THE RELIABILITY OF SPLIT RING RESONATOR BASED METAMATERIALS IN PLASMA ENVIRONMENT

A Thesis in
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by

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ABSTRACT

Metamaterials are the artificial materials with a focus on structure more than composition, and they have customized properties that are not found in nature, such as negative refractive index. Negative refractive index is achieved through negative values of the permittivity and permeability in a certain range of frequency. Currently, the metamaterials work well in the range of microwave. In this range, the negative permeability can be readily achieved by using a structure of split-ring resonator (SRR). At the same time, plasma at the gap of SRR is a novel method to provide the negative permittivity.

It is straightforward to fabricate SRRs in the laboratory by conventional processes. In this work, silver ring patterns were painted onto the alumina followed by firing at 850°C in a belt furnace. The geometry of the SRR was designed by the analytical equations and corroborated by the simulation software from CST Corporation. Plasmas in the ring gap were created by exposing the SRR to high power (300W) at the frequency of 2.45GHz in air atmosphere. However, the plasma only lasted for five minutes with serious silver erosion at the gap. Therefore, a protective dielectric layer is proposed in this thesis for SRR working in a harsh plasma environment.

The development path begins with evaluating the reliability of the dielectric substrate in a plasma environment. Several oxide substrates were investigated including alumina, silica and a commercial low temperature co-fired ceramic (LTCC). The effect of plasma treatment on surface morphology and chemistry was characterized by optical profilometry, energy dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM). A network analyzer with a split-cavity resonator was used to monitor the microwave dielectric properties before and after plasma treatment. It has found that small amounts of carbon residues will reduce the Q-factor. Commercial 96%Al₂O₃ is proven to be a suitable substrate for the SRR.
Since most serious erosion occurs at the gap of the silver section in SRR, a protective dielectric layer was investigated to prevent degradation of the resonator performance. Several approaches were tried to protect the silver ring resonator including thin coatings made through thick film deposition. All of the SRR structures were tested in a high power microwave chamber which had a 1kW power supply at 2.45GHz. All of the tests were carried out in air and it was found that a plasma could not be ignited for thick dielectric protective coatings. To support the experimental result, simulations by CST software were carried out and the results show that the electric field intensity is substantially lower for thick dielectric coatings. These results suggest that substantially higher microwave power are needed to generate sufficient electric fields to ignite and sustain a plasma as the dielectric layer thickness increases. Comparisons between the uniform dielectric coating and a protective layer with a gap show that the partially protected SRR should work much better. Experimental results show that the SRR sample with partial protection works in a microwave environment for at least 5 hours with no evidence of failure.
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LIST OF SYMBOLS

EM Electromagnetic

rf Radio Frequency (/s)

SRR Split Ring Resonator

\( \varepsilon_0 \) Permittivity in Vacuum (F/m)

\( \varepsilon_r \) Relative Permittivity

\( \mu_0 \) Permeability in Vacuum (H/m)

\( \mu_r \) Relative Permeability

\( n_{ph} \) Phase Velocity of Wave (m/s)

\( c \) Velocity of Light (m/s)

\( \omega_p \) Frequency of Plasma (/s)

\( \omega_0 \) Resonant Frequency of SRR in Lorenzian Model (/s)

\( \vec{k} \) Wave Vector

\( \vec{S} \) Poynting Vector

\( \vec{D} \) Displacement Field (C/m²)

\( \vec{B} \) Magnetic Field (A/m)

\( \vec{E} \) Electric Field (V/m)

\( \vec{P} \) Momentum of Electron (kg·m/s)

\( \tau \) Mean Free Time between the Collision (s)

\( \vec{p} \) Polarization (C/m²)

\( \Phi \) Magnetic Flux (Wb)

\( \chi_e \) Electric Susceptibility

\( L \) Inductance in Lorenzian Model (H)

\( R \) Resistance in Lorenzian Model (Ω)
$I$ Electric Current in Lorenzian Model (A)

$M$ Magnetization Density (A/m)

$D$ Diameter of the SRR (mm)

$W$ Width of SRR strip (mm)

$G$ Width of Gap of SRR (mm)

$t$ Thickness of Metal Ring (mm)

$h$ Height of Substrate (mm)

$f_{RN}$ Resonant Frequency of SRR in Design (/s)

$\varepsilon_{eff}$ Effective Relative Permittivity of SRR

$Q$ Quality Factor
This thesis involved bringing together the information from a lot of different studies and would have been impossible if not for the technical and emotional support of many people.

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Chapter 1

Introduction

1.1 Introduction

Metamaterials are composite structures comprised of dielectrics and metals. Often metamaterials are constructed from conductors that are periodically arranged on the dielectric substrate at a subwavelength spacing, in which electromagnetic (EM) wave interacts with matter in a way that is not found in nature[1]. Because of the periodicity in the subwavelength scale, metamaterials could often be regarded as a homogeneous medium with effective constitutive properties of permittivity and permeability[2].

In 1968, a new perspective in materials science came into expectation due to the Veselago’s unprecedented hypothesis that electromagnetic waves potentially are able to transmit in unconventional ways, which now we know is negative refraction[3]. The metamaterial medium has negative permittivity and negative permeability simultaneously. However, it was not until 1990s that negative refractive phenomenon was proven experimentally[4]. Since then, metamaterials as a new branch attracted more and more attention of material scientists. The research in this field developed rapidly with a large number of new applications being brought forth[5-9].

This thesis focuses on split ring resonator structures that are fundamental elements of metamaterial structures, as shown in Figure 1-1[10]. These types of structures produce unconventional electromagnetic propagation characteristics as the negative refractive wave which produces unconventional optical behavior (Figure 1-2). Another noteworthy characteristic in the
negative refraction is that the wave vector $k$ is in the opposite direction of the poynting vector $\vec{S}$.

Based on this phenomenon, several application have been developed among which the most famous are ‘cloaking materials’ and ‘perfect lenses’[4].

Negative index of refraction is the result of both negative permeability and permittivity. Negative permeability was realized through a resonant ring resonator which generates an rf current and hence magnetic field. Negative permittivity can be created through EM interaction with a wire rod which creates a resonant dipole[2,11]. Plasmas have been proven to be a more suitable negative permittivity material because the permittivity can be adjusted by varying the density of the plasma. In this manner, metamaterials can be fabricated conveniently by constructing an array of SRR, which will have both negative permittivity and negative permeability components. Figure 1-1 portray the metamaterials based on the SRR constituents.

The dimensions of the metamaterials constituents are in the range of the electromagnetic wave length and they can be quite small for optical metamaterials (380-750nm). This gives rise to the challenge in synthesizing the structure precisely, even by means of the nanofabrication technique. Therefore, a majority of metamaterials that are realized so far have been in the microwave frequency range, corresponding to a geometric size of the periodic unit between 0.1cm and 100cm, which is easy to achieve under ordinary fabrication conditions.

Plasma, although being proven to be an excellent medium for contributing to negative permittivity, has the potential to degrade the substrate and metal due to the ion bombardment of surfaces and high temperatures. The purpose of this thesis is to understand the degradation of the negative refraction metamaterials in a harsh plasma environment. For this purpose, a detailed explanation is necessary in the fundamentals of negative refraction.
Figure 1-1. The metamaterial with negative refractive index based on the split ring resonators (SRR). (a) conventional structure with metal rods or wires; (b) current structure with plasma excited in the gap of the SRR[12]

Figure 1-2. The refraction of the electromagnetic wave in two kind of medium. (a) negative refraction; (b) conventional refraction
1.2 Theory of the negative refractive index

The research deals with the propagation of electromagnetic wave through a metamaterial, with no need to considerate the source of the wave. Therefore we can begin with the Maxwell equations in differential form.

\[ \nabla \cdot \vec{D} = 0 \quad (1.1) \]
\[ \nabla \cdot \vec{B} = 0 \quad (1.2) \]
\[ \nabla \times \vec{E} = -\frac{\partial \vec{B}}{\partial t} \quad (1.3) \]
\[ \nabla \times \vec{H} = \frac{\partial \vec{D}}{\partial t} \quad (1.4) \]

Where \( \vec{D} \) is displacement field, \( \vec{B} \) is magnetic field, \( \vec{E} \) is electric field and \( \vec{H} \) is magnetizing field. Also we know the relation between \( \vec{D} \) and \( \vec{E} \), and between \( \vec{B} \) and \( \vec{H} \).

\[ \vec{D} = \varepsilon_0 \varepsilon_r \vec{E} \quad (1.5) \]
\[ \vec{B} = \mu_0 \mu_r \vec{H} \quad (1.6) \]

Insert (1.6) into (1.3) and make a curl operation,

\[ \nabla \times (\nabla \times \vec{E}) = -\mu_0 \mu_r \frac{\partial}{\partial t} (\nabla \times \vec{H}) \quad (1.7) \]

Based on the fundamental formula \( \nabla^2 \vec{E} = \nabla (\nabla \cdot \vec{E}) - \nabla \times (\nabla \times \vec{E}) \), combining \( \nabla \cdot \vec{E} = 0 \) in (1.1), the following expression is obtained,

\[ \left( \nabla^2 - \varepsilon_0 \varepsilon_r \mu_0 \mu_r \frac{\partial^2}{\partial t^2} \right) \vec{E} = 0 \quad (1.8) \]

Follow the same line of the reasoning, we can obtain as well,

\[ \left( \nabla^2 - \varepsilon_0 \varepsilon_r \mu_0 \mu_r \frac{\partial^2}{\partial t^2} \right) \vec{B} = 0 \quad (1.9) \]

We know as to a wave \( \vec{A} \) the wave equation has the form of \( \frac{\partial^2}{\partial t^2} \vec{A} = v_{ph}^2 \nabla^2 \vec{A} \), where \( v_{ph} \) is phase velocity of the wave. Compare (1.8) and (1.9) with wave equation, we can conclude,
\[
\varepsilon \varepsilon_0 r \mu_0 \mu_r = \frac{1}{v_{ph}^2} \quad (1.10)
\]

It is known that \(\mu_r\) and \(\varepsilon_r\) are 1 in vacuum, so \(\varepsilon_0 \mu_0 = \frac{1}{c^2}\). Through the fundamental optical law that \(n = \frac{c}{v_{ph}}\), we can obtain the equation,

\[
n = \sqrt{\varepsilon_r \mu_r} \quad (1.11)
\]

The equation (1.11) illustrates the relation between refractive index with the two elemental parameters of the electromagnetic wave. However, the further explanation is still needed on why negative permittivity and negative permeability are required in order to achieve the negative refractive index. Since the EM wave consists of time-varying electric field and magnetic field at certain frequency, most parameters could be expressed as complex number for the sake of convenient calculation. We define a vector

\[
\hat{n} = n' + in'' \quad (1.12)
\]

Where \(n'\) is the real part representing the refractive index of the material. \(n''\) is the parameter which should be clarified. As we already know, electric field could be formulated as below,

\[
\vec{E} = E_0 e^{i(kr - \omega t)} \quad (1.13)
\]

Where \(k = \frac{2\pi}{\lambda_0} \hat{n}\). By inserting (1.12) into (1.13),

\[
\vec{E} = E_0 e^{i(-2\pi \frac{n''}{\lambda_0} r)} e^{i \left(2\pi \frac{n'}{\lambda_0} r - \omega t\right)} \quad (1.14)
\]

We can draw the conclusion that the magnitude of \(\vec{E}\) is a decaying function dependent on the imaginary part of the refractive index in the positive medium \((n' > 0)\). Therefore, if we expect a higher transmittance, imaginary part of \(\hat{n}\) must be as low as possible. On the other hand, \(\hat{\varepsilon}_r\) and \(\hat{\mu}_r\) can as well be expressed as complex number,

\[
\hat{\varepsilon}_r = |\varepsilon_r| e^{i\theta} \quad (1.15)
\]

\[
\hat{\mu}_r = |\mu_r| e^{i\phi} \quad (1.16)
\]
We can transform the equation (1.11) to the form below,

$$
\tilde{n} = \sqrt{|\tilde{\varepsilon}_r| |\tilde{\mu}_r|} e^{i\frac{1}{2}(\theta + \phi)}
$$

(1.17)

Figure 1-3 is a complex plane representation of $\tilde{n}$, $\tilde{\varepsilon}_r$, and $\tilde{\mu}_r$. We can visually draw a conclusion that negative refractive index with high transmittance only could be achieved if $\varepsilon_r$ (real part of $\tilde{\varepsilon}_r$) and $\mu_r$ (real part of $\tilde{\mu}_r$) are negative simultaneously and imaginary part of $\tilde{\varepsilon}_r$ and $\tilde{\mu}_r$ are as small as possible.

![Complex Plane Representation](image)

Figure 1-3. The schematic of the negative refractive index in complex plane

The negative permittivity can be achieved by a plasma, which is readily explained by the Drude model in Appendix A. In this model, negative permittivity occurs when the frequency of the transmitting wave is less than the frequency of plasma, shown in (1.18).

$$
\varepsilon_r = 1 - \frac{\omega_p^2}{\omega^2}
$$

(1.18)

Where $\omega_p$ is the frequency of plasma $\sqrt{\frac{n_e e^2}{m_e \varepsilon_0}}$. The charge of electron $e$, the mass of electron $m_e$, and the permittivity of vacuum $\varepsilon_0$, are all constant. Therefore, the frequency of
plasma uniquely depends on the density of plasma \( n_e \). By varying the density of plasma, the
cutoff frequency for the negative permittivity is thereby determined. It is reported that a
diagnostics of microdischarge-integrated plasma source could produce plasma with density of
\( 10^{13} \text{cm}^{-3} \), which corresponded to plasma frequency at \( \omega_p/2\pi \sim 28 \text{GHz} \)\(^{11,13} \). In this thesis, we
begin to explore the development of plasmas in split ring resonator configurations.

The generation of negative permeability in split ring resonator structure, shown in Figure
1-4, is illustrated well by Lorenzian model in appendix B. In this model, the permeability \( \mu_r \) is
described as below.

\[
\text{Re}(\mu_r) = 1 + \alpha \frac{\omega^2 (\omega_0^2 - \omega^2)}{(\omega_0^2 - \omega^2 + \omega \beta)^2} \\
\text{Im}(\mu_r) = \alpha \frac{\omega^3 \beta}{(\omega_0^2 - \omega^2)^2 + (\omega \beta)^2}
\]

(1.19) \hspace{1cm} (1.20)

Where \( \text{Re}(\mu_r) \) and \( \text{Im}(\mu_r) \) are the real part and imaginary part of the \( \mu_r \), respectively. \( \omega_0 = \frac{1}{\sqrt{LC}} \) is resonant frequency of SRR, in which \( L \) and \( C \) are the inductance and capacitance. \( \alpha \) and \( \beta \)
are the constants dependent on the dimension of the SRR. Curves can be easily plotted in Figure
1-5 according to the formula (1.19) and (1.20). Therefore, the design of the SRR plays a crucial
role in obtaining customized frequency above which negative permeability occurs.

![Figure 1-4. Schematic Representation of a Split Ring Resonator (SRR) structure](image)

Figure 1-4. Schematic Representation of a Split Ring Resonator (SRR) structure
1.3 The reliability of the SRR Metamaterials

Metamaterials with negative refractive index are easily realized by utilizing periodic arrangement split ring resonators with a plasma in the gaps. However, the biggest disadvantage of this concept is that plasmas cause irreversible damage to the structure, including erosion of the metal rings and dielectric substrate. When such damage occurs, it gives rise to degradation of the metamaterial response, resulting in a lower reliability and a shorter life circle.

In this thesis, an additional structural feature is proposed to protect the metal rings in a harsh plasma environment. The ends of the silver rings at the gap were covered by a dielectric layer which inhibits erosion of the metal ring. It was demonstrated that the rings survive a high power microwave environment (1000W) for more than 5 hours. Besides the experimental data, simulation shows as well the variation of the electric field at the gap between original SRR and protected one. Based on these observations and kinetic data a detailed analysis is discussed on the mechanism of degradation of this type of metamaterials in plasma.
In this thesis, the overall design, fabrication and testing of split-ring resonators are covered. Chapter 2 provides an overview of the material selection with an emphasis on the dielectric substrate performance in a plasma environment. Chapter 3 introduces design and fabrication of SRR, including analytical expressions for predicting the resonant frequency and low power measurement methods. High-power SRR testing is discussed in Chapter 4 followed by an analysis of protective dielectric layers to avoid silver erosion of the metal ring. The conclusion and the future work will be discussed and given in Chapter 5.
Chapter 2

Selection of the Substrate for Split Ring Resonator

2.1 Introduction

In Chapter 1, the density of the plasma is a pivotal parameter in achieving negative refractive index because it determines the cutoff frequency below which a negative permittivity can be achieved. In practice the electron density is $10^{13}$-$10^{14}/\text{cm}^3$ range, which is sufficient for the metamaterials to operate in microwave frequency. However, such an intense plasma presents a challenge in metamaterial design. The SRR component must be able to withstand the attack of the high power plasma with no obvious physical damage and degradation of the elemental dielectric properties for sustained period of time. Since the metal ring and dielectric substrate are exposed to the plasma, the degradation of both materials needs to be explored. In this chapter, only the substrate is discussed.

Both polymer and ceramic materials are candidates for SRR substrates [14]. However, lower temperature operation makes polymer less reliable than ceramics due to heating from the plasma. Important materials parameters include dielectric permittivity, dielectric loss and high erosion resistance in a plasma environment. In this chapter a measurement technique is introduced to test the suitability of dielectric substrates in a harsh plasma environment.

2.2 PI facility and process of the plasma experiment

Three materials were tested in this experiment including Al$_2$O$_3$, ZrO$_2$ and quartz, with an approximate geometry of 50mm×50mm×0.6mm, as shown in Figure 2-1. In order to explore substrate degradation in plasma environment, the PI facility at UCLA was employed (Figure 2-2).
The sample holder in the PI facility was made of stainless steel, and the dielectric plate sample was fixed towards the plasma projector, as shown in Figure 2-2 (a). The sample assembly was placed in a sealed chamber where atmosphere could be customized, in Figure 2-2 (c). When a voltage was applied, high power ion current flowed in the direction perpendicular to the surface of sample, which ensured the sample was attacked in the highest energy, as shown in Figure 2-2 (b). The temperature of the target due to the bombardment of energetic ions was recorded as a function of time. After undergoing the bombardment of plasma for a given duration, the sample was taken out for the measurement of the physical and electric properties.

Figure 2-1. The test samples. (a) Al₂O₃; (b) ZrO₂; (c) Quartz.
Figure 2-2. Photos are from the PI Plasma Interaction facility in Dr. Wirz’s Lab at UCLA. (a) Photo of the sample within the controlled atmosphere chamber; (b) Plasma excited on the surface of the fixed dielectric; (c) PI facility chamber.

Three materials were tested as candidate SRR substrates under the condition shown as the Table 2-1. Al₂O₃ and quartz were commercial, ZrO₂ was prepared in the lab using tape-casting.

All samples showed no obvious physical damage in plasma for the duration listed. It is noteworthy that remarkably higher ion current was detected at the surface of Al₂O₃ than other two plates, although under the same voltage applied, along with an extremely higher temperature.

Table 2-1. The Pi Facility conditions for the plasma exposure.

<table>
<thead>
<tr>
<th></th>
<th>Al₂O₃</th>
<th>ZrO₂</th>
<th>Quartz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage(V)</td>
<td>52V</td>
<td>55V</td>
<td>60V</td>
</tr>
<tr>
<td>Ion current(A)</td>
<td>0.26A</td>
<td>0.09A</td>
<td>0.085A</td>
</tr>
<tr>
<td>Target Temperature(°C)</td>
<td>400</td>
<td>130</td>
<td>220</td>
</tr>
<tr>
<td>Duration(mins)</td>
<td>300</td>
<td>200</td>
<td>315</td>
</tr>
</tbody>
</table>

The most important parameters are the substrate temperature and ion current. Temperatures exceeded 100°C for all substrates, which exceed the operating temperature of many polymer substrates. Ion current is related to the overall bombardment flux of the Ar ions.
2.3 Measurement of properties of the substrate materials

In order to select the material more suitable for the substrate, a comparison is necessary of the robustness and the electric properties between the pre-excited and the post-excited states.

The robustness is easily determined. No visual damage was observed for all samples that were exposed to plasma in the PI facility. Therefore, any of these materials are suitable for SRR substrates. A precise record of weight loss was provided by UCLA, shown in Table 2-2.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Testing period (minutes)</th>
<th>Original weight (g)</th>
<th>Reduction in weight (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al2O3</td>
<td>300</td>
<td>6.2026±0.0002</td>
<td>3.4±0.3</td>
</tr>
<tr>
<td>ZrO2</td>
<td>200</td>
<td>8.1007±0.0002</td>
<td>4.3±0.2</td>
</tr>
<tr>
<td>Quartz</td>
<td>315</td>
<td>9.6821±0.0002</td>
<td>88.2±0.2</td>
</tr>
</tbody>
</table>

From the Table 2-2, the weight of loss demonstrates a limited loss of material due to erosion, even for Al2O3 after 5 hours’ of exposure. The very small weight loss could be attributed to removal of organics or moisture from the surface. Therefore all substrate materials are suitable for plasma applications.

The dielectric properties included resonant frequency $f$, relative permittivity $\varepsilon_r$ and quality factor $Q$, measured by a vector network analyzer. The measurement of dielectric properties can be carried out by vector network analyzer Anritsu 37369D in Figure 2-3 (a). The dielectric properties are measured by a split cavity technique in which the substrates is placed at the center of the cavity [15]. The electric field vector is parallel to the substrate. The transmission coefficient, S21, is measured as a function of frequency and the peak is designated as the resonant frequency, as shown in Figure 2-3 (c). The data are easily read from the panel, listed in Table 2-3.
Figure 2-3. The vector network analyzer for the measurement of electric properties. (a) Photo of vector network analyzer Anritsu 37369D; (b) Split Cavity Resonator; (c) the curve of S21 signal measured by the network analyzer.

Table 2-3. The condition of the plasma experiment on the substrate.

<table>
<thead>
<tr>
<th></th>
<th>Al₂O₃</th>
<th>ZrO₂</th>
<th>Quartz</th>
</tr>
</thead>
<tbody>
<tr>
<td>resonant frequency(GHz)</td>
<td>14.5</td>
<td>10.2</td>
<td>15.0</td>
</tr>
<tr>
<td>relative permittivity εᵣ</td>
<td>9.3</td>
<td>24.0</td>
<td>3.8</td>
</tr>
<tr>
<td>quality factor Q</td>
<td>1100</td>
<td>800</td>
<td>2900</td>
</tr>
</tbody>
</table>

From the Table 2-3, there was scarcely change in frequency and relative permittivity. However, significant reduction in quality factor needs an explanation, for which a parallel mixing rule model was introduced.

The Figure 2-4 shows the photos of the surface being bombarded by plasma. In each sample there was dark residue at the center of the surface, which EDS shows was carbon. Figure
2.5 is cross-section of the measurement, based on which the relationship of the Q of different part could be established as equation 2.1.

\[
\frac{1}{Q_{\text{Total}}} = \frac{1}{Q_{\text{Substrate}}} + \frac{1}{Q_{\text{Residue}}} \quad (2.1)
\]

At the same time, the conductivity could be calculated by Q as equation (2.2),

\[
\sigma = \frac{\omega \varepsilon_0 \varepsilon_r}{Q} \quad (2.2)
\]

Where the \(\omega\) is the resonant frequency, insert parameters in Table 2-3 into equation (2.2), we can obtain the quality factor and conductivity of carbon residue shown in the Table 2-4.

<table>
<thead>
<tr>
<th>(Q_{\text{residue}} \times 10^4)</th>
<th>(\sigma_{\text{residue}} \times 10^4 \text{S/m})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al(_2)O(_3)</td>
<td>0.29</td>
</tr>
<tr>
<td>ZrO(_2)</td>
<td>0.99</td>
</tr>
<tr>
<td>Quartz</td>
<td>1.11</td>
</tr>
<tr>
<td>1.89</td>
<td>0.56</td>
</tr>
<tr>
<td>0.50</td>
<td></td>
</tr>
</tbody>
</table>

Carbon has two allotropes in general, diamond with conductivity of \(10^{-13}\) S/m, and graphite of around \(10^{-1}\) S/m. The carbon residue of all samples are in the order of magnitude of \(10^{-4}\) S/m, indicating the residue must be graphite in which there is little inter-particle connectivity [16]. Since the graphite did not form a network, and plasma will excited actually in a very small area in the gap of SRR as discussed in next chapter, the negative effect of this residue must not be taken into account.

As a conclusion, though there is no change in resonant frequency and permittivity in all samples when excited, Al\(_2\)O\(_3\) has a higher Q and better mechanical resistance to plasma attack compared with ZrO\(_2\). Moreover, because the permittivity of the quartz is much lower than Al\(_2\)O\(_3\) and ZrO\(_2\), for the same working frequency the SRR must be designed in a much bigger geometry. Therefore, Al\(_2\)O\(_3\) is used as substrate for the split ring resonator in this thesis.
Figure 2-4. The samples of the substrate after bombardment of plasma. (a) Al$_2$O$_3$; (b) ZrO$_2$; (c) quartz.

Figure 2-5. Schematic diagram of measurement in split cavity resonator in cross-section view.

The effect of a small amount of residual carbon is shown to have a detrimental effect on the microwave loss of the substrate and ultimately the Q of the ring resonator. It is anticipated that carbon residue will be formed on polymer substrates during plasma exposure so ceramic substrates should be ideal for SRRs.

An improved exciting technology is discussed in Appendix C.
Chapter 3
Design, Fabrication and Simulation of the Ring Resonator

3.1 Introduction

In Chapter 1, metamaterials have been introduced as periodic structures with split ring resonators (SRR) elements that are spaced at the subwavelength scale, as shown in Figure 3-1[12]. Although the rings are square shape, the electromagnetic field configuration and the plasma ignition mechanism are the same as for circle rings, which will be illustrated later in this chapter.

Figure 3-1. The split ring resonators. (a) Schematic representation of SRR metamaterials on which plasma is generated by remote power from an antenna; (b) Photograph of a metamaterial board; (c) Plasma ignited at atmospheric pressure.[12]

Figure 3-1 (a) is a schematic representation of a metamaterial substrate that is exposed to high power microwave at the power in the range of 2-50W. The plasma is excited at the resonant frequency of the individual ring resonators. Figure 3-1 (b) shows the dielectric substrate (Rogers RO4003C, 0.8mm thickness) with two series of SRR made of different size, in order to produce plasmas at different frequencies. The smaller ring is designed to resonate at 2.45GHz and the
larger ring is designed at 2.1GHz. Figure 3-1 (c) is a photograph of the plasma which is ignited at atmospheric pressure and frequency of 2.45GHz[12].

3.2 Estimation of resonant frequency

Figure 3-2 shows the voltage profile along the circumference of the split-ring at the resonant frequency. The voltage starts at the highest level at one end of the split-ring and then transitions to the lowest level at the other end. The voltage difference results in a high electric field across the gap, resulting in a maximum voltage at the gap that could ignite plasma if the voltage is high enough. The resonant frequency can be determined from Equation (3.1)

$$f_{RN} = \frac{n \cdot c}{2\pi D \sqrt{\varepsilon_{eff}}}$$  \hspace{1cm} (3.1)

Where, $\varepsilon_{eff}$ is the effective permittivity, $f_{RN}$ is the resonant frequency, $n$ is the mode which is 1 in this design, $c$ is velocity of light $3 \times 10^8 \text{m/s}$, and $D$ is the diameter of SRR.

Figure 3-2. The schematic diagram of plasma in SRR generated by sinusoidal wave.
However, according to the fact that the waves along the ring go separately through both air and dielectric substrate, the value of $\varepsilon_{\text{eff}}$ should be an intermediate value of the air and $\varepsilon_r$ of the substrate. The equation (3.2) provides reasonable approximation for the relationship between permittivity of substrate $\varepsilon_r$ and effective permittivity of the device dependent on the geometry shown in Figure 3-3.

$$\varepsilon_{\text{eff}} = \frac{\varepsilon_r+1}{2} + \frac{\varepsilon_r-1}{2} \left[ (1 + 12 \frac{h}{W})^{-\frac{1}{2}} \right]$$  \hspace{1cm} (3.2)

![Figure 3-3](image)

(a) The top view showing the conductor on the dielectric substrate; (b) The side view which shows the SRR and silver bottom ground plane.

Where $h$ is the height of the substrate, and $W$ is the width of the resonator. We can calculate the diameter of SRR in particular resonant frequency based on the equation (3.2). Table 3-1 summarizes the results for six rings that resonated between 2.40GHz to 2.50GHz. The resonant response can be altered through changing the ring diameter. Therefore the diameter of the resonator should be designed for a resonant frequency of the microwave source (2.45GHz).
Table 3-1. The design of the resonator

<table>
<thead>
<tr>
<th>Sample</th>
<th>Width of the Resonator (mm)</th>
<th>Height of the Substrate (mm)</th>
<th>$\varepsilon_r$</th>
<th>$\varepsilon_{eff}$</th>
<th>Diameter of the Resonator (mm)</th>
<th>Resonant Frequency (GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td>7.72</td>
<td>2.40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td>7.65</td>
<td>2.42</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>0.63</td>
<td>7.59</td>
<td>2.44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td>7.53</td>
<td>2.46</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td>7.46</td>
<td>2.48</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
<td>7.41</td>
<td>2.50</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The width of the conducting ring was chosen to be 1 mm, which was within the process window of the thick film fabrication method. The height was taken from commercially available alumina and the permittivity was determined from split-cavity measurement. The $\text{Al}_2\text{O}_3$ for the substrate is 96% $\text{Al}_2\text{O}_3$ with the permittivity of 9.42.

The effective permittivity was calculated from Equation 3.3 and the resonant frequency from Equation 3.2.

### 3.3 Ring design and simulation

The analytical model in Section 3.2 provided an accurate estimate of the ring resonator dimensions for a specific resonant frequency. The value of the resonant frequency (2.45GHz) coincides with the commercial high power microwave sources. In order to validate the electromagnetic simulations, which will be important for calculating the magnitude of the magnetic field, the analytical results were compared with simulation. In this section, CST (Computer Simulation Technology), software for the simulation of electromagnetic fields in arbitrary 3D structures, will be described.

In simulation, the first step is to build the structure in the main view of the CST software environment which represents the actual SRR sample, as shown in Figure 3-4. All of the important parameters are defined in the build step including: dimensions of the metal and
dielectric structures, material parameters such as permittivity and conductivity, and the frequency range of interest. The gap width of 0.15 mm was taken from literature where it was demonstrated to produce sufficient field for plasma ignition [12].

![Image of SRR simulation structure](image)

**Figure 3-4.** The simulation structure of the SRR. (a) The 3D schematic view of the overall SRR with a magnified view of the gap region; The insertion is the probe location in gap which gives the spatial variation of the electric field. There are two probes within the gap as represented by a set of white and green arrows; (b) the parameters of the SRR design for simulation.

<table>
<thead>
<tr>
<th>Parameter List</th>
</tr>
</thead>
<tbody>
<tr>
<td>Name</td>
</tr>
<tr>
<td>Diameter_resonator</td>
</tr>
<tr>
<td>Height_resonator</td>
</tr>
<tr>
<td>Height_substrate</td>
</tr>
<tr>
<td>Width_gap</td>
</tr>
<tr>
<td>Width_resonator</td>
</tr>
</tbody>
</table>
The variables shown in Figure 3-4(b) were obtained from the analytical model in the previous section. Attention should be paid to the coordinate system in Figure 3-4 (a), because the following discussion refer it.

In this simulation, an electric field with default input power of 1W is projected to the surface of SRR in the direction antiparallel to the Z-axis, because this is proved to be the most efficient excitation of the resonant mode. The magnitude of the electric field vector is shown in Figure 3-5, with the highest electric field observed at the gap region.

![Figure 3-5](image)

Figure 3-5. The schematic of field distribution for the SRR at the resonant frequency of 2.45 GHz.

After the excitation signal has been generated it propagates along the ring surface and generates surface currents in the metal ring. At the resonant frequency, a standing wave pattern for the voltage is produced as shown in Figure 3-2. We established two probes in the gap, as
shown in Figure 3-4 (a). They are all at the same height along the Z axis, and at the middle region of SRR, but at each termination of the SRR. The simulated resonant frequency is 2.479GHz, as determined by the probe signal, shown in Figure 3-6. Since the two probes are in the same condition, one of them is enough to collect the data. The corroboration between the analytical and simulation mode is excellent and the simulation model will be extended in the next section to include the effect of protective dielectric layers on the overall resonant frequency of the SRR.

![Graph of frequency characteristic of the SRR](image)

Figure 3-6. The frequency characteristic of the SRR collected by the probe located as shown in Figure 3-4 (a).

3.4 Fabrication of the ring resonator

The analytical model and electromagnetic simulation provided the SRR ring dimensions. The resonator can be fabricated by a stenciling method in which the pattern is made through cutting tape to form a ring outline on the alumina substrate. Silver paste (DuPont 6160A) is painted on the substrate followed by firing at 850°C in belt furnace. The gap width was the most challenging to control by the stenciling method. However, according to the experience in lab, if the width of gap is under 0.3mm, this uncertainty has no negative effective in the resonant frequency or
exciting a plasma. The fabricated ring pattern with a well-defined gap is shown in Figure 3-7. The geometry parameters of the fabricated SRR are listed in Table 3-2.

![Photograph of the SRR made of silver ring painted on the Al₂O₃ substrate.]

Table 3-2. The list of the parameters of the fabricated SRR

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Value (mm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>D</td>
<td>7.25</td>
<td>The diameter of the SRR</td>
</tr>
<tr>
<td>W</td>
<td>1.00</td>
<td>The width of the SRR</td>
</tr>
<tr>
<td>G</td>
<td>0.10</td>
<td>The gap of the SRR</td>
</tr>
<tr>
<td>h</td>
<td>0.62</td>
<td>The height of the substrate</td>
</tr>
<tr>
<td>t</td>
<td>0.01</td>
<td>The thickness of the SRR and silver bottom</td>
</tr>
</tbody>
</table>
3.5 Low Power Measurement of SRR

In this section, the resonant frequency and Q factor of the resonators are of primary interest. They can be measured by using Agilent E8364B PNA Network Analyzer (10MHz-50GHz), shown in Figure 3-8 (a). The SRR is connected to the PNA through inductive coupling in which the magnetic field of the loop probe is placed in the region of maximum magnetic field of the SRR. When using the fixture in Figure 3-8 (b), it is crucial to make the loops of the probes parallel to the resonator. The resonant frequency and 3dB points are read from the screen in Figure 3-8 (c), that resonant frequency is 2.4627GHz and Q factor is 134.43 for this sample. A series of SRR were measured and the dielectric properties were listed in Table 3-3. Though the actual values of the parameters differ from the estimated ones in Table 3-1, the diameter is inversely proportional to the resonant frequency as predicted. The Q factor represents the electromagnetic energy stored in the ring divided by the energy loss. Q factor can be calculated by the following equation (3.3).

\[
Q_L = \frac{f_{RN}}{3d\Delta f}
\]  

(3.3)

Where \(3d\Delta f\) is the bandwidth at half power.

Table 3-3. Dielectric properties of a series of SRR

<table>
<thead>
<tr>
<th></th>
<th>diameter of SRR(mm)</th>
<th>gap width of SRR(mm)</th>
<th>resonant frequency(GHz)</th>
<th>delta f of SRR(MHz)</th>
<th>Q factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.45</td>
<td>0.16</td>
<td>2.425</td>
<td>20.46</td>
<td>119</td>
</tr>
<tr>
<td>2</td>
<td>7.39</td>
<td>0.06</td>
<td>2.435</td>
<td>18.84</td>
<td>129</td>
</tr>
<tr>
<td>3</td>
<td>7.36</td>
<td>0.12</td>
<td>2.458</td>
<td>18.20</td>
<td>135</td>
</tr>
<tr>
<td>4</td>
<td>7.33</td>
<td>0.12</td>
<td>2.476</td>
<td>19.15</td>
<td>129</td>
</tr>
<tr>
<td>5</td>
<td>7.26</td>
<td>0.12</td>
<td>2.482</td>
<td>18.48</td>
<td>134</td>
</tr>
</tbody>
</table>
In this chapter, the design of the SRR was based on an analytical equation that accounts for the dielectric properties of the substrate and geometry of the SRR. The computer simulation results closely matched the analytical model and samples were made by a thick film method. The SRRs were tested at microwave frequency and the resonant frequency of 2.45GHz were validated.
Chapter 4

Plasma Excitation of the Ring Resonator in a High Power Microwave Environment

4.1 Introduction

A split-ring resonator model was introduced in Chapter 3 that defines the materials, the geometry and the resonant frequency. Simulation of the resonant frequency via CST software were also validated through an analytical model and measurement in Chapter 3. The goal in this research is to provide SRR based metamaterial that will work in harsh plasma environment with no degradation and long operation life.

There have been several previous studies on using split-ring resonators as microwave plasma sources [17,18]. Similar to the work in this thesis, a micro-strip configuration was designed and fabricated. Different ring dimensions were made with operation frequencies between 830 MHz and 910 MHz. Figure 4-1 show the minimum rf power thresholds for igniting the plasma. The ring structures were placed in a vacuum system where the gas species and pressure were controlled.
Figure 4-1. The minimum ignition power as a function of pressure for two different gas environments[17].

As shown in Figure 4-1, there is a decrease in the ignition power as the pressure increases from 0.5 Torr to 10 Torr then the ignition power increases beyond 2 W as the pressure increases beyond 10 Torr. There is also a significant increase in ignition power as the gas species changes from noble gas (Argonne) to diatomic molecules (oxygen/nitrogen mix). The curves shown in
Figure 4-1 are closely associated with Paschen’s law which predicts the breakdown of gases as a function of pressure and distance between the electrodes. At pressures above 10 Torr the mean-free path of electrons decreases as the number of molecules increases within the volume. At low pressures the distance between the electrodes becomes the limiting factor as the energy of emitted electrons need to be sufficient for ionization[19].

The erosion properties of the dielectric substrates were studied in Chapter 2, and a novel method to protect SRR from the attack by plasma will be discussed in detail in this chapter. But in the beginning of this chapter, it is necessary to make a brief introduction on the ignition of plasma in the laboratory.

### 4.2 High Power Interaction of the SRR in Plasma

Plasma ignition requires sufficient energy which can ionize the gas atoms into ions and free electrons. In laboratory, the plasma is ignited by microwave oven shown in Figure 4-2. Figure 4-2 (a) is the photo of the microwave oven, HAMiLab-C1500 MW CHAMBER SYSTEM, which has a working area of 150mm×140mm×110mm. The insertion is the inside view where the aluminosilicate as refractory layers can be seen around the wall inside. A hole is at the center of the back wall of the chamber through which the lifted sample can be observed from outside, as shown in the Figure 4-2 (b). The insertion (b1) is the magnified view of an SRR sample in chamber. The insertion (b2) is the view of the sample through the hole from outside. It is noteworthy that only one SRR is enough since the goal of the experiment is to test the duration of the SRR working in plasma environment. The only parameters needed to be clarified are the electromagnetic frequency and power. This oven has a nonadjustable working frequency at 2.45GHz, though the SRR can be ignited with resonant frequency from 2.40GHz to 2.50GHz according to the previous experiments. The Power can be adjusted from 0 to 1kW.
4.2.1 The experiment on the bare SRR being excited

The first high power experiment was on an SRR made of silver on the Al₂O₃ substrate. The plasma with power of 300W lasted merely for 5 minutes in air and then it extinguished. The result is shown in Figure 4-3, with experimental details listed in Table 4-1. The resonant
frequency, measured by the vector network analyzer, shifted from 2.45GHz to 2.50GHz, which was beyond the upper limit of the high power microwave source. In Chapter 3, the resonant frequency is found to be inversely proportional to the circumference of the SRR according to the Formula (3.2). Therefore, we can draw a conclusion that circumference of the SRR was essentially shortened during plasma exposure, meaning an enlargement of the gap. At the same time, EDS of the composition in gap was listed in Table 4-4, where there was a large amount of Ag which could be in the form of a metal or oxide. The high resistance across the gap was maintained after plasma exposure which was confirmed by a digital multimeter.

![Figure 4-3](image)

Figure 4-3. The comparison of the SRR at the gap region. (a) Pre-ignited SRR; (b) Post-ignited SRR.

<table>
<thead>
<tr>
<th>Components of SRR</th>
<th>Silver ring, Al2O3 substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condition of ignited</td>
<td>300W, 2.45GHz</td>
</tr>
<tr>
<td>Duration of plasma</td>
<td>5 minutes</td>
</tr>
<tr>
<td>Change in width of the gap</td>
<td>0.51mm to 1.79mm</td>
</tr>
<tr>
<td>Shift of resonant frequency</td>
<td>2.45GHz to 2.50GHz</td>
</tr>
</tbody>
</table>

**Table 4-1. The condition and the results of the SRR being ignited**

### 4.2.2 The experiment on the partially protected SRR

In order to reduce the damage to the silver ring, dielectric protection layers were investigated. A commercial insulating dielectric paste (Dupont 5704) was painted over the top of
the conducting ring. As shown as Figure 4-4, two different coating configurations were explored. For convenience, we call the sample in Figure 4-4 (a) the partially protected SRR, and the one in Figure 4-4 (b) the wholly protected SRR in the following. The parameters of the Dupont 5704 coating are listed in Table 4-2.

![Image showing two samples with protective dielectrics](image)

**Figure 4-4.** The samples of the split ring resonators with protective dielectric overcoats. (a) partially protected SRR with gap exposed; (b) wholly protected SRR with gap covered.

<table>
<thead>
<tr>
<th>Protection</th>
<th>Thickness</th>
<th>Main Component</th>
<th>Relative Permittivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dupont 5704</td>
<td>0.043 mm</td>
<td>Al₂O₃</td>
<td>8–10</td>
</tr>
</tbody>
</table>

Table 4-2. The list of the parameters of the protection

In the high power microwave experiment, a plasma could not be ignited for the wholly protected SRR, even for power levels up to 1000W. Therefore in this section, only the partially protected SRR is discussed. The plasma being excited in this SRR lasted for more than 5 hours. The power sequence used to characterize the lifetime of the partially protected SRR is shown in Figure 4-5. The plasma was continuous until the high power microwave experiment was terminated. The photo of the sample after being excited is shown in Figure 4-6 and there is black residue on top of the dielectric, which did not affect the overall resonant properties. The resonator
properties of the sample were measured after high power microwave exposure and the data are listed in Table 4-3. There is scarcely a change in the resonant frequency of the partially protected SRR between before and after being excited. We can draw a conclusion that this type of protection is perfectly efficient when the silver SRR working in such highly powerful plasma for 5 hours.

Figure 4-5. The profile of exciting plasma on the partially protected silver SRR.

Figure 4-6. The photo of the partially protected SRR after being exposed to a plasma for a duration and power levels shown in Figure 4-5. (a) An optical micrograph of the SRR and gap region of the sample; (b) An expanded view of the gap shown by optical microscopy. The black region shows some change in the thick film overcoat.
Table 4-3. The dielectric properties of the sample in different stage.

<table>
<thead>
<tr>
<th></th>
<th>Resonant frequency(GHz)</th>
<th>Q factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>SRR before being coated</td>
<td>2.489</td>
<td>156</td>
</tr>
<tr>
<td>Partially protected SRR</td>
<td>2.466</td>
<td>164</td>
</tr>
<tr>
<td>Partially protected SRR</td>
<td>2.465</td>
<td>141</td>
</tr>
</tbody>
</table>

4.2.3 The EDS analysis on the composition of the gap

In Figure 4-7, a comparison of the EDS at the gaps is made between the bare SSR and partially protected SRR. And the content of major components is listed in Table 4-4. The results show that there is massive Ag observed at the gap of the bare SRR after being excited, like discussed in the section above which proves plasma bombardment results in the abnormally high Ag composition at the gap, while a very few Ag was observed at the gap of the partially protected SRR after being excited, in the same scale of the composition of Ag as of the bare SRR before being excited, meaning that Ag in partially protected SRR is beyond the influence by plasma.
Figure 4-7. The map scanning EDS of the gap. (a) Bare SRR before being excited; (b) Bare SRR after being excited; (c) Partially protected SRR after being excited. (The content of the point scanning is listed in Table 4-4)

Table 4-4. The content of the major components at the point scanning section in Figure 4-7

<table>
<thead>
<tr>
<th></th>
<th>Ag(atomic%)</th>
<th>Al(atomic%)</th>
<th>O(atomic%)</th>
<th>others(atomic%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>excited bare SRR</td>
<td>47.72</td>
<td>16.46</td>
<td>23.94</td>
<td>11.88</td>
</tr>
<tr>
<td>excited partially protected SRR</td>
<td>0.99</td>
<td>34.67</td>
<td>59.85</td>
<td>4.49</td>
</tr>
<tr>
<td>non-excited bare SRR</td>
<td>0.31</td>
<td>31.98</td>
<td>66.25</td>
<td>1.46</td>
</tr>
</tbody>
</table>
The EDX data and optical inspection of the SRR after plasma exposure suggest that a small amount of silver oxides is produced in the protected SRR. There is not enough material to cause a significant change in the resonant response for the partially protected SRR.

4.3 CST Microwave Studio Simulation of the Electric Field Intensity within the Gap for SRRs with and without Dielectric Layer Protection

The lifetime of the plasma was extended to an unfathomable extent in the partially protected SRR, as introduced in the last section. The main goal of this section is to explore the effect of the protective layer on the electric field distribution near the gap. It was experimentally determined that the fully coated SRR could not excite a plasma up to microwave power levels of 1 kW, so it is hypothesized that the electric field is not high enough to ignite the plasma.

In simulation, it is convenient to use the 96%Al2O3 as the default material, which has a permittivity of 9.6, in an approximation with the protection material used in lab, which has the permittivity of 8–10. The samples were designed in three configurations shown in the Figure 4-8. (a) is bare SRR with no protection. (b) is the SRR protected except the gap. And (c) is the SRR with gap wholly coated. Notably, there are only small regions around the gap being painted in (b) and (c), which mimicked the actual samples in the lab.
Figure 4-8. The simulation of SRRs in different protective method. (a) SRR without protection; (b) partially protected SRR with gap exposed; (c) wholly protected SRR with gap covered.

The simulation with various views is shown in Figure 4-9 (a). The Z axis is perpendicular to the surface of the resonator. The gap, the most important part of the device, is projected along the Y axis.

In order to observe the electric field distribution, we can read the information from a plane along the Y axis at the mid gap, as Figure 4-9 (b) shows.
A simple way to prove the effectivity of the simulation is comparing the shift in the resonant frequency in different conditions between the simulating and experimental results. In Table 4-5, consistency is observed, which makes the electric field simulation more convincing. The result in Table 4-5 also show that the electric field, and resonant wave behavior, above the conducting ring is influenced by the dielectric protective layer.

Table 4-5. The shift of the resonant frequency

<table>
<thead>
<tr>
<th>Protection</th>
<th>simulating(GHz)</th>
<th>experimental(GHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non</td>
<td>2.479</td>
<td>2.484</td>
</tr>
<tr>
<td>partial</td>
<td>2.464</td>
<td>2.466</td>
</tr>
</tbody>
</table>

The electric field intensity in the SRR gap is shown in Figure 4-10 for the unprotected and partially protected SRR. It is observed that the electric field is more intense and in a more elevated position for the partially protected SRR. Then, an assumption can be drawn that plasma is ignited in general at the region of the most intensive electric field and generates the erosion nearby in a very small volume, shown in the Figure 4-11. In this assumption, the dielectric protection absorbed the most energy of plasma and showed no erosion for its inert character in
atmosphere. These assumption could be supported by EDS discussed in the last section, that a smaller amount of Ag existed at the gap of the partially protected SRR than for the bare one.
Figure 4-10. The 3D simulation results of SRRs showing the electric field intensity in the gap region. (a) 3D schematic of electric field at the gap of the SRR without the Dupont 5704 protective layer; (b) 3D schematic of electric field at the gap of the partially protected SRR with gap exposed.
Figure 4-11. The schematic of the electric field showing the influence of plasma by the red arrows. (a) The bare SRR; (b) The partially protected SRR.

Another simulation was performed for the SRR with a complete layer over the conductor, as shown in Figure 4-12. The electric field intensity is significantly lower for the completely protected SRR than for the partially protected SRR as shown in Figure 4-10.

The simulation results support the experimental evidence, by the fact that no plasma was ignited to the wholly protected SRR even with the power up to 1kW. The schematic of the electric field and its influence on plasma creation is shown in Figure 4-13.
Figure 4-12. 3D schematic of electric field at the gap of the wholly protected SRR.

Figure 4-13. The schematic of the electric field showing the possible location of plasma by an elliptical circle in (b). (a) The sample of the wholly protected SRR; (b) the schematic of the electric field intensity and showing the very low field in the air above the protective layer.
In this chapter, a protective layer was added to the SRR to work with high reliability in harsh plasma environment was discussed. It was found that there was significant degradation of the silver electrode near the gap when the SRR was exposed to a plasma in a high power microwave environment. There was a significant increase in the SRR durability in a plasma environment from 5 minutes to 5 hours, when a protective layer was added to the SRR.

It was also found that a plasma could not be ignited in the SRR for samples with uniform dielectric layers above the ring. A gap in the dielectric protective layer is necessary to ignite the plasma. Electromagnetic simulation confirmed the electric field levels were substantially smaller for the uniform dielectric coating than for the dielectric layer with a gap, suggesting that the threshold power exceeds 1kW for igniting a plasma in the SRR with a uniform dielectric layer.
Chapter 5

Conclusion and Future Work

The split ring resonator (SRR) is a fundamental element in left-handed materials (LHMs). Theoretical calculations show that a negative index of refraction can be generated through combination of a negative permeability and negative permittivity. Negative permeability is created through the magnetic field from the ring current and negative permittivity is created through a plasma. Though plasma has advantage in manipulating permittivity by altering its density, its tendency to damage most materials cannot be ignored. Especially if this metamaterial is expected to be applied in ambient atmosphere, very high power will be required to excite the plasma. Al₂O₃ was selected as substrate due to excellent dielectric properties and robust performance in extreme electrical and thermal environments. Alumina was shown to have excellent resistance to plasma exposure as shown in the PI facility experiments. It was also found that small amounts of carbon residues caused significant loss, as measurement by microwave cavity measurements. This is an important result because polymer materials degrade in plasmas to form carbon residues, so ceramic substrates will be better candidates for plasma split ring resonators. In addition, polymers have lower temperature operation than ceramics so heating from the plasma may be an issue for polymers.

A simple but effective design of split ring resonator was generated with analytical and simulation models. SRRs were fabricated by depositing thick film silver on an alumina substrate and there was significant degradation of the SRR under the plasma conditions. However, this was improved by the protection of a dielectric material Dupont 5704 coated with a gap. By this
method, the reliability was found to improve dramatically. The lifetime of plasma is prolonged from 5 minutes to more than 5 hours without the sign of the plasma extinguishing.

The experimental results were supported through electromagnetic simulation with CST software. It was found that the maximum electric field was suppressed when a complete dielectric coating was placed over the silver conducting ring, which supported the experimental fact that a plasma could not be ignited in the fully protected SRR. Electromagnetic simulation for the partially protected SRR showed that maximum electric field moved away from the silver ring, thus limiting the plasma exposure to the silver.

Future work should focus on CST simulations with an actual plasma because the electric field distribution could be substantially different between the SRR with and without the plasma. A conducting volume with materials properties approximating a plasma could be added to the simulation. This type of analysis could also help the understanding between plasma ignitions and sustaining processes.

The mechanism of the silver erosion is another topic which should be clarified. As to the forms in which the Ag exists at the gap, two assumptions can be drawn. The first is that Ag was oxidized due to plasma inputting enough energy to improve the oxidation reaction, which is challenged by the EDS results that Al and O have a ratio of around 2:3, as in the Table 4-4. Therefore it seems there is no extra oxygen atom to form Ag$_2$O in a large amount. The second assumption is that the Ag diffused by absorbing enough energy from the high power plasma. However, no elemental Ag was observed in microscopy, which contradicts the fact that massive Ag atoms were observed in EDS.

The mechanism of silver erosion should be further explored. The black coating on the partially protected SRR shows higher Ag content but the oxygen stoichiometry is difficult to ascertain because of background oxygen from the dielectric material under the black erosion product. It would be interesting to carry out the same high power experiments in different gas
environments. For example it is unlikely that silver oxide will be prevalent in an argon atmosphere.

Finally, more precise control and monitoring of the microwave power input needs to be carried out so that the ignition and sustaining powers can be distinguished. It is known that the SRR conditions change substantially after a plasma is ignited and precise power at every stage of the process will help in understanding the fundamental contributions of the different materials to the ultimate metamaterial structure.
References


Appendix A

Negative Permittivity

Although negative permittivity was realized by utilizing metal rods, scientists have substituted them with plasma because of the advantages that will be discussed well based on Drude model in this section. In this model, the state of electron can be described as following,

\[ \frac{d}{dt} \vec{P} = e\vec{E} - \frac{\vec{P}}{\tau} \]  
(A1.1)

\[ \vec{P} = m_e \frac{d\vec{r}}{dt} \]  
(A1.2)

Where \( \vec{P} \) is momentum of electron, \( \tau \) is mean free time between the collisions. Integrate the equation (A1.1) with (A1.2) inserted into it, we can obtain,

\[ \frac{d\vec{r}}{dt} = \frac{e}{m_e} \int \vec{E}(\vec{r}, t) dt - \frac{\vec{r}}{\tau} \]  
(A1.3)

Since \( \vec{E}(\vec{r}, t) \) could be represented by the product of two terms dependent on \( \vec{r} \) and \( t \) respectively, \( \vec{E}(\vec{r}) \cdot e^{-i\omega t} \), we can rewrite the (A1.3),

\[ \frac{d\vec{r}}{dt} = \frac{e}{m_e} \omega \vec{E}(\vec{r}) e^{-i\omega t} - \frac{\vec{r}}{\tau} \]  
(A1.4)

By solving this differential equation, we can obtain,

\[ \vec{r} = -\frac{e}{m_e \omega^2} \left( \frac{1}{1 + \frac{i}{\omega \tau}} \right) \vec{E}(\vec{r}) e^{-i\omega t} \]  
(A1.5)

Since polarization \( \vec{p} = n_e e \vec{r} \), insert it into (A1.5) and we can obtain,

\[ \vec{p} = -\frac{n_e e^2}{m_e \omega^2} \left( \frac{1}{1 + \frac{i}{\omega \tau}} \right) \vec{E}(\vec{r}, t) \]  
(A1.6)

As we have already known \( \vec{p} = \varepsilon_0 \chi_e \vec{E} \), we can simply obtain,

\[ \chi_e = -\frac{\omega_p^2}{\omega^2} \left( \frac{1}{1 + \frac{i}{\omega \tau}} \right) \]  
(A1.7)
Where \( \omega_p \) is plasma’s frequency, defined by,

\[
\omega_p = \sqrt{\frac{n_e e^2}{m_e \varepsilon_0}} \quad \text{(A1.8)}
\]

Since \( \varepsilon_r = 1 + \chi_e \), we can simplify (A1.7) under the condition where the frequency is much greater than the characteristic frequency \( \frac{1}{\tau} \),

\[
\varepsilon_r = 1 - \frac{\omega_p^2}{\omega^2} \quad \text{(A1.9)}
\]

From (A1.9), we can realize the negative permittivity by choosing a higher plasma frequency than the EM frequency, which could be achieved by increasing the density of plasma as pointed out in (A1.8).
Appendix B

Negative Permeability

The negative permeability of split ring resonator (SRR) can be explained well by Lorenzian model of an equivalent LRC circuit, as shown in Figure A2-1.

![Diagram of SRR and its equivalent circuit](image)

Figure A2-1. The split ring resonator and its equivalent circuit. (a) The top view of SRR; (b) The LRC equivalent circuit of the SRR

In the circuit model, the induced voltage by the change of the magnetic flux conforms to the Lenz’s law. \( \vec{H} \) is in the complex form, \( \vec{H}(\vec{r})e^{-i\omega t} \).

\[
V = -\frac{d\Phi}{dt} = -A\mu_0 \frac{d\vec{H}}{dt} = i\omega A\mu_0 \vec{H} \tag{A2.1}
\]

Where A is the area of the circuit, \( \mu_0 \) is permeability in vacuum, \( \Phi \) is magnetic flux. Still, we finds by using Kirchoff’s voltage law,

\[
V = (-i\omega L - \frac{1}{i\omega} + R)I \tag{A2.2}
\]
The magnetization density $M$ is $N \times I \times A$, where $N$ is the number of SRRs per volume.

Combining (A2.1) and (A2.2), we obtain,

$$M = \frac{i \omega N A^2 \mu_0 \bar{H}}{-i \omega L - \frac{1}{i \omega t} + R} \quad (A2.3)$$

From the relation $B = \mu_0 \mu_r H = \mu_0 (H + M)$, we obtain,

$$\mu_r = 1 + \frac{M}{H} = 1 + \frac{\omega^2 N A^2 \mu_0}{\frac{1}{LC} - \omega^2 - i \omega \beta} \quad (A2.4)$$

This equation can be simplified,

$$\mu_r = 1 + \alpha \frac{\omega^2}{\omega_0^2 - \omega^2 - i \omega \beta} \quad (A2.5)$$

Where $\omega_0 = \frac{1}{\sqrt{LC}}$ is resonant frequency of the circuit, also the SRR. By further calculating (A2.5), we obtain,

$$\text{Re}(\mu_r) = 1 + \alpha \frac{\omega^2 (\omega_0^2 - \omega^2)}{(\omega_0^2 - \omega^2)^2 + (\omega \beta)^2} \quad (A2.6)$$

$$\text{Im}(\mu_r) = \alpha \frac{\omega^3 \beta}{(\omega_0^2 - \omega^2)^2 + (\omega \beta)^2} \quad (A2.7)$$

From (A2.6) and (A2.7), the dependence of $\text{Re}(\mu_r), \text{Im}(\mu_r)$ on $\omega$ can be plotted as Figure A2-2.
In Figure A2-2, it is clear that negative permeability can be achieved at the frequency higher than the resonant frequency of the SRR. However, the image part of the permeability is highest at the resonant frequency, which would cause more loss when wave transmitting through the medium. Therefore, a higher frequency should be taken into consideration in practice.
Appendix C

Improved Exciting Technology

From the discussion in the sections 2.3.1 and 2.3.2, Al₂O₃ is determined to be a suitable substrate because it has excellent dielectric, mechanical and thermal properties.

An improved method was used by UCLA that a tighter beam formed through target magnet. The comparison between the old and new method are shown in Figure A3-1. Besides, there was no metal-ceramic interface any longer in new method. As a result, the Al₂O₃ showed no unexplainable weight loss, which was 6mg from 6.1461g to 6.1455g in 5 hours. Fewer dark spot was observed at surface. Shown in Figure A3-2. Considering that the PI facility before had a large area of interface between stainless steel holder and dielectric sample, the dark part at the surface could be assumed that carbon was excited off the stainless steel holder. Therefore, the carbon residue can be neglected when the device is working in the carbon-free condition, which is majority environment where the SRR works. Therefore, Al₂O₃ is extremely excellent candidate for the substrate of SRR.

Figure A3-1. The comparison of the exciting method. (a) PI facility used before; (b) The new method with tighter beam by target magnet.
Figure A3-2. The comparison of the excited Al2O3 in plasma for 5 hours between two methods. (a) the excited sample in old PI facility; (b) the excited sample in new method.