

UNIVERSITI TEKNOLOGI MARA

**MICROWAVE NON-DESTRUCTIVE TESTING
OF SEMICONDUCTOR MATERIALS**

NOOR HASIMAH BABA

MSc

April 2005

UNIVERSITI TEKNOLOGI MARA

**MICROWAVE NON-DESTRUCTIVE TESTING
OF SEMICONDUCTOR MATERIALS**

NOOR HASIMAH BABA

**Thesis submitted in fulfillment of the requirements
for the degree of
Master of Science in Electrical Engineering
Faculty of Electrical Engineering**

April 2005

ACKNOWLEDGEMENTS

My deepest praise to Allah s.w.t. for giving me the strength and good health to pursue and complete my Masters thesis.

It is with great sincerity to acknowledge my supervisors, Associate Professor Dr. Zaiki Awang and Associate Professor Dr. Deepak Kumar Ghodgaonkar for giving me their support and encouragement during the duration of my study. This work would not have been possible without their guidance and input.

I wish to express my sincere gratitude to the management of Microwave Technology Center, where this work was carried out, for their assistance and for allowing me to carry out this investigation. I would like to thank Pn. Najiha Tamyis, Ms. Azlinda Ramli, En. Aziz Aris, En. Ermeey, En. Faisa Muhamad, En. Wan Hamdan and others who had helped me throughout the course of this project - without their invaluable help this project could not have been completed. I would also like to extend my sincere appreciation to Professor Dr Burhanuddin Yeop Majlis of Microelectronics Institute Laboratory, U.K.M., Bangi for providing the four-point probe method.

Special thanks are owed to my husband and children for their support and understanding from the very beginning of my work until my studies were completed.

TABLE OF CONTENTS

TITLE	i
ACKNOWLEDGEMENT	ii
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF PLATES	xi
SYMBOLS	xii
ABBREVIATIONS	xv
ABSTRACT	xvi
1.0 INTRODUCTION	1
1.1 Introduction	1
1.2 Problem Statement	4
1.3 Objectives	6
1.4 Scope of Research	7
1.5 Organization of Thesis	8
2.0 OVERVIEW OF MATERIALS CHARACTERIZATION METHODS	9
3.0 FUNDAMENTALS OF MICROWAVES AND SEMICONDUCTOR MATERIALS	16
3.1 Theory of Microwave	16

3.2	Advantages and Disadvantages of Microwave Testing	16
3.3	Electromagnetic Properties of Material	18
3.3.1	Dielectric Polarization	19
3.4	Electromagnetic (EM) Fields in Media	24
3.5	Complex Permittivity	26
3.6	Propagation of EM Waves in Media	27
3.6.1	Propagation of EM Waves in Lossless Medium	28
3.6.2	Propagation of EM Waves in Lossy Medium	29
3.6.3	Skin Depth in Lossy Medium	30
3.7	Scattering Parameters (S-parameters)	30
3.8	Semiconductor Materials	32
3.9	Crystal Structures and Orientation	34
3.10	Silicon Wafer Processing	35
3.11	Doping In Semiconductor	36
3.12	Conductivity	37
3.13	Effect of Doping on Polarization	39
4.0	RESEARCH METHODOLOGY	42
4.1	Introduction	42
4.2	Theoretical Consideration	44
4.2.1	The Quarter-Wave Transformer	44
4.2.2	Transmission Method	47
4.2.3	Short Circuit Method	56
4.3	Sample Specifications	60

4.4	Measurement Systems and Calibration	62
4.4.1	Free-Space Measurement Set-up	62
4.4.2	Rectangular Dielectric Waveguide Measurement Set-up	67
4.4.3	Calibration Procedure	71
4.4.4	Measurement Procedure	79
5.0	ANALYSIS AND DISCUSSION OF RESULTS	81
5.1	Introduction	81
5.2	Effect of Quarter-wave Transformer	81
5.3	Comparison Between Theory and Experiment	83
5.4	Comparison between Free-space and RDWG Methods	87
5.5	Results of Dielectric Properties	91
5.6	Results of Electrical Properties	97
5.7	Four-Point Probe Method	100
5.8	Skin Depth	102
5.9	Accuracy of Measurements	103
5.10	Summary of Findings	105
6.0	CONCLUSIONS	106
6.1	Conclusion	106
6.2	Future Research	108
	REFERENCES	110

APPENDIX A: Derivation of ABCD Matrix for Lossy Transmission line.

- APPENDIX B: Derivation of forward transmission coefficient S_{21}^c for calculation of complex permittivity.
- APPENDIX C: The Transmission Line Terminated By Arbitrary Load, Z_L
- APPENDIX D: The Muller Method [46]
- APPENDIX E: A Fortran program for calculation of complex permittivity by transmission method
- APPENDIX F: A Fortran program for calculation of complex permittivity by short circuit method.
- APPENDIX G: A Fortran program for removing effect of quarter-wave transformer for transmission method.
- APPENDIX H: Tables of Measurement Results
- APPENDIX I : A Fortran program for calculation of forward reflection and transmission coefficients from theoretical model.
- APPENDIX J: List of Publications

LIST OF TABLES

Table 3.1	Elements in the Periodic Table That Form Common Semiconductors.	33
Table 3.2	Elemental and Compound Semiconductors.	33
Table 4.1	Specifications for silicon wafers	61
Table 5.1	Magnitude and Phase for S_{11} with and without a quarter-wave transformer of 8-inch diameter Si wafer (SD2).	82
Table 5.2	Magnitude and Phase for S_{21} with and without a quarter-wave transformer of 8-inch diameter Si wafer (SD2).	82
Table 5.3	Doping concentration of silicon wafers.	99
Table 5.4	Comparison between microwave resistivity and dc resistivity.	101
Table 5.5	Error analysis for ϵ' and ϵ'' measurements at 10 GHz.	104

LIST OF FIGURES

Figure 3.1	Frequency response of permittivity and loss factor for a hypothetical dielectric showing various contributing phenomena [32].	23
Figure 3.2	S-parameters relationships	31
Figure 3.3	An illustration of valence electrons in covalent bonds (a) in the absence of an applied field (b) in the presence of an applied field [39].	40
Figure 3.4	An illustration of ionic polarization between Si and P ions (a) in the absence of an applied field and (b) in the presence of an applied field [39].	40
Figure 4.1	The quarter-wave matching transformer.	45
Figure 4.2	Typical reflection characteristics of a quarter-wave transformer [35].	47
Figure 4.3	Schematic diagram of Teflon TM -silicon- Teflon TM assembly	47
Figure 4.4	Plot of the magnitude of S_{11} of the Teflon TM quarter-wave transformer	51
Figure 4.5	Schematic diagram of planar sample.	51
Figure 4.6	Flowchart for finding complex permittivity (ϵ^*).	55
Figure 4.7	Schematic diagram of a short circuit method.	56
Figure 4.8	Schematic diagram of free-space measurement set-up.	63
Figure 4.9	Model of RDWG and sample.	68
Figure 4.10	Schematic diagram of Rectangular Dielectric Waveguide measurement set-up.	69
Figure 4.11	Schematic diagram of the error model [51].	71
Figure 4.12	Signal flow graphs in the forward and reverse directions. The dotted line defines DUT, while RP1 and RP2 are the calibration planes [51]	72
Figure 4.13	The magnitude and phase of S_{21} of a through connection used as verification element.	76

Figure 4.14	The magnitude and phase of S_{11} of a reflect connection used as verification element.	76
Figure 4.15	The complex permittivity for Teflon TM .	78
Figure 4.16	The complex permittivity for PVC.	78
Figure 4.17	The flow chart of the experiment.	79
Figure 5.1	Measured and calculated magnitude of S_{11} for sample SN15 sandwiched between two quarter-wave plates.	85
Figure 5.2	Measured and calculated phase of S_{11} for sample SN15 sandwiched between two quarter-wave plates.	86
Figure 5.3	Measured and calculated magnitude of S_{21} for sample SN15 sandwiched between two quarter-wave plates.	86
Figure 5.4	Measured and calculated phase of S_{21} for sample SN15 sandwiched between two quarter-wave plates.	87
Figure 5.5	Comparison of dielectric constant between free-space and RDWG methods for 8-in doped silicon wafers	88
Figure 5.6	Comparison of loss factor between free-space and RDWG methods for 8-in doped silicon wafers	89
Figure 5.7	Comparison of loss tangent between free-space and RDWG methods for 8-in doped silicon wafers	89
Figure 5.8	Comparison of conductivity between free-space and RDWG methods for 8-in doped silicon wafers	90
Figure 5.9	Comparison of resistivity between free-space and RDWG methods for 8-in doped silicon wafers	90
Figure 5.10	Comparison of skin depth between free-space and RDWG methods for 8-in doped silicon wafers	91
Figure 5.11	The dielectric constant ϵ' versus frequency for different silicon wafers using transmission method.	92
Figure 5.12	The dielectric constant ϵ' versus frequency for different silicon wafers using short circuit method.	93
Figure 5.13	The loss factor ϵ'' versus frequency for different silicon wafers using transmission method.	93
Figure 5.14	The loss factor ϵ'' versus frequency for different silicon wafers using short circuit method.	94

Figure 5.15	The loss tangent versus frequency for different silicon wafers.	96
Figure 5.16	The conductivity versus frequency for different silicon wafers.	97
Figure 5.17	The resistivity versus frequency for different silicon wafers.	99
Figure 5.18	Schematic of four-point probe method [64].	101
Figure 5.19	The skin depth versus frequency for different silicon wafers.	102

LIST OF PLATES

Plate 1	A photograph of the silicon wafers used in this study	61
Plate 2	A photograph of the free-space measurement system with a) spot-focusing horn lens antennas, b) mode transitions and precision coaxial cables. The sample holder located between two antennas is shown in c).	64
Plate 3	A photograph of the rectangular dielectric waveguide measurement system with rectangular rod antennas, mode transitions and precision coaxial cables. The sample is placed between the two antennas (inset).	70

SYMBOLS

V	Voltage
I	Current
C	Capacitance
Q	Charge
A	area of the plates of the capacitor
P	Polarization
\overline{D}	electric flux density (Coulombs/meter ²)
\overline{E}	electric field intensity (Volt/meter)
\overline{H}	magnetic field intensity (Ampere/meter)
\overline{J}	current density (Ampere/meter ²)
\overline{B}	magnetic flux density (Webers/meter ²)
\overline{M}	magnetic current density (Volt/meter ²)
ρ	electric charge density (Coulombs/meter ³)
f	Frequency
χ_e	electric susceptibility
ϵ_0	permittivity of free space ($= 8.854 \times 10^{-12}$ Farad/meter)
μ_0	permeability of free space ($= 4\pi \times 10^{-7}$ Henry/meter)
ϵ	Permittivity
ϵ^*	complex permittivity
ϵ'	real part of complex permittivity
ϵ''	imaginary part of complex permittivity (loss factor)
μ	Permeability
μ^*	complex permeability
u	velocity of wave
ϵ_c''	dielectric losses due to ionic conductivity
ϵ_d''	dielectric losses due to dipolar polarization
ϵ_e''	dielectric losses due to electronic polarization
ϵ_a''	dielectric losses due to atomic (ionic) polarization
ϵ_i''	dielectric losses due to interfacial polarization
∇	del operator
$\frac{\partial}{\partial t}$	partial derivative with respect to time
j	$\sqrt{-1}$

π	pi (= 3.14)
c	speed of light (= 2.998×10^8 meter/sec)
σ	Conductivity
τ	relaxation time
e	electronic charge (= 1.60×10^{-19} Coulombs)
n	Electron
p	Hole
μ_e	mobility of electron
μ_h	mobility of hole
E_{loc}	local electric field
E	electric field
N	molecules per unit atom
α_T	molecular polarizability
g	ratio between local electric field and electric field
n_i	intrinsic carrier concentration
R_s	sheet resistance
λ_0	wavelength in free-space
λ_m	wavelength in the material
α	attenuation constant
β	phase constant
ω	angular frequency
γ	propagation constant
δ	skin depth
d	sample thickness
l	distance between parallel-plate capacitor
α_e	electronic polarization
α_i	ionic polarization
S_{11}	forward reflection coefficient
S_{21}	forward transmission coefficient
S_{11}^m	measured forward reflection coefficient
S_{11}^c	calculated forward reflection coefficient
S_{21}^m	measured forward transmission coefficient
S_{21}^c	calculated forward transmission coefficient
θ_{11}	forward reflection coefficient phase

θ_{21}	forward transmission coefficient phase
TE_{10}	transverse electric mode
Z_L	load impedance
Z_{in}	input impedance
Z_0	characteristic impedance
Γ	reflection coefficient
Γ_L	reflection coefficient at the load
T	transmission coefficient
a_1	forward incident wave
a_2	reverse incident wave
b_1	forward scattered wave
b_2	reverse scattered wave
E_{DF}	forward directivity error
E_{SF}	forward source match error
E_{XF}	forward isolation error
E_{TF}	forward transmission tracking error
E_{RF}	forward reflection tracking error
E_{LF}	forward load match error
E_{DR}	reverse directivity error
E_{SR}	reverse source match error
E_{XR}	reverse isolation error
E_{TR}	reverse transmission tracking error
E_{RR}	reverse reflection tracking error
E_{LR}	reverse load match error

ABBREVIATIONS

AC	alternating current
DC	direct current
CW	continuous wave
FM	frequency-modulated
EM	Electromagnetic
PVC	Polyvinyl-chloride
VNA	vector network analyzer
NDT	non-destructive testing
DUT	device under test
LRL	line-reflect-line
TRL	thru-reflect-line
MHz	Megahertz
GHz	Gigahertz
RF	radio frequency
IC	intergrated circuit
MNDT	microwave non-destructive testing
RDWG	rectangular dielectric waveguide
m	Meter
ns	Nanosecond
in	Inch
sec	Second
mm	Millimeter

ABSTRACT

A contactless and non-destructive microwave method has been developed to characterize silicon semiconductor wafers from reflection and transmission measurements made in free space at normal incidence. The measurement system consists of a pair of spot-focusing horn lens antenna, mode transitions, coaxial cables and a vector network analyzer (VNA). Two methods were developed namely the transmission and the reflection methods. From the complex permittivity, the resistivity and conductivity of Si wafers can be obtained. Results for p-type and n-type doped silicon wafers are reported in the frequency range of 8.5 to 12.5 GHz. The measurements were conducted in the frequency domain.

The research found that, the dielectric constant of the silicon wafers are relatively constant, varying slightly over the frequency range. As the frequency increased, the loss factor of the wafers decreased. The dielectric losses were the ionic and electronic losses due to dipoles orientation. The loss factor, loss tangent and conductivity of the doped wafers are higher than the undoped type. The conductivity of silicon wafers increases with frequency. Despite lower doping concentration, the n-type wafers showed the highest conductivity due to the higher mobility of electrons compared to holes. There is no significant difference between the AC and DC resistivity of the silicon wafers. This could be due to the non-polar property of the wafers. The skin depth of the silicon wafers decrease with increasing conductivity. This technique will be a major contribution to the semiconductor industry since it is accurate, quick, broadband, contactless and non-destructive. This is highly desirable since the silicon wafer is brittle, fragile and requires high purity level.

CHAPTER ONE

INTRODUCTION

1.1 Introduction

Microwave frequencies occupy a part of the electromagnetic spectrum extending from 300 MHz to 300 GHz [1]. Applications of microwaves in various areas have been extensive and varied particularly in telecommunications and radar. The most popular application of microwave power is in microwave ovens for domestic and commercial cooking. Industrial applications of microwave power include applications in industries such as food, rubber, chemicals, plastics, textiles, building materials, oil, coal and pharmaceuticals. Others include continuous moisture-content monitoring, monitoring of geometrical dimensions and determination of solid and liquid levels.

The ability of microwaves to penetrate most dielectric materials, and their relatively short wavelengths make them suitable for non-destructive measurements (a contactless, free-space technique). Since the penetration of microwaves in good conducting materials is very small, microwave non-destructive testing (MNMT) techniques are mainly used for non-metallic materials. The spatial resolution of these techniques depends on the wavelength of the wave. For the commonly used microwave band of 3 to 100 GHz, the wavelength varies from 100 mm to 3 mm. Major studies of the dielectric properties were done in the microwave range since this method is non-invasive and non-destructive - practically no sample preparation is required. These techniques have advantages over other non-destructive methods (such as radiography, ultrasonics and eddy current) by way of low cost, good

penetration in non-metallic materials, good resolution and contactless feature of the microwave sensor.

Complex permittivity and permeability are determined by the material's molecular structure, so they can be related to other properties of interest as well. Measuring them can provide critical insight to applications in many industries. It can be useful in all stages of a product's lifecycle: design, incoming inspection, process monitoring, and quality assurance. For example, it can provide important information about materials used in state-of-the-art radio frequency (RF) and microwave electronic components. Even biomass, bulk density, bacterial content, and chemical concentration can be related to a material's electromagnetic properties.

The choice of substrate material is important for high frequency integrated circuit design. The substrate must be a semiconductor material to accommodate the fabrication of active devices. Besides substrate thickness and strip width, substrate permittivity is another important parameter for high frequency IC design. Characteristics such as complex permittivity (dielectric constant, loss tangent and loss factor) and resistivity must be evaluated because the design of these circuits rely critically on these parameters. The dielectric properties of materials dictate to a large extent the response of the devices when subjected to RF or microwave fields. In addition to polarization effects, at microwave frequencies, these properties may change significantly due to radiation loss or other spurious effects such as electromagnetic coupling, thus posing problem to the IC designers. Therefore, knowledge of these properties will contribute significant understanding and ultimately will assist high frequency IC designers.

For this reason, silicon semiconductor wafer is chosen as the sample in this study. Silicon is one of the most common substrate for high frequency ICs. The wider bandgap of silicon results in electronic devices that are capable of operating up to around 200 °C. Silicon, in contrast to germanium, readily accommodates itself a stable passivation layer on the surface by forming silicon dioxide (SiO₂), which provides a high degree of protection to the underlying devices [2]. In addition, silicon wafer can be considered a perfect planar sample due to its single-crystal property, which have a high degree of regular geometric periodicity throughout the entire volume of material and are capable of being cleaved at precise planes [3]. Thus is suitable for this technique which allows reflection and transmission measurements for normal incidence.

To date, various versions of microwave measurement techniques have been reported for the characterization of semiconductors but none have used free-space method, which is contactless and non-destructive [4-8]. This research presents a free-space method for measurement of complex permittivity of semiconductor materials at microwave frequencies using reflection and transmission techniques. It thus yields values of microwave permittivity and microwave conductivity simultaneously. Since contacts are not used, the methods have advantages over other conventional conductivity measurements method. The system consists of a pair of spot-focusing horn lens antenna, mode transitions, coaxial cables and a vector network analyzer (VNA).

The inaccuracies in free-space measurements are due to two main sources of errors; diffraction effects at the edges of the specimen and multiple reflection between horn

lens antennas and the sample. The spot-focusing antennas are used for minimizing diffraction effects while free-space TRL (thru, reflect, line) calibration method implemented on VNA along with time domain gating feature of the VNA reduces errors due to multiple reflections.

1.2 Problem Statement

An important consideration in the construction of semiconductor devices is the control and measurement of the electrical properties of the material. The semiconductor transport properties such as permittivity, resistivity, conductivity and mobility must be evaluated since at microwave frequencies these properties may change significantly due to dielectric loss or other undesired spurious effects such as electromagnetic coupling thus posing problems for high frequency IC designers. At microwave frequencies, the dielectric loss is associated with formation of electric dipoles due to electronic and ionic polarizations. These dielectric losses have contributions to the permittivity and conductivity of semiconductor materials.

Much information about the fundamental processes whereby electrons in semiconductors are scattered (i.e. make spontaneous transitions from one quantum state to another) is contained in transport properties such as conductivity and mobility. Currently, the measurement of these properties with direct current has been very useful in the study of different mechanisms, which cause transitions between the various stationary states. The ac transport properties, which are obtained by microwave measurement differ from the dc properties by having real and imaginary parts. This added information reflects many details relevant to the scattering processes, thus can be an extremely useful means of studying the detail scattering

mechanisms. Knowledge of scattering effects is also important to high frequency IC designer since the conduction mechanism affects circuit losses and contribute to noise. This is the primary advantage of characterizing semiconductors with microwaves rather than with dc.

In contrast to NDT, other methods performed using ohmic contacts introduce contact noise. This results in low signal to noise ratio, which hampers the measurement. Furthermore, the use of contacts destroys the sample. Currently, the DC four-point probe method [9] is widely used for the measurement of resistivity in semiconductor material where the probes are in direct contact with the wafer thus inducing probe damage and adding contamination. This occur even at high temperature where contacting probes could react with the semiconductor. Furthermore four-point probe method reveals dc properties only and are thus not much use for high frequency IC design. At high frequencies other physical effects such as surface wave modes add complexity and will give rise to further measurement errors.

Until today, various researches have been carried out to develop characterization tools to evaluate semiconductor materials. Waveguide methods are widely used, where the sample is precisely cut to fit inside a waveguide. In comparing rectangular and circular waveguides, rectangular samples are easier to make than circular ones, but they can only be used over a limited frequency range. Circular waveguide can extend over wide frequency bands but sample preparation is difficult. Errors associated with the waveguide methods are caused by incomplete filling of the waveguide by the sample. In addition sample preparation is destructive and time

consuming. Another microwave method is the cavity resonator. This method gives the conductivity and permittivity from measurements of changes in quality factor and frequency, which result when a sample is inserted into the cavity. Cavity methods are very accurate and can measure loss tangent of very low loss material accurately but it employs only single frequency per cavity.

For complete characterization of materials, a large number of measurements over a wide frequency band are required. This process is obviously time-consuming. Therefore the development of a technique capable of covering a broad frequency range with acceptable accuracy is highly desirable. Free-space microwave probing methods overcome many of these problems. These methods make possible a very accurate, quick, broadband, contactless and non-destructive measurement of materials. They allow sample to be of any shape provided its size is greater than three times the beamwidth of the antenna at the focus. The methods are useful above all for the measurement of plate materials such as semiconductor wafers. Furthermore, measurements by this free-space method can be made without inducing probe damage and adding contamination. These conditions have led to development of a new method of measurement which is fully contactless and non-destructive and can be carried out at low or high temperatures, and in strong magnetic and electric fields.

1.3 Objectives

The main objective of this research is to develop a non-destructive testing technique to measure the complex permittivity of silicon wafers at microwave frequencies. A computer modeling to calculate the reflection and transmission coefficients and to

correlate the dielectric properties of the wafers with the electrical properties is developed.

1.4 Scope of Research

In this work, the magnitude and phase of the reflection and transmission signals were measured. Two free-space methods were developed, namely, reflection and transmission methods.

The reflection method is also known as the short circuit method. In this method, the complex forward reflection coefficient is measured by inserting a perfectly conducting plate behind the silicon wafers at the focus of the lens antenna. By implementing an algorithm which finds the zeros of the error function, the complex permittivities are then calculated from the reflection coefficients.

The transmission method employs a pair of quarter-wavelength impedance transformer to reduce the reflectivity of the sample. By this method, the complex permittivity of semiconductor materials is calculated from the complex transmission coefficient by substituting the values of the impedance transformer. An algorithm was developed which calculates complex permittivities from measured magnitude and phase of the transmission coefficients. This algorithm involves calculation of the actual transmission coefficient due to the impedance transformer and calculation of new complex permittivity guess by using zero finding technique. From the complex permittivity, the resistivity and conductivity can be obtained.