UNIVERSITI PUTRA MALAYSIA

HIGHLY SENSITIVE METAMATERIAL BIOSENSOR OPERATING IN THE TERAHERTZ REGIME

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By

NADEEM NAEEM

Thesis Submitted to the School Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

To my encouraging parents, my siblings

My beloved wife, my children and all family members

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

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September 2016

Chairman : Associate Professor Alyani binti Ismail, PhD
Faculty : Engineering

Four metamaterial structures are presented in this thesis, which are designed to operate in terahertz frequency regime. The main objective of this study is to design terahertz metamaterial sensors that can be used as highly sensitive biosensors. Since every substance or material has its dielectric characteristics; these biosensors are designed to sense small changes appeared at the minute amount of samples called analytes when they are poured on the surface of the metamaterial structures. There is a change in transmission appeared at metamaterial structures which is due to the change in surface electric field localized in split gap areas.

The first two planar metamaterial Spiral ring resonators were designed operating in terahertz frequency band. These subwavelength structures were categorized as Type-I and Type-II (two-turn and three-turn Spiral resonators). They are designed for use as sensitive metamaterial terahertz sensors. The unloaded fundamental frequency of was observed around 3.69 THz and 3.93 THz respectively. The two-turn and three-turn Spiral resonators based biosensors exhibit very high values of unloaded *Q* factor of 159 and 689.

The other two terahertz sensors composed of split ring resoantors are also demonstrated as negative index metamaterials operating in terahertz regime. The Square and Hexagon spilt resonators are introduced as terahertz biosensors. The unloaded *Q* factor calculated for the Hexagon and Square split ring biosensors were observed at the operating frequencies of 3.2 THz and 3.31 THz are 33.64 and 42.1 respectively. There is significant shift in the transmission coeeficients were observed under loading conditions when the dielectric material as a biological sample of varrying permittivity and different material thickness were deposited on the surface of proposed biosensors. Such biosensors can be used in label free sensing of minute biological substances like protein, bacteria without harming their characteristics and the valuable indformation present in the cells due to the non ionizing characterisitcs of terahertz frequency.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

BIOSENSOR METAMATERIAL BERKEPEKAAN TINGGI BEROPERASI DALAM REJIM TERAHERTZ

Oleh

NADEEM NAEEM

September 2016

Pengerusi : Profesor Madya Alyani binti Ismail, PhD
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Empat struktur metamaterial dibentangkan dalam tesis ini. Semuanya direka untuk beroperasi dalam rejim frekuensi terahertz. Objektif utama kajian ini adalah untuk mereka sensor metamaterial terahertz yang boleh digunakan sebagai biosensor berkepekaan tinggi. Oleh sebab semua bahan atau material mempunyai ciri-ciri dielektrik, biosensor ini direka untuk mengesan perubahan kecil dalam amaun sampel kumin yang dipanggil analit apabila ia dituang ke atas permukaan struktur metamaterial. Perubahan dalam penghantaran akan dilihat pada struktur metamaterial ini disebabkan perubahan medan elektrik permukaan setempat di kawasan celah terpisah.

Dua alat resonan metamaterial gelang berpilin menyatah yang pertama direka untuk operasi dalam jalur frekuensi terahertz. Struktur sub-panjang gelombang ini dikategorikan sebagai Jenis-I dan Jenis-II (alat resonan berpilin dua dan tiga putaran). Alat resonan ini direka sebagai sensor terahertz metamaterial yang sensitif. Frekuensi asas tanpa beban dilihat pada 3.69 THs dan 3.93 Hz, masing-masing. Biosensor berasaskan alat resonan berpilin dua putaran dan tiga putaran menunjukkan nilai faktor Q tanpa beban yang sangat tinggi (159 dan 689, masing-masing).

Dua sensor terahertz seterusnya yang terdiri daripada alat resonan gelang terpisah turut terbukti sebagai metamaterial indeks negatif yang beroperasi dalam rejim terahertz. Alat resonan Segi empat dan Heksagon terpisah turut diperkenalkan sebagai biosensor terahertz dalam kajian ini. Faktor Q tanpa beban dikira untuk biosensor gelang terpisah bentuk Heksagon dan Segi empat dan diperhatikan di frekuensi operasi 3.20 THz dan 3.31 THz dengan nilai 33.64 dan 42.10, masing-masing. Anjakan signifikan dalam pekali penghantaran dapat dilihat dalam keadaan dengan beban apabila material dielektrik, sebuah sampel biologi yang mempunyai ketelusan dan ketebalan material berbeza, diletakkan pada permukaan biosensor yang dicadangkan dalam kajian ini. Biosensor ini boleh digunakan dalam pengesanan bebas-label bahan biologi kumin seperti

protein dan bakteria tanpa membahayakan ciri-ciri bahan dan maklumat penting yang terdapat dalam sel. Hal ini disebabkan oleh ciri-ciri bukan-pengion radiasi terahertz yang tak berbahaya.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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LIST OF ABBREVIATIONS

Abbreviation	Meaning
3D	Three Dimensions
MTM	Metamaterial
LH	Left-Handed
NIM	Negative Index Materials
THz	Terahertz
SRR	Split Ring Resonator
Q	Quality factor
THz–TDS	Terahertz Time-Domain Spectroscopy
EM	Electromagnetic
DNA	Deoxyribonucleic Acid
LHM	Left-Handed Metamaterial
2D	Two Dimensions
DNG	Double negative
FSS	Frequency Selective Surface
ADSR	Asymmetric Double Split Resonator
CST	Computer Simulation Technology

CHAPTER 1

INTRODUCTION

1.1 Background

The term metamaterial, coined by Walser (2001), is defined as a 3-D periodic non-natural composite, which is based on the combination of two or more electromagnetic responses. Presently, metamaterials are known as innovative synthetic materials that can surprisingly manipulate beams of light. The metamaterial structure is mainly based on sub-wavelength metallic resonators that remain in a dielectric. These resonating elements provide electromagnetic properties to metamaterials by ignoring conventional materials including atoms or molecules from these resonating elements (Pendry, 2006).

Recently, interest in the research and development of new materials with characteristics that may not be found in nature has increased. These materials include metamaterials (MTMs), left-handed (LH), and negative index materials (NIM). These materials can be applied not only in scientific areas but also in industrial areas. These applications include artificial dielectrics, lens, absorbers, antenna structures, optical and microwave components, frequency-selective surfaces, sensing devices for gas, chemical- and water- based substances (Drexler et al. 2014; Miyamaru et al. 2014; Balamati et al. 2014), and vapor sensors (Salvatore, 2015).

Metamaterial science has achieved a high degree of sophistication due to the continuous progress in design and fabrication on the submicron and nanometer scales to create novel properties of metamaterials. The interdisciplinary integration between metamaterial science and terahertz sensing technology has paved the way for new scientific and technological development including research into terahertz sensors based on metamaterials, which has led to the development of various metamaterial-based components for detecting information on substance and circumstance.

The present advances in terahertz metamaterials have extended to include optical nanostructures and other related fields including communication, microscopy, and defense. Similarly, the terahertz (THz) spectrum dealing with distinctive properties and associated with terahertz frequencies is also known as a major research area involving metamaterials and favorable applications are currently associated with human exposure including medical diagnoses and security screening.

Terahertz biosensors based on metamaterials, due to the non-ionizing properties of terahertz radiation, are suitable for biomedical sensing applications (Fitzgerald 2002; Bogomazova 2015; Ergün and Sönmez, 2015), e.g. sensing of thin layers of bio molecules without harming the cells. In metamaterials, the split ring resonators have shown promising potential in terms of their simple structures and easy implementation. Split rings exhibit left-handed properties, which are of great importance in constructing new types of metamaterials.

1.2 Statement of Research Problem

It is difficult for scientists to detect very thin and small amounts of biological and biochemical substances using conventional sensors without damaging cell structure. Labeling is the most common method used to detect the minute amount of biomolecules. Metamaterial-based sensors have been reported in the literature as a reliable solution for increasing the sensitivity of conventional sensors.

In recent years, there have been reports on metamaterial sensors designed for sensing thin dielectric layers of biological and chemical substances but most of them have suffered from low quality factor values due to their shallow transmission characteristics (Fedotov et al. 2010; Singh et al. 2014; Yang et al. 2014; Chowdhury et al. 2014). Low quality (*Q)* factors limit the sensing capabilities of metamaterial biosensors (Miyamaru et al. 2010; Moser et al. 2005; Chen et al. 2008; Tao et al. 2009; O'Hara et al. 2008; Jansen et al. 2011).

High *Q* factors are desirable in sensing applications. The *Q* factor of a sensor is generally determined by the energy loss per cycle as opposed to the stored energy. High *Q* metamaterial resonators for terahertz frequencies are difficult to design and characterize since the radiation loss in these structures increases inversely with resonator size. Several sensor designs have been reported to achieve higher *Q* factors and low losses particularly the design of terahertz metamaterials with new geometries and electrical coupling between resonating elements (Gu et al. 2010; Chen et al. 2010; Singh et al. 2008; Singh et al. 2010). However, these designs have achieved limited success in terms of sensitivity.

1.3 Motivation

Terahertz (THz) electromagnetic (EM) radiation falls between the microwave and infrared frequency bands and shares some common properties of each of these frequency bands. Materials like plastic, glass, paper, cloths, and ceramics are transparent to terahertz radiation (Fitzgerald et al. 2002). Such transparency facilitates high resolution non-invasive sensing that is useful in applications related to security and quality control. The low photonic energy of terahertz waves and their tremendous sensitivity to water absorption is the main motivation for research in the field of terahertz sensing, especially in the study of living organisms and imaging systems (Mittra et al. 1988; Pendry et al. 1996; Genet, 2007; Yan et al. 2014).

The opportunities for safe and harmless direct communication are boundless with the use of comparatively short wavelength and broad frequency bandwidth (Comparatively short wavelength along with broad frequency bandwidth creates chances for safe and harmless direct communication). High data rates can be achieved with THz frequency. Recently, the detection of biological samples and explosives with unique spectral signatures (i.e. difference in the reflectance/emittance characteristics with respect to wavelengths) has become an emerging research area (Pendry et al. 1999; Powell et al. 2008).

Terahertz biosensors have persistently been developed in terms of parallel improvement of ultrafast lasers; turnkey systems can be obtained commercially for overall spectroscopy and imaging (Pendry, 2000; Soukoulis et al. 2007; Dolling et al. 2007). These developments in terahertz systems are, however, associated with some design limitations.

The sensing and categorization of actual minute amounts of biological analyte has stimulated a rapidly increasing branch of terahertz research. However, thin biological samples induce small and immeasurable changes via relatively inadequate interaction with terahertz waves. The main focus of this thesis, therefore, is to overcome this problem by suggesting the use of a novel metamaterial based on terahertz biosensors that could detect thin layers of biological analytes with different dielectric constants and layer thickness.

1.4 Challenges in Terahertz Biosensing

Terahertz biosensing systems must be specified, so as to facilitate accurate measurable sensing. This is because traditional THz sensing techniques have yielded limited success in detecting minute amounts of biological samples and chemicals (Withayachumnankul 2009). Developed analytes in the form of a dielectric layer have different terahertz properties than its bulk counterparts due to the size of these analytes and surface roughness, which changes during the fabrication process.

Micro devices are designed with low adaptations toward planar substrates, which include sub-atomic implantation, chemical vapor deposition, and surface treatment with the help of chemicals. Moreover, biosensing techniques have helped develop a series of beneficial terahertz science and applications.

Terahertz biosensing offers several practical and fundamental challenges involving electromagnetic sensing. In order to get measurable readouts, under-test materials must have a strong interaction with the applied field. This interaction is associated with frequency-dependent constitutive material parameters and the amount of material to be tested. In spectroscopy devices, insufficient interaction between the biological samples and the applied EM field can lead to partial or incomplete information being obtained.

1.5 Research Aim and Objectives

The objective of this research work is to design and analyze metamaterial terahertz biosensors for sensing minute and thin biological samples. These terahertz sensors consist of planar metamaterial designs. The proposed terahertz biosensors are targeted for sensing thin layers of DNA samples with different dielectric constants and varying thickness. In order to achieve this aim, the following objectives have been set:

1. To design planar split rings and Spiral resonators based on metamaterial biosensors.
2. To scale down the metamaterial structures so that they will be able to operate in a terahertz regime.
3. To evaluate the effectiveness of the developed terahertz sensors in terms of their quality factors (Q) and surface electric field distributions.
4. To evaluate the performance of the proposed metamaterial biosensors and analyze the effect of thin dielectric material deposition that mimics the bio samples, which includes different thickness and dielectric constants.

1.6 Scope of Research

The scope of this research is to design metamaterial-based biosensors that operate in the terahertz regime, so as to sense any dielectric changes that appear under the loading conditions of biological samples. The performance of the proposed design is to be tested using the commercially available CST design studio version 2011 software. The sensing performance of terahertz biosensors is evaluated by measuring external quality factor, surface electric field distribution, and transmission spectra. The scope of research work is outlined in Figure. 1.1.

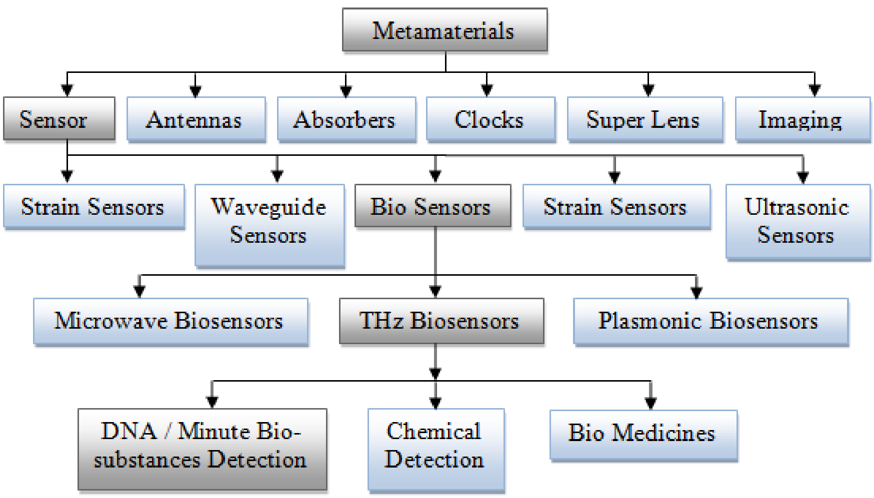

Figure 1.1 : Scope of Research

1.7 Research Methodology

The performance of metamaterial-based terahertz biosensors is tested through software simulation and Scattering parameters are extracted to observe their resonant behavior. The effective parameters are calculated in order to observe the left-handedness of the proposed biosensors in the THz frequency band.

The terahertz biosensors are modeled and simulated with depositions (loadings) of different thin layers of the biological sample with varying sample permittivity. The obtained results will then be compared with the frequency responses of reported biosensors to validate the novelty of the proposed metamaterial resonators as a terahertz biosensor. Figure 1.2 illustrates the flow chart for this study in which the procedural steps for the proposed design methodology are depicted.

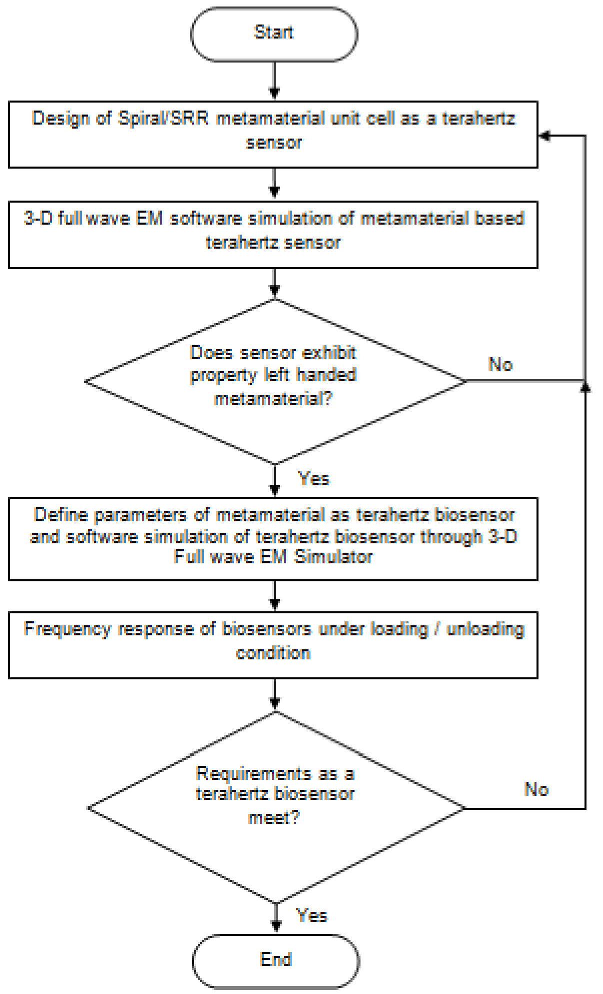

Figure 1.2 : Methodology

1.8 Organization of Thesis

This thesis is organized into six chapters. The summary of each chapter is given below:

Chapter 1 provides a general introduction to the research area and identifies the current problems in designing metamaterial-based terahertz sensors, which are the main motivation for this research. This chapter also introduces the goals, objectives, methodology, and scope of research as well as the organization of this thesis.

Chapter 2 presents a thorough literature review and theoretical background about metamaterial structures, which covers sensors operating in the terahertz frequency band. It also provides a background and history of metamaterials before detailing the salient electromagnetic features of these materials as well as their recent applications, especially as terahertz biosensors.

Chapter 3 provides an introduction on terahertz waves and its applications in the field of biosensing and imaging.

Chapter 4 and 5 describe the design aspects of Spiral, Hexagon, and Square ring resonators in the terahertz frequency region.

The testing results of the terahertz biosensors are presented in Chapter 6 and the conclusion to this study is presented in Chapter 7.

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LIST OF PUBLICATIONS

Naeem, N., Ismail, A., Alhawari, A.R.H. and Mahdi, M.A. 2015. Subwavelength negative index planar terahertz metamaterial arrays using spiral split ring resonators for near field sensing. *International Journal of Applied Electromagnetics and Mechanics* 47(3): 827-836.

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