Characterisation and Optimisation of the Variable Frequency Microwave Technique and its Application to Microfabrication

by

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Declaration

I hereby declare that the work contained in this thesis has not been previously submitted for a degree or diploma to any other university or institution, and to the best of my knowledge and belief, contains no material previously published or written by another person except where due reference is made.

Christian Antonio
June, 2006
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ABSTRACT

The benefits of microwave technology in materials processing is well documented and researched. It offers many potential advantages over conventional processing such as rapid heating, faster processing times and more consistent product quality. However the actual implementation of this technology has been lacking and the benefits have gone largely unrealised. This is due largely in part to the non-uniform heating obtained in multimode cavities in conventional microwave processing. Recently, a new processing method dubbed the Variable Frequency Microwave (VFM) Technique has been developed to overcome the inherent problems associated with conventional microwave processing. By sweeping through a bandwidth of frequencies, the limitations observed in conventional processing, and specifically the problem of heat uniformity, are avoided. With the increase in research activities in alternative processing methods for new and current materials that will provide better product quality as well as time and cost savings, the VFM technique has the potential to rejuvenate interest in microwave processing. This thesis documents the research work undertaken on the VFM technique with emphasis on its characterization, optimisation and implementation to suitable applications in particular in the upcoming area of Microfabrication.

A commercial Variable Frequency Microwave with an operating bandwidth of 2.5-8.0 GHz was investigated through modelling and experimental work to determine the energy distribution within a multimode cavity and to provide an insight of the mechanisms of the method. Modelling was found to be an efficient and cost-effective tool to simulate VFM and to examine the reported advantages of this new technique. Results obtained confirm the superiority of the VFM method over the conventional fixed-frequency processing showing a marked improvement in the heating uniformity achieved. Quantitative analysis of the three major VFM parameters that influence heat uniformity – Sweep Rate, Bandwidth and Central Frequency – indicate that although slight variation in heat uniformity was observed when changing these parameters, these variations are only small which implies that the VFM technique is quite insensitive to changes in the parameters making it quite a robust system. An analytical model of the Variable Frequency
Microwave technique was developed and it was found that the heating uniformity could be further optimised using a sweep rate that varies as the inverse of the frequency squared (weighted-sweep).

In this study, VFM Technique was successfully extended to the Micro-Electro-Mechanical Systems (MEMS) industry as an alternative method for the processing of a polymer system – negative-tone SU8 photoresist – which is gaining widespread use in Microfabrication. The VFM method was compared to conventional hotplate curing as well as a new hybrid curing method introduced in this work and the product quality assessed optically and by thermal analysis. Results from this work indicate that the Variable Frequency Microwave technique is a viable alternative to the conventional cure currently used in practice. With proper optimisation of the VFM parameters, VFM was found to provide samples that are comparable or better than conventionally cured samples in terms of properties and microstructure quality. Using the VFM method, enhancement in cure rates and drying rates, which are described by others as “microwave effects”, were observed and investigated. A significant increase on the degree of cure of up to 20% greater than conventional cure was observed when VFM was utilized and an apparent enhancement in solvent evaporation in the thin SU8 films observed. Experiments undertaken show that microwaves irradiation can enhance diffusion rates of cyclopentanone in the SU8 system by approximately 75-100%. The findings signify that SU8 curing at lower temperatures or rapid curing are possible and long drying times could be reduced significantly thus alleviating many of the problems associated with conventional thermal curing.

Outcomes of this study demonstrate the ability of the new VFM technique to provide uniform heating which is essential for materials processing. Its application to the emerging field of Microfabrication exhibits its unique advantages over conventional curing methods and establishes itself to be a versatile and robust processing tool. The experimental observations made under microwave irradiation are further proof of the existence of specific “microwave effects” which is one of the most debatable topics in the Microwave processing field. A mechanism based on the Cage Model by Zwanzig [1983] was put forward to explain the increase in transport rates.
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CHAPTER 1

INTRODUCTION

1.1 OVERVIEW

This thesis documents a Doctoral Research Program undertaken at the Industrial Research Institute Swinburne (IRIS) which is a research faculty of Swinburne University. The research program was undertaken between the periods of 2001-2004 and was funded by the Australian Research Council (ARC).

The objective of this research program was to investigate a new microwave processing method dubbed the “Variable Frequency Microwave Technique” and to apply this new technology to new applications that would benefit from microwave heating over traditional processing techniques. VFM is a new procedure that overcomes the inherent problems such as non-uniform heating and arcing experienced in traditional microwave processing. Since VFM technology is in its infancy, much research into this processing technique and its applications to potential industrial areas are required and are the impetus of this project.

The aim of this chapter is to present a synopsis of the background to the research project and to outline the research problems and objectives set out to be achieved in this research program. This chapter also provides an outline of the entire thesis.
CHAPTER 1: INTRODUCTION

1.2 BACKGROUND

The development of RADAR during the Second World War stimulated the rapid growth of microwave technology. Even in its early days its potential in heating was recognised. Investigations of industrial applications of microwave heating began in the 1940s and domestic and commercial appliances for heating and cooking foodstuffs began to appear in the 1950s. One of the most successful and widely used microwave applications comes in the form of the Microwave Ovens found in millions of household kitchens worldwide which give a simple and convenient means of thawing, heating or cooking food. From these humble beginnings the application of microwave heating has undergone a rapid growth and expansion to sectors other than food processing. Such growth areas include materials processing where microwave energy is used in sintering, drying and joining of a wide variety of new and engineered materials which includes ceramics, polymers, composites and chemicals. This growth has come about due to the increasing awareness through research of the unique benefits this technique has to offer potential users. Although still in a constant developmental stage, there has been an increase in research and development efforts throughout the last decade. Much of the research is directed at overcoming the challenges of microwave processing through modelling and experiments with the long term goal of developing microwave processing for industrial applications [NRC, 1994].

Microwave processing offers a number of advantages over conventional heating methods. Microwave energy can penetrate a sample thus achieving rapid and volumetric heating as the heat does not have to be conducted into the bulk from the surface unlike conventional heating. Along with the possibility of more controlled heating, it also has the ability to selectively heat target areas which in turn can lead to improved quality and product properties, potentially reducing processing times and energy and increasing labour savings.

Microwave processing employs several set frequencies set out by a governing body to limit its interference with sensitive applications such as mobile telephony and radar systems. Conventional microwave processing uses single frequencies and is often called
“fixed-frequency” microwave processing. Although microwave processing can bring a lot of benefit to materials processing, in the case of using the conventional fixed frequency microwave method, other processing challenges arise from this technique such as difficulty in control, the creation of hotspots which lead to non-uniform heating, arcing and thermal runaways. These have been the main key issues which limits microwave technology uptake in industry.

Recently, a new process called the Variable Frequency Microwave (VFM) technique for material processing has been developed and has alleviated many of the problems inherent in microwave processing such as non-uniform heating and arcing. The technique was developed for the purpose of processing many of today’s advanced materials, with particular applications in the area of polymer adhesives and encapsulants used in electronic packaging [Lambda Technologies, 1998]. VFM is a rapid and controlled approach to uniform heating at the molecular level, hence is volumetric, and is distributed uniformly throughout the work piece by sweeping through a wide frequency spectrum. The technique works by sweeping through a bandwidth of frequencies which are cycled through consecutively and launched into a cavity, resulting in different standing waves with many resonant modes. By sweeping through thousands of frequencies, thousands of possible cavity modes are excited, corresponding to different distributions of hotspots within the cavity. In succession, the heating patterns associated with the different resonance modes begin to overlap thus resulting in a time-averaged uniformity.

The special qualities of VFM technology offer the potential for processing material combinations that could not be simultaneously processed by any other heating technique. Successful applications have already been reported in the curing of advanced polymeric encapsulants and Flip-Chip underfills used in the electronic and semiconductor industries [Fathi et al, 1998], in the production of smartcard products and structural bonding such as optical lens glass to plastics [Lambda Technologies, 1998], joining of fibre reinforced thermoplastics matrix composite materials and materials characterisation. It has also gained attention in many industrial sectors including rubber processing where it has been proposed to improve manufacturability of virgin rubber, and is gaining attention in the automotive industry as an alternative process for car parts such as gaskets. The
application horizon for VFM technology is expanding continuously. However, much research into this new technology still has to be undertaken.

1.3 RESEARCH PROBLEM AND OBJECTIVE

Several techniques are currently employed to reduce the effects of hotspots and improve field uniformity within microwave cavities, many of which depend on the modification of the electric field within the cavity. One such example is found in domestic microwave ovens which are equipped with turntables that rotate during operation. The purpose of which is to reduce the effect of multiple hotspots by passing the food through areas of high and low power thus, achieving a time averaged uniformity. Similarly, mode stirrers are fans which rotate within the cavity to continuously redistribute the electromagnetic field by reflection off the reflective fan blades. However, with the advent of the Variable Frequency Microwave (VFM) technique, these hurdles can now be overcome and has opened new avenues for microwave processing to be applied to new materials. As previously mentioned, since VFM technology is in its infancy, much research into this processing technique and the identification of possible applications are required and are the impetus of this project.

The main aim and objectives of this research work thus include:

- undertaking a study to characterise and study the parameters affecting heating uniformity in the VFM technique using a variety of modelling and experimental methods.

- investigating the feasibility and applying the VFM technique as a rapid and or alternative tool for processing in an industrial application that could harness the potential of this new processing method
It is anticipated that the outcomes of this investigation can lead to a better understanding of the VFM technique in order to understand the underlying mechanisms to further improve and expand its capabilities.

1.4 CONTRIBUTION TO NEW KNOWLEDGE

The work undertaken in this research has made a number of specific contributions to new knowledge in the fields of Microwave Processing and Microwave Applications. These contributions are summarised as follows:

i) Characterisation of the Heat Uniformity in Variable Frequency Microwaves

The energy distribution in a commercial VFM was investigated using a variety of modelling and experimental methods. An analytical model was devised to quantify the energy distribution in a VFM cavity and was compared to a model using a commercial electromagnetic software package (CST Microwave Studio). The models were validated using experimental heat-mapping techniques. The parameters that affect the heat uniformity in the VFM technique were also investigated and quantified using thermal imaging techniques to enable better selection of these parameters.

ii) Optimisation of the Heat Uniformity in Variable Frequency Microwaves

The frequency sweep parameter of the VFM was investigated to improve the heating uniformity in the VFM cavity. A 2D model of the VFM was modelled and two algorithms to produce even heating were compared. A non-linear sweep-rate regime was developed and compared to the current linear sweep. The non-linear sweep is based upon weighing the sweep-rate, the time spent launching a particular frequency, depending on the frequency and was found to improve heat uniformity.
iii) Extension of VFM Technology to MEMS Processing

A potential application for the Variable Frequency Microwave technique was identified to exist in the Micro-Electro-Mechanical Systems (MEMS) industry in processing of photosensitive SU8 resist, a polymer that is gaining extensive use in this industry. A drying and curing regime and the best combination of processing parameters were established for the processing of SU8 using the VFM. The VFM technique was successfully utilised in the processing of SU8 and was found to be a viable alternative processing method.

iv) Discovery of Apparent Rate Enhancement in Photosensitive SU8

During VFM processing of SU8, an apparent increase solvent evaporation was experienced using VFM compared to conventional Hot Plate processing. This effect was further investigated and it was found that there is an apparent rate increase in the evaporation of the solvent from SU8 under a microwave field.

Several outcomes of this project have been reported in various papers and conferences and are listed in Appendix A.

1.5 OUTLINE OF THE THESIS

For ease of organisation, the thesis has been broken up into 11 chapters. This chapter gives a brief introduction into the background and thrust behind the research proposal and the problems that are to be addressed. The basic information about microwave heating, experimental techniques and analysis of results are outlined in the next 10 chapters as follows:

Chapter 2 gives a detailed literature review of microwave processing of materials, the Variable Frequency Microwave Technique as well as microwave processing of polymers. This chapter presents a thorough review of the past and present work in VFM processing.
including modelling and applications as well as on its status as a processing tool. Applications suited to VFM are presented and the criteria for choosing and the choice of the specific area to apply VFM technology are presented and discussed.

Chapter 3 provides an overview of the fundamentals in electromagnetic theory including microwave systems, generation and transmission. A background on the microwave heating concept is also presented and details the mechanisms of microwave-material interaction and in particular presents the mechanisms of microwave coupling with polymers.

Chapter 4 introduces experimental equipment and methodology used. The concept behind the Variable Frequency Microwave technique is outlined and the internal mechanism of the commercial Variable Frequency Microwave, MicroCure 2100 from Lambda Technologies, used in this thesis is discussed in detail. The heat-mapping techniques used to quantify the electromagnetic distribution inside the VFM cavity are also discussed as well as other equipment developments and scientific tests used in this research program.

Chapter 5 presents the results of characterisation of the Variable Frequency Microwave through modelling. The software employed (CST Microwave Studio) is introduced and the VFM model developed and all the assumptions made are discussed. Through this modelling the advantages of VFM in its capacity to heat large areas uniformly are highlighted.

Chapter 6 gives an analysis of the parameters that control the variable frequency microwave and determines their effects on heat uniformity in the VFM cavity. The parameters include frequency, sweep-rate, operating bandwidth and central frequency. Visualisation studies were undertaken using a thermal imaging camera to quantify the effect of altering these parameters.

Chapter 7 introduces a proposal for an optimised “recipe” for the best sweep-rate regime in VFM to further enhance heating uniformity. A 2D model of the VFM was modelled and two algorithms to produce even heating were compared. A non-linear sweep-rate
regime was developed and compared to the current linear sweep. Experimental results are also presented to validate the proposal.

Chapter 8 gives an overview of the new field of Micro-Electro Mechanical Systems (MEMS) and discusses in detail the properties and process requirements of photosensitive SU8 used in this thesis. The development of the processing technique required for SU8 using VFM technology and the choice of suitable parameters and operating conditions are presented. The process developed herein is used in the following chapters for the drying and curing of SU8.

Chapter 9 presents the result of processed SU8 using the VFM technique. This chapter compares the VFM technique to conventional heating and a hybrid heating method for the processing of thin SU8 films. Samples are analysed using thermal and other techniques to study the differences, if any, between the products produced from the different processing methods. Microstructures are fabricated from thin films and the process optimisation discussed.

Chapter 10 presents the results of an apparent enhancement on diffusion in polymeric systems under a microwave field which came about due to results obtained in the previous chapter. A drying experimental schedule to study the diffusion coefficients is discussed and a likely mechanism for this observed phenomenon based on the “Cage Model” by Zwanzig is proposed.

To conclude, a brief summary of the findings and recommendations for future work are discussed in Chapter 11.
CHAPTER 2

LITERATURE REVIEW

2.1 OVERVIEW

A literature review was conducted during the term of the work to acquire an understanding of the context of the research and to provide a basis on which direction the research work would pursue. A thorough understanding of the previous and current work in the area of microwave processing, particularly in the field of Variable Frequency Microwave processing, was required and undertaken to obtain an impetus for this research. This chapter is divided into three sections which include,

- Microwave Processing of Materials
- Variable Frequency Microwave Technique
- Microwave Processing of Polymers

Initially, an overview of the microwave processing field is discussed with particular emphasis on inherent problems of conventional microwave processing. Then a review of the Variable Frequency Microwave Technique, its past and present research areas and applications is presented. Finally, the emerging trend in polymer processing using microwave is reviewed and potential applications are discussed.

2.2 MICROWAVE MATERIALS PROCESSING

Microwaves are defined as electromagnetic waves in the frequency band from 300MHz to 300GHz. Originally developed during the Second World War for use in navigation and radar target detection, the use of microwaves on the industrial and domestic scenes has increased dramatically in the past thirty years. Microwave processing is an attractive alternative to conventional processing methods and provides benefits, which are not
easily obtainable otherwise. It has been used in the processing of rubber, polymers, ceramics, composites, minerals, soils, wastes, chemicals and powders. Nowadays, the use of microwaves extends to many industrial and medical sectors. Industrial microwave processing is usually accomplished at the frequencies set aside for industrial use – 915MHz, 2.45GHz, 5.8GHz and 24.124GHz which are so called ISM (Industrial, Scientific and Medical) frequencies designated by an international committee which constantly review the use of the electromagnetic spectrum.

To date, commercial application of microwaves has been limited. The area where it has been employed includes food processing, the heating and vulcanisation of rubber and in analytical chemistry. Food processing and rubber manufacture involve relatively high-volume, continuous processing. Analytical chemistry applications involve high-volume, repetitive, batch processing, often with long intermediate drying and reaction steps that can be shortened using microwave heating.

The interest in microwave as a processing tool has grown in recent years and is reflected by the number of symposiums that have been dedicated to microwave processing [NRC, 1994]. In spite of microwave processing only being adopted as a processing tool in very few commercial applications, research in the area has been growing due to advances in microwave equipment and understanding of microwave/material interaction. One area where microwave processing is receiving particular attention is in the area of polymer curing/processing because of reported benefits of shorter processing times whilst achieving better quality product and because of reported enhanced kinetics which is apparently due to a poorly understood “microwave effect”. Work has also been undertaken in the use of microwaves for the processing of a wide range of materials which includes ceramics, powders, minerals, ceramic composites and polymer composites. In addition, there has also been a lot of research in microwaves as a plasma process for surface modification, chemical vapour infiltration and powder processing.

In spite of the considerable effort that has been expended in microwave process development, there has been little industrial application to date, with most of the effort still in the laboratory stage. Some of the more significant problems that have inhibited
industrial application of microwave processing include equipment cost, limited applicability, the variation in material dielectric properties with temperature and heating uniformity [NRC, 1994]. The latter two being topics that are of great interest to researchers.

2.2.1 Heating Uniformity in Multimode Cavities

The need to process materials in large quantities has led to applicators that are capable of sustaining a number of high order modes at the same time. Called multimode cavities, the design of these cavities are often based on trial and error, experience and intuition [Thostenson and Chou, 1999] unlike their single-mode counterparts which are designed based on solutions of the electromagnetic field equations. Multimode cavities are typically more versatile than single mode applicators for batch operations and processing of large, complicated shaped objects. As the size of the microwave cavity increases, the number of possible resonant modes also increases. As energy is transferred by the electromagnetic field, non-uniformity in the electromagnetic field will result in non-uniform heating. The presence of different modes results in multiple hotspots within the microwave cavity. Similar to single-mode cavities, local fluctuations in the electromagnetic field result in localised overheating.

Non-uniform heating prevented many applications from taking up microwave technology despite its advantages, as they required multimode cavities. As a result, there has been plenty of research to create greater uniformity within the cavity. Most techniques to enhance uniformity depend on modifying the electromagnetic field within the cavity. One such example is the turntable on a domestic microwave oven which rotates the food through areas of high and low power during operation to obtain uniform heating. Similarly, mechanical techniques such as “mode stirring” are employed by using fan like reflectors inside the cavity to “mix up” the modes by reflecting waves off the irregularly shaped blades thus continuously redistributing the electromagnetic field. Addition of multiple microwave inputs into a cavity has also been found to further enhance field uniformity. Hybrid heating has also been used to achieve better heating uniformity within materials. This is achieved by combining microwave heating with conventional
heat transfer through radiation, convection and conduction. Variations of these have been successfully developed and used for sintering [Thostenson and Chou, 1999].

Recently, collaboration between researchers from the Oak Ridge National Laboratory and Lambda Technologies, Inc. has developed a new system based on variable frequency processing. Dubbed the Variable Frequency Microwave (VFM) technique, this method has been able to overcome the problems of power non-uniformity within multimode cavities as well as other problems inherent in microwave systems by utilising a bandwidth of frequencies instead of a single fixed-frequency as used in conventional microwave processing.

## 2.3 VARIABLE FREQUENCY MICROWAVE TECHNIQUE

The concept of the variable frequency microwave technique is based upon the sweeping of frequencies to obtain a time-average power uniformity within the microwave cavity [Everleigh et al., 1994; Fathi et al., 1996; Fathi et al., 2001; Johnson et al., 1994a; Ku et al., 2000a; Ku et al., 2001a; Lauf et al., 1993; Panchapakesan et al., 1997; Paulauskas et al., 1996; Thostenson and Chou, 1999; Wei et al., 1998]. During processing the parameters are processed, with the bandwidth (typically between 2.5 and 18 GHz) subdivided into thousands of frequencies. These frequencies are cycled through consecutively and launched into the cavity, resulting in different standing waves with multiple modes for each frequency. A simplistic illustration of the technique is shown in Figure 2-1. For a moment in time, the VFM works as a fixed-frequency microwave establishing a standing wave pattern and depositing high energy at different locations within the cavity (Figure 2-1a). By sweeping through thousands of frequencies, thousands of possible modes corresponding to different distribution of hotspots within the cavity are excited. In succession, the heating patterns associated with the different resonance modes begin to overlap (Figure 2-1b). The established standing wave pattern only exists momentarily, and so the heating pattern is constantly changing. Thus, hotspots and thermal runaways are avoided. The contribution of each point in depositing and
heating different parts of the cavity creates a time-averaged heating of the cavity and thus eliminates the non-uniformities in temperature experienced in fixed-frequency microwave heating.

![Figure 2-1: Comparison of the field uniformity in a multimode cavity obtained in a Fixed-Frequency (2-1a) processing to the time-averaged field uniformity obtained by the Variable Frequency Microwave Technique (2-1b) [Lambda Technologies, 2001].](image)

Each sweep cycle takes typically less than 0.5 sec. By sweeping across the complete variable frequency range in tenths of a second, the conditions that would create arcing are avoided therefore leading to damage-free processing. Arcing is the result of excessive charge build-up in metallic materials caused by the presence of standing wave patterns observed in multimode ovens powered with fixed frequency sources [Fathi et al., 1996].

The VFM technology has several adjustable parameters that distinguish it from the conventional single frequency microwave technology: variable centre frequency, bandwidth, and sweep-rate. The central frequency irradiated inside the microwave can be tuned to increase the coupling efficiency with the material to be processed, whilst the combination of bandwidth and sweep-rate can be tuned around the selected central
frequency to redistribute the microwave energy to obtain more uniform heating throughout the work-piece. Finally, the microwave incident power can be pulsed or continuously varied to provide control in real time over the heating profile and minimise reflected power and/or maintain a preset temperature or heating rate of the work-piece, as is the case for fixed frequency microwave oven.

As VFM utilises frequencies outside the assigned Industrial, Scientific and Medical (ISM) frequencies, there is a possibility of interfering with telecommunication frequencies. However, VFM processing is permitted, provided that the equipment is designed to emit less than the legally prescribed limit of Radio Frequency (RF) or Microwave power. This depends on where the VFM device is being operated as each country has different laws regarding operation outside ISM frequencies.

The cost of a variable frequency microwave facility is high compared to fixed frequency microwave facilities or conventional heating. The capital cost of equipment for VMF depends on the power rating, operating frequency, size and applicator design [Ku et al., 2001e]. Commercially available VFM units are presently limited to below 2 kW. However, in spite of these limitations, other factors make microwave processing such as VFM an attractive option. Such factors include process time savings, increased process yield and environmental compatibility [NRC, 1994].

The system makes use of a YIG (Yttrium Iron Garnet) oscillator, a voltage controlled oscillator to generate the required frequency or frequencies to enable sweeping of the microwave field as compared to magnetrons that fixed-frequency microwave systems utilise. A travelling wave tube (TWT), which is capable of giving amplification over a wide frequency band, is then used to amplify the microwave power. Originally developed for electronic warfare applications, the TWT is a linear beam device characterised by a travelling electromagnetic wave that extracts energy longitudinally along the path of an electron beam. The microwave power is then launched into a cavity, which for most systems are multimode cavities. There are several platforms available using the VFM technology, essentially all the components are similar apart from the cavity which
depends on the application. Some commercial Variable Frequency Microwaves are shown in Figure 2-2.

![MicroCure 5100 In-Line System, MicroCure 2100 Batch System and MicroCure 5300 Continuous Flow System](image)

**Figure 2-2:** Commercial Variable Frequency Microwave from Lambda Technologies. From L – R MicroCure 5100 In-Line System, MicroCure 2100 Batch System and MicroCure 5300 Continuous Flow System [Lambda Technologies, 2001].

2.3.1 Modelling of Variable Frequency Microwave

Unlike the more commonly used microwaves (fixed-frequency at 2.45GHz), there have only been a handful of models pertaining to VFM technology. Several techniques have been utilised in the modelling of microwaves including the Finite Difference Time Domain (FDTD). Several algorithms have been developed to simulate variable frequency microwave processing and its parameters. Models developed include qualitative simulations for power distribution using VFM technology to illustrate its ability to create uniformity in small cavities via the sweeping of frequencies through a bandwidth [Lauf et al., 1993; Thostenson and Chou, 1999]. A normalised power density was obtained by summing together the contributions of each excited mode and holding the assumption of equivalent coupling and assuming uniform dwell time spent at each frequency. Using a rectangular cavity of dimensions (30cm x 30cm x 24cm) with a
2GHz bandwidth, their studies indicated a vast improvement in heating uniformity due to a more even power distribution using the VFM technique as compared to the conventional fixed frequency technique. Although the model is an over simplification of the system, the results obtained provide an accurate and illuminating theoretical basis of the variable frequency approach. Similar findings were obtained by Fathi et al. [1995], using Finite Difference Time Domain (FDTD) code where they demonstrated a thermal gradient of 11°C across 20cm x 20cm Polymer Matrix Composite (PMC) plates compared to 90°C using fixed frequency irradiation. An extension of Lauf et al’s [1993] model was performed to study the effect of bandwidth on uniformity [Johnson et al., 1994b]. Results indicate that less bandwidth is required to achieve the same level of heating uniformity as the centre frequency is increased. This finding is significant to large-scale VFM processing because with centre frequency held constant, smaller bandwidth will be required to achieve the same level of uniformity as processing results are scaled up for industrial application i.e. microwave source cost will significantly decrease.

Other models are based upon processes that can take advantage of what the variable frequency technology offers. These include processes such as curing of polymeric materials and sintering. One such model based on the sintering of ceramics, where an FDTD code was coupled with a heat-transfer code to provide a dynamic simulation of this new microwave sintering technique. The coupled FDTD and heat-transfer simulations were run in 100MHz steps to account for the frequency variations in the electromagnetic field in the multimode cavity. The results show that VFM improves the heating uniformity when an optimum frequency range is chosen as compared to single frequency system running at 2.45 GHz [White et al., 1996]. A similar study was undertaken with a polymer matrix with the same results [Panchapakesan et al., 1997]. The FDTD method provides an efficient and cost-effective tool for modelling microwave applications using variable frequencies and for examining the reported advantages of this new technique.

Recently, a 3D finite element simulation of the microwave distribution inside a VFM oven was modelled using ANSYS5.5, a commercial FEM software [Yi et al., 2001]. The
program was used to simulate the microwave field on the surface of a metal-covered substrate in a multi-mode rectangular cavity excited by a source inside a waveguide. The study found that the position of the substrate could affect the resonant frequencies and therefore field distribution depending on the placement of the substrate and could sometimes eliminate resonance. The study also shows that the power density of the VFM oven can be given by the average of the peak powers of the resonant frequencies. Thus, the more resonant frequencies in the microwave oven, the better the field distribution and hence a more even temperature distribution can be achieved.

2.3.2 Application and Research in Variable Frequency Microwave Processing

Most research and development in microwave applications involves fixed-frequency sources i.e. 2.45 GHz due to the limitations of both microwave tubes and the associated microwave components [Bows, 1999]. With the increasing availability of more flexible microwave tubes such as TWTs, commercial availability of variable frequency microwaves is also starting to emerge slowly, thus, it is now possible to investigate potential uses that can benefit from this emerging technology.

The use of variable frequency microwave technology has great potential for industrial applications. The industries that will benefit from VFM technology are where conventional processing does not suffice to obtain the required quality of product. For the majority of these industries, VFM has distinct advantages over conventional processing. These benefits include arc-free processing, selective and rapid heating just to name a few. The technique has already been applied in the processing of advanced materials related to bonding and curing, polymerisation, composite processing, chemical synthesis and non-destructive evaluation. Other uses have been in curing of flip-chips, smartcards, thermoplastic matrix composites, glob-tops etc. The reported advantages in the following applications will be discussed in detail.

2.3.2.1 Plasma

The use of the variable frequency technology to generate and control plasma has been reported as a flexible plasma-processing tool. Several studies have shown that careful
selection of frequencies over a wide range can lead to a uniform distribution of microwave plasma, and can allow precise location and manipulation of the plasma. Thus dramatically improving control of processes such as diamond Chemical Vapour Deposition (CVD), chemical vapour infiltration and plasma etching.

In preliminary studies, researchers used a variable frequency microwave furnace using a high power system driven by a broadband travelling wave tube for the deposition of diamond films by CVD using a 50/50 water-ethanol solution at frequencies in excess of 7 GHz [Rudder et al., 1993]. Results showed that by controlling frequency, the size, shape and location of the water-ethanol plasma ball could be manipulated thus allowing localisation of the plasma discharge in the precise processing area in order to increase process efficiency. Further studies have shown that variation of the processing frequency generated intense, controllable, localised plasmas at pressures of 5-10 torr in virtually every location within a quartz bell jar. It was also shown that large-volume plasmas were possible by increasing the swept-frequency bandwidth about a centre processing frequency [Johnson et al., 1994a]. Thus, results indicate that it should be possible to “scan” microwave-excited plasmas over arbitrarily shaped work-pieces using the VFM technique. Currently, in-roads are being made using the tunable variable frequency technique to process coatings because more uniform plasma etchings are achievable via this method. Diamond films for semiconductors, as well as applying synthetic diamond films on industrial saw blades are being produced.

2.3.2.2 Non-Destructive Evaluation/Material Characterisation

A method using the variable frequency technique to determine the most suitable frequency bandwidth has been reported and is referred to as cavity/material characterisation [Ku et al., 2000b]. The method measures incident and reflected power as a function of frequency for a sample in-situ, thus eliminating the need for prior knowledge of dielectric properties. The frequencies where reflected power is high can therefore be avoided and the most favourable conditions i.e. maximum power absorption, suitable for microwave processing can be chosen. Thus, strong coupling of energy inside the material can be obtained. An example of which is shown in Figure 2-3.
A number of studies have been undertaken to characterise the processing of different materials. Ku et al. [2000b] have characterised a number of different materials including five different thermoplastic matrix composites (TMC) using two different VFM facilities with a combined frequency range of 2-18GHz. The materials tested were 33wt% random carbon fibre reinforced polystyrene, 33wt% random carbon fibre reinforced low-density polyethylene, 33wt% random glass reinforced polystyrene, 33wt% random glass fibre reinforced low-density polyethylene and 33wt% random glass fibre reinforced Nylon 66. The optimum frequency band for microwave processing of the five materials was in the range 8-12GHz. The group also characterised two encapsulants, Uniset adhesive A-312-20 and Hysol encapsulant EEO-1060, used in the electronics industry using the same method and the optimum absorption was found to be between 10-12GHz [Ku et al., 2001d]. Additionally, a two-part liquid araldite characterised in the 2-8GHz was found to absorb best between 6.5-8GHz [Ku et al., 2001a].

This characterisation was also undertaken on a number of thermoplastic substrates - ABS, plexiglass, PVC and Acrylic to determine the optimum processing frequency between 6-
18GHz [Soesatyo et al., 2001]. It was found the best processing bandwidth lies between 9-12GHz for all the substrates except PVC, which was between 10-11GHz. Furthermore, temperature profiles of the various thermoplastics were also measured using the optimum frequency range with the information being gathered useful for optimisation of processes that make use of these plastics.

This system can also be developed as a non-destructive evaluation tool as the spectrum obtained from product processing is purely a function of the nature of the material for a given frequency range, cavity and material dimensions and location within the cavity. This was demonstrated by Fathi et al. [Lambda Technologies, 1998] by using a VFM system to trace material status during microwave curing of Diglycidyl Ether of Bisphenol A (DGEBA)/ Diamindiphenylsupphone (DDS) epoxy samples. In general, the spectra of the epoxy changed at different stages of the curing process and are directly related to the change of the dielectric properties. Thus, the technique can be used for in-situ quality assurance or to check for extent of cure. Once fully developed, the technology can be used for polymers, composites, ceramics, chemicals, pharmaceutical products, foods, and other products pre- or post-processing to determine quality and or extent of processing.

Bows [1999] used the characterisation to examine if the bio-material variability of foodstuff i.e. shape, weight, moisture content etc., had an effect on the characteristics of the VFM system employed and therefore the suitable frequency. By analysing the reflected power vs. frequency of six foodstuffs, it was found that the small variations between the foodstuffs had no significant impact on the profile of the reflected data with frequency. Thus, suggesting that inherent variation in shape, weight and moisture content should not adversely affect the optimum choice of frequencies, and that some degree of temperature-dependent permittivity can be tolerated.

2.3.2.3 Polymer Processing

The largest area of research in VFM processing has been in the processing of polymers. A major barrier to the use of thermosetting composites in many applications is the long cure and post cure processing times.
One of the largest applications for VFM technology is in the bonding and curing of polymeric substances such as adhesives and polymer matrix composites (PMC). Studies have shown the eligibility of this method to cure polymers of different types for different applications with remarkable advantages over conventional processes, whilst bonding has been achieved with a number of material combinations such as plastic-plastic, plastic-glass, glass-ceramic, ceramic-metal, PMC-PMC and polymer-metal combinations.

At any fixed frequency, a specific pattern of electric field is established inside the entire volume of polymer which causes localised over curing and catastrophic thermal runaways during any polymer curing [Paulauskas et al., 1996]. By frequency sweeping, the ability of the technique to cure uniformly has been proven. Several research groups have cured thermoset resin (epoxy/hardener) system using frequency-sweeping characteristic of the VFM as a means of mode stirring to minimise power non-uniformity within the cavity. The approach was found to provide a means by which a uniform and controllable heating and significant reduction on curing times can be achieved. In one study [Fathi et al., 1995], VFM provided a cure time that was eight times faster than conventional non-electromagnetic heating cure cycles. To illustrate the technique of uniform heating, eight samples were scattered within a cavity and were all found to be uniformly cured after processing. Furthermore, to prove the scale-up capability an epoxy sample weighing 530g with dimensions 26.7 x 16.5 x 1.5cm has been successfully cured using variable frequency irradiation. Fixed frequency microwave treatment was not possible for this application as the material being cured was primarily used in microelectronics and contained metal parts, thus would lead to arcing in parts being treated. In another study [Surrett et al., 1994], the same system was studied with addition of some microwave-absorbing additives such as carbon black and various oxides to determine any improvements in microwave coupling. As before, significant time savings were realised using VFM compared to conventional cure, however the addition of microwave absorbing additives had no effect on the microwave curing when added at less than 1-wt%. The degree of cure was indicated by the glass transition temperature ($T_g$) measured with a differential scanning calorimeter at different positions on the sample and was found to range between 161-177°C which compared well with for
conventionally cured samples. This result also indicates a uniform cure. The technique was also investigated as a means of rapid curing of two photosensitive polyimides, polymer dielectrics which are widely used in the microelectronics industry for a variety of applications [Farnsworth et al., 2001]. Results indicate that rapid VFM curing of these polymers is feasible and complete imidization was possible. Curing time was significantly reduced from 240-300 minutes for thermally cured samples down to only 12-30 minutes using VFM depending on the cure set-up. Spectroscopic comparison with thermally cured samples indicates that VFM cured samples have the same extent of imidization and chemical structure and therefore have comparable properties apart from significant difference in the electrical properties due to slow evolution of chemical products. In a similar study on a different polymer dielectric system, Tanikella et al. [2001] demonstrated analogous results, observing comparable or improved properties for VFM cured films compared to thermally cured films and processing improvements such as shorter cure times and lower processing temperature.

Successful applications have also been reported in the curing of:

- Advanced polymeric encapsulants and Flip-Chip underfills used in the electronic and semiconductor industries [Anderson et al., 1998; Fathi et al., 1998]
- In the production of smartcard products [Clayton et al., 1998] and structural bonding such as optical lens glass to plastics [Clayton et al., 1998]
- Joining of fibre reinforced thermoplastics matrix composite materials [Ku et al., 2000b; Ku et al., 2001b] and polymer matrix composites (PMC) [Adegbite et al., 1995; Demeuse and Johnson, 1994; Fathi et al.; Fathi et al., 1995].

A number of bonding applications have been investigated using this new technology, some of which have been implemented on an industrial scale. A growth area in VFM application for bonding is in the electronics and semiconductor industry, where a continuing trend to maximise the functionality-to-size ratio of devices through the development of smaller assemblies that have faster device speeds, higher heat dissipation, higher reliability and lower cost [Wei et al., 1998]. This is achieved through a process called Direct Chip Attachment (DCA) or Flip-Chip technology. DCA or Flip-Chip is a
process where bare silicon dies are attached directly to a Printed Circuit Board (PCB) as opposed to packaging the device and then mounting it on the PCB. Flip-Chip technology requires the use of high quality advanced polymeric adhesives and encapsulants to attach and protect these devices. The process has been found to significantly reduce the curing times of the bonding adhesive or encapsulant, whilst minimising stress by lowering thermal gradients due to the nature of selective heating obtained with microwave energy. In addition, other investigations have discovered that materials such as flip-chips processed by VFM technology have improved product quality and uniformity compared with processing by conventional techniques. Moreover, this is achieved without compromising other important properties such as the glass transition temperature ($T_g$) and adhesion strength.

2.3.2.4 Future Applications

The application horizon for VFM technology is expanding continuously into different sectors of the manufacturing community as the VFM technology advances. The research community is leading this technology expansion/transfer into different sectors. Although an array of materials has been processed using variable frequency irradiation, much of the emphasis was on epoxies and polymer matrix composites [Fathi et al., 1995]. Future areas of interest include drying, binder burnout, ceramic and ceramic composite as well as heating and sintering of Alumina, SiC and Si$_3$N$_4$ [Garard et al., 1995]. It has also been proposed to improve manufacturability of virgin rubber, and is gaining attention in the automotive industry as an alternative process for parts such as gaskets and other lightweight composites that will eventually replace metallic parts to reduce the weight of the vehicle to reduce fuel consumption. Other potential bonding applications include automotive light lens to body assemblies, SMC component bonding, speaker assembly, fabric bonding and EMI/RF Gasketing [Porter, 2002]. Other applications of VFM technology being explored include its usage in the curing of polymeric mouldings in rapid prototyping technology, and the curing of thin coatings in sol-gel and other applications [Osiyemi, 2002].
One possible area of application of VFM is in the area of Micro-Electro-Mechanical Systems (MEMS) and will be discussed in section 2.4.1.

2.4 MICROWAVE PROCESSING OF POLYMERS

There are increasing demands across broad product lines for new polymeric materials and processes that are cost-effective and environmentally safe [NRC, 1994]. As polymers and their composites are usually thermally cured, and since most have low thermal conductivity, many of the technical challenges associated with conventional processing also exist for polymers. For this reason, there has been much research in the area of microwave processing of polymers. A major barrier in the use of thermosetting polymers in many applications is the long cure and post-cure times required to obtain the required mechanical and physical properties.

In traditional heating, the cycle time is often dominated by slow heating rates that are chosen to minimise steep thermal gradients that result in process-induced stresses. For materials such as polymers which have low thermal conductivities, microwave processing can result in significantly reduced processing times.

One of the first industrial applications of microwaves in polymeric processing is in the vulcanisation of rubber in the tire industry in the 1960s. The principal mechanism of coupling of microwave to the material occurred via carbon black fillers which were already present in many rubber formulations. However, the processing technology was limited due to non-uniformity of the microwave curing ovens available at the time and thermal runaway attributable to increases in dielectric loss with increasing temperature.

Since then, with the increased understanding of microwave processing due to greater research in the area and with the advent of new technologies and equipment, such as the Variable Frequency Microwave, the interest with microwave as a processing tool has resurfaced. Currently, there is significant interest in applying this technology to the processing of high performance, high cost-materials and to extend its application to other industries.
Early literature on the microwave processing of polymers and composites reported a drastic reduction in the required cure time [Thostenson and Chou, 1999]. This reduction in cure time which translates to acceleration in cure kinetics is the most commonly reported “Microwave Effect”. Since the early experimental investigations of microwave curing of polymers, there have been many attempts to understand the real effect of microwaves, if any, on the chemical kinetics and physical properties of microwave cured polymers. Hence, the existence of the microwave effect in polymer curing is quite controversial and there have been a lot of conflicting results on this topic. There have been numerous studies that show a clear enhancement in cure kinetics of polymers under microwave radiation over conventional processing for experiments undertaken at similar conditions [Mijovic and Wijaya, 1990; Lewis et al., 1992; Mallon and Ray, 1997; Nakai et al., 2002; Petchuay et al., 2003; Katakura et al., 2003]. However, for each of these studies, there have been similar amount of studies which refutes an enhancement of cure kinetics reporting similar kinetics for both microwave and conventional heating [Jordan et al., 1992; Jullien and Petit, 1992; Mijovic et al.; 1992] at similar conditions. There has been a difficulty in the comparison and rationalization of these effects as experimental conditions and the materials studied have been different from group to group. Despite this, it has been shown that microwaves can be of benefit to the processing of polymers although the exact reason and mechanism for the observed effects are still uncertain.

Polymer processing is a very broad area where VFM technology could be applied. There is a current trend in industry to decrease cycle time in processes to save on cost and to investigate alternative processing methods that can achieve this. From this point of view, it is an opening that VFM can exploit and where it can compete with traditional processing methods.

2.4.1 Micromachining/MEMS – A Potential Application of Microwaves

Micro-Electro-Mechanical Systems (MEMS) is the integration of mechanical elements, sensors, actuators, and electronics on a common silicon substrate through microfabrication technology. MEMS promises to revolutionize nearly every product category by bringing together silicon-based microelectronics circuitry with microsensors
and microactuators via micromachining technology, making possible the realization of complete systems-on-a-chip. Since MEMS devices are manufactured using batch fabrication techniques similar to those used for integrated circuits, unprecedented levels of functionality, reliability, and sophistication can be placed on a small silicon chip at a relatively low cost.

MEMS technology is based on a number of tools and methodologies, which are used to form small structures with dimensions in the micrometer scale (one millionth of a meter). MEMS are smart devices that can integrate with Complementary Metal Oxide Semiconductor (CMOS) logic to not only sense but also analyse therefore can interact intelligently with the physical and chemical world. This emerging field has seen the development of applications ranging from the micro-tweezers, cantilevers, protein analysis and cell manipulation by electrophoresis, intelligent drug delivery, RF devices and the modern inkjet nozzle in printers, some of which are seen in Figure 2-4.

![Image of MEMS devices](image-url)

**Figure 2-4:** Sample of MEMS devices – From L-R Micro Tweezers, Low-Temperature Mechanically Released Cantilevers, Ink Jet Nozzle with Square Counterbore [DALSA Semiconductor, 2004; JPSA Laser, 2004].

Furthermore, it brings new capabilities to the automotive, biomedical, RF, photonics and information technology as indicated in Table 2-1.
CHAPTER 2: LITERATURE REVIEW

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Table 2-1: Application of MEMS products in different industries.

While the electronics on MEMS products are fabricated using integrated circuit (IC) process sequences (e.g., CMOS, Bipolar, or BICMOS processes), the micromechanical components are fabricated using compatible "micromachining" processes that selectively etch away parts of the silicon wafer or add new structural layers to form the mechanical and electromechanical devices. Similar to IC’s, almost all devices are built on silicon wafers and the structures are realized in thin films which are patterned using photolithographic methods.

There are three basic building blocks in MEMS technology – 1) the ability to deposit thin films of material on a substrate, 2) application of a patterned mask on top of the films by photolithographic imaging, 3) etching the films selectively to the mask. A MEMS manufacturing process is usually a structured sequence of these operations to form actual devices. For the purpose of this thesis, it is the second step in the process which is of interest.
Photolithography in the MEMS context is typically the transfer of a pattern to a photosensitive material by selective exposure to a radiation source such as light. A photosensitive material is a material that experiences a change in its physical properties when exposed to a radiation source. If we selectively expose a photosensitive material to radiation (e.g. by masking some of the radiation) the pattern of the radiation on the material is transferred to the material exposed, as the properties of the exposed and unexposed regions differ (as shown in Figure 2-5).

![Figure 2-5: Method of patterning using photosensitive materials in MEMS production.](image)

In photolithography for micromachining, the photosensitive material used to form a patterned coating on a surface is typically a photoresist. Photoresists are light sensitive, organic polymers which become soluble when exposed to ultraviolet light. It contains a light-sensitive substance whose properties allow image transfer onto a PCB board or silicon substrate. Photoepoxies have a wide range of applications within various industries such as semiconductor, biomedical engineering, holographic, electronics, and nanofabrication. Specifically, they are used in the following application as shown on Table 2-2.
Typically after the photoresist has been spun onto a substrate and after exposure to UV light, it goes through some sort of heat treatment to remove excess solvent and to crosslink the photoresist for development. Heat treatment usually is done in conventional ovens or on hotplates. Photoresists are classified into two groups, positive resists, in which the exposed areas become more sensitive to chemical etching and are removed in the developing process, and negative resists, in which the exposed areas become resistant to chemical etching, so the unexposed areas are removed during the developing process.

There are several formulation of photoepoxies in the market however, one that is gaining a lot of attention and is gaining much use in the MEMS field is a formulation called SU8.

### 2.4.2 SU8 – Negative Tone Photoresists

SU8 is a negative tone photoresist which was first formulated at IBM as early as 1982 [Ito and Willson, 1982]. Its first application in thick film photolithography was reported in 1995 [LaBianca and Gelorme, 1995]. SU8 has been used and investigated for various high aspect ratio patterning purposes such as masking for deep reactive-ion etching (RIE), electroplating moulds, injection moulding masters, microfluidic components and structural parts for micro motors. Its main lithographic application has been in deep optical lithography and x-ray exposures to produce high aspect ratio structures for MEMS and MicroOptoElectroMechanical Systems (MOEMS) applications where the resist is
SU8 is a three compound epoxy based, negative, near-UV photoresist based on EPON SU8 epoxy resin. The SU8 monomer/prepolymer provides a very high group functionality and has a low molecular weight of approximately 7000g/mole. Each SU8 monomer has 8 epoxy functional side groups which provide a very dense three-dimensional network of crosslinks when the resin is cured, as shown in Figure 2-6. The structuring of SU8 is accomplished with UV illumination of the photoresist through a mask which activates a photo acid generator (PAG) that facilitates crosslinking. Cured SU8 forms a highly crosslinked matrix of covalent bonds which results in glass like mechanical properties. Other advantages include, relatively low price, the feasibility of batch processing, high chemical resistance and good optical transparency. The material properties and the outstanding structural anisotropy in the fabrication of micro structures open up a promising field of applications. A more in-depth study of SU8, its properties and processing method can be found in Chapter 8.
2.4.2.1 Traditional Heating and Curing Methods

In recent years, requirements for thick photoresists layers have gained increasing importance in UV lithography. This increase has led to increasingly challenging processing requirements and make control of lithographic processes exceedingly challenging. Photoresists such as SU8 has been used for this.

One of the decisive factors determining the patterning result is the prebake of the thick resists. The prebake or the softbake is where much of the solvent has to be evaporated from the coating. This step traditionally requires long bake times and/or high temperatures. These conditions can cause degradation of the photoactive compound as a consequence hence affecting the function of the resists layer.

Traditionally, two baking methods are commonly used to process SU8 layers – Oven and on a hotplate. In an oven, the resist is uniformly heated by convection from all sides. Thus, skin formation of the resists surface is often observed, which reduces solvent evaporation. This phenomenon can be avoided by employing a hotplate where the resist is heated from below by heat conduction and a temperature gradient develops in the resist layer, which has been found to have a favourable effect in thinner resist layers. For thicker layers, a uniform bake of the layer is not possible thus, poor pattern resolution, formation of micro-cracks and severe outgassing occurs as a consequence. Depending on the layer thickness, prebaking of samples to obtain the desired solvent content can take from minutes to several hours via the conventional methods.

The long prebaking and curing times together with the mismatch in coefficient of thermal expansion (CTE) between SU8 and its substrate, and the characteristically inherent rigid molecular structure of the molecule combine to generate considerable internal stress which causes cracked lithographic features [Ruhmann et al., 2001]. Thus alternative processing methods for SU8 has been investigated to speed up the process and to avoid abnormalities in the structures such as cracks. These include non-conventional curing such as infrared (IR) baking and hybrid curing methods using a combination of hotplate and IR energy. IR energy is absorbed by the resist which causes uniform heating of the
resist layer thus, reduction of process time and temperature has been observed [Kubenz et al., 2003].

Although there have been multitudes of studies in microwave curing of epoxy based polymers, there has been very little done in terms of microwave curing of photoepoxies. There has been an even smaller amount of studies using VFM. As mentioned previously, a study by Farnsworth et al. [2001] investigated VFM for rapid curing of two photosensitive polyimides, polymer dielectrics which are widely used in the microelectronics industry for a variety of applications. Their results indicate that VFM is a feasible alternative method for processing these type of polymers. Rapid curing of approximately 12-30 minutes where achieved for VFM as compared to 240-300 minutes for thermally cured samples. The VFM processed products were comparable to thermally cured samples in terms of crosslinking and final chemical structure. However, a significant difference in electrical properties was obtained due to the completely different heating mechanism under microwave radiation and conventional heating which caused slow evolution and incomplete removal of volatile reaction by-products during VFM cure. The research study they undertook is quite promising and thus, the use of VFM in the processing of SU8 in this study is a natural progression to extend VFM to other fields.

2.5 SUMMARY

Industrial use of microwaves as a processing tool has been limited due to inherent problems in large cavities. Due to limitations in available microwave sources and limitations imposed by the ISM, only several frequencies (915MHz, 2.45GHz, 5.8GHz and 24.124GHz) are allowed to be used for microwave heating. Thus, most research has been undertaken using these frequencies. Only recently has a variable frequency microwave been developed which uses a broadband of frequencies as opposed to the conventional single-fixed frequency method. This new technology has been able to overcome many of the inherent problems associated with conventional microwave
processing such as heat uniformity and the ability to process metals, however, very little work has been done on the Variable Frequency Microwave Technique thus far.

Of all the advantages of VFM technique one of the main advantages it has over single frequency processing is processing in multimode cavities where heat uniformity can be achieved. The VFM achieves better heat uniformity by controlling several process parameters. These include bandwidth, sweep-rate, and central frequency. To date, little has been done on characterizing the heat uniformity in terms of these process parameters. And even smaller amount of data has been published in the area of modelling of the Variable Frequency Microwave Technique.

Due to its advantages, several applications have been studied using VFM in the fields of polymer processing, materials characterisation and plasma processing. Most of the work has been done in polymer curing as microwaves have a long history of success in polymer curing as seen in past research using single frequency microwaves. Although there has been work published using VFM technology to cure polymers, most of the polymers used have been associated with the electronic and semiconductor industry. Several distinct advantages have been noticed which include a significant reduction in processing time, equal or better quality products, reduction in stress, etc. These are advantages which can benefit other applications.

One such area of research is the area of Micromachining or Micro Electro Mechanical Systems (MEMS). MEMS is the integration of mechanical elements, sensors, actuators, and electronics on a common silicon substrate through microfabrication technology. The micromechanical components are fabricated using compatible "micromachining" processes that selectively etch away parts of the silicon wafer or add new structural layers to form the mechanical and electromechanical devices. One micromachining process is photolithography where photoresists such as SU8 are used. SU8 is a negative epoxy based resist combined with a photo acid generator (PAG) that is capable of producing high aspect ration features with vertical profiles which are properties essential to the development and fabrication of MEMS devices. Current curing techniques for SU8 suffer from very long processing times, which can sometimes reach in the order of hours.
Due to the mismatch in the coefficient of thermal expansion (CTE) between SU8 and its substrate, and the photo initiated cross-linking reaction together with the polymers characteristically inherent rigid molecular structure combine to generate considerable internal stress which causes cracked lithographic features. Several curing techniques are currently employed to cure this type of polymer including hotplates and ovens. Although these are the conventional method of curing, ovens often produce “skin problems” due to the mechanism of heat transfer. Hotplates on the other hand have been seen to have advantages in curing thin films but not in thicker films. Non-conventional curing such as IR baking has also been studied for curing photoresists material such as SU8 which have achieved a reduction in processing time and curing temperature. Although there have been a lot of research using microwaves in general, research in the use of VFM to cure polymers have mostly been in polymers which are specific to the microelectronic and semiconductor industry. There have been no reported uses of microwaves or VFM to cure photoresist aimed at the MEMS/micromachining industry such as SU8.

Although most research in polymer curing has been done with single frequency microwaves, most of the principles can still be applied to VFM. Whilst there are conflicting results by different research group on the benefits of microwave curing of polymers and the existence of a “microwave effect”, it is clearly understood in the field that microwave processing is seen by most to be beneficial to polymer curing. Thus, VMF technology is well suited as an alternative curing method for photoresists such as SU8. There is a need to study this technology further to understand the potential benefits it can offer and at the same time extending its application horizon to other fields.
CHAPTER 3

MICROWAVE THEORY

AND

MATERIAL INTERACTION

3.1 OVERVIEW

The objective of this chapter is to give a background of microwave processing and the associated theory behind microwave heating. In this chapter, the electromagnetic theory and the underlying Maxwell’s equation which governs microwave generation and propagation are presented and typical microwave systems and their components are discussed. The material interaction with microwaves with emphasis on dielectric heating and the mechanism of microwave coupling in polymers are discussed.

3.2 INTRODUCTION

Originally developed during the Second World War for use in navigation and radar target detection, the use of microwaves on the industrial and domestic scenes has increased dramatically in the past thirty years [Thuery and Grant, 1992]. Even in its early days, its potential in heating was recognised and remains a very attractive alternative to conventional heating methods as it can provide benefits which are not easily obtainable otherwise. It has been used in the processing of rubber, polymers, ceramics, composites, minerals, soils, wastes, chemicals and powders [NRC, 1994]. Nowadays, the use of microwaves extends to many industrial and medical sectors. Such growth areas include materials processing where microwave energy gives way to process a wide variety of new and engineered materials. This growth has come about due to the increasing
awareness through research of the unique benefits this technique has to offer potential users.

Microwave processing offers a number of advantages over conventional heating. These include rapid and volumetric heating as the heat does not have to be conducted into the bulk from the surface unlike conventional heating, selective heating and self-limiting reactions which in turn can lead to improved quality and product properties, reduced processing times, and energy and labour savings. Although microwave processing can bring a lot of benefit to material processing, other processing challenges arise from this technique such as non-uniform heating, arcing and thermal runaways. However, with the advent of the Variable Frequency Microwave (VFM) technique, these problems can now be overcome and this has opened new avenues for microwave processing to be applied to new materials.

3.3 ELECTROMAGNETIC THEORY

Microwaves are electromagnetic waves which form a continuous spectrum that lies between millimetre-waves and radio waves. Specifically, they are waves having wavelengths between 0.1 m to 1 m which correspond to frequencies of 30 GHz to 300 MHz respectively [Metaxas and Meredith, 1983] as shown in Figure 3-1. Industrial microwave processing is usually accomplished at the frequencies set aside for Industrial, Scientific and Medical (ISM) Applications – 915 MHz, 2.45 GHz, 5.8 GHz and 24.124 GHz. The ISM frequencies are surrounded by bands allocated for communication purposes thus, microwave apparatus must be carefully designed to prevent interference to other uses of the electromagnetic spectrum.
3.3.1 Microwave Systems

Typical microwave systems consist of three major components: the source, the transmission lines and the applicator. The microwave source generates the electromagnetic radiation which is generated from the acceleration of charge, and the transmission lines deliver the electromagnetic energy to the applicator where it is either absorbed or reflected by the material being processed. The theoretical analysis of each of these microwave components is governed by the Maxwell equations, which describe mathematically all phenomena of electromagnetism, and appropriate boundary conditions.

\[ \nabla \times E = \frac{\partial B}{\partial t} \quad \text{Equation 3 - 1} \]

\[ \nabla \cdot B = 0 \quad \text{Equation 3 - 2} \]

\[ \nabla \times H = \frac{\partial D}{\partial t} + I \quad \text{Equation 3 - 3} \]

\[ \nabla \cdot E = \rho \quad \text{Equation 3 - 4} \]
where $E$ is the electric field vector, $H$ the magnetic field vector, $D$ the electric flux density vector, $B$ the magnetic flux density vector, $I$ the current density vector and $\rho$ the charge density.

3.3.2 Microwave Sources

To achieve the high power and frequencies required for microwave heating, most microwave sources are vacuum tubes. The most commonly used microwave generators are magnetrons, klystrons and travelling wave tube (TWT). Magnetrons, which are used in home microwave ovens, are the most widely used. They are efficient, reliable and low cost. Magnetrons tubes use resonant structures to generate the electromagnetic field and therefore can only generate fixed frequency fields. Klystrons are used for more specialised requirements where there is a need for very high continuous power. They are used in most modern radars, special materials processing equipment and in the medical field in equipment such as those used for radiation cancer therapy. Unlike magnetrons and klystrons, where the tube is used both to create the frequency of the oscillation and to amplify the signal, the TWT only acts as an amplifier. Travelling Wave Tube works by transferring energy from an electron beam to a wave propagating in a helix structure within the tube and can generate a broad band of microwave frequencies typically between 0.5 – 18GHz and are thus employed in variable frequency microwaves.

3.3.2.1 Travelling Wave Tube

The travelling wave tube as used in the variable frequency microwave consists of four major parts as shown in Figure 3-2.

1) an electron gun assembly
2) a RF interaction circuit
3) a focusing magnet
4) and a collector (anode)
When the cathode is heated, a continuous stream of electrons are generated and are drawn to the collector which is at the opposite end of the TWT. This electron beam is focused into a narrow beam by the focusing magnet and fed through the centre of a tightly wound helix structure within the tube. An RF signal is injected into the helix, the speed of which is determined by the pitch of the helix. The TWT is designed such that the velocity of the RF energy is approximately equal to that of the electron beam thus resulting in an interaction between the signal and the beam. As the electron beam travels through the TWT, some electrons are accelerated and some are slowed. As these velocity modulated electrons progress through the helix, they form bunches which in turn transfers kinetic energy onto the propagating RF wave. Consequently, the travelling wave in the tube increases in amplitude as it progresses along the length of the TWT, resulting in amplification of the initial electromagnetic wave.

3.3.3 Microwave Transmission Line

Microwave transmission lines couple the energy of the microwave source to the applicator. Coaxial cables are used as transmission lines in low power systems. At high frequencies coaxial cables suffer considerable losses, thus waveguides are often the transmission line of choice for high frequency applications such as microwave heating systems.
3.3.4 Microwave Applicators

Multimode oven applicators are by far the most widely used applicator. These are found in domestic ovens and for a large number of low power industrial units and many high power installations [Metaxas and Meredith, 1983; Meredith, 1998]. Multimode applicators are mechanically simple consisting of a closed metal box with some means of coupling in power from a generator and versatile having the ability to accept a wide range of heating loads. One of the limitations of Multimode applicators is heating uniformity which is a frequent problem. As the size of the microwave cavity increases, the number of possible resonant modes also increase. For a rectangular cavity of dimensions $a$, $b$ and $d$, in the $x$, $y$ and $z$ directions, the mode equation for the resonant frequencies must satisfy:

$$
\begin{align*}
& \frac{n^2 l^2}{a^2} + \frac{m^2 b^2}{d^2} + \frac{n^2 a^2}{c^2} \\
& \text{Equation 3 - 5}
\end{align*}
$$

where, $f_{nmn}$ is the TE$_{nmn}$ or TM$_{nmn}$ mode’s resonant frequency; $l$, $m$ and $n$ are integers corresponding to the number of half-sinusoidal variation in the standing wave pattern along the principal coordinate axes ($x$, $y$, $z$); and where, $c$, is the velocity of light ($3 \times 10^8$ m/s).

3.4 MICROWAVE INTERACTIONS WITH MATERIALS

In the case of microwaves, the energy transfer does not occur by conduction or convection as in conventional heating, but by dielectric loss. Energy is transferred to materials by interaction of the electromagnetic fields at the molecular level, and the dielectric properties ultimately determine the effect of the electromagnetic field on the material. Microwave heating lies in the ability of an electric field to polarise charges in a material and the inability of this polarisation to follow rapid reversals of an electric field. The microwave heating effect depends on the frequency as well as the power applied.
Under the influence of an alternating field dipole molecules undergo oscillations in response to the high frequency field's polarity changes as shown in Figure 3-3. Materials dissipate microwave energy by two main mechanisms: dipole rotation and ionic conduction. When molecules with permanent or inducible dipoles are exposed to an electric field, they become aligned. If this field oscillates, the orientation changes with each alternation. The strong agitation, provided by the reorientation of molecules, in phase with the electrical field excitation, causes an intense internal heating from internal resistance to the rotation and or intermolecular friction.

![Diagram of molecule movement](image)

**Figure 3-3:** Molecule movement when subjected to an alternating electric field such as Microwaves.

The total polarisation, \( \alpha_t \), is the sum of a number of individual components:

\[
\alpha_t = \alpha_e + \alpha_a + \alpha_d + \alpha_I
\]

*Equation 3 - 6*

where, \( \alpha_e \), is the electronic polarisation and arises from the realignment of electrons around a specific nuclei; \( \alpha_a \), is the atomic polarisation which is the result of the relative replacement of nuclei due to the unequal distribution of charge within the molecule; \( \alpha_d \), is the dipolar polarisation which results from the orientation of permanent dipoles by the electric field. Lastly, \( \alpha_I \), is the interfacial polarisation, or Maxwell-Wagner effect, which occurs when there is a build up of charges at interfaces.
The response of materials to oscillating electric fields depends on the time scales of the orientation and disorientation phenomena relative to the frequency of the radiation. The time scales for the polarisation and depolarisation of electronic polarisation ($\alpha_e$) and the atomic polarisation ($\alpha_a$), are much faster than the microwave frequencies and therefore their effects do not contribute to the dielectric heating effect. The timescales for dipolar polarisation and interfacial polarisation are comparable to microwave frequencies and consequently contribute to dielectric heating.

### 3.4.1 Dielectric Properties

The ability of a dielectric material to absorb and to store electrical potential energy, microwaves must be able to enter the material and transmit energy. The dielectric constant or real permittivity, $\varepsilon'$, characterises the ability of the molecule to be polarised by the electric field, and the dielectric loss factor, $\varepsilon''$, indicates the efficiency with which the energy of the electromagnetic radiation can be converted to heat. These quantify the capacitive and conductive components of the dielectric response which are commonly expressed in terms of the complex permittivity, $\varepsilon^*$, which is a measure of the ability of the dielectric to store and to absorb electrical potential energy.

$$\varepsilon^* = \varepsilon' - i\varepsilon''$$  \hspace{1cm} \text{Equation 3 - 7}

The ratio of the loss factor and the dielectric constant is also another commonly used term for the dielectric response which is called the loss tangent,

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'}$$  \hspace{1cm} \text{Equation 3 - 8}

and is indicative of the ability of the material to convert absorbed energy into heat at a given frequency and temperature.

Materials with a dielectric loss factor of between $1 \times 10^{-2}$ and 5 are well suited to dielectric heating. A loss factor $>5$ leads to a decrease in the penetration depth thus the material only absorbs the majority of the incident microwave power in the first few millimetres leaving the rest of the material unheated. Loss factors $<10^{-2}$ does not absorb microwaves...
well and very high electric fields are required to achieve a reasonable rate of temperature rise [Metaxas and Meredith, 1983].

When microwave energy penetrates a sample, the energy is absorbed by the sample at a rate dependent upon its dissipation factor. Penetration is considered infinite in materials that are transparent to microwave energy, and is considered zero in reflective materials such as metals. The dissipation factor is a finite amount for absorptive samples.

The dielectric properties of the material together with the electromagnetic field and distribution determine the conversion of electromagnetic energy into heat. The power that is transmitted to an object can be determined by the use of the Poynting vector [Metaxas and Meredith, 1983] which can be derived from the Maxwell equations. The pointing vector has unit W/m² and is given by the cross product of $E \times H^*$, where $^*$ denotes the complex conjugate. The power that is transmitted across a surface, $S$, having a volume, $V$, is given by the real portion of the Poynting power theorem:

$$P = \frac{1}{2} \int_V (\omega \mu H \cdot H^* + \omega \mu E \cdot E^* + \sigma E \cdot E^*) dV$$  \hspace{1cm} \text{Equation 3 - 9}$$

Since the magnetic permeability is usually small in dielectric materials, the first term can thus be neglected. The term $\omega \epsilon$ can be considered as an equivalent conductance [Thostenson and Chou, 1999] and hence the second and third term can be combined. If the electric field is assumed to be uniform throughout the volume, $V$, a simplified power, $P$, absorbed per unit volume is:

$$P = \frac{1}{2} \pi \epsilon_0 \epsilon'' E_{rms}^2$$  \hspace{1cm} \text{Equation 3 - 10}$$

From this equation, it can be seen that the conversion of electromagnetic energy into heat is therefore proportional to the frequency, the dielectric loss of the material and the square of the electric field strength. Materials with high conductivity and low capacitance (i.e. metals) have a high dielectric loss factor, however limit the penetration depth of microwaves and this result in a negligible amount of energy being absorbed.
These types of materials are considered as reflectors. Similarly, materials with low dielectric loss factors such as PTFE and quartz, have very large penetration depth which results in very little energy being absorbed. These materials are said to be transparent. Thus, due to this behaviour, energy transfer with microwaves is most effective in materials that have dielectric factors in the middle of the conductivity range. Figure 3-4 illustrates how microwave power is absorbed by different materials as the dielectric loss factor changes.

3.4.2 Mechanism of Microwave Coupling in Polymers

Not all polymer materials are suitable for microwave processing. However, many polymers contain groups that form strong dipoles. These include epoxy, hydroxyl, amino and cyanate groups amongst others. Thus, microwave processing can be used over a broad range of polymer products including thermoplastics and thermosetting resins, rubber and composites.

The principal mechanism of microwave absorption in a polymer is the reorientation of dipoles in the imposed electric field. The ability to process polymeric materials with microwaves depends on the dipole structure, frequency of processing, temperature and additives or fillers that have been included with the polymer. In addition, the coupling
efficiency with the material is dependent on the dipole strength, dipole mobility and the dipole’s mass.

During a processing cycle, the polymers dielectric constant, which is a measure of the ability to absorb microwaves vary as phase changes within the polymer occurs. In thermosetting polymers, the viscosity and molecular weight tend to increase during the cure as they become a highly cross-linked network. Studies have shown that the permittivity and the dielectric loss factor of these types of polymers generally increase with temperature and decrease with extent of cure [Jow et al., 1988]. Thus, initially thermosets are efficient absorbers of microwave energy as $\varepsilon''$ increases as temperature increases.

### 3.5 SUMMARY

This chapter presented a detailed background on microwave theory and gave an overview of the constituents of a microwave heating system. The basics of microwave heating were discussed and the governing equations relevant to this type of heating were introduced. Moreover, the interaction between microwaves and material and more specifically the behaviour of polymers under microwave radiation have been discussed, all of which are an important basis for this study.
4.1 OVERVIEW

This chapter presents the main experimental equipment used in this thesis. This section has been divided into three parts for simplicity. Firstly, an overview of the VFM processing equipment (MicroCure 2100) and experimental setup for optimum processing are presented. This is followed by the heat-mapping techniques used to quantify the heat uniformity in the VFM in Chapters 6 and 7. Finally, the material characterisation techniques used to analyse the conventionally and VFM cured SU8 in Chapter 9 are presented.

4.2 VARIABLE FREQUENCY MICROWAVE: MICROCURE 2100

All the microwave heat-mapping studies and SU8 curing studies were undertaken using the MicroCure™ 2100 Variable Frequency Microwave from Lambda Technologies which can operate at a maximum power of 200W and a frequency range of 2.5-8.0GHz. This section discusses the main features of the MicroCure 2100 including temperature control.

The MicroCure 2100 comprises of several separate subsystems. Foremost is the Oven Control System which maintains control over the entire system components. It is a software based system on a PC and serves as an interface to the other subsystems and as the central data collection and control point. The microwave energy required by the
equipment is generated in the Signal and High Power Amplifier (HPA) System which consist of a YIG oscillator, a voltage controlled attenuator, PC data acquisition and the VFM interface board. This system generates all of the low-level microwave energy and controls all the systems high-level energy. A 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. This high power microwave energy is routed to the curing cavity by the Transmission System comprising a high power isolator, a dual directional coupler which is combined with crystal detectors to provide power sampling for the system, a waveguide and an iris-style launcher. Finally, there is the Curing cavity, a metal enclosure where the curing process takes place. An overall system schematic diagram of the MicroCure 2100 is shown in Figure 4-1.

**Figure 4-1:** Schematic diagram of MicroCure 2100 Variable Frequency Microwave System.
4.2.1 Curing Cavity

The MicroCure 2100 cavity is a multimode applicator constructed from high-grade aluminium of size 365mm x 355mm x 482mm where curing took place as shown in Figure 4-2. The cavity features a manual hinged door with microwave seals around the door facing. The seals provide good electrical contact and prevent microwave leakage from the cavity enclosure. The cavity has inlet and outlet ports and openings for fibre optic temperature probes.

The samples being processed were placed at the bottom of the cavity on a Teflon block support of dimensions (h=45mm x d=125mm) to ensure proper microwave field distribution. Teflon was used as the support because of its negligible microwave absorption [Kingston and Haswell, 1997], thus its contribution to the heating characteristics of the material being processed is minimised.

![Figure 4-2: MicroCure 2100 cavity with Teflon block used as sample support.](image)

4.2.2 Oven Control System

The oven control system was software driven and was utilised and maintained control over all system components. An industrial PC served as the central data collection and control system and interfaced with and supervised all of the subsystems. It was
responsible for operating the graphical user-interface as well as maintaining the input and output functions of the VFM system.

The MicroCure 2100 allows a number of controllable parameters which includes central frequency, sweep rate, bandwidth, power level and ramp rate. Samples can be processed at a fixed frequency ranging from 2.5-8.0GHz or using a variable frequency sweep with a chosen central frequency between that range and bandwidth of 0-100% of the frequency range using sweep times of 0.1-60sec. Forward power or power level can be varied between 50-200W in 10 Watt increments. A feedback control system is used to control the sample temperature by using the temperature probes. The control system adjusts the power levels automatically to maintain the sample at the desired temperature thus allowing for very good control of ramp rates and hold temperatures.

4.2.3 Signal Generator and High Power Amplifier

Unlike conventional microwave systems, the Variable Frequency Microwave does not use a magnetron for signal generation. Instead, the VFM the signal generation system consists of a YIG Oscillator, a voltage controlled attenuator, PC data acquisition and a VFM interface board. This system generates all the low-level microwave energy and controls all of the systems high energy. A high power amplifier (HPA) amplifies the low-level microwave energy from the signal generation system to levels high enough to provide adequate molecular excitation in the curing cavity. The HPA consists of a Travelling Wave Tube as discussed in Chapter 3, a high voltage power supply, a solid state amplifier that serves as an intermediate power amplifier, and control logic. Communication with the PC is via an RS-232-C serial communication link.

4.2.4 Variable Frequency Microwave Furnace (VFMF) Safety PCB

This system controls the safety of the VFM from releasing high-level microwave energy. This stand-alone device senses the high-level microwave energy in the transmission system and also monitors the cavity door safety interlocks and other high-level RF safety interlocks to determine whether they are in a safe position. If the system detects
dangerous levels of RF energy in the transmission system while an interlock is tripped or if the cavity is overheated the VFMI Safety PCB will sever the safety interlock line in the HPA. The system prevents production of high-level microwave energy until a safe condition is detected.

4.2.5 Transmission System

The transmission system is used to route high power microwave energy from the HPA to the curing cavity. The system comprises of a high power isolator, a dual directional coupler which is combined with crystal detectors to provide power sampling for the system, a waveguide and iris-style launcher.

4.2.6 Temperature Monitoring System

Temperature measurement within the microwave cavity is one of the most important parameters in microwave processing. Temperature measuring devices should be able to maintain good thermal contact with the sample and should produce minimal perturbation of the electric field within the cavity whilst not being affected by the electromagnetic fields themselves. Standard to the MicroCure 2100 VFM is a four channel Nortech NoEMI-TSTM Family type fibre optic measurement system supplied by Nortech Fibronic, Inc. This consisted of four fibre optic probes with a Teflon cladding and high temperature epoxy tips within the cavity. The fibre optic probes can handle temperatures up to 250°C with an accuracy of ±1°C and resolution of 0.1°C. These were put into contact with the surface of the sample to be monitored using a high temperature Kapton tape. Although four channels are available and can monitor temperature simultaneously, only one channel can be used as a feedback control for keeping steady temperatures during microwave curing of the samples.

4.2.7 MicroCure 2100 System Software

The MicroCure 2100 VFM is controlled by proprietary software from Lambda technologies. The VFM can be run in either Automatic mode which is designed for increased ease of use with limited control on processing conditions, or Manual mode
which allows detailed and full control of the VFM’s functionality. Under manual mode, more control over the system is possible. Processing conditions such as power control, sweep setup (central frequency, bandwidth, sweep rate), processing mode (fixed-frequency or VFM) can be chosen. The aforementioned settings can be accessed through the main Graphical User Interface (GUI) of the software which also allows the user to monitor the status of the system at any time during processing through a series of status displays, quantitative displays and controls as seen in Figure 4-3.

![Graphical User Interface of the MicroCure 2100 System software](image)

**Figure 4-3:** Graphical User Interface of the MicroCure 2100 System software.

There are two important utilities that the software incorporates, namely the Cavity Characterisation and Programming features which will be described in more detail below.

### 4.2.7.1 Cavity Characterisation of VFM

The cavity characterisation feature of the VFM allows the measurement of cavity characteristics when a sample is loaded. It is a sequence whereby the ability of a sample to absorb microwave energy can be determined by measuring the incident and reflected power as a function of frequency for a given frequency range. The frequencies where
reflected power is high can therefore be avoided and the most favourable conditions i.e.
maximum power absorption, suitable for microwave processing can be chosen. Thus the
most suitable frequency bandwidth can be chosen so that strong coupling of energy inside
the material can be obtained. This feature is a quick and simple method to determine the
suitability of materials for VFM or microwave processing in general if dielectric
properties are unknown.

To minimise the number of high reflected power faults, a cavity characterisation using
the MicroCure 2100 was undertaken to determine the characteristic of an unloaded cavity
during processing. Results are shown below in Figure 4-4.

![Figure 4-4: Cavity Characterisation of an unloaded VFM cavity at 50W.](image)

The result above show that high reflectance is experienced from the cavity which trips the
safety mechanism built into the MicroCure 2100, consequently stopping microwave
generation during the cavity characterisation as illustrated above at approximately
3.05GHz after which no reading could be taken. This is extremely undesirable as high
reflectance could halt microwave generation during experiments or at worst damage the
system. To prevent this from occurring, microwave absorbing loads were placed in the
cavity so that excess power will not be reflected back to the source which may damage the HPA. Another cavity characterisation was undertaken with the load placed inside the cavity and results shown in Figure 4-5.

Figure 4-5: Cavity Characterisation of cavity with microwave absorbing loads.

Figure 4-5 shows that high reflectance in the cavity still exist however reflectance is lower due to the microwave absorbing loads. The cavity characterisation in Figure 4-5 indicate that reflectance decreases as the frequency increases, suggesting that the higher frequencies are more favoured in this particular cavity.

4.2.8 Programming Menu/Recipes

The programming menu is a useful function of the VFM Software and is where a series of commands can be defined to establish a sequence of event for a given process or processing condition. Parameters such as power, frequency, ramp time, temperature, can be written in a script for ease of use. The script or “Recipe” can be loaded and allows for the automation of the MicroCure 2100 and allows for easy change of parameters if required. Several recipes were written for the heat-mapping of the cavity presented in
Chapters 6 and 7 and for the processing of SU8 as presented in Chapter 9. These recipes are presented in Appendix C.

4.3 HEAT MAPPING

Heat-mapping is an indirect method of visualising the distribution of electromagnetic energy within a microwave cavity. Two heat-mapping techniques were used in this thesis and are described below.

4.3.1 Thermal Imaging

An AGEMA infrared system (Thermovision model 570) was used for the heat-mapping of the VFM. The IR system is a complete, stand-alone temperature measurement camera which has a spectral range of 8-12microns and a MicroBolometer detector with image size of 320 x 240 pixels. The IR camera has a spatial resolution of 1.1 mrad with an accuracy of ±2%, or 2°C. Other particulars of the IR camera and its setting are shown in Table 4-1.

<table>
<thead>
<tr>
<th>Model Type</th>
<th>THV 570</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lens</td>
<td>24</td>
</tr>
<tr>
<td>Emissivity</td>
<td>0.91</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>20°C</td>
</tr>
<tr>
<td>Atmospheric Temperature</td>
<td>20°C</td>
</tr>
<tr>
<td>Relative Humidity</td>
<td>50%</td>
</tr>
</tbody>
</table>

Table 4-1: Equipment settings for Agema Thermovision IR Camera

Microwave absorbing materials were used to facilitate in heat mapping of the VFM cavity. Thermal images of these microwave absorbing materials were taken after heating them up using a prescribed heat up sequence. Thermograms were taken at 4 second intervals for approximately 120 seconds. The resulting IR images were analysed using the software IRWin Research v2.01 to determine temperature difference in the cavity.
4.3.2 Chemical Heat-Mapping

A second type of heat-mapping technique as used by Steyn-Ross and Riddell [1990] and described in literature, was utilised to obtain a qualitative form of the heat distribution in the VFM cavity. The heat-mapping technique made use of the colour properties of cobalt chloride ($\text{CoCl}_2$) soaked paper that is pink in wet conditions and blue when dry. The heat-sensitive paper of size 210mm x 297mm was supported on a platform and placed inside the multimode cavity of the VFM. Heat maps were taken at a fixed-frequency of 2.45GHz as well as variable frequency to show and contrast the heating advantage of VFM over conventional fixed-frequency methods. A ‘recipe’ was programmed into the VFM for each case and a power was chosen to obtain drying in sufficient time.

4.4 MATERIALS CHARACTERISATION

4.4.1 Dielectric Measurements

Dielectric properties were measured using the Dielectric Probe Method. The test equipment consisted of an open-ended coaxial line connected to an Agilent 8722ES S-parameter network analyser with an operating bandwidth of 50MHz-40GHz to measure the complex permittivity of materials.

The open-ended coaxial probe consisted of a truncated section of a coaxial line and was connected to the network analyser via a coaxial cable. Before measurements of the dielectric properties were taken for a particular frequency, the dielectric probe had to be calibrated against substances with known dielectric properties. These were air, pure water and a metallic shorting block. Due care was taken not to introduce bubbles in the sample and calibration water whilst calibrating the system and whilst taking readings.

4.4.2 Thermal Analysis

Polymers typically display broad melting endotherms and glass transitions as major analytic features associated with their properties. The characterisation of polymers
requires a detailed analysis of these characteristic thermal transitions. By heating over a
temperature range certain properties can be measured and are commonly required for
characterisations of polymers. Polymers are characterised by a melting range instead of a
discrete melting temperature. They are also characterised by a glass transition
temperature \( (T_g) \) below which the polymer has “glassy” properties or hard and brittle.
Above the \( T_g \), the polymer behaves like a rubber or polymer melt. In this project, the
thermal analysis methods of Thermogravimetric Analysis (TGA) and Differential
Scanning Calorimetry (DSC) were used to characterise the photosensitive SU8 when
dried and cured using the VFM. These techniques were used to obtain properties such as
Glass Transition Temperature, Melting Temperature, Degradation Temperature, material
composition and degree of cure.

4.4.2.1 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is the study of weight gained or loss from the
decomposition, oxidation or loss of volatiles of a condensed phase due to the evolution or
absorption of gas from a sample as a function of temperature. Weight loss during heating
is a common phenomenon for polymers due to the degradation and loss of residual
solvents and monomers. TGA measures the amount and rate of change in mass of a
sample as a function of temperature or time in a controlled atmosphere. These
measurements are used primarily to determine the thermal and/or oxidative stabilities of
materials as well as their compositional properties.

The thermal stability of the SU8 was evaluated using a TGA system from Mettler Toledo
(TGA850 Thermal Analyser). Small amount of the uncured and cured SU8 were used
(<10mg) and studied at a ramp rate of 10°C/minute from room temperature to a
temperature of 1000°C under a nitrogen blanket. The weight loss and degradation
temperature were determined using the proprietary software from the manufacturer.
Minimisation of error in the thermal techniques used was conducted through the
repetition of the measurements.
4.4.2.2 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) involves the measurement of relative changes in temperature and heat or energy either under isothermal or adiabatic conditions. DSC measures the quantitative heat flow as a direct function of time or the sample temperature. A DSC system is composed of two identical cells in which the sample and a reference cell, which is empty, are placed. Both cells are heated with a constant heat flux using separate heaters and the temperatures of the two cells are measured as a function of time. The output of a DSC is a plot of heat flux versus temperature at a specified temperature ramp rate from which key physical and chemical properties such as the Glass Transition temperature ($T_g$), Melting Temperature ($T_m$) and Degree of Cure ($\alpha$) can be extracted or calculated.

The DSC used in this study was a Mettler Toledo DSC 821e. Small amounts of the cured samples (<10mg) of the SU8 were evaluated using the DSC to determine the transformation within the material during thermal processing. The temperatures of transformations as well as thermodynamics were determined at a ramp rate of 10°C/min to a temperature of 370°C, under a nitrogen blanket. As with the TGA, error minimisation was achieved through the repetition of experiments.

4.5 SUMMARY

This chapter introduces the main experimental equipment used in this project – the MicroCure 2100 Variable Frequency Microwave from Lambda Technologies. The main parts of the VFM were discussed and its operation and control were outlined. A thorough introduction to the operating software was given and the most relevant functions introduced. Moreover, the experimental techniques employed for the characterisation of the VFM and for characterising the cured and uncured polymer samples and the methodologies undertaken were discussed.
CHAPTER 5

RESULTS -

THEORETICAL CHARACTERISATION OF

VARIABLE FREQUENCY MICROWAVE

5.1 OVERVIEW

Modelling of a VFM system was undertaken using the commercial software – CST Microwave Studio. The software uses the finite integration technique (FIT), and is well suited to the high frequencies used by VFM. A commercial VFM oven operating on a broadband frequency range of 2.5-8GHz was modelled using a transient solver and the uniformity of the electric field inside its cavity was investigated through the resulting energy density distribution. Results of the modelling work emphasising the advantages of this new technique over fixed-frequency microwaves are presented.

5.2 INTRODUCTION

Solving Maxwell’s equations by numerical methods has long been used to simulate electromagnetic fields in cavities. The Finite Difference Time Domain (FDTD) first introduced by Yee [1966] and the Finite Element methods (FEM) being the most used techniques however, hybrid models have also been employed. Unlike fixed-frequency microwave, simulations pertaining to VFM have been lacking. Simulations with customised or commercial software are a practical and efficient way to visualise and understand what is occurring inside a microwave cavity. Several researchers have utilised either a customised process specific model or commercial software such as ANSYS to simulate VFM in order to visualise and predict the VFM technique [Surrett et
al., 1994; Johnson et al., 1994b; Panchapakesan et al., 1997; Yi et al., 2001]. In this paper, a commercial software package – CST Microwave Studio – which is based on the Finite Integration Technique (FIT) modelling [CST, 2002], is utilised to give a qualitative view of the aforementioned improved heating using VFM. The package is used to predict electric field (E-field), from which the energy distribution may be calculated.

5.3 METHODOLOGY

A commercial Variable Frequency Microwave from Lambda Technologies model type Micro Cure 2100 was used as a basis for simulating VFM technique using CST Microwave Studio. In the following sections, the steps undertaken to develop the model is discussed. It should be noted that the underlying principles regarding the modelling software will be discussed and the governing equations will be introduced. However, derivations of these equations are beyond the scope of this thesis and therefore will not be shown. A more detailed derivation of the formulas can be seen in the paper by Weiland [1977].

5.3.1 CST Microwave Studio

CST Microwave Studio is a general-purpose electromagnetic simulator based on the Finite Integration Technique (FIT). This numerical method was first proposed by Weiland in 1976 [Weiland, 1977] and provides a universal spatial discretisation scheme which is applicable to various electromagnetic problems ranging from static field calculations to high frequency applications such as the VFM, in the time or frequency domain.

Unlike most numerical methods, the FIT discretises the following integral form of Maxwell’s equations, as shown below, rather than the differential form.

\[ \oint_{\partial A} \vec{E} \cdot d\vec{s} = -\int_{A} \frac{\partial \vec{B}}{\partial t} \cdot d\vec{A} \]

Equation 5 – 1
In order to solve these equations numerically a finite calculation domain is defined, enclosing the considered problem. This is achieved by creating a suitable mesh system which is split up into many small cuboids or grid cells.

5.3.1.1 Solver

A transient solver of the software was used because of its flexibility in handling low and high frequency problems. The transient solver is based on the solution of the space discretised set of Maxwell’s Grid Equations.

\[
\int_{\mathcal{D}} \vec{H} \cdot d\vec{s} = \int_{\mathcal{A}} \left( \frac{\partial \vec{D}}{\partial t} + \vec{J} \right) \cdot d\vec{A} \quad \text{Equation 5 - 2}
\]

\[
\int_{\mathcal{V}} \vec{D} \cdot d\vec{A} = \int_{\mathcal{V}} \rho \, dV \quad \text{Equation 5 - 3}
\]

\[
\int_{\mathcal{V}} \vec{B} \cdot d\vec{A} = 0 \quad \text{Equation 5 - 4}
\]

In which the time derivatives are substituted by central differences yielding the explicit update formulation for a loss-free case

\[
e^{n+1/2} = e^{n-1/2} + \Delta t M^{-1} \left[ \tilde{C} M^{-1} b^n + j_s^n \right] \quad \text{Equation 5 - 9}
\]

\[
b^{n+1} = b^n - \Delta t C e^{n+1/2} \quad \text{Equation 5 - 10}
\]
In which the calculation variables are given by the electric voltages and the magnetic fluxes. Both unknowns are located alternately in time in the leap frog scheme. Which means that the magnetic flux at $t = (n + 1)\Delta t$ is computed from the magnetic flux at the previous time step $t = n\Delta t$ and from the electric voltage at half time step before, at $t = (n + 1/2)\Delta t$ as demonstrated in Figure 5-1.

![Figure 5-1: The Leap Frog Scheme for solving of discretised equations with respect to time (t).](image)

5.3.2 Model

In modelling of the MicroCure2100 VFM, the structure to be modelled essentially consists of two parts, the microwave cavity where all the materials processing occurs and the VFM horn, a double ridge waveguide through which the microwave energy from the microwave source, the TWT, is introduced into the cavity. This structure is sufficient to obtain the response of the VFM to an excitation pulse and obtain the electromagnetic fields within the cavity. All other components before the VFM horn can be neglected in the model.

5.3.2.1 Cavity

The MicroCure2100 has a bandwidth of 2.5-8GHz and a maximum operating power of 200W and has a processing volume consisting of an Aluminium (Al 6061-T6) multimode cavity of size 365mm x 355mm x 482mm. The cavity (Figure 5-2) is a highly resonant structure and contains negligible internal losses and is expected to have a very high Q factor. Only the air filled part of the structure is modelled, since the enclosing walls are conducting and the E-field on the walls is zero. This will also lower the structures Q
factor so that the energy introduced decays faster and hence a quicker solution convergence is obtained.

**Figure 5-2:** Cavity of MicroCure 2100 Variable Frequency Microwave from Lambda Technologies.

### 5.3.2.2 VFM Horn

The VFM horn is the link between the microwave source and the cavity. The VFM Horn is connected to the upper left corner of the back of the cavity and amplified electromagnetic energy is channelled through this before it enters the cavity.

The VFM Horn is a Double-Ridged Waveguide that is terminated with a pyramid funnel-shaped horn. The ridges in this waveguide increase the bandwidth of the guide at the expense of higher attenuation and lower power-handling capability. The bandwidth can easily exceed that of two contiguous standard waveguides. Introduction of the ridges mainly lowers the cut-off frequency of the TE10 mode from that of the unloaded guide, which is predicated on width alone. The pyramid funnel-shaped horn was utilised to obtain impedance matching through a gradual change in impedance at the end of the waveguide so that almost no standing waves are formed. Since the impedance of the waveguide does not match the impedance of the cavity, impedance matching is required in order to optimise the power delivered to the load from the source. Without proper impedance matching, the waveguide would have suffered from abrupt changes in
impedance which causes standing waves which can largely decrease the efficiency of the waveguide. Waveguide horns have several advantages over other impedance-matching devices such as their large bandwidth, which makes them ideal in Variable Frequency Microwaves, and their simple construction.

The VFM Horn had an overall length of 165mm with a horn opening having dimensions of 117mm x 155mm. As with the modelling of the cavity as previously discussed, only the air filled part of the structure is modelled, since the enclosing walls are conducting and the E-field on the walls is zero. A waveguide port at the end of the ridged waveguide was defined as the excitation source and a gaussian excitation pulse was used with a bandwidth of 2.5-8GHz. The model of the VFM Horn used in the software is shown in Figure 5-3.

![Figure 5-3: Model of the VFM Horn (Clockwise from Top Left – Back View, Side View, 3D view, Top View).](image)
5.3.2.3 Boundary Conditions and Meshing

The boundary conditions were set as conducting walls so that the internal can be modelled as air. An expert mesh system unique to CST Microwave Studio was utilised which discretised the structure into a mesh size of \( N_x = 98, N_y = 97 \) and \( N_z = 176 \) resulting in model consisting of approximately 1.7 million nodes.

A model of the VFM cavity and the ridged-waveguide was created in the software as per the MC2100 dimensions as shown in Figure 5-4. A summary of the model parameters is shown in Table 5-1.

<table>
<thead>
<tr>
<th>INPUTS</th>
<th>SETTINGS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solver Type</td>
<td>Transient</td>
</tr>
<tr>
<td>Excitation Type</td>
<td>Gaussian Excitation Pulse</td>
</tr>
<tr>
<td>Boundary Conditions</td>
<td>External = Conducting Walls ((E=0))</td>
</tr>
<tr>
<td></td>
<td>Internal = Air ((\epsilon = 1))</td>
</tr>
<tr>
<td>Frequency ((f_{\text{min}}, f_{\text{max}}))</td>
<td>(f_{\text{min}} = 2.5\text{GHz}, ) (f_{\text{max}} = 8.0\text{GHz})</td>
</tr>
<tr>
<td>Mesh Type</td>
<td>Expert Mesh System ((N_x = 98, N_y = 97, N_z = 176))</td>
</tr>
</tbody>
</table>

Table 5-1: Summary of model inputs on CST Microwave Studio.
5.3.3 Macro

A macro was written to run within the software in order to obtain the broadband energy distribution within the cavity to illustrate the effect of variable frequency microwave technique. The resulting energy distribution can be simulated by the summation of the contribution of each launched frequency into the cavity. Due to limitation in computing memory, only several frequencies contribution was summed within the bandwidth (2.5, 3, 4, 5, 6, 7, 8GHz). In reality, the frequency bandwidth of 2.5-8GHz is divided up into 4096 discrete frequencies each one contributing to the final time averaged E-field and hence the energy distribution.

5.4 RESULTS & DISCUSSION

Simulation of the MicroCure 2100 VFM cavity was undertaken at a broadband frequency of 2.5-8.0GHz for the purpose of characterising the cavity and analysis of the distribution of microwave energy within it.
As the internals of the cavity and ridged-waveguide were predominantly empty, the two components were simulated as one system and its internal was modelled as air. Due to the asymmetrical nature of the resulting model (i.e. cavity and waveguide), the model could not be simplified using symmetrical planes and together with its large size, simulations were quite time and CPU intensive. Apart from its size, the cavity is also a highly resonant structure and thus a considerable amount of time for the energy to reach steady state as shown in Figure 5-5 is observed. It should be noted that the model is not assumed to be free from numerical errors or numerical diffusion which are unavoidable in models such as these. However these errors have been minimised as much as possible to obtain accurate results. Quantifying these errors are out of the scope of this project but are worthy of further studies.

![Graph showing time to reach steady state.](image)

**Figure 5-5:** Graph showing time to reach steady state.

Figure 5-6 shows % Absorption vs. Frequency graph of the model as given by Equation 5-11 below. Power absorption within the bandwidth is consistent of a cavity with a very high Q factor.
To verify the “VFM Macro” that was written into the software does model the power distribution of variable frequency microwaves, a simple waveguide was simulated at two different frequencies and their respective resulting energy density distribution, which is representative of the E-field shown in Figure 5-7. The results show two distinct modes which after the macro was applied illustrate in a simplistic way the ability of the VFM technique to achieve an even power distribution. This is analogous to what some researchers [Qui, 2000] have termed “Complementary Heating”, which is based upon the alternation between two or more electromagnetic modes which have complementary heating patterns by mode-switching in fixed-frequency processing. This is usually achieved by altering the geometry of the applicator which inherently suffers from a time-delay associated with mechanically changing the dimensions of the cavity. In the VFM technique, complementary heating modes are achieved by varying the frequency using
CHAPTER 5: RESULTS – THEORETICAL CHARACTERISATION OF VARIABLE FREQUENCY MICROWAVE

the Travelling Wave Tube (TWT). The TWT can vary frequency so the cavity can accommodate a different resonant mode at a given time hence the motivation for sweeping a bandwidth of frequencies to obtain heating patterns which are complementary of each other.

![Model results verifying VFM Macro. Top images illustrates heating modes of two single frequencies and the resulting heating mode after the VFM Macro was applied (bottom).](image)

In a similar manner as above, the simulation was undertaken for the VFM cavity at a bandwidth of 2.5-8GHz and the resulting energy density distribution were plotted. To compare and determine the extent of distribution of the E-field within the cavity, the energy density distributions were examined on three levels on the $xz$-plane (above, in-plane and below the source), which is expected to be the predominant plane samples will be positioned. The E-fields at two single frequencies, 2.5 GHz and 5GHz, were acquired and a birdseye view of the energy density distribution at the three levels is shown in Figure 5-8 and 5-9, respectively, to demonstrate how the cavity behaves in fixed frequency mode. Likewise, Figure 5-10 shows the broadband energy density distribution inside the cavity during VFM processing at 2.5-8GHz after the VFM macro was applied.
Figure 5-8: Energy distribution at xz-plane for 2.5GHz  a) y = -200mm b) y = 0mm c) y = 70mm.

Figure 5-9: Energy distribution at xz-plane for 5GHz  a) y = -200mm b) y = 0mm c) y = 70mm.

Figure 5-10: Energy distribution at xz-plane for Variable Frequency Microwave of bandwidth 2.5GHz – 8.0GHz  a) y = -200mm b) y = 0mm c) y = 70mm.
As illustrated above, processing at a single frequency of 5GHz (Figure 5-9) produces a better energy distribution within the cavity than processing at the lower frequency of 2.5GHz (Figure 5-8). Overall, a more uniform E-field is achieved at all three planes. Although hotspots are still visible, at higher frequencies the wavelength is shorter and the resulting resonance modes produce hotspots which are closer in proximity to each other compared to that at the lower frequency of 2.5GHz. In general, higher frequencies processing like that in Figure 5-9 produce modes with more hotspots that are closer to each other than using low frequencies and may seem to be the solution to achieving greater E-field uniformity however, lower frequencies are able to penetrate deeper into a substance to create volumetric heating but the power absorbed per unit volume i.e. the conversion of electromagnetic energy into heat in a material, is lower. Thus, it seems imperative to use quite a wide band of frequency to achieve high penetration and power absorption as is used in the MicroCure 2100. In doing so, the low and high frequencies create complementary heating modes which will overlap as they form within the cavity. Whilst lower frequencies produce hotspots that are further apart, modes from higher frequencies will ensure that areas of low microwave energy will be occupied or filled in thus obtaining a uniform E-field. Figure 5-10 demonstrates this theory where an improvement in field uniformity can already be experienced with the summation of just seven modes from seven different frequencies. In reality, the bandwidth of the VFM is divided into thousands of discrete frequencies which are launched consecutively with each frequency contributing to the final time averaged E-field. It can therefore be concluded that more resonant frequencies in the microwave oven, the better the time averaged field distribution. In terms of heating, a more even temperature distribution can be achieved.

It is interesting to note that E-field intensities vary at different planes for both fixed and VFM processing as observed in the results. It shows that the greater E-field intensities are experienced closer to the microwave source and decreases with distance from the source. This suggests that the microwave energy dissipates as it travels outwards from the source. For materials processing, although VFM will provide a more uniform E-field that will ensure a higher probability of uniform processing than its fixed-frequency
counterpart, the actual position of the samples within the cavity with respect to the source will have effect on the amount of power exposed to sample which may have affect on processing speed or product quality. Higher E-field intensities and hence power is experienced closer to the source plane i.e. waveguide, as expected. Placing samples closer to the wave source will ensure higher E-field intensities, however this may be detrimental to the VFM system as found by Yi et al. [2001]. Their simulations using a 3D FEM model found that sample position is very important to the harmonic vibration and field distribution within a cavity and placing samples to close to the wave source can sometimes eliminate the resonance i.e. formation of any modes. Although their model is quite small (60mm x 70mm x 100mm) compared to the model studied here and utilises a rectangular waveguide instead of a ridge-waveguide and only a bandwidth of 0.2GHz, the size of their cavity makes it more sensitive to changes in the internal of the cavity. Nevertheless, the position of a similarly sized sample (30mm x 20mm x 1mm) in the cavity studied herein is also expected to have an effect on the resulting E-field although perhaps not as obvious. More pronounced changes to the E-field would occur with larger samples. Their findings are quite supportive and emphasised other findings in this area of this research program. They proved the VFM concept in a small cavity and predicted that the rapid increase in modes with an increase of size will achieve greater uniformity which has been proven here.

5.5 SUMMARY

A commercial electromagnetic modelling software was used to model the energy distribution within a Variable Frequency Microwave cavity. The software was found to be an efficient and cost-effective tool method to simulate VFM and to examine the reported advantages of this new technique. As expected a broadband frequency produced better distribution of power than a single frequency input. Hence, for heating purposes a sample will experience more even heating than heating with a single microwave frequency. However, it was also found that E-field intensity is dependent on the distance away from the microwave source thus, sample placement is still an important factor to consider when utilising the VFM technique.
6.1 OVERVIEW

In this chapter the parameters that affect the deposition of electromagnetic energy within a Variable Frequency Microwave Cavity are investigated. Unlike other microwave ovens that use mechanical means to obtain a uniform field distribution in multimode cavities, several parameters can be adjusted electronically in VFMs to achieve these. These parameters include the frequency operating mode, sweep rate, operating bandwidth and central frequency. A heat mapping technique using a thermal imaging camera and a dielectric absorber were used to quantify and give a qualitative analysis of the energy deposition in the VFM cavity and the results are presented herein.

6.2 INTRODUCTION

The VFM technology has several adjustable parameters that distinguish it from the conventional single frequency microwave technology: Variable Centre Frequency, Bandwidth, and Sweep rate. The central frequency irradiated inside the microwave can be tuned to increase the coupling efficiency with the material to be processed, whilst the combination of bandwidth and sweep rate can be tuned around the selected central frequency to redistribute the microwave energy to obtain more uniform heating throughout the work-piece. Finally, the microwave incident power can be pulsed or continuously varied to provide control in real time over the heating profile and minimise
reflected power and/or maintain a preset temperature or heating rate of the work-piece, as is the case for fixed frequency microwave oven.

6.3 EXPERIMENTAL METHODOLOGY

The experimental apparatus consisted of a urethane-based absorber (ARC-UD-11554), resonant-tuned EM material measuring 150mm x 150mm was mounted on a sheet of microwave transparent material in the middle of the cavity and irradiated with microwave energy at different VFM and fixed frequency settings. The 4.4mm thick absorber material is especially designed to absorb electromagnetic energy in the range of 0.7–18GHz which accommodates the operating range of the MicroCure 2100 and was chosen for this reason. The dielectric absorber was irradiated in the VFM at a moderate power level of 100W and a preset amount of time (60seconds) such that lateral heat diffusion is limited and that any areas of low and high microwave absorption can be clearly observed. After irradiation, the material was removed from the cavity and placed under an AGEMA infrared system (Thermovision model 570) as presented in Chapter 4, to evaluate the delivery of the electromagnetic field by measuring the temperature distribution on the surface of the microwave absorber. Thermal images of the microwave absorber where taken at a frequency of one every 4 seconds which were consequently analysed using the proprietary software IRWIn Research v2.01 also from AGEMA.

6.4 RESULTS

The following results are presented to demonstrate the effect on heat uniformity of the main VFM parameters which included:

a. Processing Mode – Fixed Frequency Mode
b. Sweeprate
c. Bandwidth
d. Central Frequency
Initially, visual interpretation of the results is presented and then a quantitative analysis is undertaken. The outcome gives a clearer understanding of the VFM parameters and how they each affect heating uniformity and also provides a basis on which the VFM technique can be optimised.

6.4.1 Effects of Processing Mode: Fixed Frequency Mode in VFM Cavity

One of the main advantages of the MicroCure 2100 VFM is the ability to switch from utilising a bandwidth of frequency to processing with using just a single or fixed frequency. This can be achieved in two ways firstly by using a “fixed-frequency” option built into the MicroCure 2100 software or by using a very small bandwidth around a central frequency. The latter option is still technically using the VFM technique as the small bandwidth will still be swept through however, if the bandwidth is small enough, it will be similar to fixed-frequency processing. For the purpose of this study, the “Fixed Frequency” option was chosen and mapped at the following Fixed Frequencies – 2.5, 4.0, 5.25, 6.0 and 8.0GHz. Results are illustrated in Figure 6-1.

The results correlate well with the energy deposition simulations presented in Chapter 5 for fixed frequency microwave processing in the MicroCure 2100 and highlight the inefficiencies of using fixed frequencies in a multi-mode cavity. As previously shown, heat uniformity greatly suffers in fixed frequency processing and this is more evident at the lower frequencies which can be attributed to the larger wavelength at these frequencies. As frequency increases the distance between the hotspots and coldspots become closer because of the decreasing wavelength therefore improving the overall heat uniformity.

The thermal images show an edge effect that can be observed at the edges of the samples. The thick material used obviously causes a distortion of the electromagnetic field such that a higher build-up of energy and hence a higher temperature is experienced at the edges. This phenomenon is common to multimode cavities such as the one used in the VFM and is termed the “Edge Overheating Effect” [Risman, 2004]. This effect is a non-
resonant diffraction phenomenon caused by the E-field component parallel to the edge of high permittivity loads and is observed in different load geometries especially loads with flat surfaces with high permittivity. As the results show, the edge effect is more evident at the lower frequencies of 2.45 and 4.0GHz than at higher frequencies.

Figure 6-1: Thermal images showing the effects of different Fixed Frequencies on heat uniformity.
6.4.2 Effects of Varying Sweeprate

The sweeprate of the Variable Frequency Microwave is the amount of time taken for a chosen bandwidth of frequencies to be launched into the cavity. For the MicroCure 2100 VFM, this can be varied from 0.1-60 seconds. As sweeprate time increases, the time spent launching each frequency increases and it is expected that the lifetime of each heating modes generated during the sweep will also increase. Experiments were undertaken at five different sweeprates and the resulting heat maps shown in Figure 6-2.

![Thermal images showing the effects of varying Sweeprate on the heat uniformity.](image)

**Figure 6-2:** Thermal images showing the effects of varying Sweeprate on the heat uniformity.
The first noticeable effect of VFM on the heat maps are their uniformity as compared to the Fixed-Frequency mode discussed in the previous section. The increase in sweep rate seems to have a detrimental effect on the heating uniformity as seen in the IR heatmaps. The results show that although all the sweep rates provide uniform heating over most of the sample area with the most uniform occurring at a sweep rate of 0.5 seconds, the sweep rate exacerbates the edge effect first observed in the fixed-frequency mode heating. This may be explained by the increase in residency time of each launched frequency in the cavity which increases with higher sweep rate times. For the MicroCure 2100, the bandwidth is divided into 4096 discrete frequencies and launched consecutively within the cavity within the chosen sweep rate time. Figure 6-3 illustrates the residence time spent at each frequency and the number of times the full bandwidth is cycled through during the 90 seconds processing time.

![Figure 6-3: Time spent at each frequency and Number of Cycles as a function of Sweep rate.](image)

Figure 6-3 illustrates how the residence time for each frequency increases linearly with the sweep rate. As sweep rate increases, the longer residency time for each frequency means that heating modes associated with the launched frequency exist longer in the cavity and thus contributing more to the edge effect resulting in increased edge
temperature. It seems that increasing the sweeprate to 1.0 second allows enough time for heating modes to affect the heating especially at the edges. Furthermore, the graph shows that for a sweeprate of 0.5 seconds, the full bandwidth would have been cycled through 180 times in the ninety second processing time, 3 times for a sweeprate of 30 seconds and 1.5 times for a sweeprate of 60 seconds and so on. It is predicted that cycling through the bandwidth a greater number of times will achieve better time-averaged uniformity as standing waves would not have a chance to establish and create hotspots during previous sweep cycles, as proven by the IR image of the sweeprate at 0.5 seconds which achieved the best heating uniformity. Thus, it seems logical to utilise faster sweeprates during processing but may be limited by the speed at which the microwave source can change frequencies. For the MicroCure 2100 VFM, this is limited to sweeprate of 0.1 seconds which may be due to limitations of the hardware used.

### 6.4.3 Effects of Varying Bandwidth

The VFM has the ability to sweep frequencies from 2.5-8.0GHz thus having a bandwidth of 5.5GHz. A central frequency was chosen and the bandwidth was changed accordingly. Table 6-1 summarises the conditions for which the IR reading were taken. The results are shown in Figure 6-4.

<table>
<thead>
<tr>
<th>Bandwidth</th>
<th>Lower Limit (GHz)</th>
<th>Central Frequency (GHz)</th>
<th>Upper Limit (GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100%</td>
<td>2.50</td>
<td>5.25</td>
<td>8.00</td>
</tr>
<tr>
<td>75%</td>
<td>3.188</td>
<td>5.25</td>
<td>7.313</td>
</tr>
<tr>
<td>50%</td>
<td>3.875</td>
<td>5.25</td>
<td>6.625</td>
</tr>
<tr>
<td>25%</td>
<td>4.563</td>
<td>5.25</td>
<td>5.938</td>
</tr>
</tbody>
</table>

Table 6-1: Experimental summary of the different Bandwidth used for heating experiments.

The results show that uniform heating was achieved for the range of bandwidth investigated with not much variation in maximum temperature achieved. The maximum temperature achieved ranged from 30.1-31.7°C which is only a difference of 1.6°C which is somewhat expected as the sample used is designed to absorb electromagnetic energy in the range of 0.7–18GHz and therefore not particularly sensitive to a change in bandwidth. As before, edge effects are again evident in the samples.
The significance of varying the bandwidth is to limit the number of frequencies being launched into the cavity which in turn limits the heating modes. A wider bandwidth is expected to increase the probability of heating uniformity. However, although the full effect of a change in bandwidth was not illustrated in this test, the MicroCure 2100 VFM’s ability to change bandwidth is advantageous when processing material which only absorbs at particular frequencies. The ability to target those frequencies will allow better use of energy and increase processing efficiency.

### 6.4.4 Effects of Varying Central Frequency

In order to determine the effect of Central Frequency, the entire bandwidth of the MicroCure 2100 VFM was divided into five segments each having a bandwidth of 1.1GHz. A central frequency was obtained at each segment and IR readings were taken.
A summary of the conditions of each test is shown in Table 6-2 and the results illustrated in Figure 6-5.

**Figure 6-5:** Thermal images showing the effects of varying Central Frequency on heat uniformity.
CHAPTER 6: EXPERIMENTAL RESULTS – EFFECTS OF VFM PARAMETERS ON HEATING UNIFORMITY

<table>
<thead>
<tr>
<th>Bandwidth Lower Limit (GHz)</th>
<th>Central Frequency (GHz)</th>
<th>Bandwidth Upper Limit (GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.50</td>
<td>3.05</td>
<td>3.60</td>
</tr>
<tr>
<td>3.60</td>
<td>4.15</td>
<td>4.70</td>
</tr>
<tr>
<td>4.70</td>
<td>5.25</td>
<td>5.80</td>
</tr>
<tr>
<td>5.80</td>
<td>6.35</td>
<td>6.90</td>
</tr>
<tr>
<td>6.90</td>
<td>7.45</td>
<td>8.00</td>
</tr>
</tbody>
</table>

Table 6-2: Experimental summary of the different Central Frequencies used for heating experiments.

The IR images show that there is very little difference in the heat uniformity achieved at different central frequencies apart from that taken at a central frequency of 7.45GHz. Results indicate that the average temperature rise generally increases with an increase in central frequency. At the minimum central frequency of 3.05GHz a temperature rise of 16°C was achieved while 22.7°C was achieved at the maximum central frequency of 7.45GHz. All of the IR images illustrate even heating within the main body of the samples except at the edges which again suffers from the edge overheating effect. Line scans which show temperature profile through the middle of the sample as shown in Figure 6-6, concurs with the IR images and demonstrates more clearly the uniformity of the heating which shows very small variations in temperature across the sample apart from the edges.

![Figure 6-6: Line scans showing temperature profile across the surface of the middle part of the sample.](image)
The temperature profile for central frequency of 7.45GHz also show that although the IR image illustrates an area of higher temperature in the middle of the sample, the temperature profile confirms that this is not a large difference in temperature and is still acceptable. The hotspot may also have been intensified visually by the scale used to illustrate the IR images. Johnson et al. [1994a] found that less bandwidth is required to achieve the same level of heating uniformity as the centre frequency is increased. Since the bandwidth was kept constant at 1.1GHz for each test, this may have led to the slight overheating of the sample at some areas as observed on the 7.45GHz sample. In retrospect, the bandwidth should have been changed accordingly as central frequency increased, so that the same number of frequencies were scanned for each case.

6.5 QUANTITATIVE ANALYSIS OF HEAT UNIFORMITY

A quantitative analysis of the results presented in this chapter was undertaken to determine the most influential factor affecting the heat uniformity in the MicroCure 2100 Variable Frequency Microwave and how sensitive it is to changes in these parameters. A search of the literature has found no measure to describe the heat uniformity apart from visual inspections. In order to establish a baseline for comparison, a value N, is introduced and was determined for each parameter and is defined as

\[ N = \frac{\Delta T}{T_{avg}} \]  

Equation 6 - 1

where \( \Delta T \) is the difference between the maximum and minimum temperature and \( T_{avg} \) is the average temperature within the sample. Essentially, the N-value that is introduced here is a measure of the heat uniformity in a sample. The N-value can be used to quickly compare any differences or improvements in heating uniformity between different processing conditions and is useful for a processing point of view. The smaller the N-value, the more uniform the heating. Initial analysis of the IR results is shown in Figure 6-7 and Table 6-4. Note that Figure 6-7 is plotted purely as a visual comparison of the N-
values calculated at the different heating conditions. The experimental conditions corresponding to each value is summarised in Table 6-3.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Experimental Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fixed Frequency</td>
<td>2.5GHz 4.0GHz 5.25GHz 6.0GHz 8.0GHz –</td>
</tr>
<tr>
<td>Sweeprate</td>
<td>0.5sec 1.0sec 5.0sec 10sec 30sec 60sec</td>
</tr>
<tr>
<td>Bandwidth</td>
<td>25% 50% 75% 100% – –</td>
</tr>
<tr>
<td>Central Frequency</td>
<td>3.05GHz 4.15GHz 5.25GHz 6.35GHz 7.45GHz –</td>
</tr>
</tbody>
</table>

Table 6-3: Experimental conditions corresponding to each value in Figure 6-7 and 6-8.

![Figure 6-7: N-values calculated at different heating conditions.](image)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>N-Value Range</th>
<th>Average N-Value</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fixed Frequency</td>
<td>0.29 – 0.76</td>
<td>0.52</td>
<td>0.17</td>
</tr>
<tr>
<td>Sweeprate</td>
<td>0.48 – 0.59</td>
<td>0.54</td>
<td>0.05</td>
</tr>
<tr>
<td>Bandwidth</td>
<td>0.53 – 0.60</td>
<td>0.57</td>
<td>0.03</td>
</tr>
<tr>
<td>Central Frequency</td>
<td>0.47 – 0.66</td>
<td>0.55</td>
<td>0.07</td>
</tr>
</tbody>
</table>

Table 6-4: Summary of N-values calculated for different parameters.

The results indicate that the main parameters of the VFM (sweeprate, bandwidth and central frequency) do not show much variation in N-Values, which suggest that the heat
uniformity is not particularly sensitive to changes in these parameters. Whilst the large variations for the fixed-frequency parameter indicate that it has the largest effect on the heating uniformity in the VFM. This is expected as the IR images of the fixed-frequency test produced the worst heating uniformity of the entire test. Although the values obtained gives an insight to the sensitivity of the heat uniformity in changes in the parameters, the values acquired maybe unrealistic as the edge-effects were not taken into account and may have increased the actual value of N due to the higher temperatures at the edges. A more practical approach was taken by excluding the edge effects by only measuring temperature for 80% of the sample surface and calculating the N values. A value of 80% was chosen as this eliminated most of the obvious edge effect for most of the samples. Results are shown in Figure 6-8 and Table 6-5.

Figure 6-8: N-values calculated at different heating conditions for 80% sample surface area.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>N-Value Range</th>
<th>Average N-Value</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fixed Frequency</td>
<td>0.16 – 0.39</td>
<td>0.28</td>
<td>0.10</td>
</tr>
<tr>
<td>Sweeprate</td>
<td>0.16 – 0.20</td>
<td>0.18</td>
<td>0.01</td>
</tr>
<tr>
<td>Bandwidth</td>
<td>0.16 – 0.19</td>
<td>0.18</td>
<td>0.01</td>
</tr>
<tr>
<td>Central Frequency</td>
<td>0.18 – 0.28</td>
<td>0.22</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 6-5: Summary of N-values calculated for 80% of the sample surface area.
The new results still show that processing at fixed-frequencies is the biggest factor that affects heat uniformity in the VFM with N-Values ranging from 0.16-0.39. As frequency increases, the N-value decreases suggesting an improvement in heating uniformity, which is reasonable as the wavelength is decreasing which means hotspots will be closer in proximity as discussed previously. Of the three main VFM parameters, central frequency is identified to be the most influential in terms of achieving heat uniformity as indicated by the variations in N-values at different central frequencies. The N-values obtained for sweeprate and bandwidth is very similar and does not vary much irrespective of the parameter value chosen. Despite the variations in N-values for central frequency, sweeprate and bandwidth, these can be considered small compared to changes observed with fixed-frequency, which means that heat uniformity will not vary significantly if the parameters are changed. Thus, making VFM a robust and versatile method for materials processing.

As a measure of heat-uniformity, the N-values calculated shows lower values for the VFM parameters than the Fixed Frequency and is therefore indicative of the degree of uniformity achieved in a sample. Taking the worst heating case scenario which occurs at a fixed frequency of 2.5GHz, and comparing this to the VFM conditions, the N-values suggest that the improvement in heating uniformity is up to two and a half times better. This is a significant improvement for materials processing application in which heating uniformity is crucial. Interestingly, the results also show that uniform heating is achieved at the fixed frequency of 6.0GHz with an N-value in the same region as the VFM parameters. This however, is quite misleading as the IR images in fact show otherwise. It can be argued that if the difference in temperature extremities i.e. $T_{\text{max}} - T_{\text{min}}$, achieved is not too great, which is the case at 6.0GHz, low N-values can be achieved. From earlier discussions about the cavity characterisation of the VFM cavity in Section 4.2.7.1 however, it was found that the MicroCure 2100 cavity favours higher frequencies as indicated by the lower reflectance of microwave energy, which probably explains the decrease in N-value as the frequency is increased. Perhaps at higher frequencies, more resonant mode can exist in the cavity and 6.0GHz is the sweetspot. Thus, it can be
concluded that cavity dimensions also play a part in the N-values achieved. Nevertheless, except for instances where the frequency is favoured by the cavity, the N-value is a quick and simple tool to quantify heat uniformity.

6.5.1 Feature Size

The analysis of the N-values above enables other important information about the heating to be gathered. By investigating how the N-Values decay as a function of time it is possible to determine the feature size or hot or cold spot size at different heating conditions. The measured decay can be correlated with the time for thermal diffusion which is related to the uniformity of the heat established in the sample after microwave irradiation and can be used to determine the spot size. It is envisaged that smaller spot sizes are more probable to result in uniform heating as they would be closer in proximity and will require shorter time for the temperature to even out. The spot sizes does not however indicate their temperature or how or if they overlap during processing.

The relationship between the spot size and thermal diffusion can be given by

\[
\text{spot size} \propto \sqrt{\gamma_\text{mat} \tau}
\]

where \(\gamma_\text{mat}\) is the thermal diffusivity of the material being processed and, \(\tau\), is the decay time which is the reciprocal of the decay rate. The decay rate was measured by obtaining N-values for each parameter at intervals of 30 seconds for 120 seconds after microwave irradiation. The decay rate is taken as the exponential factor in a logarithmic line of best fit. Typical decay curves for each parameter is shown in Figure 6-9 and a summary of the feature size obtained is shown in Table 6-6.
CHAPTER 6: EXPERIMENTAL RESULTS – EFFECTS OF VFM PARAMETERS ON HEATING UNIFORMITY

Figure 6-9: Typical N-value decay curves for each parameter.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Longest (mm)</th>
<th>Shortest (mm)</th>
<th>Mean (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fixed Frequency</td>
<td>19.6</td>
<td>14.1</td>
<td>16.9</td>
</tr>
<tr>
<td>Sweep Rate</td>
<td>20.9</td>
<td>16.4</td>
<td>18.3</td>
</tr>
<tr>
<td>Bandwidth</td>
<td>19.2</td>
<td>17.1</td>
<td>18.3</td>
</tr>
<tr>
<td>Central Frequency</td>
<td>17.4</td>
<td>15.6</td>
<td>17.7</td>
</tr>
</tbody>
</table>

The result shows that different spot sizes can exist in the VFM cavity ranging from approximately 14mm to 21mm. Although it was illustrated previously through heat-mapping that fixed frequency processing produces the worst heating uniformity of all the parameters, the spot sizes calculated in Table 6-6 do not show a significant difference between each parameters despite there being a considerable difference in heat uniformity. This could be explained by Figure 6-9 where the N-values represent the “spot amplitude” which is a measure of the intensity of the hot or cold spots. Typically, the Fixed Frequency parameters produce spot amplitudes of about a third more to double that of the
VFM parameters which means that the spots are at a higher intensity and would therefore take longer to even out, hence exhibiting highly visible hot and cold spots. Furthermore, although the spot sizes produced by the VFM parameters are roughly similar to the Fixed Frequency parameter, hot and cold spots are not visible during VFM processing probably due to a great number of overlapping spots of different spot amplitudes which decayed quicker to even out the temperature. This is expected, as the main principle of the VFM technique is the overlapping of modes to achieve time-averaged uniformity.

6.6 SUMMARY

A thermal imaging camera was utilised to investigate the parameters that control heat uniformity in the VFM technique. IR images of an irradiated sample were taken at different conditions and the results analysed. Results confirm how the heat uniformity within a sample greatly suffers with the use of fixed-frequencies as compared to VFM heating. Quantitative analysis of three main VFM parameters – Sweeprate, Bandwidth and Central Frequency – indicate that although some variation in heat uniformity was observed when changing these parameters, these variations are only slight which implies that the VFM is quite insensitive to changes in the parameters making it quite a robust system.
CHAPTER 7

RESULTS -

OPTIMISATION OF SWEEP RATE
IN VARIABLE FREQUENCY MICROWAVE

7.1 OVERVIEW

This chapter presents work focused on the sweep-rate regime used in VFM and the effect of varying this parameter. A “recipe” for a non-linear sweep-rate regime is introduced and compared with the current linear-sweep regime and conventional fixed-frequency processing in terms of heating uniformity. Theoretical modelling of the linear and non-linear sweep regimes is presented and is experimentally verified by heat-mapping of a multimode cavity with the aid of thermally sensitive material to quantify improvements in the uniformity of heating.

7.2 INTRODUCTION

There are two ways to optimise the VFM technique. The first is to optimise the highly reflective cavity by physically changing the dimensions or making inserts that would enable different electromagnetic modes to occur during heating. This is difficult to achieve as a cavity would always only work best at certain frequencies or a small range of frequencies. Having a non-static wall that is moveable may solve the problem as the cavity can change dimensions to suit each frequency. The problem though is the speed at which it has to change to keep up with the changing frequency of the VFM technique which could be as fast as milliseconds. The second optimisation option is through the optimisation of the variable parameters in the VFM – Bandwidth, Central Frequency and Sweep Rate.
As observed in Chapter 6, the widest bandwidths produced the best heating uniformity. Making the operating frequency range of the VFM wider will most certainly improve heating uniformity but as a wider bandwidth will require a new Travelling Wave Tube source which is very expensive, optimisation of the bandwidth is not practical. Similarly, optimisation of the Central Frequency is also not practical as it was shown in the previous chapter that varying central frequency actually limits the bandwidth that is useable. The most practical optimisation point is therefore the Sweep Rate parameter. Varying the sweep-rate requires no further capital investment whilst still enabling the use of the full range of the VFM bandwidth.

In Chapter 6, it was shown that hotspots occur more prevalently at lower frequencies. These hotspots could be avoided by simply using higher frequencies however, at higher frequencies there is low penetration of microwave radiation therefore low frequencies are still required to get greater penetration and heat the bulk of the material. The current linear sweep means that each frequency is introduced into the cavity for the same amount of period therefore allowing hotspots to occur. Thus, by varying the amount of time each frequency is introduced in the cavity hotspots maybe avoided whilst still achieving heating uniformity in the material. By analysing the underlying equations for energy deposition in a multimode cavity, the sweeping regime can be examined and further optimised.

### 7.3 ANALYTICAL MODELLING OF ENERGY DEPOSITION

The idea behind the Variable Frequency Microwave technique is that by sweeping through a bandwidth with consecutive frequencies will result in multiple hotspots within the microwave cavity that will lead to a time averaged heating. In modelling the VFM, a number of assumptions have to be made:
**Assumption 1: Resonant modes only**

Only the resonant frequencies of the microwave cavity will be considered. At resonant frequencies the electric field is much stronger inside the cavity than at non-resonant frequencies. Since the amount of heat absorbed by the material in the cavity depends on the electric field squared, this is a reasonable assumption.

**Assumption 2: Equipartition of Energy**

At any given frequency, all the resonant modes at that frequency share equally the power absorbed within the cavity. At any single frequency all the modes resonating at that frequency and within the bandwidth will be excited simultaneously with equal energy.

Using these two assumptions and an argument similar to that used in the derivation of Wien’s law, a sweep rate that produces the most even heating can be derived.

### 7.3.1 Field Equations

A cavity is defined as a volume enclosed by a conducting wall. Depending on their dimensions as compared to the wavelength of frequencies, cavities are said to be resonant or oversized, respectively. The electromagnetic energy trapped in a cavity is reflected by its walls and takes the form of stationary waves. For a rectangular cavity, the mode equation for the resonant frequencies is given by,

\[
f_{\text{nm}l} = c \left[ \left( \frac{l}{2d} \right)^2 + \left( \frac{m}{2b} \right)^2 + \left( \frac{n}{2a} \right)^2 \right]^{\frac{1}{2}}
\]

**Equation 7 - 1**

where \(f_{\text{nm}l}\) is the TE\(_{nm}l\) or TM\(_{nm}l\) mode’s resonant frequency; \(c\) is the speed of light; \(n, m, l,\) are the number of half-sinusoid variations in the standing wave pattern along the \(x, y\) and \(z\)-axes; and \(a, b\) and \(d\) are the dimensions of the cavity in the \(x, y\) and \(z\) directions. This equation describes the limitation on the possible frequencies of the electromagnetic radiation contained in the cavity.
The Electric field \( E \) at the resonances is given by:

\[
E = \sum_{l,m,n} A_{l,m,n} \sin(k_n x) \sin(k_m y) \sin(k_l z)
\]

Equation 7 - 2

where \( k_n = \frac{l\pi}{a}, k_m = \frac{m\pi}{b}, k_l = \frac{n\pi}{d} \) and \( A_{l,m,n} \) is the strength of the resonance, and

\[
k^2 = k_t^2 + k_m^2 + k_n^2 = \left( \frac{2\pi}{c} \right)^2 f^2
\]

Equation 7 - 3

From this, the total heat deposited at a particular wave number, \( k \), (corresponding to frequency, \( f \)) into the cavity can be calculated from:

\[
Q_k \propto \varepsilon E^2
\]

Equation 7 - 4

where \( \varepsilon \) is the imaginary part of the dielectric constant. Therefore,

\[
Q_k \propto \varepsilon \left( \sum_{l,m,n} A_{l,m,n} \sin(k_n x) \sin(k_m y) \sin(k_l z) \right)^2
\]

Equation 7 - 5

Since all the resonant modes are being excited at the same time in the frequency range from \( f \) to \( f + df \), where \( df \) is the bandwidth, then, using assumption 2, all the \( A_{l,m,n} \) constants are the same at this frequency.

\[
A_{l,m,n} = A_k
\]

Equation 7 - 6

Thus Equation 7-5 can be written as,

\[
Q_k \propto \varepsilon A_k^2 \left( \sum_{l,m,n} \sin(k_n x) \sin(k_m y) \sin(k_l z) \right)^2
\]

Equation 7 - 7
The frequency is swept through a frequency range at a rate to be determined. Therefore the total heat absorbed is given by:

\[
Q = \sum_{k_{\text{min}} < k < k_{\text{max}}} Q_k \tag{7-8}
\]

Thus the total heat absorbed over the time of the variable frequency microwave irradiation is given by:

\[
Q = C \sum_{k_{\text{min}} < k < k_{\text{max}}} \varepsilon A_k^2 \left( \sum_{l,m,n: \text{for } k=\text{constant}} \sin(k_x x) \sin(k_y y) \sin(k_z z) \right)^2 \tag{7-9}
\]

Or more compactly,

\[
Q = \frac{C}{2} \sum_{k_{\text{min}} < k < k_{\text{max}}} \varepsilon A_k^2 \left( \sum_{l,m,n: \text{for } k=\text{constant}} [1 + F_k(x,y,z)] \right)^2 \tag{7-10}
\]

where \(F_k(x,y,z)\) is a function that takes values between –1 and +1 and whose average value is zero, over the volume of the cavity.

Equation 7-10 is instructive in that, if the \(A_k^2\) coefficients are known, then an estimation of the non-uniformity of the heat absorbed may be made. If all the coefficients \(A_k^2\) are constant and there are \(K\) different discrete frequencies (of bandwidth \(dK\)) over which the variable frequency microwave is swept, then:

\[
Q = \frac{C}{2} \varepsilon A^2 \left( K + \sum_{\text{for all } k} F_k(x,y,z)^2 \right) \tag{7-11}
\]
where it is expected that $\sum_{k} F_k(x,y,z)$ will lie between $-1$ and $+1$. This would imply that constant coefficients $A_k$ are going to yield the heat absorption with the minimum non-uniformity.

### 7.3.2 Density of States

If we consider a lattice having points at coordinate $(n,m,l)$, the total number of points corresponds to the number of allowed frequencies which is given by Equation 7-1. For a given frequency interval, the number of frequencies allowed, lie between two concentric shells as shown in Figure 7-1.

![Figure 7-1: Graphical representation of Equation 7-1.](image)

The concentric circles represent the boundaries allowed in the system, the minimum ($f_{\text{min}}$) and maximum ($f_{\text{max}}$) frequencies.

The density of resonant modes per unit volume in $k$ space is unity. If the microwave power absorbed at a given frequency is $P(f)$, then the value of $A_k^2$ can be written as:
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\[ A_k^2 = \frac{P(f)}{N(k)} \delta t \]  \hspace{1cm} \text{Equation 7 - 12}

and

\[ N(k) = \frac{4\pi}{8} k^2 dk \]  \hspace{1cm} \text{Equation 7 - 13}

where \( N(k) \) is the number of resonant states lying between \( k \) and \( k + dk \).

Therefore we may express the coefficients \( A_k^2 \) in terms of the frequency sweep rate \( \frac{df}{dt} \).

\[ A_k^2 = \frac{c^3 P(f)}{4\pi^2 f^2} \frac{dt}{df} \]  \hspace{1cm} \text{Equation 7 - 14}

where equation 7-1 gives the relationship between \( l,m,n \) and frequency.

Thus, if the frequency sweep rate is constant i.e. linear sweep, the \( A_k^2 \) terms are weighted by the inverse of the frequency:

\[ A_k^2 \propto \frac{P(f)}{f^2} \]  \hspace{1cm} \text{Equation 7 - 15}

If on the other hand we would like the energy to be shared equally between all the resonant modes, that is all the \( A^2 \) terms are equal, then the frequency sweep rate is given by:

\[ \frac{df}{dt} \propto \frac{P(f)}{f^2} \]  \hspace{1cm} \text{Equation 7 - 16}
In doing so, longer time is spent at high frequencies and a shorter time is spent in lower frequencies and the sweep rate can be weighted depending on the frequency (weighted sweep). This is the expected “best” frequency sweep algorithm to obtain the most even heat absorption.

The result above is not changed if assumption 2 is slightly altered. It may be that not all the modes of the cavity at a given frequency are excited. For instance, the cavity may be excited so that only symmetric modes are excited. The density of states argument remains, it is just that a constant fraction of the possible modes are excited so that $4\pi/8$ factor is reduced. The weighting, however, remains the same preserving the form of Equation 7-16.

### 7.4 RESULTS

#### 7.4.1 Analytical Modelling

A two-dimensional model of a variable frequency microwave with a cavity cross-section of 365mm x 482mm and a bandwidth of 2.5GHz-8.0GHz was modelled using the assumptions discussed above. The situations of 1) linear sweep and 2) weighted sweep were modelled using MATLAB with results shown in Figures 7-2 and 7-3 respectively. A sample of the MATLAB code developed is shown in Appendix B.

The contour maps shown in the following figures is the power distribution or field distribution and is representative of the temperature profile that can be achieved within the cavity.
Case 1: Linear Sweep

Figure 7-2: a) Result of 2D linear sweep   b) Contour map of linear sweep.
Case 2: Weighted Sweep

Figure 7-3: a) Result of 2D weighted sweep  b) Contour map of weighted sweep.
As expected the results of the model show that a more uniform power distribution can be achieved when the sweep rate was weighted (Case 2). The weighted sweep produced a more uniform profile compared to linearly sweeping the frequencies which produced tiger-stripes like profile parallel to the longer axis as seen in Figure 7-2a and Figure 7-2b.

A cross-section in the y-direction of both Figure 7-2a and Figure 7-3a were used and normalised to obtain the typical temperature profile obtained for an average processing temperature of 100°C as shown in Figure 7-4. It should be noted that the edge effects (Gibbs Phenomena) in both process were not taken into account during normalisation.

It can be seen that the profile of the weighted sweep case is not entirely flat, thus, temperature fluctuations still exist. The peaky nature of this profile suggests that tiger stripes also exist although not as apparent as in the linear sweep case. For a target processing temperature of 100°C, the linear sweep has a peak to trough difference of about 4°C (standard deviation of 2.1°C) whilst the weighted sweep has a peak to trough difference of about 2°C (standard deviation of 0.89°C). Although a halving of the temperature non-uniformity from a linear sweep procedure may not seem significant, if the processing is being carried out to cure the material, then the cure rate will probably
follow an Arhenius law. Thus variations of a few degrees could correspond to large differences in the properties of the cured material. Also in some materials the curing reaction is exothermic, thus a positive feedback condition could be exacerbated by a slight uneven heat distribution. The temperature unevenness increases at higher processing temperatures and may be significant if processing materials, which require fairly accurate uniform heating, or when processing small substrates where material positioning will become important. Furthermore, since the peaks in the linear sweep are further apart, lateral heat dissipation to even out the heat in the material will take longer than the weighted sweep.

7.5 EXPERIMENTAL RESULTS

A commercial Variable Frequency Microwave (MicroCure 2100) from Lambda Technologies operating at a bandwidth of 2.5-8.0GHz that can be operated at a fixed or variable frequency mode, and a heat mapping technique as used by Steyn-Ross and Riddell [1990] as described in literature, were utilised to obtain a qualitative form of the heat distribution in the VFM cavity. The heat-mapping technique made use of the colour properties of cobalt chloride (CoCl₂) soaked paper that is pink in wet conditions and blue when dry. The heat-sensitive paper of size 210 mm x 297mm was supported on a platform and placed inside the multimode cavity of the VFM, which has dimensions of 365 mm x 355 mm x 482mm as shown in Figure 7-5. Heat maps were taken at a fixed-frequency of 2.45GHz as well as variable frequency to show and contrast the heating advantage of VFM over conventional fixed-frequency methods. A ‘recipe’ was programmed into the VFM for each case and a power was chosen to obtain drying in sufficient time. The resulting heat maps were converted for clarity and for further analysis and are shown in Figure 7-6 for fixed-frequency and in Figure 7-7a and Figure 7-7b for VFM technique using a linear and a weighted sweep regime respectively. In addition, the heat-maps where processed with a commercial image processing software and the wet and dry areas calculated which are indicative of the heating uniformity as shown in Table 7-1.
CHAPTER 7: RESULTS – OPTIMISATION OF SWEEP-RATE IN VARIABLE FREQUENCY MICROWAVE

**Figure 7-5:** Experimental setup for heat-mapping of VFM cavity.

**Figure 7-6:** Heat map of VFM cavity using conventional fixed-frequency method at 2.45GHz.

**Figure 7-7:** Heat map of commercial using VFM a) Linear Sweep b) Weighted Sweep.
Results highlight the inherent difficulty with using fixed-frequency in multimode cavities for uniform heating as illustrated by the dark regions in Figure 7-6, which indicates concentrated electric fields. In comparison to Figure 7-7a and Figure 7-7b, a large improvement in heat uniformity is achieved when VFM processing is utilised. Image processing results show that less than a quarter of the heat-mapping area was dried compared to >80% for VFM with a linear sweep regime and >90% for VFM with a weighted sweep regime. While the difference in the dry area between linear sweep and weighted sweep is small (~10%), the improvement in heat uniformity using a weighted sweep has halved the wet area. Although the halving of the wet area is comparable in value to the halving of the peak to trough temperature distribution predicted by the computer model, this cannot necessarily be interpreted as “good agreement”. The local vapour pressure of water at any given position on the paper will drive the drying process. This is a strong function of temperature on the paper (Clausius – Clapeyron equation) and illustrates the point that the result of microwave processing depends on the processes taking place in the material being treated. The experiment confirms that the weighted sweep has a more uniform heating distribution.

There has been very little VFM optimisation presented in literature. Mostly all optimisation reported concerns the optimisation of an actual process using the VFM method instead of the actual VFM method which is not always transferable or adaptable to other processes. The study undertaken in this chapter shows an optimisation of the actual VFM method and can be applied in any processing situation. A simple software
modification of the current VFM software is all that is required to implement this process.

7.6 SUMMARY

A two-dimensional computer model of the Variable Frequency Microwave technique was investigated and it was found that a more even temperature distribution could be achieved by using a sweep rate that varies as the inverse of the frequency squared (weighted-sweep). Results show that although a linear-sweep rate produced uniform heating over a large area, temperature fluctuations in the order of 4% of the temperature rise still exist. The use of a weighted sweep further improved heating uniformity by reducing these temperature variations by a factor of two. An experimental comparison through heat-mapping of the different microwave heating procedures confirmed the model predictions for an area of size 210mm x 297mm and also highlighted that a much greater heating uniformity can be achieved using the VFM method than conventional fixed-frequency method which in terms of materials processing, translates to better processing and hence better product quality.
8.1 OVERVIEW

This chapter introduces the photosensitive polymer SU8 and investigates the parameters to enable its processing with microwave energy. Important material properties such as its dielectric constant and the dielectric constants of its constituents where analysed to determine its compatibility with microwave energy. Furthermore, its behaviour under microwave field are also investigated and reported. Microwave parameters such as operating frequency, power, and other relevant operating microwave parameters were also investigated and optimised to determine the most suitable microwave parameters to be utilized for the SU8 systems processing.

8.2 SU8 NEGATIVE TONE PHOTORESISTS

SU8 is a negative tone, epoxy-based, near-UV (350-400nm) photoresist widely used in MEMS production for many years. SU8 is prepared from commercially available components by dissolving an EPON SU8 (shell chemical) resin in organic solvents such as Gamma Butyrolactone (GBL) or Cyclopentanone. Several formulations of SU8 exist for different purposes. The type of solvent and its quantity determines the possible resist thickness which could vary from <1µm to >200µm with single spin coat processes. SU8 has the highest epoxide functionality per molecule commercially available (Figure 8-1) [LaBianca et al., 1993; Eyre et al., 1998]. The functionality is defined as the number of reactive epoxy groups per molecule and is the result of epoxidation of novolac resin
formed from the condensation polymerisation of Bisphenol-A and formaldehyde. The resist is combined with an appropriate photoacid generator (PAG), in this case a triaryl salt ($\text{SbF}_6$) which facilitates crosslinking after exposure to UV. The final result is a highly crosslinked structure with excellent physical properties as shown in Figure 8-2. Table 8.1 shows a typical formulation of SU8.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>CAS #</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclopentanone</td>
<td>120-92-3</td>
<td>23-85%</td>
</tr>
<tr>
<td>Mixed Triarylsulfonium / Hexafluoroantimonate Salts</td>
<td>89452-37-9 / 71449-78-0</td>
<td>0.5-5%</td>
</tr>
<tr>
<td>Propylene Carbonate</td>
<td>108-32-7</td>
<td>10-75%</td>
</tr>
<tr>
<td>Epoxy Resin</td>
<td>28906-96-9</td>
<td>10-75%</td>
</tr>
</tbody>
</table>

Table 8-1: Typical formulation of SU8 2100 as obtained from MicroChem Inc. MSDS

![SU8 Monomer](image)

Molecular Weight =1397.73  
Exact Mass =1396  
Molecular Formula = C$_{87}$H$_{96}$O$_{16}$  
Molecular Composition =C 74.76%  H 6.92%  O 18.31%

Figure 8-1: SU8 monomer showing eight epoxide functionality per molecule.
8.2.1 Chemical Reaction

Polymerisation of SU8 is based on chemical amplification. A single photo event, also known as protolysis, initiates a cascade of subsequent chemical reactions that induce the crosslinking in the SU8 resin. Cationic photoacid generation is responsible for initiating image formation. After exposure to UV, a photoacid (which is a strong Lewis acid) is generated and acts as the catalyst in the crosslinking which is thermally driven during Post Exposure Bake (PEB). The epoxy groups on the SU8 monomers undergo cationic ring opening initiated by the photoacid. The photoacid is not consumed in the reaction and can initiate several chains. The crosslinking reaction produces a rigid molecular structure where an epoxy monomer is connected to an average of seven other monomers. The reaction is shown in Figure 8-3.
Lewis Acid Photogeneration

\[ \text{Ar}^+\text{SbF}_6^- \xrightarrow{\text{UV}} \text{H}^+\text{SbF}_6^- \]

Crosslinking

Figure 8-3: Chemical reaction showing Lewis Acid photogeneration after UV exposure and the subsequent crosslinking of the SU8 monomer.

8.2.2 SU8 Physical Properties

SU8 and its properties have been extensively investigated and the most interesting mechanical, physical and electromagnetic properties are summarized in Table 8-2.
### Property | Value | Source
--- | --- | ---
Young’s Modulus ($E$) | 4.4 GPa (Postbake @ 95°C) | Sotec Micro., 1998
| 4.02 GPa (Postbake @ 95°C) | Lorenz, 1998c
Poisson coefficient | 0.22 | Sotec
Film Stress | 16-19 MPa (Postbake @ 95°C) | Lorenz, 1998b
Coefficient of Thermal Expansion (CTE) | 50ppm/K | Sotec
| 52.0 +/- 5.1 ppm/K | Lorenz et al, 1998
Glass Temperature ($T_g$) | ~50°C (unexposed film) | LaBianca et al, 1993
| >200°C (fully crosslinked – hardbaked) | LaBianca et al, 1993
Degradation Temperature ($T_d$) | ~380°C | LaBianca et al, 1993
Polymer Shrinkage | 7.5% (Postbaked @ 95°C) | Guerin et al, 1997
Thermal Conductivity | 0.2 Wm/K (general value) | Guerin et al, 1997
Absorption coefficient ($\alpha_b$) | ~2 cm$^{-1}$ @ 100 GHz | Arscott et al, 1999
| ~40 cm$^{-1}$ @ 1.6 THz | Arscott et al, 1999
Loss tangent ($\tan \delta$) | 0.08 @ 100 GHz | Lucyszyn, 2001
| 0.14 @ 1 THz | Lucyszyn, 2001
Relative dielectric constant ($\varepsilon_r$) | 4 @ 10 MHz (postbaked at 100°C, may be valid between 20-40 GHz) | Thorpe et al, 1998
| 4.5 @ 10 MHz | Thorpe et al, 1998
| 4.2 @ 10 GHz | Thorpe et al, 1998
| 3 @ 10 MHz (postbaked at 95°C) | Sotec Micro

Table 8-2: Common SU8 properties as found in literature.

### 8.2.3 SU8 Processing

SU8 is processed as a negative photoresist with an additional post-exposure step where crosslinking of the monomers occurs. Processing steps include SU8 deposition, softbake, exposure, Post-exposure bake, development and an optional hardbake. Figure 8-4 shows a schematic of the processing steps required for SU8. Multilayer structures are possible by depositing additional layers of SU8 after post-exposure bake or development steps. Literature describes a wide variation in values for processing parameters which is due to the many different SU8 formulations [LaBianca et al., 1993, Despont et al., 1997]. For this study the recommended processing parameters from the SU8 manufacturer MicroChem Inc. were used.
There are three main applications of VFM in the processing of SU8. The first is during the Softbake (SB), the second is during the Post Exposure Bake (PEB) and finally the Hard Bake (HB) step.

### 8.2.3.1 Deposition (Spin-Coating)

The simplest and most convenient method of depositing SU8 onto a substrate is by Spin-coating. Spin coating of the SU8 on substrate is required to obtain an even thickness on the substrate. The thickness is determined by the viscosity of the resists used and by the spin coating parameters of speed and time.

#### 8.2.3.1.1 Silicon Substrate

The substrates used in this work were boron doped Silicon (Si <100>) wafers from Motorola. The wafers had a resistivity of 7.0-17.0 ohm/m and thickness of 14-16mm.
8.2.3.2 Soft Bake (SB)

After the resist has been applied to the substrate a softbake step is required to evaporate the solvent and densify the film. It is recommended that ramping or stepping the SB temperature is best [MicroChem, 2004]. Softbaking times depend on the thickness of the film. For this thesis contact hotplate as well as Microwaves were used to softbake the samples at a temperature of 95°C. Since the Softbake temperature is greater than the glass transition temperature, reflow of the resists occurs during the first few minutes thus, it is critical that the sample is placed levelled in order to produce smooth and uniformly coated substrates.

A two-step softbaking process as seen in Figure 8-5 is recommended and is commonly used in SU8 processing. The lower initial bake temperature of 65°C allows the solvent to evaporate out of the film at a more controlled rate thus resulting in better coating fidelity, reduced edge-beading and better adhesion. Hold temperatures are achieved using a ramp rate of 250°C/hr. A ramp down rate of 70°C/hr is used during cool down to minimize the internal pressure build up within the thin film.

![Figure 8-5: Recommended two-step Softbaking process for SU8.](image-url)
8.2.3.3 UV Exposure

SU8 is sensitive in the near-UV region (365nm) and exposure is required to generate the Lewis acid from the Photo Acid Generator (PAG) to induce crosslinking of the SU8 photoresists. The exposure is done using a broadband mask aligner with soft contact mode. The exposure time is calculated using the intensity value of the 365nm spectrum line of the mask aligner. The exposure dose required depends on the layer thickness.

8.2.3.4 Post Exposure Bake (PEB)

Following exposure, a Post-exposure bake is performed to selectively crosslink the exposed portions of the film. During this step, the exposed area of SU8 is polymerised through a cationic photoamplification mechanism. This bake is necessary because very little reaction can take place in the solid state where molecular motion is effectively frozen and has to be carried out at temperatures greater than $T_g$ in order to be effective. As crosslinking occurs, the resist film properties will change in several ways. Firstly, some shrinking will occur due to densification during crosslinking formation and outgassing of the solvent. The glass transition temperature will rise as the film becomes increasingly crosslinked. As crosslinking proceeds and the network gradually approaches completion, the crosslinking reaction will slow down and eventually stop. Therefore, the final $T_g$ of the material is dependent on the PEB temperature. Typically, a PEB temperature of 95°C is used for this step. A stepwise temperature heat-up and cool-down regime as shown in Figure 8-6 is recommended to minimize stress, wafer bowing and resist cracking.
8.2.3.5 Development

Photoresists development is done by means of the immersion technique using MicroChem SU8 developer Propylene Glycol Methyl Ether Acetate (PGMA) at room temperature. Each sample is immersed in the developer for several minutes depending on the layer thickness. Manual agitation of the Developer can be performed to speed up development. In some cases mechanical agitation can be used. Following development, each sample should be rinsed briefly with isopropyl alcohol (IPA) then dried with a gentle stream of nitrogen.

8.2.3.6 Hard Bake (HB)

After development, another baking step called the Hardbake can be undertaken in an oven at 200°C which will lead to a very hard material as crosslinking can continue to total completion. Hardbake is an optional step as the high temperature used can lead to significant stresses in the structures due to the difference between the linear thermal expansion coefficients (CTE) of the substrate and the resists which can lead to high internal stresses and generation of cracks. For this thesis, the hard bake was not done due to this reason.
8.3 VFM PARAMETER DETERMINATION AND OPTIMISATION

Although the VFM software has experienced a huge advance in terms of control from previous versions, where it lacked facilities such as temperature ramp-up and ramp-down, it is still necessary to investigate how the materials to be processed behave under different processing conditions i.e. power and frequency, and not blindly use random parameters that may have unexpected outcomes.

Different techniques were utilized to determine the material properties important for MW heating and were used to determine the optimum parameters to be used in VFM processing. The results and problems encountered and how they were overcome are discussed in the following sections.

8.3.1 Dielectric Properties

Dielectric properties of SU8-2100, its solvent cyclopentanone and the Si<100> substrate were measured using the open probe method. Initially, the dielectric constants ($\varepsilon'$) and dielectric loss ($\varepsilon''$) of known materials were tested to determine the accuracy and reliability of the results obtained. The results water and acetone were measured at 2.45GHz and compared with those found in literature as shown in Table 8-3.

<table>
<thead>
<tr>
<th></th>
<th>Measured</th>
<th>Literature</th>
<th>% Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water $\varepsilon'$</td>
<td>79.9</td>
<td>80</td>
<td>0.125%</td>
</tr>
<tr>
<td>Water $\varepsilon''$</td>
<td>5.63</td>
<td>6.1</td>
<td>7.7%</td>
</tr>
<tr>
<td>Acetone $\varepsilon'$</td>
<td>22.1</td>
<td>20.7</td>
<td>6.8%</td>
</tr>
<tr>
<td>Acetone $\varepsilon''$</td>
<td>1.1</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 8-3: Comparison of measured and literature dielectric properties of Water and Acetone at 25°C.

Results show good agreement with the literature values to within <10% which suggest the dielectric measurement setup is correct and outputs can be trusted.
Dielectric Constants were measured for SU8-2100 and its solvent cyclopentanone at range of frequencies ranging from 2.45GHz to 8GHz and results shown in Figures 8-7 and 8-8 respectively.

Figure 8-7: Dielectric properties of liquid SU8-2100 using the Dielectric Probe Method.

Figure 8-8: Dielectric properties of Cyclopentanone solvent using the Dielectric Probe Method.
The dielectric properties above only show the dielectric properties of SU8 at a constant temperature of 25°C. Since the temperature will be constantly changing during processing, the dielectric properties are expected to constantly change. Thus, dielectric properties were measured at different temperatures ranging from 18-100°C at two different frequencies to determine how they change with temperature and frequency. An enclosed heating apparatus especially designed for liquids was used as a heat source whilst dielectric measurements were being taken. Figure 8-9 illustrates that the dielectric properties of SU8 does not vary a lot with temperature over the range studied at both 2.45 and 3.50 GHz.

Figure 8-9: Dielectric properties of liquid SU8 at varying temperatures using the Dielectric Probe Method.
Similarly, dielectric measurements were also undertaken on the Si \(<100>\) substrate with the following results as shown in Figure 8-10. Results show that the loss factor of the substrate varies over the range of interest. Microwave absorption increases as the frequency increases and peaks at around \(\varepsilon'' = 4.8\,\text{GHz}\) and decreases slightly over the rest of the bandwidth. The dielectric loss factor of the substrate is several times that of the bulk SU8 which means that most of the microwave absorption will be by the substrate.

![Figure 8-10: Dielectric properties of Silicon \(<100>\) substrate using the Dielectric Probe Method.](image)

**8.3.2 Best Operating Frequency**

A sample of the spun SU8 was used in the VFM to determine the best possible frequency to be used for dielectric heating of the SU8. Although the dielectric values of the SU8 were previously obtained, this does not take into account the actual processing environment i.e. the microwave cavity. Thus it is essential to determine how the samples will behave in the processing environment. The characterisation function of the VFM is an indirect way of determining the best possible frequency to be used for the processing.
A cavity characterisation process was performed with the sample atop a cylindrical Teflon block located centrally within the cavity using the VFM parameters as shown on Table 8-4. The Teflon block is a low loss material and thus will have minimal affect on the cavity characterisation.

<table>
<thead>
<tr>
<th>Central Frequency</th>
<th>Power</th>
<th>Bandwidth</th>
<th>Sweep Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.25 GHz</td>
<td>100 W</td>
<td>2.5-8.0 GHz</td>
<td>0.1 sec</td>
</tr>
</tbody>
</table>

Table 8-4: VFM parameters used for Cavity Characterisation of SU8.

Figure 8-11 shows the result of the cavity characterization for the sample. As with the dielectric measurements, the results show no particular frequency to be optimum. A narrow band between 6-8GHz suggests that this is the best possible frequency as this is the range of lowest reflectance, however, for the purposes of this research and because there is no significant change in power absorbance for a particular bandwidth, it was chosen to use the whole bandwidth to utilize the full operating potential of the VFM.
This will minimise temperature differences in the cavity and increase the possibilities of time-average heating previously discussed as the size of the samples being processed are quite small compared to the cavity. In an industrial situation, it may be better to limit the operating bandwidth as the production quantity is much larger and the cost of a microwave source with a narrower bandwidth is much less than ones with wider operating bandwidth.

The decision to use the full bandwidth of the VFM can be further supported by the following simulations. The VFM model used in Chapter 5 was modified to simulate the VFM cavity loaded with a layer of polymer material resting on a Ø125 x 45mm teflon block as shown below in Figure 8-12.

![Figure 8-12: Left) VFM model of polymer material inside a cavity. Right) Teflon block and polymer sample dimensions.](image)

Figure 8-13 illustrates the results of the simulation for fixed frequency processing (2.5GHz and 5GHz) and VFM processing. The simulations highlight the improvement in the energy distribution in the load when VFM is utilized as compared to fixed-frequency processing.
8.3.3 Heating Profile

The heating behaviour of the liquid SU8 and the Silicon substrate was investigated to determine the individual heating abilities of each component and the best parameters to use for its processing. A fibre optic temperature probe was used to determine the temperature change and was attached to the samples using kapton tape. Temperature profiles were gathered using the inbuilt data acquisition hardware of the VFM. Temperature profiles were obtained for different frequencies and powers as summarized in Table 8-5 at a maximum processing time of 120 seconds.

<table>
<thead>
<tr>
<th>Material</th>
<th>Power (W)</th>
<th>Frequency (GHz)</th>
<th>Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU8 on glass</td>
<td>50, 100, 150</td>
<td>2.5, 3.5, 4.5, 6.0, 8.0, VFM (2.5-8.0GHz)</td>
<td>120</td>
</tr>
<tr>
<td>Silicon</td>
<td>50, 100, 150</td>
<td>2.5, 3.5, 4.5, 6.0, 8.0, VFM (2.5-8.0GHz)</td>
<td>120</td>
</tr>
</tbody>
</table>

Table 8-5: Summary of VFM parameters used to examine heating behaviour of SU8 on Glass and Si <100> substrate.

The heating profiles for SU8 at different frequencies and powers are shown in Figures 8-14 and 8-15 respectively. A glass substrate was used as it is virtually transparent to microwave energy and will not substantially contribute to the rise in temperature of SU8.
Figure 8-14: SU8 on Glass substrate – Effects of changing Frequency on temperature rise (P=50W).

Figure 8-15: SU8 on Glass substrate – Effects of increasing Power on temperature rise (f=2.5GHz).
The temperature profiles for the SU8 by itself suggest and supports the dielectric measurements undertaken that it is not very susceptible to microwave energy as the maximum temperature rise obtained over a 120s interval is only approximately 4°C even for the highest power used of 150W. It does show however that it is more absorbing at higher frequencies (6GHz and 8 GHz). This temperature rise is most likely due to the solvent component (cyclopentanone) of the SU8 which was found to be more susceptible to microwave power between 6-8 GHz as discussed previously.

Unlike the SU8, the Si substrate is more susceptible to microwave energy as the following results illustrate. Figure 8-16 shows the result of silicon heating as a result of changing frequencies. The heating profiles show that the sample strongly absorbs at 3.5GHz and 4.5GHz which resulted in highest temperature rise and the lowest occurring at 2.5GHz. The heating profiles show that the temperature rose rapidly at the beginning and plateaus as a maximum temperature is reached. Figure 8-17 illustrates the temperature rise as a function of power. Results show that the rate of initial temperature rise is proportional to the power input. As expected, a higher input power generates a more rapid ascent to the final temperature as well as achieving a greater temperature rise. This also shows that for a particular power, there is a limit to the maximum temperature that can be achieved due to heat losses.

A few common trends can be concluded in the heating profiles study of both the SU8 and Silicon substrate. The first is that higher power results in a higher temperature. Secondly, utilizing VFM frequencies (i.e. the whole bandwidth) does not necessarily mean that it will result in a higher temperature rise than using single frequency. As mentioned before, VFM results in a time averaged heating uniformity. Finally, for a particular power and/or frequency, there is a limiting temperature that can be reached as shown by the plateauing of the temperature profiles.
Figure 8-16: Silicon <100> substrate – Effects of changing Frequency on temperature rise (P=50W).

Figure 8-17: Silicon <100> substrate – Effects of increasing Power on temperature rise (f=2.5-8.0GHz).
8.4 VARIABLE FREQUENCY MICROWAVE RECIPE DEVELOPMENT AND OPTIMISATION

The version of the MicroCure software used in this research has had the advantage of significant advancement in temperature control and feedback control over previous versions. New developments in the software allows control of temperature ramp-up, hold times and temperature ramp-down which made it easier on the user, however despite this, the correct parameters must still be chosen to obtain the best possible processing conditions.

From the heating profiles gathered in the previous section, it was evident that the system reacts to different power levels and frequencies quite differently. It was therefore required to tailor and program a “Recipe” to obtain the required temperature profile. Since it was decided that VFM would be used i.e. 2.5-8.0GHz, the only other parameter that could be adjusted was the power level. Hence, different Recipes for the softbake and the post-exposure bake were developed to closely match the regime used in conventional processing.

It was initially thought that the use of higher power processing was more advantageous to allow the system to reach the desired temperature faster however, it was quickly realized that high power processing also means rapid temperature rise and higher temperature which led to larger temperature overshoots especially during the ramp and constant hold temperatures. Although the software allows for a temperature feedback which will cease microwave input into the cavity to control these overshoots, the feedback loop was not fast enough to turn off the power and unfavourable elevated temperatures were still attained particularly when using power settings >100W. High power processing had an unfavourable and detrimental effect on the sample which led to excessive outgassing of the solvent which is discussed further in the next chapter.

To overcome this problem, different powers were used at different stages of the processing. The use of low power was required during the ramp-up to get the system into
the correct temperature range without overshooting the required temperature profile. Microwave power <30W was not used as it was insufficient and took quite a long time to reach temperature. During the time of constant temperature, it was found that utilizing moderate power levels (~100W) to keep a constant temperature and limit temperature overshoots worked quite well. The recipes developed for the conventional softbake and post exposure bake that follows the desired temperature profile for the processing are shown in Appendix C. Representative temperature and power graphs obtained for the recipes are shown in Figures 8-18 and 8-19 for the softbake and post exposure bake respectively.

It should be noted that despite Figures 8-18 and 8-19 appearing to show constant power input during the constant holding temperatures (65°C and 95°C), oscillations in the temperature is observed due to the power being momentarily switched on and off when the set temperature reaches a maximum and minimum allowable threshold. This threshold was set at ±1°C above and below the constant hold temperature to avoid large temperature overshoots. The pulsing of microwave power into the cavity to maintain the set temperature may have been too rapid to be reflected in the power input graphs.

Figure 8-18: Temperature and Power profile obtained for VFM Softbake Recipe.
Figure 8-19: Temperature and Power profile obtained for VFM Post Exposure Bake Recipe.

A thermal image was taken of the sample after processing in the VFM using the developed recipes shows that uniform heat is achieved by the VFM as shown in Figure 8-20.

Figure 8-20: A thermal image of an SU8 sample straight after processing shows the heat uniformity within the sample using the recipe developed above.
8.5 SUMMARY

The investigation found that although the MicroCure 2100 has had a significant upgrade in its software for control of the VFM parameters and it was quite simple to program a “Recipe” for a particular cure regime, it wasn’t the case of simply programming and blindly choosing parameters in the hope that the software will compensate for events such as temperature overshoots and ramp time. The choice of the parameters greatly affects the processing and hence affects the final quality of the sample. It was found that with any microwave process, the material properties are quite important and the material’s behaviour within the cavity it is to be processed in, must be investigated and taken into account during the choice of VFM parameters. The MicroCure 2100 Cavity Characterisation sub-routine was found to be a quick way of determining the best frequency or bandwidth of frequency to be used and which can be indirectly used to approximate how the dielectric properties of the material varies at different frequencies.
9.1 Overview

This chapter introduces the results of SU8 curing using conventional, hybrid and VFM curing. The MicroCure 2100 is investigated as an alternative to conventional methods. To determine the quality of processing and the mechanisms eventuating from the different curing process, samples are characterised with respect to their, material properties such as dielectric properties ($\varepsilon'$, $\varepsilon''$) and thermal properties such as glass transition temperature ($T_g$), degradation temperature ($T_d$) and degree of cure ($\alpha$). Finally, sample structures are investigated using optical methods to determine the quality of the structures obtained from different cure regimes.

9.2 Introduction

Conventional curing of the epoxy based photosensitive SU8 photoresist is frequently done in an oven or on a hotplate. This type of curing is often time intensive and results in non-uniform products. For thick layers of SU8, a uniform bake of the layer is not always possible due to the mechanisms of heat transfer conventional curing offers, leading to poor pattern resolution, formation of micro-cracks and severe outgassing occurring as a consequence. Depending on the layer thickness, prebaking of samples to obtain the desired solvent content can take from minutes to several hours via the conventional methods. The long prebaking and curing times together with the mismatch in coefficient
of thermal expansion (CTE) between SU8 and its substrate, and the characteristically inherent rigid molecular structure of the molecule combine to generate considerable internal stress which causes cracked lithographic features. Thus alternative processing methods for SU8 are required to speed up the process and to avoid abnormalities in the structures such as cracks.

Microwave processing of polymers has existed since the 1940s but has only gained momentum in the early 1980s when advances in microwave technology came about. Although not all polymeric materials are suitable for microwave processing, many polymers contain functional group that form strong dipoles and are susceptible to microwave radiation. These include epoxy, hydroxyl, amino and cyanate groups among others. Thus, microwave processing can be used over a broad range of polymer products including thermoplastics, rubbers, composites and thermosetting resins such as SU8. For these materials, microwave processing can offer a new form of energy transfer which offers distinct advantages such as volumetric, rapid, direct, selective and instantaneous and controllable heating.

The principal mechanism of microwave absorption in a polymer is the reorientation of dipoles in the imposed electric field. The ability to process polymeric materials with microwaves depends on the dipole structure, frequency of processing, temperature and additives or fillers that have been included with the polymer. In addition, the coupling efficiency with the material is dependent on the dipole strength, dipole mobility and the dipole’s mass.

Although there have been multitudes of studies in microwave curing of epoxy based thermoset polymers, there has been very little done in terms of microwave curing of photoepoxies like SU8 or in the use of VFM for curing these. Microwave cured thermoset systems, including, polyesters [Gourdenne et al, 1979; Hottong et al, 1991], polyurethanes [Jullien and Valot, 1985], polyimides [Lewis, 1994] and epoxies [Gourdenne et al., 1979; Lewis et al., 1987; Jow et al., 1989; Wei et al., 1993; Demeuse and Johnson, 1994] have been studied with positive results. Many of the results showed enhanced curing speed during microwave curing compared to thermal curing. Other
advantages include improved or similar chemical and mechanical properties and more uniform product quality. Hence the interest in applying the Variable Frequency Microwave Technique to the processing of SU8 photoresist resins.

9.3 EXPERIMENTAL METHODOLOGY

An experimental schedule to investigate the suitability of VFM technology as an alternative to conventional SU8 processing was initiated and discussed below.

9.3.1 Sample Preparation

Commercially available Negative Tone SU8-2100 photoresist from MicroChem Corporation was used in this research for its ability to cover a wide range of thicknesses required in this study. SU8-2100 contains approximately 75% of high functionality SU8 resin, 25% cyclopentanone as the solvent and a very small percentage of a cationic photoinitiator based on mixed triarylsulfonium hexafluoroantimonate salts.

Thin SU8 films were prepared according to the steps outlined in Chapter 8. SU8 was statically dispensed on a Si <100> substrate and spin coated at a predetermined rotation rate according to Figure 9-1 to obtain a film thickness of approximately 260µm. Prior to spin coating, the Si <100> substrate was dehydrated in an oven at 150°C for approximately 15 minutes. The spun samples were allowed to settle for a few minutes on a levelled surface before going through the Softbake process.
Three different curing methods were investigated in this study 1) Conventional Curing  2) Hybrid Curing 3) Variable Frequency Microwave Curing. Conventional curing utilized a temperature controllable hotplate for sample curing during both the SB and PEB whilst VFM curing employed the MicroCure 2100 Variable Frequency Microwave. Hybrid curing utilized the hotplate for SB and the VFM for PEB. The use of a controllable hotplate ensured that heat input to the sample during the cure regime especially during the ramp-up and ramp-down would be similar to that of the VFM which is equipped with a more sophisticated software driven control system.

The recommended SB and PEB regimes as described in Chapter 8 were followed and the recipes developed for VFM curing were used. The SB regime stayed constant for all the experiment however, the PEB were undertaken at different temperatures by varying the second holding temperature (Hold2).

After softbaking, the samples were allowed to cool to room temperature after which the SU8 samples were UV exposed using a broadband mask aligner with soft contact mode to generate the Lewis acid from the Photo Acid Generator (PAG) to induce crosslinking of the SU8 photoresists. Each sample was exposed for approximately 120s with an
exposure strength of 275W, at a pressure of 350mmHg unless otherwise stated. A mask with a repeated pattern as shown in Figure 9-2 was applied to fabricate structures on the photoresists for the experiments.

![Pattern used during UV exposure for the fabrication of microstructures on SU8 films.](image)

Figure 9-2: Pattern used during UV exposure for the fabrication of microstructures on SU8 films.

Following the exposure, a PEB was undertaken on the samples. Finally, the samples underwent development by means of an immersion technique using MicroChem SU8 developer, PGMA at room temperature. Each sample was immersed in the developer for several minutes whilst manually agitating the developer to speed up development. In some cases mechanical agitation was used. Following development, each sample was rinsed briefly with Isopropyl Alcohol then dried with a gentle stream of nitrogen.

For this thesis, a hardbake which is an optional step in SU8 processing was not undertaken on any of the samples. This is to avoid high internal stresses that may cause cracking within the sample due to the difference between the linear thermal expansion coefficients (CTE) of the substrate and the resists and so that a residual cure could be calculated.

A summary of the experiments undertaken are summarized in Table 9-1. For simplicity, the following conventions will be used from here on in, Conventional curing on the hotplate is specified HP, Hybrid curing will be HY and Variable Frequency Curing is VFM.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness</th>
<th>Soft Bake (SB)</th>
<th>Exposure</th>
<th>Post Exposure Bake (PEB)</th>
<th>XX (PEB Hold Temperature –Hold2)</th>
</tr>
</thead>
</table>
| HP-XX   | 260µm     | HP R1: 250°C/hr to 65°C  
Hold: 65°C for 7min  
R2: 250°C/hr to 95°C  
Hold2: 95°C for 60min  
R3: -70°C/hr to 25°C  | 275W, 350mmHg for 120sec | HP R1: 250°C/hr to 65°C  
Hold: 65°C for 1min  
R2: 250°C/hr to XX°C  
Hold2: XX°C for 15min  
R3: -70°C/hr to 25°C  | 1) 65°C  
2) 75°C  
3) 85°C  
4) 95°C |
| HY-XX   | 260µm     | HP R1: 250°C/hr to 65°C  
Hold: 65°C for 7min  
R2: 250°C/hr to 95°C  
Hold2: 95°C for 60min  
R3: -70°C/hr to 25°C  | 275W, 350mmHg for 120sec | VFM R1: 250°C/hr to 65°C  
Hold: 65°C for 1min  
R2: 250°C/hr to XX°C  
Hold2: XX°C for 15min  
R3: -70°C/hr to 25°C  | 1) 65°C  
2) 75°C  
3) 85°C  
4) 95°C |
| VFM-XX  | 260µm     | VFM R1: 250°C/hr to 65°C  
Hold: 65°C for 7min  
R2: 250°C/hr to 95°C  
Hold2: 95°C for 60min  
R3: -70°C/hr to 25°C  | 275W, 350mmHg for 120sec | VFM R1: 250°C/hr to 65°C  
Hold: 65°C for 1min  
R2: 250°C/hr to XX°C  
Hold2: XX°C for 15min  
R3: -70°C/hr to 25°C  | 1) 65°C  
2) 75°C  
3) 85°C  
4) 95°C |

**Table 9-1:** Summary of curing experiments undertaken for SU8 photoresists processing. Please note that XX indicates the second holding temperature (Hold2) during PEB as shown in the last column.
9.3.2 Material Characterisation

9.3.2.1 Thermal Analysis

Thermogravimetric Analysis (TGA) was conducted using a Mettler Toledo TGA850 Thermal Analyzer to study sample thermal stability and weight loss as a function of time and temperature. Small samples of uncured SU8 were contained in an alumina crucible and heated using a ramp rate of 10°C/minute from room temperature to a temperature of 1000°C in a nitrogen atmosphere. From the data collected, sample composition and degradation temperature were obtained. The degradation temperatures, $T_d$, are reported as the onset of degradation as calculated from the intersection baseline of 100% sample mass and the line tangent to the inflection point.

A Differential Scanning Calorimeter from Mettler Toledo (DSC 821e) was used to study the thermal calorimetric properties of the cured and uncured resin and monitor the curing process via measurement of the heat of reaction. SU8 samples between 5-10mg were enclosed in an 40 microlitre aluminium pan with a pierced lid to prevent excessive pressures from building up during the solvent evaporation phase and were tested under a nitrogen purge of 50 ml/min to inhibit oxidation during heating. Samples were heated at a rate of 10°C/minute to a temperature just below the degradation temperature of 380°C [La Bianca and Gelorme, 1995].

The DSC was used to determine the heat of cure which is measured as the integral of a residual exothermic peak which occurs on the DSC thermogram. The heat of cure can be used to assess the Extents of Cure (%cure) using the following formula:

$$\%\text{cure} = \left( \frac{\Delta H_{\text{uncured}} - \Delta H_{\text{cured}}}{\Delta H_{\text{uncured}}} \right) \times 100$$

Equation 9 - 1

where $\Delta H_{\text{uncured}}$ is the heat of cure of the unreacted SU8 resin and $\Delta H_{\text{cured}}$ is the heat of cure of the partially cured sample. The heat of cure was normalized by the mass of each sample.
9.3.2.2 Dielectric Measurements

Dielectric measurement were carried out using a Dielectric Probe kit coupled to an Agilent S-Parameter Network Analyzer (Model 8722ES) with an operating bandwidth of 50MHz-40GHz. Prior to taking dielectric measurements, the dielectric probe was calibrated against substances with known dielectric properties. These were air, distilled water and a metallic shorting block. Dielectric properties of uncured SU8 resin as well as cured SU8 resin were taken at 2.5-8.0GHz. Appropriate care was taken not to introduce bubbles in the liquid sample and calibration water whilst calibrating the system and whilst taking readings. For solid samples, it was ensured that the probe face was resting parallel to the cured film and had good contact without any gaps.

9.3.2.3 Microstructure Analysis

After development of the thin SU8 films the microstructures were analysed visually for the quality of structures using a variety of methods. Visual analysis of the structures was undertaken at a variety of magnification using a Laser Scanning Confocal Microscope and a normal optical microscope both from Olympus.

Film thickness and surface profile of the samples were also obtained using a Mahr Perthometer Profilometer.

9.4 RESULTS

Thin SU8 films were cured as described on Table 9-1. DSC and TGA analyses of the SU8 films were conducted in order to determine the effect of the different curing techniques on the quality of cure and the effects on material properties such as the degree of cure (\( \alpha \)), glass transition temperature (\( T_g \)), degradation temperature (\( T_d \)) and solvent content of the thin films.
9.4.1 Characterisation of Uncured Photosensitive SU8

Thermal analysis was performed on the uncured SU8 as a reference for comparison to determine the extent and quality of cure using the different types of curing techniques undertaken in this section.

As a baseline, a DSC analysis was carried out on the as supplied resin to determine the heat of cure of the unreacted resin (\(\Delta H_{uncured}\)). Figure 9-3 shows typical DSC results for uncured SU8 at a heating rate of 10°C/min to a temperature just below the degradation temperature of 380°C. Multiple test were undertaken to show repeatability.

![Figure 9-3: Typical DSC thermogram of as received SU8-2100 resin from MicroChem Corporation.](image)

Under the conditions used in this experiment, the curing reaction begins at approximately 150°C and is observed as a large exothermic peak in the temperature range up to 350°C. Integration of the exotherm results in a Total Heat of Cure (\(\Delta H_{uncured}\)) required for a full (100%) cure of the SU8 is approximately 252 J/g. This value was used as the reference
value to evaluate the extent of cure for the remainder of this chapter. To ensure the resin was fully cured, the cured resin was cooled down to 0°C at a rate of -10°C/min and reheated back to 370°C at the same heating rate. The resulting exotherms (Figure 9-4) show the total disappearance of any exothermic peak therefore indicating that the cross-linking has gone to completion when heated to 370°C during the first scan and thus the resin was assumed to be fully cured.

![Figure 9-4: Exotherm illustrating disappearance of exothermic peak indicating full crosslinking of the SU8. Cool down (Top), Heat up (Bottom).](image)

The thermal stability of the uncured SU8 was studied by TGA. From the TGA data gathered as shown in Figure 9-5, it is evident that there are two distinct transformation regions, all indicating mass loss, which corresponds to the boiling point of each of the components in SU8. The first significant weight loss is due to the evaporation of the solvent, cyclopentanone, which coincide to a boiling point of approximately 130°C. Results indicate that the average solvent content of the uncured SU8 is between 18-21%
w/w. The second transformation region occurs at approximately 435°C and corresponds to the epoxy content which is measured to be between 53-57% w/w. Overall, uncured SU8 decreased an average of 73-75% of its weight during the TGA scans. From this figure, the onset of degradation temperature ($T_d$) is 385°C which is just above the value measured by other researchers [LaBianca and Gelorme, 1995; Feng and Farris, 2002 & 2003] of approximately 380°C, and the char yield at 1000°C is between 25-27%. Also shown in the figure is the numerical derivative TG trace (DTG), which is a smoothed plot of the instantaneous slope of the specimen mass with respect to time. The DTG does not contain any new information, however it clearly identifies the temperature at which mass loss is at a maximum (the DTG Peak). This corresponds to a temperature of 430°C.

![Figure 9-5: Thermogravimetric Analysis of raw SU8 resin.](image)

### 9.4.2 Characterisation of Cured SU8

SU8 films where processed using the 3 different curing methods and were characterized as discussed in the following section.
9.4.2.1 *Differential Scanning Calorimetry*

Thin SU8 films were cured according to the settings outlined on Table 9-1. Figures 9-6, 9-7 and 9-8 show the calorimetric response of the samples to a heating rate of $10^\circ$C /min. In all curing techniques used, the resulting DSC curve shows a large exothermic peak which indicates that the films were not fully cured after processing. This result is common with the findings of other researchers [Feng and Farris, 2003] whose work have found that an exothermal peak still exist after PEB of SU8 and the disappearance of this exothermal peak was only observed after the Hard Bake which was not undertaken in this study. Typically, a Hard Bake is undertaken at a temperature of $>200^\circ$C to fully crosslink the polymer.

![Figure 9-6: Exotherm for Hotplate (HP) cured SU8 resin at different temperatures.](image-url)
Figure 9-7: Exotherm for Hybrid (HY) cured SU8 resin at different temperatures.

Figure 9-8: Exotherm for VFM cured SU8 resin at different temperatures.
In all the DSC curves, a small endothermic peak at approximately 70-80°C can be observed and is possibly due to moisture and trapped solvents within the densified film. Unlike the thermally cured raw SU8 (Figure 9-3) which had a large exothermic peak between 150-350°C, the exothermic peak observed in the cured samples initializes at approximately 100°C and ends at roughly 270°C. A likely explanation for the shift in the exothermic peak may be the presence of the strong Lewis acid used as a catalyst for the crosslinking of the polymer, and which was generated during the UV Exposure step of the processing which was not taken into account during the DSC of the uncured SU8. Nevertheless, it is still valid to use the Total Heat of Reaction gained for the uncured SU8 as a baseline as the amount of heat required is irrespective of any catalyst in the system. The main purpose of the catalyst is to initiate the crosslinking by lowering the activation energy for the reaction to proceed.

By integrating the area under the exothermic peaks, a residual heat was obtained and was used together with Equation 9-1 was used to calculate the degree of cure for the different processing conditions as illustrated in Figure 9-9.

![Figure 9-9: Degree of Cure vs. PEB Processing Temperature for HP, HY and VFM cured SU8.](image)
The graph shows that the degree of cure increases with increasing PEB temperature for all curing techniques. The Conventional (HP) curing was found to achieve lower %Cure and hence lower crosslinking than the Hybrid and VFM curing at all temperatures used. The hybrid curing achieves an average of 3% better cure than the conventional cure and the VFM at approximately 12% higher.

The higher conversion rates obtained during the Hybrid and VFM processing exemplifies the advantage of MW energy in polymer processing and its major effect on curing rates of SU8. The increase in curing rates over the conventional HP curing maybe explained in two ways. One is a purely heat transfer explanation and the other brought about by the unique ability of MW energy to couple with the sample.

For thermosetting polymers such as SU8, conversion is slow due to its poor thermal conductivity when processed by conventional means [Day and Zainol, 2003]. In conventional curing the coated substrate is laid flat onto the surface of the Hotplate such that full contact can be obtained to achieve an even temperature distribution. During SB and PEB, the curing reaction is typically associated with significant shrinkage of the SU8 coating due to the molecular chains being pulled closer together by the crosslinks. Due to the large mismatch in Coefficient of Thermal Expansion (CTE) between SU8 (50ppm/K) and the silicon substrate (2.33 ppm/K), bowing of the substrate is a common occurrence in conventionally cured SU8 films [Ruhmann et al, 2001]. As a result of the wafer bowing, the surface contact between the substrate and hotplate is compromised resulting in only partial contact or at worst, point contact with the hotplate surface therefore affecting the heat transfer to the sample and thus influencing the curing rate. A surface profile of a Hotplate and a VFM sample at 95°C was obtained using a profilometer and is shown in Figure 9-10. The profiles obtained confirm the bowing of the wafer which is more significant at higher temperatures.
Figure 9-10: Surface profiles of cured samples at 95°C showing bowing of the substrate and film due to the mismatch in CTE – Hotplate (top) and VFM (bottom).

After the softbake, only a slight bowing of the wafer is observed. The main stress occurs during the post exposure bake and is anticipated to increase even more during the hard bake which is undertaken at a very high temperature. Bowing of patterned films i.e. of exposed films are expected to be better as there is some stress release due to the unpatterned areas [Despont et al, 1997].

The limitations of heat transfer are eliminated as MW radiation is absorbed directly by both the substrate and the SU8 coating. Therefore bowing of the substrates will not have an effect on the heat transfer to or within the sample. It is likely that the heat transfer is a contributing factor in the difference in the degree of cure observed. If this is the case, the HP samples would have the lowest degree of cure followed by the Hybrid and then the VFM which was observed experimentally. In Hybrid curing, the heat transfer effect is partially eliminated as a combination of hotplate and microwave heating is used. In VFM
curing, the heat transfer effect is completely eliminated as it is purely a non-contact method of heating thus achieving the highest conversion.

The use of an oven instead of a hotplate for conventional curing may overcome the problem of good surface contact and will produce curing rates similar to those of the VFM technique. As far as the heat transfer is concerned, it may be similar to the VFM technique. However, the major difference is in the VFM interaction with the coating material. The difference between the external heat sources causing linear translational movement of the molecules, versus the additional rotational movement of the molecules caused with VFM, is what enhances the cure rates. In SU8, these functional groups are the epoxide groups which are well known to be microwave absorbers.

The difference in the degree of cure experienced in the different curing methods is most likely a combination of the reasons explained above. Since the samples where quite thin, one might argue that a temperature gradient may not exist long enough and that thermal equilibrium is reached quite rapidly to cause such a significant difference in the degree of cure between the three different curing techniques. In fact, a heat transfer calculation as shown in Appendix D, shows that the characteristic time to heat up is 68ms. If this is the case, it becomes more evident that the use of MW energy has a real effect on the curing of SU8. From a processing point of view, the significance of a higher conversion is advantageous effect in that low temperature curing can be used to obtain the same degree of cure as conventional processing. Furthermore, rapid curing of the films is possible to an otherwise lengthy cure.

Another indicator of the degree of cure is the Glass Transition Temperature ($T_g$). As a thermosetting resin cures two main items occur, 1) the heat of cure decreases and, 2) $T_g$ increases, thus changes in $T_g$ can also be used to quantify the degree of cure of the resin. $T_g$ represents the region in which the resin transforms from a hard, glassy solid to a viscous liquid in a DSC curve. The cure reaction is typically associated with significant shrinkage because of the stress induced as molecular chains are pulled closer to each other during crosslinking. The network of crosslinked molecules increases as the conversion increases, subsequently impeding more crosslinking and a further increase in
$T_g$. Depending on the segmental mobility of the molecules through the crosslinked network, a further increase in $T_g$ may or may not be significant as conversion reaches 100%. This is true whether the process of curing is thermal or radiation-induced such as microwave curing, since the nature of the curing process involves the mobility of the individual growing chains. While generally the $T_g$ increases with an increase in the degree of conversion, the relationship is not necessarily linear [Rath et al, 2004].

Unfortunately, several attempts were done to determine the $T_g$ of the samples by using different heating scans using the DSC but due to the large exothermic peak in the DSC curve which is a measure of the total heat flow, the $T_g$ was overlapped and was very difficult to detect. In hindsight the use of different techniques such as Dynamic Mechanical Thermal Analysis (DMTA) [Feng and Farris, 2003] or a Modulated Differential Scanning Calorimeter (MDSC) [Rath et al, 2004] which are more sensitive to $T_g$ detection could have been used to obtain the $T_g$ of the samples but were unavailable to this study. Different $T_g$ has been reported in literature due to the differences in processing conditions. However, the reported $T_g$ for a fully cured, hardbaked, SU8 films is over 200°C [LaBianca et al, 1993]. Feng and Farris [2003] found that the $T_g$ is approximately identical to the baking temperature when the processing temperature is less than 220°C and increasing in deviation at higher temperature ranges to a maximum $T_g$ of 238°C at a baking temperature of 300°C, which corroborates with the finding of LaBianca. The $T_g$ of post cured SU8 films have been reported to be always higher than the PEB curing temperature of about 40-70°C [Rath et al, 2004].

The $T_g$ is uniquely related to the conversion for a thermosetting system. Di Benedetto has derived from theory an equation relating the shift in the $T_g$ temperature to the degree of crosslinking [Nielsen and Di Benedetto, 1969]. By assuming an idealized system consisting of a mixture of a fully cured network and an unreacted monomer phase, a more commonly used version of the well-known Di Benedetto equation was derived from entropic considerations as shown in equation 9-2 [Simon and Gillham, 1993]

$$\frac{T_g - T_{g0}}{T_{g0} - T_{g0}} = \frac{\lambda \alpha}{1 - (1 - \lambda) \alpha}$$

Equation 9 - 2
where $\alpha$ is the degree of polymer conversion or degree of cure, $T_{g0}$ and $T_{g\infty}$ are the $T_g$ values at zero and 100% conversion respectively and $\lambda$ is an adjustable structure-dependent parameter which was found to be approximately 0.88 by Rath et al. [2004] for SU8. Using this modified Di Benedetto equation and the results of the degree of cure obtained from the DSC studies, the glass transition temperature for the cured SU8 films were calculated and shown in Figure 9-11. The values for $T_{g0}$ was 50°C which was previously measured for the uncured SU8 resin using the DSC, and the reported literature value of $T_{g\infty} = 238^\circ$C for a fully cured SU8 [Feng and Farris, 2003] was utilized for $T_g$ prediction.

![Figure 9-11: Glass Transition Temperature of cured SU8 as predicted by the Di Benedetto Equation.](image)

Observation of the results above show that $T_g$ increases with increasing PEB temperature as expected and is generally associated with a higher degree of crosslinking attained at higher processing temperatures. The Di Benedetto Model predicted a range of $T_g$ values between 144°C for a conversion of about 53% at 65°C PEB temperature, to 197°C for the highest degree of conversion attained of 80% at 95°C PEB temperature. On average, the $T_g$ values for VFM processed samples are higher than the other processing methods by approximately 14-27°C. The difference in $T_g$ reflects the high amount of crosslinking in the VFM cured films as compared to the HP and HY samples at similar PEB temperatures. Predicted $T_g$ values for HP and HY films are more or less identical at
processing temperatures >85°C but start to deviate at 95°C. An interesting observation may be made about the values of the glass transition temperatures predicted and the PEB temperatures used in the experiments. It can be observed that the $T_g$ is always higher than the PEB temperatures by about 80°C for the HP and HY processed samples and approximately 100°C for the VFM samples. Similar findings have been made by Rath et al, [2004] who reported $T_g$ of about 40-70°C above the PEB temperature. The $T_g$ values predicted by the Di Benedetto equation seem to be reasonable, although maybe higher than those found in literature. The difference between the predicted and literature values may be due to the differences in processing conditions used and therefore literature values can only be used as a guide. Moreover, it must be noted that the Di Benedetto equation is a models and all models make certain assumptions and have their limitations.

### 9.4.2.2 Thermo Gravimetric Analysis

Thermogravimetric Analysis of the HP, Hybrid and VFM cured SU8 were undertaken to study the thermal stability of the cured films. A typical curve for the three processing methods is shown in Figure 9-12 and the results summarized in Table 9-2.

The thermal degradation profiles reveal that all samples experience similar degradation. Initially, a small weight loss of <5% can be observed at the lower end of the temperature range below 150°C. This initial rapid yet small weight loss is most probably due to the evaporation of trapped residual solvent and or photoproducts and was ignored when determining the degradation temperature ($T_d$) of the samples. At approximately 395-405°C the onset of thermal decomposition of the samples occurred in a single step during which most weight loss occurs. The rate of weight loss during this period is rapid and continued up to approximately 470°C peaking at around 430°C as indicated by the DTG peak. The average char yield after heating to 1000°C is 26.2%. The measured data also suggest that after complete reaction, the TGA curve was more stable than that of the unreacted SU8 resin which has a $T_d$ of 385°C.

The results of the $T_d$ values for the HP, HY and VFM do not differ significantly to each other and the differences are within experimental error. Therefore it can be concluded
that the stability of the cured films are similar at all processing temperature using any of the processing methods investigated. A value of $T_d = 398^\circ$C has been reported in literature which compares well with the ones measured.

From the results obtained, the residual solvent content shows no particular relationship between processing temperature and residual solvent which was not expected. It was anticipated that solvent content will be lower at higher processing temperatures however are not reflected in the results. The anomalous result may have been due to the inherent sensitivity of the TGA analyser to sampling technique. Although sampling was done quite consistently, contaminations could not be fully avoided and occurrence such as uptake of moisture from the environment in some samples may have occurred thus producing different results.

![Figure 9-12: Typical Thermogravimetric Analysis profile for processed SU8.](image)
9.4.2.3 **Dielectric Properties**

The dielectric properties were evaluated for the samples cured by the different processing technique and compared to the dielectric properties at the conventional microwave frequency of 2.45GHz. The results of the test are summarized in Table 9-3 and the variation in dielectric loss ($\varepsilon''$) factor in Figure 9-13.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\varepsilon'$</th>
<th>$\varepsilon''$</th>
<th>Loss Tangent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uncured (2.45GHz)</td>
<td>6.62</td>
<td>1.50</td>
<td>0.2266</td>
</tr>
<tr>
<td>HP-65</td>
<td>8.69</td>
<td>1.24</td>
<td>0.1430</td>
</tr>
<tr>
<td>HP-75</td>
<td>11.15</td>
<td>1.92</td>
<td>0.1723</td>
</tr>
<tr>
<td>HP-85</td>
<td>10.05</td>
<td>1.56</td>
<td>0.1556</td>
</tr>
<tr>
<td>HP-95</td>
<td>8.14</td>
<td>1.01</td>
<td>0.1239</td>
</tr>
<tr>
<td>HY-65</td>
<td>8.81</td>
<td>1.15</td>
<td>0.1304</td>
</tr>
<tr>
<td>HY-75</td>
<td>6.47</td>
<td>0.67</td>
<td>0.1034</td>
</tr>
<tr>
<td>HY-85</td>
<td>6.98</td>
<td>0.74</td>
<td>0.1066</td>
</tr>
<tr>
<td>HY-95</td>
<td>8.47</td>
<td>0.24</td>
<td>0.0286</td>
</tr>
<tr>
<td>VFM-65</td>
<td>3.00</td>
<td>0.15</td>
<td>0.0510</td>
</tr>
<tr>
<td>VFM-75</td>
<td>3.64</td>
<td>0.14</td>
<td>0.0393</td>
</tr>
<tr>
<td>VFM-85</td>
<td>3.86</td>
<td>0.14</td>
<td>0.0355</td>
</tr>
<tr>
<td>VFM-95</td>
<td>2.99</td>
<td>0.05</td>
<td>0.0157</td>
</tr>
</tbody>
</table>

Table 9-3: Dielectric Properties of processed SU8 as measured at 2.45GHz using the Dielectric Probe Method.
The most important dielectric property is the dielectric loss ($\varepsilon''$) and therefore will be focused on in this section. Results show that apart from the HP processed samples, there is a general decrease in $\varepsilon''$ that was observed as compared to a value of 1.5 for unprocessed SU8.

During a processing cycle, the polymers dielectric constant, which is a measure of the ability to absorb microwaves vary as phase changes within the polymer occur. In thermosetting polymers, the viscosity and molecular weight tend to increase during the cure as it becomes a highly cross-linked network. Studies have shown that the permittivity and the dielectric loss factor of these type of polymers generally increase with temperature and decrease with extent of cure [Jow et al, 1988]. Thus, initially thermosets are an efficient absorbers of microwave energy as $\varepsilon''$ increases as temperature increases. However, as the polymer crosslinks the materials become relatively less and less susceptible to microwave heating owing to the decrease in dipolar mobility and, hence, dielectric properties with extent of reaction. During the SB and PEB, initial curing involves the gelation of the liquid SU8 to form longer but initially linear chains, thus resulting in an increase in viscosity but not entanglement of the growing chains. As

![Graph showing relationship between Dielectric Loss Factor ($\varepsilon''$) and PEB Processing Temperature for different processing methods.](image-url)
the curing process proceeds, the entanglements increase significantly to isolate the reactive epoxy groups within the matrix, together with some of the residual protons and any other reactive species. As the curing proceeds the epoxy groups decrease hence the lower $\varepsilon''$ measured for the HY and VFM samples.

For the HP samples, it seems that a decrease in $\varepsilon''$ does not occur till a processing temperature of 95°C is used. The initial increase in $\varepsilon''$ may be due to the residual solvent and photoproducts such as radical initiators generated during UV exposure, remaining in the film. Unremoved solvents and portions of the photopackage including initiators would result in higher dielectric properties. The presence of these unremoved species are observed as the initial weight loss detected in the TGA results of the cured samples as discussed in the previous section. Although the weight loss is also observed in the HY and VFM cured samples, any unremoved solvents and portions of the photopackage did not increase the measured $\varepsilon''$ as the molecular structure of the polymer were already rigid due to the higher degree of cure achieved using these methods compared to HP processing. In general, the Dielectric loss factor decreases with the processing temperature as seen in Figure 9-13 for each processing method which is expected.

9.5 MICROFABRICATION OF MICROSTRUCTURES

Ultimately, the curing of SU8 films ends in the fabrication of microstructures required in the MEMS industry. Microstructures were fabricated using the curing techniques described above and their quality, in terms of structure features, resolution i.e. definition and sharpness of edges or corners, and adhesion were assessed using a variety of optical methods. Since there were a large number of images taken of the microstructures, only selected images have been chosen to illustrate what is being described. It should be noted that black spots on the surface of the samples are dirt particles which could not be avoided as the VFM facility was not in a clean room environment where such fabrication normally is undertaken and should be ignored.
The results and problems that were overcome are discussed in the following sections. A quick summary of the cure results and findings are shown in Table 9-4.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Microstructure Quality</th>
<th>Problems / Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP-65</td>
<td>unsatisfactory</td>
<td>Poor resolution, swelling, tapering, semi-developed</td>
</tr>
<tr>
<td>HP-75</td>
<td>unsatisfactory</td>
<td>Poor resolution, swelling, tapering, semi-developed</td>
</tr>
<tr>
<td>HP-85</td>
<td>satisfactory</td>
<td>Tapering of edges</td>
</tr>
<tr>
<td>HP-95</td>
<td>satisfactory</td>
<td>Tapering of edges, cracks developing</td>
</tr>
<tr>
<td>HY-65</td>
<td>unsatisfactory</td>
<td>Poor resolution, swelling, tapering, semi-developed</td>
</tr>
<tr>
<td>HY-75</td>
<td>unsatisfactory</td>
<td>Slightly better resolution, swelling, tapering, small</td>
</tr>
<tr>
<td></td>
<td></td>
<td>undeveloped areas, structure failure</td>
</tr>
<tr>
<td>HY-85</td>
<td>satisfactory</td>
<td>Tapering of edges, cracks developing</td>
</tr>
<tr>
<td>HY-95</td>
<td>satisfactory</td>
<td>Slight tapering of edges</td>
</tr>
<tr>
<td>VFM-65</td>
<td>unsatisfactory</td>
<td>Very poor resolution, swelling, cracking, structure failure,</td>
</tr>
<tr>
<td>VFM-75</td>
<td>satisfactory</td>
<td>Tapering, cracking</td>
</tr>
<tr>
<td>VFM-85</td>
<td>satisfactory</td>
<td>swelling</td>
</tr>
<tr>
<td>VFM-95</td>
<td>satisfactory</td>
<td>cracking</td>
</tr>
</tbody>
</table>

Table 9-4: Summary of the experimental findings for curing of SU8 photoresists.

9.5.1 Hotplate Processed Microstructures.

The hotplate processing method was found to be quite straightforward and no processing problems were faced during the study. The processed microstructures at different temperatures are shown in Figures 9-14.
CHAPTER 9: RESULTS – VFM PROCESSING OF PHOTOSENSITIVE SU8

The above results show that Hotplate microstructures obtained at low processing temperatures of HP65°C and HP75°C were of very poor quality after development which indicates films where insufficiently cured. At these processing conditions, the degree of cure was measured to be <60%. The microstructures produced are of very poor resolution with the existence of undeveloped resist in between structures are visually evident thus suffering from distorted features such as curved edges, un-straight lines and semi-developed structures. Furthermore, developer induced swelling of parts of the patterned images as seen in Figure 9-15 is another indication of the poor cure achieved as SU8 is known to adhere quite well and is said to be difficult to remove from the substrate when a good level of polymerisation is achieved [Koukharenko et al, 2005]. If left longer
in the developer solution adhesion failure and perhaps structure failure of these parts would result as the developer gets in between the film and substrate.

Microstructures at HP85°C and HP95°C showed improved resolution over the lower temperature cured samples. The structures are well developed with all unexposed areas etched away during development resulting in defined edges and straight lines. However in both samples, tapering or bevelling of the edges can be observed. This tapering is also a result of a low degree of cure and hence crosslinking of the polymer due to low curing temperatures used or insufficient post-baking time. The tapering seems to decrease at HP95°C however, minuscule cracks can be seen to appear on the inner diameter of the large ring structure. This is most likely due to stress introduced by the difference

![Figure 9-15: SU8 microstructure illustrating poor resolution.](image-url)
between the CTE of the Si substrate and SU8 and to stress due to resist polymerization hence predisposing SU8 samples to mechanical stress which could lead to adhesion problems. However, it may also be attributable to uneven cross-linking, which could result in cracks occurring at potential stress concentration areas as observed here. The stresses being brought on by thermal gradients during heating or cooling. An even cross-linking distribution would not be prone to cracking due to thermal stresses.

9.5.2 Hybrid Processed Microstructures

As a result of preliminary studies on Hybrid processing of SU8, one of the problems that influenced the choice of VFM power was encountered. After the post-exposure bake using the VFM, small bubbles at the intersection of the straight channels or pathways surrounding the main circular structures were observed which consequently affected the surface directly in the vicinity of the bubble after development of the sample. Figure 9-16 illustrates the effect of the bubble on the surface finish after development and after the bubble had burst.

![Figure 9-16](image)

**Figure 9-16:** Left – Optical image of effect of bubble formation due to excessive outgassing during processing. Right – 3D Confocal image of affected surface.
Any trapped solvent that is evaporated during PEB will take the path of least resistance to exit the film. When the samples are heated during PEB, the polymer matrix becomes rigid as polymerization proceeds and makes it difficult for solvents to evaporate from the polymerized areas. The unexposed areas, which are designed not to crosslink, and especially the region where these channels intersect are an easy route for evaporating solvents. The bubble is thought to be caused by residual solvents evaporating during PEB. However, although the Hotplate and VFM follow the same PEB regime, the bubbles were not observed in Hotplate processed samples. The main difference between the two techniques was rapid heating of the sample due to overshoots in the set temperature during VFM processing thus causing rapid evaporation of solvent and creating the bubbles observed. This was due to the high power initially chosen in VFM which was eluded to and discussed in Chapter 8 and is the main reason for choosing lower microwave powers. The disappearance of the bubbles due to lower microwave power is illustrated in Figure 9-17.

![Figure 9-17: Disappearance of bubble using modified VFM recipe.](image)

The Hybrid processed microstructures at different temperatures are shown in Figure 9-18.
Similar to results obtained for Hotplate samples, cured samples at lower temperature were poor in quality. HY65°C suffered from poor resolution, undeveloped resin and swelling. The sample processed at HY75°C achieved a slightly better resolution though undeveloped resin is still visible in some parts. Swelling is also obvious as well as structure failure at some sections.

At higher temperatures, structure integrity was improved although still suffering from tapered edges and miniscule cracking. For HY95°C, the effect of tapering is not as pronounced as in HY85°C therefore, it can be concluded that the HY95°C is close to the optimum degree of cure to eliminate tapering.
9.5.3 VFM Processed Microstructures

The initial trials for the VFM cured SU8 all produced poor quality microstructures. Early observations found that a noticeable difference between the HP and Hybrid cured SU8 is the tackiness of the films after softbaking. After SB using the hotplate, the films where still quite soft and tacky which means a higher solvent content compared to the VFM SB films which were a lot harder. During exposure, the mask aligner contacts the film and exposure is undertaken four times at 30s interval each. Any slight movement of the sample during exposure or in between exposures results in misalignment from the previously exposed areas. Due to the tackiness of the hotplate samples, it was found that it was easier to expose these and no misalignment occurred. The VFM softbaked samples were difficult to expose due to their hardness and mostly resulted in misaligned exposures resulting in multiple patterns on the sample which did not develop well as demonstrated in Figure 9-19. As a result, it was decided to do the whole exposure at once i.e. 120s, which proved to overcome the misalignment problem encountered.

![Figure 9-19: Effect of slight movement in sample during UV Exposure.](image)
The VFM processed microstructures at different temperatures are shown in Figure 9 - 20.

![Figure 9-20: Hybrid cured SU8 films clockwise from top a) 65°C, b) 75°C c) 85°C d) 95°C.](image)

The above shows that VFM curing at 65°C resulted in catastrophic failure of the structures. The sample illustrated shows the centre structure has lifted off most likely from swelling during development. At this processing temperature, a degree of curing of just above 60% was achieved which was similar to the HP and Hybrid curing where unsatisfactory microstructures were observed thus, it can be concluded that a degree of cure above 65% is required to achieve satisfactory microstructures. One difference with the VFM65°C cured samples is the appearance of micro cracks on the surface of the
structure. This may have been caused by the longer exposure time used in the VFM curing which generates a bit more heat and therefore stress to the surface of the film.

At the higher processing temperatures, improved resolution was achieved. Edges are well defined and all unexposed areas etched away during development as required. Although the images do not illustrate this due to the appearance of black spots, it should be noted that these are actually dust particles which were difficult to remove and not undeveloped SU8. As previously mentioned, the samples were processed outside of a clean-room for VFM processing and during imaging with a microscope, where dust is present and contamination could not be avoided. Contamination due to dust and dirt is particularly greater for VFM method as both SB and PEB were undertaken outside a clean-room. One noticeable absence in the VFM cured microstructure is the tapering observed in the HP and HY methods suggesting that the degree of cures are sufficient which substantiates the higher degree of cure measured using the DSC. However, it seems that the better resolution microstructure were achieved at the cost of miniscule cracks appearing on the surface of the microstructures which could be due to the initially much harder film after the softbake which could be because of low solvent levels within the film after the softbake. Nevertheless, the results show that full VFM curing is possible at lower temperatures and can be further optimized to give better structures.

9.5.4 Optimisation of VFM Processing of SU8

Although miniscule cracks were present in the VFM cured samples, the microstructures obtained were quite satisfactory. As eluded in the previous section, the harder film obtained after the VFM softbake step may be the cause of the cracks and suggest that solvent content is a possible optimisation point for the VFM method. The softbake is a simple but important step in the process and has been found to influence the final quality of microstructures [Liu et al, 2005] which may include elimination of cracks.

In order to optimise the process, an analysis of the weight change during the different steps of processing was undertaken and the result shown in Figure 9-21.
The results confirm that most of the weight loss due to solvent evaporation occurs during the softbaking step which is expected as the main purpose of this step is to reduce the solvent level and densify the film. After this step, minimal solvent removal is experienced during the exposure and the post exposure bake. It also shows that VFM achieves the highest weight loss followed by the Hybrid then the HP processing. As before, the difference in weight change is most likely due to direct coupling of microwave radiation to the SU8 film. The lower solvent content means a denser film which is what was observed with the VFM cured SU8 samples.

In this study, the normal softbaking time used was approximately 60 minutes at 95°C. The change in solvent content as a function of time at 95°C is shown in Figure 9-22. The rate of solvent evaporation is high at the beginning and slowly decreases as solvent content within the film reduces. At the normal softbaking time of 60 minutes, the solvent content is approximately 6-7% which is higher than the TGA results of about <5%. However, it must be noted that the softbake regime is a multiple step process with additional ramp-up and ramp-down steps thus the actual SB time is actually greater than
60 minutes. It should be noted that the results in Figure 9-22 do not take into account these ramp-up or ramp-down times and hence results in the slightly higher solvent content. It has been suggested in literature that a solvent content of approximately 8-10% is ideal. This corresponds to a SB time of approximately 36 minutes.

![Graph showing change in solvent content vs. time for SU8 film at 95°C.](image)

**Figure 9-22**: Change in Solvent Content vs. Time for SU8 film at 95°C.

The improved soft baking time was incorporated in a VFM cure of the SU8 with the resulting microstructures as shown in Figure 9-23.
CHAPTER 9: RESULTS – VFM PROCESSING OF PHOTOSENSITIVE SU8

Figure 9-23: Comparison of microstructure features obtained using Full VFM cure (left) vs. Full Hot Plate Cure (right).

As seen above, the optimized VFM method produced a vast improvement on the microstructures fabricated using the normal VFM regime. TGA analysis of the film indicates that solvent content is approximately 8% which is within the targeted solvent range. Very good resolution is obtained with well defined structures with sharp and...
straight edges and no signs of tapering. The problem of the cracks has also been greatly improved with almost all cracks eliminated. In comparison to the normal SU8 processing technique (HP95°C), the microstructures fabricated seem to compare well and even better as no tapering is present.

### 9.6 VFM CURING OF THICK SU8 PHOTORESIST FILMS (>500 MICRONS)

VFM curing of thick SU8 films (>500µm) was attempted in this study to determine the ability of VFM to remove solvent and to determine the degree of cure. Only a full Hotplate Cure (HP95°C) and VFM cure (VFM95°C) were studied as time was limited. Due to the thickness of the films the curing holding time were adjusted accordingly, the SB time was increased to a hold time of 720minutes and PEB was increased to 30minutes. A flood exposure was undertaken on the samples in a UV cabinet as the samples were too thick to be exposed in the UV mask aligner and consequently could not be patterned to for the fabrication of microstructures.

After the SB, the samples were observed to be hard and dense which indicated high solvent loss and after exposure and PEB became even harder which suggested a high degree of curing. However, the results shown in Table 9-5 suggest otherwise.

<table>
<thead>
<tr>
<th>Curing Method</th>
<th>Degree of Cure</th>
<th>Solvent Content</th>
<th>$T_d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hotplate</td>
<td>48.5%</td>
<td>7.5%</td>
<td>405°C</td>
</tr>
<tr>
<td>VFM</td>
<td>56.2%</td>
<td>6.3%</td>
<td>403°C</td>
</tr>
</tbody>
</table>

Table 9-5: Thermal analysis result for thick film photoresist.

One possible reason for the low degree of cures measured despite the appearance of being highly polymerized, maybe that at the beginning of the SB there is a large difference in evaporation rate between the bulk (interior) and the surface of the film and as the solvent near the surface evaporates the surface densifies, and a ‘skin’ appears on the surface of the film. As the baking continues, the skin becomes thicker and evaporation of solvent from the interior halts. Thus resulting in a hard top layer but quite a soft inner layer
which was observed when obtaining samples for thermal testing. Nevertheless, despite the low degree of cures obtained, the degree of cure was still found to be higher using the VFM and the solvent content lower as compared to the conventional hotplate processing as experienced with the thinner films.

9.7 SUMMARY

The Variable Frequency Microwave Technique was successfully extended to the MEMS industry as an alternative method for the processing of negative tone SU8 photoresist. The VFM method was compared to the conventional processing method, which utilises a Hotplate, and a hybrid method utilizing both Hotplate and the VFM. A significant increase on the degree of cure was observed using the VFM over the Hybrid and Hotplate curing which means that SU8 curing at lower temperatures or rapid curing is possible. The increase in cure rates can be attributed to a combination of heat transfer and the unique capability of microwave to couple with the sample. Thermal analysis of the cured samples suggested that there is very little difference in the thermal properties of the films cured. Optical studies of the microstructures fabricated suggest that films that have a degree of cure of <60% resulted in poor quality microstructures. The VFM was found to achieve satisfactory microstructures at most of the temperatures used compared to only the high temperatures for the Hotplate and Hybrid methods, although these last were prone to microscopic cracking most probably due to the low solvent content and high degree of curing. An optimisation of the VFM process which resulted in the decrease of the softbaking time to alleviate the cracking was successful with microstructures obtained better than those produced by conventional processing. Along with the increase in cure rates, another significant finding in this chapter is the apparent enhancement in solvent evaporation in the films when VFM was used. This phenomenon will be further investigated in the next chapter.
CHAPTER 10

INVESTIGATION OF
RATE ENHANCEMENT
IN MICROWAVE PROCESSING

10.1 OVERVIEW

As a result of the observed phenomena in Chapter 9 during the softbaking of SU8 samples using the VFM, this chapter focuses on investigating the rate of diffusion of the cyclopentanone solvent in SU8 which is essentially a drying mechanism. An experimental methodology was designed and undertaken to investigate the rate enhancement in microwave processing. A comparison of the rate of the solvent evaporation in conventional (hotplate) and under a microwave field is reported and explanations of the results are discussed.

10.2 INTRODUCTION

Moisture content of epoxy systems such as SU8 is an important aspect of its properties as it can adversely affect its performance and reliability. Traditionally, drying of polymeric materials such as epoxy systems is done thermally in conventional ovens, hotplates, etc. and may take long periods of time. In recent times, the increasing need for more efficient and faster alternatives to conventional thermal drying has led to an interest in using microwave energy for drying polymeric materials. Microwave heating offers many advantages over conventional methods including volumetric and selective heating. The difference between microwave heating and conventional heating requires investigation of the mechanism of drying, which can be limited by the process of diffusion.
In order to dry something, the solvent has to be transported to the surface of the material and then evaporated. This chapter looks at the case when the drying rate is limited by the diffusion of the solvent to the surface. Diffusion is the movement of matter from an area of higher concentration to an area of lower concentration and is a result of the kinetic properties of particles of matter. This phenomenon can be described by

$$ F = -D \frac{dc}{dx} \quad \text{Equation 10 - 1} $$

where $F$ is the rate of transfer per unit area, $dc/dx$ is the change in concentration ($c$) over distance ($x$), and $D$ is the diffusion coefficient.

There have been many reports of microwave-assisted diffusion in the literature. Some studies [Mallon, 1997; Nakai et al., 2002] report increased diffusion due to microwave interactions with polar functional groups. Similarly, Levskovsek et al. [1994] also found that microwaves assisted in increasing transport processes in the system they studied compared to conventional heating at comparable temperatures. Gibson et al. [1988] studied the kinetics of the desorption of ethylene oxide from PVC and found an increase in the overall diffusion coefficient due to disruption of the EO/PVC hydrogen bonding resulting in a significant reduction of immobilised diffusant molecules at any instant. The current work investigated enhancement in transport properties in an epoxy system irradiated with microwaves. Microwave-enhanced diffusion can assist by significantly lowering drying times in epoxy systems without increasing the temperature.

**10.3 EXPERIMENTAL**

An experimental schedule to investigate the apparent enhanced diffusion was undertaken and the methodologies are discussed in the following sections.
10.3.1 Sample Preparation

The same commercial high-epoxide functionality SU8-2100 resin formulation used in Chapter 9 was used for the diffusion experiments. The SU8 formulation consisted of approximately 75%w/w epoxy resin in 25% w/w of cyclopentanone solvent as confirmed using TGA. To obtain consistent samples, the SU8 resin was spun onto silicon substrates that were pre-dried at 150°C for 15 minutes prior to spinning. A spin coater was used for applying the resin and a procedure chosen to obtain a film thickness of approximately 260 microns. Therefore the samples had low thermal inertia.

10.3.2 Drying Procedure

Two drying methods were employed to enable comparison of microwave and conventional heating. It was decided that drying in a waveguide setup, which is a single-mode cavity (TE10), would be ideal rather than using the multimode cavity of the MicroCure. The reason for this choice was due to the constant and known electromagnetic field that a single-mode cavity can provide making placement of the sample uncomplicated as the electromagnetic field is not constantly changing like in a multi-mode cavity. Consequently, temperature measurements become easier and sample weight can be measured in-line.

The microwave setup consisted of a voltage-controlled 1kW microwave generator (1) and a 6-port analyser with a built in autotuner (2), which measures incident and reflected power. The system was connected to a circulating water-load (3) to absorb any excess power. The sample was placed in the middle of a TE10 waveguide (4) on a Teflon block (5) to expose it to the peak electromagnetic power. Temperature measurements were obtained using an Infrared non-contact probe from Raytek (6). Control of temperature was done manually by varying the voltage into the generator, thus varying the power introduced into the waveguide. Weight was measured online using an analytical balance (7). A schematic of the setup is shown in Figure 10-1. Weight readings were taken at predetermined intervals, initially every minute for the first 20min and then at gradually increasing intervals.
Conventional drying was undertaken on a programmable hotplate with an aluminium surface. As the weight could not be measured online, the sample was placed on a piece of aluminium and weighed together to minimise heat loss when weighing. Temperature was recorded as before using the same IR probe from Raytek for consistency.

10.4 RESULTS & DISCUSSION

Drying of the spun SU8 samples on Si <100> substrate was undertaken at different temperatures ranging from 60°C to 101°C for conventional and MW drying with results shown in Figure 10-2. A rapid decrease in weight is experienced at the initiation of drying and decreases. If dried for a longer period, the plots are expected to plateau until no solvent is left in the film. By normal diffusion theory, the drying process can be divided into three steps. At the beginning of drying, the solvent evaporates quickly due to the very high solvent level. The diffusion coefficient is large enough to evaporate the solvent from the bottom of the film. During this step, drying is evaporation controlled and the concentration of the solvent within the film is still constant. As the solvent level decreases further, the evaporation rate decreases rapidly and results in a concentration...
gradient within the film and drying becomes diffusion controlled. Finally, the lower solvent level brings upon the slower diffusion rate and the evaporation is eventually stopped.

![Graph of normalised % Solvent vs. Time for Hotplate and MW drying of SU8.](image)

**Figure 10-2:** Graph of normalised % Solvent vs. Time for Hotplate and MW drying of SU8.

Results indicate that SU8 drying behaves as it should under normal diffusion theory for both drying methods from the shape of the plot. As expected lower solvent content and more rapid weight loss are achieved at higher temperatures. In comparison, MW heating shows more rapid weight loss at the start of the process compared to the hotplate dried samples at equivalent temperatures most probably because the sample is heated volumetrically and thus achieves thermal equilibrium more rapidly. At the drying temperature of 60°C there is only a slight deviation in the drying curves for both MW and HP which suggests that drying is still evaporation limited at this temperature for the duration of the drying time. As the temperature increases, the deviation between MW and HP increases during the diffusion controlled stage indicating that microwaves have an effect during this stage of the drying.
The initial slopes of the drying curves suggest that microwaves enhance diffusion at the start of the process when solvent content is high. However, a plot of final solvent Concentration vs. Temperature (Figure 10-3) shows very little difference in the final solvent concentration, which suggests that they reach the same endpoint suggesting that the form of heating does not alter the final thermodynamic state, only the transport properties.

![Figure 10-3: Graph of Final Solvent Content vs. Temperature for Hotplate and MW drying of SU8.](image)

It can be shown that for one-sided drying as used in these experiments, the diffusivity can be given by

$$D_e = I^2 \frac{dM}{dt}$$  \hspace{1cm} \text{Equation 10 - 2}

where $M$ is the mass of solvent, $t$ is time and $I$ is the thickness of the polymer layer.

Regression techniques were used to determine the Diffusion Coefficient for the two drying methods and results shown in Table 10-1. Calculated results show that irradiating with microwaves leads to an increase of between 75% and 100% of the diffusion.
coefficient over the conventional technique which is significant and would mean that long drying times can be reduced considerably. The increase in drying rate is akin to the findings of other researchers who studied drying rates of other polymeric materials [Gibson et al., 1988].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$D_c$ – Hotplate (10⁻⁹m²/s)</th>
<th>$D_c$ – Microwave (10⁻⁹m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>0.99</td>
<td>2.06</td>
</tr>
<tr>
<td>70</td>
<td>1.22</td>
<td>2.16</td>
</tr>
<tr>
<td>75</td>
<td>1.40</td>
<td>2.70</td>
</tr>
<tr>
<td>95</td>
<td>1.99</td>
<td>3.90</td>
</tr>
</tbody>
</table>

Table 10-1: Average calculated values of the Diffusion Coefficient ($D_c$) at various temperatures.

The dependence of diffusion on temperature in liquids and solids is governed by the Arrhenius equation. Thus an equation of the form of Equation 10 - 3 can be established

$$D_c = A e^{\frac{-\Delta E}{RT}} \quad \text{Equation 10 - 3}$$

where $T$ is the temperature, $R$ is the Gas Constant (8.3144 Jmol⁻¹K⁻¹) and $D_c$ is the diffusion coefficient. The pre-exponential, $A$, is the “free diffusion” coefficient that would arise if no local potential well existed to delay the random walk. Finally, $\Delta E$ is the activation energy required for a molecule to jump out of the local potential well and undertake a random step to the next site [Gibson et al., 1988].

An Arrhenius plot of the logarithm of Diffusion Coefficient versus Inverse Temperature illustrates a reduction in the activation energy when microwaves were utilised as shown in Figure 10-4. The corresponding constants for the Arrhenius plot are tabulated in Table 10-2.
An analysis of variance was undertaken on the data collected and confirms the results are significantly different with a p-value of <0.001. This means that there is less than one chance in a thousand that the two sets of data (MW and HP) come from the same distribution. The standard deviation of the \( \Delta E \) measurement is about 20% and the \( \ln(A) \) about 10%. The data here suggests that both \( \Delta E \) and \( A \) have changed between the two different drying techniques. However, additional data is required to establish this with certainty, as the limited data here could be interpreted as only a change in \( A \) (with a 14% probability).

A decrease in \( \Delta E \) suggests that the energy required for a molecule to undergo diffusion is reduced. Furthermore, a decrease in the pre-exponential factor, \( A \), suggests that microwave irradiation has also decreased the mean free path of solvent molecules within
the system. The results above are consistent with results obtained by Gibson et al. [1988] who also found a reduction in the activation energy and an increase in diffusion in a polymeric system they were studying.

The analysis here is similar to that of Loupy [2002], who summarises work on increased reaction rate constants with microwaves. This and similar work has been the subject of controversy, which could be explained if microwave enhanced transport processes were the rate-limiting step.

### 10.5 POSSIBLE MECHANISM FOR RATE ENHANCEMENT

There is an abundance of reports which have demonstrated the advantages of microwave processing over thermal processing. Advantages include enhanced polymerisation rates, increased mechanical properties and improved product quality among many others. These so called ‘Microwave Effects’ have caused much debate and polarisation of views in the microwave community where the anomalous results have been commonly attributed to molecular hotspots, inverted temperature gradients or inaccurate temperature measurements due to the lack of a proven mechanism [Deam, 2006]. These enhancements may be due to one or more potential microwave effects at the molecular level which includes increased molecular temperature, enhanced segmental vibration, creation of ‘energetic species’ and preferential alignment of dipolar segments. The latter is quite interesting and is the basis of the possible mechanism put forward for the enhancement in transport properties observed in the drying of liquid systems such as SU8.

The mechanism being put forward to explain the enhancement in transport properties under microwave radiation is based on the Cage model by Zwanzig [1983]. Zwanzig stipulates that the rate of transport on a molecular scale can be determined by assuming individual molecules are caged by its nearest neighbours and the rate at which the molecule can hop out of its cage determines the diffusion coefficient. Mathematically, this can be expressed as
\[
D_c = \frac{kT}{M} \int_0^\infty \rho(\omega) \frac{\tau}{1 + \omega^2 \tau^2} d\omega
\]

Equation 10 - 4

where \(\rho(\omega)\) is the density of vibrational states of the molecule in the potential well of the cage that the molecule is located, and \(\tau\) is defined as the hopping time constant which is the effective potential seen by the molecule to remove itself from the cage and is dependent on temperature. A diagrammatic representation of the model is given in Figure 10-5.

![Diagram](10-5.png)

**Figure 10-5:** The Cage Model arrangement under a) normal conditions and b) under a microwave field.

The above representation considers a group of molecules surrounding a molecule which is ‘caged’. The cage represents the average inter-atomic potential and is represented by the dotted line. In the model, the molecule has certain probability of hopping out of the cage as shown by the arrow. Under normal conditions, molecule orientation is random as illustrated in Figure 10-5a. To remove itself from the cage, the molecule must break away from the binding forces of its own site and squeeze between neighbours to hop out and diffuse. In liquids, the surrounding molecules offers elastic (shear) resistance to such a distortion i.e. attempt to hop out, but this resistance relaxes as similar movements occur nearby. Under a microwave field the average inter-atomic potential is modified as shown.
in Figure 10-5b. The cage experienced by the molecule in the centre is still present, but because the molecules are all responding to the alternating electric field, they have a long range alignment correlation that was not present without the microwave field. This leads to the average potential of the cage having valleys or openings through which the caged molecule is more likely to pass, thus resulting in enhanced diffusion.

The mechanism put forward is believed to be the underlying mechanism for the microwave enhanced diffusion that was observed in this thesis and maybe applicable to similar findings reported in literature. Further studies are required to validate this model.

10.6 SUMMARY

The experiments presented here have shown that microwaves can enhance diffusion of cyclopentanone in the SU8 system over thermal drying. Increases of 75-100% on the diffusion coefficients were observed which means that long drying times could be reduced significantly, if drying is diffusion limited. Further analysis found that the activation energy, $\Delta E$, was lower under microwave irradiation suggesting that the energy required for a molecule to undergo diffusion is reduced. Furthermore, a decrease in the pre-exponential factor, $A$, suggests that microwave irradiation has also decreased the mean free path of solvent molecules within the system. A mechanism based on the Cage Model by Zwanzig [1983] was put forward to explain the increase in transport rates. The proposal is based around the concept of microwave irradiation altering molecular orientation which increases the chance of a caged molecule to hop out of the cage thus increasing diffusion.
CHAPTER 11

CONCLUSION

AND

FUTURE WORK

11.1 OVERVIEW

The Variable Frequency Microwave Technique is a new processing method which while still in its infancy, has the capabilities to overcome many of the obstacles that conventional fixed frequency microwave processing has faced. Hence, the thrust of this research was to investigate and characterise this new technology with respect to heating uniformity and to advance its use into industry by applying it to a real world application, which is both problematic and will benefit from the uniqueness of the VFM technology. In this case, VFM was investigated as an alternative processing method for photosensitive SU8, a negative tone photoresist widely used in the MEMS industry. This final chapter of the thesis outlines the specific contribution of the research undertaken and future work arising from the findings.

11.2 SUMMARY OF FINDINGS

The research work was undertaken in accordance with the aims stated at the very beginning of this project. During the course of the research program, a number of important development and findings were made with respect to the VFM technique and its application to SU8 processing as well as observation of apparent “Microwave Effects” which is quite a controversial topic within the microwave processing community. The following are the major outcomes of this work.
11.2.1 The Variable Frequency Microwave Technique

The Variable Frequency Microwave Technique was investigated by studying the energy distribution in a commercial VFM from MicroCure Corporation (MicroCure VFM 2100) using a variety of modelling and experimental methods. An analytical model was devised to quantify the energy distribution in a VFM cavity and was compared to a model using a commercial electromagnetic software package (CST Microwave Studio) and was validated using experimental heat-mapping techniques. Analysis showed that a vast improvement in heating uniformity could be obtained in multimode cavities as compared to the conventional fixed-frequency microwave processing when employing the VFM technique. Several parameters can be varied in the VFM technique (frequency, bandwidth, sweeperate and central frequency) and were investigated and quantified using thermal imaging techniques to enable better selection of these parameters. Results show that the choice of frequency whether be it the central frequency or the frequency during fixed frequency processing has the greatest effect on heating uniformity in the VFM cavity. The choice of bandwidth and sweeperate had little effect on the heat uniformity but are an integral and essential part of the VFM technique to obtain uniform heating.

Of the VFM parameters, the sweeperate parameter was further investigated, as it was potentially the best parameter to optimise and further improve the heating uniformity in the VFM cavity without physically redesigning the VFM cavity. A 2D model of the VFM was modeled and two algorithms to produce even heating were compared. A non-linear sweep-rate regime was developed and compared to the current linear sweep. The non-linear sweep is based upon weighing the sweeperate, the time spent launching a particular frequency, depending on the frequency and was found to improve heat uniformity.

Overall, the VFM technique was found to be very versatile. Even with no prior knowledge of the dielectric properties of a material, the VFM can quickly determine the best possible operating bandwidth samples to be processed by using the cavity characterisation function of the MicroCure VFM 2100. Thus, with little modification to the system, the VFM can be setup for the processing of a host of different materials.
11.2.2 VFM as an Alternative Processing Tool for Photosensitive SU8

Significant work was undertaken to determine the feasibility of VFM as an alternative to the conventional curing on hotplates for the SU8 photoresist. Initial investigations on the dielectric properties of SU8 and its components as well as the Si substrates were undertaken at different frequencies to determine their response to MW energy. Following this, a study of how the VFM parameters affect the samples was carried out and it was found that the choice of the parameters, particularly microwave power and frequency greatly affect processing and based on this, an optimised VFM recipe for the processing of SU8 was established.

The VFM technique was used for the processing of SU8 and was compared to the conventional heating (Hotplate) and a hybrid (Hotplate/VFM) method. Thermal analysis was carried out on the cured SU8 samples with very positive results towards VFM processing. Thermal analysis of cured samples suggests that more thermally stable films can be obtained using VFM technology than by conventional means.

The VFM technique consistently achieved higher degree of cures than the hotplate and hybrid method whilst the hotplate constantly had the lowest degree of cure for the same temperature. Thus, suggesting that microwave energy had a beneficial effect on the processing. Dielectric studies of the sample materials suggest that substrate heating as one of the main contributors to sample heating however it is more likely that a combination of substrate heating and dielectric heating is the reason for the increase in cure rates. The increase in cure rates means that SU8 can be processed at lower temperatures thus alleviating internal stress due to the mismatch of CTE between the SU8 film and the Si substrate and/or decreasing cure times of an otherwise lengthy cure. Furthermore, microstructures fabricated using the VFM technique compared very well against those processed using conventional and hybrid means. VFM succeeded in satisfactory microstructure fabrication at temperatures as low as 75°C which was not possible using the Hotplate or Hybrid methods. Apart from severe out-gassing experienced due to rapid heat up using microwave energy during the first trials of VFM
curing, microstructures obtained were of very good resolution with very little signs of cracking, swelling or structure failure, after proper optimisation of the VFM technique.

One hurdle the VFM faces is the initial cost outlay of a VFM oven. At over AU$100,000 for a unit such as the one used in this research project, it may be difficult to justify its cost when the material it will be used for is a low-cost material. In this case, it may be more economical to employ the use of mechanical means (i.e. mode stirrers or turntables) to obtain heat uniformity.

### 11.2.3 Rate Enhancement in Microwaves

The existence or non-existence of the so-called “Microwave Effect” is still one of the most debatable topics in the Microwave processing field. In this research work, cure rate enhancement was observed during SU8 curing using the VFM and an apparent increase in solvent evaporation was experienced using VFM compared to conventional Hotplate or Hybrid processing. The latter effect was further investigated and it was found that there is an apparent rate increase in the evaporation of the solvent from SU8 under a microwave field. It is proposed that this is due to microwave enhancement of the diffusion of the cyclopentanone in the SU8 epoxy resin.

The mechanisms for the observed “microwave effect” such as the ones observed in this work are not fully understood and there is considerable debate whether even they do exist or are the result of anomalies due to experimental errors. Despite this, a mechanism based on the Cage Model by Zwanzig was put forward to explain this phenomenon. This proposal is based on the ability of preferential alignment of dipolar segments in sample materials which is unique to microwave processing and is believed to be the principal mechanism for the microwave enhanced diffusion observed in this work.

### 11.3 PROPOSED FUTURE WORK

As a result of the research study undertaken, new ideas and questions have arisen that are out of the scope of this study but are worthy of further studies. These pertain to the VFM
technique, the VFM software, SU8 processing and the Microwave Effect. The following are a summary of proposed future work.

The use of a commercial VFM in this project proved to be very useful. Although the current version of the MicroCure VFM 2100 software has been a vast improvement on previous versions especially with regards to temperature control, further improvement could be made by incorporating a function that can vary the type of sweeping so that the non-linear sweep rate proposed in this research work could be integrated into the control system. Another software improvement could be the automatic analysis of the Reflectance vs. Frequency curve obtained during the Cavity Characterisation function of the VFM software so that the best frequency range could be used. Software changes should also be made to allow constant analysis of the reflected power so that frequencies resulting in high reflection of microwave power will be recognised and automatically omitted during frequency sweeping. Therefore these frequencies will not be unnecessarily introduced into the cavity consequently saving time and power and preventing unnecessary emergency shut down of the system as experienced earlier in this work. This can be achieved using similar feedback loops such as used to control the temperature. As the VFM technology is in its infancy, the full implication to industry is still unknown. Apart from advantages in terms of product quality, other manufacturing concerns related to production throughput, factory space, inventory levels as well energy requirements are also issues that can be addressed by VFM technology and should also be further investigated.

One of the most significant outcomes of this study has shown that the VFM technique is a viable alternative to SU8 processing. The increased curing and drying rate observed when VFM was utilised suggest that processing at lower temperatures and rapid processing of SU8 is possible, thus further studies should be undertaken in this area as it would result in significant time and energy savings. Additional work should also be undertaken on the material properties of VFM cured SU8 films. In particular, examination should be undertaken to quantify the effects of VFM radiation on important mechanical properties such as residual stress, tensile strength and adhesion quality; optical properties such as birefriengence and refractive index; and chemical properties
such as chemical stability and moisture uptake. The study of the material properties is important for the VFM to be fully accepted as an alternative processing method not just for SU8 but also for other polymer systems that are designed to be cured via conventional means.

It was shown that the use of microwave radiation in the processing of polymers such as SU8 offers a number of unique possibilities related to the idea that microwave energy is coupled directly to the material. As previously mentioned, curing rates of SU8 was observed to be enhanced under the VFM technique. Although it is still not clear what causes this enhancement, the use of equipment such as the Microwave Calorimeter developed in UMIST in the UK [Nesbitt et al., 2004; Navapour et al., 2006] can give an insight into the kinetics of SU8 curing using this microwave based calorimeter. The other microwave effect observed in this study is the enhanced drying rates of SU8 under the VFM method. As it is not yet possible to measure temperature at the atomic level, indirect measures could be used such as the measurement of viscosity, which is directly related to temperature. Furthermore, it has been suggested in this research work that if microwaves affect diffusion, other transport properties may also be affected and hence should also be investigated. Finally, further studies should be invested in the validation of the Cage Model as presented in Chapter 10, which is thought to be the underlying mechanism for the microwave enhanced diffusion that was observed in this thesis.

11.4 CONCLUSION

The Variable Frequency Microwave Technique was investigated and characterised and was found to be a versatile tool which overcomes the inherent difficulty of obtaining uniform heating using the conventional fixed-frequency microwave processing in multimode cavities. Modelling and experimental studies provided an insight on how the VFM technique works and a greater understanding of how each VFM parameter affects the resulting heating uniformity was achieved. With this knowledge, the VFM was optimised and investigated as an alternative processing technique to negative-tone SU8 photoresist used in the MEMS industry, and was a significant part of this study.
The results from this work indicate that the Variable Frequency Microwave Technique is a viable alternative to the conventional cure currently used in practice. With proper optimisation of the VFM with respect to parameters such as frequency bandwidth and temperature, VFM was found to provide samples that are comparable or better than conventionally cured samples in terms of properties and microstructure quality. Using the VFM method, enhancement in cure rates and drying rates, which are described by others as “microwave effects”, were observed and investigated.

Outcomes of this work prove that the microwave effect is a benefit to microwave processing of materials albeit not being fully understood. The observed microwave effect has had a positive effect on the work undertaken herein and the proposed Cage Model to explain the observations is believed to be quite valid and promising. It is anticipated that the work undertaken in this study will renew interest in the topic and provide a new path in which new research in this area can follow.
REFERENCES


APPENDIX A

LIST OF PUBLICATIONS & PRESENTATIONS

A.1 JOURNAL ARTICLES AND CONFERENCE PROCEEDING


A.2 CONTRIBUTIONS TO TEXT BOOKS ON PHD RELATED WORK


A.3 RESEARCH PRESENTATIONS

APPENDIX B

MATLAB PROGRAM FOR MODE CALCULATION AND ENERGY DISTRIBUTION IN THE VFM

The following program calculates the modes and the resulting energy distribution in a Variable Frequency Microwave with cavity dimensions 365mm x 355mm x 482mm as presented in Chapter 7.

% Initiation

close all           % closes all windows opened during the previous runs
clear all           % clears the memory from the results of previous runs
clc                  % clears screen

% VFM specifications

a=0.365;    % length (x) of cavity 0.365m (n)
b=0.482;    % height (y) of cavity 0.482m (m)
d=0.355;    % depth (z) of cavity 0.355m (l)
fmin=2.5E9;    % minimum frequency
fmax=8E9;    % maximum frequency

% Setup Surface Matrix

step=50;
x0=0; xf=a;    % setup time range
da=(xf-x0)/step;   % time-step
x=[x0:da:xf];    % time matrix – from t0 to tf in intervals of dt

y0=0; yf=b;    % setup time range
db=(yf-y0)/step;   % time-step
y=[y0:db:yf];    % time matrix – from t0 to tf in intervals of dt

z0=0; zf=d;    % setup time range
dd=(zf-z0)/step;   % time-step
z=[z0:dd:zf];    % time matrix – from t0 to tf in intervals of dt

[X,Y,ZZ]=meshgrid(x,y,z);
E=[X,Y,ZZ];

% Constants

c=3*10^8;    % speed of light
\[
\begin{align*}
\text{kmin} & = \frac{2\pi}{f_{\text{min}}/c}; \\
\text{kmax} & = \frac{2\pi}{f_{\text{max}}/c}; \\

% \text{Setup n-vector} \\
\text{ni} & = 0; \quad \% \text{initial n} \\
\text{nf} & = 20; \quad \% \text{final n} \\
n & = [\text{ni}:1:\text{nf}]; \quad \% \text{n-vector – from mi to mf in intervals of 1} \\

% \text{Initialise matrices and functions} \\
Z & = []; \quad \% \text{Z matrix} \\
\text{count} & = 0; \quad \% \text{initialise counter} \\
\text{tic;} & \quad \% \text{start timer} \\

% \text{Mode calculation} \\
\text{for l=0:nf} \\
\quad \text{for m=0:nf} \\
\quad \quad \text{fnml} = c*\left(((l/(2*d))^2) + ((m/(2*b))^2) + ((n/(2*a))^2)\right)^{1/2}; \\
\quad \quad \% \text{calculate mode} \\
\quad \quad Z = [Z;fnml]; \quad \% \text{store each harmonic as a row in a matrix for retrieval} \\
\quad \quad \text{count} = \text{count} + 1; \quad \% \text{count} \\
\quad \text{end} \\
\text{end} \\

k = (2\pi/c).*Z; \\
Kcount = 0; \\
\text{Anm} = 1; \\
N = 10; \\
E & = [];
E2 & = \text{zeros(length(x),length(y),length(z))}; \\
Eadd & = \text{zeros(length(x),length(y))}; \\

\text{for l=0:nf} \\
\quad \text{for m=0:nf} \\
\quad \quad \text{if kmin} \leq (k((l*(nf+1))+(m+1),n+1)) \leq (k((l*(nf+1))+(m+1),n+1)) \leq \text{kmax} \\
\quad \quad K(m+1,n+1,l+1) = k((l*(nf+1))+(m+1),n+1); \\
\quad \quad K2((l*(nf+1))+(m+1),n+1) = k((l*(nf+1))+(m+1),n+1); \\
\quad \quad \text{Kcount} = \text{Kcount} + 1; \\
\quad \quad E = ((\text{Anm}))/((K(m+1,n+1,l+1)^N))^{2})*((\sin ((n*pi)/a)).*((\sin ((m*pi)/b)).*Y)).*((\sin ((l*pi)/d)).*ZZ)).^{2}; \\
\quad \quad \text{else } E(:,:,)=0; \end{align*}
\]
end
E2=E+E2;
end
end
end
for l=0:step
    Eadd=E2(:,:,l+1)+Eadd;
end
tfinal=toc

% Write data to file
csvwrite('3D-20N10-tfinal.dat',tfinal)
csvwrite('3D-20N10-Eadd.dat',Eadd)
csvwrite('3D-20N10-E2.dat',E2)
csvwrite('3D-20N10-Kcount.dat',Kcount)
csvwrite('3D-20N10-Kb2.dat',K2)
csvwrite('3D-20N10-Kb.dat',K)
csvwrite('3D-20N10-Ka.dat',k)
APPENDIX C
MICROCURE 2100 VARIABLE FREQUENCY MICROWAVE RECIPES

C.1 SOFTBAKE RECIPE FOR SU8 PHOTORESISTS

Event Steps List

Vari Freq = 5.2500 GHz, 5.5000 GHz, 0.1 Secs
Power Output = 30 Watts, Active
RF On
Control Parameters, Integral = 10 Clamp = 10
Auto Ramp = 65 Degs C, 0.07 D/S, 0:0:0 HH:MM:SS
Power Output = 50 Watts, Active
Wait Temp = 65 Degs C
Set Temp = 65 Degs C, 1 Degs C, 0:7:0 HH:MM:SS
RF Off
Vari Freq = 5.2500 GHz, 5.5000 GHz, 0.1 Secs
RF On
Control Parameters, Integral = 5 Clamp = 30
Auto Ramp = 95 Degs, 0.07 D/S, 0:0:0 HH:MM:SS
RF Off
RF On
Power Output = 100 Watts, Active
Wait Temp = 97 Degs C
Set Temp = 97 Deg C, 2 Degs C, 1:0:0 HH:MM:SS
RF Off
Auto Ramp = 33 Degs, 0.02 D/S, 0:0:0 HH:MM:SS
Wait Temp = 33 Degs C

C.2 POST EXPOSURE BAKE RECIPE FOR SU8 PHOTORESISTS

Event Steps List

Vari Freq = 5.2500 GHz, 5.5000 GHz, 0.1 Secs
Power Output = 30 Watts, Active
RF On
Control Parameters, Integral = 10 Clamp = 10
Auto Ramp = 65 Degs C, 0.07 D/S, 0:0:0 HH:MM:SS
Power Output = 50 Watts, Active
Wait Temp = 65 Degs C
Set Temp = 65 Degs C, 1 Degs C, 0:1:0 HH:MM:SS
RF Off
Vari Freq = 5.2500 GHz, 5.5000 GHz, 0.1 Secs
RF On
Control Parameters, Integral = 5 Clamp = 30
Auto Ramp = 95 Degs, 0.07 D/S, 0:0:0 HH:MM:SS
RF Off
RF On
Power Output = 100 Watts, Active
Wait Temp = 97 Degs C
Set Temp = 97 Deg C, 2 Degs C, 0:15:0 HH:MM:SS
RF Off
Auto Ramp = 33 Degs, 0.02 D/S, 0:0:0 HH:MM:SS
Wait Temp = 33 Degs C
APPENDIX D

HEAT TRANSFER IN THIN SU8 PHOTORESISTS FILMS

The following analysis determines how rapidly thermal equilibrium is achieved in a 260µm SU8 photoresist. The time required to attain thermal equilibrium time can be described by a characteristic time, $\tau$, determined by the relationship,

$$\tau \sim \frac{L^2}{\gamma_{film}}$$

Where $L$ is the SU8 film thickness and $\gamma_{film}$ is the thermal diffusivity. If we consider $\gamma_{film}$ as being the low end of the scale ($1 \times 10^{-6}$), then the characteristic time to heat up is

$$\tau \sim \frac{(0.26 \times 10^{-6})^2}{1 \times 10^{-6}}$$

$$\therefore \tau = 0.0676 \text{ seconds}$$

$$\approx 68ms$$

Therefore, it can be assumed that thermal equilibrium is reached quite rapidly, compared with the time taken by the experiment (minutes).