MICROWAVE TEST CHAMBER FOR MEASURING THE RELATIVE PERMITTIVITY OF THIN FILMS

A Thesis
Presented to
The Faculty of Graduate Studies and Research
The University of Manitoba

In Partial Fulfillment
of the Requirements for the Degree
Master of Science
in
Electrical Engineering

by
Harinder Pal Singh Ahluwalia
September, 1970

ACKNOWLEDGEMENTS

I wish to express my gratitude to Dr. W.M. Boerner under whose able guidance the project was conducted. I am pleased to acknowledge my indebtedness to Professor M.A.K. Hamid and Dr. S.S. Stuchly for many fruitful discussions and to Dr. A. Petkau of Atomic Energy of Canada Ltd. for his encouragement and for supplying the test films. I also wish to thank Dr. C.W. Searle of the Physics Department for his assistance in component fabrication and Mr. Bodo Meyer of the Physics Workshop for his skilled and precise machining of the test chamber.

Last but not the least, I am particularly appreciative of the tireless efforts of Miss. A.M. Korol for careful and cheerful typing of this material.

This research was supported by the National Research Council of Canada, the Defence Research Board of Canada and the Faculties of Agriculture and Graduate Studies of the University of Manitoba.

(H.P.S. Ahluwalia)

CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF PHOTOGRAPHS

Abstract

A microwave test chamber for measuring the dielectric constant of thin films on substrates using the cavity perturbation technique is described. Special emphasis is placed on testing biochemical phospho-lipid membranes, whose properties resemble those of living tissues; therefore their rupture by microwave irradiation may determine microwave safety levels. A novel feature of the test chamber is the use of a resonant iris to enhance the field in the aperture where the sample is suspended. It is shown that significant shift in the center frequency and change in the Q factor of the X-band cavity are obtained for films of thicknesses as low as a few hundred microns.

CHAPTER I

INTRODUCTION

The work presented here resulted from a fundamental research on rf safety standards to establish criteria for defining the transition between macro and micro-thermal effects on tissue layers caused by electromagnetic irradiation. The behaviour of such membranes at high power densities is of particular importance since the power density at which they rupture is an indication of the critical level which may determine the safety standard for microwave irradiation [1]. The study is being carried out on artificial phospho-lipid membranes which resemble living tissues very closely in their chemo-physical properties [2]. It may also be possible to electrically bias the two bipolar membrane layers and to create membranes of electro-active properties by proper ion doping of the two bipolar phospho-lipid membrane layers.

Although the basic incentive of this research is to study the rupture of biochemical membranes and their electrical permittivity, the test chamber described in this work may also be used for dielectric measurements of samples of different semiconductor materials. These measurements at microwave frequencies enable one to deduce information regarding the properties of carriers not obtained by dc techniques. Even in those cases where the properties can be determined by dc as well as microwave techniques, the latter have the advantage of not requiring contacts ,thus eliminating the possiblity of altering the sample characteristics during the measurement. It has been shown [3] that for semiconductors, the conductivity measured at microwave frequencies is the same as the dc conductivity but the dielectric constant obtained by microwave measurements differs from the dc value by $\sigma_s \tau/\varepsilon_o$. Here σ_s is the microwave conductivity, τ the momentum relaxation time and ε_o the free space permittivity. Furthermore, measurement of the complex permittivity as a function of frequency and temperature yields information on the scattering mechanisms controlling the transport of charge carriers.

A number of methods for measuring the dielectric constant of semiconductors using substitution, standing wave ratio, transmission bridge, and resonant cavity techniques have been described in the literature [4-7]. However none of these methods is applicable to thin films of thicknesses in the range of a few hundred microns deposited on substrates. It should be noted that the study of microwave properties of thin films of semiconductors has been of increasing importance due to their potential application in microwave devices. Attempts are being made to increase the frequency range of transistors and other semiconductor devices. One of the main limiting factors in the construction of transistors to operate at higher frequencies is the base width which has to be extremely small, thus approximating a thin film. Since the dielectric constant of such thin films is anticipated to be considerably different from the tabulated bulk value, the technique described in this thesis could be suitable for such studies.

The cavity perturbation method has been found suitable for the measurement of the dielectric constant of thin films. The measurement is performed by inserting the sample film into a cavity resonator and determining the properties of the sample from the resultant change produced in the resonant frequency and the quality factor of the cavity. The aim of the present scheme is to create a high electric field in the plane of the sample in order to enhance the changes in the measured quantities. For this the sample is suspended in a resonant iris which is placed at the maximum longitudinal field in a resonant rectangular cavity. The measured shift in the resonant frequency and Q factor of the cavity are utilized in the perturbation equation to determine the complex permittivity.

CHAPTER II

MEASUREMENT TECHNIQUE

A variety of methods for measuring dielectric constants are available [4-8]. Since it is required to measure the dielectric constant of 'thin' films, the conventional methods based on transmission and reflection of power in a waveguide, containing a sample approximately a quarter wavelength long, cannot be employed. In this case the cavity perturbation method, which is highly sensitive and versatile with regard to the type and shape of the sample, is suitable. When the perturbation method is used in conjunction with the quasi static approximation, the accuracy of the results may be surprisingly high. Since the cavity perturbation methods involve approximations in their formulation, the sample to be tested has to satisfy certain conditions. The sample must be very small, compared with the cavity itself, so that the induced frequency shift and change in Q are small compared with the centre frequency and Q of the unloaded cavity and also for the quasi-static approximation to be valid. Our thin films satisfy the above requirements thus justifying the use of the cavity perturbation technique. A great advantage of the perturbation approach is that it is no longer necessary to account for many of the details of the cavity which are the same for the loaded and unloaded cases and hence their effect is cancelled out.

The change in the Q factor and resonant frequency due to the sample are utilized to calculate ε_r using the perturbation equation:

$$\frac{f_o - f_s}{f_s} - j\left(\frac{1}{Q_o} - \frac{1}{Q_s}\right) = (\varepsilon_r - 1)\,\frac{\int_{V_s} \vec{E}_o \cdot \vec{E}_2 \, dv}{\int_{V} |\vec{E}_o|^2 \, dv} \qquad (1)$$

Here f_o and Q_o are the resonant frequency and quality factor for the unloaded cavity, respectively, whereas f_s and Q_s are the corresponding quanitities for the loaded cavity. $\vec{E}_o$ is the transverse electric field in the unloaded cavity volume v_o and $\vec{E}_2$ is the electric field in the sample volume V_s.

When the sample extends over the entire cross-section, and is tangential to the electric field, $\vec{E}_2 = \vec{E}_o$ for small perturbations. However, when the sample is suspended in the iris aperture only, it is more difficult to determine $\vec{E}_2$ with satisfactory accuracy since the field distribution in such a resonant aperture is not available. A resonable estimate for this field may be obtained with the aid of a shape factor γ which is determined experimentally using films with precisely known dielectric constants as discussed later.

There are two general methods for the measurement of Q of a cavity. One method depends on observing the response of the resonator to CW signals in the neighborhood of its resonant frequency and the other involves observing the transient response of the resonator to the sudden application or removal of an exciting signal at or near resonant frequency. The first method includes a number of different measurement techniques such as transmission methods, impedance measurement method and dynamic methods. The transient decay or the decrement method is particularly applicable to high Q cavities because it does not need a high degree of frequency stability required in other types of measurements, but at lower values the decay period is too short for convenient measurement. In the present measurements, the reflectometer method which applies particularly to the single-ended resonators was found to be convenient because it lends itself readily to oscilloscopic presentation and has the advantage of speed.

The reflectometer method simply requires the measurement of the voltage reflection coefficient $|\rho_o|$ at the resonant frequency f_o and at two other frequencies f_1 and f_2, symmetrically spaced about f_o. If there are appreciable losses in the input coupling structure, the reflection coefficient magnitude $|\rho_1|$ far from resonance must also be measured. The Q of the resonator is then given by the relation:

$$Q_L = \frac{\alpha f_o}{\Delta f} \tag{2}$$

where $\Delta f = f_2 - f_1$ (3)

and $$\alpha = \left[\frac{|\rho|^2 - |\rho_o|^2}{|\rho_1|^2 - |\rho\ |^2} \right]^{1/2} \tag{4}$$

CHAPTER III

TEST CHAMBER

The microwave test chamber consists of an X-band rectangular cavity operated in TE_{10n} mode and coupled to the main waveguide through a circular iris. The radius of the iris is such that the cavity is critically coupled to the main waveguide. A sliding short is employed to vary the cavity length as shown in Fig. 2. Two requirements need to be satisfied simultaneously, the first is to create a high electric field in the applicator along the sample and the second is to place the sample in the region where the highest electric field values exist. The need to create a high electric field in the sample arises from the fact that films of a few micron thickness, when suspended in the total cross section of the cavity, induce a very small perturbation in the complex frequency (obtained from the resonant frequency and the Q factor of the cavity). By suspending the films in a resonant iris, and thus enhancing the electric field in the sample, an increase in the change of complex frequency is obtained. The requirements mentioned above are satisfied by placing the resonant iris, containing the sample, an odd multiple number of quarter wavelengths away from the sliding short. Since the frequency perturbation for thin films is generally small, utmost care must be exercised in very accurate machining of the various components of the chamber. Furthermore special care must be taken during the experiment to align the chamber assembly in both transverse and longitudinal directions and ensure accurate positioning of the rectangular iris with and without the sample. This is achieved with the aid of guide pins which are used to align the two sections of the chamber.

Photograph 1 shows the test chamber. It consists of two wave-

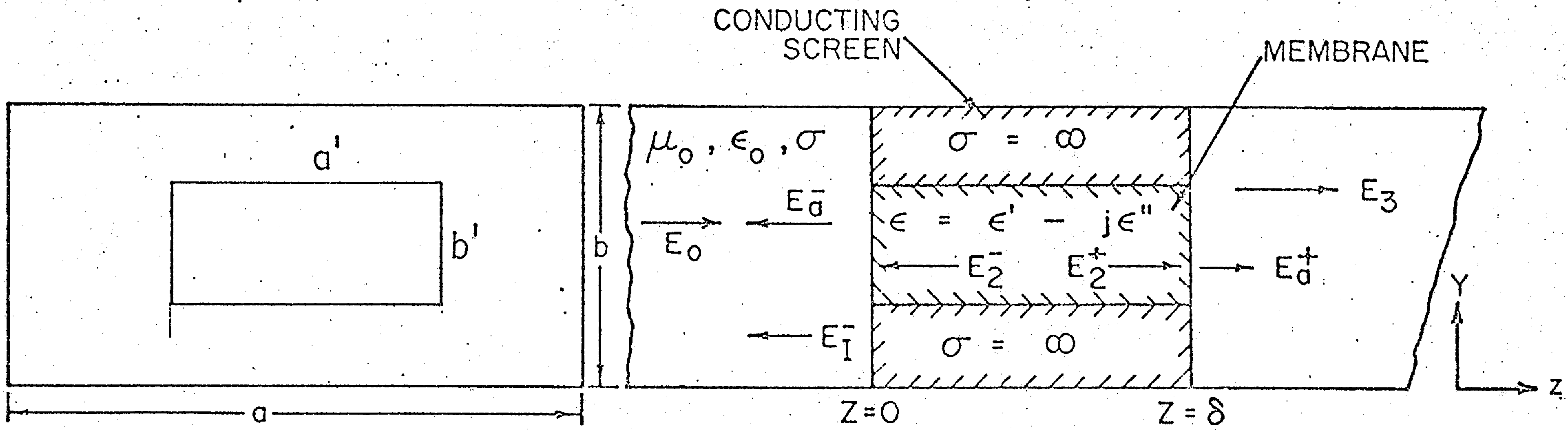


Fig. 1 CONDUCTING SCREEN WITH FILM APERTURE

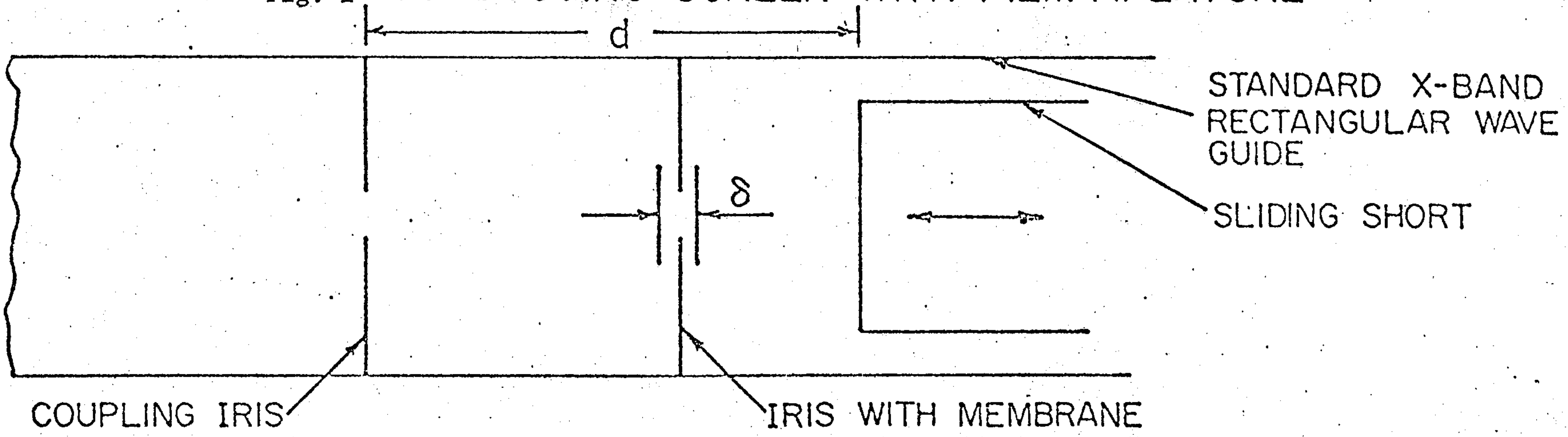


Fig. 2 RESONANT TEST CHAMBER

guide sections, one fixed and the other variable in length. The iris containing a sample in the aperture is placed between the two sections and the three parts are aligned with the help of pins on the fixed section. The cavity is coupled to the main waveguide through a circular iris of radius $\frac{15}{128}''$.

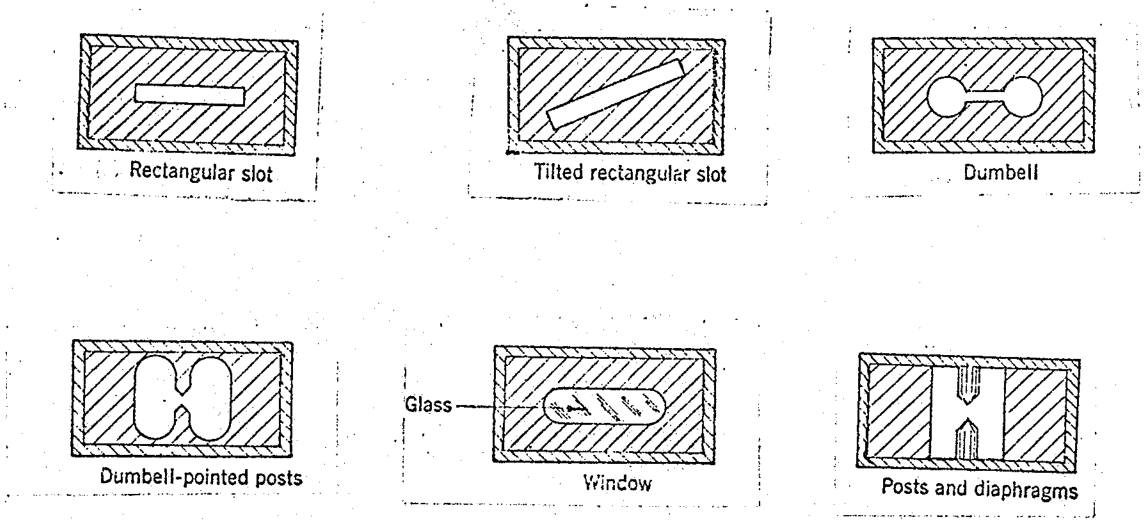
For the sake of convenience in calculations some samples are prepared to extend over the whole cross section of the cavity (i.e. without the iris) while the other samples of the same material are suspended in the resonant iris. This provides a check on the calculated results and allows experimental determination of the shape factor γ associated with the resonant iris as will be discussed in section 3.4.

As has been mentioned in the introduction, the work presented here resulted from fundamental research on rf safety standards which involves the study of the rupture of the phospho-lipid membranes which resemble living tissues in their properties. Although the work described here concerns only measurement of dielectric constant of these membranes, the set up shown in Fig. 6 is also suitable for rupture test. In order to perform the rupture tests, the cavity may be placed in a temperature controlled chamber and connected to a medium power microwave generator and the measurement set up via an electrically controlled waveguide switch which changes the mode of operation from 'measurement' to irradiation.

The Resonant Iris:

3.2 Theoretical Consideration:

A metal diaphragm with a rectangular opening exhibits a susceptance-frequency characteristic similar to that of a parallel resonant circuit shunting the guide. In fact elements of a great variety of shapes may be made to resonate at any microwave frequency depending upon the dimensions of the element. A resonant element of the dumb-bell shape may be made with a number of variations as shown in Fig. 3. Tunable resonant elements can be made using posts, screws etc.



Rectangular slot

Tilted rectangular slot

Dumbell

Dumbell-pointed posts

Window

Posts and diaphragms

FIG. 3 RESONANT IRISES

To understand the behaviour of these elements, it is desirable to calculate the field in the junction so that one can know the dependence of the resonant frequency on the geometrical parameters of the elements, the frequency dependence of the transmission or reflection of power and also the energy dissipated in the element because of currents in the metal posts. Unfortunately, even the simplest resonant element-the rectangular slot-has not been analyzed theoretically to the extent of finding the field in the aperture. The method of attack at present is limited to finding experimentally the equivalent circuit of the resonant element. The equivalent circuit of a resonant iris has been derived in Appendix 2 and is helpful in understanding the behaviour of the iris.

3.3 Design of Rectangular Resonant Iris:

The approximate dimensions of a centrally located window in a rectangular guide operating in a dominant mode are given by the empirical' relation given below [10].

$$\frac{a}{b}\sqrt{1-\left(\frac{\lambda}{2a}\right)^2} = \frac{a'}{b'}\sqrt{1-\left(\frac{\lambda}{2a'}\right)^2} \qquad (5)$$

where a and b are the guide dimensions, a' and b' are the dimensions of the opening (with a' measured parallel to a and b' parallel to b) and λ is free-space wavelength.

The design can be carried out in two ways. In one case the dimension b' is assumed and the corresponding value of a' is calculated for a given value of a, b and λ. The second method depends on a geometrical construction: In the centre of the cross section of the guide, lay out a line of length $\lambda/2$, parallel to larger dimension of the guide, and centered with respect to the walls as shown in Fig. 4. Two hyperbolas are now drawn with their axes parallel to the guide walls, passing through the ends of this line

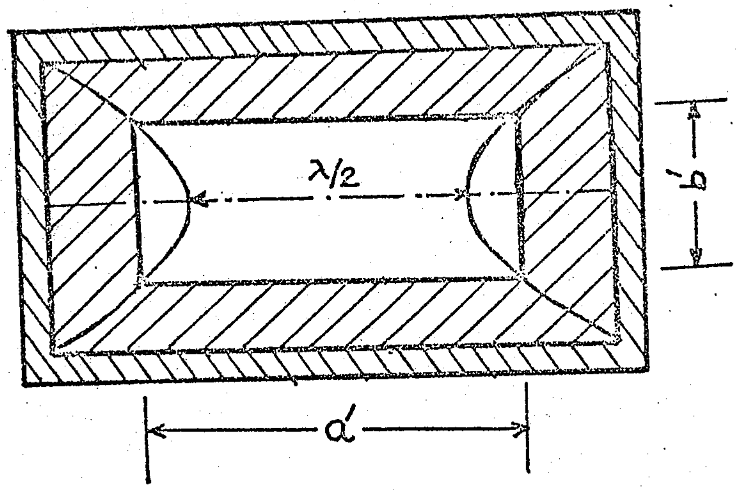


FIG. 4. DESIGN OF A RESONANT IRIS.

and also through the corners of the waveguide. All the centrally located rectangles whose corners fall on these hyperbolas will be found to resonate at the same frequency but will have a different normalized characteristic admittance or loaded Q, which will increase with decreasing height of the window and will also increase with increasing thickness of the diaphragm. The design carried out using equation 2, usually has an error of much less than 5 percent of the predicted value. The effect of thickness on the dimensions required for resonance is negligible as long as it is below 0.04λ.

Four rectangular resonant irises were designed to study the behaviour of their γ factor as described in section 3.4. The dimensions of these irises are given in Table 1. It has been proved that the resonant frequency of the iris of Figs. 5a and 5b are the same provided their areas are equal [9]. The four resonant irises used are shown in Photograph 2.

3.4 Shape Factor γ of the Resonant Iris:

It has been mentioned in Section 3.2 that the solution for the field in a rectangular iris is not available and is very difficult to determine. Therefore, it is necessary to devise some other means to evaluate the integral in the numerator of the right hand hand side of equations 1. This can be achieved by relating the field in the iris to the field in the waveguide without the iris. We define the ratio of these two fields by a shape factor called γ. It was observed experimentally that the shape factor is approximately constant for a given iris as long as the sample thickness is less than a few hundred microns. If γ is assumed to be constant, the ratio of the two fields will be observed as a proportional increase in the resonant frequency of the test chamber as can be seen from equation 1. This then gives a convenient method for determination of this shape factor.

Table 1

Dimensions of four resonant irises with resonant frequency = 10 GHz

Iris	a' in inches	b' in inches	c in inches
A	0.727	0.250	0.531
B	0.671	0.188	0.524
C	0.647	0.156	0.525
D	0.628	0.125	0.529