predicted from the equivalent circuit model. It also demonstrates the effect of the gap between the heating element and bottom waveguide on internal heat generation rate. As mentioned earlier, reducing the gap increases the power absorption. Since the measurements are performed under a standing wave condition, if the sample is placed away from the quarter wave position, the voltage (electric field) is reduced. The field strength is lower inside the heating element thus resulting in less heating. The maximum power absorption occurs at a quarter \( \lambda g \), and a minimum occurs at half \( \lambda g \). The heating element used in this case is a 60% PANI + 40% HDPE, 0.45mm thick, and 100 watts of input power is applied into the model.

5.5 Microwave Adiabatic Heating Experiment

To verify the accuracy of the equivalent circuit model, a microwave heating experiment is performed where adiabatic heating is assumed. The adiabatic heating equation is
Figure 59: Effect of gasket thickness on internal heat generation rate predicted from equivalent circuit model
Figure 60: Initial heat generation rate as a function of sample location predicted from equivalent circuit model.
\[ \rho C_p \frac{\partial T}{\partial t} = \dot{Q} \quad \text{(Eq. 5-34)} \]

where \( \rho \) is density, \( C_p \) is specific heat, \( \partial T/\partial t \) is the heating rate, and \( \dot{Q} \) is internal heat generation rate. Power absorption from microwave heating (\( P_{mw} \)) is \( \dot{Q} \times \text{Volume} \) (in watts). The thermal properties are all temperature dependent as discussed in chapter 4. Also it should be noted that the impedance measurement is performed at room temperature, thus the results are valid only at initial heat generation rate.

5.5.1 Microwave Heating System

Microwave source operates at different frequencies depending on power output. It was found that the frequency varies from 2.41 GHz at low power to 2.45 GHz at high power as shown in figure 61[56]. Due to the limits of the temperature measurement device, microwave heating can only be performed at low power levels. The frequency at this low power level is very close to the cut-off frequency for WR 284 waveguide. Therefore, a improved microwave heating system is used. Figure 54(a) shows the experimental system for adiabatic microwave heating. The microwave adiabatic heating system uses two 3-port circulators. The system contains the power generator which produces 3000 watts of the power. Waves propagate from the generator into the first port of the first 3-port circulator as shown in figure 54(a), this 3-port circulator prevents the reflected wave from damaging the source. A power meter (PM-1) is then connected to the second port of the first circulator to monitor the input power. A dummy
Figure 61: Relation between operating frequency and power for the magnetron [56]
load (DL-1) is connected to the third port of this circulator to absorb the reflected energy. The first port of the second 3-port circulator is connected after the power meter (PM-1). A four screw tuner is then connected to the second port of the second 3-port circulator serving as a reflector which reflects the wave into the third port of the second 3-port circulator. This reflected energy is the input power for microwave heating experiments. A second power meter (PM-2) is then connected to the third port of the second 3-port circulator to monitor the input power in microwave heating. A 24 inches waveguide which is used in network impedance measurements is then connected to the second power meter and shorted at the end with a small hole for the temperature measurement probe. However, the 4 screw tuner may not reflect all the incoming wave; therefore, a second dummy load (DL-2) is connected to absorb the forward wave at the end of the tuner.

The microwave power is controlled by the magnetron current; however, changing the power output, i.e., changing magnetron current, causes a change in the operational frequency. Thus, the microwave power is set to the high end which means the frequency is centered at 2.45GHz and the signal has a narrow band width [56]. The advantages of using this system is that the power output from the generator can be kept constant which results in a constant frequency output for microwave heating. It is very important to have constant frequency since the optimal design for power absorption is done at a single frequency. Furthermore, by adjusting only one screw, the power can be changed to a desired level without changing the operating frequency, which gives important advantages for good impedance matching operation and resonator operation. The microwave power
output from the generator was then set at 1600 watts but only 100 watts was delivered to the heating waveguide.

5.5.2 Microwave Heating Results

A Luxtron 755 fluoroptic thermometer was used to measure the heating element temperature during microwave adiabatic heating. Due to the fast initial heating rate, a non-contact measurement was used. This technique involves a coating process in which a phosphor powder and a binder was used to coat the sample surface. Usually, a small amount of phosphor powder (0.02g) is mixed with 4 drops of binder. The sample is coated with this mixture on one side. A fiber optical probe is placed at a distance 3mm from the sample during heating. The main advantage of this remote temperature measurement is that the temperature probe response time is much faster than for typical contact temperature measurements. The coating was always applied at the center of the sample to achieve the best reading. A typical non-contact heating temperature curve is shown in figure 62. There are two series of curves, one with contact measurement and one with non-contact measurement. It is clear that the non-contact measurement has a very short response time compared to the contact method. Note that at the end of heating, these two curves give the same results. A non-contact heating curve is then selected to find the heating rate (slope of temperature curve). The range of the selection is based on the initial heating rate. It is difficult to define "initial"; therefore, a maximum temperature was used as a limit to restrict this range. Previous studies showed that HCl doped polyaniline still
Figure 62: Comparison between contact and non-contact temperature measurement

\[ y = 25.055x - 0.8731 \]
\[ R^2 = 0.9924 \]
retains its conductivity within the same order of magnitude of the room temperature value up to 150°C [57]. Thus, the temperature limit was selected at 120°C. The insert picture in figure (62) shows the selected range and linear regression line. The plotted slope is strongly correlated to the data points. Once the heating slope is obtained, equation (5-34) is used to calculate the initial heat generation rate. The density and specific heat of the heating elements are determined by the rule of mixture as discussed in chapter 4. Figure 63 shows typical heating rate as a function of polyaniline concentration in the heating element. It is clear that increasing the polyaniline loading in the element increases the heat generation rate, and all of the linear regression curves agree well with the data points. Figure 64 shows a comparison of initial heat generation rate between the equivalent circuit model and the temperature measurement results as a function of polyaniline concentration in the heating element. All samples were first analyzed using the network analyzer and then heated by microwave radiation. Both results agree well for the initial internal heat generation rate. It is noticed that the 50% PANI element has almost the same initial heat generation rate as compared to 60% PANI element. It is found that at elevated temperature the 60% loading element has better heating ability than that of 50% PANI element. Figure 65 shows a comparison of the power absorption between the equivalent circuit model and the temperature measurement results as a function of the heating element thickness. It was found that thicker elements has less initial power absorption than
Figure 63: Typical heating rate as a function of polyaniline concentration
Comparison Between Model and Experimental Measurement

Figure 64: Comparison of initial heat generation rate between the equivalent circuit model and experimental measurement as a function of polyaniline concentration
Figure 65: Comparison of initial heat generation rate between the equivalent circuit model and experimental measurement as a function of composite thickness
thinner element for both 50% and 60% PANI elements. The reason for that was explained in the previous section by using the magnitude of the reflection coefficient and equation (5-28). The magnitude of the reflection coefficient for 50% PANI samples, 5014 and 503, was 0.979 and 0.987 respectively. Therefore, sample 5014, the thinner one, has higher power absorption. It is the same situation for the 60% PANI composite where the magnitude of the reflection coefficient for sample 604 (0.41mm) and 6011 (0.69mm) are 0.978 and 0.986, respectively. Thus, sample 604 has a higher power absorption.
CHAPTER 6

SINGLE MODE MICROWAVE WELDING

6.1 Introduction

Multi-mode microwave welding of thermoplastics has been demonstrated in Chapter 4 with superior joint strength; however, the heating time is relatively long compared to some of the welding techniques. In order to make microwave welding a more competitive joining methods, the heating time must be reduced. There are several methods which can reduce the heating time such as using a higher microwave power, using more lossy composites, or increasing the power transfer efficient. The peak heat generation rate of a 60% PANI composite in a multi-mode microwave is on the order of $2 \times 10^8$ w/m$^3$ as shown in figure 38; thus, the peak power absorption is only about 4 watts. Compared to the power delivered to the cavity, i. e., 600 watts, the energy transfer efficiency is extremely low. Since it is not an easy task to manipulate the PANI material properties to become more lossy, increasing the microwave power is the simplest method to reduce the heating time. However, the energy transfer efficiency remains the same if no other changes are made to the welding system. Consequently, by arranging the sample
orientation parallel to the electric field direction is one of the way to improve the power absorption efficiency. The only possibility to know the electric field direction is through a wave guide system in which the microwave propagation is restricted in a metallic conductor or waveguide system. Hence the wave patterns can be solved by using Maxwell’s equations and the field pattern is no longer a mystery. The single mode microwave system is one which satisfies the above requirements.

6.2 System Description

A single mode microwave heating system consisting of a microwave power source, a controller, a 3-port circulator, a 4-stub tuner, an applicator, two dual power meters and two dummy loads as shown in figure 66 was used. The microwave power source uses a magnetron to deliver 3000 Watts of power (Gerling Laboratories GL-103A) operating at 2450±50 MHz. It connects to a 3-port circulator which prevents the reflected wave form damaging the source. A 4-stub tuner is used to match the impedance between the source and the load. One dual power meter is located between the circulator and tuner to monitor the forward and reflected power. Welding is done in the double slotted applicator which is connected to the tuner. Another power meter and dummy load are connected after the applicator to absorb the transmitted power. Water is used to cool the power source and the dummy loads.

Normally, the system operates in a traveling wave pattern. However, the second power meter and dummy load can be replaced by a solid plate to generate a standing wave
Figure 66: Schematic representation of a single mode microwave welding system
pattern. One advantage of using this system compared to a multi-mode system is that the electromagnetic field inside the applicator can be calculated; thus, the sample can be aligned parallel to the electric field direction. Therefore, maximum heating efficiency can be achieved. The field distribution can be calculated by using Maxwell’s equations with the associated boundary conditions. Due to the dimensions of the waveguide, the number of wave modes inside the applicator is limited. The system used in this study is a WR 284 rectangular waveguide which has a cross section of 72 mm x 34 mm.

Waveguides are always used for high power transmission mainly because of the enclosed metallic structure which provides no energy leakage and low attenuation during transmission. One special property for the waveguide system is that it is a high-pass filter since transmission is possible when the wave length (\( \lambda \)) is smaller than the cut-off wave length (\( \lambda_c \)). In other words, for the wave to travel inside the waveguide, the frequency must be larger than the cut-off frequency (\( f_c \)). The cut off wave length is two times the broad waveguide dimension which is 144 mm in the air-filled case, and the cut-off frequency is 2.08 GHz. The wave pattern inside the waveguide is controlled by the waveguide geometry, filling media, and operating frequency. For an air-filled WR 284 waveguide operating at 2.45GHz, the dominant mode existing in the waveguide is \( \text{TE}_{10} \) mode. In order to understand the field pattern, it is necessary to solve Maxwell’s equations, which are:
\[ \nabla \cdot B = 0 \]
\[ \nabla \cdot E = \rho \]
\[ \nabla \times H = J + \frac{\partial D}{\partial t} \]  \hspace{1cm} \text{(Eq. 6-1)}
\[ \nabla \times E = -\frac{\partial B}{\partial t} \]

where \( B \) is magnetic flux density (tesla), \( E \) is electric field intensity (volt/meter), \( \rho \) is charge density, \( H \) is magnetic field strength (ampere/meter), \( J \) is current density (ampere/meter\(^2\)), \( D \) is displacement current (coulomb/meter\(^2\)). Equation (6-1) can be simplified for the case when no free charge is present so that \( \rho = 0 \) and assuming the field is harmonic in both time and distance. The general solution can be obtained using the boundary conditions which are tangential components of electric field vanish at the waveguide wall surfaces, at the side wall, \( E_y = 0 \) at \( z = 0 \) and \( z = z_l \), and at the top and bottom walls, \( E_z = 0 \) at \( y = 0 \) and \( y = y_l \), as shown in figure 67. The general solutions for \( \text{TE}_{mn} \) mode are as following,

\[
H_x(x, y, z, t) = H_0 \cos\left(\frac{n \pi y}{y_l}\right) \cos\left(\frac{m \pi z}{z_l}\right) e^{-\gamma x} \\
H_y = \frac{\gamma H_0}{k^2} \frac{n \pi}{y_l} \sin\left(\frac{n \pi y}{y_l}\right) \cos\left(\frac{m \pi z}{z_l}\right) e^{-\gamma x} \\
H_z = \frac{\gamma H_0}{k^2} \frac{m \pi}{z_l} \cos\left(\frac{n \pi y}{y_l}\right) \sin\left(\frac{m \pi z}{z_l}\right) e^{-\gamma x} \\
E_x = 0 \\
E_y = \frac{\gamma Z_{yy} H_0}{k^2} \frac{m \pi}{z_l} \cos\left(\frac{n \pi y}{y_l}\right) \sin\left(\frac{m \pi z}{z_l}\right) e^{-\gamma x} \\
E_z = -\frac{\gamma Z_{yz} H_0}{k^2} \frac{n \pi}{y_l} \sin\left(\frac{n \pi y}{y_l}\right) \cos\left(\frac{m \pi z}{z_l}\right) e^{-\gamma x}
\]
WR 284 Waveguide, $z_l = 72\text{mm}, y_l = 34\text{mm}$

Figure 67: WR 284 waveguide geometry
where \( m \) and \( n \) represent the number of half \( \sin \) variations along the \( Z \) and \( Y \) direction, respectively, and

\[
\gamma = \text{Propagation constant} = \alpha + i\beta
\]

\[
\gamma = \sqrt{\left(\frac{n\pi}{y_1}\right)^2 + \left(\frac{m\pi}{z_1}\right)^2} - \omega^2 \mu \varepsilon
\]

\[
k = \sqrt{\left(\frac{n\pi}{y_1}\right)^2 + \left(\frac{m\pi}{z_1}\right)^2}
\]

\( Z_{yz} = \text{Transverse wave impedance} \quad (\text{Eq. 6-3}) \)

\[
Z_{yz} = \frac{\omega \mu}{\beta} = \frac{376.7}{\sqrt{1 - \left(\frac{\lambda_0}{\lambda_c}\right)}}
\]

\( \lambda_0 = \text{wavelength in unbounded medium} \)

\( \lambda_c = \text{cutoff wavelength} = \frac{2\pi}{k} \)

For specific \( \text{TE}_{10} \) mode, where \( m = 1 \) and \( n = 0 \), the equation (6-2) reduces to

\[
E_x = 0
\]

\[
E_y = \frac{\gamma Z_{yz} H_0}{k^2} \frac{\pi}{z_1} \sin \frac{\pi z}{z_1} e^{-\gamma x}
\]

\[
E_z = 0
\]

\[
H_x = H_0 \cos \frac{\pi z}{z_1} e^{-\gamma x} \quad (\text{Eq. 6-4})
\]

\[
H_y = 0
\]

\[
H_z = \frac{\gamma H_0}{k^2} \frac{\pi}{z_1} \sin \frac{\pi z}{z_1} e^{-\gamma x}
\]

\( e^{i\omega t} \) is omitted

The \( H_0 \) is the amplitude of the wave and it is proportional to the input power. As shown in equation (6-4), the only non-zero electric field is \( E_y \). Figure 68 shows a plot of the instantaneous electric field with respect to an arbitrary power input. It is found that the
Figure 68: Electric field distribution for an air filled waveguide with TE$_{10}$ mode at 2.45 GHz.
electric field is a function of location and time. The electric field has a half sinusoidal variation across the z direction and a sinusoidal variation along the propagation direction and it has no variation in the y direction. The maximum electric field in the Y-Z plane is located at the center of the waveguide. Figure 69 shows an instantaneous 3-D plot of the electric field distribution for two wave lengths along the waveguide at 2.45 GHz. The above solution is the traveling wave solution which means that the waveguide length is infinitely long, i.e., no reflected wave.

As mentioned before, the electric field strength is associated with the input power.

The power transmitted through the waveguide is given by

\[ P_{\text{wg}} = \frac{1}{2} \left( \mathbf{E} \times \mathbf{H}^* \right) \cdot ds \quad \text{(Eq. 6-5)} \]

Therefore, for a WR 284 waveguide, the power delivered into the guide is

\[ P_{\text{wg}} = \text{Re} \left[ \frac{1}{2} \int_0^{y_1 \lambda_l} \int_0^{z_1} \left( \mathbf{E} \times \mathbf{H}^* \right) \cdot d y \, d z \right] = \frac{1}{4} \frac{E_y^2 \beta}{\omega \mu_0} y_1 \cdot z_1 \quad \text{(Eq. 6-6)} \]

Rearranging equation (6-6), the peak electric field strength can be found as

\[ E_y = \sqrt{\frac{4 P_{\text{wg}} \omega \mu_0}{\beta \cdot z_1 \cdot y_1}} \quad \text{(Eq. 6-7)} \]

The phase constant, \( \beta \), can be found from equation (6-3). Equation (6-7) shows that the electric field strength is proportional to the square root of the input power. Thus, increasing the input power results in increasing the electric field. Figure 70 shows the
For air filled S band, \( f = 2.45 \text{GHz} \), \( (\lambda/4) = 58 \text{mm} \)

Figure 69: Three dimensional plot of the electric field distribution for a TE_{10} mode
Figure 70: Electric field strength as a function of input power for traveling wave
calculated and the measured electric field strength as function of input power in the traveling wave condition. The electric field strength was measured by using a Luxtron E-Field probe (MEF-1.5) and the maximum measurable electric field strength was 42KV/M. It contains one pre-calibrated susceptor temperature sensor and one reference temperature sensor and by measuring the temperature differences between the two, the electric field strength can be determined. Figure 71 shows the electric field strength along the waveguide which was measured at 400 watts of the power in traveling wave condition. It was found that the voltage standing wave ratio (VSWR) equals to 1.12 which indicates the reflection from the dummy load is acceptable and the electric field strength is quite uniform. In general, a traveling wave is used in heating of large structures and in continuous processing. Another basic operation in a single mode microwave system is the standing wave pattern. The standing wave can be generated by removing the second dummy load (as shown in figure 66) and replacing it by a solid plate (shorted plate) which reflects the wave, generating an interference between the forward and the reflected wave. Figure 72 shows the electric field strength measured at 100 watts input power in the standing wave condition. It is noted that the electric field strength is a half sinusoidal variation. For a standing wave, the first minimum electric field occurs at the shorted plate, and the consecutive minimum occurs at a distance of $\lambda_d/2$ away from the shorted plate. The first maximum electric field is located at a quarter wave length away from the shorted plate and the consecutive maximum occurs at a distance of $\lambda_d/2$ from the first maximum.
Figure 71: Measured electric field strength at 400 watts traveling wave
Figure 72: Measured electric field strength at 100 watts standing wave
The maximum electric field location will not change with time but the amplitude will vary with time. The standing wave pattern has uneven field distribution which results in non-uniform heating. However, it is beneficial for welding of thermoplastic using conducting composite because it enables maximizing the field strength at the interface thereby producing very localized heating.

6.3 Welding Aperture

As mentioned before, increasing the microwave power, i.e., increasing the electric field strength, reduces the heating time in microwave welding; therefore, the heating composite should be placed at a quarter wave length from the shorted plate. However, there are three possible orientations to place the sample in the waveguide as shown in figure 73, the bottom diagram represents the interaction between heating composite and incoming electric field. The boundary conditions for electromagnetic wave are that the normal displacement current should be continuous and the tangential electric component should be continuous. Due to the large dielectric constant of the heating element, if the sample is placed as orientation 1 in figure 73, that results in a very small electric field inside the gasket. If the sample is placed as orientations 2 and 3 in figure 73 that should have higher and equal electric fields strength. Figure 74 shows the effect of orientation on adiabatic heating of the 60% PANI composite for 100 watts traveling waves. The sample that was perpendicular to the incoming electric field experienced less heating than the ones
Figure 73: Microwave welding orientation in waveguide
Figure 74: Effect of orientation on adiabatic heating of 60% PANI composite in waveguide system
that were parallel to the incoming electric field. There is no difference in heating ability between two parallel samples. Therefore, orientation 1 was eliminated in fixture design. The use of orientation 3 needs hole drilling on the side walls which causes the microwave to radiate; therefore, orientation 2 was selected as the sample orientation.

A fixture similar to the one used in multi-mode microwave welding was constructed with the sample interface located at quarter wave length ($\lambda_g$) from the shorted plate. As shown in figure 72, the first electric peak is located at 58mm from the shorted plate for an air filled waveguide; thus, the joint interface was designed at that position. However, the waveguide wave length was affected by the dielectric constant of the sample holder (HDPE). Therefore, the electric field distribution was changed and it is necessary to re-measure with the sample holder and the sample in the waveguide to determine the quarter wave length location during welding. Figure 75 shows the measured electric field strength as a function of location inside the waveguide. It was found that the sample holder reduces the waveguide wave length; thus, the new location for the joint interface is at 38mm from the shorted plate. A HDPE bar through the waveguide and sample holder was used to hold the sample holder position during the pressuring stage. Also, a small hole was drilled on the shorted plate to apply the pressure.

There are two methods to control the microwave system operation. First, is through the “push button” which was provided by the system controlled. Second, is remote control which uses an external current or voltage to control the power level and a voltage source to control the microwave power on/off. For most of the test, the first
Figure 75: Electric field strength distribution in waveguide with sample and fixture
method was used. However, the controller does not provide a digital timer to control the heating time. Therefore, two external relays were installed, one controls the heating time and the second controls the pressure. There are eight functions on the relay which provides for more versatility in operating the system. For example, if the post heating time is 10 seconds, the heating timer can be set at 10 seconds in mode 1, and the pressure timer and be set at 10 seconds in mode 3. During welding, the heating timer will on for 10 seconds and turned off; however, the pressure timer will have a delay 10 seconds and then turn on to trigger the solenoid valve and turn on the pressure. For the constant pressure method, both timer can be set at the same modes. However, the timer operation can only be used in one pre-set power level. The second control method is through a computer with a data acquisition board (Metrabyte DAS-20). The DAS-20 provides a DC signal at 3.15 volt which can turn the microwave on and off. Further, the DAS-20 has the digital to analog function which provides a variable voltage output to determine the microwave power. The data acquisition program that was developed is listed in Appendix D. The advantage of using a computer to control the microwave system is that multi-step power levels can be used during heating. When heating with polar polymers, such as nylon, the loss tangent increases dramatically at Tm. Therefore, it is beneficial to reduce the power level at elevated temperature to reduce the heating rate and prevent thermal run-away.
6.4 Welding of HDPE

6.4.1 Heating Results

The heating elements and samples used in single mode microwave welding is exactly the same as in multi-mode microwave welding. As mentioned before, placing the gasket in parallel with the electric field increases the power transfer efficiency and increasing the microwave power reduces the welding time. Figure 76 shows the adiabatic heating results of a 40%, a 50% and a 60% PANI composite using 100 watts standing wave condition. It is clear that the higher PANI concentration provides faster heating and higher temperatures. It should be noted that the power level in this case is only 100 watts while the maximum microwave power that is available is 3000 watts. Thus, high microwave power will reduce the heating time dramatically during welding. Figure 77 shows the temperature history of the gasket/sample interface during welding for single mode (standing wave) and for multi-mode (600W) conditions. It is clear that higher power levels result in higher temperatures and faster heating rates (slope of the temperature profile) at the joint interface. It is noted that in less than 5 seconds the temperature at the interface exceeds 200°C. This may shorten the welding cycle dramatically. The heating rate changes with increasing temperature due to an irreversible loss in electromagnetic absorption which is caused by loss in polyaniline conductivity at high temperatures. As discussed in multi-mode microwave welding, the internal heat generation rate during welding can be calculated from FEM using the temperature measurements. Figure 78 shows the FEM predicted internal heat generation rate using 60% PANI composite and
Figure 76: Adiabatic heating results of 40%, 50%, and 60%, PANI, 0.5mm thick composites at 100 watts standing wave
Figure 77: Temperature histories at gasket/sample interface during welding
Figure 78: Heat generation rate for 60% PANI, 0.5 mm thick gasket using 1800 watts standing wave
1800 watts standing wave. It was found that the maximum heating rate is $6.5 \times 10^8$ w/m$^3$.

In order to verify the accuracy of the FEM model, a second temperature probe was placed at 1.54mm away from the interface to measure the temperature during welding. Figure 79 shows the measured and the FEM predicted temperature profile. Due to the finite diameter of the temperature probe, the predicted temperature for two adjacent nodes is plotted. It was found that the FEM prediction is in good agreement with the temperature measurement. Figure 80 shows a comparison between multi-mode 600 watts and single mode 1800 watts internal heat generation using 60% PANI composite. It was found that use of single mode 1800 watts provides very high initial heat generation rate than the multi-mode 600 watts which reduces the welding cycle dramatically.

6.4.2 Welding Results

Welding of HDPE was performed in a single mode microwave applicator as described in the previous section. Figure 81 shows the effect of heating time and power on joint strength for single mode microwave welding using 60% PANI 0.5mm gasket. It is clear that 2400 watts provides faster heating and a shorter welding cycle. It is noted that at 15 seconds, the joint strength is $26.50 \pm 0.74$ MPa which is 96% of the bulk material strength ($27.58 \pm 0.32$ MPa). Previous studies shows that the joint strength can be increased by increase the welding pressure. Thus, by increase the welding pressure, the welding cycle can be on the order of 15 seconds with good joint strength. The maximum
Figure 79: Comparison between measured and FEM predicted temperature histories at 1.5mm from gasket/sample interface using 1800 watts standing wave
Figure 80: Comparison of internal heat generation rate between multi-mode 600 watts and single mode 1800 watts
Figure 81: Effect of heating time and power on joint strength using 60% PANI 0.5mm gasket in single mode
joint strength using 600 watts power was 25.63±0.55 MPa which is 93% of the strength of HDPE. Figure 82 shows a comparison of the joint strength between single mode and multi-mode microwave heating at 600 watts of power. Single mode heating results in stronger joints with shorter cycle time. Figure 83 shows the effect of concentration of PANI in the composite on joint strength. It was found that using 50% PANI loading gasket can provide a good joint quality using 20 seconds of heating time. The joint strength can be further increased either by increasing the heating time or increasing the joining pressure. Using a 30% PANI gasket with 20 seconds of heating time results in 55% of the joint strength. Therefore, increasing the heating time was attempted. Figure 84 shows the effect of heating time using 30% and 60% PANI composite. It was found that increasing the heating time for the 30% concentration PANI composite does not improved the joint strength significantly. The reason for that is because too little heat was generated in the 30% PANI composite even with long heating time. The only way to improve this is by increasing the energy transfer efficiency. As shown in figure 66, there is a 4 screw tuner between applicator and generator which can change the impedance of the heating section to match the characteristic impedance of the waveguide. Therefore, the impedance can be changed by turning the screws. A preliminary study showed that 85% joint strength can be obtained by using 10% PANI composite with 800 watts and 30 seconds of heating time. Tuning the microwave system is the most efficient way in reducing the heating time. More efforts should be given to this area, especially for disassembly applications where less PANI composite is present at the joint interface.
Figure 82: Comparison of joint strength between single mode and multi-mode microwave welding using 600 watts, 60% PANI 0.5mm gasket
Figure 83: Effect of polyaniline concentration on joint strength

2400W, 20 seconds, 0.69 MPa

Weight % of Polyaniline
Figure 84: Effect of heating on joint strength using 30% and 60% PANI composite
As demonstrated in multi-mode microwave welding, joining of nylon using PANI composite results in 60% of the joint strength which was also attempted in single mode microwave. However, due to the mismatch in material properties, the joint strength is still low. Therefore, a nylon and PANI composite should be used to improve the joint quality.

6.5 Welding of Nylon 6/6

6.5.1 Introduction

Welding of Nylon is different from welding of HDPE because a different conducting composite heating element is needed and nylon contains polar groups. The polar groups heat up under microwave radiation without the need for a heating element. Thus, selecting an appropriate heating time is crucial in order to prevent over heating and thermal run-away. The conducting composite in this case were conductive polyaniline/nylon films, which were cast on Pyrex glass by doping polyaniline base with d,l-camphorsulfonic acid (HCSA) in m-cresol solution [58,59]. The films can be made with different polyaniline concentrations to obtain different conductivity, and the thickness can also be controlled to provide the required dimension. The concentration of the PANI in the films can be controlled from 0%, i.e., pure nylon film, to 100%, i.e., pure PANI film. The HCSA serves as a primary dopant and m-cresol serves as a secondary dopant with a combination of these two producing a conductivity increase by several orders of magnitude. The presence of m-cresol changes the conformation of the polyaniline from
compact coils to expanded coils even after removal of the m-cresol. There are several advantages in using these films such as, compatibility with the bulk material, high conductivity, low PANI concentration and film flexibility. It has been reported that pure polyaniline film can have conductivity up to 400 S/cm [59]. However, the experimental conditions in film processing affect the conductivity dramatically [58,59,60]. For example, processing time, residual m-cresol in the film, drying time, drying temperature and storage time will all affect the final conductivity. It was found that processing parameter, especially timing, affect the conductivity dramatically. The detailed procedure for making the conducting films is classified for discussion at the present time. Therefore, the procedures are omitted.

6.5.2 Welding Aperture

The films used in this study were: Nylon with 4.57% PANI by weight with 0.05 mm in thickness, Nylon with 9.72% PANI by weight with 0.025, 0.05 and 0.075 mm in thickness and Nylon with 17.72% PANI by weight with 0.013 mm in thickness. The effect of heating and re-heating of these films, effect of heating time, effect of concentration and effect of film thickness on joint strength were studied using the single mode microwave system. The microwave system and welding experimental setup were discussed in Chapter 6.3. The adiabatic temperature rise in the films and the temperature increase at the joint interface were measured using a Luxtron 755 fiber optical temperature

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measurement system. The nylon6/6 bar had the dimensions of 6.35 x 6.35 x 50 mm. The nylon6/6 bars were dried in a vacuum oven at 90°C for 72 hours before welding. A fixture was made from HDPE to hold the nylon bars and a 9.5 mm circular hole was drilled in the shorted plate to apply the pressure. As discussed in section 6.3, orientation of the film in the waveguide affects the heating result dramatically. Therefore, the heating element has to be placed parallel to the incoming E-field in order to have better heating efficiency. Also the joint interface has to be at a quarter wave length from the shorted end in a standing wave pattern.

6.5.3 Results

The temperature rise in the film is always of concern in microwave welding. Figure 85 shows the temperature histories of a 4.57%-0.05 mm film in a traveling wave condition. Curve 1 represents the adiabatic heating temperature history under 100 watts; curve 2 and 3 are the re-heating curves (same film) at 100 watts but in different orientations see the insert in figure 85. Curve 4 is the re-heating temperature profile using the same film but at 500 watts. It is noted that after the first heating the conductivity of the film was reduced; however, it remained at a high enough level to achieve the consecutive heating (curve 2 and 3). When the power level is increased (curve 4), the film reaches higher temperature which is essential for some recycling applications. For example, in order to disassemble a welded structure, one must first: make the joint and second: reheat the sample to separate the joint. The embedded film at the joint interface not only provides the heat required during welding but also generate the heat for disassembly. Figure 86
Figure 85: Temperature histories of a 4.57%, 0.05mm nylon film in a traveling wave condition
Figure 86: Adiabatic heating results of a 17.72%,0.013mm thick nylon film
shows the adiabatic re-heating results for 17.72%-0.013mm film. It shows the same phenomena as 4.57% film even with five heating-cooling cycle.

The heating rate and the temperature profile are related to the conductivity of the film which is related to the polyaniline concentration in the film. Figure 87 shows the heating results of the films in different concentrations at 100 watts in the traveling wave condition. It shows that increasing the PANI concentration in the film results in a higher temperature and faster heating rates. It is noted that 17.72%-0.013 mm reduces its heat generation at elevated temperatures which prevents thermal run away. Figure 88 shows the effect of film thickness on heating using 9.72% film and a 100 watts traveling wave. It shows that increasing the film thickness increases the heating rate and temperature. One aspect that should be addressed here is that too high or too low a conductivity of the film will reduce the heating rate and temperature. Figure 89 shows the adiabatic heating of PANI nylon films from another film processing date. The PANI concentration in the films was varied from 0.8% to 50%. The best heating films were 1.6% and 7.7% PANI. The 0.8% and 50% PANI films resulted in low heating rate and low temperatures. This means that there is an optimal concentration for microwave heating. As mentioned before, the conductivity depends on the processing parameters. Therefore, the optimal concentration range may be different depending on the processing parameters. Figure 90 shows the temperature increase at joint interface (ΔT) without films for different power levels under standing wave condition. When the power is low, the temperature at the interface is close
Figure 87: Effect of PANI concentration in films on adiabatic heating
Figure 88: Effect of film thickness on adiabatic heating using 9.72% nylon film
Figure 89: Effect of PANI concentration (0.8% - 50%) in the film on adiabatic heating
Figure 90: Temperature increase at joint interface without film as a function of power level using nylon sample
to the bulk temperature; however, at higher power levels, the temperature rises dramatically when the bulk temperature exceeds 100°C. This phenomenon is the reason for thermal runaway. It is crucial to control the heating time in microwave welding. This is one of the reasons that the power level is controlled by computer which can change the power immediately. Figure 91 shows the interface temperature history under different powers with the 4.57%-0.05mm film inserted. It is clear that low power requires longer time to reach the melting temperature (~250°C), in order to shorten the welding cycle, high power is preferred. It is noted that for 2000 watts of power in less than 8 seconds the temperature already exceeded 300°C which indicates a short welding cycle is possible. The changes in heating rate reflect the changes in the conductivity of the film at elevated temperature. This irreversible loss in electromagnetic absorption is caused by loss of both primary and secondary dopant in the film.

Figure 92 shows the effect of heating time on joint strength using 4.57%-0.05 mm film at 2000W, 1 MPa joining pressure, and standing wave condition. At 10 seconds of heating time the average joint strength is only 50% of the bulk material strength (73.93±1.37 MPa). Increasing the heating time to 14 seconds, the joining strength was 87% of the bulk material strength. However, dripping of molten bulk material occurred at this heating time due to the high temperature and low viscosity at the joint interface. Figure 93 shows the effect of polyaniline concentration in the film on joint strength at 2000W, 10 seconds of heating and 1 MPa of joining pressure.
Figure 91: Temperature increase at joint interface with 4.57%, 0.05mm film as a function of power level.
Figure 92: Effect of heating time on joint strength using 4.57%, 0.05mm thick film at 2000 watts, 1 MPa joint pressure
Figure 93: Effect of PANI concentration on joint strength using 2000 watts power
The thickness of the films is 0.05 mm except for 17.72% that is only 0.013 mm. Under this condition, the best joining result was $71.45 \pm 2.64$ MPa which is 97% of the bulk material strength using 17.72% film. Figure 94 shows the effect of the film thickness on joint strength using 9.72%, 2000W and 1 MPa pressure. Increasing film thickness from 0.05 to 0.075 mm decreases the joint strength. One possible reason for that is that the thicker film initial temperature increase is too fast and it losses its conductivity dramatically and produces less heat subsequently; therefore, total heat generation is lower resulting in lower joining strength. As mentioned before, the conductivity of the film varies by storage time, the film used in this study were stored for one month after film processing. When using a fresh film, 96% of the joint strength can be reached by utilizing 4.57%-0.05mm film and 10 seconds of heating time at 2000 watts. Further study is needed to understand this phenomenon completely.
Figure 94: Effect of film thickness on joint strength using 9.72% PANI loading and 2000 watts and 1 MPa joint pressure.
The use of plastics and their composites in structure applications is rapidly growing due to the many advantages that these materials offer. However, the use of these materials is often limited by the ability to quickly produce high quality joints with good repeatability and predictable properties. Therefore, it is critical to the development and usage of polymers and polymeric composites that new and faster joining techniques be developed. Intrinsically conducting polymer, such as polyaniline, offers this opportunity in developing new joining technologies. These polymers, like semi-conductors, conduct electricity through doping. This enables the designer to manipulate the electrical properties of the material specifically for the application like for joining. Microwave energy has been used in drying and heating of materials for many years. Industrial applications of microwave heating and drying include paper, printing, textile, wood, rubber, and plastic industries. The high penetration ability of the microwave energy provides fast heating and a short processing cycle in industrial applications. Therefore, the main objective of this work is to develop a novel joining technique which combines microwave energy and
intrinsically conducting polymers. The effect of welding parameters on joint strength were studied and an equivalent circuit heating model was also constructed to predict the power absorption of the conducting composites. The finite element method was also used to calculated the heat generation during the welding process.

The conducting polyaniline powders which were synthesized from chemical processing cannot be used in welding directly; thus, a conducting composite containing conductive polyaniline and HDPE powder was made by microwave molding, ultrasonic molding, and compression molding. It was found that microwave and ultrasonic moldings were fast molding processes, but molding quality and consistency were worse than the compression molding. Therefore, the conductive composite gaskets were compression molded.

A feasibility study on resistance (0 - 60 Hz), induction (9 KHz - 4 MHz), radio frequency (27.12 MHz), and microwave heating (2.45 GHz) of conducting composites revealed that increasing the heating frequency provided faster heating rates and resulted in higher temperatures. Nevertheless, all of these heating methods can raise the gasket temperature to exceed 100°C, which is a favorable indication for future development. It was found that gasket with higher polyaniline concentration absorb more energy at microwave frequency; however, at radio frequency, an intermediate polyaniline concentration composite (40% in this case) absorbs more power. A more intensive study is needed to understand the phenomena.
Multi-mode microwave welding of HDPE was successfully demonstrated by placing the conducting composite at the joint interface. It was found that increasing the gasket thickness results in higher joint strength under constant pressure. Also, increasing the PANI concentration in the gasket results in higher joint strength. It was also found that increasing the welding pressure results in higher joint strength. However, the constant pressuring method squeezes out the heating composite in the middle of the heating stage; thus, less heating occurs in the latter stages. The maximum joint strength is 86% of the molded HDPE strength for both 50% PANI, 1mm thick gasket and 60% PANI, 0.5mm thick gasket.

A modified multi-mode microwave welding method with pressure application after heating, which improves the joint quality was also demonstrated. The pressure was applied by an air cylinder outside the oven after the heating stage (post heating pressuring method). The maximum joint strength was equal to the bulk strength of HDPE. It was found that post heating pressuring method not only increases the maximum joint strength but also reduces the heating time to reach a specific joint strength. The FEM calculation of the heat generation rate shows that 60% PANI, 0.5mm gasket provides faster heating rates and higher temperatures than the 50% PANI, 1mm gasket although the 50% PANI, 1mm gasket has more polyaniline powders. This reveals that the heating ability is determined by the intrinsic properties of the gasket not by the amount of polyaniline. Welding of other thermoplastics were also attempted using polyaniline and HDPE composites gasket. The joint strength depends on the compatibility of the HDPE and the
material being joined. For example, PP with the HDPE/PANI gasket produced a high joint strength but nylon has low joint strength. Therefore, in order to achieve the maximum joint strength, the polyaniline should be blended with the polymer being joined. Although the joint strength is high for HDPE, the welding cycle is still relatively long. Thus, increasing the power level and increasing the power transfer efficiency were attempted using a single mode microwave system.

A single mode microwave system has two operating patterns: traveling wave and standing wave. It was found that the standing wave provides higher electric fields than the traveling wave but with non-uniform field distribution. However, this non-uniform electric field becomes an advantage because localized heating is preferred in welding. The orientation of the heating composite inside the waveguide affects the heating rate. It was found that the heating element should be placed in parallel with the incoming electric field to achieve a higher heat generation. For the standing wave, the maximum heating location is at the quarter wave length from the shorted plate. Welding of HDPE was successful, reducing the heating time from 80 seconds in multi-mode to 15 seconds in single mode due to the higher power source and better energy transfer efficiency. The maximum heat generation rate using 1800 watts and 60%, 0.5mm PANI composite was estimated by FEM calculation to be $6.5 \times 10^8$ (W/m$^2$).

An equivalent circuit model representing the single mode microwave heating of a conducting composite was constructed to predict the initial power absorption (heat generation rate). It is in very good agreement with experimental temperature
measurements. In addition, the equivalent circuits model uses a single parameter, the impedance, to predict the power absorption, which is more convenient than computer simulations. Furthermore, the circuit model provides design guidelines for future development in conducting composites.

Single mode microwave welding of nylon6/6 using polyaniline and nylon film was also studied. The maximum joint strength was 97% of the nylon6/6 strength. It was found that a longer heating time causes temperature rise in the bulk material and thermal runaway; therefore, short heating times using PANI composites are preferred in welding of polar materials. Higher PANI concentration in the films provides faster heating rates and higher temperatures. A too high or too low PANI concentration in the films results in low heating rates and temperatures. An optimal concentration can be found either by adiabatic heating experimental measurement or by using the equivalent circuit model.

In the future, the microwave energy transfer efficiency could be improved by matching (tuning) the load impedance to the characteristic impedance of the waveguide. There are several ways to improve the matching such as changing the composite impedance, changing the waveguide dimension to change the characteristic impedance of the line, or to insert some dielectric into the waveguide. This can reduce the power consumption and equipment cost by using a smaller power source. The conducting PANI should be blended with other substrates to achieve better joint strength with other polymers, such as PC, PMMA. In fact, those blends have been developed by researchers and they are similar to the nylon film processing method.
The electrical properties of the heating element should be measured as function of temperature and time to understand the change in the conductive composite during welding. This will be critical to gasket development and design. Due to the fact that a 50%, 0.5mm PANI composite has less heating than a 60%, 0.5mm PANI composite at elevated temperatures, the equivalent circuits model should be extended to a higher temperature range for adiabatic heating measurements. Also, the new model should include the welding fixture and sample to predict the power absorption during welding. The temperature dependent impedance measurement can be achieved by using an infrared heater or hot gas chamber to raise the sample temperature during measurement. The calibration process during the measurement can also be modified by using an S-band waveguide instead of the APC-7 calibration set. This way, the tuning process can be achieved easily, thereby producing more efficient heating.

Furthermore, welding of a large and/or complex structure with multiple joints is impossible with other plastics welding methods. However, large microwave cavities and high power sources are already available, it may be easier to weld large structure using conductive PANI gaskets. This innovative and exciting approach of microwave welding using intrinsically conductive polyaniline will serve as the basis for a new future in welding of large and complex polymeric and polymeric composite structure.
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United States Patent, Patent No. 4711983


APPENDIX A

The Basic program for controlling the DAS-20 data acquisition board to measure the displacement during microwave welding
'DATA ACQUISITION FOR LVDT USING DAS20 MODE 4

'Sampling rate = 200/sec, total time = 20 seconds

'DATE: 3/18/92 by: CHUN-YUAN WU

DIM DIO%(10)
DIM DT%(10000), CH%(10) 'set up integer arrays for data/channel #

COMMON SHARED DIO%(), DT%(), CH%
DECLARE SUB DAS20 (MODE%, BYVAL dummy%, FLAG%)

'$DYNAMIC
DIM dat%(2000)
'$STATIC

----------- Initialize section -------------------------------

SCREEN 0, 0, 0: CLS : KEY OFF: WIDTH 80

200 'STEP 2: Initialize with mode 0 ------------------------

220 OPEN "DAS20.ADR" FOR INPUT AS #1 'get base l/o address
230 INPUT #1, DIO%(0)
240 DIO%(1) = 2 'interrupt level
250 DIO%(2) = 1 'DMA level
270 FLAG% = 0 'error variable
280 MD% = 0 'mode 0 - initialize
290 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
300 IF FLAG% <> 0 THEN PRINT "INSTALLATION ERROR": STOP'Halt on error

320 'STEP 3: Prompt for scan sequence and set using mode 1 ----------

340 'INPUT "Enter channel number : ", L%
350 'INPUT "Enter gain range : ", U%
360 'INPUT "Enter 2-first, 0-next, 1-last: ", C%
370 DIO%(0) = 0 'set channel 0
380 DIO%(1) = 1 'set gain range -10---+10V

208
390 DIO%(2) = 1  'set command last
400 MD% = 1  'mode 1 - set scan sequence
410 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
420 IF FLAG% <> 0 THEN PRINT "Error #"; FLAG%; " in setting scan sequence": STOP
430 IF C% <> 1 THEN GOTO 340
440 ' "
450 FILES = "c:\wu\disp.dat"
455 OPEN FILES FOR APPEND AS #2

456 '------ STEP 4: Set timer rate to trigger A/D using mode 24  "

460 'Alternatively you can externally trigger the A/D, in which case this step
470 'can be skipped (see Step 5).
480 ' "
490 'Setting timer to 200Hz
500 DIO%(0) = 5000  'you can set another rate here if you want,
510 DIO%(1) = 5  'this divides 5MHz by 5,000 (DIO%(0)
520 'if timer word 2 (DIO%(1))=0 then word not used
530 MD% = 24  'mode 24 - timer set
540 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
550 IF FLAG% <> 0 THEN PRINT "Error #"; FLAG%; " in setting timer": STOP
560 ' "

570 '------ STEP 5: Do 2000 conversions to array DT%(*)  "

580 N = 2000  'number of conversions required
600 DIO%(0) = N  'number of conversions required
610 DIO%(1) = VARPTR(DT%(0))  'provide array location
620 DIO%(2) = 2  'trigger source, 0=external on IP0
630 ' 1=internal timer with ext gate
640 ' 2=internal timer start as result
650 ' of executing mode 4
660 MD% = 4  'mode 4 - A/D to array program control..
670 'will not return from call until all conversions
680 'have been made.
690 'Note: If the timer is used as a trigger source then holding input IP0
700 'low will delay starting conversions.
710 PRINT "Performing "; N; " conversions. Please wait."
720 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
730 IF FLAG% <> 0 THEN PRINT "Error #"; FLAG%; " in mode 4": STOP
740 ' "
750 '------ Stop internal timer if running (not always necessary)  "
760 'Needed if Internal timer was select (dio%(2) was a 1 or 2 for Mode 4

209
770 MD% = 26
780 DIO%(0) = 0       'Stop A/D Timer
790 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
800 '

810 '----- STEP 6: Display results ------------------------------------------

820 'Alternatively here you could file them, turn data into real units etc.
830 PRINT "SAVING 4000 data points PLEASE WAIT!"
840 FOR I = 0 TO (N - 1)
     PRINT #2, USING "#####.#####"; I / 200;
850 PRINT #2, USING "#####.#####"; DT%(I) * 10 / 2048;
     PRINT #2, USING "#####.#####"; DT%(I) * 5 / 2048
860 NEXT I
870 PRINT : PRINT : END
APPENDIX B

The input file for ANSYS Finite Element Program to calculate the initial heat generation rate during microwave welding
1-D FEM-ANSYS-INTERNAL HEAT GENERATION RATE CALCULATION
WRITTEN BY: CHUNG-YUAN WU

/PREP7
/TITLE HEAT GENERATION FOR 50% 0.8g(1.0mm thick)

C***-----------------------------FILE:d508.DAT---------

C***-----------------------------------------------June, 1993
KAN,-1
ET,1,32

C***-------------------------MATERIAL PROPERTIES FOR COMPOSITE GASKET
R,1,0.0000403225
KXX,1,0.28
DENS,1,950

C***--------------------------MATERIAL PROPERTIES FOR HDPE BAR
R,2,0.0000403225
KXX,2,0.28
DENS,2,900

C***-----------------Cp vs. TEMP TABLE(DSC rule of mixture)(gasket)--MAT 1
MPTEMP,1,20,40,60,80,100
MPDATA,C,1,1,2200,2298,2511,2748,3123
MPTEMP,6,110,120,130,132,140
MPDATA,C,1,6,3379,4534,10766,11477,2881
MPTEMP,11,150,160,170,180,190
MPDATA,C,1,11,2917,3123,3653,4071,4404
MPTEMP,16,200,210,220,230,240,300
MPDATA,C,1,16,4230,4077,3692,3570,3313,3000

C***-----------------Cp for HDPE bar(DSC)-MAT-2
MPTEMP,1,20,40,60,80,100
MPDATA,C,2,1,2400,2474,2749,3052,3596
MPTEMP,6,110,120,130,132,140
MPDATA,C,2,6,4034,6220,18725,20294,3168
MPTEMP,11,150,160,170,180,190

212
MPDATA,C,2,11,2949,2968,2961,3019,3076
MPTEMP,16,200,210,220,230,240,300
MPDATA,C,2,16,3071,3157,3066,3158,3088,3000

C***-----------------------first 0.5"(0.010"/element)

n,1
N,51,0.0127
fill

C***-----------------------rest of the bar(0.100"/element)

n,52,0.01524
N,66,0.0508
FILL

C***------------------------ELEMENTS FOR GASKET

MAT,1
E,1,2
E,2,3

C***------------------------ELEMENTS FOR HDPE BAR

MAT,2
E,3,4
EGEN,63,1,3

C***----------------------I.C.
TUNIF,25
KTEMP,-1
KBC,1

C***------------------------LOADING
MPLIST,1,2,1,C
MPPLOT,C,1,0,20000
C***----------------------0-2
QE,1,16000000
qe,2,16000000
TIME,2
ITER,40,10,10
LWRITE
C***------------------------4sec
QEDELE,1,2,1
qe, 1, 29500000
QE, 2, 29500000
TIME, 4
ITER, 40, 10, 10
LWRITE
C***--------------------- 7 sec
QEDELE, 1, 2, 1
QE, 1, 40000000
qe, 2, 40000000
TIME, 7
ITER, 60, 10, 10
LWRITE
C***----------------------- 10 sec
QEDELE, 1, 2, 1
QE, 1, 45000000
qe, 2, 45000000
TIME, 10
ITER, 60, 10, 10
LWRITE
C***------------------------ 14 SEC
qedele, 1, 2, 1
qe, 1, 50000000
qe, 2, 50000000
time, 14
iter, 80, 10, 10
LWRITE
c***----------------------- 18 SEC
qedele, 1, 2, 1
qe, 1, 77000000
qe, 2, 77000000
time, 18
iter, 80, 10, 10
LWRITE
c***------------------------ 20
QEDELE, 1, 2, 1
QE, 1, 42000000
QE, 2, 42000000
TIME, 20
ITER, 40, 10, 10
LWRITE
C***------------------------26
qedele, 1, 2, 1
QE, 1, 43000000
QE, 2, 43000000

214
TIME,26
ITER,120,10,10
LWRITE
C***------------------------30
QDELE,1,2,1
QE,1,40000000
QE,2,40000000
TIME,30
ITER,40,10,10
LWRITE
C***------------------------40
qedele,1,2,1
qe,1,42000000
qe,2,42000000
time,40
iter,200,10,10
lwrite
C***------------------------43
qedele,1,2,1
qe,1,42000000
qe,2,42000000
time,43
iter,60,10,10
lwrite
C***------------------------48
qedele,1,2,1
qe,1,44000000
qe,2,44000000
time,48
iter,100,10,10
lwrite
C***------------------------53
qedele,1,2,1
qe,1,41000000
qe,2,41000000
time,53
iter,100,10,10
lwrite
C***------------------------
qedele,1,2,1
qe,1,43000000
qe,2,43000000
time,57
iter,80,10,10
lwrite
c***
qede,1,2,1
qe,1,39000000
qe,2,39000000
time,60
iter,60,10,10
lwrite
c***
AFWRITE
FINISH
/INPUT,27
FINISH
APPENDIX C

MathCad program to calculate the Transition impedance, Gasket Impedance and Gasket power absorption
load = 0 (short) coaxial impedance Z0 of waveguide waveguide wave length
ZL = 0 Z01 = 50 Z0 = 341.99 λg = 236.19

free space measured data
λ0 = 122.95 Zina = 0.375 + j 33.904 f = 2.44·10^9

dw1 = 0.152 dw2 = 0.0497 dc1 = 0.4615
dw1 = \frac{35.89}{λg} dw2 = \frac{11.73}{λg} dc1 = \frac{56.74}{λ0}

dw1 = 0.152 dw2 = 0.0497 dc1 = 0.4615
er = 2.365

\frac{3·10^{11}}{\sqrt{er}} = 79.9495 λ4 = 79.9495

Z02 = 60 - \frac{1}{er}\ln\left(\frac{10.95}{3.04}\right) dc2 = \frac{13.21}{λ4}

Z02 = 49.9975 dc2 = 0.1652

waveguide has attenuation of α coaxial line has attenuation α1=0, α2=0

α = 0.0034 Np/m β = 2π

γ = α + j · β γ1 = 0 + j · β γ2 = 0 + j · β

Zin1a = Z00ZL + Z0 · tanh(γ dw1) Zin2a = Z00ZL + Z0 · tanh(γ dw2)

Zin1a = 0.5292 + 483.0705i Zin2a = 0.0638 + 110.5702i

Zin12a = \frac{1}{Zin1a + Zin2a} Zin12a = 0.0605 + 89.81i

Zin3a = Z01 \frac{Zina - Z01 · tanh(γ1 · dc1)}{Z01 - Zina · tanh(γ1 · dc1)}

Zin3a = 0.5738 + 55.5383i Zin = 2.378 + j 70.6
Find the N type to waveguide losses($Z$)

if $Z$ is in series

$$Z_s = Z_{in4a} - Z_{in12a}$$

$$Z_s = 0.2069 - 99.9596i$$

We have two answers in the solution; therefore, find the correct answer is needed. By connecting 24" waveguide and short the circuit, $Z_s$ and $Z_p$ can be verified.

dw3 = total waveguide length from shorted side, with short $ZL=0$

$$dw3 = 24 \frac{25.4}{\lambda g} + dw1$$

$$Z_{dw3} = Z_0 \frac{ZL + Z0 \cdot \tanh(\gamma dw3)}{Z0 + ZL \cdot \tanh(\gamma dw3)}$$

$$Z_{dw3} = 275.1488 + 3.152 \cdot 10^3i$$

$$Z_{indw23} = \left( \frac{1}{Z_{dw3}} + \frac{1}{Z_{dw2}} \right)^{-1}$$

IF in series

$$Z_{in4s} = Z_{indw23} + Z_s$$

$$Z_{in4s} = 0.5789 + 6.656i$$

$$Z_{inde2} = Z02 \frac{Z_{in4s} + Z02 \cdot \tanh(\gamma2 \cdot dc2)}{Z02 + Z_{in4s} \cdot \tanh(\gamma2 \cdot dc2)}$$

$$Z_{inde2} = 3.7434 + 118.0675i$$

$$Z_{inde1} = Z01 \frac{Z_{inde2} + Z01 \cdot \tanh(\gamma1 \cdot dc1)}{Z01 + Z_{inde2} \cdot \tanh(\gamma1 \cdot dc1)}$$

$$Z_{inde1} = 1.585 + 66.8156i$$

IF in parallel

$$Z_{in4p} = \left( \frac{1}{Z_{indw23}} + \frac{1}{Z_p} \right)^{-1}$$

$$Z_{in4p} = 0.2607 - 9.9719i$$

$$Z_{in3p} = Z02 \frac{Z_{in4p} + Z02 \cdot \tanh(\gamma2 \cdot dc2)}{Z02 + Z_{in4p} \cdot \tanh(\gamma2 \cdot dc2)}$$

$$Z_{in3p} = 0.5644 + 55.9213i$$

$$Z_{inp} = Z01 \frac{Z_{in3p} + Z01 \cdot \tanh(\gamma1 \cdot dc1)}{Z01 + Z_{in3p} \cdot \tanh(\gamma1 \cdot dc1)}$$
Therefore Z is in series measured data=1.584+i 66.93

\[ \Gamma_N = \frac{Z_{\text{indcl}} - Z_01}{Z_{\text{indcl}} + Z_01} \]
\[ |\Gamma_N| = 0.9775 \]
\[ \text{arg}(\Gamma_N) = 73.5973 \text{ deg} \]

\[ |P_s| = 0.9775 \]
\[ \text{arg}(P_s) = 73.5973 \text{ deg} \]

\[ Z_{\text{inp}} = 0.3677 + 34.1539i \]
\[ \text{not matched with measured data} \]

Gasket Impedance Calculation

\[ Z_L = 0 \]
\[ Z_{\text{in6}} = Z_{\text{in5}} - Z_{\text{in6}} \cdot \tanh(\gamma_1 \cdot d_6) \]
\[ Z_{\text{in5}} = Z_{\text{in6}} - Z_{\text{in2}} \cdot \tanh(\gamma_2 \cdot d_5) \]
\[ Z_{\text{in4}} = Z_{\text{in5}} - Z_{S} \]
\[ Z_{\text{ind4}} = \frac{Z_L + Z_0 \cdot \tanh(\gamma d_4)}{Z_0 + Z_L \cdot \tanh(\gamma d_4)} \]
\[ Z_{\text{ind3}} = \frac{1}{\left( \frac{1}{Z_{\text{in4}}} - \frac{1}{Z_{\text{ind4}}} \right)} \]
\[ Z_{\text{in2}} = Z_0 - \frac{Z_{\text{in3}} \cdot \tanh(\gamma d_2)}{Z_0 - Z_{\text{in3}} \cdot \tanh(\gamma d_2)} \]

\[ Z_{\text{in6}} = 5.949 + 127.3995i \]
\[ Z_{\text{in5}} = 0.8131 + 8.0303i \]
\[ Z_{\text{in4}} = 0.6062 + 107.9899i \]
\[ Z_{\text{ind4}} = 0.0638 + 110.3202i \]
\[ Z_{\text{ind3}} = 1.1589 \cdot 10^3 + 4.8426 \cdot 10^3i \]
\[ Z_{\text{in2}} = 7.2386 + 209.2914i \]
\[ Z_{\text{in2}} = 1.5035 \cdot 10^3 - 8.1503 \cdot 10^3i \]

220
\[ Z_{in1} := Z_0 \frac{Z_L + Z_0 \tanh(\gamma d_1)}{Z_0 + Z_L \tanh(\gamma d_1)} \]

\[ Z_g = 1.3422 \times 10^3 - 8.2081 \times 10^3 i \]

\[ \sigma = 0.7761 \]

**Power calculation**

\[ Z_{in1p} := Z_0 \frac{Z_L + Z_0 \tanh(\gamma d_1)}{Z_0 + Z_L \tanh(\gamma d_1)} \]

\[ Z_{in2p} = \frac{1}{Z_g} + \frac{1}{Z_{in1p}} \]

\[ \Gamma_L = \frac{Z_{in2p} - Z_0}{Z_{in2p} + Z_0} \]

\[ Z_{in2p} = 1.5035 \times 10^3 - 8.1503 \times 10^3 i \]

\[ Z_{in1p} = 4.0234 \times 10^5 \]

\[ Z_{in2p} = 1.5035 \times 10^3 - 8.1503 \times 10^3 i \]

\[ \Gamma_L = 0.9819 - 0.0798 i \]

\[ |\Gamma_L| = 0.9852 \]

\[ Z_{imp} := Z_0 \frac{Z_{in2p} + Z_0 \tanh(\gamma d_2)}{Z_0 + Z_{in2p} \tanh(\gamma d_2)} \]

\[ P_a = 100 \]

\[ V_g := \sqrt{P_a \cdot 4 \cdot Z_0} \]

\[ V_0 := \frac{V_g}{2} \]

\[ V := V_0 e^{\gamma d_2} (1 + \Gamma_L) \]

\[ I := \frac{V}{Z_g} \]

\[ I = 0.0332 - 0.0286 i \]

\[ \Gamma_L = \text{Re}(V \cdot I) \]

\[ P_{ZL} = 2.5697 \]
Appendix D

The Basic Program for controlling the DAS-20 data acquisition board to control the single mode microwave welding
QBPOWER.BAS - Modes 7 D/A output 09/12/95
-10 VOLTS TO +10 VOLTS (-2048 TO +2048)
FOR REMOTE CONTROL OF MICROWAVE POWER
WRITTEN BY CHUNG-YUAN WU

DIM DIO%(10)
DIM DT%(1000), CH%(1000) 'set up integer arrays for data/channel #

COMMON SHARED DIO%(), DT%(), CH%()
DECLARE SUB DAS20 (MODE%, BYVAL dummy%, FLAG%)

'$DYNAMIC
DIM dat%(2000)
'$STATIC

'---------- Initialize section -------------------------------

SCREEN 0, 0, 0: CLS : KEY OFF: WIDTH 80

200 '------ STEP 2: Initialize with mode 0 -------------------------

220 OPEN "DAS20.ADR" FOR INPUT AS #1 'get base I/O address
230 INPUT #1, B%
240 DIO%(0) = B% 'base I/O address
250 DIO%(1) = 7 'interrupt level
260 DIO%(2) = 1 'D.M.A. level
280 FLAG% = 0 'error variable
290 MD% = 0 'mode 0 - initialize
300 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
310 IF FLAG% <> 0 THEN PRINT "INSTALLATION ERROR": STOP'Halt on error

320 '------ STEP 3: Output data to D/A channel 0 ---------- INPUT PARAMETERS ----

322 DIO%(0) = 0 'SET OUTPUT CHANNEL = 0

324 PRINT " TWO STAGES MICROWAVE POWER SETTING :"
325 PRINT
326 PRINT
332 INPUT " 1. ENTER FIRST HEATING TIME : ", T1
333 PRINT
334 INPUT " 2. ENTER SECOND HEATING TIME : ", T2

223
335 PRINT
'PRINT : COLOR 0, 7: PRINT " *** NOTE: (-205) = MIN. POWER, 0 MAX.
POWER ***": : COLOR 7, 0
336 PRINT
INPUT " 3. ENTER FIRST POWER LEVEL (100 - 0)% : ", K1 '100%= 0 VOLT,
0% = -1 VOLT
L1 = 2.04 * K1 - 204
IF (L1 + 205) < 0 THEN GOTO 336
IF L1 > 0 THEN GOTO 336
337 PRINT
339 INPUT " 4. ENTER SECOND POWER LEVEL (100 - 0)% : ", K2
L2 = 2.04 * K2 - 204
IF (L2 + 205) < 0 THEN GOTO 337
IF L2 > 0 THEN GOTO 337
400 'PRINT : INPUT "enter FIRST d/a data in bits (-2048-+2048): ", DIO%(1)
401 '2048 MEANS -10 VOLT TO +2048(+10 V), FOR MICROWAVE CONTROL IS -
1 VOLT TO 0 VOLT

404 TIMER ON
406 T0 = TIMER

407 '------------------------FIRST HEATING -------------------------
409 DIO%(1) = L1
410 MD% = 7
420 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
422 IF FLAG% <> 0 THEN PRINT "Error # "; FLAG%; " in D/A output."
    SOUND RND * 1000 + 47, 4: SOUND RND * 500 + 47, 4
424 IF ABS(TIMER - T0) < T1 THEN GOTO 410

425 '------------------------ SECOND HEATING ------------------------
426 DIO%(1) = L2
432 MD% = 7
433 CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)
434 IF FLAG% <> 0 THEN PRINT "Error # "; FLAG%; " in D/A output."
    SOUND RND * 1000 + 67, 3: SOUND RND * 600 + 67, 3
'SOUND RND * 1000 + 67, 3: SOUND 3500, 3
435 IF ABS(TIMER - T0) < ABS(T1 + T2) THEN GOTO 426

438 '--------------------- RESET MICROWAVE POWER TO ZERO ----------------- 
439 DIO%(1) = -204
440 MD% = 7
CALL DAS20(MD%, VARPTR(DIO%(0)), FLAG%)

IF FLAG% <> 0 THEN PRINT "Error # " ; FLAG%; " in D/A output."

TIMER OFF  '----------------------------- RESET -----------------

PRINT : COLOR 0, 7: PRINT " DO ANOTHER HEATING (y/n)? "; : COLOR 7, 
0: PRINT " ";

A$ = INKEY$: IF A$ = "" GOTO 459

PRINT A$

IF A$ = "Y" OR A$ = "y" THEN GOTO 326

END