Electromagnetic characterization of photo-definable ferrite loaded polymers and their applications in micro-rectangular coaxial phase shifters

Olusegun Samuel Sholiyi

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ELECTROMAGNETIC CHARACTERIZATION OF PHOTO-DEFINABLE FERRITE LOADED POLYMERS AND THEIR APPLICATIONS IN MICRO-RECTANGULAR COAXIAL PHASE SHIFTERS

by

OLUSEGUN SAMUEL SHOLIYI

A DISSERTATION

Submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in
The Department of Electrical and Computer Engineering to
The School of Graduate Studies of
The University of Alabama in Huntsville

HUNTSVILLE, ALABAMA

October, 2014
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Olusegun Samuel Sholoyi

11/07/2014

(date)
Submitted by Olusegun Samuel Sholiyi in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Electrical Engineering and accepted on behalf of the Faculty of the School of Graduate Studies by the dissertation committee.

We, the undersigned members of the Graduate Faculty of The University of Alabama in Huntsville, certify that we have advised and/or supervised the candidate of the work described in this dissertation. We further certify that we have reviewed the dissertation manuscript and approve it in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Electrical Engineering.
As the demand for smaller size, lighter weight, lower loss and cost of communications transmit and receive (T/R) modules increases, there is an urgent need to focus investigation to the major subsystem or components that can improve these parameters. Phase shifters contribute greatly to the cost of T/R modules, and thus this research investigation examines a new way to reduce the weight and cost by miniaturizing the phaser design. Characterization of hexaferrite powders compatible with the sequential multilayer micro-fabrication technology and numerical simulations of a novel rectangular micro-coaxial phase shifter are investigated. This effort aims to integrate ferrite material into a rectangular micro-coaxial waveguide at Ka-band using electromagnetic finite element numerical tools. The proposed technique exploits rectangular coaxial waveguide with a symmetrically placed inner signal conductor inside an outer conductor connected to the ground. Strontium ferrite-SU8 composite is used as an anisotropic material of choice in the modelled design. Numerical modeling is employed using High Frequency Structure Simulator, HFSS, a 3-D full wave electromagnetic solver for analyzing the performance of the device. Two model structures
were designed for reciprocal and non-reciprocal applications. The first model (Model A) produced a tunable phase shift of almost 60 degrees /cm across 0 to 400 kA/m applied field and at 1800 Gauss. In model B, a non-reciprocal phase shift performance of 20 degrees /cm from a reference phase of 24 degrees at 0 A/m was realized at the same saturation magnetization. A return loss better than 20 dB and an insertion loss less than 1.5 dB were obtained for both models.
ACKNOWLEDGMENTS

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<td>$\mu$-RCP</td>
<td>Micro-rectangular coaxial ferrite Phase shifter</td>
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<td>$\gamma$</td>
<td>Gyromagnetic ratio</td>
</tr>
<tr>
<td>$\mathbf{m}$</td>
<td>Magnetic moment vector</td>
</tr>
<tr>
<td>$\mathbf{s}$</td>
<td>Spin angular momentum vector</td>
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<tr>
<td>$m_e$</td>
<td>Mass of the electron</td>
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<tr>
<td>$\mathbf{T}$</td>
<td>Torque</td>
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<td>$\hat{z}$</td>
<td>Unit vector along z-axis</td>
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<td>Permittivity in vacuum</td>
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<td>$m_x$</td>
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<td>$m_z$</td>
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<tr>
<td>$\omega_0$</td>
<td>Precession, or Larmor frequency</td>
</tr>
<tr>
<td>$\theta$</td>
<td>Precession angle</td>
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<tr>
<td>$\mathbf{M}$</td>
<td>Total magnetization vector</td>
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<tr>
<td>$\chi$</td>
<td>Susceptibility</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
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<tr>
<td>( \kappa )</td>
<td>kappa, Permeability in a material, ( \mu = \mu_0 \mu_r )</td>
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<td>( \mu )</td>
<td>Effective permeability, ( \mu_e = \frac{(\mu^2 - \kappa^2)}{\mu} )</td>
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<td>( k_c )</td>
<td>Cut-off wave number</td>
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<td>( \mu'' )</td>
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\( v_{\text{phase}} \)  Phase velocity, m/s

\( C_{\text{perlength}} \)  Capacitance per unit length, F/m

\( L_{\text{perlength}} \)  Inductance per unit length, H/m

\( \mathbf{E} \)  Electric field vector, V/m

\( \mathbf{D} \)  Electric flux density vector, Coul/m²

\( \mathbf{B} \)  Magnetic flux density vector, Wb/m²

\( \mathbf{H} \)  Magnetic field vector, A/m

\( \beta \)  Propagation constant

\( \nabla \)  nabla
To

Mosunmola, Olufemi, and Michelle
CHAPTER 1

INTRODUCTION

1.1 Objective of the work

The objective of this dissertation is to investigate a novel method to adapt a rectangular micro-coaxial waveguide to function as a phase shifter by integrating compatible ferrimagnetic micro-particles such as M-type barium and strontium hexaferrites using \( \mu \)-coaxial PCB technologies. In this work a comprehensive characterization and analysis of the ferrimagnetic materials mixed into a photo-patterned resist will be carried out so as to determine the percentage loading of ferrite into the bonding polymers that will be used in micro-fabrication of ferrite phase shifter. Also different configurations of the compatible ferrimagnetic material are investigated. Finite element modelling of the 3-Dimensional structures of different configurations of Micro-rectangular coaxial ferrite Phase shifter (\( \mu \)-RCP) are carried out using finite element full-wave electromagnetic computer aided design tools such as high frequency structure simulator (HFSS), CST studio 3D full-wave electromagnetic solver.
This dissertation examines primarily the following research works: First, the characterization of hexagonal structured barium and strontium ferrimagnetic materials for possible micro-fabrication applications at Ka-band. This involves the investigation of permeability and permittivity of barium and strontium ferrite powders and composites with SU8 negative photo-resist polymer using Nicolson-Ross-Wier (NRW) algorithm. Also the stoichiometric composition of these materials and their composites are carried out in order to determine the percentage composition of the elements making up the samples using Edax equipment. The ferromagnetic resonance and magnetic hysteresis loops of the materials are determined using FMR equipment and vibrating sample magnetometer respectively. Secondly, the photolithographic patterned micro-fabrication of ferrite-polymer magnets on wafers using micro-machining technology is presented. This work involves the fabrication of different shape ferrite polymer magnets on wafers from ferrite-SU8 composites using photolithographic patterned micro-fabrication process. Next, the design and modeling of a micro-rectangular coaxial transmission line at Ka-band frequency range is presented. This research entails the design and modeling using finite element modeling CAD tools. The fourth activity is the design and modeling of micro-rectangular coaxial ferrite phase shifter, $\mu$-RCP using ferrite-SU8 composite and 3D full wave electromagnetic adaptive solvers. Finally, after the modeling of the structures, analysis of the designed models are investigated for different quasi-planar configurations so as to compare generic rectangular ferrite phase shifter and $\mu$-RCP. Before any of the major tasks is embarked upon, it is necessary to delve into some of the backgrounds of early ferrite phase shifters as extensively presented in the next section.
1.2 Background

Phase shifters are microwave components, broadly used in a variety of instrumentation, measurement, communication, satellite and radar systems. These low-loss two port devices change the phase of the radio frequency signal. They can be classified as mechanical or electronic depending on whether the phase tuning is mechanical or electronic in nature [1]. Until the late 1950s, all phase shifters were mechanical in nature, either in the fixed or variable form such as those proposed by Gardner Fox in 1947 and Louis Stark in 1957 [2, 3]. In 1957, the first electronically controlled ferrite phase shifter was reported by Reggia and Spencer [4] and was used in a volumetric scanning radar. Thus the year 1957 represents the beginning of modern ferrite phase shift technology. Since then, there has been intense and continued research and development designs and technology required to meet widespread demand for the modern communications and radar market. These efforts include semiconductor diode phase shifters [5, 6], gallium arsenide field effect transistor (GaAs FET) active phase shifters [7, 8], bulk semiconductor phase shifters with optical control [9, 10], and true time delay element phase shifters. The two most prominent phase shifter technologies available today are the ferrite and semiconductor diode phase shifters. The advent of microwave integrated circuits (MICs) and monolithic microwave integrated circuits (MMICs) has provided an easy way to realize MMIC phase shifters with this technology. The benefits of modern MMIC phase shifters are the cost reduction due to batch processing, higher reliability, improved reproducibility, size and weight reduction, circuit design flexibility and multifunction performance [11]. Apart
from aforementioned applications, current research and development on electronic phase shifters have opened up new applications where micro-sized phased arrays are required, such as in areas of traffic control and collision avoidance radar devices. Ferrite phase shifter being one of the most developed phase shifters, often has the advantages of lower cost, higher power handling capacity and power requirement benefits. Together, these advantages make ferrite a better choice in a phased array applications, where every element in the array is connected to a ferrite phase shifter for beam steering in space.

To date, numerous research and development efforts on ferrite phase shifters for both reciprocal and non-reciprocal have been reported [12–15]. Boyd Jr. [12] employed a pair of similar non-reciprocal phase shifters with circulators at both input and output to produce a latching reciprocal phase shifter over a 10% bandwidth at X-band. Although the fabricated device supported the peak power of up to 1 kilowatt at room temperature, the design suffered considerable loss attributed to alignment errors in the polarizers and fabrication processing. Later, Ince and Stern [13] employed a complete analysis for three different configurations of non-reciprocal remanence ferrite phase shifters in a rectangular waveguide: the first configuration has a toroid centrally placed inside a rectangular waveguide, the second is made of double toroids symmetrically placed and separated with a dielectric rib, while the third arrangement is a composite circuit phaser separated with a dielectric rib inside the rectangular waveguide where the flux path is completed outside the microwave circuit. Also in year 2007, Hui et al [14] proposed a microstrip-line-based ferrite phase shifter at 3 GHz by arranging three microstrip lines to produce circularly polarized
waves in the ferrite region by feeding them with different phases. These authors were able to achieve approximately 360 degrees per wavelength of phase shift, but at the expense of a large magneto-static circuit. Abuelmaatti, et al, [15] used a finite element method to model the non-uniform magnetization and partially magnetized state of a twin toroid ferrite phase shifter, and then fabricated a homogeneous toroid made from viscous plastic processing (VPP) ferrite to achieve a reasonable return loss and phase shift agreement within 10% between simulation and measured values over 9.5-10.3 GHz.

Despite all this development on ferrite phase shifters, to date ferrite in the forms of industrial blocks have mostly been inserted in the square, rectangular, or cylindrical waveguiding microwave devices to achieve the desired phase changing. These waveguides are one conductor two-port transmission lines, which can only support transverse electric, (TE) modes and not both TE and TEM modes of electromagnetic propagation as in the case of two conductors. As the current demand for minimal loss, low cost, small size, and good performance microwave devices for transmit and receive, (T/R) modules increases, the possibility of implementing the ferrite phase shifter in two-conductor transmission lines and waveguides could be a welcome research effort. This implementation can lead to a better performance of what the present one conductor waveguide structures can offer. Higher isolation, better dispersion, broadband operations, loss reduction, and increase in power handling capacity are some of the major benefits that can be realized from a two-conductor waveguide over one-conductor waveguide. Moreover, the current micro-fabrication technology such as Nuvotronics PolystrataTM [16–18] and EFAB [19, 20] techniques for two-
conductor rectangular coaxial waveguides could be successfully utilized and adapted to realize the solutions to the current challenges encountered in the communications and radar systems where ferrite phase shifters are numerously used.

Current advances in research and development on rectangular coaxial cable/waveguide have been made possible by the enormous research work carried out and reported by the following scholars [21–28]. Several attempts have been made by Chen [21], Cohn [22, 23], Anderson [24], Getsinger [25], Garver [26], to derive expressions for the characteristic impedance of a rectangular coaxial waveguide for different ratios of inner conductor width to vertical gap between inner and outer conductor (w/h), and inner conductor height to the horizontal gap between the inner and outer conductor (b/g). Their derivations and methods have different degrees of accuracies but Cruzan and Garver [27] successively expanded on work by Skiles and Higgins [28] to compute and catalog large number of cases in graphical forms so as to obtain the desired information directly for coaxial rectangular waveguides. Also [29–31] contributed in no small way in developing air-filled rectangular microcoaxial waveguides. The first rectangular coaxial transmission line compatible with existing MMIC technology was fabricated and characterized by Bishop, et al, [29] in 1991. This line is made up of a center conductor surrounded by a homogeneous dielectric (probimide) and then covered entirely by a gold plated ground plane as an outer conductor. The result, in the form of S-parameters, was measured through on-wafer microwave measurement and then the data was converted to ABCD parameters for extraction of two important transmission line parameters, namely characteristic impedance and propagation constant. Similarly, Filipovic, et al, [30], between fre-
quency range 26-35 GHz, analyzed, designed, fabricated and evaluated performance of copper-air rectangular coaxial lines, branch line hybrid, cavity resonator and cavity-backed patch antenna using copper as the structural material and polymer for inner conductor support. These authors obtained a very low loss of about 0.22 dB/cm and isolation better than 60 dB at 26 GHz. Lancaster, et al., [31], reported the design, fabrication and measurement of performance of a five-layer low-loss rectangular air-filled coaxial cable using micromachining technology. The cable has a symmetrically located conductor inside a rectangular hollow outer conductor. The whole cable is divided into five layers, fabricated separately using gold coated SU8 photoresist, aligned with optical microscope and eventually bonded together with a silver loaded polymer. The results reported were encouraging with about 0.6 dB insertion loss due to the coaxial-coplanar transitions connected to the input and output ports for on-wafer microwave measurements. Furthermore they reported a return loss better than 15 dB within the measured pass band of 14-36.3 GHz.

Continued research by other scholars also contributed in no small way to the development of rectangular micro-coaxial lines and applications such as directional couplers and antennas [32–35]. Having studied all the above developments with respect to one of the most developed phase shifters (ferrite phase shifters), and the requirements to fulfil low-loss, low-cost, good isolation, better dispersion, broad band operation, power handling capability, and compatibility and adaptability with MMIC and MEMs technology, there appears no record to date of \( \mu \)-RCP. The main interest of this project is to investigate a novel technique of achieving a better ferrite phase shifter
adapting the current rectangular coaxial waveguide using technologies compatible with sequential micro-fabrication.

The compatible ferrimagnetic materials should be a ferrite-composite and should demonstrate a remanence ratio close to unity, a dielectric constant between 4 and 13, and low dissipative permeability. Finally, the high frequency performance of the composite material should provide the required effective permeability needed for non-reciprocity in microfabricated phase shifting applications. However, saturation magnetization ($4\pi M_s$) of the composite should be large enough to achieve a high degree of magnetic activity, although its actual value depends on the operating frequency and the peak power requirements. In general for a Ka-band application, the ferrite composite must have a saturation magnetization value on the order of 5000 G [1]. Other characteristics such as good power handling capability, good temperature stability (considerably below Curie temperature), switching time of 1s to tens of microseconds, and negligible attenuation at about 1 dB per cm, are necessary factors to be met by the ferrite composite in this application area.
CHAPTER 2

CURRENT TECHNOLOGY OF FERRITE PHASE SHIFTERS AND
THE NEW APPROACH

2.1 Current technology and challenges

The agility afforded by the electronically controlled phase shifters is an important factor in making phased array antennas very attractive to radar applications [36]. Phase arrays possess the capability to multitask functions simultaneously interlaced in time by using signal processing circuits and computer control capabilities. Generally the functions are three-dimensional (3D) scanning, formation of multiple beams of similar and non-similar beamwidths and simultaneous switching of multiple beams [1]. The beam is formed in part by every element of the array. Connecting each component to an independent phase shifter allows one to control the direction of the intended beam as shown in Figure 2.1 (a) [37]. Current technology uses a ferrite phase shifter based array. These phase changers are manufactured using industrial ferrite blocks that are machined and polished to sizes with the examples shown in Figure 2.1 (b) [38]. These blocks are machined and polished to size and then incorporated into the waveguides to form a complete ferrite phase shifter. However, this manufacturing technique is not sufficient for integrating ferrite materials into miniaturized
**Figure 2.1:** Antenna array and ferrite blocks: (a) antenna phased array schematic and graphics [37], and (b) ferrite blocks [38]

**Figure 2.2:** Rectangular coaxial waveguide applications [39]

or micro-fabricated rectangular or coaxial waveguides in Figure 2.2 [39]. Another challenge associated with classical machining is unattainable mechanical resolution required for a critical dimensions less than 200 microns [40].
To achieve ferrite integration on this scale, this work has endeavored to establish a technique by which ferrite and photo-polymer can be mixed into a uniform suspension and then spin cast, baked, exposed, and developed using MEMs techniques. These micro-electromechanical systems (MEMs) assembly technologies have already been used to develop micro coaxial transmission lines, waveguides, cavity resonators, and cavity-backed patch antennas \cite{30,32-35} and have all been patterned onto single substrates. Examples of rectangular micro-coaxial transmission line applications are depicted in Figure 2.3. Prior to this effort, ferrite integration had not yet been achieved using manufacturing processes that can be easily adapted into commercial micro-fabrication techniques. In part this dissertation focuses on mixing micron-sized ferrite powders (Barium or strontium or both) with lithographically patternable polymers to create microscale ferrite composites that will be integrated into rectangular micro coaxial transmission lines, to form a complete ferrite phase shifter module using the micro-fabrication process.

2.2 Anisotropic materials chosen for the ferrite-polymer composites

The application space, or frequency, often dictates the choice of ferrite used. In this investigation, a crystalline hexagonal shape, M-type barium ferrite (BaFe$\text{$_{12}$O$_{19}$}$) and strontium ferrite (SrFe$\text{$_{12}$O$_{19}$}$) powders are the baselines for the ferrimagnetic materials that were chosen based on their excellent performances and reasonable losses within the frequency range of interest (30-60 GHz). Hexagonal ferrites of these types have closely related and complex crystal structures. Description of a hexagonal
crystal is made using four Miller indices h, j, k and l. The crystal has its principal, c-axis in the (0001) direction along its length (c lattice parameter), and the three other axes are across the basal (0001) plane of the hexagonal polyhedron at angles of 120° to each other. Since the three axes of the hexagonal plane are equal, thus only one parameter, a is required for a regular hexagonal crystal which is the length of one of the sides [41]. In other words, for a dimensional description of a crystal, two basic lattice parameters, a and c are required. The compound BaM, BaFe$_{12}$O$_{19}$, with a melting point of 1390°C was confirmed to be in existence in 1936. The compound was first studied and magnetically characterized in the early 1950s. BaM has coercivity within 160-255 kA per meter, but lower saturation magnetization than the existing alloy magnets. It is much cheaper to produce, and demonstrates a high magnetic uniaxial anisotropy along the c-axis [42]. The anisotropy property often gives ferrite its ability to react differently with an electromagnetic field when a magneto-static
biased field is applied to the material either transversely or longitudinally. This property is what is being utilized in nonreciprocal ferrite phase shifters to control the phase shift electronically by varying the biased static field. In its simplest form, the basic properties: permeability tensor, circularly polarized fields, and losses of ferrimagnetic materials will be discussed in the following section.

2.3 Permeability tensor of ferrimagnetic materials

Considering a simple microscopic view of the atom, the permeability tensor of a ferrite material can be deduced. This tensor can be affected by losses and demagnetization field inside an infinitesimally sized piece of the material. A non-magnetic material has its relative permeability to be unity but a magnetic material on the other hand has its relative permeability to be a tensor. A Magnetic dipole moment of an electron can be established according to the vector relation between the magnetic moment, \( \mathbf{m} \) and the spin angular momentum, \( \mathbf{s} \) as

\[
\mathbf{m} = -\gamma \mathbf{s}.
\]

(2.1)

where gamma \( \gamma \) is the gyromagnetic ratio \( (\frac{q}{m_e}) \) and \( q \) and \( m_e \) are charge and mass of the electron respectively. As the negative sign indicates, the magnetic moment is oppositely directed to the spin angular momentum. With an applied bias field along the z-axis, \( \mathbf{H}_0 = \hat{z} H_0 \), to the atom as shown in Figure 2.4, then a torque \( \mathbf{T} \) will be exerted on the magnetic dipole which is presented in Equation 2.2. This torque is determined using Figure 2.4 (a) of a spinning electron. In this diagram, it
is assumed that the direction of precession of the magnetic dipole moment is counter-clockwise about the z-axis and also the spin angular momentum is in line with the magnetic moment but oppositely directed. Figure 2.4 (d) [44] shows the sinusoids of left and right circular polarizations experienced by the magnetization vector during the precession in Figure 2.4 (a and b). Thus this torque $T$ is expressed as

$$T = m \times B_0$$

$$= \mu_0 m \times H_0$$

$$= -\mu_0 \gamma s \times H_0$$ \hspace{1cm} (2.2)$$

But this torque can equally be expressed in terms of time rate of change of spin angular momentum. On the other hand, it can be explained as being negatively proportional to the time rate of change of the magnetic dipole moment

$$\frac{ds}{dt} = -\frac{1}{\gamma} \frac{dm}{dt} = T$$

$$= \mu_0 m \times H_0$$ \hspace{1cm} (2.3)$$

Rearranging the terms,

$$\frac{dm}{dt} = -\mu_0 \gamma m \times H_0$$ \hspace{1cm} (2.4)$$
Figure 2.4: A spinning electron and circular polarizations [43]: (a) vectors of magnetic dipole moment, $\mathbf{m}$ and angular momentum $\mathbf{s}$, (b) RHCP, (c) LHCP, and (d) sinusoids of RHCP and LHCP.

which is the equation of motion for the magnetic dipole moment, $\mathbf{m}$ and solving it indicates that the magnetic dipole precesses around $\mathbf{H}_0$, the field vector. Expressing the equation in terms of the three vector components $x,y$ and $z$, it is expressed as

$$\frac{dm_x}{dt} = -\mu_0 \gamma m_y H_0 \quad (2.5)$$

$$\frac{dm_y}{dt} = \mu_0 \gamma m_z H_0 \quad (2.6)$$

$$\frac{dm_z}{dt} = 0 \quad (2.7)$$
Using Equations (2.5) and (2.6) to obtain two equations in terms of \( m_x \) and \( m_y \), we therefore can obtain the second order equations:

\[
\frac{d^2 m_x}{dt^2} + \omega_0^2 m_x = 0 \tag{2.8}
\]
\[
\frac{d^2 m_y}{dt^2} + \omega_0^2 m_y = 0 \tag{2.9}
\]

where

\[
\omega_0 = \mu_0 \gamma H_0 \tag{2.10}
\]

is called precession, or Larmor frequency. The solutions to Equations (2.8) and (2.9) compatible with Equations (2.5) and (2.6) can be written as

\[
m_x = C \cos \omega_0 t \tag{2.11}
\]
\[
m_y = C \sin \omega_0 t \tag{2.12}
\]

where \( C \) is a constant. Also for (2.7) to be zero, \( m_z \) must be a constant, thus

\[
|\mathbf{m}|^2 = m_x^2 + m_y^2 + m_z^2 = C^2 + m_z^2 \tag{2.13}
\]
Therefore the precession angle, \( \theta \) that exists between \( \mathbf{m} \) and \( \mathbf{H}_0 \) (z-axis) is given as

\[
\sin \theta = \frac{\sqrt{m_x^2 + m_y^2}}{|\mathbf{m}|} = \frac{C}{|\mathbf{m}|}.
\]

(2.14)

However, for \( N \) magnetic dipoles per unit volume, the total magnetization, \( \mathbf{M} \) of a saturated ferrimagnetic material is

\[
\mathbf{M} = N \mathbf{m}
\]

(2.15)

provided there is a strong bias field to perfectly align all the magnetic dipole moments from unbalanced electron spins. Now relating this to the equation of motion of the magnetic dipole moment in (2.4), we can obtain

\[
\frac{d\mathbf{M}}{dt} = -\mu_0 \gamma \mathbf{M} \times \mathbf{H}
\]

(2.16)

where \( \mathbf{H} \) is the internal applied field. As the strength of the bias field, \( H_0 \) increases, more magnetic dipole moments align with \( H_0 \) until all are aligned, and \( \mathbf{M} \) reaches the maximum point and the ferrimagnetic material is termed magnetically saturated, denoted as \( M_s \). But if a small signal (AC magnetic field: \( \mathbf{H} \)) approximation is assumed, and this signal is applied to a saturated ferrimagnetic material, and also assumed that
the $|H| \ll H_0$, then the total magnetic field and magnetization in the material are

\[ H_t = \hat{z}H_0 + H \quad (2.17) \]
\[ M_t = \hat{z}M_s + M \quad (2.18) \]

where $M$ is the additional AC magnetization in the x-y plane, transverse to the direction of propagation. By substituting (2.17) and (2.18) into (2.16) and since $\frac{dM_s}{dt} = 0$, it can be assumed that $|H|M \ll |M|H_0$ and $|M|H \ll M_s|H|$. Ignoring MH products, the equations of motion can be reduced to

\[ \frac{dM_s}{dt} = -\omega_0 M_y + \omega_m H_y \quad (2.19) \]
\[ \frac{dM_y}{dt} = -\omega_0 M_x - \omega_m H_x \quad (2.20) \]
\[ \frac{dM_z}{dt} = 0, \quad (2.21) \]

where $\omega_0 = \mu_0 \gamma H_0$ and $\omega_m = \mu_0 \gamma M_s$. Solving (2.19) and (2.20) for $M_x$ and $M_y$ gives

\[ \frac{d^2 M_x}{dt^2} + \omega_0^2 M_x = \omega_m \frac{dH_y}{dt} + \omega_0 \omega_m H_x \quad (2.22) \]
\[ \frac{d^2 M_y}{dt^2} + \omega_0^2 M_y = \omega_m \frac{dH_x}{dt} + \omega_0 \omega_m H_y. \quad (2.23) \]
If the AC $\mathbf{H}$ and $\mathbf{M}$ fields have $e^{jwt}$ time harmonic dependences as

\[
H_x = H_x e^{jwt}
\]
\[
H_y = H_y e^{jwt}
\]
\[
H_z = H_z e^{jwt}
\]
\[
M_x = M_x e^{jwt}
\]
\[
M_y = M_y e^{jwt}
\]
\[
M_z = M_z e^{jwt},
\]

then the steady state forms of (2.22) and (2.23) reduce to the phasor equations as

\[
(\omega_0^2 - \omega^2)M_x = \omega_0 \omega_m H_x + j \omega \omega_m H_y
\]
\[
(\omega_0^2 - \omega^2)M_y = -j \omega \omega_m H_x + \omega_0 \omega_m H_y,
\]

which can be expressed in terms of susceptibility tensor, $[\chi]$,

\[
\mathbf{M} = [\chi] \mathbf{H} = \begin{bmatrix} \chi_{xx} & \chi_{xy} & 0 \\ \chi_{yx} & \chi_{yy} & 0 \\ 0 & 0 & 0 \end{bmatrix} \mathbf{H}.
\]
From equation (2.32), the elements of the susceptibility are written as follows

\[ \chi_{xx} = \chi_{yy} \]
\[ = \frac{\omega_0 \omega_m}{\omega_0^2 - \omega^2} \] \hspace{1cm} (2.33)

\[ \chi_{xy} = -\chi_{yx} \]
\[ = \frac{j\omega \omega_m}{\omega_0^2 - \omega^2} \] \hspace{1cm} (2.34)

and

\[ \mathbf{B} = \mu_0 (\mathbf{M} + \mathbf{H}) \]
\[ = \mu_0 ([\chi] \mathbf{H} + \mathbf{H}) \]
\[ = \mu_0 ([U] + [\chi]) \mathbf{H} \]
\[ = [\mu] \mathbf{H}, \] \hspace{1cm} (2.35)

where the tensor permeability from equation (2.35) is written as

\[ [\mu] = \begin{bmatrix} \mu & j\kappa & 0 \\ -j\kappa & \mu & 0 \\ 0 & 0 & \mu_0 \end{bmatrix} \text{ for } \hat{z} \text{ bias.} \] \hspace{1cm} (2.36)
From (2.36), it is obvious that the magnetic moment of the ferrimagnetic material in this case is not affected by the z-component of the AC magnetic field, $H$. However, if the bias field is applied other than in the z-direction, we have

$$[\mu] = \begin{bmatrix} \mu & 0 & j\kappa \\ 0 & \mu_0 & 0 \\ j\kappa & 0 & \mu \end{bmatrix}$$ for $\hat{y}$ bias, and

$$[\mu] = \begin{bmatrix} \mu_0 & 0 & 0 \\ 0 & \mu & j\kappa \\ 0 & -j\kappa & \mu \end{bmatrix}$$ for $\hat{x}$ bias. \hspace{1cm} (2.37)

The notations $\mu_0$, and $\mu$, are permeability in vacuum and material respectively,

$$\mu = \mu_0 \left(1 + \frac{\omega_0 \omega_m}{\omega_0^2 - \omega^2}\right)$$ \hspace{1cm} (2.39)

and

$$\kappa = \mu_0 \frac{\omega_0 \omega_m}{\omega_0^2 - \omega^2}.$$ \hspace{1cm} (2.40)

The parameters, $\mu$ and $\kappa$ are essential in the modeling of anisotropic materials because they are used to calculate the effective permeability of the material which subsequently determines the polarizations of the EM waves propagating through the material. For an infinite ferrite-filled region with static bias field given by $H_0 = \hat{z} H_0$, the effective permeability for left and right hand circular polarization waves are $\mu - \kappa$.
and $\mu + \kappa$ respectively. However, if the applied bias field is in either the x or y axis direction and provided the direction of propagation is in the z-direction, the effective permeability is determined as $\mu_e = \frac{(\mu^2 - \kappa^2)}{\mu}$. In order to determine $\mu$ and $\kappa$, extensive characterization of the ferrimagnetic material chosen in this research work is required, as detailed in the next chapter.
CHAPTER 3

BARIUM HEXAFERRITE AND COMPOSITES

3.1 Synthesis and structure

Microwave ferrites are magnetic ceramic materials that refer to the entire family of iron oxides such as garnets, spinels, hexaferrites, and orthoferrites [42, 45–47]. The most studied ferrites with different crystal structures and physical properties are presented in [48–54]. Hexaferrites have become very important materials for commercial and technological development, with BaM hexaferrite alone accounting for about 50% of the total magnetic materials manufactured globally, at over 300,000 tons per year [41]. Also since their discovery in the 1950s, there has been an increasing interest in the hexagonal ferrites, as depicted in Figure 3.1, as indicated by the number of hexaferrite papers published in 2011, while applications have drastically exploded as shown in Figure 3.2. This explosion of interest in hexaferrites in the last decade was a result of the rapid increase in demand for electronic components for mobile and wireless communications at microwave frequencies, electromagnetic wave absorbers for electromagnetic compatibility (EMC), radar absorbing material (RAM) and stealth technologies.
**Figure 3.1:** Yearly number of hexaferrites publications from 1959 to 2011 (search using Scopus) [41]

**Figure 3.2:** Yearly number of hexaferrites papers by specialised theme since 1975 (search using Scopus) [41]
Similarly the recent interest in the development of nano-fibers and fiber orientation as related to nanotechnology contributed to this explosion. The M-type hexaferrite \( \text{BaFe}_{12}\text{O}_{19} \), being the chosen anisotropic material in this effort, can equally be called barium ferrite, hexaferrite, barium hexaferrite, ferroxdure, \( \text{M ferrite} \) and \( \text{BaM} \). This material consists of a hexagonal structure as described by Went et al. [55]. The general structure of M type hexaferrite \( \text{AFe}_{12}\text{O}_{19} \), where \( \text{A} \) is a divalent ion (\( \text{Ba}^{2+} \), \( \text{Sr}^{2+} \), \( \text{Pb}^{2+} \)), is constructed from 4 building blocks, namely S, \( S^* \), R, and \( R^* \). The oxygen atoms are closely packed with the A and Fe ions in the interstitial sites. The structure consists of ten layers of oxygen atoms along the c-axis, and also positioned at five crystallographically sites are the Fe atoms. The S (\( \text{Fe}_6\text{O}_8 \)) and \( S^* \) blocks are spinels with 2 oxygen layers and six iron III ions. Two of the six \( \text{Fe}^{3+} \) ions are in tetrahedral sites with their spins counter parallel to the spins of the remaining four \( \text{Fe}^{3+} \) ions in octahedral sites. Those four \( \text{Fe}^{3+} \) ions have their spins aligned parallel to each other as shown in the schematic structure in Figure 3.3 [56]. Hexagonal \( \text{R (BaFe}_6\text{O}_{11}) \) and \( R^* \) blocks consist of three oxygen layers with one of the oxygen anions. Each \( \text{R block} \) contains six \( \text{Fe}^{3+} \) ions, of which five are in octahedral sites, three having spin up and two having spin down polarization. Barium ferrite belongs to the group of hexagonal ferrites which are all ferrimagnetic materials, and their magnetic properties are intrinsically linked to their crystalline structures. Hexagonal ferrites have a magnetocrystalline anisotropy (MCA), that is the induced magnetization has a preferred orientation within the crystal structure, and can be divided into two main groups namely uniaxial hexaferrites (with an easy axis of magnetization), and the ferroxplana or hexaplana ferrites (with an easy plane (or cone) of magnetization).
Figure 3.3: The schematic structure of the hexaferrite BaFe$_{12}$O$_{19}$. The arrows on Fe ions represent the direction of spin polarization [56]
Magnetic properties of hexaferrites can be controlled by a doping procedure. For instance, it is documented that magnetization and the anisotropy of barium ferrite can be increased by the substitution of Al$^{3+}$, Ga$^{3+}$ or Cr$^{3+}$; while substitution of Ti$^{4+}$Me$^{2+}$ or Ge$^{4+}$Me$^{2+}$ (Me$^{2+}$=Zn, Co, Ni) will decrease the magnetization and the anisotropy field. Generally, in order to lower the coercivity ($H_c$), decrease the positive temperature coefficient of $H_c$ and obtain fine particle size, the divalent-tetravalent metal elements such as La-Co [57–60], Ti-Co [61–63], Zn-Co [64], Zn-Ti [65, 66], Ir-Co [63, 67], Gd-Co, [68] and Ni-Zr [69] have been used. Also many other scholars [70–80] have reported that trivalent metal elements such as La, Es, Si, Ga, In, Co, Bi, Sb and Al must be substituted in order to increase the $H_c$ of the hexaferrites for the fabrication of permanent magnets but saturation magnetization $M_s$, or the anisotropy field $H_a$, or both decrease [56]. Also generally, M-type hexaferrite films can be deposited by various methods such as evaporation, metal-organic chemical vapor deposition (MOCVD), pulsed laser deposition (PLD), rf sputtering, sol-gel techniques, and liquid phase epitaxy (LPE) deposition technique [81–92]. For the sake of this research work, the barium ferrite powder employed in this investigation is as delivered by BGRIMM MAGMAT of China and is used directly with SU8 polymer and other viscosity controlled solvent for all the experiments.

### 3.2 Barium ferrite powder SU8 polymer

Ferrite material and organic polymer composites exhibited good magnetic properties and these materials found applications in high performance electronic systems operated at high frequencies [93]. Since micro-fabrication of these systems re-
quires that the ferrite material must be in a compatible form for processing, some polymer matrix is needed for binding and structural support. There are numerous polymers that could be used for this purpose. However, Microchem SU8 photoresist is chosen for its ability to be photo-defined and its high chemical resistance after exposure. Thus, one can pattern the ferrite and not worry about its dissolution during the remaining lithographic processes. Also other impressive properties of SU8 includes high aspect ratio, high contrast, and can it be used where chemically and thermally stable images are desired [94]. During the mixing process, this polymer alongside with other solvents, ethyl acetate and 1-cyano-ethyl-2-ethyl-4-methylimidazole (2E4MZ-CN), were used as viscosity control and catalyst respectively. The composite samples were then baked and cured in the oven at 90\(^{\circ}\) C for five hours. After the preparation of the composites, scanning electron micrograph images of both the powders and the composites were taken and presented in Figure 3.4. Figure 3.4a shows an fine barium ferrite powder (0.8-1 \(\mu m\)) compactly packed together, while in Figure 3.4b, the particles are considerably larger in size and termed coarse barium ferrite powder (3-6 \(\mu m\)). Particles are well adhered to the polymer and evenly distributed in Figure 3.4c as well as in Figure 3.4d of ratio 3:1 barium ferrite fine composite and ratio 1:1 barium ferrite coarse composite respectively. From Figure 3.4c and Figure 3.4d, it is clearly shown that ferrite particles are more in ratio 3:1 than in ratio 1:1. The resulting samples were cooled naturally to room temperature.
Figure 3.4: Scanning electron micrograph images: (a) 0.8-1 \( \mu m \) fine barium ferrite (BMS-3) powder, (b) 3-6 \( \mu m \) coarse barium ferrite (BMS-3) powder, (c) 0.8-1 \( \mu m \) fine barium ferrite (BMS-3) powder plus SU8 (3:1 fine barium ferrite powder to SU8 by mass), (d) 3-6 \( \mu m \) coarse barium ferrite (BMS-3) powder plus SU8 (1:1 coarse barium ferrite to SU8 by mass)

3.2.1 Magnetic measurements

A MicroSense EV9 Vibrating Sample Magnetometer, (VSM) shown in Figure 3.6 (a and b) was used to obtain data for saturation magnetization, magnetic remanence, and coercivity, which were in turn used to generate hysteresis loops of the powders and the composites. The preparation and mixing of the samples were carried out according to Section 3.2. An empty cylindrical glass tube was filled with a certain amount of the sample, and a glass rod stopper rolled with PTFE tape at one
end was inserted into the tube to hold the sample in place. The resulting unit was then inserted into the magnetometer for measurements, which then generated hysteresis data automatically. This experiment provided a direct comparison between SU8, ferrite/SU8 composite and the ferrite powders. In Figure 3.6, magnetic hysteresis loops of fine barium ferrite powder and its composite with photoresist were plotted. The measurements were taken with maximum applied static magnetic field of 21 kOe at room temperature. Approximately 45.8 emu/g at 6 kOe of saturation magnetization was measured for the fine barium ferrite powder and about 35.8 emu/g at 3.5 kOe for its composite with SU8. The result demonstrates an important property for the ferrite-SU8 composites in that upon removal of the applied field, the sample (Ferrite-SU8) retains up to 90% of its magnetization compared to approximately 65% retentivity of the powder alone. The high remanent magnetization of the composite is a desirable characteristic in microfabrication of microwave devices [95]. For instance, a latching phase shifter maintains its retentivity when the driving pulse is removed.
This is achieved by maintaining the active state made between the two (+$M_r$ and -$M_r$) stable states until a reverse bias field is applied [12]. Also because of this ferrite retentivity property, the need for external magnets for biasing may be eliminated under certain design conditions, thus a reducing weight and cost for microwave ferrite devices.

There is a reduction in coercivity, $H_c$ from 1570 Oersted fine (0.8-1 $\mu$m powder) to 461 Oersted (composite) which makes the composite less magnetically hardened. Figure 3.7, shows the magnetic hysteresis loops of two ratios of coarse barium ferrite powder to SU8 photoresist. The higher ratio of the powder (3:1) has higher saturation magnetization than the lower ratio (1:1), which is expected due to higher density of the powder in the mixture. Also, the lower ratio (1:1) sample has a lower coercivity, $H_c = 118$ Oersted compared to ratio 3:1 with coercivity, $H_c = 467$ Oersted. The reason
Figure 3.6: Magnetic hysteresis loops measured with the applied magnetic field for fine barium hexaferrite powder and its composite with SU8

Figure 3.7: Magnetic hysteresis loops measured with the applied magnetic field aligned perpendicular to the sample plane for two ratios of coarse barium hexaferrite powder to SU8
for the differences in the coercivity of the two ratios when the Barium ferrite powder is mixed with SU8 requires future investigation. Depending on the applications, the concentration of the ferrite material could be determined based on the available saturation magnetization and retentivity of the hysteresis loop of the ferrite material. In both figures, the SU8 control experiment demonstrates no magnetization under the applied field, which is expected for a nonmagnetic material. Having examined the static magnetic properties of these samples, it is important to determine the RF characteristics of the materials as well, and this is detailed in following subsection.

3.2.2 Electromagnetic characterization of ferrite powder and composites

In this measurement, only one size (0.8-1.0 micron) of the powder was used since the two particle sizes belong to the same ferrite type. At room temperature, the composite was packed into the sample holder for the WR28 waveguide for S-parameter measurements using the Agilent E8363C programmable network analyzer. The set up consists of the E8363C PNA with two 2.4 mm microwave cables, the coaxial/waveguide adapters and the sample holder of thickness 2.997 mm which was supplied by Maury Microwave, Ontario California, USA. The prepared ferrite samples were carefully loaded into a sample holder and then inserted into Ka-band rectangular waveguide (WR28: 7.112 mm by 3.556 mm) as shown in Figure 3.8. Several measurements were taken to be sure the sample thickness was the same as the holder thickness. Index pins were utilized to establish a proper flange alignment of the two adapters and sample holder. Based on the frequency of this waveguide and the sample holder, a TRL calibration was performed to establish the calibration planes of
Figure 3.8: PNA E8363C and 0.118 inch-sample holder for measurement

the waveguide before making the transmission and reflection measurements. For this effort we worked collaboratively to borrow a programmable network analyzer (PNA) from a NASA laboratory located at National Space Science and Technology Center (NSSTC) building, Huntsville Alabama, United States. Figure 3.8 shows model E8363C PNA employed in this experiment.

The data of the S-parameters measured with the PNA were then fed into Mat-

lab program written for solving Nicholas-Richardson-Wier (NRW) algorithm [96,97] to extract the complex permittivities and permeabilities of the samples. The expres-
sions for the determination of these parameters using the algorithm are summarized below as

\[ S_{11} = S_{11}^0 e^{j(0 \times \sqrt{k_0^2 - k_c^2})} \]  
\[ S_{21} = S_{21}^0 e^{j(L-t) \times \sqrt{k_0^2 - k_c^2}}, \]

(3.1) (3.2)

where \( k_0 = \omega \sqrt{\mu \epsilon} \), \( S_{11} \) and \( S_{21} \), are modified S-parameters while \( S_{11}^0 \) and \( S_{21}^0 \) are initial S-parameters at zero reference plane. Also \( k_c = \sqrt{(m \pi / a)^2 + (n \pi / b)^2} \). The complex permeability and permittivity of the tested material are \( \mu \) and \( \epsilon \) respectively, while \( \omega \) is the operating frequency, \( a \) and \( b \) are the dimensions of the waveguide, while combinations of \( m \) and \( n \) give the waveguide modes. \( L \) assumes the typical quarter wavelength difference between thru and line in air used to realize zero planes. For a \( TE_{10} \), \( k_c = \pi / a \), the complex propagation of the incident wave through the ferrite sample in the rectangular waveguide is obtained as

\[ \gamma = \frac{1}{t} \left[ \ln \left( \frac{1}{|T|} \right) + j(2\pi n - \varphi) \right], \]

(3.3)

where \( n = 0, \pm 1, \pm 2 \cdots \). For \( n = 0, -2\pi < \varphi < 0 \). The transmission coefficient is evaluated from the reflection coefficient and the modified scattering parameters as

\[ \Gamma = K \pm \sqrt{K^2 + 1}, \quad \text{and} \quad K = \frac{S_{11}^2 - S_{21}^2 + 1}{2S_{11}} \]
yielding,

\[ T = \frac{S_{11} + S_{21} - \Gamma}{1 - (S_{11} + S_{21})\Gamma}. \]

From above, the complex permeability and permittivity in terms of the thickness, \( t \), of the sample in the sample holder, reflection coefficient, \( \Gamma \), transmission coefficient, \( T \), wavelength in free space, \( \lambda_0 \), and the guide dimension, \( a \), corresponding to the complex propagation factor, \( \gamma \), is given as

\[
\mu = -j \frac{1}{\sqrt{1/\lambda_0^2 - (1/2a)^2}} \left( \frac{1+\Gamma}{1-\Gamma} \right) \left( \frac{\gamma}{2\pi} \right),
\]

(3.4)

\[
\epsilon = -j \frac{1}{\sqrt{1/\lambda_0^2 - (1/2a)^2}} \left( \frac{\epsilon}{j} \right)^2 \left( \frac{1+\Gamma}{1-\Gamma} \right) \left( \frac{\gamma}{2\pi} \right),
\]

(3.5)

The results obtained from return and insertion losses of the PNA measurement are presented in Figures 3.9 and 3.10, it is noticed that from these figures, the treatment of the powder with the solvent and SU8 polymer, increased the losses of the composites and also shifted the resonance frequencies relative to the resonance frequency of the powder. Although there are ten samples of composites measured using the PNA, but only few ratio samples were actually of interest to this investigation for the purpose of clarity. In Figure 3.9, resonance frequencies for ratios 3:5, 1:4, and 2:5 are shifted to lower frequencies of about 37 GHz, 36.5 GHz, and 36.3 GHz respectively. However, ratio 1:1 composite indicated two resonance frequencies of about 34 GHz and 31 GHz which are respectively termed as natural and domain wall resonance frequencies [98]. This shift in resonance frequency can be explained as
a result of changes in the crystalline anisotropy of the material samples when mixed with the polymer and solvents. All other resonance frequencies of other ratios occurred outside the swept frequency range. These outside resonance frequencies might have either occurred below or above the present frequency range. Also in Figure 3.10, the insertion loss increases from less than 1 dB to about 7 dB as the concentration of the powder increases in the composites.

![Figure 3.9: Measured S_{11}-parameter plots for Barium powder and composites](a)

Figure 3.9: Measured S_{11}-parameter plots for Barium powder and composites
Finally, the permittivity and permeability of the fine barium ferrite powder and its four composites are extracted using the Matlab codes and presented in the following figures. In Figure 3.11 and Figure 3.12, over a range of frequencies between 26.5 GHz and 40 GHz, the range of complex relative permittivity is deduced as

$$\epsilon_r = (3.5 - 6.32) - j(0.65 - 2.63)$$

for the ratios 1:10, 2:5, 7:10, and 4:5 of the ferrite polymer samples compared to the powder. The real part of the permittivity of the powder is extracted to be in the range of $$\epsilon' = (6.5 - 8.5)$$ within 26.5 GHz and 31.5 GHz, while $$\epsilon''_{max} = 6.9$$ at about 33.4 GHz.
**Figure 3.11**: Real part of complex permittivity of barium powder and four composites

SU8 mixed with barium hexaferrite powder (0.8 - 1 micron); ratio = mass powder to mass SU8

**Figure 3.12**: Imaginary part of complex permittivity of barium powder and four composites
The corresponding complex relative permeability extracted from Figure 3.13 and Figure 3.14. The complex relative permeability over the entire frequency range were deduced to be $\mu_r = (1.1 - 1.5) - j(0.1 - 0.4)$ for composites while the powder permeability (real part) in Figure 3.13 decreases from $\mu' = 3.8$ at 26.5 GHz to about $\mu' = 2.5$ at 32 GHz before the dip at about 34 GHz. The maximum imaginary permeability of the pure powder in Figure 3.14, $\mu''_{max} = 3.1$ at 34 GHz was due to the natural resonance, $f_{res} = (\mu_0 \gamma H_A)/(2\pi)$ with $\mu_0 = 4\pi \times 10^{-7}$ H/m the permeability of free space, $\gamma$ the gyromagnetic ratio, and $H_A$ the c-axis magnetocrystalline anisotropy field. The spin-orbit interaction is the primary source of magnetocrystalline anisotropy. The magnetocrystalline anisotropy is basically the orbital motion of the electrons that couples with the electric field of the crystal giving rise to the first order contribution. The second order arises due to the mutual interaction of the magnetic dipoles of the material. Thus, any shift in frequency as a result of mixing the powder with the polymer matrix is basically a function of the c-axis magnetocrystalline anisotropy field, $H_A$. Also from plot Figure 3.14, there exists another imaginary permeability peak of 0.4 at 29 GHz, but this might have been due to the domain wall resonance of the powder.
Figure 3.13: Real part of complex permeability of barium powder and four composites

Figure 3.14: Imaginary part of complex permeability of barium powder and four composites
From these results, as the concentration was increased, the permittivity increased up to 40% of the powder concentration but after which the permittivity began to decrease as a result of additional increase in powder concentration. Some of the data of absolute values for the relative permittivity and permeability are presented in Table 3.1.

**Table 3.1:** Permittivity and Permeability of barium hexaferrite powder (0.8-1.0) µm and composites

<table>
<thead>
<tr>
<th>Freq. (GHz)</th>
<th>$\mu_{eff}$, (pwd)</th>
<th>$\mu_{eff}$, 1:1</th>
<th>$\mu_{eff}$, 3:1</th>
<th>$\epsilon_{eff}$, (pwd)</th>
<th>$\epsilon_{eff}$, 1:1</th>
<th>$\epsilon_{eff}$, 3:1</th>
</tr>
</thead>
<tbody>
<tr>
<td>26.5</td>
<td>4.0</td>
<td>1.5</td>
<td>1.2</td>
<td>6.4</td>
<td>3.5</td>
<td>7.1</td>
</tr>
<tr>
<td>28.0</td>
<td>3.4</td>
<td>1.3</td>
<td>1.2</td>
<td>6.9</td>
<td>3.9</td>
<td>7.7</td>
</tr>
<tr>
<td>30.0</td>
<td>2.9</td>
<td>1.2</td>
<td>1.3</td>
<td>7.7</td>
<td>4.3</td>
<td>7.3</td>
</tr>
<tr>
<td>32.0</td>
<td>2.0</td>
<td>1.1</td>
<td>1.5</td>
<td>8.2</td>
<td>4.6</td>
<td>6.3</td>
</tr>
<tr>
<td>34.0</td>
<td>3.1</td>
<td>1.1</td>
<td>1.4</td>
<td>5.9</td>
<td>4.7</td>
<td>6.8</td>
</tr>
</tbody>
</table>

The dielectric and magnetic loss tangents of permittivity and permeability are presented respectively in Figure 3.15 and Figure 3.16. In the Figure 3.15, permittivity has a range of dielectric loss tangent, $\tan \delta_\epsilon = 0.11 - 0.55$, for ratios 1:10, 2:5, 7:10, and 4:5 composites compared to the powder alone with $\tan \delta_\epsilon = 0.01 - 0.43$ outside the resonance zone.
On the other hand, magnetic loss tangent, $\tan \delta_{\mu} = 0.01 - 0.43$ was recorded in Figure 3.16. There is a sudden increase in the magnetic losses of the powder within the frequency range 32 GHz through 36 GHz in Figure 3.16; this was due to ferromagnetic absorption of the material at this frequency range. From this result, it shows that the treatment of the powder with the polymer made the composites a little lossy and also made the composites resonance occur outside the frequency range 26.5-40 GHz.

Figure 3.15: Loss tangent of permittivity for barium powder and four composites
The four composite samples dielectric losses fluctuate between 0.1 and 0.5 in the entire frequency range. The lowest dielectric loss was recorded in ratio 2:5 at about 37 GHz. The composite samples experienced lower magnetic losses compared to the dielectric losses as shown in Figure 3.16. The magnetic losses fluctuate between 0.01 and 0.3 within the frequency band. Generally speaking it can be concluded that the losses experienced by the composite samples are mostly from the dielectric.

3.2.2.1 Characterization using Quasi-optical millimeter wave spectroscopy

High RF powered Backward Wave Oscillator (BWO) technique is a form of free space quasi-optical spectrometry. In this case, a coherent high source of power is generated by using backward wave oscillators tunable in the 35-80 GHz frequency
range. The backward wave oscillator method showed that a variation in the chemical composition of ferrites leads to a change in the magnetic properties and absorption of materials in the millimeter waves. Formation and focusing of the Gaussian beam on the sample were achieved by a full-band horn antenna and a set of polyethylene lenses [99,100]. Disc-shaped planar samples were made and placed between the poles of the magneto-static circuit which can provide a variable static magnetic field in excess of 7.5kOe (60,000 A/m). The measurements were performed in the frequency sweep mode to obtain the transmittance of different samples of barium hexaferrite powder and composites. The composite samples used in this measurement were prepared according to Section 3.2 of Chapter 3. Transmittance data are used for the computation of effective permeability, thus, effective complex magnetic permeability is given as [101,102]:

\[
\mu_{eff} = \frac{1}{3} \left( \frac{(H_A + 4\pi M_s)^2 - \left( \frac{\omega}{\gamma} \right)^2}{H_A^2 - \left( \frac{\omega}{\gamma} \right)^2} \right)^{\frac{1}{2}},
\]

where \( \omega \) is the angular frequency, \( H_A \) is anisotropy field, \( 4\pi M_s \) is the saturation magnetization, and \( \gamma \), the gyromagnetic ratio. Demagnetizing factors are determined by the theory of Schlomann’s model for non-ellipsoidal bodies. The real and imaginary parts of the complex magnetic permeability of the powders and the magnetic composites are shown in Figures 3.17, 3.18, 3.19 and 3.20. The permeability spectra appear to be almost the same, although the transmittance spectra of the coarse ferrite photoresist composite exhibit lower levels compared to transmittance spectra for coarse barium
powder. The reflectance from the photoresist composite appears to be much higher than the one for the barium ferrite powder causing the lower transmittance spectra. The strength of ferromagnetic absorption of fine barium photoresist composite is higher than the fine barium powder. The ferromagnetic resonance frequency shifts to 49.2 GHz which may be due to the bonding effect from the photoresist between the fine barium powder particles. The existence of photoresist enhances the interaction between particles and prevents reduction of the crystalline anisotropy from thermal activation.

Figure 3.19: Real part: BWO complex permeability of barium powder (0.8-1 µm) and composite
Figure 3.17: Real part of complex permeability of barium powder (3-6 µm) and composite using BWO.

Figure 3.18: Imaginary part of complex permeability of barium powder (3-6 µm) and composite using BWO.
Figure 3.20: Imaginary part: BWO complex permeability of barium powder (0.8-1 \( \mu \text{m} \)) and composite

Transmittance spectra of the fine barium ferrite and its photoresist composite are shown in Figure 3.21. Transmittance for coarse barium powder and its photoresist composite is shown in Figure 3.22. The ferromagnetic resonant frequencies of coarse powder and its composite appear at the same range as the bulk hexagonal barium ferrite magnets. The resonant frequency of fine barium ferrite powder shows a shift to lower frequencies compared to resonance frequency for the coarse powder. This is expected due to the submicron particle size of the fine barium powder \[103\].
As the particle size reduces to be comparable to single domain size, the thermal activation is becoming dominant and canceling out part of the magneto crystalline anisotropy [104]. The very fine ferrite particles have a relative large air gap between each other which reduces the strength of the interaction between separated particles. When the fine hexagonal barium powder is mixed with the photoresist to form the composite, the ferromagnetic resonance shifts back to the original frequency. The fine powder composite behaves similar to the bulk hexagonal barium powder. The values of the strong uniaxial anisotropy, saturation magnetization are acquired by employing the curve fitting method for ferrite with isotropic dielectric permittivity.
described in [105]. The ferromagnetic resonant frequencies and anisotropic magnetic fields are shown in Table 3.2

<table>
<thead>
<tr>
<th>Material</th>
<th>FMR (GHz)</th>
<th>Anisotropic field, $H_A$ (kOe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BaM powder (0.8-1 micron)</td>
<td>46.3</td>
<td>16.5</td>
</tr>
<tr>
<td>BaM powder composite (0.8-1 micron)</td>
<td>49.2</td>
<td>17.6</td>
</tr>
<tr>
<td>BaM powder (3-6 micron)</td>
<td>49.2</td>
<td>17.6</td>
</tr>
<tr>
<td>BaM powder composite (3-6 micron)</td>
<td>49.3</td>
<td>17.6</td>
</tr>
</tbody>
</table>

In conclusion, the fine barium ferrite with 0.8 to 1 micron particles size exhibits ferromagnetic resonance at 46.3 GHz which is much lower than the resonance frequency position for the coarse barium powder with 3 to 6 micron particle size. The coarse powder and its composite have ferromagnetic resonance similar to the bulk hexagonal barium ferrite. The composite of the fine ferrite powder also shows higher ferromagnetic resonance frequency than the fine barium powder. The blending of the SU8 photoresist with the fine powder shifts the ferromagnetic resonance from 46.3 GHz back to 49.2 GHz. Similarly, these composites combine the high frequency anisotropic magnetic field, $H_A$ sensitivity, with photolithographic fabrication capability. Thus Barium ferrite powders and their composites with SU8 will be useful in a variety of high frequency applications especially in on-chip magnetic components and microwave phase shifters for MMIC circuits, where classically machined and polished
monolithic ferrite materials are too large to be incorporated. Also it is noticed that
the results obtained with this method are in agreement with the results obtained
when the waveguide TRL method was used in obtaining the magnetic permeability.

![Transmittance spectra: coarse barium ferrite powder (3-6 µm) and composite using BWO](image.png)

**Figure 3.22:** Transmittance spectra: coarse barium ferrite powder (3-6 µm) and composite using BWO
CHAPTER 4

STRONTIUM HEXAFERRITE POWDER AND COMPOSITES

4.1 Strontium ferrite powder and composite with SU8 polymer

The strontium (SrFe$_{12}$O$_{19}$) ferrite powders, from Bgrimmm magmat, China used in this research investigation are anisotropic hard ferrite powders for wet processing. For this research work, coarse and fine samples used have the following mass ratios of the powders to the SU8 photoresist: 1:1 ratio by mass was prepared from 3 grams each of both strontium (BMS-4) ferrite powder and SU8 polymer, while 3:1 by mass composite was prepared from 6 grams of powder and 2 grams of SU8. After the preparation of the composites, scanning electron micrograph images of both the powders and the composites were taken and are presented in Figure 4.1. In Figure 4.1a, the image shows an inhomogeneous granular structure (uneven sized particles) simply because the particles are coarse with structural particle size within 3-6 microns. In Figure 4.1b, the particles are compactly packed together and the grain boundaries are visible in the structure. Particles are well adhered to the polymer and evenly distributed in Figure 4.1c as well as in Figure 4.1d. The gaps in the composites in Figure 4.1(c and d) can greatly be reduced with higher concentration of strontium ferrite powder where higher magnetic flux is required depending on the applications.
Because of the ferrimagnetic nature of the powder, a nonmagnetic stirrer was used to blend the strontium ferrite powder with SU8 polymer to prevent unwarranted magnetization.

The EDAX equipment was used to verify the strontium ferrite stoichiometry resulting in the determination of the percentage compositions of the compound. The percentage composition actually obtained matched the stoichiometric percentage of strontium ferrite as shown in Table 4.1. Energy Dispersive X-ray spectrometry of strontium ferrite powder in Figure 4.2 showed the detail of the sample composition. From the plot, all the elements are well resolved by K- and L-shell X-rays with fewer artifacts. Energy of the emitted X-rays is a characteristic of the element, since the energy levels of the shells are directly related to the number of charges in the nucleus of the element. The peaks associated with K-shell X-ray correspond to atomic numbers 4 through 32 and L shell X-ray peaks correspond to atomic number 22 through 79. From Figure 4.2, Fe is the highest energy element with about 5.5 keV on the Ka-shell while Strontium has 1.9 keV on the La-shell. The weight percent of Fe is about 80% while Strontium is roughly 20% as shown in Table 4.1. This composition conforms to the theoretical stoichiometry of the compound.

4.1.1 Ferromagnetic resonance - FMR

FMR experiment is a spectroscopic method employed to investigate and obtain information about the magnetization of magnetic materials. It is a standard technique for investigating spin waves and spin dynamics. The method probes the material
Figure 4.1: Scanning electron micrograph images: (a) 3-6 µm coarse strontium ferrite (BMS-4) powder, (b) 0.8-1.0 µm fine strontium ferrite (BMS-4) powder, (c) 3-6 µm coarse strontium ferrite (BMS-4) powder plus SU8 (3:1 coarse strontium ferrite to SU8 by mass), (d) 0.8-1.0 µm fine strontium ferrite (BMS-4) powder plus SU8 (3:1 fine strontium ferrite to SU8 by mass).

Figure 4.2: Spectrum of strontium ferrite powder collected at 20 keV using Edax
magnetization that occurred as a result of magnetic moments of unpaired dipolar-coupled electrons. Ferromagnetic resonance develops from a precessional motion of magnetization of ferromagnetic materials when an external field is applied. The external field exerts a twisting force on the magnetization of the magnetic sample, and this in turn makes the magnetic moments in the sample change their orientation with respect to the rotational axis. In this research, a broadband ferromagnetic resonance (FMR) was used to characterize the dynamic properties of the samples. In FMR experiments, the sample is subject to a strong static external magnetic field and a small radio frequency magnetic field typically oriented perpendicular to the static field \[106\]. Here we show results for field-swept FMR, for which the microwave frequency is held constant while the external magnetic field is swept through the resonance of the sample under investigation. The broadband FMR setup in this study utilizes a coplanar waveguide \[107\] that enabled the measurement of the ferromagnetic resonance spectra up to 67 GHz. Field-swept spectra were fitted to accurately extract both the resonance field and the peak-to-peak linewidth.

**Table 4.1: Results of Figure 4.2**

<table>
<thead>
<tr>
<th>Element</th>
<th>Line</th>
<th>Energy [kEv]</th>
<th>Weight [%]</th>
<th>Compound</th>
<th>Cmpd Wt [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>KA1</td>
<td>6.403</td>
<td>56.26</td>
<td>Fe\textsubscript{2}O\textsubscript{3}</td>
<td>80.44</td>
</tr>
<tr>
<td>Sr</td>
<td>LA1</td>
<td>1.806</td>
<td>16.54</td>
<td>SrO</td>
<td>19.56</td>
</tr>
<tr>
<td>O</td>
<td>KA1</td>
<td>0.523</td>
<td>27.20</td>
<td>\cdots</td>
<td>\cdots</td>
</tr>
<tr>
<td>Total</td>
<td>\cdots</td>
<td>\cdots</td>
<td>100</td>
<td>Total</td>
<td>100</td>
</tr>
</tbody>
</table>
4.1.2 FMR results and discussion

In Figure 4.3, ferromagnetic resonances of fine strontium ferrite powder and its composites are compared. In the Kittel graph, Figure 4.4, the resonance field shifted from 3.52 kOe (Sr powder) to 2.74 kOe and 2.83 kOe at 60GHz respectively for 3:1 and 1:1 composites. Fine composite, ratio 3:1, shift rate decreases and then approaches the fine powder resonance field at 65 GHz. From this characteristic, it is recommended to operate below the resonance frequency of 60 GHz which implies that any micro or millimeter wave device could be designed to operate up to 55 GHz in order to have a good margin from the absorption zone. This zone should be completely avoided otherwise most of the wave energy would be converted to losses in the ferrite medium for $\mu$-RCP application. Also Figure 4.5, shows the ferromagnetic resonances of coarse strontium ferrite powder and its composites. From the figure, the peak decreases as the concentration was increased.
Figure 4.3: Ferromagnetic resonance comparisons of fine powder and composites

60GHz

Figure 4.4: Resonance field comparison for fine samples
On the other hand, resonance fields shifted towards the lower fields for both 1:1 and 3:1 coarse composites as shown in Figure 4.6 at 60 GHz. Comparing 1:1 and 3:1 coarse composites at 60 GHz, it could be implied that the higher the strontium ferrite powder concentration in the composite, the more the shift towards lower resonance fields. However, it could also be submitted that mixing of SU8 with fine and coarse strontium ferrite powders, has strong effects in shifting resonance fields towards the lower fields as depicted between 56 and 65 GHz in Figure 4.6.

![Figure 4.5: Ferromagnetic resonance comparisons of coarse powder and composites 60GHz](image)

There is a strong interest in the magnitude of the linewidth, because it is a measure of the width where the electromagnetic fields are absorbed in the material. In Figure 4.7, linewidth comparisons are made on the samples within the 57 and 67
GHz frequency range based on the respective composite ratios and the particle sizes. Three out of four samples showed little change over the frequency range while the fourth sample (Fine strontium ferrite composite, 3:1), demonstrated a significantly increased linewidth with frequency. Coarse and fine composite samples, 1:1 and coarse composite sample, 3:1 have their linewidth between 1,750 and 1,950 Oe while fine composite sample, 3:1 had the highest linewidth. Reasonable selection could be made among the composite ratios, although low values of FMR linewidth provide better performance for practical millimeter-wave device applications. A broadened linewidth can also be beneficial in absorbing applications.

4.1.3 Static magnetic characterization - SrM powder and composites

Static magnetic properties of strontium ferrite powders and their corresponding composites with SU8 were characterized with the Vibrating Sample Magnetometer. The measurements were taken with a maximum applied magnetic field of 21 kOe at room temperature. Figure 4.8 shows magnetic hysteresis loops of the two different sized strontium ferrite powders and the SU8 photoresist. At the maximum field of 21 kOe, the strontium ferrite powders are not fully saturated due to large intrinsic magnetic anisotropy field (H_k). Magnetization (Ms) at 21 kOe was measured to be 65.5 emu/g for fine powder and 76.2 emu/g for coarse powder. In addition, fine strontium ferrite powder showed a larger coercivity (H_c) of 1.6 kOe than 860 Oe for the coarse powder. This result indicates that more energy is required to demagnetize the fine sample material than the coarse sample. Also as expected, the SU8 photoresist is non-magnetic and therefore not likely to have serious adverse effects on the
Figure 4.6: Resonance field comparison for coarse samples

Figure 4.7: Linewidth $\Delta H$ comparison of fine and coarse strontium powder composite with SU8
composites when used for the fabrication of micro and millimeter wave devices. The squareness ratio, SQR values (Mr/Ms), were determined by the ratio of remanent magnetization to magnetization at 21 kOe and recorded for both the fine and coarse powders. They are 0.451 and 0.392, which are the measures of the squareness of their hysteresis loops. Also from this figure, 3:1 composite has the largest squareness ratio of 0.72, and 1:1 composite the lowest with a squareness ratio value of 0.391, which actually approached the squareness of the coarse Strontium ferrite powder. The remanence values on the other hand for 1:1 and 3:1 composites are 5.80 and 26 emu/g respectively indicating that the 3:1 composite will have a better retentivity than the 1:1 composite if the two composites are adopted in a memory related application.

![Figure 4.8: Hysteresis loops for coarse and fine strontium ferrite powder and SU8](image)

Figure 4.8: Hysteresis loops for coarse and fine strontium ferrite powder and SU8
Figure 4.9: Hysteresis loops for two ratios of fine strontium ferrite powder and composites

In Figure 4.9, $H_c$ of fine strontium ferrite composites were controlled with the mass ratio of strontium ferrite powder to SU8. This means that for an application where reversal of the magnetization at low fields is required, this variational dosing method could be a great choice. The pure strontium ferrite powder has the highest coercivity of 1.6 kOe and ratio 1:1 composite shows the lowest coercivity of 73 Oe while ratio 3:1 composite measured about 340 Oe. Saturation magnetization, $M_s$, observed at 21 kOe for fine powder, 1:1 and 3:1 composites are 65.5, 14.72 and 36.10 emu/g. Figure 4.10 compared three magnetic hysteresis loops of the coarse strontium powder and composites. From the figure, increasing the ferrite concentration and the mixing
powder with SU8 greatly improved the squareness ratio values of the composites, as depicted in the two ratios 1:1 and 3:1 with the squareness 0.43 and 0.51 respectively. The squareness ratio values obtained were a result of saturation magnetization and remanence of both 1:1 and 3:1 composites. The saturation magnetization for 1:1 and 3:1 are respectively 25.35 and 41.71 emu/g, while 10.9 and 21.0 emu/g were correspondingly measured for the remanence. In terms of hardness, it is shown in Figure 4.10 that the composite 3:1 sample exhibited a harder tendency than the 1:1 composite as a result of its 648 Oe coercivity which is about 240 % larger.

The summary of the hardness for the two powders, fine and coarse, with their respective composite ratios is clearly illustrated in Figure 4.11. Here the magnetic hardness increases from the fine composite 1:1, followed by coarse composite 1:1, then fine composite 3:1 and finally coarse composite 3:1. Their coercivity values respectively are 73, 193, 344, and 648 Oe. On the other hand, the remanence value progressively decreases from coarse composite 3:1, followed by fine composite 3:1, coarse composite 1:1 to the fine composite 1:1, which has the lowest average remanence, $M_r = 5.8$ emu/g. Also the largest squareness ratio value was recorded for fine Strontium ferrite composite 3:1 as 0.72. This largest squareness value has an improvement percentage of 85% relative to fine composite 1:1 with the smallest squareness of 0.39. This analysis serves as a guide for the selection of the right ferrimagnetic material and composition percentages required for any microwave application. Finally the saturation magnetization of the two powders dropped when they are mixed with the SU8. Coarse powder had the highest saturation magnetization followed by the fine powder and each powder composite behaves the same way with the same ratio.
Figure 4.10: Hysteresis loops for two ratios of coarse strontium ferrite powder and composites

Figure 4.11: Comparison of magnetic hysteresis loops for the two ratios of fine and coarse strontium ferrite powder composites.
relative to their fine counterpart. Thus, it is reasonable to conclude that saturation magnetization is a function of the particle sizes, homogeneity of the structure, and the concentration of the powder in SU8.

4.2 Ferrite-SU8 saturation magnetization and the previous published work

In this section, comparisons are made between the static magnetic properties obtained in this investigation and already published work. In particular, saturation magnetization, $\mu_s$ values are compared. From the comparison results in Table 4.2, the results obtained in this work are in total agreement with already published work.

**Table 4.2: Saturation magnetization and the previous published work**

<table>
<thead>
<tr>
<th>Reference</th>
<th>Saturation magnetization, $M_s$ (emu/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Xu, et al, [108]</td>
<td>SrM and $\alpha$-Fe$_2$O$_3$: 26-58</td>
</tr>
<tr>
<td>Yang, et al, [109]</td>
<td>Ni-Zn-Cu, Co$_2$Z-ferrite + photoresist:</td>
</tr>
<tr>
<td></td>
<td>30-41</td>
</tr>
<tr>
<td>Dong, et al, [111]</td>
<td>Cobalt ferrite: 39.7 @ 0.98 g cm$^{-3}$, 53.7</td>
</tr>
<tr>
<td></td>
<td>@ 2.77 g cm$^{-3}$</td>
</tr>
<tr>
<td>This work (Ferrite-SU8 composites)</td>
<td>BaM and SrM: 14-76</td>
</tr>
</tbody>
</table>
4.3 RF measurement of strontium ferrite powder and composites

Figure 4.12 shows the plots of insertion and return losses of the powder (3-6 µm) and its composites using Agilent programmable network analyzer (PNA) model E8363C. The coaxial/waveguide adapters (2.4 mm) and the sample holder of thickness 0.118 inches were supplied by Maury Microwave, Ontario California, USA. The powder and the cured composites are prepared and put into the sample holder to fill up the entire thickness. Several measurements were taken to be sure the sample thickness is the same as the holder thickness. A TRL calibration was performed to establish the calibration planes of the waveguide before making the measurements. The measured S-parameter data are also used in extracting the permittivity and permeability of the powder along with the composites using Nicolson-Ross-Wier algorithm [96,97]. From Figure 4.12, it is evident that mixing of the powder with SU8 made the natural resonant frequency of the material to be shifted towards the higher frequency zone as the concentration of the powder increases. This resonance effect is determined by the valleys of return loss (S11) plots.
Figure 4.12: Measured S-parameters of the powder and the composite ratios of strontium ferrite material to SU8 photoresist.

The permittivity and permeability of the coarse strontium ferrite samples were extracted from the measured S-parameters using the Nicolson-Ross-Wier algorithm written in Matlab m-file. Accuracy of the complex permittivity and permeability largely depends on how accurate the reflection coefficients are measured using the Agilent programmable network analyzer. In Figure 4.13 and Figure 4.14, the real and imaginary permittivity of the powder and the two composites are plotted. In the figure, the permittivity of the powder is extracted to be in the range of $\epsilon_r=(0.11-4.65)-j(0.13-0.67)$ over 27-40 GHz frequency range. The composite complex permittivities
are \( \epsilon_r = (2.83-7.24) - j(2.67-7.10) \) and \( \epsilon_r = (3.8-12.15) - j(1.56-6.47) \) for ratio 1:1 and 3:1 respectively over the entire frequency range. From these results, increasing the concentration of the powder in the composite shifts the natural resonance frequency to the higher frequency zone.

**Figure 4.13**: Real part of complex permittivity for strontium ferrite powder and composites
These results show that depending on the application of interest, a complex permittivity could be obtained for a specific frequency within the frequency band. The results also indicate that mixing of the powder with SU8 photoresist for the ultimate micro-fabrication would not have adverse effect on the dielectric property of the powder. In Figure 4.15, the permittivity loss tangent of the fine strontium ferrite powder and composites are plotted where the powder has a loss factor in the 0.09-0.20 range within 28.5 and 40 GHz, but with a significant loss factor of 27 at
about 27 GHz. The ranges of loss factor experienced by ratios 1:1 and 3:1 composites are 0.5-1.8 and 0.28-0.67 respectively in the entire frequency range.

Figure 4.15: Permittivity loss tangent of strontium ferrite powder and composites
Figure 4.16: Real part of complex permeability for strontium ferrite powder and composites
The real and imaginary parts of complex permeability and their corresponding losses in the samples are presented as follows. From Figure 4.16 and Figure 4.17, permeability values are extracted from the plots as $\mu_r=(0.81-1.88)-j(0.43-1.20)$ and $\mu_r=(0.57-1.79)-j(0.05-0.57)$ at 26.5-40 GHz for ratios 1:1 and 3:1 respectively. Also the complex permeability obtained for the strontium ferrite powder is $\mu_r=(0.63-2.3)-j(0.18-0.24)$ within 29 and 40 GHz frequency range. Similarly, the magnetic losses experienced by the powder when mixed with SU8 for the two ratios are not very different from that of the original strontium ferrite powder within 28-40 GHz frequency range, as shown in Figure 4.18.
In this figure, magnetic loss factor ranges are 0.06-0.13, 0.27-1.1, and 0.005-0.28 respectively for powder, composite 1:1, and composite 3:1. From these results, the RF performances of these composites are not adversely affected by mixing the powder into SU8 polymer matrix.

4.4 Ferrite-SU8 RF characteristics and the previous published work

RF characterization involved the determination of permittivity and permeability of the ferrite-SU8 composites. In the observed frequency range, 26.5-40 GHz, different complex values were extracted using NRW algorithm. These values were then compared with the results of the studies carried out by Jia, et al, [113] and Kim,
et al, [114]. From Table 4.3, the results reported in this work for both the barium and strontium ferrite composites are in total agreement with the published work of these scholars.

**Table 4.3:** Permittivity/permeability and the previous published work

<table>
<thead>
<tr>
<th>Reference</th>
<th>Permittivity, permeability (Real and imaginary), 26.5-40 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jia, et al, [113]</td>
<td>Ti-doped BaM: $\epsilon' = 4.2-5.4$, $\epsilon'' = 0.1-0.2$, $\mu' = 0.8-1.5$, $\mu'' = 0.01-0.55$</td>
</tr>
<tr>
<td>Kim, et al, [114]</td>
<td>Ti, Co substituted BaM: $\epsilon' = 7.8-8.0$, $\epsilon'' = 0.1-0.2$, $\mu' = 1.0-5.0$, $\mu'' = 0.01-0.50$</td>
</tr>
<tr>
<td>This work</td>
<td>SrM: $\epsilon' = 0.1-4.5$, $\epsilon'' = 0.01-0.5$, $\mu' = 0.5-1.8$, $\mu'' = 0.01-0.05$ (28-40 GHz), BaM: $\epsilon' = 0.5-8.5$, $\epsilon'' = 0.2-1.0$ (excluding 32-36 GHz), $\mu' = 0.5-3.6$, $\mu'' = 0.1-0.4$ (excluding 32-36 GHz)</td>
</tr>
</tbody>
</table>
CHAPTER 5

MICRO-RECTANGULAR COAXIAL PHASE SHIFTER, (μ-RCP)

5.1 Rectangular coaxial waveguides

Rectangular coaxial waveguides consist of a rectangular inner conductor located inside a rectangular outer conductor. The inner conductor is placed such that it is either symmetric or asymmetric inside the hollow guide such that the depth of the two conductors are equal, similar to a typical cylindrical coaxial transmission line. Electromagnetic fields inside this geometry are very close to those in a cylindrical coaxial transmission line, especially when the inner conductor is very small [21]. However, if the ratio of width to depth in both conductors is large, and the inner conductor forms a flat strip, then the field patterns in the rectangular transmission line approach those existing in a shielded strip line. The type of transmission line could serve to transmit microwave energy and also be used as an impedance transformer. A typical rectangular coaxial transmission line two-dimensional schematic is shown in Figure 5.1. It consists of two coaxial rectangular copper conductors which propagate electromagnetic energy in and out of the structure. The outer conductor is grounded while the inner conductor acts as the signal line. The gaps between the outer conductor and inner conductor dimension are used in the determination of capacitance and
inductance per length of the guide, which in turn are employed in the characteristic impedance calculation. The gaps, widths, and thicknesses of the waveguide shown in the schematic are as follows:

\[ w = \text{inner conductor width} \]
\[ w_g = \text{Gap between the inner conductor and the sidewall of the waveguide} \]
\[ t = \text{Thickness of the inner conductor} \]
\[ t_g = \text{Gap between the inner conductor and the lower or upper ground conductor}. \]

In this transmission line, TEM waves are propagated. Characteristic impedance of such a line is calculated as

\[ Z_0 = \sqrt{\frac{L_{\text{perlength}}}{C_{\text{perlength}}}} = \frac{1}{(v_{\text{phase}} \times C_{\text{perlength}})}, \quad (5.1) \]

where the velocity of propagation or phase velocity, \( v_{\text{phase}} \) can be written as

\[ v_{\text{phase}} = \frac{1}{\sqrt{\mu \varepsilon}} = \frac{1}{L_{\text{perlength}} \times C_{\text{perlength}}}, \quad (5.2) \]
Figure 5.1: 2-D schematic rectangular coaxial transmission line

Figure 5.2: 3-D air-filled rectangular coaxial waveguide
Here $L_{\text{per length}}$ is the inductance per unit length, and $C_{\text{per length}}$ is the capacitance per unit length. According to Chen [21], the capacitance per unit length for a line with $t > t_g$ and $w > w_g$ is given as

$$C_{\text{per length}} = 2\epsilon \left( \frac{w}{t_g} + \frac{t}{w} \right) + \frac{4\epsilon}{\pi} \left[ \ln \left( \frac{w^2 + t^2}{4t^2_g} \right) + \frac{2t_g}{w_g} \tan^{-1} \left( \frac{w_g}{t_g} \right) \right] + \frac{4\epsilon}{\pi} \left[ \ln \left( \frac{w^2 + t^2}{4w^2_g} \right) + \frac{2w_g}{t_g} \tan^{-1} \left( \frac{t_g}{w_g} \right) \right]. \quad (5.3)$$

While the lower cutoff frequency of TEM transmission line is 0 Hz, the upper cutoff frequency is set by the lowest mode that will propagate on the transmission line. The mode with the lowest cutoff frequency for a rectangular coaxial transmission line is either TE$_{10}$ or TE$_{01}$ mode [115]. However, because of symmetry, these modes are the same as the modes in a ridged waveguide. Thus, the cutoff frequency of a TE$_{10}$ mode in a ridged waveguide is calculated from the transcendental equation as

$$\cot \left( \frac{2\pi t_g f_c}{c} \right) - \left( 1 + \frac{w}{2w_g} \tan \left( \frac{t\pi f_c}{c} \right) \right) - \frac{B}{Y_c} = 0 \quad (5.4)$$

where $\frac{B}{Y_c}$ is the shunt susceptance associated with the discontinuity created by the ridge and approximated as shown in equation (5.5) [116] using quasistatic conformal mapping

$$\frac{B}{Y_c} = \frac{f_c}{c} (w + 2w_g) \left[ 1 - \ln (4u) + \frac{u^2}{3} + \frac{(1 - u^2)^4}{2} \left( \frac{f_c(w + 2w_g)}{2c} \right)^2 \right], \quad (5.5)$$
and

\[ u = \frac{2w_g}{w + 2w_g}. \]  \hfill (5.6)

Assume a TE_{10} wave propagating in the z-direction at an operating frequency 34 to 36.5 GHz, and with the base parameters: inner conductor thickness, \( t = 27 \ \mu m, \) \( t_g = 24 \ \mu m, \) \( w_g = 52 \ \mu m, \) \( w = 50 \ \mu m, \) \( \epsilon = \epsilon_0 (\epsilon_r = 1), \) and \( c = 3 \times 10^8 \ m/s. \) Numerically, \( C_{\text{perlength}} \) was approximated to be 70 pF/m yielding a characteristic impedance, \( Z_0, \) of 48-50 Ω. These initial parameters were used to model the base structure in the Ansys high frequency structure simulator, HFSS. Ansys HFSS is a 3-D electromagnetic solver that is based on a finite element numerical technique, it will be used throughout the modeling of the structures. The finite element numerical method (FEM) has its origin in the structural analysis, and was applied to electromagnetic problems in 1968 for the first time. Although other numerical methods such as Finite difference method (FDM) and method of moments (MOM) are easier to program than FEM, FEM is more powerful for handling complex structures and inhomogeneous media. Finite element analysis of any problem is divided into four main steps:

1. Discretize the solution region into a finite number of elements
2. Derive the governing equations for a typical element
3. Assemble all elements in the solution region
4. Solve the system of equations obtained
For the sake of this modeling and investigation, a high frequency structure simulator (HFSS) is used as a 3-D electromagnetic solver. To generate an electromagnetic field solution for a high frequency structure, HFSS employs the finite element method. FEM divides the full problem space into thousands of smaller regions and represents the field in each sub-region with a local function. In HFSS, the geometric model is automatically divided into a large number of tetrahedra, where a single tetrahedron is a four-sided pyramid. This collection of tetrahedra is referred to as the finite element mesh. The value of the H-field or E-field at points inside each tetrahedron is interpolated from the vertices of the tetrahedron. At each vertex, HFSS stores the components of the field that are tangential to the three edges of the tetrahedron. Also, it can store the component of the vector field at the midpoint of selected edges that is tangential to a face and normal to the edge. The field inside each tetrahedron is interpolated from these nodal values as shown in Figure 5.3 [117]. By representing field quantities in this way, the system can transform Maxwell's equations into matrix equations that are solved using traditional numerical methods.

There is a trade-off among the mesh size, level of accuracy, and available computing resources. The accuracy of the solution depends on the size of each of the individual elements. Typically, solutions based on meshes using thousands of elements are more accurate than solutions based on coarse meshes using relatively few elements. To generate a precise description of a field quantity, each element must occupy a region that is small enough for the field to be adequately interpolated from the nodal values [117]. The 3-D structure in Figure 5.2 was drawn, modeled, and analyzed using HFSS. The S-parameter results obtained for this figure are presented
in Figure 5.4 by sweeping the height, \( t \) of the inner conductor between 0.056 mm and 0.06 mm. In order to achieve optimized parameters, an HFSS optimetrics analysis tool was invoked on the base structure. The following optimized parameters were obtained as a result of this action: \( t = 28 \mu m \), \( t_g = 23 \mu m \), \( w_g = 47.5 \mu m \), and \( w = 55 \mu m \), for a waveguide length of 10 mm. In Figure 5.5, different shapes of rectangular coaxial transmission lines are modelled. The S-parameter performances for these shapes are presented in Figure 5.6 proving that any of these structures can be adapted as a \( \mu \)-RCP.

### 5.2 Micro rectangular coaxial phase shifter

Despite the development in Micro-electromechanical systems (MEMS) and modern ferrite technologies, to the best of the author’s knowledge, no ferrite inte-
Figure 5.4: S-parameter performances of air-filled rectangular coaxial waveguide by varying the inner conductor height

gration has been achieved using manufacturing processes that can be easily adapted into commercial micro-fabrication techniques. In this work the effort is focused on numerical modeling of ferrite integrated micro-coaxial phase shifter. Consider the diagram in Figure 5.7, [21,43,118,119].
Figure 5.5: Possible shapes and combination of rectangular coaxial waveguides

Figure 5.6: S-parameter performances of air-filled rectangular coaxial waveguide for different shapes of 0, 45, 90 and 180 degree bends.
In the ferrite slab, Maxwell’s equations can be written as

\[ \nabla \times \mathbf{E} = -j\omega [\mu] \mathbf{H}, \]  
\[ (5.7) \]

\[ \nabla \times \mathbf{H} = j\omega \varepsilon \mathbf{E}, \]  
\[ (5.8) \]

\[ \nabla \cdot \mathbf{D} = 0, \]  
\[ (5.9) \]

\[ \nabla \cdot \mathbf{B} = 0, \]  
\[ (5.10) \]

where \([\mu]\) is the permeability tensor for \(\hat{y}\) bias as given in (2.37). Given the waveguide with the two symmetrical ferrite slabs as shown in Figure 5.7. However, if we assume that \(\mathbf{E}(x, y, z) = [e(x, y) + \hat{z}e_z(x, y)] e^{-j\beta z}\) and \(\mathbf{H}(x, y, z) = [h(x, y) + \hat{z}h_z(x, y)] e^{-j\beta z}\), for \(\text{TE}_{m0}\) modes, then the solutions for \(e_y\) in the air-ferrite-air regions of the waveguide...
are [43]

\[
e_y = \begin{cases} 
  A_1 \sin k_a x & 0 < x < c \\
  A_2 \sin k_f (x - c) + A_3 \sin k_f (c + t - x) & c < x < c + t \\
  A_4 \cos k_a (a/2 - x) & c + t < x < a/2 
\end{cases} 
\] (5.11)

to facilitate enforcement of boundary conditions at \( x = 0, c, c + t \) and at \( a \). However, \( h_z \) can equally be evaluated as

\[
h_z = \begin{cases} 
  (j k_a A_1 / \omega \mu_0) \cos k_a x & 0 < x < c \\
  -\kappa \beta (j / \omega \mu_e) [A_2 \sin k_f (x - c) + A_3 \sin k_f (c + t - x)] \\
  +(j / \omega \mu_e) \mu k_f [A_2 \cos k_f (x - c) - A_3 \cos k_f (c + t - x)] & c < x < c + t \\
  (j k_a A_4 / \omega \mu_0) \sin k_a (a/2 - x) & c + t < x < a/2 
\end{cases} 
\] (5.12)

where \( k_f^2 = \omega^2 \mu_e \epsilon - \beta^2 \), \( k_a^2 = k_0^2 - \beta^2 \), and \( A_1, A_2, A_3, A_4 \) are constants. In order to obtain these constants in these four equations, \( e_y \) and \( h_z \) must be mode matched at
the boundaries at \( x = c, x = c + t = (a/2) - d \) to give

\[ A_1 \sin k_a c = A_3 \sin k_f t, \quad (5.13) \]

\[ A_2 \sin k_f t = A_4 \cos k_a d, \quad (5.14) \]

\[ A_1 \frac{k_a}{\mu_0} \cos k_a c = A_2 \frac{k_f}{\mu_e} - A_3 \frac{1}{\mu_0 \mu_e} (-\kappa \beta \sin k_f t + \mu k_f \cos k_f t), \quad (5.15) \]

\[ A_2 \frac{k_a}{\mu_0 \mu_e} (\kappa \beta \sin k_f t + \mu k_f \cos k_f t) - A_3 \frac{k_f}{\mu_e} = A_4 \frac{k_a}{\mu_0} \sin k_a d. \quad (5.16) \]

Finally solving equation (5.13) and equation (5.14) for \( A_3 \) and \( A_4 \), and substituting \( A_3 \) and \( A_4 \) into equation (5.15) and equation (5.16), and then eliminating either of \( A_1 \) or \( A_2 \), we produce an equation called a transcendental equation containing trigonometric functions as

\[ \left( \frac{k_f}{\mu_e} \right)^2 + \left( \frac{\kappa \beta}{\mu_0 \mu_e} \right)^2 - k_a \cot k_a c \left( \frac{k_f}{\mu_0 \mu_e} \cot k_f t + \frac{\kappa \beta}{\mu_0 \mu_e} \right) + \left( \frac{k_a}{\mu_0} \right)^2 \cot k_a c \tan k_a d + k_a \tan k_a d \left( \frac{k_f}{\mu_0 \mu_e} \cot k_f t - \frac{\kappa \beta}{\mu_0 \mu_e} \right) = 0. \quad (5.17) \]

Close examination of the resulting equation (5.17) shows that the only unknown is the propagation constant, \( \beta \) and this needs a numerical method to solve completely. All other geometrical parameters and properties of ferrite material are known from characterization measurements in Chapter 3 and Chapter 4.
5.2.1 Ferrite composite and $\mu$-RCP modeling

At this stage, after designing the rectangular coaxial air-filled transmission line, the ferrite material is then partially introduced into the structure illustrated in Figure 5.8. The resulting structure is then modeled in HFSS version 13, with the necessary static bias field applied transversely to the direction of electromagnetic field propagation as shown in Figure 5.9. For the purpose of this investigation, a composite of ferrite-polymer material is modeled as being patterned on the floor of the waveguide. The composite consists of strontium hexaferrite, $\text{SrFe}_{12}\text{O}_{19}$ powder and SU8 negative photoresist as a binder.

Figure 5.8: Modelling of a micro-rectangular coaxial ferrite phase shifter
The ferrimagnetic material has a crystalline hexagonal shape, M-type strontium ferrite powder with an excellent performance and reasonable loss within the frequency range of interest. The powder and the composite material were prepared and characterized as discussed in Chapters 3, 4 and 6. The measured data extracted from hysteresis loops of ferrite-SU8 composites presented in Figure 4.10 were used in the structure modelling. Approximately, 0.6 Tesla saturation magnetization, and a fairly low coercivity range, 0.6 kOe - 2 kOe were read from the plot for the ratio, 3:1 of 3-6 \( \mu m \) ferrite/SU8 composite size. These parameters fall within values for applications in a digital (or remanence) ferrite phase shifter, thus satisfying a required performance of order 5,000 Gauss for a common ferrite phase shifter intended to operate at Ka-band [1]. Because the direction of wave propagation is along the z-axis, the bias static magnetic field should be applied transversely in y-direction (-Y-direction) as shown in Figure 5.9. Also a dielectric constant value of 5 was used for the ferrite composite in the simulation. This value was taken from experimental TRL measurements recorded in Chapter 4.
Figure 5.9: Ferrite Y-directed static magnetic field applied to $\mu$-RCP

The model was setup in HFSS with the saturated magnetization, $M_s = 1800$-$5000$ Gauss, dielectric constant, $\varepsilon_r = 5$, variable static applied field, $H_0 = 0$-$400$ kA/m, linewidth, $\Delta H = 62$ Oe, and lande G factor of 1.98. A 3-D finite element numerical analysis solver invocation with adaptive mesh refinement was used to solve the electromagnetic fields in the structure. A solution frequency of 36.5 GHz was set for the simulation and 30% maximum refinement per pass. The ports only simulation was first invoked to determine the correctness of the excitation at the ports. This excitation performance is presented in Figure 5.10, and it shows that the E-field at the input wave port is correctly aligned with x-axis and transverse to the direction of propagation. After proper sizing of the wave port, a 0-40 GHz frequency sweep was setup with a first order iterative solver. The relative residual of this solver was set
at 0.0001. Using the default initial mesh and the subsequent adaptive refinements by the solver, the last adaptive mesh plot is presented in Figure 5.11 while the solution convergence plot is shown in Figure 5.12. The convergence plot indicates that the solution converged to the maximum magnitude Delta S of 0.02 after about 5 adaptive passes. Figure 5.13 shows the electric field vectors rotation inside the phase shifter due to the circular polarization initiated by the ferrite material.

**Figure 5.10**: Port sizing for micro-rectangular coaxial ferrite phase shifter

**Figure 5.11**: Meshing of the $\mu$-RCP 3-D structure with sacrificial holes
Figure 5.12: Convergence plot extracted from the solution data

Figure 5.13: Perspective of electric vectors from input port to output port
The structure in Figure 5.9 with the model size: \( t = 28 \, \mu m, t_g = 23 \, \mu m, \)
\( w_g = 47.5 \, \mu m, w = 55 \, \mu m, \) and ferrite length, \( L_f = 10 \, mm \) produced the phase shift of Figure 5.14 [120]. Table 5.1 shows the saturated magnetization values and the corresponding phase shifts as extracted from Figure 5.14 when the applied field values 0-400 kA/m were swept at 36.5 GHz. Also shown in this figure are the phase shift plots of air-filled rectangular coaxial waveguide and equivalent alumina-filled \( \mu \)-RCP with the same dielectric constant of 5. The phase shifts of the air-filled and alumina-filled structure are respectively 47 and 64 degrees at the same frequency. From this result, the effect of the ferrite composite material is noticed across the applied field as shown by the progressive variations of the phase shift with respect to the nominal phase at 0 A/m which is contrary to the effects of air-filled micro-rectangular coaxial waveguide and alumina-filled \( \mu \)-RCP. The nominal phases of the air-filled and alumina-filled structures are constant across the entire applied field range.

One of the performance indicators of a phase shifter is the S-parameter performance which is depicted in Figure 5.15. In this figure at 36.5 GHz, return loss is better than 20 dB while the insertion loss is less than 1 dB for every saturation magnetization, \( M_s \), value used. The non-reciprocity of the \( \mu \)-RCP can be visualized by obtaining a differential phase between the phases of insertion losses (S21, S12) of a two port micro-RCP as shown in Figure 5.16. In this figure, the maximum phase difference between the phases of the insertion loss measured at port 2 when the input is at port 1 and insertion loss measured at port 1 when the input is at port 2 is less than 2 degrees. From the performance of this model size (Model A), there is a possi-
Table 5.1: Magnetization and corresponding Phase shift for model A $\mu$-RCP

<table>
<thead>
<tr>
<th>Saturated magnetization (Gauss)</th>
<th>Phase shift (degree per centimeter) at 36.5 GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>1800</td>
<td>54</td>
</tr>
<tr>
<td>2800</td>
<td>56</td>
</tr>
<tr>
<td>3800</td>
<td>76</td>
</tr>
<tr>
<td>5000</td>
<td>57</td>
</tr>
</tbody>
</table>

bility of achieving a good reciprocal phase shifting at this frequency. However, for a non-reciprocal performance, a further investigation on the model parameters will be required.

Figure 5.14: Phase shift of a micro-rectangular coaxial ferrite phase shifter, $\mu$-RCP (Model A)
Figure 5.15: S-parameter performances of \( \mu \)-RCP (Model A)

Figure 5.16: Differential phase, \(|\arg(S_{21}) - \arg(S_{12})|\) of a micro-rectangular coaxial ferrite phase shifter, \( \mu \)-RCP (Model A)
In order to achieve non-reciprocal performance, practical realization and ease of fabrication of $\mu$-RCP, the model size was scaled by a factor of 10. The new model size (Model B) now reads: $t = 280 \ \mu m$, $t_g = 230 \ \mu m$, $w_g = 475 \ \mu m$, $w = 550 \ \mu m$, with ferrite length, $L_f = 10 \ mm$ and height, $h_f = 230 \ \mu m$. The simulation was re-run based on the same ferrite data and frequency of operation. The performance plots of the scaled model of $\mu$-RCP are presented in Figure 5.17, Figure 5.18, and Figure 5.19. Table 5.2 shows the saturated magnetization values and the corresponding phase shifts as extracted from Figure 5.17 when the applied field values 0-400 kA/m were swept at 36.5 GHz.

![Figure 5.17: Phase shift of a micro-rectangular coaxial ferrite phase shifter, $\mu$-RCP (Model B)]](image)

Table 5.2 shows the saturated magnetization values and the corresponding phase shifts as extracted from Figure 5.17 when the applied field values 0-400 kA/m were swept at 36.5 GHz.
Figure 5.18: S-parameter performances of $\mu$-RCP (Model B)

Figure 5.19: Differential phase, ($|\arg(S_{21}) - \arg(S_{12})|$) of a micro-rectangular coaxial ferrite phase shifter, $\mu$-RCP (Model B)
In Figure 5.18, the length of the ferrite and the coaxial waveguide were simultaneously varied to establish the impact. In this plot, the model with 20 mm ferrite length produced the highest phase shift across the applied field, 0-400 kA/m. This highest phase shift of 88°/cm was recorded from the nominal phase of 44°. This result indicates that by varying the ferrite length, the phase shift produced is altered from the nominal. Non-reciprocal performance was described by the plot in Figure 5.19. There is a clear non-reciprocity between the two ports of the proposed device for this model scale at 36.5 GHz operating frequency. From the figure, across the applied field, the differential phase between S$_{21}$ and S$_{12}$ increases from 6° to about 22° for different magnetization values from 1800 G to 5000 G. This result emphasizes the possibility of using this device to achieve a non-reciprocal micro-rectangular ferrite phase shifter.

### 5.2.2 Validation of $\mu$-RCP with the previous published work

In order to validate the simulated results using ferrite-SU8 composites characterized in this investigation, previous published work by some scholars are presented in
Table 5.3. The models designed in this investigation are compared with the different phase shifters at Ka-band frequency range. The relevance and promising performance of $\mu$-RCP is established by comparing the figure of merit, return, and insertion losses of the simulated models to already published work of the scholars. From the table, the simulated performance of the $\mu$-RCPs is promising and favorably compared to already published research investigations.

**Table 5.3: Comparison of $\mu$-RCP with previous published work**

<table>
<thead>
<tr>
<th>Reference</th>
<th>$S_{11}$ [dB]</th>
<th>$S_{21}$ [dB]</th>
<th>Figure of merit [deg./dB]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Erker, et al, [121]</td>
<td>12</td>
<td>5.8</td>
<td>27.1 @ 30 GHz</td>
</tr>
<tr>
<td>Hacker, et al, [122]</td>
<td>15</td>
<td>2.2</td>
<td>20 (45° phase state) @ 35 GHz</td>
</tr>
<tr>
<td>Hayden, et al, [123]</td>
<td>11</td>
<td>1.5</td>
<td>59 (89° phase shift) @ 37 GHz</td>
</tr>
<tr>
<td>Malmqvist, et al, [124]</td>
<td>...</td>
<td>1.5</td>
<td>64 (45° phase state) @ 30 GHz</td>
</tr>
<tr>
<td>Pillans, et al, [125]</td>
<td>15</td>
<td>2.2</td>
<td>20 (45° phase state) @ 35 GHz</td>
</tr>
<tr>
<td>Siegel, et al, [126]</td>
<td>15</td>
<td>2.2</td>
<td>13.25 (measured) @ 34 GHz</td>
</tr>
<tr>
<td>Wang, et al, [127]</td>
<td>12</td>
<td>2.35</td>
<td>19 (45° phase state) @ 35 GHz</td>
</tr>
<tr>
<td>$\mu$-RCP (Reciprocal)</td>
<td>20</td>
<td>1.5</td>
<td>36 (54° phase shift) @ 36 GHz</td>
</tr>
<tr>
<td>$\mu$-RCP (Non-reciprocal)</td>
<td>20</td>
<td>0.4-0.8</td>
<td>117 (47° phase shift) @ 34.5 GHz</td>
</tr>
</tbody>
</table>
CHAPTER 6

FERRITE-SU8 MAGNETIC COMPOSITE AND
PHOTOLITHOGRAPHY

6.1 Processability of the magnetic composite

Having designed and simulated the 3-D \( \mu \)-RCPs, then the ferrite-SU8 composites are patterned as micro-magnets on wafers. Multilayer microfabrication procedures are required for ultimate patterning, \( \mu \)-RCP, on wafer using PolystartaTM process [128]. The magnetic polymer composite is composed of barium ferrite particles of chemical composition BaFe\(_{12}\)O\(_{19}\) with an average particle size 0.8-1.0 \( \mu \)m and SU8-2000 negative photoresist (MicroChem Corp. USA). SU8 2000 has excellent imaging characteristics and is capable of producing very high aspect ratio structures. The polymer, SU8 2000 is ideally suited for imaging near vertical sidewalls in very thick films as a result of its very high optical transmission above 360 nm [94]. Before mixing various quantities of the ferrite particles into the SU8 matrix, the photoresist is treated by addition of ethyl acetate and 1-cyano-ethyl-2-ethyl-4-methylimidazole (2E4MZ-CN) [129–131] ordered from Fisher Scientific Company and Wako Chemicals. These additional solvents of ethyl acetate and 2E4MZ-CN are used for viscosity control and faster chemical binding respectively. Mixing was performed in the micro-
fabrication lab, using a nonmagnetic stirrer shown in Figure 6.1. The mixture was left for 24 hours in order to ensure homogeneity of the composite solution.

Figure 6.1: Ferrite SU8 preparation and magnetic stirrer

Samples of the different ratios of the composite were made and spin-coat on quartz glass and then soft baked on hot a plate for three minutes before being tested for transmittance using ultraviolet (UV)-visible spectrometer. The transmittance results are plotted in Figure 6.3. These results indicate for a particular reference wavelength, from 365 nm to 500 nm, the transmittance increases as the concentration of the ferrite powder decreases in the composites. Also the figure shows that the SU8 coated glass has a transmittance closest to that of blank quartz glass, and this is due to the transparency of the polymer. Their transmittances are 63 and 86 % for both SU8 coated glass and the blank quartz glass respectively. Composite-90 and composite-100 have, 0.5 and 0.4 % transmittance respectively, which are relatively close to zero. Thus a wavelength 365 nm was chosen as the UV light wavelength used for all the
fabrication exposure. Figure 6.3 shows the test samples and a fabricated test device. This experiment simply proved the possibility of using ferrite/SU8 polymer matrix in fabricating planar magnetic devices.

**Figure 6.2**: Percentage transmittance of strontium ferrite powder concentrations in SU8 on quartz glass.
Figure 6.3: Test samples for transmittance and fabricated device using strontium ferrite/SU8 polymer

Ferrite/polymer composite was equally deposited on a 4-in silicon wafer substrate, and then spin-cast on a model 5100 Solitec wafer spinner. The deposition was soft baked using Ogden ETR-9090 hotplate at 95°C for three minutes. The spread and spin parameters of the spinner are 500 rpm (for 60 s) and 3000 rpm (for 60 s) respectively. Before patterning, dark and light field masks of ferrite-SU8 composite micro-magnets were designed at UAH and produced by Front range photomask LLC under a contract agreement. The masks are in Figure 6.4 and Figure 6.5.
Figure 6.4: Light field mask for ferrite-SU8 composite micro-magnets

Figure 6.5: Dark field mask for ferrite-SU8 composite micro-magnets
These masks were designed and then drawn using the electromagnetic simulation software, (CST STUDIO). Each of the square toroids was drawn with the size, 4 x 4 mm and inner diameter, 0.6-1.5 mm. The circular toroids have both the inner and outer diameter, 4-10 µm and 16 µm respectively. Also, the bar composite magnets designed to have the sizes, 2 x 10 mm (32 magnets) and 4x3 mm (1 bar composite magnet). Finally, the horse shoe shaped magnets were assigned the sizes 8 mm (13 pcs) and 4 x 3 mm (2 bar magnets). Ultra violet light exposure of the ferrite/polymer was achieved using the OAI model J500/vs mask aligner, by first exposing the baked sample to ultra-violet light of 365 nm wavelength with a dose of 50 mJ/cm². After ultra-violet light exposure, the sample was then post baked for five minutes at 95⁰ C before being developed in SU8 developer with ultrasonic agitation, as recommended by [132,133] for 5 minutes. Following the development, the substrate and the patterns were rinsed with isopropyl alcohol (IPA), then dried with a gentle stream of nitrogen.

Other fabricated devices from plain SU8 photoresist and ferrite-SU8 composites are shown in Figure 6.6 and Figure 6.7 respectively. In Figure 6.6, SU8 was spin cast on a 4-in silicon wafer and then fabricated according to the previous procedure but with light field mask. The structures in Figure 6.7 are fabricated with some other dark field masks.
Figure 6.6: fabricated structure with SU8

Figure 6.7: Fabricated devices using ferrite-SU8 composites
A SEM image of one of the patterned ferrite composite magnets of Figure 6.7 is shown in Figure 6.8. The bottom image of Figure 6.8 shows the cross section of the top image. From the cross section, the particles of the ferrite powder are clearly shown as being encapsulated by the SU8. The ferrite powder was evenly distributed in the composite, making it easier to pattern. Thus, fabrication of the proposed phase shifters, $\mu$-RCPs, can be effectively realized due to homogeneity of the particles in the ferrite-SU8 mixture. The average height of the cross section of one of the patterned magnetic structure was measured to be 15 $\mu$m.

**Figure 6.8:** Patterned ferrite composite and its cross-sectional view
6.2 Scanning Electron Microscopy

The Scanning Electron Microscope (SEM) is used to image the topography and composition of a sample surface using a high-energy electron beam. The system operates by focusing the electron beam onto the sample in a sequence of horizontal strips. The electron beam interacts with the atoms on the sample surface, and the signals from these interactions are interpreted by the interface computer system as information about the properties of the sample. There are four types of signals that are recorded from SEM scans, and they include backscattered electrons, secondary electrons, x-rays, and cathodoluminescence (visible light). The SEM images of the powder and cured ferrite composite were taken using LEO model 1550 scanning electron microscope. The measurement started by venting the vacuum chamber of the equipment and inserting the ferrite sample in any convenient sample holder which snapped to the stage of the chamber. After the sample insertion, the chamber is closed and then vacuum to $1.55 \times 10^{-5}$ Torr pressure. Also the EHT value was set to 2.0 kV while the working distance, and magnification are equally set at convenient values. The sample holder and the chamber scope of the equipment employed in this investigation are shown in Figure 6.9
Figure 6.9: Vacuum chamber and stage assembly views: (a) Vacuum chamber showing the scope, sample holder and ferrite sample during measurement, (b) Bottom view of the stage assembly, and (c) Top view of the stage assembly showing the sample holders magnetised to it.
CHAPTER 7

CONCLUSIONS AND FUTURE RESEARCH

This work introduced a novel Micro-rectangular coaxial phase shifter, $\mu$-RCP, design at Ka-band using ferrite-SU8 composite as an anisotropic material. This new design will greatly promote the miniaturization and resolution of the passive components at this frequency. The $\mu$-RCP, has promising return and insertion losses, making it a viable candidate technology for application in the front end of microwave devices. The design was made possible by a comprehensive characterization of the ferrimagnetic materials and the organic polymer described in Chapter 2, Chapter 3 and Chapter 4 of this work.

7.1 Summary

**Barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) powder and SU8 polymer:** Micron-sized barium hexaferrite powder materials have been suspended in SU8 2000 negative photoresist. The electromagnetic characteristics of these composites have been investigated to determine their magnetic and RF properties using a RF waveguide technique, VSM and energy dispersive x-ray analysis tool. This work provides a method to cast and pattern ferrite-polymer composites for use in microwave applications be-
tween 26.5 GHz and 60 GHz. Unlike conventional ferrite blocks, which are bulky and incompatible with current micro-fabrication processes, the new composite material approach can be directly integrated into photolithographic and laser patterned printed circuits. The composite still retained characteristics commonly expected of ferrite material needed for the anisotropic microwave transmission through $\mu$-RCP. The primary effect on the electromagnetic properties of the barium hexaferrite is the resonance frequency shift due to the c-axis magneto-crystalline anisotropy field effect.

The hysteresis loop results in Figure 3.6 and Figure 3.7 also provide sufficient evidence that 5,000 Gauss magnetization requirement [1] for a typical Ka-band ferrite phase shifter can be achieved using a ratio 1:1 barium ferrite composite for instance. The effective permittivity range of 3.5-7.7, and permeability range of 1.0-1.8 as a function of frequency extracted from measured data and presented in Table 3.1 provide the required diagonal and off-diagonal components of the permeability tensor for microscaled reciprocal and nonreciprocal phase shifter applications. In summary, the composite is recommended in micro and nano-fabrication of high frequency devices such as on-chip magnetic components for millimeter wave integrated circuits, circulators, isolators, and phase shifters where current machined and polished monolithic industrial ferrite blocks are too large to be incorporated into the fabrication procedure.

**Strontium ferrite (SrFe$_{12}$O$_{19}$) powder and SU8 polymer:** Similarly to the barium ferrite, (BaFe$_{12}$O$_{19}$) powder, micron-sized strontium ferrite powders were mixed with SU8-2000 negative photoresist and the electromagnetic and ferromagnetic characteristics of the samples were investigated using the Energy Dispersive
X-ray spectrometry, EDAX, Vibrating Sample Magnetometer, VSM, Ferromagnetic resonance, FMR, and waveguide measurement methods. Ferromagnetic resonance was centered around 60 GHz where absorption of the signal was noticed for both strontium ferrite powder and its composites with SU8 photoresist. The shift in field strength resulting from the different percentage ferrite concentration within the composite has made it possible to suggest how Strontium ferrite powder could be blended with SU8 for a spintronic devices. Similarly to barium ferrite measurements, the only noticeable effect in the composites is the progressive shift in the natural resonance of the composites towards the higher frequency zone as the concentration of the powder increases. Therefore strontium ferrite/SU8 composites are also recommended for micro and nano-fabrication of microwave devices such as on-chip magnetic components for millimeter wave integrated circuits, circulators, isolators, and phase shifters.

Micro-rectangular coaxial phase shifter, ($\mu$-RCP):

A detailed characterization of ferrimagnetic powders and composites with some additives has been presented. The results obtained from the experiments and $\mu$-RCP simulations show that both reciprocal and non-reciprocal phase shifting are possible and realizable. This technology can be implemented on planar substrate by employing sequential lithographic and plating processes. The Proposed strontium/barium ferrite composite will provide the non-reciprocity and necessary mechanical support for the inner conductor of the $\mu$-RCP simultaneously. The two model structures designed for reciprocal and non-reciprocal applications presented in Chapter 5 demonstrate tunable phase shifts of almost $60^\circ$/cm across 0 to 400 kA/m applied field and at 1800 Gauss for Model A and a non-reciprocal phase shift performance of $20^\circ$/cm for model
B at the same saturation magnetization and 36.5 GHz operating frequency. Also, promising performance indicators were obtained, a return loss better than 20 dB, and an insertion loss less than 1 dB for the two model structures. Hence, because of the enumerated performances, the structure, \( \mu \)-RCP is therefore recommended as a ferrite phase shifter that can be used for microwave applications at Ka-band.

7.2 Future work

The new technique introduced in this dissertation work can be extended to the design of a variety of ferrite passive components and devices such as micro-rectangular coaxial circulators, \( \mu \)-RCCs and micro-rectangular coaxial isolators, \( \mu \)-RCIs which can be used in the front-end of a wireless communication systems alongside \( \mu \)-RCPs. Improved performance with respect to isolation, peak and average powers are expected due to the coaxial arrangement of the conductors.

Another project may be the replacement of off-the-shelf ferrimagnetic material with an in-house synthesis ferrite materials. This will allow for proper control of the characteristics of the materials as to reduce losses and improve anisotropic performances. In the near future, this technique may be presented to Nuvotronics LLC for possible development, scalability and commercialization under DARPA 3-D MERFS program (DARPA - Defense Advanced Research Projects Agency, 3-D MERFS 3-Dimensional Micro-Electromagnetic Radio Frequency Systems). DARPA is an agency of the United States Department of Defense responsible for the development of new technologies for use by the military.
REFERENCES


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