STUDY OF DIELECTRIC PROPERTIES OF FILMS USING MICROWAVE TECHNIQUES

by

R. NATARAJAN

Thesis submitted to the Indian Institute of Technology, Delhi
for the award of the Degree of
DOCTOR OF PHILOSOPHY

Department of Physics
Indian Institute of Technology, Delhi

October, 1975

ACKNOWLEDGEMENT

I wish to express my deep sense of gratitude to Dr. D.C.Dube for his invaluable guidance, kind encouragement and keen interest during the course of this work.

I also wish to express my gratefulness to Dr. A.Bhaskara Rao (ECIL, Hyderabad) for his help in the early stages of the work.

My sincere thanks are due to Prof. M.S.Sodha and Prof. A.K. Ghatak (Head of Physics Department) for their interest and encouragement.

I am indebted to Prof. K.L.Chopra for many stimulating and valuable discussions and for his generous help in the preparation of thin films.

I also take this opportunity to sincerely thank Dr. V.K.Tripathi for his personal touch, inspiration and valuable suggestions at various stages of the work. I am thankful to Prof. S.K.Garg (NRC, Ottawa) for many helpful discussions and to Dr. Ashutosh Singh, Dr.A.C.Rastogi, Dr. D.K. Pandya and Dr. Krishna Kumar for their cooperation.

I also thank Mr. T.N.Gupta for his efficient typing of the manuscript with care and interest.

The financial assistance rendered by the Council of Scientific and Industrial Research, India and by the Director, I.I.T., Delhi are gratefully acknowledged.

PREFACE

Recent advances in the state of the art of both integrated circuit technology and solid state devices have led to the integration of microwave circuits. Dielectric films find applications in a variety of devices as well as in integrated circuits and a knowledge of the parameters of the films at microwave frequencies is essential for circuit design and material evaluation.

Conventional techniques for the measurement of complex permittivity of materials in bulk at microwave frequencies have been extensively reviewed by several authors. In general, the basic measurement techniques can be divided into four categories. These are:

(a) Standing Wave Techniques, in which standing wave patterns formed by filling section of the waveguide with the material are studied.

(b) Reflection Techniques, wherein the waves reflected from the specimen are studied and a measurement of the reflection coefficient leads to the determination of the parameters of interest.

(c). Transmission Techniques, wherein waves transmitted through the material are investigated.

(d) Cavity resonance Techniques, in which resonance is observed in a partially or completely filled cavity and the corresponding change in resonant frequency because of filling, is made use of for the determination of dielectric parameters.

The final choice of technique, or combination of techniques among the above mentioned will depend on frequency, the value of complex permittivity and loss tangent availability of the quantity and form of the material and the accuracy of results required.

The methods of dielectric measurement listed above though have proved very useful but they have their own limitations. Method (b) is subject to considerable errors in measuring the complex reflection coefficient as the dielectric constant becomes large and so is the case with method (c) when the measurement of complex transmission coefficient is involved. Methods (a) and (d) are commonly employed for making measurements on bulk samples; they are extremely difficult to be performed satisfactorily for materials in the form of thin films. This is mainly because of the lack of strong interaction of the rf signal with the specimen. To achieve accuracy in the measurements with the above methods, the specimen thickness should normally be a sizable fraction of the wavelength ($\sim \lambda/4$ and its multiples in most cases). The details are discussed in chapter I.

In the recent past, attempts have been made to study thinner samples by using specific forms of cavities and other forms of sample holders. With the objective of providing a quick, handy and accurate method for the measurement of dielectric properties of films at microwave frequencies, a different technique has been proposed in the present thesis. In this method, the specimen in the form of a thin long strip, or film deposited on it, is put longitudinally at the centre of a hollow rectangular waveguide excited in the normal dominant TE_{10} mode. The surface of the film is parallel to the narrow wall of the waveguide in much the same way as in an ordinary vane type attenuator.

In this geometry, by virtue of being positioned in the maximum electric field, the interaction of specimen with the microwave field in the guide is quite strong. Thus, the film impedance changes the propagation characteristics of the wave to a measurable degree.

One of the advantages of the present method is that it does not impose any restriction on the length of the sample, unlike in cavity resonance methods. Thus by choosing proper lengths depending on material characteristics, measurable phase shift and attenuation can be obtained. Solutions of Maxwell's equations have been discussed to obtain the dispersion relation in the appropriate geometry. The complex dielectric constant can be calculated from the measured phase shift and attenuation introduced by the specimen using the given expressions.

Experimental results on two materials, namely Mylar and Teflon confirm the ease and accuracy in the determination of dielectric parameters for films by mounting the specimen longitudinally at the centre of the waveguide. The standard two channel technique (Bridge Technique) used in the above measurements has been discussed in details. Further, some numerical analytical results have also been given to show the variation of phase shift and attenuation as a function of film thickness for various dielectric parameters.

Also, another circuit arrangement is proposed for the measurement of phase and attenuation using a slotted line, retaining the geometry of the film in the waveguide as above. Experiments have been performed on Mylar and Teflon films for a range of thicknesses from 75 to 250 microns. The experimental results are in good agreement with the accepted values (within $\sim$2%). This experimental set-up is simpler than the previous one and it does not require calibrated components such as calibrated variable phase shifter and variable attenuator, unlike in the conventional bridge method. However, the accuracy in this method is limited due to the general problems of measurement with slotted line such as the possibilities of higher mode generation and power leakage through the slot. These are the contents of the second chapter.

The rest of the three chapters have been devoted to the study of electrical properties of different substances of varying characteristics and conductivities ranging from highly insulating films (polyvinyl chloride) to highly conducting thin films (aluminium and gold) using the proposed experimental technique.

In chapter III, dielectric properties of pure, iodine doped and metal ion doped polyvinyl chloride (PVC) films are discussed. Measurements have been made at X-band and at K-band frequencies. The loss behaviour of these films as a function of temperature are studied. The results obtained have been explained within the framework of the existing theories.

In chapter IV, the electrical properties of a semiconducting material, namely silicon wafers have been discussed. An important aspect in the construction and fabrication of semiconductor devices is to have an intimate knowledge of the electrical properties of semiconductors; conductivity being an important property among them, has been chosen for investigation in these microwave measurements.

Microwave methods for the study of electrical properties of semiconductors that have been developed in recent years are found to have certain advantages over dc methods. Making use of the proposed technique, the complex permittivity and microwave conductivity of silicon wafers of different resistivities have been studied. Measurements have also been made on the temperature variation of the conductivity of these samples.

In chapter V, studies made on thin semimetal and metal films are reported. As far as the semimetal films are concerned, the choice fell on bismuth, which is a popular semimetal since the properties of thin films are quite different from those of the bulk, it is of interest to have measurements on thin films. Also, the skin depth of bismuth is of the order of few microns for microwaves; therefore microwaves would be most suitable for the investigation of thin films.

Thin bismuth films were prepared by vacuum evaporation, deposited on mica substrates and varied in thickness from 240 - 720 angstroms. The real and imaginary parts of the complex permittivity (ϵ_r and ϵ_i) have been calculated from the measured phase and attenuation constants. High values for ϵ_r and ϵ_i are obtained as expected because of free carrier effects. The permittivity data has been used for the computation of other transport parameters like relaxation time of the charge carriers in the films.

The microwave behaviour of thin metal films has not been studied much so far. Different though related, studies such as the optical and dc properties on metal films have been made; nevertheless the study of collective behaviour of free charge carriers would necessitate the microwave investigations of these films. Thin films of gold and aluminium ($\sim$80 to 200 angstrons thick) were obtained by evaporation on mica substrates and were investigated in the same manner as bismuth films.

The work has resulted in the following publications:

1. "Determination of Dielectric Parameters for Films at Microwave Frequencies", D.C.Dube and R. Natarajan, J.Appl.Phys. 44, 4927 (1973).

2. "Measurement of the Permittivity of Films at Microwave Frequencies", D.C.Dube and R.Natarajan, J.Phys.E: Scientific Instruments, 7, 256 (1974).

3. "Measurement of Complex Permittivity of Thin Bismuth Films", D.C.Dube and R.Natarajan, paper presented in the Symposium on 'Electronic Measurements and Instrumentation', Bangalore, India (Jan. 1975).

4. "Electrical Behaviour of Pure and Doped PVC Films at Microwave Frequencies", D.C.Dube and R.Natarajan, (To be communicated, 1975).

5. "Complex Permittivity and Microwave Conductivity of Silicon", D.C.Dube and R.Natrajan, (To be communicated, 1975).

6. "Microwave Investigations of Thin Metal Films", D.C.Dube and R.Natarajan, (To be communicated, 1975).

CONTENTS

CHAPTER		Page
	PREFACE	(i)
I	INTRODUCTION AND REVIEW OF MICROWAVE DIELECTRIC MEASUREMENT TECHNIQUES	
	1.1 Introduction	1
	1.2 A Summary of Microwave Techniques for Dielectric Measurement	3
	i) Methods using waves in free space	3
	ii) Cavity resonance methods	4
	iii) Methods using guided waves (Transmission method)	5
	1.3 Measurement Techniques for the Dielectric Constant of Films	8
	REFERENCES	11
II	PROPOSED TECHNIQUE FOR THE MEASUREMENT OF DIELECTRIC PARAMETERS OF FILMS	
	2.1 Introduction	13
	2.2 The Evaluation of the Phase and Attenuation Constants	14
	2.3 Experimental Verification	31
	2.3.1 Method I: Bridge method	31
	2.3.2 Method II: Standing wave ratio method	38
	REFERENCES	45
III	DIELECTRIC BEHAVIOUR OF PVC FILMS AT MICROWAVE FREQUENCIES	
	3.1 Introduction	46
	3.2 Experimental Details	48
	3.2.1 Preparation of pure and doped PVC films	48
	3.2.2 Measurement of dielectric parameters at different temperatures at X-band (8.2 - 12.4 GHz)	49

3.2.3 Measurements at K-band frequencies (18-26 GHz) 53

3.3 Results and Discussion 54

3.4 Conclusions 76

REFERENCES 78

IV DIELECTRIC AND CONDUCTION BEHAVIOUR OF SILICON FILMS

4.1 Introduction 80

4.2 Experimental Details 81

4.3 Results and Discussion 83

4.4 Conclusions 95

REFERENCES 97

V STUDY OF DIELECTRIC BEHAVIOUR OF HIGHLY CONDUCTING FILMS

5.1 Introduction 99

5.2 Experimental Details 100

5.2.1 Preparation of films 100
5.2.2 Measurement details 105

5.3 Results and Discussion 106

5.4 Conclusions 116

REFERENCES 117