UNIVERSITI PUTRA MALAYSIA

FREE-SPACE MICROWAVE CHARACTERISTICS OF NATURAL RUBBER COMPOSITES FILLED WITH CARBON BLACK FOR MICROWAVE APPLICATION

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A contactless and non-destructive microwave method has been developed to characterize natural rubber composites from reflection and transmission measurements made at normal incidence. Microwave non-destructive testing (MNDT) using free-space microwave measurement (FSMM) system involves measurement of reflection and transmission coefficient in free-space. The measurement system consist of a pair of spot focusing horn lens antenna, mode transition, coaxial cables and vector network analyzers (VNA). The inaccuracies in free-space measurements are due to two main sources of errors. 1) Diffraction effects at the edges of the material specimen. 2) Multiple reflections between horn lens antennas and mode transitions via the surface of the sample. The spot-focusing antennas are used for minimizing diffraction effects and we have implemented free-space TRL calibration technique by establishing three standards, namely, a through connection, a short circuit connected to each port and a transmission line connected between the test ports. This calibration along with time domain gating feature of the VNA can eliminate effects of multiple reflections.

In this method, the free-space reflection and transmission coefficients, S_{11} and S_{21} are measured for natural rubber composites sandwiched between two Teflon plates of 10.64 mm thickness which act as a half-wave transformer at mid-band. The actual reflection and transmission coefficient, S_{11} and S_{21} of the natural rubber composites are then calculated from measured S_{11} and S_{21} by using ABCD matrix transformation in which the complex permittivity and thickness of the Teflon plates are known. From the complex permittivity, loss tangent, conductivity, wavelength, velocity and skin depth can be obtained. Result for natural rubber composites filled with different concentrations of carbon black are reported in frequency range 9-11 GHz.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains.

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Fakulti: **Kejuruteraan**

Penyelidikan yang menggunakan kaedah gelombang mikro yang tidak menyentuh dan memusnahkan sampel telah dicipta untuk mengenalpasti ciri-ciri elektrik dan bukan elektrik yang ada didalam sebatian getah asli dengan menggunakan pengukuran pembalikan dan penghantaran pada laluan terus.

Kaedah gelombang mikro ini telah menggunakan pengukuran koefisien pembalikan dan penghantaran dalam ruang 'free-space'. Sistem pengukuran terdiri daripada sepasang horn antenna, pengalih mod, kabel sepaksi dan Vector Network Analyzer (VNA)

Ketidaktetapan dalam pengukuran dalam diruang 'free-space' adalah disebabkan dua sebab utama 1) Kesan pembelauan pada pinggir sample 2) Terlalu banyak pembalikan signal yang melalui sampel diantara dua horn antenna. Antena fokus telah digunakan untuk meminimakan pembelauan signal dan kami juga telah menggunakan kaedah'free-space' penentukur TRL. Kaedah penentukur ini akan digunakan bersama 'time domain gating' daripada VNA untuk mengurangkan kesan pembalikan signal.

Dalam kaedah 'free-space' pembalikan dan penghantaran koefisien ini S_{11} dan S_{21} telah digunakan untuk mengenalpasti ciri-ciri elektrik dan bukan elektrik sebatian getah asli. Sampel sebatian getah asli ini diletakkan diantara dua kepingan Teflon yang bertindak sebagai pemegang kepada sampel dan sebagai laluan 'half-wave'. Koefisien sebenar S_{11} dan S_{21} dapat dikira dengan menggunakan ABCD matrik transformasi apabila ciri-ciri elektrik , bukan elektrik dan ketebalan Teflon diketahui. Daripada pengukuran tersebut, ciri, kadar pengaliran elektrik, panjang gelombang dan kelajuan gelombang didalam sampel dapat diketahui. Kesemua keputusan yang diperolehi untuk sebatian getah asli dengan konsentrasi karbon yang berlainan ini adalah pada had frekuensi 9-11 GHz.

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LIST OF ABBREVIATIONS

ε^*	-	Complex Permittivity
ε	-	Complex Permittivity
μ^*	-	Complex Permeability
μ	-	Complex Permeability
ω	-	Angular Velocity, rad/s
σ	-	Conductivity
α	-	Attenuation Constant
γ	-	Propagation Constant
β	-	Phase Constant
δ	-	Skin Depth
λ	-	Wavelength
S_{11}	-	Reflection Coefficient
S_{21}	-	Transmission Coefficient
HAF	-	Carbon Black N330
SMR	-	Standard Malaysian Rubber
NR	-	Natural Rubber
pphr	-	Part per hundred rubber
MNDT	-	Microwave Nondestructive Testing
FSMM	-	Free-Space Microwave Measurement
TRL	-	Thru, Reflect, Line
LRL	-	Line, Reflect, Line
FSMS	-	Free-Space Measurement System
VNA	-	Vector Network Analyzer
DUT	-	Device under Testing
IF	-	Intermediate Frequency
RF	-	Radio Frequency
Γ	-	Complex Reflection Coefficient
$\lvert\Gamma\rvert$	-	Magnetic of Reflection Coefficient
GHz	-	Giga Hertz

CHAPTER 1

INTRODUCTION

1.1 Introduction

The measurement of complex permittivity of materials at microwave frequencies can be performed in many ways. Waveguide methods are popular, where the sample is precisely machined to fit inside the waveguide. Rectangular and coaxial waveguides are used, rectangular samples are easier to produce than coaxial ones, and however they can only be used over a limited frequency range. Coaxial waveguide allows extremely wide-band measurements but sample preparation is difficult. A vector network analyzer is generally used to collect the reflected and transmitted signals, and permittivity value can be extracted from the results.

Waveguide measurements suffer from errors caused by incomplete filling of the waveguide by the sample. Corrections for this have been proposed with some success but are not perfect. Sample preparation is destructive and often time consuming. Some materials are not suitable for waveguide measurements, such as those containing long fibers, or foams with large voids. High frequency measurements are especially difficult due to waveguide size. Free space techniques overcome many of these problems [1].

The main objective of this project is to develop a free space measurement system at microwave frequencies in the frequency range of 8 GHz to 12 GHz (X-band) for a measurement of Reflection Coefficient, S_{11} and Transmission Coefficient, S_{21} of the

Natural Rubber filled with different concentration of Carbon Black (HAF N330). The Complex Permittivity (ε^*) is then calculated from the measured values of S_{11} and S_{21}.

A Free-Space Microwave Measurement (FSMM) method is a non-destructive and contactless; hence, it is suitable for measurements of the complex electric permittivity of the rubber composites under high-temperature conditions. In the past, free-space methods have been used by several investigators for the measurement of electrical properties of materials [1]-[4]. Recently, a new free-space method was developed for the accurate determination of the dielectric constant and loss tangent of non magnetic materials such as Teflon and quartz at microwave frequencies using reflected fields from metal-backed samples [3]. This paper presents an extension of the free-space method for simultaneous measurement of complex permittivity of non magnetic materials at microwave frequencies using reflected and transmitted fields.

FSMM method is used because the reflection and the transmission coefficients (S-parameters) of planar specimens of rubber composite materials are measured in free-space instead of coaxial line method. The errors in free-space measurements are presumed to be due to diffraction effects at the edges of the sample and multiple reflections between the horn lens antennas and the mode transitions. Diffraction effects at the edges of the sample are minimized by using spot-focusing horn lens antennas as transmitters and receivers. In addition, a free-space TRL (Through-Reflect-Line) calibration technique which eliminates errors due to multiple reflections is developed in the research work.

A conventional method for ε* measurement consist of measuring complex reflection coefficients of short-circuited and open-circuited (quarter wavelength short-circuited line) samples [2]. Using this method, the measurements are difficult and time consuming to make because the open- circuited sample has to be established at each measurement frequency. In this project, a method for determination of ε* from reflection and transmission coefficients of a planar sample is used. This method is especially suitable for quick, routine, and broad-band measurement of ε* and of high-loss materials. For materials with dielectric (or magnetic) loss tangent less than 0.025, the loss factor measurements are found to be inaccurate because of errors in reflection and transmission coefficient measurements[11].

In this method, the free-space reflection and transmission coefficients, S_{11} and S_{21} of a planar sample are measured for a normally incident plane wave. The complex constitutive parameter ε* is calculated from the measured S_{11} and S_{21}. For flexible samples of non-magnetic materials, the accuracy of measurement of S_{11} and S_{21} is poor because of sagging of the sample when mounted on the sample holder. Hence, the sample had to be sandwiched between two half-wavelength (at midband) Teflon plates, to eliminate the effect of sagging. The actual reflection

and transmission coefficients, S_{11} and S_{21} of the sample are calculated from the measured S_{11} and S_{21} of the Teflon plate-sample-Teflon plate assembly from knowledge of the complex electric permittivity and thickness of the Teflon plates.

The key components of the measurements system consists of transmit and receive spot-focusing horn lens antenna, and the vector network analyzer. Because of the far-field focusing ability of horn lens antennas free-space measurement can be made at microwave frequency in a relatively compact and simple measurement setup

1.2 Scope of Work

The scopes of work that have been studied and done are listed below:

1.2.1 Study of Measurement Equipment and Dielectric Properties

The study is including the basic theory on microwave to improve skills and knowledge in doing a calibration and choose a better method to given a better result for the calibrations. This study is also including the basic theory on the rubber materials to give some ideas.

1.2.2 Choose Type of Measurement Technique

The Free Space Measurement System (FSMS) at microwave frequencies in the frequency range 8 GHz to 12 GHz was chosen to measure the Reflection Coefficient, S_{11} and Transmission Coefficient, S_{21} of the natural rubber filled with different concentration of carbon black.

1.2.3 Calibration using Chosen Measurement Technique

Implement two-port TRL (Through-Reflect-Line) calibration technique along with time domain-gating to remove the effect of multiple reflections.

1.2.4 Develop Computer Programs

To develop computer programs for the calculation of complex permittivity of the composite.

1.2.5 Analysis of results

Analyze the data using FORTRAN Software to reduce the effect of Teflon permittivity on the Reflection Coefficient S_{11} and Transmission Coefficients S_{21} of the samples.

The Fortran 77 programming is used to determine the complex permittivity ε^* of the sample by using measured S_{21} only.

1.3 Objectives

1. To measure dielectric properties of natural rubber with different concentration of carbon black using microwave non-destructive technique.

2. To establish a correlation between the dielectric properties of natural rubber composites evaluated from microwave non-destructive technique with the electrical properties.

3. To develop computer programs for the calculation of complex permittivity.

1.4 Thesis Organization

To achieve the objectives mentioned earlier, a microwave non-destructive measurement system using free-space method has been developed. Natural rubber with various parameters were cast and prepared.

The parameters are the concentration of the carbon black as filler and the thickness of the samples. The system was used to measure the electromagnetic properties of natural rubber with different concentration of carbon black in frequency range 8-12 GHz. The effects of the carbon black concentrations to the electromagnetic properties of natural rubber were investigated.

Finally, the relationship between the electromagnetic properties and the basic properties was established.

Chapter two describes the theory of microwaves which are related to this measurement and investigation. It discuss the S-parameters, two-port network, transmission line and electromagnetic properties of the sample.

Chapter three discuss about the theory of natural rubber and the effect of the carbon black to the dielectric materials.

Chapter four focuses on the experimental program. It describes the material used in this research and the methods of the testing. Also, this chapter describes the measurement system developed for the purpose of this research, calibration system and methods of calculating the properties of the composites from measured reflection and transmission coefficient.

Data analysis and results together with their interpretation are presented in chapter five. Chapter six summaries the findings , provides conclusions and future development.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The first free-space method for measurement of dielectric constant using transmission technique was proposed by R.M Redheffer in 1966 [4],who reported that free-space methods are non-destructive and contactless techniques which are suitable for dielectric measurement of material.

Harrold L.Basset [5] was the first researcher to measure complex permittivities of fused silica as a function of temperature from ambient to 2500 ^{O}C in free-space employing spot- focusing horn lens antennas at a frequency at 9.4 GHz [2],[3]. This technique has been used for dielectric, reflection and transmission coefficient measurements of material such as cement, bricks, textile, wood and human skin in the frequency range of 8-40 GHz by Ghodgaonkar and others [24],[25].

A free-space method by Ghodgaonkar and others [3] was developed for accurate measurement of dielectric constants and loss tangents of metal-back planar samples in microwave frequencies. For low-loss materials the accuracy of this method is better than ± 2% and 20 x 10^{-4} for dielectric constants and loss tangents measurement respectively.