STUDIES OF MICROWAVES APPLICATIONS
FOR
ADHESIVE, WELD JOINT
AND
NDT TECHNIQUE

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Abstract

One of the most important areas of the application of industrial microwave processing for materials processing is curing polymers and polymer based composites. This research effort has been made to fast process and assesses the joining of thermoplastic substrates. Polycarbonate and Acrylic sheet substrates were chosen. Two adhesives were selected, namely the C-245 General Purpose Epoxy Adhesive and Toughened Acrylic Adhesive respectively in order to provide heterogenous bonding. Teflon jig and wooden clamp jig were designed and used to assist the adhesive joining process. Single lap shear joints were prepared for characterisation to ASTM procedures. Adherend were prepared and the joining process was performed in the Variable Frequency Microwave Furnace (VFMF). Curing was input with various power levels and irradiation duration. Inside the cavity of the VFMF, a temperature sensor probe was used to automatically monitor the temperature level of the load. Operational parameters, such as temperature and power levels, were recorded and saved as a file on the CPU unit.

Tensile testing was performed on the all adhesive joints using Instron Machine and the tensile bond strength (Stage I) was calibrate and recorded. Another set sample (Stage II) was performed again, but the tensile testing measurements were performed after six months since the bonding was performed. These two sets of data has recorded and further studied.

Scanning Electron Microscopy (SEM), photostress analysis using a polariscope, and infrared spectroscopy analysis were performed on selected fractured joints to explore the detailed fractured surface characteristics and stress distribution of the tested to destruction joints. Results show the average of both set of the bond strength and the different percentage deviations from the parent material strength. In general, stage II seemed to yield weaker bond strength. This provided a strong indication that the microwave curing bonding strength is time (duration) sensitive. The polariscope results showed an even stress distribution and the bond strength being close to the parent material strength.
Towards the end of the thesis, a new NDT (non-destructive testing) methodology is demonstrated based on the microwave characterisation technique. This form of quality testing can prove to be a new evaluation tool for assessing bond integrity using the same processing and quality testing facilities, ie the VFMF.

In summary, this thesis presents the results of many adhesive bonded experimental studies as well as a new methodology for evaluating bonded joints. Together with the new findings about the duration sensitivity of the microwave curing process and NDT technique, the associated limitations are outlined. The recommendations arising from the research outcomes suggest ways for extending the work of this research studies and suggest further developments for enhancing the capabilities and applications of the techniques.
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To my parents, who are very patient and motivated me, especially during the years of my study in Australia and away from them.
Declaration

This thesis:

1. no material which has been accepted for the award to the candidate of any other degree or diploma, except where due reference is made in the text of the thesis;
2. to the best of my knowledge contains no material previously published or written by another person except where due reference is made in the text of the thesis; and
3. where the work is based on joint research or publications, discloses the relative contributions of the respective workers or authors.

Siu, Yan Kit
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1. Introduction

Microwave energy is a unique energy source and offers an alternative means by which rapid processing for an array of advanced materials performed. Research teams worldwide are investigating the use of Microwave energy in the area of materials processing—all of which use either single mode or multi mode, variable-frequency Microwave processing techniques.

Microwave heating is more efficient than conventional thermal processing because the Microwave (electromagnetic) energy is directly coupled into the material at the molecular level, bypassing the air medium in the cavity chamber. The demonstrated “global advantages” of Microwave heating over conventional heating include direct and volumetric heating, high selectivity, fast heating rates, and reaction kinetics enhancement. Unfortunately, these potentials have not yet been attained at an industry scale due to non-uniform energy distribution inside Microwave cavities. The electromagnetic energy distribution is directly related to the magnitude and configuration of the electric field pattern inside the microwave cavity and is usually localized in specific regions. This condition is represented by a typical multi-mode cavity energy distribution pattern. In other words, fixed-frequency Microwave processing systems can only provide either a small, high-efficiency processing volume or a large, low-efficiency processing volume.

There are several advantages resulting from the use of variable frequency microwave energy. Some of these advantages include selective and volumetric heating, and rapid curing. These advantages make microwave processing an appealing method, which can be find in automobile and aerospace industries, provided the process is scalable to full production. The variable frequency microwave systems meet these scale-up requirements.
1.1 Brief Outline of Microwave Research

-To assess the applicability of harnessing variable frequency microwave energy for joining polymers using adhesives.

-To develop suitable microwave applicators and peripheral hardware facilities for lap joining of thermoplastics

-To optimize input parameters in order to obtain a high quality and testing methodology of bonding.

-To explore the potential of the microwave process for applications in, PCB, automotive and aerospace industries.

-To evaluate various curing process according to its advantages and limitations.

-To establish microwave pattern (signature curve) as a mean of Non-destructive evaluation method.

-To study different welding jointing methodologies and its bonding optimization.

1.2 Aims and Objectives of the Research and Development Work

The proposed research work aims at developing a novel technique for adhesive bonding dielectric polymers through the application of microwaves. Lap joining of the specimens took place in a microwave field using a 200W variable power magnetron operating at 2.45 GHz or variable frequency (1-6GHz) and specimens have enclosed in applicator. Bonding took place due to controlled and fast curing of the adhesive in the microwave field.
The frequency agile microwave applicators offer considerable advantages when compared to fixed frequency applicators. Electronic tuning of frequency, frequency bandwidth and sweep rates are integral features of variable frequency microwave systems. A closed loop computer control system allows for intelligent processing. In addition to heating uniformity and frequency tuning capability, the variable frequency microwave processing technology leads to successful processing in areas otherwise unexplored in the microwave community: such as surface mount applications in the PCB assembly methodology.

A Microwave system that combines efficiency with large heating volume has been needed to boost the commercial application of Microwave technology. New development has been established to focus on the exploitation of a unique Microwave processing approach that provides a uniform large heating volume at a high energy coupling efficiency. Using variable frequency irradiation, this approach specifically targets the processing of high-performance materials such as polymers, ceramics and monolithic composites, and providing solutions to the inherent scale up limitations of fixed-frequency microwave irradiation.

1.3 Thesis Structure

This dissertation is consists of a number of chapters that follow on from this introduction. In summary, the contents of these chapters are as follows:

-Chapter 2- Literature review. This chapter provides a detail discussion of the research literature covering microwave field, its mechanisms of interaction, dielectric properties, model of the microwave process, and polarization. These topics are essential to understand the basis of microwave technique of these research studies.
-Chapter 3- Innovative Breakthrough: Variable Frequency Microwave (VFM) interaction with materials. This chapter covers the newly invented variable frequency microwave technology, its advantageous features, such as the rapid heating, overcome the thermal runaway, frequency control, selective and volumetric heating, even distribution of energy distribution.

-Chapter 4- Composite Materials and Mechanisms of Joint Adhesion. This chapter introduces various types of epoxy and acrylic adhesives as adhesives material and polymeric matrix composite as adherend materials. Adhesive and welding joints and their curing process are also explained in this chapter.

-Chapter 5- Experimental Facilities and Processing. This chapter has revealed the latest models of VFMF (Variable Frequency Microwave Furnace), which capable to overcome a lot of drawbacks of the conventional microwave process methodology. Adhesives, adherends, and other accessories are introduced.

-Chapter 6- Adhesive Curing with Microwave Technology. Industrial applications of the adhesive joints are introduced. Adhesive curing technology has become popular in the automotive and aerospace industry, utilized encapsulation technique (the PCB manufacturing), better weight-to-cost ratio and better manufacturability of PMC materials with VFM technology.

-Chapter 7: Microwave Adhesive and Welding Joint. Experimental result of lap joint and welding joint was caliber. Bonding strength and elongation of the adhesive and welding joints valued have collected via the Intron Machine. Statistical studies of these data obtained reveal the findings of the experimental research.

-Chapter 8: Non-Destructive Evaluation. This chapter applied the newly invented Non-Destructive Testing methodology via the microwave radiation. With the workload characterization process, different materials and product will generate a
unique reflective curve as their identity. These signature curves will act as a standard reflection value during production. This unique NDT technique will offer another alternative methodological testing for online production in the future.

-Chapter 9: Conclusion and Recommendations for further work. This chapter restates the core objectives and aims of the thesis intended. The experimental results and findings of this research are illustrated. Recommendation of future researches are proposed, and highlights the limitation and deficiencies also addressed.
2. Literature Review

In 1945, when T. L. Spenser of Raytheon Co. use microwave on food heating, has marked the first commercial microwave processing in history. Again in 1952, Spenser introduced a conveyor microwave system, which led to industrialise of microwave heating technology. During the 1960s and 1970s, several companies first applied microwave equipment and applications for industries, particularly food production. Until the 1980s, large-scale production achieved commercial success. Some important achievements, including the three major applications: meat tempering, bacon cooking, and rubber production. Smaller-scale applications can be found in the pharmaceutical, forest product, ceramics, textile, and chemical industries-mostly for the curing or drying of basic materials. Many new developments also in the area of bio-medical research for testing of human organ and body and sterilization of virus in closed chambers.

There are several reasons for slow growth of microwave processing, including a limited choice of operating frequencies, a lack of understanding of microwave/material interactions, the reluctance of industry to accept the risk of a new technology, and a lack of communication between microwave engineers and manufactures.

As the influence of key processing parameters affecting the heating of materials with microwaves are better understood, process optimization and control has become more precise. In recent time, improvement of microwave production device has developed into variable-frequency which by computerise, improved data collection and modelling efforts are helping to illuminate the complex physical mechanisms of microwave/material interactions. Once materials can be adapted to meet industry standards and customer needs, microwave applications can be expanding rapidly/such as the automotive and aerospace industries.
2.1 The Microwave Spectrum

Figure 2.1 is an expanded view of the microwave spectrum, showing the frequency locations of some microwave systems. Long-range military search radar operates at 450 MHz, UHF broadcast TV at 470-870 MHz, and the cellular telephone band at 900 MHz. Just above 1 GHz is the air traffic control transponder, which allows aircraft to repeat an identification code to the air traffic control radar. Just below 2 GHz are space telemetry systems, which transmit data from deep-space probes to earth. Just above 2 GHz are troposcatter communication systems, where the microwave signal is scattered off of the troposphere to achieve long-distance communication.

Microwave heating equipment, including home microwave ovens, is fixed at 2.45 GHz. Just above 3 GHz is airport search radar, and just below 4 GHz is point-to-point microwave relay, carrying thousands of telephone channels and television programs.
across the country. Communication satellite downlinks are at 4 GHz, and the uplinks are at 6 GHz. Just above 7 GHz is STL, the studio transmitter link, which transmits radio and television programs from the downtown studio to the transmitter site.

Airborne fire control radar is at 10 GHz, and just above that is another microwave relay band for telephone transmission. Just below 20 GHz is another satellite communication downlink band, and just above 30 GHz is the corresponding uplink band. These bands are allocated for future satellite communication.

Heating with microwave is fundamentally different from traditional heating because electromagnetic energy is directly transferred to and absorbed by the material being processed. Because microwave radiation is penetrating, materials are internal heating, when properly controlled, more rapidly and evenly than traditional heating. This is particularly desirable for thick sections of low thermal-conductivity materials, such as polymers and ceramics, the penetrating radiation also cause materials with interiors hotter than their surfaces.

As microwave heating mechanism different from the traditional process, microwave processing equipment and processing conditions often bring new challenges. Employing recent advances in the understanding of microwave/material interactions is imperative for the successful processing of many materials and should be factored into the design of new microwave equipment.

In the electromagnetic spectrum, the microwave frequency range lies between the radio and infrared regions. To avoid interference with telecommunications, defence, and maritime applications, the state governments allocate discrete frequencies for industrial, scientific, and medical uses purposes. Allowable microwave operating frequencies for industrial processing vary. In the United States, the two most commonly used frequencies are 0.915 and 2.450 GHz. The longer wavelength of the 0.915 GHz frequency, commonly used in industrial ovens, allows deeper penetration into an object,
distributing heat more evenly. The much shorter operating wavelength corresponding
the 2.45 GHz, however, is more practical for more-compact household ovens. A new
generation of variable-frequency microwave furnaces can sweep across a frequency
range of 0.9 to 18 GHz.

2.1.1 Evolution of Microwave Processing

Although microwave heating was conceived about fifty years ago, it is a relative
newcomer to the field of materials processing. The first commercial microwave ovens
for heating food were manufactured in the early 1950s. However, they were not used
widely in industry until 1962 when practical choke systems were developed for
conveyor ovens. Presently, microwave heating is used extensively only in a few
processing industries, with industrial and domestic food preparation comprising the
majority of applications. Meat tempering, bacon cooking and rubber vulcanization
account for about 90% of the industrial microwave was processing systems in the
United States. In the 1970s and 1980s, the highest growth segment of the industry was
home microwave ovens, which first introduced on a large scale in 1965.

2.2 The Nature of Microwaves

Microwaves travel in free space at the speed of light. The space wavelength \( \lambda_0 \) is
related to the frequency by the equation:

\[
\lambda_0 = \frac{c}{f}
\]  

(2.1)

where,
\( \lambda_0 \) is the free space wavelength in centimetres,
c is the speed of light (3x10^10 cm/sec) and
\( f \) is frequency in Hz (cycles/sec).
Microwave radiation is non-ionizing as distinguished from ionizing radiation. Examples of ionizing radiation are x-rays, gamma rays and cosmic rays. Microwaves interact with dielectric materials to generate heat by the agitation of molecules in an alternating electromagnetic field.

The direct association of dielectric and capacitance is definitely a valid one. We called a dielectric an insulator in the capacitor example, and the dictionary definition said it was a material or substance that would not conduct electricity. These statements mean that dielectric is a material that causes the path of the electrical energy to be obstructed.

2.2.1 Microwave Energy Processing Technology

Microwave energy interacts with materials at the molecular level. Heat has generated by the movement of the molecules which radiated by the electromagnetic energy into the irradiated material. The microwave energy absorbed within materials depends, among other things, on the incident microwave frequency, the dielectric constant and the effective dielectric loss of the material and the distribution of electric fields within the material.

Microwave processing is generally functioned within a metallic applicator. There are three groups of applicators: resonant applicators, travelling wave applicators and multimode applicators. The travelling wave applicator and the single mode applicator (resonant cavity) are successful in processing simple material geometrize such as fibres. However, the multimode applicator has the capability of forcing electromagnetic energy onto large and complex shaped materials. Thus, the multimode applicator is suited with the production of large and complex functional material components.

Multimode applicators can be powered with fixed and/or variable frequency sources. The fixed frequency microwave oven is alike to home kitchen models. When a fixed frequency microwave signal is launched within the microwave cavity (the metallic box),
it suffers multiple reflections and results in the establishment of several modal patterns. The overall distribution of electromagnetic energy is varying throughout the microwave cavity resulting in high and low energy field areas, i.e. hot and cold spots. Heating using multimode applicators has been successful in various foods and rubber related applications. The thermal gradients established owing to non-uniform electromagnetic energy distribution can be adjusted for to some extent and are tolerated for food applications, but this is not the case for advanced materials.

Multimode microwave furnaces powered with variable frequency sources are better appropriated for advanced materials applications related to polymerisation, composites processing, bonding and others. The electric field distribution, on a time averaged basis, is uniform throughout the entire cavity volume leading to uniform exposure of the processed material to the microwave energy. In addition, the incident microwave frequencies can be changed to optimize the microwave energy abortion by the material of interest.

### 2.3 Fixed-frequency Microwave Processing

At room temperature, many polymers do not absorb appreciably at 2.45 GHz. However, their absorption can be increased by increasing the temperature (in contrast to water), adding absorbing constituents, altering their microstructure and defect structure, by changing their form (e.g., bulk versus powder) or by changing the frequency of the incident radiated. The wide range of interactions that occur between microwaves and various materials provides many challenges to the processor. On the other hand, it is because of these different interactions that microwaves can be transported, contained, and used at room temperatures as well as elevated temperatures.

Many materials do not couple well with 2.45 GHz microwave energy at room temperature. However, coupling efficiency can be improved by increasing their temperature of the target material. Once the material is heated to its critical
temperature, $T_c$, microwave absorption becomes sufficient to cause self-heating. This hybrid method can result in more uniform temperature gradients since the microwaves heat volumetrically and the external heat source minimizes surface heat losses. Hybrid heating can be achieved either by using an independent heat source such as a gas or electric furnace in combination with microwaves, or through the use of an external susceptor that couples with the microwaves.

Another means of heating a non-coupling material is to incorporate absorbing additions. Reinforcements, binders, fillers, plasticizers and dispersants are often added to polymers and ceramics as processing aids or to improve their properties, proper selection of these additives can improve absorption and make them more favourable for microwave processing. The trend here should be to tailor parameters such as composition and additives specifically to facilitate microwave processing rather than to try to use microwave processing with established parameters. This latter scenario is most likely to fail and cause the processor to miss the real benefits of microwave processing.

2.3.1 Dielectric Properties

At the macroscopic level, the dielectric properties control the microwave processible of a wide range of non-magnetic materials through the quantitative relationships of absorbed power per unit volume (PA) and depth of penetration (DP).

$$P_A = 2\pi f\varepsilon_0\varepsilon''_{eff}|E|^2$$  \hspace{1cm} (2.2)

$$D = \frac{\lambda_0}{2\pi(2\varepsilon')^3} \left[ \sqrt{1 + \left( \frac{\varepsilon''_{eff}}{\varepsilon'} \right)^2} - 1 \right]$$  \hspace{1cm} (2.3)
where,

\[ f \]: Frequency of the field;

\[ \varepsilon_0 \]: Permittivity of free space;

\[ \varepsilon' \]: Free space wavelength of the microwave radiation;

\[ E \]: Electric field;

\[ \varepsilon'' \]: The relative values of the dielectric constant;

\[ \varepsilon'' \text{ and } \varepsilon''_{\text{eff}} \]: The relative values of loss factor;

The dielectric constant is composed of a real part and an imaginary part. It represented the real component, called the real permittivity (\( \varepsilon' \)), gives a measure of the material’s ability to store electrical energy, whilst the imaginary component, called the loss factor (\( \varepsilon'' \)), determines the material’s ability to respond to changes in an electric field or its ability to be polarized. The dielectric loss factor provides the most instructive measure of a material’s ability to absorb electromagnetic radiation.

2.3.2 Accelerated and Self-Limiting Heating

Metaxas and Meredith (1983), as well as many others, have shown that when low loss materials exhibit an abrupt increase in \( \varepsilon''_{\text{eff}} \) with increasing temperature, the potential exists for very rapid changes in temperature. The temperature where this abrupt change in \( \varepsilon'' \) occurs is called the critical temperature, \( T_c \). A more precise definition of \( T_c \) based on difference in absorbed and dissipated power is provided by Johnson, 1993.

A potential consequence of the rapid increase in \( \varepsilon'' \) with temperature (\( d\varepsilon''/dT \)) is that, unless the sample is heated uniformly, localized hot spots can develop. Equation 2.1 shows that the amount of power absorbed and converted to heat is strongly dependent on the electric field. If the electric field varies significantly over the dimensions of the material non-uniform heating can occur. The inverted temperature profile that is
characteristic of microwave processing also can cause non-uniform heating between the internal and external surfaces. The development of hot spots is usually referred to as thermal runaway (unstable accelerated heating). This terminology is unfortunate since it suggests a process that cannot be controlled. In fact, it is this very property (large $d\varepsilon^*/dT$) that permits the microwave processing and rapid heating of many materials that are poor absorbers at ambient temperatures.

Some materials exhibit the exact opposite of thermal runaway. That is, upon heating above a certain temperature, $d\varepsilon^*/dT$ decreases significantly and heating becomes self-limiting. Dishes fabricated from these materials are used in home microwave ovens for searing meats. Polyester films loaded with graphite and used in the food packaging industry also rely on self-limiting heating. Other applications include phase changes in which the product is a poor absorber. Many experiments (Wei and Hawley, 1993, 1996) also point out that microwaves offer self-limiting heating during polymer processing because materials are less susceptible to microwave heating once polymerized, and heating will focus automatically on the unreacted part.

For polymeric systems (Chen, 1993) has correlated accelerated heating behaviour for a series of polymers with a polymer relaxation at microwave frequencies. Above which the molecular dipoles can respond to the applied electric field with a corresponding increase in the loss. Above the critical temperature, molecular dipoles call respond faster to the applied electric field with a corresponding increase in the loss and therefore, in the heating rate.

### 2.3.3 Ionic Polarization

Ionic polarization occurs when ions in solution move in response to an electric field. Ions carry an electric charge and are accelerated by the electric field. Kinetic energy is given up by the field to the ions, which collide with other ions, converting kinetic energy into heat. The more concentrated the solution, or the more dense the solution,
the greater the frequency of collisions, and the more kinetic energy that is released. When the electric field is alternating at microwave frequencies ($2.45 \times 10^9$ cycles/sec, for example) numerous collisions occur, and large heat is generated. This is recognizable as an increase in the temperature of the dielectric material. On a microscopic scale ionic polarization can occur in cellular fluids.

2.3.4 Dipole rotation

The dipole rotation heating mechanism as illustrated in figure 2.2. In the presence of all electric field, the polar molecules line up with the field. As an alternating field is applied, the polarity of the field is varied at the rate of the microwave frequency and the molecules attempt to align themselves with the changing field. Heat is generated as a result of the rotation of the molecules. When the field is removed, the molecules return to their random orientation. The amount of time required to realign depends on the size of the molecule. Microwave heating is also dependent on the physical state of the microwave technology material. In ice, the movement of water molecules in a microwave field is restricted and, therefore, ice is a poor microwave absorber. As the temperature of a material increases, the molecules tend to line up more rapidly and return to the random state more rapidly.

![Figure 2.2 Dipole rotation due to changing field (Decereau, 1986)](image-url)
2.3.5 Polarization

Polarization is the displacement of positive and negative charges as they align within a dielectric material under the influence of an electromagnetic field. There are four types of polarization:

1. Ionic polarization,
2. Electronic polarization,
3. Maxwell-Wagner (or space charge) polarization,
4. Orientation (or dipolar) polarization.

The types of polarization experienced by a material depend upon the type of dielectric material and the frequency of the electromagnetic field. Thus ionic and electronic polarization occurs, under the influence of an electromagnetic field, the neutral molecules or atoms become molecules or atoms with induced charges. Ionic polarization is when molecules become induced dipoles as a result of the distortion of the molecules and the unequal distribution of the charges in molecule formation. This type of polarization occurs at infrared frequencies. Electronic polarization results from the redistribution of electrons around an atom’s nucleus and occurs in non-polar dielectrics at frequencies close to the ultraviolet region. Maxwell-Wagner polarization shown in figure 2.3 arises from charge build up at interfaces between components in heterogeneous materials (Metaxas, Meredith, 1983).

![figure 2.3 A schematic of the rotation theory.](image-url)
The type that is important to microwave heating is orientation (or dipolar) polarization. Orientation polarization occurs at lower frequencies (HF, microwaves) at the molecular level in polar dielectrics containing very little moisture. The electric field produced by the microwave causes the permanent dipoles to rotate and re-orientate themselves as the field alternates.

Microwaves can interact with materials either through polarization or conduction processes. Polarization involves the short-range displacement of charge through the formation and rotation of electric dipoles (or magnetic dipoles if present). Conduction requires the long-range (compared to rotation) transport of charge. Both processes give rise to losses in certain frequency ranges as illustrated by figure 2.4. In this figure, the dielectric losses \( \varepsilon'' \) are due to ionic conduction, dominant at low frequencies, and rotation of permanent dipoles at higher frequencies. The ionic conduction losses are due to the ohmic losses that occur when ions move through the material and collide with other species. Ionic conduction decreases with increasing frequency because the time allowed for transport in the direction of the field decreases with increasing frequency. Both processes contribute to the losses, and it is not always easy to experimentally identify between the two losses mechanisms. Consequently, losses are typically reported as effective losses \( \varepsilon''_{\text{eff}} \) or \( \varepsilon'' \) when the loss mechanisms are unknown or cannot be clearly separated.

![Figure 2.4 Effective loss factors due to dipolar ionic conduction (Clark & Sutton, 1996).](image)
In addition to dipole rotation other widely recognized polarization processes include interfacial and ionic polarization. These processes are discussed in detail by (Chen et al, 1993), for polymers and ceramics materials processing. To adequately characterize the response of ceramics and polymers to the EM spectrum, measurements should be made over a broad frequency range. Frequency ranges, where each of these can contribute to losses, vary considerably over the EM spectrum depending on the material and the temperature. Furthermore, increasing the temperature increases the kinetic energy of the dipoles, making it easier for them to respond to the oscillating field and shifting the absorption curves to higher frequencies. It is because of the frequency dependence of absorption that a single frequency such as 2.45GHz can not be used to heat all materials. To efficiently heat a material that does not have an absorption mechanism close to 2.45GHz requires changing either the frequency of the radiation or the composition of the material, or using hybrid heating. Likewise, materials can be designed to shield from microwave; these materials have insufficient absorption to be self-heating at the operating frequency of the microwave oven. Booske et al. (1992) have performed and extensive modelling/experimental study on microwave interactions with a few ionic solids. Using single-crystal NaCl as a model system, losses from three possible mechanisms have been calculated and experimentally verified using a cavity perturbation technique.

### 2.4 Reason to Use Microwave Process

Many different aspects of the microwave process have been imitated, drying and heating rates, temperature profiles, electric field distributions, power absorption and hybrid heating. Most research has been focused on imitating the electric field inside a loaded cavity, and then coupling these data with Equation 2.2. From these results, the change in temperature can be predicted. In some cases, RF and induction heating have also been modelled.
2.4.1 Microwave Processing Applications

There are numerous research groups world-wide that are applying microwave energy to different types of materials processing. Much of this work has been presented in various symposia and publishing work. In general, the areas of microwave processing can be divided into:

- Recovery (beneficiation of minerals and waste remediation);
- Synthesis (pyrolysis, combustion, decomposition, catalysis);
- Removal of phases (drying, solvent vocalization, binder burnout);
- Fabrication (joining, CVI, coatings);
- Consolidation (drying, sintering, melting, curing); and
- Post-fabrication treatments (annealing, surface modification, nucleation/crystallization, sealing, drying).

Researchers (Tinga, 1992) have developed an open coaxial microwave spot which appears useful for joining applications. The applicator can be moved along a surface to rinse its temperature in a very narrow joint region to over 1000°C. In this design it is also not necessary to heat the entire assembly only the joint region.

2.4.2 Prerequisites of Microwave Processing

Although many companies are using microwave energy successfully, there is still need many new developments in order to establish microwave technology into mainstream industrial processing. These are:

a. A better understanding of the capabilities and limitations;
b. Identification of incentives (e.g. reduced product costs, significant value-added to products);
c. Better availability of fully integrated processing equipment and supporting services;
d. More demonstrations of successful scale-up; and
e. Competitive capital equipment and operating costs.
As discussed in the previous sections, the first two requirements are being aggressively addressed through research activities and technical meetings. The last four requirements are the primary responsibility of the materials processors and equipment manufacturers. However, supporting services will undoubtedly involve universities to help design equipment and provide personnel trained in the field of microwave energy and its applications to materials processing.

2.4.3 Microwave System

A microwave system is composed typically of a generator to produce the microwaves, a waveguide to transport the microwaves and an applicator (usually a cavity) to manipulate microwaves for a specific purpose and a control system (tuning, temperature, power, etc.). Mostly, only fixed frequency single mode or multimode systems (home microwave ovens) were readily available. Single mode system have had limited (but successful) application in industry because of a limited processing volume over which the electric field is useful, but have been particularly effective in plasma processing and joining. In a multimode system, the fixed frequency microwaves excite resonant modes over a narrow frequency band around the operating frequency. The modes result in regions of uneven electric fields (non-uniformity) within the cavity. If the cavity size is large compared to the wavelength of the microwaves, a large number of overlapping modes can be produced resulting in better field uniformity over a large processing volume.

2.4.4 How Microwaves Produce Heat

When microwaves were applied to the dielectric materials such as food, they interact with the dielectric material, giving up energy which results in a temperature increase of the material. There are two main mechanisms by which microwaves produce heat in dielectric materials: ionic polarization and dipole rotation. There are other mechanisms which prevail in unique situations, for example, in the heating of
ferromagnetic materials and with gases under reduced pressure. The discussion will be limited, however, to ionic polarization and dipole rotation since this volume is primarily concerned with the heating of liquids and solids.

2.4.5 The Unique Features of Use Microwave Process

The unique property of microwaves to penetrate deeply into materials of interest provided the opportunity for substantial reductions in process time, often by as much as 10 to 1. From this unique property of deep energy penetration, many other advantages accrue. Noticeable benefits of improved quality and increased yield were often observed. Other reasons to use of microwaves are cause by processing of a product not possible by conventional heating methods and it is economical process. The improved economics usually come as a result of the combination of minimal microwave energy and some other form of energy. One case in which microwave energy is used alone and is particularly economical use of this form of energy is in the tempering frozen foods. In this process, minimum energy is required to raise the temperature of food from the solidly frozen condition to a few degrees below 32 °F. At this temperature it is possible to further process the foods without the need to thaw completely.

Space saving is another feature for choosing microwave application. Stretched out processing lines can be compressed into a fraction of the space such as pasta drying; segments of the process can be stacked, on top of the other, to permit gravity flow of the product being processes from start to finish in one-fourth the floor space. There are many reasons to opt for a microwave process when considering replacement of old equipment or construction of a new food plant. Not only space saving, but also can be in view of the plant construction cost, it can represent a substantial economic saving. The energy requirement can be substantially reduced due to the efficiency application of the microwave energy.
A number of other factors that may be taken into account in the decision whether to use microwave processing are:

1) Geometry: materials that are thick and therefore difficult and time consuming to process by conventional heating means;

2) Temperature sensitivity: materials adversely affected by usually processing temperature conditions;

3) Cost: costly materials generally can tolerate the higher cost microwave processing;

4) Uniqueness: a unique production of a product is obtained by the use of microwave heating;

5) Batch to continuous processing: batch process can convert into a continuous process which substantially reduced process time.
3. Innovative Breakthrough: VFM Interaction with Materials

Adhesives technology is commonly used as a fabrication option in new designs and has shown great potential in certain areas of structural and mechanical engineering. New structures such as polymers, composites and ceramics demand new fabrication techniques and adhesives play an important role in the joining of dissimilar materials such as metals to non-metals and in applications such as the joining of composite panels. A preparation of the surface of the adherents, wetting the surface to be joined, joint assembly, and then allowing the adhesive to cure over a period of time will make a successful bonding. Reduce the curing time by speeding up the rate of chemical reactions or by more rapidly evaporating solvents in the adhesive, the applications of certain environmental condition, generally involves exposure to heat and improvement in the overall bond strength.

A great deal of research has been devoted to understanding the chemistry and kinetics of curing reactions, due to the technological importance of thermosetting plastics. Microwave heating is one successful method that has been shown to improve reaction during the curing process of thermosetting plastics. The microwave attraction stems from the ability to deposit energy directly into a body, and generates heat without the need for thermal conduction. Compared with conventional heating methods that rely on conductive heating, microwave technology combined with suitable process control strategies obtains improved process efficiency. To understand how adhesives work, it is necessary to understand their mechanical properties and the chemistry used for creating those properties.

3.1 Variable-Frequency Microwave (VFM)

Microwave has several characteristics that different from conventional surface-heating methods. These features-penetrating radiation, controllable energy distributions, raid heating, selective heating, and self-limiting reactions allow great flexibility and control
for processing materials, and they can not only lead to improved quality and product properties, but also reduce processing times, with resultant energy and labor savings. In addition, entirely new types of materials that cannot be produced by alternative methods, but can be created and synthesized. Microwave furnaces are quiet, safe, and provide clean working conditions (which reduce contamination risk) with small floor-space requirements. Commercial opportunities for using microwaves to produce engineered materials are just at the beginning of development stage.

The internal electric fields generated within the volume of the material induce translational motion to ionic dipoles, as microwaves penetrate and propagate through the material. The resistance of these induced motions causes losses, attenuating the electric field and volumetrically heating the material. The degree of absorption can be quantified by the electromagnetic properties of the material, such as electrical conductivity, permittivity and microwave frequency. Metals have very high electrical conductivity and are thus opaque to microwaves, performing the function of good reflectors for oven walls.

The range of dielectric responses of materials and their ability to absorb microwaves is perhaps one of the most widely used features of microwaves and is perhaps one of the most widely used features of microwave processing. For example, water is a strong, broad-band absorber of microwaves and is, for that reason, widely used in selective heating for processing food. Besides, microwaves effectively remove water from all types of wood products, chemicals, and many other materials. The processing of rubbers, asphalt, ceramics, and polymer composites all depend on the selective heating of at least one of their constituents.

3.1.1 Microwave Heating with Polymers Materials

Polymers are made of long chain-like molecules with characteristic branches that are repeated along the length of the chain. Many of these branches are from dipoles.
Dipoles that possess an asymmetric charge centre, such as H₂O molecules in water, will experience a torque when placed in an electric field. When the electric field is caused to alternate, the dipole will endeavour to line up with the field direction during each cycle, its ability to do this depends on how constrained it is in the dielectric. If it is totally constrained and cannot move, no energy will be lost due to the frictional movement. However, if it can move but cannot stay in phase with the field, energy will be lost in the form of friction and converted to heat. A measure of a material’s ability to absorb energy in the form of electromagnetic radiation is given by its dielectric constant. It depends on temperature and generally increases with temperature in the case of polymers. An abrupt increase in the loss factor at elevated temperatures creates the potential for rapid changes in temperature. The consequence of this is that; unless the sample is heated perfectly uniformly, localized hot-spots can develop leading to a thermal runaway phenomenon.

3.1.2 Interaction of Microwave Radiation with Adhesives

For adhesives, the dielectric constant and the dielectric loss factor increase with temperature and decrease with the extent of cure. At the initial stages of cure, the loss factor is very temperature dependent. At the later stages, this dependence is less pronounced because a large amount of cross-linking decreases the mobility of the dipoles. The opposite is true for a low cross-linking structure. For optimum microwave processing, a dielectric should ideally possess a real permittivity that permits adequate penetration and high loss factors facilitate rapid heating.

3.1.3 Advantages Features of Variable Microwave Furnace (VMF)

1) volumetric heating
   a) Lower surface temperatures: lower, often reversed thermal gradients-stress reduction,
   b) Partial pressure control of reactive gases for selective oxidation/reduction of
certain phases; controlled environments,
c) Power/temperature response,
d) Rapid removal of water, binders, and gases with reduced rupture/cracking effects overall:
e) Improved product quality/uniformity/yield

2) Coherence and control
   a) High energy concentration,
b) Precise heating of (irregular) sections: dielectric brazing, sealing, welding, fibre drawing, plasma generation,
c) Material densification at lower temperatures: sintering and diffusion,
d) Potential for automation: energy savings

3) Differential heating
   a) Coupling to transparent materials-thermal seeds; “active” containers;
b) Synthesis of new materials
c) Selective zone heating (welding)
d) Oxidation control

4) Accelerated heating
   a) Rapid: 2 to 50 times faster than conventional heating metrology,
b) Very high temperatures, > 2000°C possible in controlled atmospheres,
c) Accelerated sintering,
d) Cost/time reductions plus new products

5) Drying:
a) Self-limiting, self-regulating
b) Effective in combination with other methods
The unique microwave heating characteristics of different materials can be utilized to selective heat target layers in thin multilayer material systems, for examples, selective processing surface coatings on sheets, fibres or fabrics; selective curing adhesives between layers and elevating temperatures of catalytic surfaces. Microwave heating offers the possibility of elevating the temperature of the surface treatment or coating of the substrate material. Heat transfer modelling is used for identifying the fundamental parameters and providing guidance for evaluating the feasibility of selective heating for candidate material combinations. A transient, multi-layer and lumped-capacitance model can be develop that temperature disparities result from known material properties and processing conditions.

Since microwave power is dissipated volumetrically within a dielectric material in proportion to the loss factor of the material, the power is delivered selectively when different materials are present in the same microwave field. This unique deposition of power offers the potential of processing material combinations that could not simultaneously processed by any conventional technique.

Conventional heating methods require that energy propagates through the materials by diffusion. Thermal diffusion is proportional to the temperature gradient in the material. Hence, when the required temperature is finally reached throughout the material, most of the material is at a higher temperature, which is often destructive. Thermal diffusion is hard to control both spatially and temporally. Temperature overshoots and soaking times are usually required. In many cases, the surface characteristics of the material being processed suffer since the surface experiences the highest temperatures.

These characteristics of conventional techniques make heating system of multiple materials, such as coated substrates, difficult or impossible. For example, the coating on a polymer fibre could not typically be treated above the softening temperature of the fibre polymer, especially after the fibre has been incorporated in a fabric. However, with selective and uniform heating, such as variable frequency microwave heating,
processing on new material combinations may be possible.

Microwave curing of adhesives is another situation that preferential heating is desired and the adjacent material may not tolerate elevated temperatures. In microwave assisted catalysis the catalytic surface may be at a much higher temperature than the substrate.

Microwave heating is fundamentally different from conventional heating since microwaves penetrate many polymeric materials and heat volumetrically. During microwave processing, temperature gradients are unavoidable since the energy is absorbed by the bulk and dissipated at the surface of the specimens. In the case of low temperature microwave processes, as the case of polymers processes, these temperature gradients are easily corrected by using microwave-transparent insulation around the samples or by controlling the temperature inside the microwave cavity using hot-air jets.

That fixed continuous power dissipation into a dry, passive workload causes its average temperature to rise linearly with time, distinguishing it from conventional heating, in which the average temperature of the workload asymptotically reaches the oven temperature, and cannot rise above it. In principle, with volumetric heating, the average temperature of the workload continues to rise as long as power is applied.

Unlike conventional heating ovens, microwave ovens are very efficient in converting energy into heat in the workload. In a large industrial oven, which is dissipated as heat in the workload, the microwave efficiency can be in the region of 95%, and the conversion of electrical power into microwave power can have an efficiency of 85%. Moreover, a conventional oven has to be heated to a temperature substantially in excess of the required temperature in the workload, while a microwave oven, if heated at all, is normally heated to a temperature no greater than the required surface temperature of the workload. Therefore the radiation and convection heat losses from the microwave oven are significantly less because of its lower temperature.
Further energy saving arises because a microwave oven has instantaneous control of power, which means that equilibrium conditions are rapidly established after a change, and start-up can be rapid. Very fast feedback control loops can be used to control processing parameters accurately, which leads to product quality improvement.

Microwave heating is more efficient than conventional thermal processing because the microwave (electromagnetic) energy is directly interacts with the material at the molecular level; bypass the air medium in the cavity chamber. The non-uniform distribution of electromagnetic energy results in processing problems such as non-uniform processing and/or sample size limitations. In other words, fixed-frequency Microwave processing systems can only provide either a small, high efficiency processing volume or a large, low-efficiency processing volume.

3.1.4 Batch and Continuous Processes

Volumetric heating by microwave is either a batch or a continuous process. In a batch process a fixed mass of workload is placed inside the oven and a predetermined amount of energy is injected as

\[ E = \int_{0}^{T} P \, dt \]  

where,

- \( E \) = total energy injected into the workload, (Joules, or watt seconds)
- \( P \) = applied power to the workload, which may be a variable function with time (watts)
- \( t \) = total time of the process (seconds)

This energy \( E \) can then be used directly to calculate the thermal energy in the workload as arranged. In a continuous-flow process the workload is fed through a microwave chamber, normally fed with a constant power whilst the workload travels through at a constant mass-flow rate.
Lots of factors determine whether a process should be continuous or batch, such as:

a) type of workload, e.g. blocks, discrete items, powders, liquids or particles in containers for batch processing, extruded solids, deposited powders or particles on a conveyor, or liquids flowing inside a microwave-transparent pipe for continuous processing;

b) Ease of control of the workload parameters, e.g. batch weight, rate of mass-flow, or starting temperature, may be decisive in choosing the best procedure. Accurate control of continuous mass flow is often very difficult, precluding a continuous process;

c) existing up-stream and down-stream product handling and control systems;

d) Available space for equipment and materials. Continuous-process plant is usually elongated in floor plan, whereas batch systems have a generally square layout with space allocated nearby for product storage, both that awaiting treatment and treated material;

e) manpower requirements a continuous-flow system usually requires fewer operators;

f) automatic monitoring and control of process parameters is usually easier with a continuous process;

g) start-up and shut-down wastage of product may be unacceptable in a continuous-flow process;

h) the duty-cycle of actual heating time to total process time, i.e. heating time plus loading/unloading time, may be unacceptable in a batch process; the peak microwave heating capacity becomes greater than that of the corresponding continuous process, because the mean power is reduce by the duty cycle, resulting in a higher capital cost of the generator equipment;

i) Management control in one process may be easier than in the other, depending on local conditions.
3.1.5 Controllable Processing

A metallic applicator is necessary in a microwave processing performance, and there are three categories of applicators: resonant applicators, travelling wave applicators and multimode applicators. With variable frequency sources, multimode microwave furnaces powered are better suited for advanced materials applications related to polymerization, composites processing, bonding and others. The electric field distribution, on a time averaged basis, is uniform throughout the entire cavity volume leading to uniform exposure of the processed material(s) to the microwave energy. Furthermore, the incident microwave frequencies can be changed to optimize the microwave energy absorption by the material of interest. Tuning of frequency, frequency bandwidth and sweep rates are regulated by electronic devices or computer control system, which are integral features of variable frequency microwave systems.

With the VMF processing procedure, there are three variable parameters, central frequency, frequency bandwidth and sweep rate, which are electronically tuneable and are vitally important for controllable processing and energy spread. The central frequency selection and tuning for a given range of products are automatically performed through a program that defines the range of operating frequencies that are optimal for curing the adhesive. Then the frequency is swept over a bandwidth that provides uniformity for large samples or multiple small ones. The sweep rate simply determines how quickly the bandwidth is swept. Another parameter, power is adjusted during processing to affect the rate of cure.

In addition to heating uniformity and frequency tuning capacity, this unique microwave processing approach that provides a uniform large heating volume at a high energy coupling efficiency.
3.2 Microwave Processing of Composite Material

Microwave processing of polymers is a relatively new area in the broader field of microwave processing of materials. There has been comparatively little research conducted in the field of polymer processing. Various studies have claimed that microwave curing of polymers have cost advantages over conventional processes.

This VMF processing technique has the ability to heat materials volumetrically in contrast to conductive heating as in conventional processes. In addition to productivity and economy gains, this technique can produce additional product properties that are impossible to achieve through conventional processing. There has been an increased worldwide demand for new polymer materials with a concomitant demand for economical and environmentally safe methods for processing them. Microwave processing techniques can satisfy both of these demands.

The electric field component of EM energy makes the most significant contribution to microwave heating in non-magnetic materials. The ability of a material to absorb microwave energy and convert it into heat is determined largely by its dielectric properties. Materials that exhibit high electrical conductivity reflect microwave radiation. Non-conductive materials are transparent to microwave radiation. Between these two extremes, some materials permit partial penetration of the energy and subsequently convert that energy into heat. The ability of a material to be penetrated by microwave energy and to convert it into heat is described by a quantity known as the complex dielectric constant (or complex permittivity). The real permittivity describes the ability of the material to store electrical charge as well as the energy penetration characteristics. The imaginary permittivity (also known as the loss factor) describes the ability of the material to convert the stored energy into heat. Thus, in materials processing, it is essential to pick a parameter which can balance the two values and provide both adequate penetration and adequate heating. It is important to note that the dielectric constant is both a function of temperature and frequency. For
the computational model used in this research, the real and imaginary parts are separated for their individual effects.

3.2.1 High Energy Penetration

Because microwaves are capable of penetrating dielectric materials, heating occurs throughout the volume rather than on the surface alone. The volumetric nature of microwave heating gives rise to some unusual effects. During heating, the interior of a specimen cannot lose energy to its environment as fast as its surface loses energy subsequently. An inverse temperature profile is formed in which the interior is hotter than the surface. The penetration characteristics can be predicted using the real part of permittivity.

3.2.2 Rapid Heating

Because heat is generated directly from within the specimen, the rate of temperature rise is limited only by the available microwave power, the distribution of the resulting EM field and the thermal inertia of the specimen. A limitation of this feature is that the very rapid rise in temperature can lead to a phenomenon called thermal runaway if temperature and power absorption are not carefully monitored and controlled (Senko, 1996). The temperature of a specimen will continue to rise until an electro-thermodynamic equilibrium is reached at the time that the product may have already been destroyed.

3.2.3 Thermal Runaway

In thermal runaway, the increase in temperature of a material causes an increase in the dielectric loss factor. This in turn increases the microwave absorbance, the heating rate and so on. In materials of a low thermal conductivity localised regions of intense heating can occur (known as hot-spot formation), which leads to undesired defects in
the product. Generally polar polymers are weak microwave absorbers at room temperature, but may increase their absorbance of microwave energy with increasing temperature because heating increases molecular mobility. In some polymers a phase change occurs rapidly from solid to a rubbery phase at elevated temperature. Rapid changes in phase can lead to thermal runaway caused by the large increase in molecular mobility. Low molecular weight polymers exhibit these characteristics and thus are vulnerable to this kind of irradiation. Amorphous polymers with a high molecular weight exhibit a rubbery characteristic after the glass transition temperature.

3.3 Polymer Processing

The preliminary work in polymers was focused on using microwave energy to cure thick cross-sections of rubber. Since rubber is a poor thermal conductor, long curing times in conventional ovens are required. However, rubber can be pre-heated rapidly and uniformly, thereby shortening final curing times required in a conventional oven. This work led to the first industrial application of microwave energy in polymer processing-vulcanization of rubber. It also demonstrated that a combination of microwave/conventional heating was preferable to either used separately for this application.

In the late 1960s, microwaves were first used to process polymeric materials, such as certain rubbers; in general, elastomers are poor thermal conductors and require complex and costly heating procedures. Microwave energy plays a considerable role in this field as the carrier of energy independent of the thermal properties of the material. Microwave processing has replaced traditional processes for certain polar elastomers specifically, polychloroprene and nitrile butadiene rubber and has the potential to provide new applications, such as the vulcanization of thick and irregularly shaped sections.
For example, microwave energy has become the preferred method for foaming and preheating the rubber that used in automobile seals. Through careful process development, the technology became a commercial success because of the high heating rates (substantially shortening process time) and enhanced product quality that results from uniform energy deposition. A significant aspect of this effort was tailoring the microwave absorption properties of carbon filler, a major component of filled rubbers.

The use of microwave energy in polymerization is in the development stage and plays only a minor role in industrial production, but this may change in the near future. Microwave technology has been applied to the processing of both thermosetting and thermoplastic polymers. Thermosetting materials (such as polyesters, polyurethanes, epoxies, and polycarbonates) have all been successfully processed with microwave energy. In the last 10 years, there has been much interest in using microwaves to process high-performance polymeric composites, such as carbon-and/or glass-fibber reinforced epoxies, which act as lightweight, high-strength structural components.

Flexibility in achieving temperature control is especially important in maintaining uniform temperature throughout a work piece, which is essential for curing polymer-matrix composites. For example, absorption properties of many polymeric materials depend on both temperature and frequency. Absorption characteristics throughout a processed sample may change as it is heated, making uniform heating more difficult. Changing the cavity mode, however, either by moving the cavity wall or changing the frequency will change the energy deposition pattern within the sample. Frequency control enhances the ability to achieve more uniform temperatures. The prospects for developing mode-tuning devices to improve process control may have a profound impact on the number and quality of microwave-based applications for polymers.

There is growing evidence to support that the use microwave energy in certain industrial processes, are increasing observed. In contrast to conventional furnaces, the material
to be processed in a microwave furnace (oven) interacts with the microwaves ray instead of radiant heat. Because the material itself converts the energy to heat, heating is more volumetric, and can be very rapid and selective. In addition, heating is instantaneous (on/off) with power. When properly controlled, these features result in better product uniformity, faster production throughput, less floor space, and reduction in wasteful heating (i.e., furnace walls, conveyor belts and kiln furniture). An even more compelling reason (beyond economics) for a manufacturer to select microwave processing is that certain products can only be fabricated or synthesized by microwave heating, or by a combination of microwave and conventional heating.

3.3.1 Existing Curing Methods

Most processing methods are used thermal cure as methods to activate the cross linking process and promote the liquid to solid phase transition of the electronic grade polymer encapsulants, including glob-tops and underfills. A non-thermal curing method based on, microwave irradiation can be used to rapidly cure specially tailored glob-top chemistries.

Thermal curing methods are based on convection heating which make use of the flow of hot air. The hot air molecules in the oven strike violently against microelectronic assemblies (for example, the surfaces of the PWB, the silicon-chip and the underfill fillet). Thermal energy transfer to the various surfaces happens during air/molecule collisions. Materials conduct the thermal energy from the surface into the bulk through intrinsic thermal transport mechanisms, depending, among other things, on the specific heat and thermal conductivity. The thermal conductivity of most substrate materials (including ceramics and organic boards) and that of the underfill materials is low which results in poor thermal transfer from the hot air environment to the various parts of the microelectronic assemblies. In addition, heat absorption results in an increase in the average interatomic distances and in general, an overall increase in the physical dimensions of the materials on the macroscopic level. Thus, PCB and silicon
has different expansion ratio, the interlayer, when exposed to a given process temperature is subjected to tensile and compressive stresses during the heating and cooling stages, respectively.

3.3.2 Innovative Curing-VFM Process

The VFM process has been qualified in terms of compatibility with electronic device functionality. Several electronic devices were successfully tested from a functionality perspective in terms of leakage current, breakdown voltage, logic, electrostatic discharge sensitivity, and memory retention before and after VFM exposure and process cycling. In previous work, the VFM process was found not to induce any deviation from the designed specifications of the electronic devices. While that study is the subject of a different publication, the present paper focuses on the evaluation of the polymer's properties after VFM rapid processing.

The variable frequency microwave technique is different from conventional microwave processing, which uses a fixed microwave frequency. Through this sweeping process, the problems that have been observed in conventional microwave ovens with non-uniformities in temperature and arcing are avoided. VFM hardware (microwave 2100) utilizes three major parameters with the purpose of providing processing reliability. These parameters are central frequency, frequency bandwidth and sweep rate. By using a user-friendly computer routine, these parameters are processed such that the bandwidth is divided into 4096 points. During an adhesive cure, the computer cycles through these 4096 frequencies consecutively with each cycle corresponding to the sweep rate input (typically 0.1 sec.). For each swept, there are standing wave patterns, consisting of tens of modes inside the cavity. By cycling through the frequencies, the VFM in effect launches 4096 different frequencies into the cavity with each sweep cycle (e.g., 0.1sec). This large number of frequencies excited during processing results in a uniform energy distribution throughout the oven.
In addition to uniform energy distribution, the VFM irradiation leads to damage free processing because the dynamics of charge build-up are reached when sweep rates less than 0.5 sec. are utilized. Indeed, when 4096 frequencies are consecutively excited over a period of 0.1 seconds, the residence time of any given, established wave pattern is less than 20 µsec, during a 20 µsec. residence time, a given component is exposed to an alternating electric field operating in the range of 5.85 to 7.0 GHz and having a magnitude between 2 to 20KV/m (0.1 to 2 V/mm). Current generation, if any, brought about by the high frequency, alternating electrical field is random and consists of eddy currents rather than being of an AC or DC nature.

### 3.3.3 VFM and Heating Mechanisms

There are four adjustable parameters that show the difference between VFM processing and conventional, single frequency microwave based technologies. These controllable processing parameters include: central frequency, bandwidth, sweep-rate and power. During processing, the central frequency irradiated inside the microwave cavity can be tuned to increase the coupling efficiency with the material to be processed. The combination of bandwidth and sweep rate around the selected central frequency provides the necessary distribution of microwave energy to carry out uniform heating throughout the work piece. The sweep rate is electronically controlled and can be varied from 100 seconds to milliseconds. The microwave incident power can be pulsed or cont sinuously varied to provide control over the heating profile of the work piece.
4. Composite Materials and Mechanisms of Joint Adhesion

4.1 Nature of Adhesives

When two solids with plane surfaces are brought together they make contact on the molecular scale only at rare points at their common interface. Mostly there is air between them. The application of force across their interface can cause distortion of the contact points making them into areas but some of the energy applied in doing this is stored as elastic energy and, on removal of the load, is utilized in breaking any union formed. The introduction of a liquid to displace the air can create a situation in which real contact is made throughout the interface and made, not between body and body but between body and liquid. If this liquid can solidify, the desired state of solid body contact is achieved although the solid body contains a plane of chemically different substance. This material used to bring about the change from occasional touch to intimate contact is an adhesive. The adhesive is essentially a solid but to achieve the essential intimate contact it must be applied in some liquid form.

Most adhesives are organic in nature and the traditional glues and gums acquired from natural sources are long chain molecules, soluble in water and are either proteins or polysaccharides. Although they are linear molecules they set in a strict manner with cross-links or hydrogen bonds between neighbouring molecules or parts of the same molecule thus giving a three-dimensional structure with consequent rigidity, a rigidity which is lost when water is evaporated.

4.1.1 Thermoset Adhesives

Today, there are a great range of commercial adhesives (over 100 types and several thousand products are listed in the Adhesives Handbook) and most of the principal thermoset and thermoplastic synthetic polymers and elastomers find some application in adhesive formulations. Adhesive formulations are of five general types;
1) Solutions of thermoplastics (including unvulcanized rubbers) which bond by loss of solvent.

2) Dispersions (emulsions) of thermoplastics in water or organic liquids which bond by loss of the liquid phase.

3) Thermoplastics without solvents (hot-melt adhesives).

4) Polymeric compositions which react chemically after joint assembly to form a crosslinked polymer (thermoset).

5) Monomers which polymerize in situ.

Adhesives of types (4) and (5) are converted by chemical reaction within the assembled joint. Chemical bonding between the adhesive and the adherend has sometimes been assumed; but adhesive bonding does not generally entail primary chemical bond formation across the interface. The reaction usually involves only components of the adhesive, which is cold-cured or heat-cured, with or without pressure. Thermoset adhesives which evolve water during curing (such as phenolic and amino resin types) usually require application of pressure. Adhesives which are sufficient polarize to show large dielectric losses at high frequency may be successfully heat-cured by exposing the assembled joint to a radiation field. Dielectric heating is widely used in bonding wood with adhesives. Cyanoacrylate adhesives are based on alkyl 2-cyanoacrylate monomers such as

\[
\text{CH}_2=\text{COOOCH}_3\text{ methyl 2-cyanoacrylate}
\]

polymerize extremely rapidly in the presence of traces of moisture normally present on adherend surfaces. For adhesive usage, polymer hybrid (two-polymer) adhesives are of considerable importance. Elastomers are added to thermoset formulations to improve flexibility and impact resistance: examples are EP-polysulphide, PF-CR and PF-NBR. PF-EP blends have exceptional performance at temperature up to 200°C.

As the new adhesives have emerged the performance (especially the strength and permanence) which can be achieved in adhesive-bonded structures has greatly improved.
The rational design of bonded joints has become a matter of concern to design engineers; there has also been parallel progress in the underlying science of adhesion, so that the factors which control adhesive action are becoming clear. It is essential that the adhesive should wet the adherend surface during the assembly of a bonded joint; and this depends on the surface energy of the adherend and the surface tension of the fluid adhesive. The ideal strength of the joint is controlled by the micro-structure of the interfacial zone, and the Van der Waals’ forces acing within it. Adherend surfaces may be porous or impermeable, and vary widely in roughness. The adhesive/adherend line of contact may be microscopic sharp or may be diffuse, through mutual solubility. In practice joint strengths are reduced by the presence of flaws, which may lie either within the adhesive or sometimes within a weak surface layer in the adherend.

4.2 Nature of Epoxy Adhesives

Epoxy resins are considered to be the most multifunction of the structural adhesives. In general, epoxy adhesives are strong but brittle; however they can be formulated to be more flexible without a significant loss in tensile strength. They may be formulated to cure at room temperature or at elevated temperatures, under dry or wet conditions (Behm, 1990).

Epoxy resins have a number of advantages over other adhesives:

- Adhesion: Epoxy resins have the ability to adhere to metals, glass and ceramics as a result of their epoxide, hydroxyl, amine and other polar groups. These adhesives have a variety of functional groups which give the epoxy adhesive an affinity for metals and plastics. They may also be formulated to vary their viscosity for improved wetting, spreading and penetration action.

- Cohesion: When properly cured, failure is more likely to occur in the joined members, rather than in the adhesive itself. This happens with glass and aluminium as well as with weaker materials such as concrete and wood.
• 100% Solids: Epoxy resins release no by-products while curing many of the phenolics and other resinous adhesives do. This makes it possible to cure the adhesive with little or no pressure. Since no water (as with rubber lattices) and no volatile solvent (as with nitro-cellulose cements) is formed (both of which must be removed), the epoxies are convenient for assembly-line bonding of impervious surfaces of metals and glass.

• Low shrinkage: Compared with vinyl-type adhesives (e.g. polyesters and acrylics) epoxy resin shrinkage is minimal. Consequently, there are no internal stresses within the adhesive-adhered bond which usually result in a weaker bond. The amount of shrinkage may be further reduced by the addition of silica, aluminium and other organic fillers.

• Low creep: Like most other thermoset resins, cured epoxies maintain their shape under prolonged stress much better than thermoplastics such as acetate, nitro-cellulose and polyvinyl butyral.

• Amiable to modification: Epoxy adhesives are easily modified to suit any application. An example is one mentioned afore where silica may be added to reduce shrinkage (Ellis, 1990).

• Improved fatigue performance: The adhesives allow a relatively uniform stress distribution resulting in improved fatigue performance.

• The ability to join dissimilar substrate materials. This allows the joining together of materials that when joined together by conventional methods creates the possibility of electrolytic corrosion.

• Allows the joining together of thin-gauge metals together, in particular honeycomb assemblies resulting in lightweight structures exhibiting high strength to weight ratios.

• Allows both increased design flexibility and the ability to design more complex shapes.

• Reduced production costs in comparison to welding and riveting.

• Good wetting properties facilitating the formation of good adhesion bonding.
4.2.1 Composite Resins

Resins with an oxirane ring as their reactive moiety are known as epoxy resins. Epoxy resins form the largest variety of structural adhesives currently available. Many epoxy resins are based upon the reaction of phenols with epichlorohydrin. As shown in figure 4.1, reaction of 2, 2'-isopropylidene diphenol, otherwise known as “Bis-Phenol-A” (BPA) with epichlorohydrin leads to the most common epoxy resin, the Diglycidyl Ether of Bis-Phenol-A (DGEBPA). It can be further reacted with Bis-Phenol-A to generate higher molecular weight resins which can be either epoxy or phenol terminated. High molecular weight products of DGEBPA and BPA terminated with phenol are known as “phenol resins”. The chemistry of epoxy resin-based adhesives consists primarily of the choice of mixtures of molecular weights of epoxy resins with the correct curatives. Bis-Phenol-F (BPF) can also be reacted with epichlorohydrin to generate resins analogous to the DGEBPA-based resins. BPF-based resins have the important attribute of lower viscosity than the DGEBPA resins. DGEBPF resins find use as viscosity and other property modifiers of adhesives based upon DGEBPA.

![Figure 4.1](image)

**Figure 4.1 Equation describing the synthesis of the diglycidyl ether of bis-phenol-A**

Another major chemical class of epoxy resin is the epoxidized phenolic resin. Novolac phenolics of the type described in figure 4.2 can be reacted with epichlorohydrin in much the same way as shown in figure 4.1. In general, the number of epoxides per molecule is two or three. Other resins can be generated by the reaction
of epichlorohydrin with aromatic amines or with aromatic amino alcohols. Thus, materials such as tetra-glycidyl methylene di-aniline (TGMDA) and tri-glycidyl amino phenol (TGAP) are also available as materials for the formulation of epoxy resin-based adhesives. These materials are not as widely used as those based upon DGEBPA because of problems with storage stability. Both TGMDA and TGAP contain tertiary amines and possibly residual alcohol group which can catalyze alcohol-epoxy reactions to occur slowly at room temperature. Adhesives which contain these materials usually need to be refrigerated even when used as two-parts.

Figure 4.2 Equation of synthesis of a novolac phenolic resin

Part of the reason for the wide use of epoxy resins in structural adhesives is the substantial number of crosslinking reactions to which oxirane groups are sensitive. Figure 4.3 provides a list of reactions used to cure epoxy resins. Epoxy resins can react with alcohols to generate ether alcohols. The source of the alcohol can be from phenol or from alcohols generated by the reaction of oxirane with alcohols. This reaction does not normally take place at room temperature, but rather at temperatures higher than 120°C, in the presence of an amine catalyst. The reaction of oxirane with phenols or alcohols is an important curing reaction and can compete with or occur simultaneously with the reaction of oxirane with amines.
Mercaptans react with oxirane groups even at room temperature to create mercapto-ether alcohols. This reaction is accelerated in the presence of tertiary amine catalysts such as tris(dimethyl amino)phenol.

Anhydride curing of epoxy resins is infrequently used in adhesives. The reaction of anhydrides with epoxy resins leads to very hard materials and the reaction occurs at elevated temperatures. It should be noted that the reaction of an oxirane with an anhydride does not occur directly. Rather, the anhydride, in the presence of a base catalyst, reacts with alcohol to form an ester and a carboxylic acid. The carboxylic acid then reacts with another oxirane group to form the crosslink.

Lewis acids can also act as curing agents for epoxy resins, although it would be more correct to say that Lewis acids act as catalysts for the cationic polymerization of oxirane-based resins. Examples of Lewis acids which can induce cationic polymerization of epoxy resins are BF$_3$ and SnCl$_4$. Lewis acids usually are not used in their pure state, but are complexed with an amine to render them latent or to slow down their reactivity.

Epoxy resins can also be polymerized anionically. One type of anionic catalyst which has become increasingly popular is based upon imidazole. Imidazole or
2-ethyl-4-methyl imidazole can be admixed with epoxy resins to generate a material with moderate stability at room temperature. The polymerization occurs rapidly at temperatures of 82°C or above. Complexes of imidazole with metal ions such as silver can be used to create a latent catalyst which causes epoxy resins to polymerize at more elevated temperatures.

By far the most common curing agent used with epoxy resins is based upon amine. Primary amines react with epoxy resins at room temperature but rapidly at elevated temperatures. For room temperature cure of epoxy resins, the most common curing agent is based upon the primary aliphatic amine. Useful primary aliphatic amines are based upon the polymerization products of aziridine (e.g. diethylene triamine and triethylene tetramine), the reaction products of dimmer acids with diamines, polyether diamines, as well as a host of other amines. One early epoxy adhesive was sold as a two-part material with one part a DGEBA while the other part was a dimmer acid-based diamine. The most common agent for high temperature cure of epoxy resins is dicyandiamide whose structure is shown in figure 4.4. Examination of the structure of this molecule indicates that there are at least four amines available for reaction with epoxy resins. At elevated temperatures, the nitrile group can also react with alcohol to generate an amide. This reaction leads to the potential for a five-fold crosslink for this rather low molecular weight material. A substantial number of elevated temperature curing epoxy resins use dicyandiamide as the sole curative or in combination with other curatives.

![Figure 4.4 Structure of Dicyandiamide](image)

4.2.2 Elastomer Formation

The thought behind the adhesive formulations described above is that the properties of a
brittle resin can be modified with an elastomer to yield a more energy absorbing material. Phenolic resin adhesives have been used with great success in the generation of durable structures. However, the cure conditions for many phenolic adhesives require temperatures too high for some substrates and pressures high enough to crush or deform other substrates. Epoxy resins do not require high pressures for cure although some may require high cure temperatures. Therefore, many of the applications which precluded the use of a phenolic structural adhesive could be handled by an epoxy structural adhesive. Formulators of epoxy-based adhesives faced the same problems encountered by formulators of phenolic adhesives: cured epoxy resins are inherently brittle, inextensible materials. Formulators of epoxy resin adhesives were able to use two mechanisms to obtain energy absorption: flexibilization and toughening by phase separation.

When an epoxy adhesive is “flexibilized” the elastomer is soluble in the resin before and after cure. Such a situation was described above for phenolic resins. The cured, formulated, flexibilized adhesive has a single glass transition temperature lower than that obtained with the unmodified epoxy resin. Elastomer modification reduces the modulus of the adhesive so that some lap shear properties, especially those measured at elevated temperatures, decrease from those for the unmodified cured epoxy resin. The resulting adhesive is also more flexible, has higher extensibility, and thus, is better able to absorb energy as the adhesive bond is loaded.

The second method of obtaining an epoxy adhesive capable of absorbing energy is toughening by phase separation. In the method, an elastomer is chosen that is soluble in the uncured epoxy resin but insoluble in the cured epoxy resin. Such an elastomer phase separates from the epoxy resin as it cures. If the elastomer is chosen properly, the elastomer phase exists as discrete balls of elastomer uniformly dispersed in the cured epoxy resin (which we now call a “matrix” resin). The epoxy resin becomes opaque as the cure progresses.
For the elastomer phase to be effective as a toughening agent, the particle size of the balls must be about 0.2-2 microns in diameter, thus scattering visible light. In general, flexibilized adhesives remain clear unless otherwise modified by fillers or other additives. Cured, toughened epoxy resins also exhibit two glass transition temperatures, one for the cured epoxy matrix and another for the phase separated elastomer. The glass transition temperature of the epoxy resin matrix is lowered in proportion to the amount of elastomer dissolved in the matrix. In comparison to flexibilized adhesives, toughened epoxy adhesives exhibit only a moderate increase in extensibility. Flexibilized adhesives can exhibit ultimate extensions in excess of 10% (up to 50%), toughened epoxy adhesives usually only extend up to about 10%, although this depends upon the temperature of the measurement. The approximate numbers provided here are based on room temperature measurements. Toughness of a matrix resin is essentially unchanged, but able now to absorb energy and to blunt cracks which may form.

Flexibilized adhesives are obtained by using elastomers soluble in the matrix before and after cure. Thus, it is necessary to use elastomers with solubility parameters close to those of epoxy resins. This criterion is met by acrylonitrile butadiene random copolymers of high molecular weight and with acrylonitrile contents in excess of 25%. A typical formulation for a heat curing epoxy resin-based adhesive formulated for flexibilization is in Table 4.1.

<table>
<thead>
<tr>
<th>Table 4.1 Typical Formulation of a Flexibilized Epoxy Structural Adhesive</th>
</tr>
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<tbody>
<tr>
<td>Component</td>
</tr>
<tr>
<td>DGEBPA</td>
</tr>
<tr>
<td>DGEBPA-based resins</td>
</tr>
<tr>
<td>Dicyandiamide</td>
</tr>
<tr>
<td>3-(p-chloropheny1)-1, 1-dimethylurea</td>
</tr>
<tr>
<td>Hyrax rubber</td>
</tr>
</tbody>
</table>
The above describes the use of a curative, 3-(p-chloropheny1)-1, 1-dimethylurea used in conjunction with dicyandiamide. Dicyan, which was described earlier, is widely used as a latent catalyst for epoxy resin cures. Its latency is due to its insolubility in epoxy resins at room temperature and below. Dicyan cures the epoxy resin when it dissolves at temperatures in excess of 150°C. 3-(p-chlorophenyl)-1, 1-dimethylurea which is also a latent source of isocyanate, reduces the temperature at which dicyan cures epoxy resins.

4.2.3 Resin Chemistry

Epoxy resins have the epoxy or oxirane group, these are a group of thermosetting materials shown in figure 4.5 and transform into three dimensional structures by a variety of curing reactions (Behm, Gannon, 1990). This section described the uncured intermediates which are either honey-coloured liquids or brittle amber solids; they become liquid when they are heated.

![figure 4.5 Epoxy or oxirane group](image)

Most epoxy resins are formed from bisphenol A and epichlorohydrin. In the synthesis of epoxies, the liquid resins are derived by two parts: the combination of two epichlorohydrin molecules and one bisphenol A molecule, in the presence of sodium hydroxide, as shown in figure 4.6 (Behm, Gannon, 1990). It is produced the crude diglycidyl other of bisphenol A by this reaction (DGEBA). Products other than epoxies have formed including hydrolysable chlorine, bound chlorine and alpha-glycol; representing only 20 to 30% of the total volume mass of products formed. The molecular size must be kept to a minimum to keep the epoxies in their liquid form (Skeist, 1962). Solids may be formed by lengthening the chain, i.e. enlarging the alpha-glycol molecule by further reaction with bisphenol A.
4.2.4 Cure Reaction

The internal structure of epoxy resins resembles a disorderly linking of chains (or threads). These chains become longer and link up with other chains at their ends and at several intermediate positions along the chain when the epoxy is cured. A popular curing reaction is the polyaddition of epoxy groups with amines or polyamides whereby primary or secondary amine add to the epoxy ring, forming a tertiary amine. The secondary hydroxyl groups can also add to the epoxy ring, hydroxyl groups are then formed which accelerate the curing process where there is excess epoxy present.

The epoxies are thermoset in the cured state, never to return to liquid state. In this state the cross-linking of chains is dense causing better electrical properties and resistance to heat-softening, better hardening and resistance to attack by chemicals and water.

4.3 Acrylic Adhesives

Acrylic adhesives are formulated from functional acrylic monomers, which achieve excellent bonding upon polymerization. Cyanoacrylates are obtained by depolymerization of a condensation polymer derived from a malonic acid derivative and
formaldehyde:

\[
\text{CN} \quad \text{CH}_2 \quad \text{COOR} \quad + \quad \text{H}_2\text{O} \quad \xrightarrow{\text{H}_3\text{O}} \quad \left[ \begin{array}{c}
\text{CN} \\
\text{COOR}
\end{array} \right]_n \quad \xrightarrow{\Delta} \quad \text{CN} \quad \equiv \quad \text{CH}_2
\]

Cyanoacrylates are marketed as contact adhesives, and they have found numerous applications. Notable uses include surgical glue and dental sealants; morticians use them to seal eyes and lips.

Di-glycol methacrylates are obtained by esterification of ethylene glycol oligomers with meg hacrylic acid.

\[
\text{HO} \left[ \begin{array}{c}
\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \\
\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}
\end{array} \right] \quad \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH} + 2\text{CH}_3\text{C} = \text{COOH} \quad \xrightarrow{\Delta} \quad \text{CH}_2\text{C} = \text{COO} \quad \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \\
\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}
\]

These materials form highly crosslinked and therefore brittle polymers. Modifications with polyurethanes or addition of other polymers, such as low molecular weight vinyl-terminated butadiene-acrylonitrile copolymers or chlorosulfonated polyethylene, have been used to formulate tough adhesives with excellent properties.

The modified dimethacrylate systems can be formulated as two-component adhesives, with a catalyst added just prior to use or with a polymerization catalyst applied separately to the surface to be bonded. Also, one-component systems have been formulated which can be conveniently cured by ultraviolet irradiation.

\[
\left[ \begin{array}{c}
\text{CH}_3 \\
\text{CH}_3
\end{array} \right] \quad \quad \text{O} \\
\text{CH}_3
\]

4.3.1 Rapid Cure of Acrylics

Acrylic structural adhesives are important because of their rapid cure and structural strength. In comparison to epoxy and urethanes adhesives, acrylics cure in less than a minute under the proper conditions. This rapid cure property is used to great
advantage in the electronics industry (such as in bonding magnets into speaker assemblies) among others in maintaining high assembly line speeds.

Acrylic structural adhesives fall broadly into two classes: redox-activated and cyanoacrylate adhesives. The basic monomers used in the generation the first type are usually combinations of methyl methacrylate, methacrylic acid, and crosslinking agents such as ethylene glycol dimethacrylate. The cyanoacrylate adhesives are based upon a particular type of acrylic monomer. The key difference between reduction-oxidation (redox) activated and a cyanoacrylate structural adhesive is how cure is affected. In both cases, the reaction mechanism is an addition polymerization, but the redox-activated adhesives react by a free radical polymerization, while the cyanoacrylate adhesives react by an anionic addition polymerization.

Redox-activated structural adhesives rely upon some form of a redox reaction to generate the free radical initiator necessary for the inception of the polymerization reaction. The oldest structural adhesive of this type is the anaerobic adhesive. Anaerobic acrylic adhesives are based upon an initiator with a long half-life and acrylic monomers whose polymerization is inhibited by the presence of oxygen. The polymerization, however, could be inhibited by a small amount of oxygen. The adhesive was stored in oxygen-permeable bottles which provided an extended shelf life. If the adhesive was brought in contact with a surface which could initiate reduction of the peroxide (such as an iron surface containing Fe$^{2+}$ or other transition metals) and oxygen was excluded, the material polymerized. The initial use of this type of adhesive was in thread locking which involves iron bolts and the exclusion of oxygen by application of the nut.

Free radical initiators could be produced in other ways besides surface-induced reduction of peroxide. The polymerization rate of the original anaerobic acrylic adhesive depended upon the type of metal used to form the bond. For example, Fe$^{2+}$ initiated a rapid polymerization but with Cu$^{2+}$ it was relatively slow. Substantial
advances were made in acrylic structural adhesive technology when it was found that certain organic species also acted as redox couples to cause reduction of peroxide. One of the redox couples was N, N-dimethyl-p-toluidine and saccharin. These materials, in the presence of hydroperoxide, initiated polymerization of acrylic monomer, although the presence of either of these materials alone did not. In one type of two-part adhesive developed based upon this redox couple, either the saccharin or the N,N-dimethyl-p-toluidine is dissolved in a solvent and this solution is applied as a primer to one of the adherends to be bonded. After the solvent has evaporated, the remaining adhesive mixture (monomers and the other half of the redox couple) is applied to the other adherend. Joining the adherends initiates the polymerization in the zone where the two parts of the adhesive meet. Another type of an adhesive using this technology is a two-part mix adhesive in which half of the redox couple is mixed in each part. When the two parts are combined, initiation and polymerization ensue.

Cyanoacrylate adhesives are unique in structural adhesive technology. The structure of the basic monomer is shown in figure 4.7. The double bond, due to its proximity to two strong electron withdrawing groups (the nitrile and the ester group) is extremely sensitive to nucleophilic attack. In fact, this monomer, during its synthesis from alkyl cyanoacetylene and formaldehyde in the presence of base, polymerizes immediately upon formation. The monomer is obtained by phosphorus pentoxide/phosphine-induced depolymerization and distillation from the formed polymer. The susceptibility of the double bond to nucleophilic attack provides a monomer whose polymerization can be initiated by species as weakly nucleophilic as water. Various bases can also be used to induce polymerization of cyanoacrylates but acids inhibit the process. The extreme reactivity of the monomer results in fast cure. Cyanoacrylate monomers can contain ester groups from methyl, ethyl to isobutyl and ethoxy ethyl, although the most commonly used monomers for adhesives are the first two. All of these monomers are clear, colorless, low viscosity liquids with pungent odours. Free radical stabilizers such as hydroquinone are added to improve storage stability as is sulfur dioxide which reduces the possibility of ionic polymerization. One key factor not normally
recognized regarding cyanoacrylate adhesives is that these materials, unless specially formulated, are thermoplastics after cure. This makes the cured cyanoacrylate susceptible to creep as well as attack by moisture. Despite these limitations, they have become quite popular commercially and many private label packagers of adhesives have made this product available to the consumer under trade names such as “Crazy Glue” and “Super Glue.”

![Figure 4.7 Structure and anionic polymerization of a cyanoacrylate](image)

4.4 Structural Adhesives

The first major type of adhesive discussed here is the structural adhesive. This group encompasses those materials with high cohesive strength used to bond adherends also with significant cohesive strength. A reasonable definition of a structural adhesive is a material used to bond other high strength materials, such as wood, composites, or metal, so that the practical adhesive bond strength is in excess of 6.9 Mpa (1000 psi) at room temperature. Because of the demands on performance, structural adhesives are usually cross-linkable (thermoset) organic compounds, are usually polar, and of high surface energy. They are usually resistant to many types of environmental attack. In fact, in many applications, structural adhesives are required to survive in adverse environments for years, if not decades.

Newer adhesives of the acrylic, anaerobic or radiation-curable types must, if they are structural and to compete with or challenge epoxy adhesive systems, have a relatively high degree of toughness and durability. Newer radiation-curable, pressure-sensitive
adhesive systems must exhibit the properties of permanence largely associated with cross-linked adhesive masses deposited from and acrylic solution polymer base similarly. Epoxy resin structural adhesives largely define the existing area on the one hand; the cross-linked acrylics deposited on plastic or metallic films the other.

4.4.1 High Performance Structural Adhesives

The adhesives described above are limited in their resistance to high temperatures. As already discussed, the thermoplastic nature of cyanoacrylate adhesives prevents them from resisting creep at high temperatures. The upper temperature limit for addition polymerized adhesives is normally the glass transition temperature of the cured monomer. For urethanes, at the upper temperature limit, usually between 120 and 150°C, the urethane unblocks. Epoxy structural adhesives usually exhibit a decrease in performance at about 30°C above the cure temperature. Phenolics can resist temperatures substantially higher than the cure temperature as demonstrated by the ablative material used in the Mercury and Gemini space programs. For the most part, epoxies and phenolics have an upper temperature limit for long term exposure of about 204°C. However, there are uses for materials which exhibit resistance to long term exposure to temperatures in excess of 204°C such as in internal combustion and turbine engines and adhesives for fuselage and other structure in high speed commercial airplanes. Many of the adhesives which we discuss shortly were developed to meet the needs of the SST (supersonic transport) program of the late 1960s. One key feature which characterizes high temperature performance adhesive materials is their highly aromatic character (which provides oxidative resistance) and the plethora of bonds formed upon polymerization. To sever the polymer chain, two bonds have to be broken.

The polymer type most examined for high temperature applications is based upon polyimides. The general polyimide reaction is shown in figure 4.8. The diamines are aromatic and can range from methylene diamine to diaminldiphenylether. The
anhydrides can range from acid anhydride to benzophenone tetra-carboxylic acid dianhydride to 4, 4'-hexafluoropropylidene-bis- (phthalic acid). Various structures of polyimide components have been studied to improve the performance of these structural adhesives. The key feature is polyimide polymerizations that each step involves the elimination of water. Both high temperature ad high pressures are involved in each stage of cure to force the water out of the bond when the adherends are non-porous. Polyimide cure temperatures are usually above 220°C. Polyimides are inherently thermoplastic and creep under high loads at high temperatures.

4.5 Configuration of the Polymer Molecule

The majority of polymeric molecules which already have some form and structure are based on organic carbon from naturally occurring hydrocarbons. Most of them are short chains with repeating units or monomers, with carbon comprising the backbone of the chain and other atoms or groups of atoms satisfying the remaining bonding possibilities. Processing is carried out, to extend the length of the backbone chain. The other bonds may also the modified through processing. The extended chain
configuration has proven to have useful properties and as a consequence a great deal of effort has gone into development of procedures through which the resulting chains conform to a specific range of lengths.

The reaction is usually controlled so that a linear polymer results when short chains are reacted to produce longer ones. This means that the backbone atoms will have another backbone atom joined to them on each side but not in other directions. Having a bond on each side of the backbone does not result in the backbone being a straight line necessarily, since the bonding angles vary with the mature of the bond. The most common form of bonding is the single bond with the $p$ orbital being involved. Since these results in a $110^\circ$ bond for most backbone forming atoms, the resulting backbone has a zigzag arrangement. This is illustrated in figure 4.9.

![figure 4.9 A schematic old part of a carbon chain](image)

4.5.1 Crystallization

Polymers can conform to a crystalline array, but the difficulties of arranging long chains in three dimensions are such that crystallinity cannot be completely achieved. The degree of crystallinity and the size of the crystals are dependent on how energetically favorable the arrangement is. For polymer chains that have dipoles or are polarizable the intermolecular forces (van der Waals bonds) will encourage close and regular proximity, encouraging crystallinity. On the other hand, if the chain is atactic or has bulky pendant groups, then the spacing between the chains will be large due to the spatial relations of fitting these irregularities into space. The increase in distance between the chains reduces the effectiveness of the intermolecular attracting forces.
When this occurs, the polymer is capable of very limited crystallinity. The ability to crystallize does have an effect on the size of the crystals as well. If the polymer has irregularities that make alignment for crystallizing difficult, these irregularities will also impede the number of chains that can be brought into alignment within a space. Thus, polymers which have limited crystallizing ability also tend to produce crystals of small size.

The long chains of a polymer demand more time and mobility to arrange themselves into a crystalline array than other types of materials. As such it is possible to quench a polymer from the melt past the glass transition temperature without producing a crystalline structure. Once a polymer is below its glass transition temperature, the chains do not have sufficient mobility to rearrange for crystallization or any other diffusion-dependent change. A depression of the molten polymer to approximately nine tenths of the melting point (on the absolute scale) will usually result in the maximum crystallization rate. Once the conditions are favorable for crystallization, the process of nucleation and growth is similar to other crystalline materials. The nucleus usually starts as a small alignment of chain segments, which have come together due to random thermal motion. Growth of the nucleus can be encouraged by a directional external force. The force tends to align the chains in its direction, thus helping to order the chains for crystallization. Once crystallization is attained, the polymeric chains are closer together than they would be in their amorphous state. Consequently, the intermolecular attraction between the chains is stronger and the material is more stable. A manifestation of this is the increased melting point of a crystalline polymer compared to the same polymer in the amorphous condition. The arrangement of crystalline polyethylene in a unit cell can be seen in figure 4.10.
This schematic shows the alignment of polyethylene chains to form part of the unit cell of a crystal. The arrows indicate the continuation of the three chains shown. The center chain is only outlined so as to indicate that it is not in the plane of the other two chains shown. To complete the unit cell (partially marked by the solid straight lines) would require two more chains of polyethylene aligned as the two painted chains are but in a plane farther beyond the outlined chain. The creation of just one unit cell can serve as the nucleation point for the subsequent crystal growth.

4.6 Adhesive Joint of Composite

In figure 4.11 illustrated a number of popular techniques for joining composite to composites, metals to composite materials. Many other possibilities exist, particularly when including honeycomb or foam-type structures such as a component in a composite structure. It is often impossible to access to a particular surface to use a particular non-destructive testing technique. The purpose of a non-destructive inspection procedure is therefore to develop a technique; it can obtain a practical measurement once the structure is completely bonded for an evaluation of its integrity, or to possibly predict performance or strength. Joint design should therefore think over inspection potential in the design process as well as strength and other performance values.
Three common bond situations are shown in figure 4.12. The voids and debondings are easily discovered with classical ultrasonic inspection techniques and with continuous wave resonant testing such as microwave signature curve method which will discusses at chapter 8. The most perplexing situation is the so-called good bond where bond material is continuous between the base and substrate, but the joint performs poorly. This can be caused by possible surface roughness, by faulty surface preparation (for example, omission of the acid etch or primer), or even other processing anomalies. It is the special situation that we are addressing with sophisticated signal processing and pattern recognition analysis, since classical ultrasonic techniques cannot detect these subtle anomaly possibilities.

Assuming that every parameter or variable could be monitored accurately throughout the entire substrate preparation and bonding process, reliability could still not be assured which is in acquiring consistently high-quality bonds because of the possibility of
contamination or equipment malfunction at any stage of the process. Therefore, the goal for bond classification is to obtain a non-destructive measurement and evaluation of bond quality once the bond is completely manufactured and then to predict the performance potential with a high degree of confidence.

In order to proceed with this goal, an understanding of the microwave wave interaction mechanisms with an adhesive bond or a layered composite structure must be thoroughly understood. Physical models of a bondline or a composite material structure must be formulated.

Many theoretical models that treat wave propagation travelling across a layered media representing an adhesively bonded structure have been formulated in the past. The model illustrated in figure 4.13 can be used with governing wave equations and appropriate boundary conditions for either normal or oblique incidence to calculate amplitude and phase changes of an radiation signal, as well as attenuation variations as a function of frequency that can assist us in both transducer and test selection and also in feature selection for advanced analysis in pattern recognition. A situation is illustrated that modifies the reflection factor at the interface which involving an area of discontinuity. Alternatively, a thin layer simulating the interface could be choosing in an alternative physical model. This idea qualitatively handles surface preparation problems. The center portion with several layers treats the cohesive problem of under cure and over cure. A traditional approach of handling an individual interface as “smooth” or “welded” can also be handled from a mathematics and mechanics point of view.

<table>
<thead>
<tr>
<th>DISCONTINUITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>ADHESIVE INTERFACIAL LAYER</td>
</tr>
<tr>
<td>(modeled as layers with property gradients)</td>
</tr>
</tbody>
</table>

Figure 4.13 Possible theoretical model for studying radiation interaction with an adhesive (containing cohesive and adhesive weaknesses)
4.6.1 Surface Roughness

Surface roughness can affect the spread of adhesive either because the adhesive cannot penetrate into the pits or because it does so too slowly and sets before it completes the contact. The first condition will be determined by the contact angle between the liquid adhesive and the surface, the shape of pits, and by whether air or vapour in pits in the surface can escape in front of the advancing liquid. The latter question is whether the pits are interconnecting or not.

4.6.2 Joints, Rigid and Flexible

In general, adhesives are used to make joints between solid materials, shape or stiffness; the solid materials may be of any matter. The adhesive is present to keep from a load separating the bodies. If there is no load, adhesive is unnecessary. To study the effectiveness of adhesives in securing bodies the loading conditions that can exist need to be perfected so that situations can be represented by models in which the stresses on the adhesive and its interface with the adherends can either be calculated or control by experimental test. Thus, the cleavage stresses tending to open the ends of a simple lap joint and the maxima of shear and other stresses concentrating at or very close to the limit of several types of joint are present however the members of the joint are secured.

4.6.3 Lap-Shear Joints

The lap-shear joint is that used almost in testing adhesives or surface preparation techniques generally. If an adhesive is to be used in a known stress situation, data derived from experiments in torsional shear will enable design forecasts to be made. Frequently, however, although the principal stress is known, there are complicating effects which arise from bending or twisting and which lead to cleavage stresses at the ends of one or both adherends. Such a state of affairs exists when a stiffening member is added to a stresses surface. The member is naturally applied in the direction of the
important stress but necessarily terminated before the stress is delivered from the surface. Because of its ubiquity, the lap-shear joint has been the subject of a number of studies to determine the nature of the stress distribution when it is loaded. The final aim of such studies is to calculate the breaking load of a joint from its geometry and the known ultimate tensile strength of the adhesive material (Bikerman, 1968).

It has been found that the tensile stress in adherend is linearly related to position along the joint length. With free body diagram for the adhesive will show a tensile stress in the centre will be a small portion of the total tensile stress across the joint because most of it will be borne by the adherends (figure 4.14). The stress will be almost exactly one half of the stress at the far ends of the adherends which across the centre of the joint.

![figure 4.14 Stresses in lap joint adherends.](image)

The existence of this tensile stress will give rise to a shear in the adhesive which is non-uniform and is superimposed upon the shear due to loading of infinitely rigid adherends. This arises from the increased strain where the load borne in the centre by two adherends is transferred to one adherend (figure 4.15).

![figure 4.15 Shear in adhesive due to extensibility of adherends.](image)
4.6.4 Adhesive Stress-Strain Characterization

The measurement has presented much more of a challenge which has of adhesive peel stress-strain characteristics. Difficulties arise because the adhesive layer, in a bonded joint, is neither homogeneous nor isotropic. It is forced by the adherends, so there are both edge effects and boundary layer effects. It is filled with special fibres to toughen its resistance against peel failures. And, in many cases, the adhesive is actually a two-phase system of a rubbery phase (vinyl, nylon, or nitrile compound) in a relatively brittle matrix (epoxy or phenolic). The situation is often overlooked in using interaction criteria which established on the basis of in-plane (tensile) and twisting (torsion) testing of cured adhesive film specimens. The adhesive peel modulus can be used easily enough by compressing a sandwich of adherends and adhesive. But the measurement of the associated microscopic tensile strain to failure is far more difficult. The same difficulty has arisen in regard to fibrous composites. A further complexity is bonded which is due to the biaxial restraint of the adherends to which the adhesive. Adhesives are close-to-incompressible materials which like most long-chain polymers. If the adherends prevent deformation in two perpendicular directions and the material were truly incompressible, there could be no deformation in the third mutually perpendicular direction either. Thus, the present state of the art permits no more than linear elastic modelling of the adhesive under peel loads. This is not as great a handicap as it might see, since the prime interest in adhesive peel stresses is in identifying when they have become a problem so as to redesign the joint and eliminate them. In the case of fibrous composite adherends, the location of any peel stress failure will be in the matrix rather than the adhesive, so there is no need to develop models for adhesive behaviour which are prevented from occurring by prior failure elsewhere in the joint.

Figure 4.16 shows a typical adhesive stress-strain characterization in shear. It is apparent that the one drawback of the elastic-plastic model is that it violates from the elastic behaviour. Of course, one can use the linear elastic characteristic whenever the
applied loads are low enough. Thus, the elastic-plastic model can be made as accurate as can be demanded. In any event, the greatest interest for which a precise elastic model is not important is usually in the failure condition. The basic advantage of the bi-linear model is that it is closer to the true characteristic of the adhesive throughout its entire range of deformations, so a single model can be used to compute the internal stresses associated with a specified external load for which it is not known, \textit{a priori}, to what degree the adhesive is being stretched. The case for the bi-linear model, or any other more precise model, is therefore based on convenience rather than precision.

![figure 4.16 Adhesive shear stress-strain curves and mathematical models.](image)

The adhesive characteristics change with temperature, as shown in figure 4.17, and similarly with moisture content. It is important that the strain energy to failure does not vary with the environment anywhere near as much as the individual characteristics do. Therefore, the strength of structural joints is not as sensitive to the environment as is the strength of typical short-overlap test coupons. Nevertheless, the changes in characteristics are sufficiently pronounced that the joint proportions which lead to the longest total overlap must be established on the basis of those conditions in figure 4.17, if that insensitivity is to be attained. Figures 4.16 and 4.17 pertain to typical modified epoxy ductile structural adhesives.
Sometimes, much more brittle adhesives are used because of high operating temperatures. The difference in behaviour of such adhesives is shown in figure 4.18. However, even the brittle adhesives show proportionally significant non-linear behaviour near the upper limit of their operating environments. So the linear theory does not suffice for even brittle adhesives. Since the brittle structural adhesives are remedied at temperatures even higher than their peak operating environment in general, whenever they are used to bond dissimilar materials together (as with metal fittings to fibrous composites) there is influence about fracture under internal residual stresses at the low end of the operating environment.

In steep-lap joint analyses in which the steps are in the adhesive, rather than the adherend, show the consequences of non-uniform adhesive moisture absorption.
Examples include moist edges and dried out edges after the interior has absorbed moisture. In the theory developed, no provision is made for variation in adhesive properties. However, by breaking up the joint into a series of segments, with uniform adhesive properties throughout each segment, such more complex problems can be analysed.

4.6.5 Definition of Mechanical Stresses for Materials Testing

There are three types of forces which can be applied to an adhesive (or to any material, for that matter). These forces are shown schematically in figure 4.19 and figure 4.20. The first is the tensile measurement (figure 4.19). In this test, a bar of material of known dimension is firmly clamped into a tensile testing machine and a force is applied to the axial dimension of the sample.

![figure 4.19](image_url) a bar of material to which a tensile force, F, is being applied.

In the second measurement shown in figure 4.20, a sample of known dimension is subjected to a force, $F$, on its faces in such a way that the forces oppose one another. This force is known as a shearing force which is of great importance in the testing and performance of adhesive bonds. Most adhesive bonds are designed to subject the adhesive to shear, rather than tension or cleavage forces.

![figure 4.20](image_url) Application of a tangential force to a rectangular shaped material.
4.6.6 Tensile Forces

In the tensile test described in the previous section, if the sample has a known cross sectional area, $A$, and $F$ is the force applied by the tensile testing machine, then we define the tensile stress, $\sigma$, as

$$\sigma = \frac{F}{A}$$  \hspace{1cm} (4.1)

The tensile stress is an important engineering concept that the force, $F$, is applied over a specific cross sectional area of sample. Many different materials could have the same elongation with applied force $F$ if the cross sectional area of the sample was the appropriate size. Elongation is the change in the length of the sample as the result of tensile forces. The elongation of the sample is defined in terms of the original dimensions of the specimen. Thus, if the original length of the sample is $l_0$ and the length (displacement) of the sample after a certain amount of tensile stress was applied is $l$, then we can define the engineering tensile strain ($\varepsilon$) as follows

$$\varepsilon = \frac{(l - l_0)}{l_0}$$  \hspace{1cm} (4.2)

It is important to note that the engineering tensile strain is a dimensionless number and is usually reported as a fraction or multiplied by 100 to obtain a percentage called elongation.

A stress-strain plot can be generated using a tensile testing machine. A known cross-sectional area specimen is subjected to a tensile force and the elongation is measured, as shown in figure 4.21.

![Schematic stress-strain curves](figure 4.21 Schematic stress-strain curves.)
The tensile stress is plotted on the y axis and the engineering tensile strain is plotted on the x axis. In some temperature of tests, many materials have stress-strain plots similar to that shown in figure 4.21. Stress-strain plots for individual material differ in the initial slope, the position of the knee, the length of the plateau portion of the curve, and the elongation at break, which is indicated by X-axis.

As shown in figure 4.21, the stress point at which the stress-strain curve exhibits a “knee”, which is known as the yield stress of the material. This parameter plays an important role in our understanding of resistance to crack propagation as well as one of the parameters necessary for the proper design of adhesive bonds. The tensile yield stress is given the symbol \( \sigma_y \) and it marks the stress or strain that the material no longer follows Hooke’s Law. After the yield stress is reached, the material in non-elastic is said to have been plastically deformed. Plastic deformation is a sign that the material is absorbing energy.

Not all materials display the plateau region is shown in figure 4.21. The plateau is seen in materials which “neck-in” or “draw down” as the sample elongates. Note that in the plateau region, the stress on the sample can actually decrease.

Eventually, the material can no longer sustain the stress and it breaks. The stress at this point is known as the stress at break while the strain at this point is known as the strain at break or the elongation at break of the material. The ultimate tensile strength of the material could be the stress at break. However, the ultimate tensile strength of the material could occur when the material begins to neck-in. All of these properties govern the potential uses for a material and also guide the adhesive-user in determining what kind of adhesive could be made from the material.

When most materials are subjected to a tensile stress, not only they sketch but also become thinner in cross section in order to conserve volume. We can define a
parameter which describes how much the material thins in response to a tensile stress in the following manner:

$$v = \frac{(r_0 - r)}{r_0 \varepsilon}$$

(4.3)

where $r$ is the radius of a cylindrically shaped tensile specimen at a certain stress, $r_0$ is the original radius and $\varepsilon$ is the tensile strain. The quantity $v$ is the ratio of the lateral strain to the tensile strain and is known as Poisson’s ratio. This quantity is measured while the material is within its elastic region. For isotropic materials, i.e., materials that have the same properties in every direction, Poisson predicted that the ratio should be 0.25. In fact, many materials have a Poisson’s ratio close to 0.25, as shown in Table 4.2. The unit volume expansion, $\Delta V$ of a material in tension can be calculated from the expression: $\Delta V = \varepsilon (1 - 2v)$. Most materials do not exhibit a decrease in volume when put into tensile stress. Therefore, $v$ has an upper value of 0.5. Materials such as rubber have Poisson’s ratio approaching 0.5. Steel has a Poisson’s ratio of 0.26.

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus (Pascal = N/m²)</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>$7 \times 10^{10}$</td>
<td>0.33</td>
</tr>
<tr>
<td>Mild steel</td>
<td>$2.2 \times 10^{11}$</td>
<td>0.28</td>
</tr>
<tr>
<td>Silicon</td>
<td>$6.9 \times 10^{10}$</td>
<td></td>
</tr>
<tr>
<td>Glass</td>
<td>$6 \times 10^{10}$</td>
<td>0.23</td>
</tr>
<tr>
<td>Polymethylmethacrylate</td>
<td>$2.4 \times 10^{9}$</td>
<td>0.33</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>$1.4 \times 10^{9}$</td>
<td></td>
</tr>
<tr>
<td>Low density polyethylene</td>
<td>$2.4 \times 10^{8}$</td>
<td>0.38</td>
</tr>
<tr>
<td>Natural rubber</td>
<td>$2 \times 10^{6}$</td>
<td>0.49</td>
</tr>
</tbody>
</table>
4.6.7 Shear Forces

Figure 4.21 shows the application of stress to the surfaces rather than the ends of a bar. We need to analyze this situation differently from tensile stress assume that we know the area over which the force is applied. The name shear stress is given to the force per unit that is applied tangentially as shown in figure 4.21. The symbol \( \tau \) denotes the shear stress and has the same units as the tensile stress.

4.7 Theories of the Adhesion

There are many theatrical illustrations for theories of adhesion:

- Mechanical interlock theory;
- Physical adsorption theory;
- Electrostatic theory;
- Diffusion theory.

4.7.1 The Mechanical Interlocking Theory

The idea of this theory is that the interlocking or keying of the adhesive with the surface irregularities of the adherend is the foundation of the adhesive reaction. Roughened surface adherend, according the theorical indication which yield sound adhesion. Figure 4.22 shows a mechanical interlocking of an adhesive joining system.

figure 4.22 Mechanical Interlocking of an Adhesive Joining (Allen, 1987)
4.7.2 The Absorption Interaction Theory

The adsorption interaction theory tries to explain the adhesion is attributed by different forces between the adhesive and the adherend. These forces are ionic attractions, covalent bonds between the adhesive and the adherend. The forces interacts included ionic attractions, covalent bonds, hydrogen bonds, and Van der Waals’ force. Figure 4.23 shows an adsorption interaction of the adhesive joining system.

![figure 4.23 Adsorption Interaction of an Adhesive Joining (Allen, 1987)](image)

4.7.3 The Electrostatic Theory

The electrostatic theory which proposed by Derjaguin et al. (1957) explains that the formation of charges of opposite signs across the interphase between dissimilar materials is the cause of adhesion. An electrical double layers, prior to adherend separation, has been created at the interface. The adhesive bond is analysed as a capacitor and the adhesive strength is the force responsible for separating the plate of charged capacitor as different charges attracts each other. Figure 4.24 shows an electrostatic adhesive joining system.

![figure 4.24 An Electrostatic Adhesive Joining System (Allen, 1987)](image)
4.7.4 The Diffusion theory

The diffusion theory, which was proposed by Voyutskii (1963), describes adhesion as a result of inter diffusion between the chain of molecules on the surface of the materials. Diffusion mechanism theory actually contributes greatly to an understanding of adhesive strength, which in many cases involving polymer bonds. This theory is not able to explain metal-polymer bonds because polymers are not soluble with metals. Figure 4.25 shows diffusion of an adhesive system. The adhesive polymeric molecules diffuse into the polymeric molecule substrate creating a strong joint.

![Diffusion Adhesive System from the Adhesive Molecule into the Polymeric Adherend (Allen, 1987).](image)

4.7.5 The Weak Boundary Layer Theory

The weak boundary layer theory suggests that clean surfaces are necessary requirement to create a strong adhesive joint but it often prevents this from occurring, due to a layer of contaminant, which is cohesively weak. The present of oil, grease, and dirt can cause a weak boundary layer between the adherend an adhesive. A strong adhesive joint can be made on a dry adherend only. It is not possible to make a strong joint on low surface energy adherend polymers such Polyethylene, Polypropylene, Polytetrafluorethylene (Teflon), and Silicon. Figure 4.26 shows a weak boundary layer adhesive joining system.
Summaries of the above mentioned theories are illustrated by Table 4.3 & 4.4 (A. J. Kinloch, 1987). Criticism of the above adhesion theories have been made by experts and are summarised in the Table 4.3.

Table 4.3 the Critics of the Adhesion Theories.

<table>
<thead>
<tr>
<th>Adhesion Theory</th>
<th>Critics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical interlocking</td>
<td>An improved adhesion due to greater roughness in the adherend does not necessarily come from the mechanical keying mechanism because it is true when the topography of the adherend surface is in favor of the mechanical interlocking</td>
</tr>
<tr>
<td>Adsorption interaction</td>
<td>No critics is found</td>
</tr>
<tr>
<td>Electrostatic</td>
<td>No critics is found</td>
</tr>
<tr>
<td>Diffusion</td>
<td>Limited application for polymer to polymer joining only because polymer can not diffuse into metallic molecules</td>
</tr>
<tr>
<td>Weak Boundary Layer</td>
<td>No critics is found</td>
</tr>
</tbody>
</table>
Table 4.4 the Strengths of Forces Which May Contribute to Adhesion.

<table>
<thead>
<tr>
<th>Type of adhesion forces</th>
<th>Strength (kJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Van der Waals</td>
<td></td>
</tr>
<tr>
<td>□ Induced dipole-induced dipole (dispersion or London Forces)</td>
<td>0.08-40</td>
</tr>
<tr>
<td>□ Dipole-induced dipoles (Debye Forces)</td>
<td>&gt;2</td>
</tr>
<tr>
<td>□ Dipole-dipole (Keesom Forces)</td>
<td>4-20</td>
</tr>
<tr>
<td>Chemical Bonds</td>
<td></td>
</tr>
<tr>
<td>□ Covalent</td>
<td>60-700</td>
</tr>
<tr>
<td>□ Ionic</td>
<td>600-1100</td>
</tr>
<tr>
<td>□ Metallic</td>
<td>110-350</td>
</tr>
<tr>
<td>Hydrogen Bonds</td>
<td></td>
</tr>
<tr>
<td>□ Involving Fluorine</td>
<td>Up to 40</td>
</tr>
<tr>
<td>□ Not involving Fluorine</td>
<td>10-25</td>
</tr>
<tr>
<td>Acid-base interaction</td>
<td></td>
</tr>
<tr>
<td>□ Conventional Bronsted</td>
<td>Up to 1000</td>
</tr>
<tr>
<td>□ Lewis</td>
<td>Up to 80</td>
</tr>
</tbody>
</table>
5. VFM Experimental Facilities and Processing

The most commonly used facilities for microwave processing materials are of fixed frequency, e.g. 2.45 GHz. This research presents a state-of-the-art review of microwave technologies, processing methods and industrial applications, using variable frequency microwave (VFM) facilities. The technique is geared towards advanced materials processing and chemical synthesis. It offers rapid, uniform and selective heating over a large volume at a high energy coupling efficiency. This is accomplished using a reselected bandwidth sweeping around a central frequency employing frequency agile sources such as travelling wave tubes as the microwave power amplifier. Selective heating of complex samples and industrial scale-up are now viable. During VFM processing, a given frequency of microwaves would only be launched for less than one millisecond. Two such facilities are available in the Industrial Research Institute, Swinburne (IRIS), Swinburne University of Technology, in Melbourne, Australia. Microwave 2100 Model 250 (figure 5.1) with a maximum power output of 250W generates microwave energy in the frequency range of 2- 7 GHz and the other, Vari-Wave 1500 (figure 5.2), operates at 6-18 GHz with a maximum power level of 125W. The cavity dimension of VW1500 was 250 mm x 250 mm x 300 mm; while, Microcure 2100 model 250 had a cavity size of 300 mm x 275 mm x 375 mm.

![figure 5.1 Variable Frequency Microwave. Power: Max 250 Watts. Frequency Range: 2.5 to 8 GHz. Sweeping Rate: Min 0.1 sec (Bandwidth Dependent)](image)
In this research, one type of adhesive joints, namely, single lap shear was chosen and prepared in accordance to ASTM D 3176-72. The resulting adhesive joints were then subjected to tensile bond shear test using an Instron machine 4303 which was run automatically using specialised software.

A Polariscope was also used to assist in the identification of thermal stresses which present in the adhesive joints soon after curing was performed in the fixed variable frequency microwave unit. Prior to adhesive joining, photostress was initially taken for the polycarbonate sheet-strip adherends after they were mounted into a Polariscope using a tripod-mounted camera loaded with 35mm film. This was to reveal and record any residual stresses which might be present in the adherend strips prior to adhesive joining. Photostress was subsequently taken on the adhesive joints after curing was performed to reveal any possible thermal stress which might be present on the adhesive joint area as a result of curing. Thermal stresses were indicated by the presence of fringes from the analyser lens. Furthermore, all adhesive joints were taken for tensile testing to determine the individual tensile bond strengths using at tensile strength testing (Instron) machine.
The fracture joints were also examined visually to determine whether joint failures occurred cohesively (in the adherend) or adhesively (in the adhesive). Selected fracture joints then underwent surface analysis using Scanning Electron Microscope (SEM) to see physically the adhesive layer and or the heat affected zones of the welding joint.

Results of all individual tensile testing were recorded and analysed. An average value was calculated from the number of tensile bond strength data for further analysis. Comparison of the average tensile bond strengths was made for those adhesive joints which were cured using the microwave heating technique and convectional oven respectively to identify the highest and lowest average tensile bond strengths. Those results were then justified and analysed using data obtained from the photostress, SEM, and spectroscopy to find correlation and/or trends.

5.1 Microwave Equipment

The Microcure 2100 (figure 5.3) is comprised of several separate subsystems. The subsystems include the curing cavity, oven control system, signal generator and high power amplifier system, transmission system, and temperature monitoring system.
This section provides detailed installation and operation instruction for the Vari-Wave Model LT 1500, designed and built by Oak Ridge National Laboratory for process research and development on a wide range of materials, chemical analysis and measurements of electromagnetic interaction. The system is intended to operate over a broad range of process parameters for development on a variety of application into a variable frequency. The system is also designed for interface with a number of options, including computer data acquisition and characterization routines.

The VW 1500 incorporates Vari-Wave operator control system and provides for setup of process parameters consisting of level of microwave power provided to the processing cavity (nominally 0-120 W). Incident center frequency (6.0 to 18.0 GHz), frequency bandwidth (0 to 100%), and bandwidth sweep rate (100-0.1 second). The set point for incident power is via a manual potentiometer on the front control panel. Power level output from the TWT will vary from a minimum at the band edges to a maximum peak near the center of the band. All other parameters are programmable via the Vari-Wave microprocessor control module. Sample loading is by a manual door opening on the front of the multi-mode cavity. The entire system is housed in a single, tabletop cabinet with convenient controls and interface panels for data and/or service connections. An overall pictorial schematic of the LT 1500 Vari-Wave system is shown in (figure 5.4) below.

figure 5.4 Vari-Wave Model LT-1500 Interface Panel (operator manual VW1500 model, undated)
Requirements in the electronics industry for smaller size, higher level interconnect and densities are leading to new demands on packaging and a greater use of polymeric materials for bonding and for protective and compliant joints. This trend has added increased time and thermal management considerations for many manufacturing processes within the industry. As a result, design tradeoffs between materials selection and production rates are common. A new processing technique introduced by VFM. These systems enable rapid and controllable curing without compromising properties is now available.

This well-established processing method for advanced polymeric composites, known as “Variable Frequency Microwave (VFM) Energy” has been applied to electronic materials processing applications, such as curing encapsulant or adhesives, polyimides and bonding electronic components onto flexible and/or rigid circuit boards. A closed loop computer control system allows for intelligent processing and programmable thermal cycles.

The unique advantage of variable frequency microwave processing over conventional fixed frequency microwave processing is its ability to provide uniform heating over a large volume at a high energy coupling efficiency. When microwave energy of a given frequency is launched into a multimode cavity, many resonant modes are established. Those areas with higher electromagnetic field strength result in more heating, creating hot spots. During fixed frequency microwave heating, the electric field pattern is fixed. In other words, the hot spots tend to stay at relatively fixed locations. Therefore, fixed frequency microwave heating normally results in a non-uniform temperature distribution within the load. With variable frequency microwave heating, more than one thousand frequencies are launched into the cavity sequentially. Each incident frequency establishes different electric field patterns and therefore results in hot spots at different locations. Different areas are heated under different frequencies. When a sufficient bandwidth is used, every element of the load will experience hot spots at one or more frequencies during sweeping. Therefore,
time-averaged uniform heating can be achieved with proper adjustment of the frequency sweep rate and sweep range, though the electromagnetic-field pattern has hot and cold spots at any given instant. In other words, electric field patterns inside the material are changing constantly during variable frequency microwave heating and the heating pattern at any given moment is based on the frequency launched. The uniform temperature rise during variable frequency microwave heating is a time-averaged result.

5.1.1 Subsystems

(1) Curing Cavity

The cavity (figure 5.5) is the metal enclosure where the curing process takes place. The cavity features a manual hinged door with microwave seals around the perimeter of the door facing. The door seals provide good electrical contact and prevent microwave leakage from the cavity enclosure. The cavity is constructed from high-grade aluminium, and contains inlet and outlet vent ports, pass through ports for the fibre optic temperature probes, which yield a precise east minute temperature measurement of the heated load.

![Cavity Chamber Microwave 2100 model](image)

The sample being processed should not be placed directly on the bottom of the curing cavity. To ensure proper microwave field distribution, the sample was placed on the top face of the Telfon Base above the metal cavity floor (figure 5.6) and a Telfon Slot
Locator (figure 5.7). Since Telfon is zero dialectical loss material, all these Telfon fixtures are ensuring that the sample will have all the energy radiation within the cavity. The temperature probe will be attached on the adhesive area, which reflects the temperature condition during the curing process.

Metallic enclosures called cavities are used instead because of their relatively large size and high efficiency. The cavity is resonant, therefore, at this wavelength. Cavities can take any shape, but the most common shape used in microwave processing is rectangular.
(2) Software System

The oven control system (figure 5.8) is utilized to maintain control over all system components. It is software driven system that supervises and control.

An industrial computerize workstation (ICW) serves as the central data collection and control point. It interfaces with, and monitoring, all of the subsystems of the VFM facilities. It is responsible for operating the graphical user interface as well as maintaining input and output functions throughout the system.

![Diagram of variable frequency microwave control and data acquisition](image)

**figure 5.8 Variable Frequency Microwave Control and Data Acquisition.**

(3) Signal Generator and High Power Amplifier System

The signal generation system is comprised of an oscillator: a voltage controlled attenuator. ICW data acquisition and the VFM interface board. It generates all of the system’s low-level microwave energy and controls all of the systems high-level energy. The high power amplifier amplifies the low level microwave energy generated from the signal generator section to levels high enough to provide adequate molecular excitation in the curing cavity.
The high power amplifier (HPA) is comprised of a Travelling Wave Tube, a High voltage Power Supply, a solid-state Amplifier that serves as an Intermediate Power Amplifier, and control logic. It communicates with the ICW via an RS-232-C serial communications link.

5.2 Cavity Characterization Function of VFMF

Once access is achieved the VFMF Cavity Characterization screen (figure 5.9) is displayed. Cavity characterization is a sequence whereby the operator can graphically see how the cavity with material loaded will operate over the operating frequency range.

Data (figure 5.10) is displayed on graphs showing forward and reflected power versus frequency or reflectance versus frequency. From this data, the operator can make an educated estimation for bandwidth values used during event programming.

*figure 5.9 Manual Control Program Main Screen (operators manual, microwave 2100, 1998)*

*figure 5.10 Cavity Characterization Menu Selections*
First, the operator selects the power level that will be used during the sequence; Second, the operator can select whether they want to save the sequence data to a file for later viewing or not. If the operator has selected not to save the sequence data, the sequence will commence as soon as the START button is pushed. If the operator has chosen to save the sequence data, another screen is displayed for naming and saving the data. The system uses standard MS Windows, file naming protocols.

It is convenient now to define an adhesive as a polymeric material which, when applied to surfaces, can join them together and resist separation. A structural adhesive is one used when the load required to cause separation is substantial such that the adhesive provides for the major strength and stiffness of the structure.

5.3 Materials

5.3.1 Adhesive: Adherend

Adhesive: Two types of adhesive were used, an epoxy adhesive and an acrylic adhesive. Epoxy adhesive has a trade mark C-Bond-245, a general purpose adhesive. The epoxy adhesive was a 2-part general-purpose epoxy resin. The 2-part ratio of the epoxy adhesive was 1:1 by volume-Epoxy adhesive (figure 5.11); the second adhesive was a cyanoacrylate RS 514-600 acrylic resin consisting of a liquid and a paste (see Appendix 2). Epoxy adhesive were prepared in accordance with manufacturer instruction, mixing part A (resin) and part B (hardener) in equal volume prior to mixing using a 5ml syringe. The mixture was prepared on a tray and has a tack life of approximately two hours after mixing.
Adherend: The substrate was polycarbonate sheet (table 5.1). It is a transparent material and has a brittle property and is potential to replace glass. Other supporting materials were emery paper and methanol solution to clean the substrate prior to bonding. Polycarbonate sheet, 1.5mm thick was used for making single lap shear bonded samples (101.6mmx25.4mm). The length and width of these lap shear samples followed the ASTM D-3163-73 requirements. The polycarbonate sample was then cleaned with methanol solution, before the experimental irradiation begun, all the sample surface need to be clean and prepare for better surface cohesiveness. Adhesive was applied to the prepared surfaces and the two sheets were manually joined to form a lap joint with an effective bond area of 25.4 mm².

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness(unit: mm)</th>
<th>Length(unit: mm)</th>
<th>Width(unit: mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrylic</td>
<td>1mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>Acrylic</td>
<td>2mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>Acrylic</td>
<td>5mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>Acrylic</td>
<td>10mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>Acrylic</td>
<td>15mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>MICA</td>
<td>2mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>MICA</td>
<td>2mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>MICA</td>
<td>3mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
<tr>
<td>MICA</td>
<td>3mm</td>
<td>101.6mm</td>
<td>25.4mm</td>
</tr>
</tbody>
</table>
5.3.2 Other Experiment of Accessories

Other accessories material are needed for the VFM processing, such as the Telfon fixture-I for the lap joint (figure 5.12) and Telfon Fixture II, which for the welding joint (figure 5.13). They are the same size, one is open pit, and another one has the slot locater for the adhesive and adherend to hold. Two sets of the Telfon slot locator and Telfon base to adjust and fix the location of the samples and made the lap joint with complete contact. During the experimental processing a microwave leakage detector (figure 5.14) was monitoring if any microwave leakage are presented outside of the cavity chamber.

figure 5.12 Telfon Fixture-I (for lap joint)

figure 5.13 Telfon Fixture II (for welding)
Dispensing Syringe (figure 5.15) measured and maintain constant amount of (drops) of adhesive on the glue area. Adhesive need to used (figure 5.16) to inject into Bond area for fixed amount of bonding adhesive onto the bonded surface.

5.3.3 Lap Shear Sample

The joint were prepared in the form of a single lap shear fashion. This is a simple method to characterize the bond strength of the adhesives under investigation and
common method. Its dimensions are in accordance with ASTM D 3163-72 Tensile Strength having 101mm length 25.4mm width 1.5mm thickness and 25.4 sq mm effective bond area. The average glue line thickness was maintained at 0.0127 mm by using a metal pin for spread out the uneven area (figure 5.17).

![A Glue Line Substrate](image)

**figure 5.17 A Single lap shear specimen**

### 5.3.4 Surface Analysis and Testing

A scanning electron microscope (SEM) was used in the interpretation of the morphology of the fractured joint. Samples of the fractured adhesive joint were cut into 10x10 mm square sections and sputter coated with carbon and gold before examination by the SEM at magnifications of between 100 or 1000. Photo-image of the surface image which indicate as different of gray level image for interpretation and photo-analysis. Also polariscope, adherends need to clamp as showed (figure 5.18) by a wooden clamp. Clamp sample was for visual inspection. An Instron tensile testing machine (type 4303) with a crosshead speed of 10mm. min\(^{-1}\) was used to determine the ultimate bond strength of each sample. This model of Instron equipped with 25KW maximum load capacity and run with a pre-programmed input data of the specimen dimension and type.

![figure 5.18 Wooden Sample Clamp](image)
6. Adhesive Curing with Microwave

6.1 Overview

Adhesive have been used for thousands of years to join various materials. Their use for bonding metals in structural (load-bearing) applications, however, did not begin until World War II. Structural adhesives were first used in the aircraft industry, but are now being applied in a variety of industries.

Now, the major users of adhesive bonding by microwave curing are found in automotive, aircraft, PCB manufacturing, and packaging industries. These four groups consume a major portion of the adhesive raw materials sold today. Other industries using adhesives include the shoe, apparel, furniture, bookbinding, electrical, railroad, shipbuilding, and medical industries. The following few specific examples serve to illustrate the importance of the adhesive joining process as an assembly technique. Some of the industrial has studied ready apply microwave technology into their manufacturing process.

This novel processing technique is now being applied to various applications in the electronic packaging industry: Die-Attach, Glob-Top, Underfill, Polyimide Cure and other surface mount applications.

Epoxy adhesives hold a large percentage of the consumer adhesive market. Most of the adhesive consumer epoxy adhesive available in the market. This is because this type of adhesive (epoxy) may be used to bond nearly any substrate and consequently have a wide variety of applications. The adhesives for the consumer market are inexpensive and are used as general purpose or structural adhesives. In industrial applications, epoxy adhesives offer a number of advantages over other fasteners for metal-to-metal bonding such as reduced labour costs. Epoxy adhesives are also used in the automotive industry where they are used in subcomponent (engine and non-engine)
assemblies and metal-to-metal bonding of parts (For example: constructing doors and hoods).

The aerospace and defence sectors use epoxy adhesives in missile assembly, composite repair and bonding of aluminium skins to aircraft frame. The foam form of the adhesive is used in composite panel assembly and repair.

6.1.1 Variable Frequency Microwave Curing

Microwave processing of materials is a relatively new advance technology that provides new alternative approaches for enhancing material properties as well as economic advantages through energy savings and accelerated product development. Factors that hinder the use of microwaves in materials processing are declining, so that prospect for the development of this technology seem to be very promising (Sutton, W.H., 1989). The two mechanisms of orientation polarisation and interfacial space charge polarisation, together with dc conductivity, form the basis of high frequency heating (Siores, 1994). Clearly, advantages in utilising microwave technologies for processing materials include penetrating radiation, controlled electric field distribution and selective and volumetric heating (Metaxas and Meredith, 1983). However, the most commonly used facilities for microwave processing materials are of fixed frequency, e.g. 2.45 GHz or 915 MHz.

Mackay (1979) first conceptualised the idea of variable frequency microwave (VFM) facility and it was not until (Bible, 1992) designed and built the first VFM processing system using a high power travelling wave tube (TWT) amplifier capable of supplying up to 2.5 kW power over the frequency range of 4-8 GHz. The frequency range can be extended by the addition of other TWTs. Microwave-based processing approaches can be broadly divided into either single-mode or multimode cavities. The single mode cavity approach makes use of a tunable microwave capacity specifically designed to support a single resonant mode at the frequency of the microwave source. This
ensures maximum coupling of the microwave energy into the load. However, the single mode nature of the cavity limits the area of high electric fields intensity and, thus, the size, shape and positioning of the material to be processed. The multimode cavity approach makes use of a cavity that is over-mode, which means it is large enough to support a number of high-order modes, often at the same frequency. However, the power distribution at a single frequency is uneven and can result in multiple hot spots (Lauf and Bible, 1993).

The VFM uses a TWT high power, broadband amplifier to sweep a range of frequencies of approximately an octave in bandwidth. The concept behind this approach is that continuous sweeping through several cavity modes within a period of a few microseconds, e.g. 20 µs results in time-averaged uniformity of heating throughout the load. The resulting relative power distribution in a plane with fixed frequency (2.45 GHz) heating is not uniform as depicted in figure 6.1. There is no mode control and hence the coupling efficiency is uncontrolled. On top of it, there is limited scale up and high potential for hot spots and thermal runaway. On the other hand, the resulting relative power distribution in a similar plane with VFM heating is uniform as shown in figure 6.2 (Ku et al, 2000b). There is selective frequency control, high energy coupling efficiency, scaleable to large processing volumes and uniform heating throughout (Lambda Technology, undated). An extra benefit of VFM facility is that components with metal parts can be processed without causing arcing (Ku et al, 2000).

![Relative Power Distribution Pattern for Fixed Frequency Heating](Lambda Technology, undated)

**figure 6.1: Relative Power Distribution Pattern for Fixed Frequency Heating** (Lambda Technology, undated)
Variable frequency microwave (VFM) processing is geared towards advanced materials processing and chemical synthesis. It offers rapid, uniform and selective heating over a large volume at a high energy coupling efficiency. This is accomplished using a preselected bandwidth sweeping around a central frequency employing frequency agile sources such as travelling wave tubes as the microwave power amplifier (Ku et al, 1999; 2000; Siu et al, 1999). Selective heating of complex samples and industrial scale-up are now viable. During VFM processing, a given frequency of microwaves would only be launched for less than one millisecond. Successful applications are in the areas of curing advanced polymeric encapsulants, thermoplastic matrix composite materials characterisation, adhesive characterisation; rapid processing of flip-chip (FC) underfills, joining reinforced thermoplastic matrix composites materials, and structural bonding of glass to plastic housing.

6.1.2 Processing and Bonding of Polymers and Polymer-matrix Composites

Innovations in microwave processing systems having started to emerge, through developments in microwave sources and applications that deliver power to the load, and integrated adaptive control hardware and software that allow the material to conform to the process. Magnetrons and Klystrons are the most commonly used microwaves sources, whilst the most commonly used applicators are multi-mode where numerous modes are excited simultaneously and single-mode where one resonant mode is excited. Such modes are induced using single electromagnetic power sources operating at two of the frequencies designated by Federal Communication Commission, usually 915 MHz.
or 2.45 GHz. In addition to these conventional microwaves technological developments, variable frequency microwaves based on the travelling wave tube principle have been introduced.

As a general way of heat processing, microwave techniques may have limited usefulness. However, in specialized applications such as processing of polymers and polymer-matrix composites the technology has distinct advantages over conventional processing methods which are mainly associated with factors such as timesaving, increased process yield and no environmental heating. Failure to realize expected benefits from microwave processing is often associated with inadequate system integration such as system design, special applicator design, rapid equipment prototyping and scale-up, monitoring and control, and above all a thorough understanding of the specific microwave interactions with materials.

An important element of microwave process development and system design is the capability to model these interactions. Understanding the variations of dielectric properties with temperature and processing state is crucial for simulation and process modelling design. Modelling design can be used to develop applicator systems and optimise process parameters for given dielectric properties, load size and shape. It can also help develop desired processing conditions for hybrid heating applications. Hybrid microwave heating may be provided actively using a separate conventional heating source, or passively using higher dielectric loss susceptor, primers, coupling agents, sacrificial intermediaries, or coatings that more readily absorb the incident microwave power.

6.2 Microwave Technology Applications in the Automotive Industry

Microwave research in the automotive industry has long been engaged in the microwave applications laboratory at the Industrial Research Institute Swinburne (IRIS). It provides an introduction into Microwave technology, which encompasses
state-of-the-art equipment and facilities, the underpinning principles involved in processing materials, and new developments in the field. Research which focuses on breaking down car exhaust gases thus minimising emissions and contributing to a cleaner environment. Emphasis is placed on the considerable advantages that microwave technology offers and the importance that microwave interactions with materials/loads has on designing and integrating effective and efficient microwave engineering systems for automotive industry applications.

6.2.1 Automotive Application

Beginning in 1960, automobile companies began to use heat curing plastisol mastic adhesive to bond the inner and outer panels of hood and trunk compartment lids. This system eliminated the flutter and rattles of hood and trunk lid components over rough roads or at high speeds and reduced the weight of the lid assemblies. The adhesive is oil tolerant and can be dispensed from automated applicators in an assembly line operation. Because the adhesive may be applied in conjunction with spot welding on the same part, it can be applied in a part stamping operation and then heat cured later in the paint ovens. The joining method combining structural adhesives and resistance spot welding, called weld bonding, is discussed later in this section.

Structural adhesive is also used for bonding interior reinforcements to door panels and framing members to the inside of van-chassis vehicles. The automotive industry also uses adhesives for joining metal to fabric for interior trim, vinyl-to-metal exterior trim, and improved performance of tire cords, adhering brake linings, and general fastening. For adhere the textured vinyl coverings to metal roof surfaces, the contact neoprene-phenolic cements have shown excellent resistance to exterior weathering condition. Hot-melt adhesives are used to bond various carpet materials for floor mats and interior kick panels. Other research such as (Bernard and Sabran, 1996; Bernard and Barboteau, 1996) has been used the dielectric heating technology for composite materials of rear doors; and also window encapsulation for Citroen car in automotive industries.
6.3 Microwave Technology Applications in the Aerospace Industries

The aircraft industry started using fibreglass components in the early 1960s but they were only used for tertiary structures such as fairings or non-structural doors. It was in the mid to late 1960s that companies started examining composites for use on structural components and this coincides with the introduction of carbon fibre which probably had some bearing on the policy change. It was realized that the fibre reinforcement would allow designers to tailor the strength of a component in the direction most needed by strategic orientation of the fibres. There was also a realization that this technology had the potential of reducing the weight of components.

Composite materials (Wick, 1987) are now used extensively on Class I (Chemically Reactive Adhesives) structures for fixed wing aircraft and helicopters, and, as can be seen from figure 6.3, a modern helicopter has a high proportion of its structure manufactured from composite materials.

![Polymeric-based composite materials](image)

*figure 6.3 Composites in EH101 (Pocius, A. V., 1997).*
6.3.1 Aerospace Application

One of the lightest and strongest constructions is the laminated or bonded sandwich consisting of thin, high-density facing members adhesively joined to a relatively thick, lightweight core. Both the facing and core materials can vary from metals like aluminum and steel to nonmetals such as wood, paper, and plastics. The basic elements of a bonded sandwich are shown in figure 6.4. Many structural honeycomb panels are made with reticulating adhesive where surface tension causes the adhesive to move up or down the walls of the honeycomb and form a fillet of adhesive. Applications of adhesively bonded sandwich structures are ever increasing in number.

![Elements of a bonded sandwich](image)

figure 6.4 Elements of a bonded sandwich (Charles Wick, 1987).

There is an increasing need for new polymer materials and processes, which are cost-effective and environmentally safe in most industrial applications. In the automotive industry, more and more metallic parts are being replaced by lightweight composites in order to reduce vehicle weight (figure 6.5). There are at least 15 locations in which adhesives and sealants could be used or are being used for automotives body. Particular note should be made of the windshield (8) which is considered a load bearing structure in modern automobiles and is adhesively bonded.
Flange bonding (1) in which adhesives are used to bond and seal. Adhesives are used to bond friction surfaces in brakes and clutches (10). Anti-flutter adhesive bonding (2) helps control deformation of hood and trunk lids under wind shear. Thread-sealing adhesives are used in engine applications (12). Assembly of such parts is achieved using different joining processes. Microwave technology can play a significant role as a competitive alternative to current processing methods and traditional practices.

![Figure 6.5 Application of adhesives in automotive industries (Pocius, A. V., 1997)](image-url)

One of the early industrial applications of microwaves for processing polymers was the treatment of elastomer and in particular vulcanisation of rubber during the late 1960s. The principal application connection between microwaves and rubber occur via carbon black fillers present in the rubber. Carbon black grade concentrations and distribution influence the heating pattern through the multi-layered product. Increased throughput and reduced operating costs helped establish microwave technology as the preferred rubber fabrication method in the 1980s. The process offers advantages including improved product uniformity, reduced extrusion line length, minimum material waste, advanced process control and automation, continuous vulcanisation rather than conventional batch processing, and improved cleanliness and environmental compatibility when compared to steam autoclaves, hot air, or salt bath heat processing techniques.
6.3.2 Why Composite Materials are chosen for Aerospace Industries

Aerospace manufacturers are always trying to improve the performance of their product which must take into account weight, stress, strain and cost. How do polymeric composite materials compare with other materials with regard to this policy?

As has already been stated, probably the greatest advantage of long fibre reinforced composites is the ability to place the strength and/or stiffness in the direction most suited to an individual component. The fibres being used for reinforcement are extremely light in comparison with metallic components – carbon fibre has a density of 1.8 compared with aluminium with a density of 2.71. Also the matrix materials are very light – epoxy resin has density of the order of 1.2. The fibres also have very high strength and stiffness, therefore the specific strength and modulus of the composite is much greater than a metallic one, as the following comparison shows:

<table>
<thead>
<tr>
<th></th>
<th>Specific tensile strength</th>
<th>Specific tensile modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium alloy</td>
<td>0.23</td>
<td>26</td>
</tr>
<tr>
<td>Carbon reinforced composite</td>
<td>1.1</td>
<td>205</td>
</tr>
</tbody>
</table>

This therefore illustrates one of the major advantages of composite materials.

What other advantages are there? A main advantage, for example, helicopter main rotor blades, is the ability to produce complicated shapes. The main rotor blade for an EH101 (figure. 6.3) helicopter has a continuous change of profile along its length and has a twist from one end to the other-this would have been impossible to manufacture as a metallic structure.

Composites have a higher fatigue endurance than metals and the damage tolerance is normally better, plus, under normal conditions, when they are impacted by a sharp object they do not suffer catastrophic failure. Even after being struck by a sharp object,
a composite component will often have sufficient residual strength to complete a task. These properties are extremely important in the aerospace industry.

It is much easier to tailor properties of the composite component, not only by fibre orientation, but also by hybridizing the fibres. Polymeric composites do not corrode but some matrixes do absorb water, which can affect their properties. It has been proved, however, that the extent of the effect on the properties of epoxy laminates can be estimated fairly accurately. Care must be taken when carbon fibre composites are joined to light alloy structures since galvanic cells can be formed and corrosion of the alloy can take place.

A property of carbon fibre which has been used to advantage in the space industry is the low coefficient of expansion of the composite structure. This is very important on structures subjected to large extremes of temperatures such as can be the case on space components. Glass and aramid fibre reinforced composites are often used for radomes because they are electronically transparent.

In summary, these types of composite materials can normally offer a great advantage in respect of weight of the finished component and other properties, but it is very important that the cost of manufacture is controlled in order that they can compete in regard to cost of ownership.

6.3.3 Honeycomb Sandwich Structure

The honeycomb sandwich structure used on a large military transport plane, the Lockheed C-5A, is shown in figure 6.6. It illustrates the strength-weight effectiveness of honeycomb by the large area in this one craft of 24,000 ft$^2$ (more than half an acre), which equals 2230m$^2$. A complete and comprehensive investigation of adhesive bonding in aircraft has been conducted by Douglas Aircraft Co. under an Air Force contract. In this investigation, a full-sized main fuselage of a large transport aircraft
was entirely joined with adhesive and instrumented for every kind of test applicable to the service life of such an aircraft. The study is known today as the PABST program, which stands for primary adhesive bonding structural technology.

6.3.4 Reasons of Adhesive Joint in Aerospace Manufacturing

Advantages of sandwich structures include the following:

- High strength-to-weight ratios.
- High stiffness-to-weight ratios.
- Compound-contoured fabrications.
- High fatigue resistance as a result of the even distribution of stresses.
- Smooth exterior surfaces.

It should be of particular interest to manufacturing engineers to note an example of the ability of structurally bonded metal joints for resisting fatigue-type failures. For
example, the PABST program showed that a deliberately machined crack in the adhesively bonded area did not propagate into catastrophic failure after all the fatigue testing was concluded. In contrast, it is common to find riveted aircraft construction in which catastrophic failures are induced by fatigue stresses radiating out from the drilled rivet hole. For adhesive joint, it has much advantage than rivet joint. When install the metal part on the aircraft structural trance, the ends of the stiffener are subjected to a peel or cleavage stress, where stress starts being transferred from surface to stiffener. Some aircraft manufacturers have in the past ensured that striping is unlikely to start by inserting a rivet at this position. The rivet is in tension, its most effective mode, and thus resists cleavage. Adhesives which are peel resistant are quite capable of performing well in these situations even if, as is generally admitted, they perform best when the stress is applied in the shear mode. The lap-shear joint owes its popularity to its convenience of manufacture and test as well as to the fact that the adhesive is subjected to cleavage as well as shear. Possible drawbacks with respect to the use of sandwich structures include the following:

- Cost is usually higher than for conventional structures.
- Making attachments requires special attention.
- The thin skin provides poor resistance to punctures and dents.

6.4 Package Application: Encapsulate

In printed circuit board assembly (PCBA), dies are attached onto the printed circuit board (PCB) using epoxy. After connecting the die pads to the PCB tracings with gold or aluminium wires, utilising ultrasonic wire bonding machine, the die will be coated with thermally cured epoxy. The chip-on-board (COB) will then be placed in an electrical oven for baking at certain temperature for a certain period of time, depending upon the type of epoxy used until the adhesive is completely cured. The baking, using electrical oven is time-consuming and costly. An alternative way of curing the epoxy is therefore searched and variable frequency microwave (VFM) source is identified as
the possible solution. The first step is curing the epoxies using VFM facilities is to characterise the adhesives. Two VFM facilities are employed to perform the characterisation. One is for the frequency range of 2 – 8 GHz and the other is for the frequency range of 6.5 – 18 GHz. By using these two facilities, the best frequency range to process an epoxy by VFM can be identified and carried out.

Printed circuit board assembly (PCBA) has been widely used in electronic industries. In the production of PCBA, in order to reduce the size of the product and save space, a bare integrated circuit (IC) chip is widely bonded onto a printed circuit board (PCB). Dies are usually placed either manually or automatically on the PCB using thermal and electrical conductive epoxy to fix relative position of the former to the latter (figure 6.7). Aluminium or gold wires are then connected between PCB tracings and die pads using ultrasonic welding wire bonding machine. After function tests, the die will be coated with thermally cured epoxy. This is referred to as encapsulation. In order to decrease the viscosity of the epoxy, the PCB board will be placed on top of a hot plate during the dispensing process of the adhesive (figure 6.8). The amount of epoxy used will depend on the products (PCBs) and the type of the epoxy used. The chip-on-board (COB) will then be placed in an electrical oven for baking at certain temperature for a certain period of time, depending on the epoxy used, to completely cure the epoxy. After curing, the PCBs are bent to a pre-selected curve for bending test to simulate real application conditions. The test is related to the dispensing process of the epoxy. The strength of the adhesive force between the PCB substrate, integrated circuit (IC) surface and the bonded wires are tested. The main purpose is to ensure the epoxy coats all these electronic items without any room for displacement against mechanical shock or impact. If either the curing temperature or time is incorrect, or if the workmanship is poor, air bubbles will be trapped in the PCB and the epoxy will not fill up all these spaces and this will result in weak mechanical interference resistance. If the strength of the epoxy is adequate the bonded wires will not break. The PCBs are then tested for electrical properties.
The curing process is energy and time-consuming. Microwave irradiation is therefore identified as an alternative source to cure the epoxy with less energy and time. As there are considerable amount of metallic items in the PCB, eg, the gold-plated or copper plated mass, the traditional fixed frequency, eg 2.45 GHz or 915 MHz microwave energy source cannot be used, as arcing will result. This paper describes a new technique for microwave processing, known as variable frequency microwave (VFM) processing (Mackay, et al, 1979; Bible et al, 1992; Lauf et al, 1993; Bows, 1999; Ku et al, 1999; 2000; Siu et al, 2000; Siores et al, 2000), which gets rid of the problems brought about by fixed frequency microwave processing.
6.4.1 Packaging Application

In the packaging industry, hot-melt adhesives are used for a large number of applications such as carton (Hasna, 1999; Lye 1999). Encapsulate (Ku and Siu, 2002) and the lamination of films, papers, and foils. The advantages of hot melts for such applications include rapid setup on cooling, which permits the use of high-speed fabrication machinery, and no volatile by products during the joining process, which permits their use on nonporous materials such as plastic film and metal foil. As a result, a number of rapid coating techniques have become feasible for manufacturing. In the heat sealing of plastic bags and containers, the plastic itself acts as the adhesive by sticking to itself in the melted state.

6.4.2 Encapsulate Used in PCB Assembly

There are more than ten types of epoxy adhesives available in the market but only two are considered to be of excellent quality. They are Uniset high temperature die attach adhesive (Uniset adhesive) and Hysol chip-on-board encapsulant (Hysol encapsulant). They are the epoxies used by Approach Holdings Limited and in this research. The Uniset adhesive (Amicon, undated) is a one component heat cured, slightly thixotropic 100% solids, liquid epoxy adhesive and insulation compound. It is black in colour and can be cured at as low as 100°C but the curing temperature used in Approach Holdings Ltd is 120°C and the curing time required is 60 minutes. Its service temperature is up to 232°C. It has good dielectric properties and is very suitable for microwave processing. The Hysol encapsulant (Dexter, undated) is a unique one component epoxy encapsulant with excellent shell stability, flame-out and fast curing capability at moderate temperature. The cured material survives severe thermal shock and offers continuous service to 177°C. It is particularly suited for use on transistors and similar semiconductors. It is black in colour and is cured at 150°C for two hours.
6.4.3 Experimental process of the Characterisation

The first step to use microwave energy to cure the adhesives is to characterise the epoxies using VFMF. This is to find out the best frequency range over which the adhesives will absorb most of the microwave irradiation. The characterisation option of the VFM facilities is used to measure the characteristics of the cavity when a sample (epoxy in this case) is loaded. The procedure followed is a sequence of operations whereby the user graphically sees how the cavity, with material loaded, will operate over the selected frequency range. The input power is selected on the basis of the measured or sometimes estimated loss tangent of the material (Ku et al, 1999). The higher the loss tangent, the lower the power level selected. During characterisation of the loaded cavity, temperature variations were obtained as well as incident power and reflected power levels from the cavity containing the sample via a monitor. The incident and reflected power levels versus frequencies together with the percentage of reflectance against frequencies were monitored and recorded.

The total operation bandwidth for VW1500 is from 6.5 GHz to 18 GHz. For the sake of keeping the temperature low, the characterisation bandwidth was broken into four equal sections. The reason for dividing the bandwidth into four sections was because the forward power, for a given setting, changed with respect to frequency, is the amplified signal changed with respect to frequency. To minimise the error across the total operating bandwidth, the total band was therefore divided into smaller sections of roughly equal power, for a given power setting.

In the characterisation of Uniset adhesive from a frequency range of 6.5 GHz to 18 GHz, the power selected was 50 W and the amount of energy reflected varied from 10W to 35W is 20% to 70% (figure 6.9). The maximum temperature reached was set at 100°C. In the characterisation of Hysol encapsulant, using the same frequency range and the same maximum temperature setting, the input power was 50 W and the reflected energy ranged from 12.5W to 45W is 25% to 90% (figure 6.10). The near zero reflectance...
(figure 6.10) in the frequency range of 16.5-18 GHz was wrong because the incident power was found to be around 10W and this meant that the machine had been automatically switched off as the maximum temperature setting of 100°C had been reached. The Uniset adhesive is loser than Hysol encapsulant in the frequency range selected.

![Characterisation of Uniset Epoxy, 6.5 - 18 GHz](chart1)

**Figure 6.9:** Percentage of Reflectance against Frequency for Uniset Epoxy in the Frequency Range of 6.5-18 GHz

![Characterisation of Hysol Encapsulant, 6.5 - 18 GHz](chart2)

**Figure 6.10:** Percentage of Reflectance against Frequency for Hysol Encapsulant in the Frequency Range of 6.5-18 GHz

Microcure 2100 was used and the frequency range of the machine was from 2-8 GHz. The power level selected for both epoxies was 50W. This was to ensure that the interaction of microwave energy and the sample was not too vigorous and that the facility could provide a complete sweep of frequency from 2.5 GHz to 8 GHz in a certain period of time without making the temperature in the cavity dangerously high. The temperature adjacent to the sample was monitored during the cavity
characterisation process and the machine would be switched off once the temperature was over 100°C, which was not too far from the curing temperature of the adhesives. Figure 6.11 illustrates the percentage of reflectance against frequencies for Uniset adhesive. It was found that the percentage of reflectance was lowest in the frequency range of 6.5 GHz to 8 GHz. The percentage of reflectance ranged from 30% to 50%. Uniset adhesive was therefore best processed in this frequency range for the frequency selected because it absorbed a greater proportion of the incident power. The best frequency ranges to Hysol encapsulant was also found to be from 6.5 GHz to 8 GHz. The percentage of reflectance of the encapsulant was from 30% - 50%.

6.4.4 Experimental Result of the Characterisation

The Encapsulation project has prove that, the values of the percentage of power reflectance of the two epoxies in the frequency range of 2-8 GHz were higher than their counterparts in the frequency range of 6.5-18 GHz. This implies that the dielectric losses of these adhesives are higher at higher frequencies. Within the frequency range of 2.5 GHz to 18 GHz, the lowest reflectance values for Uniset adhesive is 20% and is in the frequency range of 10-12 GHz, whilst that of Hysol encapsulant is 25% and is in the same frequency range. The best frequency range to cure the two epoxies is therefore from 10-12 GHz. By performing materials characterisation, the best range
for microwave processing of a material can be discovered and followed by selective heating and processing using variable frequency microwave sources.

It is, however not possible to directly relate the power reflectance of the microwave cavity to dielectric properties of the epoxy within. The reason for this is that any particular frequency, a number of different modes, each having a different field pattern, will be excited within the cavity. The extent to which each one of these is excited depends on the coupling of the source to each mode, and is not susceptible to measurement. Therefore the electric field pattern within the cavity is extremely complex and unpredictable (Ku et al, 2000). Faced with such a complex situation, the best way to proceed is on a semi-empirical basis. To reduce the time consuming experimental empiricism that was required to develop relatively simple heating procedures, computer simulations of variable frequency heating may be employed (Dibben and Metaxas; 1994; Bows, 1999).

6.5 Summary

Microwave processing of polymer materials for use in the automotive and aerospace industries can provide a powerful technology with which materials can be processed and components manufactured with improved performance characteristics. One of the major advantages of adhesive bonding is that it enables dissimilar materials to be joined, even when one of these is non-metallic. A major application of bonding (Boey, 1999) is therefore where composite materials are concerned. Composites take many forms. In aerospace applications, these materials usually consist of highly aligned layers of carbon or glass fibres, each oriented to accommodate the expected loads. Some aerospace composites are woven or stitched so that the fibres are not perfectly aligned. High-quality chemical plant may be made from satin-weave glass fibre reinforced polyester or epoxy resin, while lower grade composites usually consist of random glass fibres in polyester resin. Thus, composites can be highly anisotropic in respect of both stiffness and strength and, although a unidirectional composite may be very strong and
stiff in the fibre direction, its transverse and shear properties may be very much poorer. Bolts and rivets can sometimes be used with composites, but it is then often necessary to have load-spreading inserts bonded into the structure. Adhesive bonding is attractive since it allows for a more gentle diffusion of the load into the structure, thus reducing the localized stresses encountered in the use of bolts and rivets. In the so-called co-curing technique, the composite is bonded without the use of an adhesive. To do this, the composite is prepared in its uncured (pre-impregnated fibre) form and heat and pressure applied. As the composite cures, the excess matrix material (usually a high-performance laminating epoxy) is squeezed out in a liquid form and contacts the adjacent component. When curing is complete, the bond is made with due saving in production costs. If the parts proof the manufacturability, composite component can be made in one part with minimum shrinkage and hence minimum internal stresses. Quality of the composite component is thus improved (Fuller, 1990).

The trend for the Polymeric materials in the automotives industries is to reduce the body weight of the car. In turn, it gives the automotive manufacturer to meet the strict government requirement for better fuel consumption. Another trendy way is the flutter and rattles of the hood and trunk for weather resistance for the automobile parts. Microwave curing for the window encapsulation in automobile (Bernard and Sabran, 1996) in recent time have proved the microwave technology has successfully applying in the automobile industry.

In the aerospace industry, laminated and bonded sandwich structure has been largely used in the manufacturing of wing and body structure of the aircraft. The microwave process proved to improved product uniformity, minimum material waste, better control and automation of the assembly. Some of the intangible benefit is the improvement of the green manufacturing features, such as the cleanliness and environmental friendly compared with the conventional curing of the honeycomb fabrication.
With the new developed composite, the specific tensile modulus can reached 205 compared with 26 of aluminium alloy. The composite material some can provide up to 1.1 specific tensile strength compared with the 0.23 of aluminium alloy, demonstrated how important of the composite material can offer to the automotive and aerospace industry. With the better and advance manufacturing technology, more economic composite material can made available to the industries for mass production scale. Most important the strength-to-weight ratio and stiffness-to-weight ratio was so vital for any aircraft manufacturer to be competitive in the world market. For the encapsulation process, the curing process is very time consuming (hours of the curing compare with minutes of microwave curing). The microwave irradiation has offered a rapid, selective and volumetric heating or the epoxy adhesive curing methodology.
7. Microwave Adhesive & Welding Joint

Overview

Fabrication and process of materials by microwave technology is a distinct and also new way to process and produce components. Microwaves are electromagnetic waves occupying 300 MHz to 300 GHz frequency spectrum. For most of the industrial microwave processing applications, frequencies range between 915 MHz to 24.1 GHz are ones common used. Microwaves were first used in radar telecommunications systems during the Second World War while the first industrial applications by heating materialised soon after the introduction of the first domestic oven in 1952. For the last two decades, microwave oven has become part of the “white goods” family of appliances and common commodity in modern households. However, despite the long history of development and widespread usage, industrial applications are only now starting to emerge. A major hindrance in this respect has been the limited understandings of the complexity of microwave technology, particular the interactions with materials. Consequently, the identification of microwave technology applications where significant cost, energy and space savings, together with resulting material properties that are unique or enhanced, as compared to conventional heating, have not been fully realised.

During the nineties, considerable efforts have been made by researchers in Australia, to advance the technological frontier and applications of microwave technology. In brief, innovations in microwave processing systems have started to emerge through the developments in microwave sources and applications that deliver power to the load, and integrated adaptive control hardware and software that allow the material to conform to the process. Magnetrons and Klystrons are the most commonly used microwaves sources, whilst the most commonly used applicators are multi-mode where numerous modes are excited simultaneously and single-mode where one resonant mode is excited. Such modes are induced using single electromagnetic power sources operating at a fixed
frequency, usually 915 MHz or 2.45 GHz. In addition to these conventional microwave technological developments, variable frequency microwaves based on the travelling wave tube principle has been very recently introduced. It offers a unique capability in providing uniform and rapid heating over a large volume at high-energy efficiency.

7.1 Adhesive Joining Theory

Adhesive joining is a complex issues involved engineering, chemistry and physics expertise in order to be able to create good adhesive joint and technically sound applications. Since it requests such broad discipline field of knowledge’s, a multidisciplinary approach is need to handle this demanding task.

Most of the engineering requirement is to design the adhesive joints with the minimum stress concentration as much as possible. Hence, the design effort is to make an optimum joint shape for best stress distribution in the joint area; such as:

1. the loading direction,
2. the bonding area,
3. the bond-line thickness,
4. the type of materials or substrates to be joined, and
5. the surface cleanliness of the substrates prior to bonding

After the design stage, it needs to choose the correct adhesive which able not only for the joint stress, but used for the specific condition intended. It is very important for the designer to understand the specifications which the joint expected to serve. The selection of the right adhesive to serve need sound knowledge of chemistry. There are huge types of adhesive, such as Exposited, Acrylic or Phonetic to choose under different conditions. Researcher has attempted to make a computer program available which can decide the type of adhesive to take if service temperature, gap-filling requirements,
sharp and size of the joint, peel and impact forces were inputted. This computer program (Lees, 1993) has proved beneficial in term of quick, interesting and at ease.

7.1.1 Types of Adhesive Joining Common for Engineering Design

Adhesive joints must design to meet the specifications in practice. There is some common adhesive joints design which (figure 7.1) might suitable for different engineering applications

![Common Engineering Adhesive Joints](http://example.com/figure7.1)

**figure 7.1 Common Engineering Adhesive Joints (Adams, 1997)**

7.2 Single Lap Shear Joint

Among those types of adhesive joining, the lap joint is one of the most commonly occurring joints because it is the simplest joint configuration and could handle large loads. However, the stress state of this joint is complex because the load in the single lap is not collinear, thus a bending moment is present and the joint will rotate. Hence, the adhesive layer will no longer be solely in shear, but will have tearing stress at the end of the joint. In contrast, due to its simplicity in the construction, this joint configuration is the most often used for testing adhesive and is still being widely used in the manufacturing industry today to check adhesive quality. Single lap shear joints are
made of two sheets which are joined together with an overlay. Figure 7.2 shows a single lap shear based on the ASTM-D-3163-72 Single Lap Shear Joint for the polymeric adherend.

![figure 7.2 A Single Lap Shear Joint Specimen.](image)

### 7.3 Weld Joint

Tensile specimens are normally cut from welded PMC piece with the line of the weld normal to the loading axis. A satisfactory test will be one in which tensile failure occurs neither in the weld material, nor in the heat-affected zone (HAZ), but rather in the cross section of the bulk material (figure 7.3). In such a case it is commonly stated that the tensile ‘strength’ of the welded joint exceeds that of the bulk PMC pieces.

![figure 7.3 A tensile weld specimen may fail within the weld bead, in the heat-affected zone (HAZ) or in the bulk metal.](image)
However, when a welded joint, which has the same basic geometry as a weld, fails in the region of the weld, it does so in a region of maximum plastic constraint, and it is incorrect to regard the nominal failure stress (the failure load divided by the cross sectional area of the joint) as a measure of the strength of the weld. The measured strength is a function of the thickness of the welded joint and the surface finish of the components, increasing as the thickness of weld decreases, with the surface roughness limiting the minimum thickness of weld.

The thermal history of a welded joint has a large influence on its ductility. An effective quality control test is used to check for loss of ductility. The welded joint is bent about an axis parallel to the weld line on a mandrel of standard radius. If a U-bend can be produced without any sign of visible cracking, as illustrated at figure 7.4, in either the weld bead or the HAZ, then the weld is deemed satisfactory.

7.3.1 Microwave Welding

Microwave welding differs from induction welding in that no conductive materials are involved and it operates with dipole movement rather than induction welding. It works particularly well on plastics having a high dielectric loss factor, such as PVC, ABS and cellulose acetate, but polyethylene and polystyrene do not weld well. The sheets to be joined are pressed together between metallic electrodes connected to a power source for heat generation.
Dielectric heating softens the material between the electrodes, to form a weld on cooling. Typical applications are for manufacture of toys, bags, water proof garments, protective covers and other mass produced items. It is a fast and reliable process but equipment costs are high.

7.3.2 Welding Lap-Joints

The formation of the welded joints is random, the chance to make a welding joint depend on the closeness of the two adherends, the weight object (such as a Telfon block/Telfon rod). The Telfon block is heavier than Telfon rod, if Telfon block applied, which means better contact between the two adherends, the successful yield increase. Other parameters which contribute to the success rate are the temperature, duration and pressure. The higher the temperature, the longer the exposure of the microwave radiation, the better joint can be obtained. If the jointed are formed with the pressure (clamp) apply, the yield of the joints successful rate increases sharply. The success formation of the welding joint is an art; it is not much of the science yet. Sometime it formed the welding joint, sometime it don’t. But if the wooden clamped pressure has applied (figure 5.18), the successful rate is much higher (98% successful rate). The air bubble can be avoid is by lower the temperature and but with longer hours (figure 7.5), which only stayed at 60°C but 24 hours. If the welding joint (figure 7.6) apply at 120°C, the duration is less but the joints will normally have the air bubbles trapped in.

figure 7.5 Weld Joint at 60°C (24hrs)
7.4 Bonding Strength of Lap Joint by VMF

The tensile bond strengths is proportional to the temperature and curing time within the VFMF cavity. The coding of the tensile test pieces are: x-xx-xxx-xxxx

Table 7.1 Coding of the Tensile Strength Testing Samples

<table>
<thead>
<tr>
<th>x-Adhesive type:</th>
<th>(1) Perma bond</th>
<th>(2) 514-600</th>
<th>(3) 159-3991</th>
<th>(4) 159-3957</th>
<th>(5) C-245</th>
</tr>
</thead>
<tbody>
<tr>
<td>xx-material</td>
<td>(A) Acrylic</td>
<td>(P) Polycarbonate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>xxx- Additives</td>
<td>(N) Normal</td>
<td>(B) Carbon black power added</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>xxxx-Duration (s)</td>
<td>30s</td>
<td>60s</td>
<td>90s</td>
<td>120s</td>
<td>150s</td>
</tr>
</tbody>
</table>

Full data sets of the tensile test of the single lap test (stage I and stage II) are presented in Appendix 3; it is not much of the correlation with the tensile stress and elongation. Stage I data is obtained after the curing of the samples; the result has been calibre via the tensile strength testing machinery. For the stage II, it represented the testing result are obtained after six months of the bonding. There are some variations of the two actual sample set outcomes. But one major finding is that, 95% of the tensile strength result has declined, some even drop more than 10% of the same sample set result. Therefore, it has a very strong indication that although, the microwave irradiation provides a rapid, uniform heating and controlled electric field distribution without thermal runaway. But after duration of time, the bonding become weakens by as much
as 10-15%. Is it a natural phenomenon? Or is it only an incident by random, which need to address and empirical studies is needed.

7.4.1 Tensile strength testing

Since there is huge data volume of the tensile strength testing, there is a need to group the sample into groups.

7.4.1.1 Group A: Tensile strength (TS) testing comparison

For stage I:

1P group: There is not much of the difference among the group

![Tensile stress(MPa) STAGE I](image)

*figure 7.7 Tensile strength testing comparison:-polycarbonate- Perma bond adhesive*

2P group: The 2PN45 has a sharp drop of strength from 29.15 to 18.11 MPa.

![Tensile stress(MPa) STAGE I](image)

*figure 7.8 Tensile strength testing comparison:-polycarbonate/514-600 adhesive*
5P group: All curves perform steady has level up till 90°C, except 5PN30 and 5PN35 has decline of TS values.

![Figure 7.9 Tensile strength testing comparison: Polycarbonate/C-245 adhesive](image)

1A group: The value of the group seem to be normal responded

![Figure 7.10 Tensile strength testing comparison: Acrylic/Perma Bond adhesive](image)

2A group: All curves are steady upswing except 2AN45

![Figure 7.11 Tensile strength testing comparison: Acrylic/514-600 adhesive](image)
5A group: It is one of which is abnormal, 5AN30 seem have evaluated the TS of all, but all the curve showed the same pattern.

![Tensile Stress Stage I](image)

**Figure 7.12 Tensile strength testing comparison: Acrylic/C-245 adhesive**

**Stage II**

1P group: All curve performed same pattern.

![Tensile Stress Stage II](image)

**Figure 7.13 Tensile strength testing comparison: Polycarbonate - Perma bond adhesive**

2P group: The 2PN40 has a sharp drop of strength from 150c then start to sharp up climb from 10.54 to 24.59 MPa.
5P group: All curves perform steady has same pattern.

1A group: The value of the group seems to be normal responded with variation within group. 1AN50 sharp fall of TS value from 150°C, from 27.57 MPa to 180 MPa.
2A group: All the variation seem to be variation from within group

![Tensile stress testing comparison](image1)

**Figure 7.17** Tensile strength testing comparison: Acrylic/514-600 adhesive

5A group: Again, it is one of which is abnormal, 5AN30 seem have evaluated the TS of all, but the entire curve showed the same pattern.

![Tensile stress testing comparison](image2)

**Figure 7.18** Tensile strength testing comparison: Acrylic/C-245 adhesive

Summary: The TS value is fluctuation is not strong, only notice pattern is the 5AN group. The 5AN30 out perform other values for stage I and stage II, some of difference as much as 100Mpa.

7.4.1.2 Another group of the summary tensile strength curve

1AN30: the four curves is not much of the variation showed. But one features need to notice is the average tensile strength trend with extremely close match of the behaviour pattern.
1AN35: Very close approximate of the stage I and stage II

1AN40: Stage I and Stage II matches well, but the difference of the TS value is huge, the stage II have drop the TS value substantively.

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**Figure 7.19** Summary Tensile strength test-1AN30

**Figure 7.20** Summary Tensile strength test-1AN35

**Figure 7.21** Summary Tensile strength test-1AN40
1AN45: Stage I and Stage II perform the same. Again, TS values have dropped sharply at the stage II.

![figure 7.22 Summary Tensile strength test-1AN45](image)

1AN50: Stage I and stage II perform similar fashion. Again, TS values have dropped sharply at the stage II (except 150°C)

![figure 7.23 Summary Tensile strength test-1AN50](image)

2AN30: Stage I and Stage II demonstrated the same trending pattern. It has show higher percentage of drop in TS values at stage II.
2AN35: Same pattern of stage I and stage II, this time, it indicated the stage I has dropped TS value instead of Stage II.

2AN40: Close match of stage I and Stage II value, especially the TS value between 90°C to 120°C. But it showed the stage II are weaker in TS values.
2AN45: Close match of TS values of the two stages, and the TS values have same variation among the two.

![Figure 7.27 Summary Tensile strength test-2AN45](image)

2AN50: The stage I and stage II seem have upward movement with random percentage of different of both.

![Figure 7.28 Summary Tensile strength test-2AN50](image)

5AN30: Same pattern for stage I and stage II

![Figure 7.29 Summary Tensile strength test-5AN30](image)
5N35: All the TS values show large variation. And there is a large drop of TS value from stage II compared with stage I.

![Figure 7.30 Summary Tensile strength test-5AN35](image)

5AN40: Same pattern, and the difference of the TS value of stage I and Stage II is confirm that the stage II is weaker by time.

![Figure 7.31 Summary Tensile strength test-5AN40](image)

5AN45: Group lines shows same pattern, and so did the TS value difference.
5AN50: stage I and stage II have a downward trend as the time duration up. One noticeable feature, again, stage II TS value show negative standard.

1PN30: Normal pattern.
1PN35: show normal pattern, but confirm the less strength value of stage II.

![Figure 7.35 Summary Tensile strength test-1PN35](image)

1PN40: All curve converge the same trend. Stage II TS value has indicate negative pattern.

![Figure 7.36 Summary Tensile strength test-1PN40](image)

1PN45: Curves were upward trend, and the TS values are declining at stage II compared with TS value of stage I.
1PN50: Normal of the curve TS values.

2PN30: Almost same value, only TS percentage difference value with negative trend in stage II.
2PN35: Normal set up and trend of all value.

![figure 7.40 Summary Tensile strength test-2PN35](image)

2PN40: Normal showed pattern.

![figure 7.41 Summary Tensile strength test-2PN40](image)

2PN45: Average value has stand at the centre of the two lines, the stage II have a negative value again.

![figure 7.42 Summary Tensile strength test-2PN45](image)
2PN50: The stage I and stage appeared same pattern and the % of different is negative trend.

![Graph of 2PN50](image)

**Figure 7.43 Summary Tensile strength test-2PN50**

5PN30: up and down swing of the stage I and stage II TS trend, but pattern the same.

![Graph of 5PN30](image)

**Figure 7.44 Summary Tensile strength test-5PN30**

5PN35: Seem random but with close approximation. Stage I and stage II TS value and in positive this time.
5PN35: The TS is also close matched and stage I and stage II TS value are in negative value zone.

5PN40: Same value on both stages, so as their difference.

5PN45: Same value on both stages, so as their difference.
5PN50: Same pattern of TS values of both stages.

Summary: The pattern seem very actual, as most of the time, the stage I and stage II of different set up are very much match, which indicate that the microwave radiation have a steady input of energy input for workload. A main message has spell out that stage II have a lower TS values, which mean that the microwave curing will be decline as time elapsed.

7.4.1.3 Grand average of tensile strength-Polycarbonate

Stage I:

1) For the grand total average the curve, polycarbonate, as the duration increase, the TS is increase.
2) For the grand total average of the acrylic curve, the time of irradiation is also indicating positive relationship among these two parameters.

Stage II:

3) For the grand total average of the polycarbonate curve, as the time increase, it shows a downward trend of the TS value.
4) For the grand total average of the acrylic curve, the TS value is decline, increase after the 120°C.

Summary: After the detail studied of the stage I and stage II TS value, it is conclusive that the time duration has some affection factor on the bonding strength. On the long run, the longer of the exposure of the microwave radiation, the stronger bonding strength it can achieve.

7.4.2 SEM Analysis of Lap Joints.

SEM images of the lap fractured joints (figure 7.53) and (figure 7.54) showed that the bonded area is relatively smooth (area A) as compared with that of the welded joint. The fractured area (area B in figure 7.53) illustrates the mirror image configuration of the two halves. The bonded area C in figure 7.54 and area D in figure 7.55 are very even due to exposure in uniform and volumetric heating, a feature of the VFM. The C area has experienced a weak shear force to break the joint and this is indicated by the smooth and even surface of the C-D sides depicted in both figures 7.54 and 7.55. The fractured surface uniformity is shown in both sides with the peak and valley arrangement. The smooth surface and roughness exhibited only on the edge suggests that the uniform heat present cured the adhesive volumetrically by providing heating
from within and towards the adhered surface. No signs of hot spots build up was observed and this provided a better adhesive joint morphology. This finding has been supported by photostress results (Soesatyo, 1999) which claimed that the adhesive has better, more uniform stress distribution in the adhesive joint area when treated with microwaves rather than conventionally heat treated samples.

figure 7.53 SEM of Fracture Lap Joint (5KVx500)

figure 7.54 SEM of Fracture Lap Joint (5KVx500)
7.5 Experimental Results and Discussion - Welded Joints by VMF

Welded joints are very different than adhesively bonded joints. The energy level required in curing is generally much higher than for obtaining lap joints. A set of samples was treated in the VFM cavity in order to obtain butt joints without any adhesive applied onto the surfaces (figure 7.5).

Even at microwave curing temperature of only 120°C, but with long exposure time, without any adhesive, the butt joint depicted in figure 7.6 was achieved. However, the overlapped lap area contained entrapped air (figure 7.56) which suggested inferior quality in the bonded structure.
7.5.1 SEM Analysis and Results

SEM images of the welded joints (figures 7.57 to 7.60) were compared with the lap joints and found quite different in many respects. Figures 7.57 and 7.60 represent the mirror image of the welded joint. The image taken at 5KVx20 magnification depicts the fractured surface profile to be very sharp, which indicates that bonding obtained was very strong. There is no fragmentation showing along the contour line of both mirror images (figure 7.57 and 7.60). The peak and valley (F area in figure 7.58) is very solid, and the fractured surface of the adhesive area of the both sides is very rough (G area in figure 7.59). Such surface morphology indicates that the effective bonding area is well formed and strong; a great deal of shear strength is needed in order to break the joint area of the tensile test specimen. The resultant fragmental structure, indicated in G-area (figure 7.59), demonstrated the strong bond established between the two bonded sides. It is certain that the welding joint (Yussuf, 2005) is yielding superior quality of bonded area accompanied by better bonding strength than the lap joint.

figure 7.57 SEM Image of Welding Joint (5KVx20)
figure 7.58 SEM Image of Welding Joint (5KVx100)

figure 7.59 SEM Image of Welding Joint (5KVx200)

figure 7.60 SEM Image of Welding Joint (5KVx20)
7.5.2 Polariscope

A Polariscope was used to analyze the stress distribution of the adhesive joints. Sample parts were mounted on the wooden clamping jig, and placed in front of polarscope. The one without the wooden clamp had no circle marks (figure 7.14) and it formed a perfect welding joint (figure 7.5). Welding joints with air bubbles entrapped (figure 7.6) inside the HAZ (Heat Affected Zone) were discarded since they were made under intense temperature conditions and at short exposure radiation time intervals. Acceptable results obtained with the wooden clamped jig (figure 5.18) were formed using higher than 60°C temperature input, but only applied over 10-15 hours time intervals (figure 7.15).

7.6 Summary

Both lap and weld joints were tested using the tensile strength testing (Instron Machine 4303), polarscope and Scanning Electron Microscope (SEM). It is conclusive that welding joints showed a much stronger bond than the adhesive joint. As showed by the SEM, the welded joint was characterized by a rough bonded surface, which in turn showed higher bond strength value. It was also confirmed by the two sets of the tensile strength results that the adhesive joint gives rise to a decrease in the bonding strength as time elapsed.
8. Non-destructive Testing Methodology

8.1 Introduction

The basic advantage of variable frequency processing is to enable precise frequency tuning to optimise the coupling efficiency during initial heating. During heating, continuous and/or selective sweep through several cavity modes within a period of a few milliseconds, results in time-averaged uniformity of heating throughout the processing cavity. Multimode microwave furnaces powered with variable frequency sources are better suited for advanced materials applications related to polymerisation, composites processing, bonding and others.

This research presents a novel microwave assisted non-destructive testing (MANDT), a quality evaluation system for industrial applications. Microwave detection have also applied in aerospace industrial (Kharkovsky, 2006) and, crack detection in construction field (Rhim, 2001) (Khanfar, 2003) and (Nadakuduti, et al., 2006) in recent time. This technique provides rapid, on-line, non-intrusive, non-destructive, and volumetric monitoring of microwave curable epoxy test pieces. Material property variables such as physical geometry, chemical and physical properties, internal or surface defects, and dielectric properties can contribute to its unique output characterisation. The established reflected and input signals form a ratio, which can be monitored and plotted as a function of the frequency. The signal ratio versus frequency curve is called the microwave reflective spectrum. The spectrum generated can be used as a signature curve for assessing bond quality during processing. This way, the same material under the same processing environment provides a common characteristic curve, which can be traced. Therefore, the quality of adhesively bonded products can be detected by the intrinsic spectrum signals generated.
8.1.1 Characterisation of the Adhesive

It was also found that the best frequency to process the adhesive was in the frequency range of 9.5-12.5 GHz (Siu et al, 1999), which was also conventionally cured at 65°C isothermally for 40 minutes. Microcure 2100 model 250 VFM facility was therefore employed for the experiment. The central frequency selected was 11 GHz with sweeping bandwidth of 1.1 GHz and sweeping rate of 0.1 second. The power output was 200 W. Polycarbonate samples of 101 mm x 25 mm x 1 mm were joined and the lapped area was 6.5 cm². After applying a calculated amount of the adhesive to the lapped area, the samples were then placed in the centre of the cavity on top of a Teflon block. Microwave reflective spectra were then taken at the following moments during the cure status of the adhesive: A) before cure with sample at 45°C for 6 minutes, B) isothermally cured sample at 45°C for 10 minutes, C) isothermally cured sample at 65°C for 6 minutes, D) after sample isothermally cured for 20 minutes, and E) after sample isothermally cured for 45 minutes. All the spectra were taken in a frequency range from 10.8 GHz to 10.9 GHz as shown in (figure 8.1), with 20% off-set in the y-axis between each spectrum.

![Microwave Reflective Spectra during Isothermal Adhesive Curing at 65°C](image)

In general, the spectra shift to the left during curing and the changes in peak location and shape are directly related to the change of the dielectric properties. Comparing spectrum A to B, the peaks not only shift to the left but also change in shape and...
become flattened. Comparing spectrum B to C, the peaks become more flattened. Spectrum change between C and D is not much. Spectrum change between D and E is even less. In other words, the adhesive almost reached ultimate extent of cure after isothermal curing at 65°C for 6 minutes by VFM irradiation and essentially no further reaction occurred after 20 minutes.

8.2 Principles of the Resonant Mode MANDT

The resonant mode MANDT technique is based on a variable frequency microwave (VFM) concept and interactions between microwaves and materials. When a microwave signal of a given frequency is launched into a cavity which is fully or partially filled with materials, the microwaves will reflect back and forth between cavity walls and travel through the material many times before establishing a final standing wave condition. There are three possible outcomes for the interactions of these waves: i) partially confined and partially reflected, ii) totally reflected back to the launcher, and iii) totally confined within the cavity. The final condition depends on the cavity dimensions, frequency launched and material properties. The ratio between the reflected signal and the input signal can be monitored and plotted as a function of the frequency.

Materials vary in dielectric property parameters over a wide frequency range, from high absorption with high penetration to extreme loss with a very small skin depth penetration (Ahmed et al, 1999). The dielectric properties of as-cured and post-cured samples show changes in high power microwave fields. The polymer chain motion can take place at temperature far below the $T_{m}$, melting point. The polymer chain can vibrate and restrict crankshaft motions, if sufficient thermal energy is available (Pocius, 1998). A general trend appears in that there is an increase in dielectric parameters as the temperature rises. The test becomes reliable once the structure and quality bonding are identified through its impedance obtained from the bonded area. In general, the impedance is higher over the sound joint zone and lower over the disbonded area. One
advantage of this characterisation testing method is the volumetric and rapid monitoring feature for which a complaint or a primer is not necessary. For a given frequency range and cavity dimension, the dimension and geometry of a material, the location of the material within the cavity, and the reflective spectrum are purely a function of the intrinsic properties of that material. Such spectrum generated can be used as a signature curve for assessing the product conformity during the curing processing. In other words, products of conforming quality will share a common characteristic curve, which is different from that of lesser quality (non-conformity) products.

The study focused on the non-destructive testing method for adhesive bonding application of polycarbonate by microwave irradiation. Characterisation studies through utilisation of an appropriate frequency band for microwave processing of polycarbonate were performed. In a VFM environment, the microwave oscillator generated a signal, which was launched into the multimode microwave cavity. A directional coupler identified the strength of the forward and reflected microwave signals. A controller compensated for the gain of the amplifier to ensure a constant forward power level across a range of frequencies, while sweeping occurred. The generated data was transmitted to an off-line ICW, which recorded the information including, frequency forward and reflected powers, and also calculated a percentage of reflected energy to form the microwave reflective spectrum. This signal ratio output at a given frequency was used as an indication of all the interactions with materials inside the cavity.

8.2.1 Technique of the Resonant Mode for NDT

Microwave Assisted Non-destructive Evaluation techniques have been applied in the field of product quality control for materials characterisation and defects diagnosis. A broadband variable frequency based resonant mode methodology is used as an alternative for characterisation of materials within a cavity. The resonant mode is a non-intrusive feature and enables volumetric diagnosis of the material. As other imaging
instrument testing methodology, microwave imaging is also based on the concept of point response. The image is based on the back-calculated dielectric properties of the materials by utilising reflection coefficients or by comparison to the response of the adhesively bonded polymeric materials.

The signal ratio at a given frequency is an interact-resultant between the adherend, the adhesive and microwaves at that frequency. However, the contribution to the final condition of such resultant stage is not equal for every interaction. The signal ratio is mainly determined by the interactions at location of high electric fields. Therefore, dielectric properties at those high field locations can be back calculated from the final conditions of related frequencies. When sufficiently broad, variable frequency microwaves are launched into the cavity, every point inside the mould could be have high fields under certain frequencies as expected. In other word, a microwave reflective spectrum over a proper frequency range, it is possible calculate the distribution of the dielectrically properties inside the mould.

8.2.2 Microwave Interaction with Materials-Curing of Epoxy Adhesive

Microwave processing of polymeric adhesives is based on the interaction of an electromagnetic field with both the adherend and the adhesive. The amount of power absorbed ($P$) by the adhesive greatly influences its curing and resulting bond strength and is dependent on both the permittivity and the loss tangent of the adhesive as follows:

$$P = \frac{1}{2} \varepsilon_0 \varepsilon' \tan \delta \varepsilon E^2 \text{(Watt m}^{-3}) \quad (8.1)$$

Where, $\varepsilon_0$, is the dielectric permittivity, $\varepsilon'$ is the dielectric constant of the adhesive, $\tan \delta$ is the loss tangent, $E$ is the electric field strength.

When travelling from one dielectric material into another, microwaves change direction similar to the way light rays are refracted when passing through different media. The
wave travelling toward the polymer sample is called the incident wave and the wave travelling back to the source is called the reflected wave. The resonant wavelength for a particular mode and cavity shape depends only on cavity size. A rectangular cavity, such as in (figure 8.2) [Decareau & Peterson (1986)] has a theoretical resonant wavelength, $\lambda$, of a particular mode and cavity shape, where $\ell$ is the number of half wave variations of field along the x-axis, $m$ is the number of half wave variations along the y-axis, $n$ is the number of half-wave variations along the z-axis, equation (8.2).

For rectangular cavities, the number of modes $N$ over a band of wavelength, $\Delta \lambda$, $V$ is the cavity volume, and $\lambda_m$ is the wavelength at the centre of the band (equation 8.3).

\[
\lambda = \frac{4}{\sqrt{\left(\frac{\ell}{a}\right)^2 + \left(\frac{m}{b}\right)^2 + \left(\frac{n}{c}\right)^2}} 
\]

\[
N = 8\pi \frac{V}{\lambda_m^2} (\Delta \lambda) 
\]

**figure 8.2 - Rectangular Cavity**

8.2.3 Experimental Result and Discussion-Acrylic

To demonstrate the resonant mode evaluation concept, characterization of acrylic specimens was performed using two variable frequency multimode furnaces. One operating in the frequency interval from 2.5 to 8 GHz with output power adjustable
from 0 to 200W and the other operating in the frequency interval from 6.5 to 18 GHz with output power adjustable from 0 to 150 W. Both systems have the capability to sweep the generated frequency with minimum sweeping time being 0.1 second.

Acrylic samples 126mm in length, 25.4mm in width, and 1.5mm in thickness with 6.45cm$^2$ bond overlap area (lap joints) were exposed to a microwave field at a broadband variable frequency range of 2.5-18GHz. A flexible high strength epoxy adhesive was utilised which conventionally cured at 80°C for 10 mins. The samples were mounted onto a microwave-transparent Teflon Block (127mm diameter and 35mm height) and located at the centre of the multimode chamber. The temperature inside the cavity was monitored by a fibre-optic temperature probe. The initial temperature was set at 45°C allowing the sample to cure as the temperature was raised. A closed-looped temperature control mechanism was used to maintain the sample temperature at the desired temperature range. Once the adherend and the adhesive reached the desired isothermal and exposure conditions, characterisation of the sample took place.

Samples were characterized by the variation of reflectance with microwave frequency in the frequency range from 2.5 to 18 GHz as illustrated in (figure 8.3). Due to the complexity and large data obtained, regression trend lines were drawn. These trend lines showed that the sample was continuously absorbing the microwave energy with curve (A) and (B), which are still undergoing dielectrically absorption stage. Curves (C) (D) and (E) started to cure and the dielectric changes were stable. They also indicate that the samples were continuously absorbing the microwave energy until the maximum absorption zone of about 7.0 to 7.2 GHz was reached. Once the process reached a critical point, the energy absorption started to diminish.
Microwave reflective spectra were taken during the curing process. These spectra are shown in figures 8.4, 8.5, 8.6, 8.7 and 8.8. Curves A, B, C, D and E represent the following:

Curve A - Before curing at 45°C for 6 mins;
Curve B - Isothermally cured sample at 50°C for 6 mins,
Curve C - Isothermally cured sample at 80°C for 6 mins,
Curve D - After sample isothermally cured for 20 mins, and
Curve E - After sample isothermally cured for 30 mins.

As illustrated in (figure 8.4), the 3.25 to 3.35 GHz frequency zone within the bandwidth of 2.5 to 8 GHz was adopted for the study. The curves form a steady pattern in all five states (A), (B), (C), (D) and (E). The spectra were frequency depended, and with some expected minor variation. On the whole, the pattern is very much visible in the entire five situations.
As depicted in (figure 8.5), the 8.65 to 8.75 GHz was selected within the bandwidth of 6.5 to 9.375 GHz. As curve (A) shows, the energy absorption of the sample is low, this being due to the initial impedance of the energy reflected from the sample. The second curve (B) indicates that the modular resistance of the dielectric coupling is lower and thus the material absorbs the energy. Curves (C) and (D) shows that the dielectric parameter of the sample has changed due to the increase in temperature to 80°C. The increase of the higher reflected level of energy means that structural changes have occurred. Curve (E) shows that the adhesive has been cured. One interesting point to note is that curves (B), (C) and (D) show the same pattern, thus indicating that the dielectric properties are at a steady equilibrium state.
As shown in (figure 8.6) from 9.30 to 12.25 GHz frequency region, the 10.80 to 10.90 GHz zone was selected for further examination. Curves (A), (B), (C), (D) and (E) all exhibit the same pattern, but each has shifted to its left as the temperature rise. The indication of higher reflectance level can be in all curves. This is mainly due to the ongoing stabilization of dielectric structure changes. In curve (E), the energy absorption seems to have a sharp slow down at one incident point, this being indicated by the absorption zone at 10.9 GHz.
In (figure 8.7), from the 12.25 to 15.125 GHz frequency region, the 12.75 to 12.85 GHz zone was selected for further examination. Once again, curves (A), (B), (C), (D) and (E) show the same pattern with a higher reflectance ratio over all. Curves (D) and (E) demonstrate that curing of the adhesive have taken place which is indicates by the constant zero reflectance power in the 12.85 GHz zone by the curves.

![Signature Curve (14.50 to 14.60 GHz)](image)

As exhibited in (figure 8.8), from the 15.125 to 18 GHz frequency region, the 16.10 to 16.20 GHz zone was selected for further study. Curves (A) and (B) indicate that there is not only a slow absorption rate but also a zero reflectance from the 16.15 to 16.20 GHz range. Curves (B), (C) and (D), all shift slightly to the left as temperature is increased. Curve (E) confirms that curing of the adhesive with zero absorption of microwave energy has taken place.
From the spectra results, all the samples have higher reflection power due to their early stage of dielectric heating. Once the temperature increased, from 45°C to 50°C, curves (B) and (C) show that there is a shift in higher reflection, but the shape of the curve remains almost the same. This indicates that the dielectric structure and properties have changed. As the temperature remains increased, the dielectric structure changes again, therefore, a new curve shape is obtained. Curve (D) is different from curves (B) and (C) in shape. Once the sample was cured 80°C, the adhesive started not absorbing much microwave energy. This is evident from the curves which illustrates that there are many zero reflections within the frequency range examined.

8.2.4 Experimental Result and Discussion-Polycarbonate

In order to demonstrate the resonant mode evaluation concept again another, characterisation of polycarbonate specimens was performed using two variable frequency multimode furnaces.

Polycarbonate samples of 101mm in length, 25.4mm in width, and 1mm in thickness with 6.45cm² bond overlap area were used for the experimental task. A flexible general-purpose epoxy adhesive was utilized which conventionally cured at 65°C.
isothermally for 40 min. Experimental measurements of the loss factor of the polycarbonate showed that the dielectric constant of the polycarbonate increased slightly between 25°C and 50°C and then steadily decreased until it returned to its original value. Another indication was the loss factor, which decreased slightly between 25°C and 40°C and rose again reaching a peak at 65°C. The dielectric constant of the epoxy adhesive was measured in the range of 500 Hz to 3.5x10^9 Hz. The sample was mounted onto a microwave-transparent Teflon Block (127mm diameter and 35mm height) located at the centre of the multimode chamber. The temperature inside the cavity was monitored by a fibre-optic temperature probe during the process.

The initial temperature was 45°C and thus the sample started to cure as the temperature rose. A closed-looped temperature control mechanism with an on/off microwave power switch was used to maintain the sample temperature at the desired temperature range. Once the adherend and the adhesive reached the desired isothermal conditions and exposure duration, characterisation of the sample was immediately pursued. All spectra were taken in the frequency range from 2.5 GHz to 18 GHz as illustrated in figure 8.9. Due to the complexity and large data point obtained, trend lines were considered since they proved to give a better illustration. Trend lines showed that the sample was continuously absorbing the microwave energy until the maximum absorption zone of about 12 GHz to 12.2 GHz was reached. Once the process reached a critical point, the energy absorption started to diminish.

![figure 8.9 Reflectance Polycarbonate Adhesive Joint 2.5 GHz to 18 GHz Trend Lines (45-65°C)]
Microwave reflective spectra were taken during the curing process. These spectra are demonstrated in figures 8.10, 8.11, 8.12, 8.13 and 8.14. Curves A, B, C, D and E represent the following:

Curve A - Before curing at 45°C for 6 min.,
Curve B - Isothermally cured sample at 45°C for 10 min.,
Curve C - Isothermally cured sample at 65°C for 6 min.,
Curve D - After sample isothermally cured for 20 min., and
Curve E - After sample isothermally cured for 45 min.

As illustrated in figure 8.10, the 3.00 GHz to 3.10 GHz frequency zone within the bandwidth of 2.5 to 8 GHz was adopted for the study. The curves form a steady pattern on all five states (A), (B), (C), (D) and (E). The spectra were frequency depended, and with some expected minor variation. On the whole, the pattern is very much visible in the entire five situations.

As depicted in figure 8.11, the 7.63 GHz to 7.73 GHz was selected within the bandwidth of 6.5 to 9.375 GHz. As curve (A) shows, the energy absorption of the sample is low, this being due to the initial impedance of the energy reflected from the sample. The second Curve (B) indicates that the modular resistance of the dielectric coupling is lower and thus the material absorbs the energy. The temperature rise to 65°C, which is
illustrated in Curve (C), indicates that the energy has changed to the next level. The shape of the curve has also changed and the level of reflected power is increased. The temperature in the cavity reached 65°C for 20 min. Curve (D) shows that the dielectric parameter of the sample has changed due to the increase in temperature. The increase of the higher reflected level of energy means that structural changes have occurred. Curve (E) shows that the adhesive has been cured. The frequency range used indicates that there is zero absorption of the microwave energy in particular within the range of 7.68 to 7.70 GHz interval. One interesting point to note is that Curves (B), (C) and (D) show the same pattern, thus indicating that the dielectric properties are at a steady equilibrium state.

As shown in figure 8.12 from 9.3 GHz to 12.25 GHz frequency region, the 11.07 GHz to 11.17 GHz zone was selected for further examination. Curves (A), (B), (C) and (D) all exhibits the same pattern, but each has shifted to its right as the temperature rise. The same indication can be observed in the reflectance level in Curves (A), (B) and (C). This is mainly due to the dielectric structure changes. In curve (E), the energy absorption seems to be slow down having only one incident point, this being indicated by the zero absorption rates at 11.1 GHz.
In figure 8.13, from the 12.25 GHz to 15.125 GHz frequency region, the 14.49 GHz to 14.59 GHz zone was selected for further examination. Once again Curves (A), (B), (C) and (D) show the same pattern with a higher reflectance ratio over all. Curve (E) demonstrates that curing of the adhesive has taken place which is indicated by the constant zero reflectance power in the curve.

As exhibited in figure 8.14, from the 15.125 GHz to 18 GHz frequency region, the 16.24 GHz to 16.34 GHz zone was selected for further study. Curve (A) indicates that there is not only a slow absorption rate but also a zero reflectance from the 16.26 to 16.30 GHz range. Curves (B), (C) and (D), all shift slightly to the right as temperature is increased. Curve (E) confirms that curing of the adhesive with zero absorption of microwave energy has taken place.
It has been illustrated by the graphical behaviour of the microwave generated reflected curves that initially the sample has higher reflection power due to its early stage of dielectric heating. Once the temperature increased, from 45°C to 65°C, Curves (B) and (C) show that is a shift in higher reflection, but the shape of the curve remains almost the same. This indicates that the dielectric structure and properties have changed. As the temperature remains increased, the dielectric structure changes again, therefore, a new curve shape is obtained. This Curve (D) general is different from Curves (B) and (C) in shape. Once the sample was cured 65°C for 40 minute, the adhesive started not absorbing any more microwave energy. This is another evident from the curve which illustrates that there are many zero reflections within the frequency range examined.

8.3 Summary

In order to test the suitability of an adhesive joint for a particular application, a representative sample joint (adherend and adhesive) was used for evaluation. In pre-production stage, previous experience with adhesives, surface treatments, and joint designs were called on so that a high probability of successful conditions could be obtained. During the production phase, particular with service of critical structures, it is essential to use a non-destructive test to assess the quality and fitness for purpose of the product joining processes. Most of the non-destructive test will have a good
indication of the non-conformity of the product. In this case, if there is a representative sample’s characterization as a base to compare and trace can be generated. Traditional methodological technique is to use the standard representative samples to compare the output of the production. Such evaluation has proved not economical and very time consuming. Cost saving can be achieved as this signature curve data can be input to a computerised device to assist the evaluation process in the automated assembly line. By this way, the same material under the same processing parameters provides a common characteristic curve, which can be used as a tool to provide a rapid, on-line, non-intrusive, non-destructive and volumetric monitoring of adhesively bonded polymer materials.

Microwave irradiation is an offer an alternative means to provide rapid processing times for data feed back in seconds, or multimode variable frequency. The unique feature about microwaves is their deep penetration into materials with substantial reduction in process time, often by as much as 10 to 1 (Siu et al, 1999). Microwave assisted non-destructive testing (MANDT) techniques have been used in many areas under different names, e.g. microwave thermography, microwave imaging technique, and microwave sensors (Barrett and Myers, 1986; Bolomey and Pichot; 1992; Nyfors and Vainikainen, 1989; Wei et al, 1996). All these methods share many common theories, principles and measurement tools. All MANDT systems can be divided into two groups: active and passive systems (Steinberg and Subbaram, 1991). In the active systems, microwaves are shined on the object and property information is extracted from the reflected energy. On the other hand, in the passive systems, microwave energy or noise emitted from the body is used to extract the property information, e.g. temperature. This experimental task uses the lap joint of two samples in a microwave field for Non-Destructive Testing (NDT).

In order to test the suitability of an adhesive joint for a particular application, a representative sample joint (adherend and adhesive) was used for evaluation. Microwave heating is basically volumetric and provides an even temperature
distribution throughout the material. It is based on the interaction of an electromagnetic field with the adhesive where the microwave energy interacts with the bond adherend (polycarbonate) and the adhesive. The adherend, polycarbonate, has a low loss and much of the microwave energy is expected to interact to a great extent with the adhesive (Siu et al, 1999). The result of such microwave energy penetration permits a fast curing and strong joining of the two sheets of polycarbonate.

Microwave assisted non-destructive evaluation technique using a resonant microwave mode is thus presented. Such an evaluation system provides an on-line, volumetric, non-contact, non-intrusive and non-destructive monitoring feature. By comparing a microwave reflective spectrum during the production processes to the standard spectra, a computerised monitored system can be used to regulate the process-input parameters for proper adjustment and compensation. Such methodology can be used for assessing and evaluating product quality. Successful application of this technique depends largely on the database gathered, which can greatly reduce or eliminate products with defects generated during the manufacturing process.
9. Conclusions and Recommendations

Overview

The variable frequency microwave technique has several special features and it is capable of overcoming the non-uniformities in temperature and arcing associated with traditional microwave processing. It utilises the sweeping frequency concept, which is based on parameters including central frequency, frequency bandwidth, and sweep rate. The total bandwidth, typically between 2.5 GHz to 18 GHz is divided into a few thousand points and during operation a computerized controller cycles through these frequency points consecutively, with each cycle corresponding to a chosen sweep rate input, typically less than 0.5 seconds. For each frequency launched into the applicator, there exist standing wave patterns consisting of a number of modes. By cycling through the frequencies into the applicator, it is possible to generate thousands of different frequencies that can be launched with each sweep cycle. The large number of frequencies excited during processing results in a uniform energy distribution throughout the applicator.

Furthermore, the irradiation can lead to damage free processing because the dynamics of charge build up can cause thermal runaway unless sweep rates higher than 0.5 seconds are utilised. The residence time of any given, established wave pattern, when thousands of frequencies are consecutively excited over a period of less than 0.5 seconds, is typically less than 0.2 seconds. During this short interval, a given load is exposed to an alternating electric field operating in the range of 2.5 GHz to 18 GHz. Any current generation due to the high frequency alternating electrical field is random and consists of eddy currents rather than AC or DC. Heating of dielectric loads using microwave sources is substantially accelerated and propagates from “within” the load outwards thus resulting in minimal thermal stress build up.
Moreover, the process can be selective, especially in the case of variable frequency microwave based processing where different frequencies being operated can be triggered by parameters like dielectric loss tangent changes and also by differential absorption that the load undergoes at elevated temperatures. This selectivity of heating characteristic also extends to polymer matrix composites, and multi-layered components and structures.

Microwaves have several characteristics that provide features not available with conventional through surface heating methods. They include features such as penetrating radiation, controllable energy distributions, rapid heating, selective heating, and self-limiting reactions. They allow great flexibility and control for processing materials, and they can lead to improved quality and product properties, as well as reduced processing times, with resultant energy and labour savings. In addition, entirely new types of materials that cannot be produced by alternative methods can be created and synthesized. Microwave furnaces are quiet and safe and provide clean working conditions (which reduce contamination risk) with small floor-space requirements. Commercial opportunities for using microwaves to produce engineered materials are just beginning to be realized.

In addition to heating uniformity, variable frequency applicators eliminate the arcing problems experienced in microwave ovens when a metal or a semi-conducting material is irradiated. Arching is the result of excessive charge build-up in metallic materials in the presence of standing waves patterns. In the variable frequency microwave technique, the electric fields are electronically stirred and the microwave energy is not focused at any given location more than a fraction of a second. The dynamics of charge build-up that lead to arcing are never achieved, hence leading to no arcing and defect-free processing. Furthermore, the ability to select the incident frequencies to tune to a given material, such as the adhesive at the interface joint, makes this generation of microwave applicators suitable for different industrial application such as, surface mounts technology.
9.1 Industrial drive of the PMC composites

Composites have many special features which are great for microwave radiation. Most of the PMC are light weight, yet offer unusual strength savings up to 50% of conventional materials, can be tailored to specific mechanical requirements thus open the horizon for new applications, cost saving in overall manufacturing process and wider materials selection available to manufacturing engineers.

Aviation: fairings, ailerons, spoilers, rudders, stabilizers, blades, cabin interiors.
Automotive: drive shafts, connecting rods, piston pins, motor fastenings, springs.
Space: antenna parts, robot arms, carries for solar cells.
Electronics: a substrate for printed circuit boards.
Leisure time: tennis racket, sailboat, fishing rods, etc.

9.1.1 Improvement Microwave Techniques

Industrial microwave heating of any material has numerous advantages. These include:

1) Direct heating in which the energy used to heat materials is generated within the material not from outside the material. Therefore, heating is more efficient i.e. it is quicker than conventional heating.
2) Savings in space and manpower.
3) Easy operation using instantaneous on and off controls.
4) Easy adaptation to existing processes and may be used in combination with other heating processes.
5) There is no energy loss through radiation and therefore it is more efficient.
6) Better thermal efficiency than other heating processes.
7) Improved product quality.
The advantages particularly important to curing of epoxy adhesives are:

1) Rapid curing
2) Good thermal efficiency
3) No hot spot
4) No thermal runaway
5) Selective heating

9.1.2 The NDT Trend in Production

A microwave non-destructive method for evaluating the quality of the adhesive bonds was used to develop a signature curve for each unique joint. The experimental method presented a novel microwave assisted quality assessment system for industrial applications in the field of product quality compliance, materials characterisation and defect diagnosis. The success of utilizing the Microwave assisted Quality Evaluation technology requires a good understanding of the strengths and limitations of this method. Among the strengths are rapid, on-line, non-intrusive, non-destructive, and a volumetric monitoring of product properties. One must build up and collect spectra data to match the microwave-processing characteristic to the required material properties. Proper design of appropriate process conditions within the resonant chamber is a crucial factor for the accuracy and repeatability of data generated.

It can be said therefore that although microwaves are unlikely to occupy a position in the non-destructive testing field comparable with ultrasonic, radiography or conventional eddy current testing, they nevertheless are capable of providing quality evaluation data related to the bond integrity.

9.2 Finding and Conclusion

The findings in the literature review suggested that there is scope in the evaluation of the VFM effect on the polymers materials, especially after curing, which warrants
further investigations. With the special features of the microwave processing and characterization, it was found through the work in this thesis that both the adhesives and adherents can be processed by optimizing process input parameters. With the same energy input, bonding strength can be increase with longer duration and lower temperature exposure instead higher temperature less exposure time.

Lap and welding joint were obtained with the VFMF technique which offers thermal efficiency, no thermal runaway effect and selective heating features. With other conventional weld joint techniques, specimens are heated from surface inward, and thus curing energy will not be evenly distributed as in the case of VFM curing methodology. Welding technology using microwave assisted joint has proof as a promising new trend for joint development.

The NDT method of bonds evaluation by microwave radiation established that it is possible to carry out defect diagnosis and quality evaluation during the production process. A signature curve can be established as a unique bond feature for quality detection. Such microwave assisted quality system can offer rapid, on-line, non-intrusive, non-destructive and volumetric monitoring of bonded production items.

The VFMF technique developed can be applied to different industrial scenarios such as automotive, aerospace, and encapsulation of the PCB manufacturing. When applying the VFM curing process, the rapid, selective and volumetric heating offers unique advantages, especially in the PCB encapsulation.

9.3 Suggestions for Future Work

1) In order to improve the tightness of the bonding area, a new fixture (figure 9.1) was recommended. The fillet on the top of each slot can ensure that the full lap area is able to lap together.
2) Curing and hardening of plastics and applications is a potential future target for the VFMF technology. The principles of microwave heating can be adapted for these purposes, depending on the required physical shape of the plastics to be cured or hardened. Many of the parameters need to further studied, a).pressure vs. non-pressure; b) temperature variation; c) duration variation in order to gain better understanding of the welding processes.

3) NDT measurements can be made and compared with the standard signature curve and compared with the single samples. Strength of the welded specimen can be measured by investigating the stress standard and curve pattern. Joint efficiency can also be determined by measuring the tensile strength of the joint and the parent metal by means of smart programming and thus a faster response for Non-Destructive Test (NDT) purposes can be obtained.

The above are a few of the potential areas where further research can be done. With a great team at IRIS, microwave and medical system group, there is unlimited potential to explore further usages of microwaves and especially VFMF for the benefit of mankind.
10. Reference


**Ahmed, A.** MacRobert, M. and Siores, E., Characterisation of Alumina Based Ceramics for Microwave Processing and Joining, Melbourne, 1999.


APPENDIX 1: TELFON FIXTURE FOR MICROWAVE IRRADIATION

APPENDIX TWO TELFON FIXTURE

[Diagram of Teflon fixture for microwave irradiation]

Not to Scale

Designed By: [Name]

FIXTURE

RIS
C – BOND 245 is a flexible general purpose epoxy adhesive which can be cured at room temperature. Adhesion to metals, plastics, glass and ceramics is excellent.

The flexibility of C–BOND 245 may be adjusted by the amount of curing agent (Part B) which is used, as shown in the schedule below:

<table>
<thead>
<tr>
<th>Flexibility</th>
<th>Parts A to Parts B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexible</td>
<td>70 : 100</td>
</tr>
<tr>
<td>Semi-rigid</td>
<td>100 : 100</td>
</tr>
<tr>
<td>Rigid</td>
<td>100 : 70</td>
</tr>
</tbody>
</table>

However, for most purposes, the semi-rigid formulation is preferred, and this is the ratio in which the two parts are standard supplied.

C – BOND 245 is ideal in applications where shock, shear and peel resistance are required.

TYPICAL PROPERTIES (for semi-rigid formula, as applicable)

Color: clear
Usage Temperature: -55 to + 120°C (-70 to + 250°F)
Hardness, Shore A: 70
Lap Shear Strength: 20 MPa (300 psi)
Dielectric Strength: 15 kV/mm (400 V/mil)
Volume Resistivity: $3 \times 10^{13}$ Ohm-cm
Dielectric Constant ($10^2$ to $10^9$ Hz): 5.0 to 3.5
Dissipation Factor ($10^2$ to $10^9$ Hz): 0.03 to 0.04
INSTRUCTIONS FOR USE

Clean the surfaces to be bonded prior to application of the adhesive. Alcohol or acetone work well to remove grease, dirt, and fingerprints.

Weigh out the desired amounts of Parts A and B into a suitable container. Mix thoroughly. Pot life is approximately two hours at room temperature.

Apply the adhesive using a brush, roller of squeegee. Mate the surfaces and apply pressure to squeeze out the excess. Ideally, the pieces being bonded should be held together under pressure during cure.

Cure will take place within 8 hours, but bond strength will continue to increase during the following 24 hours. A rapid cure can be made at: 65ºC (150ºF) for 40 minutes or at 95ºC (200ºF) for 15 minutes. It is advisable to postcure a bond at a temperature equal to or above its intended usage temperature. Handling of this product should present no health hazard if ordinary care is exercised to avoid breathing vapors, if skin and eyes are protected, and if material is not ingested. Consult the Material Safety Data Sheet for more information.

ADHESIVE MATERIAL 2 RS 514-600

PRODUCT DESCRIPTION

LOCTITE® Product 406 is a low viscosity, fast curing, single component cyanoacrylate adhesive. It is specifically formulated for difficult to bond substrates.

TYPICAL APPLICATIONS

Rapid bonding of a wide range of metal, plastic or elastomeric materials. Particularly suitable for bonding plastic or rubber (EPDM) parts where very fast fixturing is required.
PROPERTIES OF UNCURED MATERIAL

Typical Value Range

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Type</td>
<td>Ethyl Cyanoacrylate</td>
</tr>
<tr>
<td>Appearance</td>
<td>Clear liquid</td>
</tr>
<tr>
<td>Specific Gravity @ 25°C</td>
<td>1.05</td>
</tr>
<tr>
<td>Viscosity @ 25 MPa, s (cp)</td>
<td>20</td>
</tr>
<tr>
<td>Flash Point (TCC). °C</td>
<td>&gt;80</td>
</tr>
</tbody>
</table>

TYPICAL CURING PERFORMANCE

Under normal conditions, the surface moisture initiates the hardening process. Although functional strength is developed in a relatively short time, curing continues for at least 24 hours before full chemical/solvent resistance is developed.

Cure speed vs. substrate

The rate of cure will depend on substrate used. The table below shows the fixture time achieved on different materials at 22°C, 50% relative humidity. This is defined as the time to develop shear strength of 0.1 N/mm² (14.5 psi) tested on specimens according to ASTM D1002.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Fixture Time, seconds</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel (degreased)</td>
<td>10 to 20</td>
</tr>
<tr>
<td>Aluminium</td>
<td>2 to 10</td>
</tr>
<tr>
<td>Zinc dichromate</td>
<td>30 to 90</td>
</tr>
<tr>
<td>Neoprene</td>
<td>&lt;5</td>
</tr>
<tr>
<td>Nitrile rubber</td>
<td>&lt;5</td>
</tr>
<tr>
<td>ABS</td>
<td>2 to 10</td>
</tr>
<tr>
<td>PVC</td>
<td>2 to 10</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>15 to 50</td>
</tr>
<tr>
<td>Phenolic materials</td>
<td>5 to 15</td>
</tr>
</tbody>
</table>
**Cure speed vs. bond gap**

The rate of cure will depend on the bondline gap. High cure speed is favored by thin bond lines. Increasing the bond gap will slow down the rate of cure.

**Cure speed vs. activator**

Where cure speed is unacceptably long due to large gaps or low relative humidity applying activator to the surface will improve cure speed. However, this can reduce the ultimate strength of the bond; therefore testing is recommended to confirm effect.

**Cure speed vs. humidity**

The rate of cure will depend on the ambient relative humidity. The graph below shows the tensile strength developed with time on Buna N rubber at different levels of humidity.

![Graph showing cure speed vs. humidity](image)

**TYPICAL PROPERTIES OF CURED MATERIAL**

**Physical Properties**
- Coefficient of thermal expansion, ASTM D696, K\(^{-1}\) $80 \times 10^{-6}$
- Coefficient of thermal conductivity, ASTM C177, W.m\(^{-1}\)K\(^{-1}\) 0.1
- Glass transition temperature, ASTM E228, °C

**Electrical Properties**
- Dielectric constant & loss, 25°C, ASTM D150:
Constant Loss

Measured at 100Hz 2.65 <0.02
1kHz 2.75 <0.02
10kHz 2.75 <0.02

volume resistivity, ASTM D257, $\Omega$, cm: $1 \times 10^{16}$
Surface resistivity, ASTM D257, $\Omega$: $1 \times 10^{16}$
Dielectric strength, ASTM D149, kV/mm 25

PERFORMANCE OF CURED MATERIAL
(After 24 hr at 22°C) Typical Value Range

Shear Strength, ASTM D1002, DIN 53283
Grit Blasted Steel, N/mm² (psi) 22 (32000) 18 to 26 (2600 to 3800)
Etched Aluminium, N/mm² (psi) 15 (2200) 11 to 19 (1600 to 2800)
Zinc dichromate, N/mm² (psi) 10 (1450) 6 to 14 (870 to 2000)
ABS, N/mm² (psi) 13 (1900) 6 to 20 (870 to 2900)
PVC, N/mm² (psi) 13 (1900) 6 to 20 (870 to 2900)
Polycarbonate, N/mm² (psi) 12.5 (1810) 5 to 20 (730 to 2900)
Phenolic, N/mm² (psi) 10 (1450) 5 to 15 (730 to 2900)
Neoprene rubber, N/mm² (psi) 10 (1450) 5 to 15 (730 to 2200)
Nitrile rubber, N/mm² (psi) 10 (1450) 5 to 15 (730 to 2200)

Tensile Strength, ASTM D2095, DIN 53283
Grit Blasted Steel, N/mm² (psi) 18.5 (2680) 12 to 25 (1740 to 3630)
Buna N rubber, N/mm² (psi) 10 (1450) 5 to 15 (730 to 2200)

TYPICAL ENVIRONMENTAL RESISTANCE

Test Procedure: Shear Strength ASTM-D1002/DIN 53283
Substrate: Grit blasted mild steel laps
Cure procedure: 1 week at 22°C
**Hot Strength** Tested at temperature.

![Hot Strength Test Graph](image)

**Heat Ageing** Aged at temperature indicated and tested at 22°C

![Heat Ageing Graph](image)

**Chemical / Solvent Resistance**

Aged under conditions indicated and tested at 22°C.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Temp</th>
<th>% Initial</th>
<th>Strength</th>
<th>Retained at</th>
</tr>
</thead>
<tbody>
<tr>
<td>Motor Oil</td>
<td></td>
<td>100</td>
<td>500rh</td>
<td>1000rh</td>
</tr>
<tr>
<td>Leaded Petrol</td>
<td>40°C</td>
<td>100</td>
<td>100</td>
<td>95</td>
</tr>
<tr>
<td>Water/Glycol (50%/50%)</td>
<td>22°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Ethanol</td>
<td>22°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Isopropanol</td>
<td>22°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Freon TA</td>
<td>22°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Humidity 95% RH</td>
<td>22°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Humidity 95% RH</td>
<td>40°C</td>
<td>80</td>
<td>75</td>
<td>65</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>40°C</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
GENERAL INFORMATION

This product is not recommended for use in pure oxygen and/or oxygen rich systems and should not be selected as a sealant for chlorine or other strong oxidizing materials. For safe handling information on this product, consult the Material Safety Data Sheet, (MSDS).

Directions for use

For best performance surfaces should be clean and free of grease. This product performs best in thin bond gaps, (0.05mm). Excess adhesive can be dissolved with clean up solvents, nitro-methane or acetone.

Storage

Products shall be ideally stored in a cool, dry location in unopened containers at a temperature between 8º-21ºC (46º-70ºF) unless otherwise labeled. Optimal storage conditions for unopened containers of cyanoacrylate products are achieved with refrigeration: 2º-8ºC (36º-46ºF). Refrigerated packages shall be allowed to return to room temperature prior to use. The refrigerated shelf-life period for a 500g container is 18 months based upon the date of manufacture. To prevent contamination of unused product, do not return any material to its original container. For further specific shelf-life information on other package sizes, contact your local Technical Service Centre.
APPENDIX 3: COMPLETE TENSILE STRENGTH DATA (TWO STAGES)

STAGE I

1PN30, Tensile Stress and Elongation

1PN35, Tensile Stress and Elongation

1PN40, Tensile Stress and Elongation

1PN45, Tensile Stress and Elongation

1PN50, Tensile Stress and Elongation

1AN30, Tensile Stress and Elongation

1AN35, Tensile Stress and Elongation

1AN40, Tensile Stress and Elongation
5PN50, Tensile Stress and Elongation

5AN30, Tensile Stress and Elongation

5AN35, Tensile Stress and Elongation

5AN40, Tensile Stress and Elongation

5AN45, Tensile Stress and Elongation

5AN50, Tensile Stress and Elongation
<table>
<thead>
<tr>
<th>Time(sec)</th>
<th>Tensile stress(Mpa)</th>
<th>Elongation(%)</th>
<th>Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>17.56</td>
<td>0.642</td>
<td>1PN30, Tensile Stress and Elongation</td>
</tr>
<tr>
<td>60</td>
<td>18.53</td>
<td>0.836</td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>20.35</td>
<td>0.9952</td>
<td></td>
</tr>
<tr>
<td>120</td>
<td>21.62</td>
<td>0.812</td>
<td></td>
</tr>
<tr>
<td>150</td>
<td>20.35</td>
<td>0.762</td>
<td></td>
</tr>
<tr>
<td>180</td>
<td>20.21</td>
<td>0.948</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>13.26</td>
<td>1.210</td>
<td>1PN35, Tensile Stress and Elongation</td>
</tr>
<tr>
<td>60</td>
<td>15.17</td>
<td>0.821</td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>15.18</td>
<td>0.6291</td>
<td></td>
</tr>
<tr>
<td>120</td>
<td>16.12</td>
<td>0.7168</td>
<td></td>
</tr>
<tr>
<td>150</td>
<td>18.21</td>
<td>0.6217</td>
<td></td>
</tr>
<tr>
<td>180</td>
<td>19.26</td>
<td>0.3121</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>12.31</td>
<td>0.5462</td>
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<tr>
<td>60</td>
<td>16.21</td>
<td>0.6226</td>
<td></td>
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<tr>
<td>90</td>
<td>18.60</td>
<td>0.8722</td>
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</tr>
<tr>
<td>120</td>
<td>18.21</td>
<td>0.5913</td>
<td></td>
</tr>
<tr>
<td>150</td>
<td>18.35</td>
<td>0.762</td>
<td></td>
</tr>
<tr>
<td>180</td>
<td>17.20</td>
<td>0.921</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>11.21</td>
<td>2.171</td>
<td>1PN45, Tensile Stress and Elongation</td>
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<tr>
<td>60</td>
<td>13.26</td>
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<td>90</td>
<td>15.27</td>
<td>0.862</td>
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</tr>
<tr>
<td>120</td>
<td>16.21</td>
<td>0.914</td>
<td></td>
</tr>
<tr>
<td>150</td>
<td>18.21</td>
<td>0.817</td>
<td></td>
</tr>
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<td>180</td>
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<td>0.721</td>
<td></td>
</tr>
<tr>
<td>30</td>
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<td>0.9121</td>
<td>1PN50, Tensile Stress and Elongation</td>
</tr>
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<td>17.85</td>
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<td>19.96</td>
<td>0.712</td>
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<td>120</td>
<td>20.15</td>
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<td></td>
</tr>
<tr>
<td>150</td>
<td>18.17</td>
<td>0.629</td>
<td></td>
</tr>
<tr>
<td>Angle</td>
<td>2PN30, Tensile Stress and Elongation</td>
<td>Angle</td>
<td>2AN30, Tensile Stress and Elongation</td>
</tr>
<tr>
<td>-------</td>
<td>-------------------------------------</td>
<td>-------</td>
<td>-------------------------------------</td>
</tr>
<tr>
<td>30</td>
<td>24.15, 1.521</td>
<td>30</td>
<td>15.16, 1.621</td>
</tr>
<tr>
<td>60</td>
<td>15.31, 1.012</td>
<td>60</td>
<td>16.17, 10.12</td>
</tr>
<tr>
<td>90</td>
<td>20.16, 1.510</td>
<td>90</td>
<td>16.21, 11.12</td>
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<td>120</td>
<td>21.17, 1.210</td>
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<td>17.84, 11.26</td>
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<td>150</td>
<td>18.18, 1.600</td>
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<td>18.99, 12.36</td>
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<td>18.62, 10.21</td>
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<td>28.17, 1.421</td>
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<td>26.81, 3.150</td>
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STAGE II

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1PN45, Tensile Stress and Elongation

1PN50, Tensile Stress and Elongation

1AN30, Tensile Stress and Elongation

1AN35, Tensile Stress and Elongation

1AN40, Tensile Stress and Elongation
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5AN30, Tensile Stress and Elongation

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**5PN30, Tensile Stress and Elongation**

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**5AN30, Tensile Stress and Elongation**

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**5PN35, Tensile Stress and Elongation**

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**5AN35, Tensile Stress and Elongation**

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**5PN40, Tensile Stress and Elongation**

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**5PN50, Tensile Stress and Elongation**

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