BASIC DETECTION OF X BAND ELECTRON SPIN RESONANCE SIGNAL USING STRIPLINE RESONATOR

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

In this work, the basic detection of electron spin resonance (ESR) signal by using stripline resonator was demonstrated. The signal was detected by using a basic homemade ESR setup, stripline resonator and a sample known as 2,2-diphenyl-1-picrylhydrazyl (DPPH). Two types of stripline resonators were designed, simulated and fabricated to detect ESR signals by varying externally applied static magnetic field strength or the microwave frequency. These respective designs were called as straight and U-shape design. The techniques used to fabricate the stripline resonators were ultraviolet (UV) lithography and milling by computer numerical controlled (CNC) machine. These resonators were named as UV, M and U resonator, UV and M resonator have straight design while U resonator is a U-shape design. UV resonator has unloaded resonance frequency at 9.12 GHz and Q-factor is 88. M resonator has unloaded resonance frequency at 9.70 GHz and Q-factor is 70. U resonator has unloaded resonance frequency at 9.70GHz and Q-factor is 121. Using these resonators, the ESR signal was successfully detected from the DPPH. By fitting using Lorentzian equation to the ESR signal, the experimental *g*-factor was determined. U-shape resonator had the highest accuracy with *g*-factor of 2.017 or 0.66% difference with the theoretical value. Simulation and experimental results conclude that resonator with the higher microwave magnetic field gives a stronger signal.

ABSTRAK

Untuk penyelidikan ini, pengesanan isyarat resonans spin elektron dengan resonator jenis garis strip telah digajikan. Isyarat tersebut dikesan dengan spektrometer ESR buatan sendiri, alat resonans dan sampel 2,2-diphenyl-l-pierylhydrazyl (DPPH). Dua alat resonans jenis garis strip telah direkabentuk, disimulasi dan dibangunkan untuk mengesan isyarat ESR. Teknik fabrikasi yang digunakan adalah litografi ultraviolet (UV) dan pengilangan dengan mesin kawalan berangka komputer (CNC). Tiga resonator telah difabrikasi dan bagi nama sebagai UV, M dan U, frekuensi resonan yang diperolehi adalah 9.079 GHz, 9.652 GHz dan 9.126 GHz manakala faktor Q adalah 88, 70 dan 121. Dengan menggunakan resonator ini, isyarat ESR telah dikesan dari DPPH. Dari data uji kaji, faktor g ditentukan dengan menggunakan kaedah pemadanan dan taburan Lorentzian. Berdasarkan nilai-nilai faktor-g yang diperolehi, resonator berbentuk U mempunyai ketepatan tertinggi dengan nilai sebanyak 2.017 iaitu perbezaan sebanyak 0.66% berbanding dengan nilai teori. Keputusan simulasi dan eksperimen membuktikan bahawa resonator yang mempunyai medan magnet teraruh yang lebih kuat daripada gelombang mikro mampu menghasilkan isyarat ESR dengan lebih kuat.

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LIST OF ABBREVIATIONS

2D	-	2-dimension
3D	-	3-dimension
AWG	-	Arbitrary wave generator
BDPA	-	1,3-biphenylene-2-phenylallyl
CNC	-	Computer numerical control
CCD	-	Computer controlled drilling
CW	-	Continuous wave
dB	-	Decibel
DC	-	Direct current
DPPH	-	2,2-diphenyl-l-picrylhydrazyl
EDMR	-	Electrically detected magnetic resonance
EM	-	Electromagnetic
ENDOR	-	Electron nuclear double resonance
EPR	-	Electron paramagnetic resonance
ESR	-	Electron spin resonance
FWHM	-	Full-width half-maximum
HFSS	-	High-frequency simulation software
HWHM	-	Half-width half-maximum
HCl	-	Hydrochloric acid
$FeCl_3$	-	Ferric (III) chloride
NMR	-	Nuclear magnetic resonance
PCB	-	Printed circuit board
RF	-	Radio frequency
SNR	-	Signal-to-noise ratio
TEM	-	Transverse electromagnetic
UV	-	Ultraviolet

LIST OF SYMBOLS

δ	-	Loss tangent
ϵ	-	Permittivity
ϵ_{r}	-	Dielectric constant
ϵ’	-	Real dielectric constant
ϵ”	-	Imaginary dielectric constant
λ	-	Wavelength
μ	-	Permeability
μ_{B}	-	Bohr magneton
γ	-	Scale parameter
ω	-	Spinner rotational speed in rpm/1000
a	-	X-coordinate of the peak in Lorentzian distribution
A	-	Radius of the dielectric resonator
b	-	Coefficients produced by the fit
B	-	Magnetic field
BW	-	Bandwidth
c	-	Speed of light
C	-	Confidence bounds
e	-	Tube gap size
f	-	Frequency
fr	-	Resonance frequency
g	-	g-factor
g_{c}	-	Coupling gap size
G	-	Mask-photoresist gap size
h	-	Planck constant
H	-	Dielectric height
k	-	Boltzmann Constant

K	-	Spinner constant
l	-	Length of resonance strip
Δl	-	Fringing field at both ends
n	-	Wavenumber
n_i	-	Integer number
p	-	Viscosity of photoresist
Q	-	Quality factor
Q_c	-	Q-factor of coupling
Q_i	-	Internal Q-factor
R_s	-	Smallest resolution
s	-	Vector of the diagonal elements
S	-	S-parameter
t	-	Thickness
t_{cu}	-	Copper thickness
t_{ph}	-	Photoresist thickness
T	-	Confidence level
w	-	Trace width
X	-	Current supply in Ampere
Y	-	Static magnetic field strength in mT
Z	-	Impedance

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Electron paramagnetic resonance (EPR) or electron spin resonance (ESR) spectroscopy is a technique that mainly applied in the study of unpaired electrons in the materials. In quantum mechanic, ‘resonance’ refers to the phenomena that transmission of molecular spin energy levels caused by the externally applied microwave radiation [1]. It is widely used to study the unpaired electron and free radial. These are normally found in metal complex and an organic compound. ESR had been used in many science branches such as biology, physics and food science. The basic principle of ESR known as Zeeman effect and was discovered by Soviet physicist Yevgeny Zavoisky in 1944 at Kazan State University. Another technique is known as nuclear magnetic resonance (NMR) shared the same working principle but the atom nuclei spins are excited in NMR instead of electron spin in ESR [2].

There are 2 types of basic ESR spectrometer which is a continuous wave (CW) and pulsed ESR [3]. The difference is the excitation source used in the spectrometer. In CW ESR, continuous electromagnetic (EM) wave act as the excitation source while pulsed ESR uses nanoseconds-long pulse instead. Pulsed ESR can excite the targeted sample while CW ESR is the most basic experiment to perform absorption of microwave wave by the paramagnetic electron. In the varying static magnetic field, CW ESR spectroscopy can show the presence of unpaired electron in material and its resonance field at specified energy. From the result of CW ESR, the presence of an unpaired electron can be detected easily because it absorbs electromagnetic (EM) wave energy. Due to the continuous nature of the excitation source, CW ESR is limited in lack of time resolution. To overcome this limitation, pulses are used to replace CW as an excitation source whereby a pulse can be represented as several frequencies superposed with each other at specific phases and amplitudes [4]. Compared to CW ESR, pulsed

ESR spectroscopy is used to excite the material by using a sequence of pulses to differentiate the electron spin interaction and also obtain information in the time domain [5]. This can be further developed to investigate the interaction between electron spin and surrounding nuclear spins [6].

In physics, ESR has been used to provide a theoretical basis for studying modification and detection of electronic structure by the surrounding atom. The electron directional spin can be controlled by using pulses, for example for pulse spectroscopy and quantum computing applications [7]. In chemistry, ESR is used to study the electronic structure and chemical where the free radicals can be determined the free radicals form and the process of the chemical reaction can be observed [8]. Furthermore, by differentiating the g-factor, the oxidation state of the transition metal can be determined [9]. In biology, ESR spectrometer can be used to detect specific chemicals in the human body. Yamato et al. used X band frequency ESR spectroscopy to detect the generation of reactive oxygen species in the brain [10], where the formation and redox reaction of nitroxyl radicals were observed and detected using ESR. Similar to NMR imaging, ESR can also be used in imaging which is based on the same principle as its NMR counterpart. It can provide information that is complementary to NMR imaging. One such example is the detection of oxygenation level of a mouse undergoing CW ESR at 300 MHz [11].

Nowadays, ESR has been widely used in industry such as ESR imaging [11], medical [10] and semiconductor [12]. It can be further developed by adding more components and modifying excitation signal or even combined with NMR into electron-nuclear double resonance (ENDOR). It is a technique that can identify the molecular and electronic structure of the paramagnetic sample. The nuclear transition can be detected by ENDOR by measuring the intensity changes of irradiated ESR transition [13]. Another technique that involves ESR principle is electrically detected magnetic resonance (EDMR). It is used to detect impurities in the semiconductor. When the sample undergoes the ESR test, the donor electron spin orientation can be turned by a pulse at its resonance frequency. The flip can decay the donor to acceptor energy level and recombine with the hole in the valence band. The energy level difference can be detected and its accuracy can down to a few hundreds of atoms [12].

All the advance device were developed from basic CW ESR spectrometer. In this research, a basic homemade setup will be built as a preliminary stage to build a more advanced pulsed spectrometer.

1.2 Problem Statement

To build an advanced pulsed ESR in the future, the resonator is a part of the whole ESR spectrometer that is essential to excite the electron spins and to reciprocally detect the ESR signal [14]. One particular design, known as a stripline resonator, has not been tested at X band frequencies. To test it, an ESR setup was needed. The other resonators such as cavity and dielectric resonator can also operate in X band but its Q-factor is too high (up to 10000) and the bandwidth is too small compared to stripline resonator [15, 16]. The low Q-factor resonator such as microstrip resonator can also operate in X band but the open structure causes field leakage and sensitive to the surrounding [17]. In stripline resonator, the resonant strip is surrounded by copper ground plate to prevent field leakage and reduce noise from the surrounding. These type of stripline resonators were successfully built and used to detect ESR signal at S band (3 GHz) and K_u band (17 GHz) [7, 18, 19].

While advanced and sensitive X band ESR spectrometers are commercially available, such as JES-X3 series from JEOL [20] and micro ESR from Bruker [21] such instruments are costly. These commercial units are not well understood and the applications are limited. Nowadays, commercial EPR spectrometer in Malaysia is only available in UPM (JES-FA200 from JEOL) [22] and an older system in CSNano in UTM (JES-FA100 from JEOL) [23]. Compared to developed countries, a lot of ESR spectrometers for physics experiments are homemade [4, 5, 24]. Some of the researchers even built custom made ESR to be used in quantum computing [7]. In Malaysia, there has been a similar attempt to build a homemade ESR spectrometer [25]. Hence, this research aims to build a basic homemade ESR setup and use it to detect ESR signal at room temperature.

1.3 Objective of Study

There are three objectives for this research:

1. To design and fabricate the stripline resonators for X band ESR spectrometer.
2. To investigate the performance of stripline resonators by simulation and actual fabrication.
3. To detect ESR signal at X band using a basic, homemade CW ESR setup.

The ESR setup is not a full spectrometer but is instead a minimal setup, capable of detecting such weak signals. Due to this reason, it is important to enhance the ESR signal as much as possible. Therefore, sensitive resonator has to be prepared first.

1.4 Scope of Study

In this research, a homemade ESR setup will be built. The continuous wave (CW) source is a commercial microwave synthesizer with X band (8 - 10 GHz) frequency. The resonators used is transmission type stripline resonator fabricated from Roger Duroid 6035 HTC and Arlon DiClad 880. The resonators were fabricated by computer numerical controlled (CNC) milling machine and ultraviolet (UV) lithography. The sample used to detect CW ESR signal is 2,2-diphenyl-1-picrylhydrazyl (DPPH) powder. During the experiment, the resonator was placed in an electromagnet with a magnetic field strength of 0.3 - 0.4 T at room temperature.

1.5 Significant of Study

The resonator size and its resonance frequency can be designed and estimated by using simulation software. This will increase the ESR signal strength and help to detect the ESR signal easily. By preparing resonator in different shape, the suitability of resonator can be determined in different purpose. It is a preliminary stage to build a complex homemade pulsed ESR spectrometer.

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