DEVELOPMENT OF PDMS-BASED MICROMACHINING PROCESS FOR MICROFLUIDIC RECONFIGURABLE ANTENNAS

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Submitted by **SEYEDEHNASIM SEYEDPOUR ESMAEILZAD** in partial fulfillment of the requirements for the degree of **Master of Science in Electrical and Electronics Engineering Department**, **Middle East Technical University** by,

Prof. Dr. Gülbin Dural Ünver _____
Dean, Graduate School of **Natural and Applied Sciences**

Prof. Dr. Gönül Turhan Sayan _____
Head of Department, **Electrical and Electronics Eng.**

Prof. Dr. Haluk Külah _____
Supervisor, **Electrical and Electronics Eng. Dept., METU**

Assist. Prof. Dr. Kağan Topallı _____
Co-Supervisor, **UNAM, Bilkent University**

Examining Committee Members:

Prof. Dr. Tayfun Akin _____
Electrical and Electronics Eng. Dept., METU

Prof. Dr. Haluk Külah _____
Electrical and Electronics Eng. Dept., METU

Prof. Dr. Özlem Aydın Çivi _____
Electrical and Electronics Eng. Dept., METU

Dr. Özge Zorlu _____
Electrical and Electronics Eng. Dept., METU

Assist. Prof. Dr. Kağan Topallı _____
Material Science and Nanotechnology Institute, Bilkent University

Date: 06/01/2015

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Name, Last name: SeyedehNasim, Seyedpour Esmaeilzad

Signature:

ABSTRACT

DEVELOPMENT OF PDMS-BASED MICROMACHINING PROCESS FOR MICROFLUIDIC RECONFIGURABLE ANTENNAS

Seyedpour Esmaeilzad, SeyedehNasim
M. S., Department of Electrical and Electronics Engineering
Supervisor: Prof. Dr. Haluk Külah
Co-Supervisor: Assist. Prof. Dr. Kağan Topallı
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The objective of this thesis is to develop fabrication methods to implement microfluidic-based reconfigurable antennas. As the initial structure, a microfluidic based reconfigurable antenna is developed for transmitarrays, which consists of a multi-layered structure incorporating a microfluidic channel to confine liquid metal. The microfluidic channels are fabricated using soft lithography techniques where the channel material is PDMS.

PDMS-to-glass and PDMS-to-PDMS bonding processes are optimized to achieve a reservoir for the fluidic material. In particular, a split-ring resonator and a complementary split ring for transmitarray applications are manufactured using the optimized process. Instead of using direct metallization approach, which is inadequate because of the low surface strength of PDMS, a novel method is proposed for the bonding of two different surfaces. A thin layer of silicon dioxide

(50 nm) is deposited on the metal layer before the oxygen plasma treatment. The oxygen plasma treatment is done for duration of 20 seconds at an RF power of 20 W. Bonding strength measurements are also performed in the frame of this thesis. It is noted that the bonding quality can be enhanced with the use of silicon dioxide intermediate layer.

The other device that has been designed and manufactured is a frequency tunable patch antenna, which is formed by reconfiguring its dimension employing two rectangular patches that are joined to each other with a liquid metal channel. The microfluidic channel is created by applying the bonding method between PDMS and metallized glass layers. The antenna has two different operation modes: (i) The channel is empty and the antenna resonates at 10.68 GHz and (ii) the condition in which the channel is full of liquid metal and the antenna resonates at 9.62 GHz.

In conclusion, microfluidic transmitarray and microfluidic reconfigurable antenna are fabricated using liquid metal channel embedded in the PDMS layer.

Keywords: Microfluidics, reconfigurable antenna, transmitarray, split ring resonator

ÖZ

MİKROAKIŞKAN TABANLI AYARLAMABİLİR ANTENLERİN GELİŞTİRİLMESİ

Seyedpour Esmaeilzad, SeyedehNasim
Yüksek Lisans, Elektrik ve Elektronik Mühendisliği Bölümü
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Ortak Tez Yöneticisi: Yrd. Doç. Dr. Kağan Topallı
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Bu tezin amacı mikroakışkan tabanlı ayarlanabilir antenleri gerçekleştirmek amacıyla üretim yöntemleri geliştirmektir. İlk yapı olarak, ileti diziler için, içerisinde sıvı metali sıkıştırmak üzere çok katmanlı bir yapı olarak oluşturulan bir mikroakışkan tabanlı ayarlabilir anten yapısı geliştirilmiştir. Bu yapıdaki mikroakışkan kanallar PDMS kullanılarak yumuşak litografi teknikleri ile üretilmiştir.

PDMS malzemeyi cam tabanlara yapıştırarak bir hazne oluşturma süreçleri optimize edilmiştir. Özellikle, ileti dizi uygulamaları için bölünmüş halka rezonatörü ve tamamlayıcı bölünmüş halka yapıları optimize edilmiş bu süreçler kullanılarak üretilmiştir. Doğrudan metal kaplama süreçlerinin kullanılması yerine, iki yüzeyi yapıştırmak için yeni bir yöntem geliştirilmiştir. Metal yüzeyin üzerine ince bir silisyum dioksit (50 nm) tabakası oksijen plazma öncesinde kaplanmıştır. Oksijen plazma süreci 20 W RF güç kullanılarak 20 saniye uygulanmıştır. Tez

kapsamında, bu yapının yapışma kuvveti ölçümleri de gerçekleştirilmiştir. Metal üzerinde silisyum dioksit ara katmanın kullanılmasıyla yapışma kalitesinin artırılabildiği görülmüştür.

Tasarlanmış ve üretilmiş olan diğer yapı ise, bir sıvı metal kanalı yardımı ile birbirine birleştirilmiş iki dikdörtgen yamanın kullanıldığı, boyutu ayarlanarak, frekansın da ayarlanabildiği yama anten yapısıdır. Mikroakışkan kanal, PDMS ve metal kaplanmış cam katmanları yapıştırmak için kullanılan yöntem kullanılarak oluşturulur. Antenin iki çalışma modu bulunmaktadır: (i) Kanalın boş olduğu ve antenin 10.68 GHz frekansında çalıştığı durum ve (ii) kanalın sıvı metal ile doldurulduğu ve antenin 9.62 GHz frekansında çalıştığı durum. Sonuç olarak, PDMS içerisinde sıvı metal mikroakışkan kanalların gömülü olduğu ileti dizi anten ve ayarlanabilir anten yapıları üretilmiştir.

Anahtar Kelimeler: Transmitarray, mikroakışkanlar, yeniden konfigüre anten, bölünmüş halka resonator

Dedicated to my family for being the reason who I am today

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CHAPTER 1

INTRODUCTION

Wireless communication devices, radar systems, satellite communication tools, and medical devices require antennas that can reconfigure their radiation characteristics such as polarization, radiation pattern and resonance frequency [1].

The necessity of improvement in gain, bandwidth and direction of antennas has led to the appearance of reconfigurable antennas [2]. Systems employing a reconfigurable antenna provide the probability of the size reduction and ease of integration with other electronic components. There are different kinds of reconfigurable antennas:

- Frequency reconfigurable
- Radiation pattern reconfigurable
- Polarization reconfigurable
- Tunable Antenna
- Switchable Antenna
- Multifunction Reconfigurable Antenna

In the frequency reconfigurable antennas, the resonance frequency displacement takes place in a return loss data. Radiation pattern reconfigurable antennas remodel the configuration, gain, or directivity of their radiation pattern. Utilization of polarization reconfigurable antennas results in an alteration in their polarization pattern. Multifunction reconfigurable antennas represent many

characteristics synthetized together to yield, for instance, a reconfigurable frequency with reconfigurable radiation pattern.

This thesis will present a reconfiguration mechanism, namely, microfluidics, that has been explored in the literature recently as a tuning and reconfiguration method for antennas. The following parts of the chapter present other methods used previously and compares it with this new reconfiguration modality. Some examples in the literature are also mentioned in the following subsections.

1.1 Reconfiguration Methods for RF devices and Antennas

There is different reconfiguration methods employed in the structure of antennas:

1. Employment of tunable materials
2. Utilization of physical/mechanical alteration
3. Manipulation of electronic switches

1.1.1 Tunable Materials

Natural electrical characteristics (conductivity, permittivity or permeability) of tunable materials can be regulated by application of an electric, magnetic, or optical bias. There are variety kinds of tunable materials such as liquid crystals; ferroelectrics, ferrites and semiconductors, which can be integrated to the structure of antennas to apply a variation in the operational characteristics of antennas.

Liquid crystals demonstrate the liquid state at high temperatures and crystalline solid-state behavior at low temperatures. Application of the electric field can change the dielectric constant of the liquid crystals, which make them desirable in the field of microwave devices. However, the main disadvantage associated with liquid crystals is that they are only in liquid phase in the temperature range of 20°C to 35°C [3].

The permeability of ferrites materials can be changed by operation of a DC magnetic field on their structure. Ferrites offer high tuning range. However, they require constant bias currents for having a movable permanent magnetic field.

The permittivity of the ferroelectric materials can be changed by execution of a static electric field. These kind of materials can be utilized in the structure of polarization reconfigurable antennas [3].

1.1.2 Structural Changes

Frequency reconfigurability can be accomplished by changing the physical structure of the antennas. Employment of actuators in the structure of antennas can offer desired alteration in the dimensions of the antenna. The other group of antennas, where structural changes play a role in the characteristics is flexible and textile antennas.

The main application of flexible and textile antennas are summarized as follow:

- Textile antennas are constructed from conductive textiles and they function near to the skin of human body. They can be utilized in diverse areas like health-care monitoring tools for patients.
- The main application area of the flexible Radio-frequency identification (RFID) antennas, is in the tracking, inspecting, general monitoring and locating of nearby objects
- The other main challenge in the antenna technology is to design reconfigurable antennas. The proposed method for the fabrication of tunable antennas is to apply microfluidics approaches. These kinds of antennas can be utilized in automotive radars, military operations and wireless communication devices.

Diverse methods are proposed for the construction of flexible, textile, and deformable antennas. The features of every method are discussed below.

Screen Printing: The screen printing method is one of the easiest techniques to fabricate the flexible antennas. During the process, a squeegee blade is utilized to apply force on the screen to move it near to the fixed substrate layer. Because of the applied force, the ink is ejected through the determined regions and the favorable model is created. Screen-printing can be utilized in three different models: flat bed, cylinder, and rotary [4].

The difference between the flat bed technique and cylinder screen-printing is in the way of pattern deposition. The desired template can be produced by turning the substrate while it is on the screen roll. In rotary screen method, ink and squeegee are rotated inside a rolled screen. The mentioned method is a useful technique in the fabrication of flexible antennas. However, the resolution of the achieved pattern is not high enough in this technique.

Chemical Etching: One of the practical methods in the fabrication of flexible antennas is to use chemical etching and photolithography. During the process, higher solution metallic templates are achieved. The main advantage of this process is the production of high accuracy samples. However, the procedure requires large amount of time, energy and cleanroom utilities. In addition, the process is not efficient enough and getting rid of the waste leftovers is the main difficulty of this technology [4].

Ink Jet Printing: In the ink jet printing method, the ink droplets are deposited in the required surface to generate the required pattern. The ink can be a conductive material such as silver nano-particle based ink. The inspection of the process is done from the computer, and there is no need to clean-room facilities [4].

1.1.3 Electronic Switches

Different switching mechanisms such as PIN diodes, Varactor diodes, GaAs field-effect transistor (FET) switches, and RF MEMS switches can be employed in the structure of reconfigurable antennas [5]-[8].

PIN Diodes: PIN diodes can be utilized as RF switches in diverse applications such as reconfigurable antennas. In the PIN diodes, wide region of intrinsic semiconductor is implanted between the heavily doped p-type and n-type regions. PIN diodes behave differently based on the biasing conditions. In the reverse bias mode, PIN diodes demonstrate lower capacitance. However, in the forward bias mode they behave like a resistor.

PIN diodes have been applied in reconfigurable antennas because of their high switching speed, low cost, and availability. However, there is considerable power dissipation due to the necessity of current bias existence in the on-state of a PIN diode. In addition, the efficiency of the reconfigurable antennas gets worse due to the resistance of the PIN diode. The main drawbacks of PIN diodes are their nonlinearity and intermodulation distortion, which affect the performance of antenna at high power levels.

Photoconductive Switches: Photoconductive switches are introduced to eliminate the parasitic problems caused by the electrical biasing. The photoconductive switches are actuated by the incident light and their operation speed is in the range of microseconds, which make them applicable in the reconfigurable antennas such as patches and dipoles. However, considerable amount of power is required to preserve the switches in the "ON" state. Moreover, the amount of loss in the photoconductive switches is quietly high and it is not negligible.

RF MEMS Switches: RF MEMS switches apply Microelectromechanical forces to displace micro machined conductors to create or break an electrical contact. There are different designs related to the RF MEMS switches. However, the main RF switches are constructed by utilization of conductive cantilevers or bridges.

The control electrode applies an electrostatic field to dislocate the cantilever or bridge.

The main property of RF MEMS switches is their lower power consumption. However, the switching speed of the RF MEMS switches is slower than the solid-state switches. In addition, RF MEMS switches outwear due to the mechanical stresses, which cause the limitation in their lifetime. RF MEMS switches may be curled or welded due to the application of high DC voltage level.

There is a considerable distinction between microfluidic technology and RF MEMS. Microfluidic devices do not suffer from the mechanical failures and self-actuation, which exist in RF MEMS switches. Microfluidic reconfigurable antennas are low resistant, lightweight, flexible, stretchable and low cost. Compared to semiconductor-based devices, microfluidics offer a platform to be used in high power applications since the materials have linear RF characteristics.

This chapter gives a brief introduction about microfluidic devices and their application in different areas such as RF field and antennas. There is a brief introduction about microfluidic switches. Since, these kinds of switches play an important role in the fabrication of reconfigurable antennas.

Microfluidic Switches: Microfluidics technology provides the opportunity to study and analyze the fluid behavior in sub-millimeter dimensions. The microfluidic devices are mainly employed in the biological and medical applications. Another important application of microfluidics is in the field of reconfigurable antennas. Microfluidic technology is proposed to solve the problems associated with the RF MEMS switches.

Liquid metal microfluidic switches are placed in the category of MEMS technology. However, these switches do not suffer from the mechanical fatigue, because of their fluid-fluid or fluid-solid feature. The utilization of microfluidic switches can solve the wear and mechanical fatigue problems that exist in RF MEMS switches. Microfluidic switches can offer new topologies and actuation possibilities, which can solve the existing challenges.

Although, the microfluidic switches are much slower than the RF MEMS and solid-state switches, they are still advantageous to be manipulated in the reconfigurable antennas.

Materials Used in the Structure of Microfluidics: The recent progress in the field of microfluidics is the utilization of flexible substances like polydimethylsiloxane (PDMS) in the fabrication procedure, which is explained in the following section [9], [10]. Microfluidic systems can be constructed from different kinds of substances such as silicon, glass, metals, ceramics, hard plastics, and elastomers. Liquid metals are injected inside the microchannels to implement microfluidic antennas.

PDMS: The most important property of PDMS, which makes it applicable in microfluidic devices, is its elasticity. PDMS has many favorable characteristics such as optical transparency, gas permeability, and simplicity of surface treatment. The behavior of the fluid through PDMS layers can be clearly observed due to the transparency of PDMS. Such devices are applicable in fluorescent detection and imaging because of their transparency.

Another superiority of PDMS is the ease of connection with the outer world. The accommodation of metal or plastic components is possible by making the holes in the PDMS layer.

Liquid Metal: The most popular liquid metals used in the fabrication of reconfigurable antennas are EGaIn, Galinstan, and Mercury. However, mercury is not appropriate because of its toxicity. Galinstan consists of (Ga 68.5%, In 21.5%, and Sn 10%) and EGaIn (eutectic gallium indium alloy) consist of Ga 75.5% and In 24.5%) [11]-[13].

The main characteristics of Galinstan, which make it applicable in reconfigurable antennas, are as follows:

- The liquid state of Galinstan at room temperature makes it injectable to the microfluidic channel.

- A thin layer of Ga_2O_3 is created due to the air exposure, which can prevent the creation of cracks.
- Other desirable properties of Galinstan include its low electrical resistivity (29.4×10^{-6} Ω.cm), high thermal conductivity (16.5 $W.m^{-1}.K^{-1}$), low toxicity, low vapor pressure, lightweight, and acceptable cost.

The main struggle in the complicated microfluidic systems is to find a technique to send the fluid in a controlled style between various on-chip parts. There is no chance to control the state of microfluidic switches and the liquid metal is pumped by syringe. The disadvantages of different switching methods are summarized in Table 1.1.

Table 1.1 Disadvantages of different switching methods

Switching methods	**Disadvantages**	**Switch speed**
RF MEMS	Limited lifetime, Complexity of fabrication, Mechanical fatigue, Low power handling capability, Slow switching speed, High cost	μs
FET	Non-linearity, Low power handling	ns
PIN Diodes	High DC bias current	ns
Varactor Diodes	Non-linearity, low dynamic range	μs
Photoconductive	Constant light bias	μs
Microfluidic	Slow	ms

1.2 Microfluidics for RF and Antenna Applications

The development of non-toxic and safe liquid metal such as Eutectic Gallium-Indium (EGaIn) and Galinstan offers new applications in reconfigurable RF elements. Liquid metal can be embedded inside the PDMS substrate for construction of flexible RF components. However, liquid metal antennas showed one-time reconfiguration by displacing liquid metal from one location to another. Potential advantages and disadvantages of Galinstan are summarized as follow:

Advantages of Galinstan

- Low cost and low power consuming devices can be produced.
- The deterioration of performances of the systems will be decreased due to the absence of parasitic effects caused by metallic bias lines or cabling.
- It does not require challenging fabrication processes such as etching or plating.
- In the case of microfluidic antennas, it can be quickly unified with other fluidic systems for sensing and tuning [13].

Disadvantages of Galinstan

- The main disadvantage of the Galinstan is its restricted application due to its sharp reaction with other metals.
- Fast oxidation of Galinstan makes it adhesive to microchannels of microfluidics devices.

Methods Utilized to Solve the Problems Associated with Galinstan

- Exploitation of Teflon micro tubes inside the channels can solve the existing problem. Utilization of Teflon solution with Galinstan inside the Teflon tube can intercept the oxidation progress [14].

- There are some other physical and chemical methods, which ameliorate the viscosity of oxidized Galinstan inside the microfluidic channel. In the physical method, four kinds of nanoparticles are deposited on the surface of paper textures embedded inside the PDMS channels. PDMS with titanium oxide (TiO_2) nanoparticles is more applicable in this method [15].
- In the chemical technique, the inner part of the PDMS microchannel is treated with sulfuric acid (H_2SO_4) [15]. Utilization of HCl-Suffusion papers in an inner wall of the microfluidic channel provides super-lyophobic channels against liquid metals.
- The employment of carrier liquids such as DI water, 1% polytetrafluoroethylene (PTFE, Sigma-Aldrich, Saint Louis, MO) in water, mineral oil (Amresco, Solon, OH), and 5% Novec 4300 (3M, St. Paul, MN) in mineral oil can erase the residue of Galinstan. Utilization of microchannel coating materials like Novec 2702 (3M, St. Paul, MN), 2% Pluronic F108NF (BASF, Florham Park, NJ), and 2% TWEEN 20 (Sigma, Saint Louis, MO) minimizes the hydrophobicity of PDMS [16].

Different Applications of Microfluidic Devices: The utilization of microfluidic technology offers variety of approaches in antenna technology such as wearable and textile antennas [15]-[20]. Liquid metal is loaded on a flexible substrate to change the operation frequency of the antenna by altering the length of the antenna.

Microfluidic channels filled with liquid metal are utilized in different applications such as flexible coils [21], beam steering antennas[22]-[24], liquid metal temperature or pressure sensor devices [25]-[27].

There are some new structures such as frequency selective surface (FSS) [28]: In the structure of tunable FSS, micropump is employed to move a Teflon solution and a liquid metal inside the tubes to adjust the surface impedance.

Liquid metal movement can be utilized in the structure of tunable band pass filters and transmitarrays [29]. For example, in the design of Reconfigurable nested ring-split ring transmitarray unit cell, circulation of the liquid metal together with the split around the ring provides 360° linear phase shift range [30].

Dislocation of liquid metal inside the pressure-driven capacitive microfluidic switches provides frequency reconfigurability in the structure of antennas. The switches are achieved by altering the displacement of conductive fluid within the channel, which reactively loads the slot [31].

Novel configuration of reconfigurable dual-band antenna, which consists of two slot antennas and four fluid metal channels on top of each antenna, is proposed. Different states of four empty and filled channels can offer digital frequency tuning [32].

Liquid metal switch is exploited in the structure of microstrip patch antenna, which can switch between the two antenna states [33]. However, liquid metal reconfigurable antennas need accurate emplacement inside the microchannels to acquire desired frequency, which is very challenging and slow. The utilization of micropumps and pressure-driven capillary devices can accelerate the process flow.

Combination of liquid metal reconfigurable patch antennas can propose a microfluidic enabled focal plane array (FPA). Pump improvised in the structure of FPA, can displace the liquid metal inside the reservoir [34].

Different materials can be exploited in the structure of frequency tunable liquid metal antennas. For example, liquid crystal polymer (LCP) layer is employed in the bottom side of PDMS microfluidic substrate. Capacitive coupling of LCP can offer an antenna with a microstrip feed line [35].

1.3 Objective of the Thesis

The aim of this thesis is to propose methods in order to design and fabricate microfluidic reconfigurable antennas. During this study, frequency reconfigurable microfluidic antennas are proposed and manufactured. A comprehensive list of objectives is as follows:

- Design and fabrication of microfluidic reconfigurable antennas. PDMS is used as a main material in the construction of microfluidic antennas.
- Optimization of PDMS-to-glass bonding processes using Oxygen Plasma tool. One of the main challenges in the field of microfluidic technology is to develop reliable devices, which can be obtained by high quality bonding.
- Optimization of PDMS-to-PDMS bonding processes using Oxygen Plasma tool The structure can offer different types of flexible antennas.
- Study and development of transmit arrays, which include a multi-layer structure with liquid metal inside the channels.
- Optimization of oxygen plasma parameters for bonding of Ti/Au deposited glass and PDMS layer. The process is possible by deposition of thin layer of silicon dioxide, without affecting the RF performance of the devices.
- Development of frequency tunable patch antenna with utilization of liquid metal channel. The antenna resonates at two different frequencies depending on the channel state.

1.4 Outline of the Thesis

This thesis is divided into five chapters. In chapter 1, there is a brief introduction to the microfluidic devices and their application in different areas. A brief literature survey is done related to the wearable antennas and their fabrication methods.

Chapter 2 describes the fabrication methods of flexible antennas. There is a brief introduction to (PDMS) and the Plasma Treatment method. In the PDMS preparation procedure, cost effective master production is employed. The optimization of the bonding process is explained in this chapter. Besides, the novel method is proposed regarding to the bonding of metallized glass samples to the PDMS material. For the bonding strength measurement, tensile strength test is performed. The cost-effective mold preparation is also done for PDMS casting.

Chapter 3 gives some brief introduction about the transmitarrays. The mentioned fabrication technique is deployed in the development of transmit arrays. The structure is a multi-layer microfluidic device, which is constructed by PDMS-to-glass bonding process. The fabrication of a split-ring resonator and a complementary split ring transmit arrays is presented in this chapter.

Chapter 4 explains the design and fabrication of a frequency tunable patch antenna. In this design, metallized glass sample and PDMS microfluidic channel are used. The antenna employs two rectangular patches, which connected to each other by a liquid metal channel. The antenna resonates at two different frequencies according to the channel condition.

Chapter 5 finalizes this thesis by summing up the accomplishments during this study and illustrating the future work related to this research.

CHAPTER 2

FABRICATION

In this chapter, there is a concise explanation to PDMS preparation method. In addition, the optimized condition is set for high quality bonding with plasma treatment method. In this study, bonding of metallized glass sample to the PDMS layer is conducted by depositing silicon dioxide layer before the plasma treatment method. Besides, peeling test is executed to measure the bonding strength.

2.1 Introduction to Polydimethylsiloxane

The substance employed in this research, is PDMS. PDMS is extensively used in the industry as a silicon-based organic polymer. The only polymer, which contains both organic and inorganic components, is PDMS. PDMS contains the groups of vinyl and hydrosiloxane as shown in Figure 2.1. The inorganic part is created by the oxygen and silicon atoms. This Si-O bond is nominated as siloxane bond, which grants the title of polysiloxane to this polymer. When a cross-linking factor is added to the primary material of the uncured PDMS, counteraction establishes affecting the organization of lengthy polymer chains and then the solidification of the mixture happens.

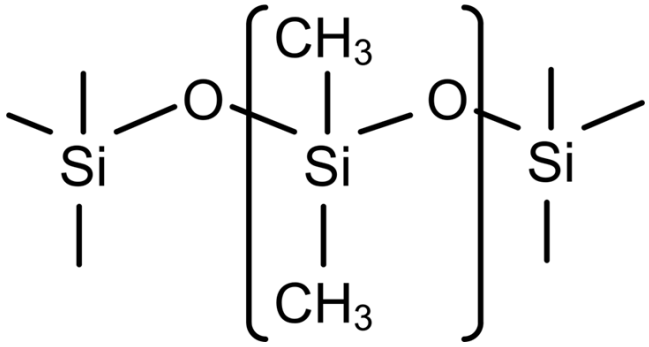


Figure 2.1 Polydimethylsiloxanechemical formula.

PDMS has attained prevalent application as a primary substance for microfluidics because it is transparent, flexible, and inert. The elasticity of PDMS makes it suitable in microfluidics application. One of the important advantages of this material is creating sophisticated models in micrometer range.

Flexible electronics need dense packaging and flexible electrical connections (as in cell phones). In order to introduce flexibility in the devices, polymers have appeared as remedy materials for flexible electronics and RF applications. The polymer layers are specified based on the necessity of their application.

In this study, a fabrication method is demonstrated for bonding of PDMS to metallized glass, which is used for reconfiguration of antennas. The fabrication technique implicates PDMS surface modifications such as oxygen plasma for adhesion improvement and stress management.

2.1.1 The Advantages of PDMS Utilization

PDMS has many advantages which make it best choice in flexible devices.

- The primary liquid state of PDMS provides the opportunity to make blend substrates of diverse condensation so that materials with different dielectric constants can be created. The other important advantage is that the thickness of the substrate can be verified because of the fluid state of elastomers. This property is extremely significant for flexible antennas because the thickness of the substrate plays a key role in the operation frequency. Compact packaging is one of the main demands of special systems. This property can be provided by using silicon elastomers.

- The compliance of PDMS is the principal incentive for bendable structures. Most of the flexible antennas are fabricated by the purpose of implanting. Therefore, the materials used in the fabrication of such devices should be biocompatible and bio-durable. PDMS is well-known because of possessing these properties. Therefore, it can be exploited for medical system applications, where infection inhibition has a great importance. Another issue to be considered is making a specific condition in which the mechanical property of the biological tissue is as same as the junction matter. PDMS with a Young's modulus between 360-870 KPa [50] is a preferable choice for body tissues. With this method, probable damages to the body tissues will be decreased.

- Silicon elastomer is an inert and hydrophobic matter because of its slight polar plane. Inertness and low-water retention are the most important characteristics of this matter. These properties reduce the possibility of erosion and warrant a long-term, permanent system operation. Therefore, silicon can be employed as a sealant and a waterproof cover in the fabrication process and as an electrical insulation in the semiconductor industry [51].

- Silicone elastomer is a polymer, which is extensively used in a fabrication process applicable at room temperature. Therefore, selecting silicone elastomer as a substrate makes the utilization of micro fabrication devices and cleanroom facilities possible.

According to the above-mentioned privileges, PDMS is a suitable material for the fabrication of the special sets mentioned here.

2.1.2 The Disadvantages of PDMS Utilization

Metal printing is quite a challenging process due to the low adhesion of inert PDMS layer. During the bending procedure, the printed metal layer may be peeled from the scheme. In order to solve this problem, another adhesion layer can be applied as a middle layer between the metal layer and the PDMS surface.

- The disagreement in the intrinsic characteristics of silicon elastomer and metals forms a mechanical inconformity between them. From the stiffness point of view, Young's modulus of silicon elastomers and metals should be considered. PDMS has a Young's modulus of 360-870 KPa, whereas copper has a Young's modulus between 110-128 GPa and platinum has a Young's modulus of ~168 GPa [51].

- The other critical issue is the existing variety in the thermal properties between silicon elastomer and metals. There is a thermal mismatch in the expansion coefficient between metals and PDMS. One executable method of metal deposition is the evaporation technique where the metal is heated until it changes to gas mode. The amount of thermal energy conveyed from the copper vapor to the PDMS cause an expansion, during the evaporation procedure [53]. However, during the cooling process, the

contraction of PDMS happens which leads to the compressive stresses. This results in the buckling of the copper film [54].

The PDMS specimens used in this study were mixed with the same mixture, which are Sylgard 184 silicon elastomer base and Sylgard 184 silicone elastomer curing agent. In order to obtain reliable results, different samples with variable base/agent ratios should be prepared. The amount of cross-linking agent can determine the PDMS stiffness. If it is added in a lower ratio, the softer PDMS is achieved.

The most extensively used PDMS samples are prepared with the ratio of 10:1. In this study, ten mass of Sylgard 184 silicone elastomer base mixed with one mass of Sylgard 184 silicone elastomer curing agent during the experiments.

2.2 Fabrication Methods

One of the most important criteria in the fabrication of PDMS microfluidic devices is to initiate a new trend for device mass production. The constructed sets in the laboratory are really time consuming and cannot be reduplicated easily. In order to solve the mentioned problems, Professor George M. Whitesides proposed a new method at Harvard University [38].

The technique related to fabrication of components from PDMS is introduced as a soft lithography method. The repetition of a topographically defined (typically in photoresist) structure on a master is called soft lithography. The procedure is performed in the laboratory conditions for many times. Therefore, it is fast, easy and inexpensive fabrication procedure.

2.3 PDMS Fabrication Process using Soft Lithography

To form a component with a special design, the required pattern should be sketched in a CAD Tool. In order to transfer the pattern of the mask to the silicon wafer, photolithography method can be used. After doing all steps, the patterned master is achieved. Since the aim of this process is to pour PDMS on the master, the fabricated master should be the negative of the required segment. Accordingly, if the demanded PDMS sample have channel, it should appear as a protuberance on the negative master.

The mixed material is located in a vacuum chamber to be degassed. The duration of degassing process is from 30 minutes to 2 hours [38]. In the next step, the fluid is poured onto the master. In order to increase the rapidity of the process, PDMS and master should be heated. After the curing step, PDMS is peeled off from the master.

2.4 Fabrication Steps for the Preparation of PDMS Samples

PDMS casting process has two basic steps. In the first step, the negative of a master is replicated. The next step is to pour and cure the required PDMS samples. Each step will be explained separately.

2.4.1 Manufacturing of Masters

Master fabrication plays a key role in the preparation of different PDMS samples. There are some steps involved in the manufacturing of masters. The process flow is as follow:

Mask Drawing: The required design must be drawn with a CAD tool. This 2-D layout is directly transferred to the polymer sheets using commercial laser-assisted image-setting systems [56]. The chrome masks are more generalized

because of their durability. However, the utilization of the polymer-based masks is more economical.

Photolithography: The common progress steps for a typical photolithography procedure are as follows: Substrate preparation, spin coating, soft baking, exposure, post-exposure bake, development, and hard baking

DRIE: After the photolithography, DRIE-etching process is utilized to form the microchannels on the silicon mold. Microfluidic channels are formed by using soft lithography techniques by using a silicon mold wafer for shaping the PDMS

2.5 PDMS Preparation

After the mask fabrication, it is the time to pour PDMS on the master and obtain the required pieces. There are some stages involved in creating the PDMS mold. The detailed explanation is as follow:

Combining Process: PDMS consists of two basic parts: (1) Prepolymer base material and (2) Curing agent. The PDMS package used in this study is Dow Corning Sylgard 184. In order to initiate the curing process, the prepolymer material is mixed with the curing agent. Since it is necessary to achieve a uniform mixture, the material should be mixed at least about 15 minutes. Sometimes, in order to minimize the created air bubbles, mixer can be used in the process. The preparation method is shown in Figure 2.2.

Figure 2.2 (a) Prepolymer base material and curing agent, (b) Pouring prepolymer,

(c) Pouring curing agent, (d) Mixture of the materials.

Degassing Process: One of the most important steps in the PDMS preparation is the degassing procedure. Due to the mixing operation, some air bubbles are created in the liquid substance. The developed bubbles should be eliminated, because they may form unfavorable holes in the cured sample. The degassing process solves the mentioned problems.

Figure 2.3 shows the placement of the prepared mixture in the vacuum chamber to be degassed. Thirty minutes is enough to remove the air bubbles. However, the viscosity of the liquid plays a key role in determining the required time. To eliminate the bubbles thoroughly, the fluid left in the chamber at least for 2 hours.

Figure 2.3 Degassing process.

Casting the Master: The silicon master is illustrated in Figure 2.4. As shown in Figure 2.5, the master is washed with the mixture of detergent, Alcohol and deionized water to reduce the adhesion of the master to PDMS. In order to remove the residues from the master, it should be rinsed with deionized water one more time. A specified amount of PDMS is casted over the master in order to obtain the required thickness. The prepared liquid fills the existing channels in the master.

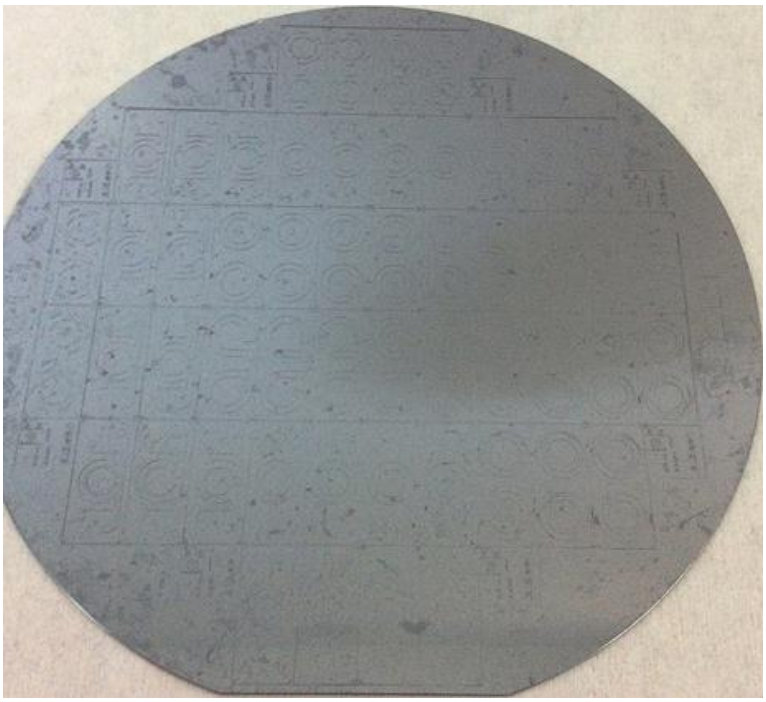


Figure 2.4 Silicon master.

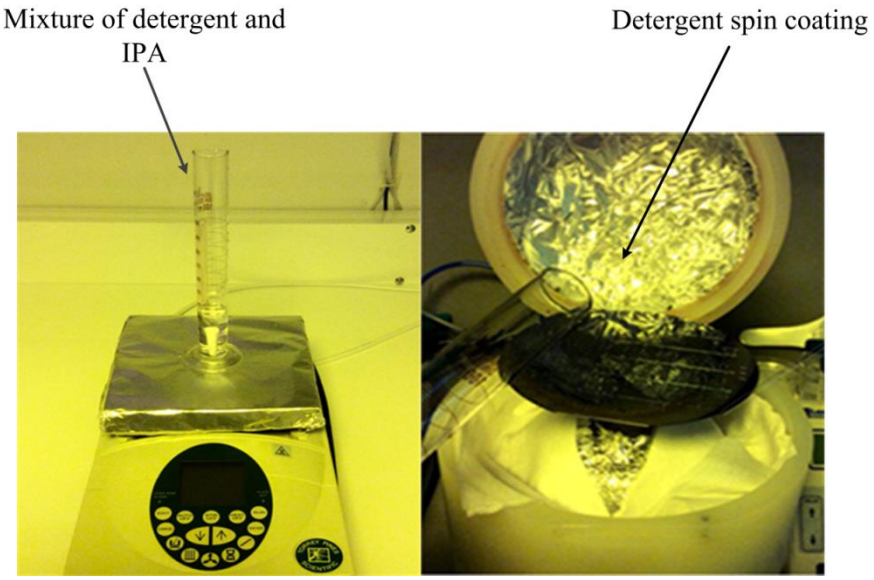


Figure 2.5 (a) Preparing detergent, (b) Spin coating of the detergent.

PDMS Curing: In order to reduce the curing duration, the prepared PDMS can be heated in an oven at 150°C for 10 minutes shown in Figure 2.6. However, it is better to cure the PDMS in the room temperature for 24 hours to achieve high quality samples.

Figure 2.6 Curing the PDMS in the oven.

Separation of the PDMS from the Master: In some critical cases, vacuum can be used for separation of the PDMS from the master. In this way, PDMS is separated thoroughly without any problem. However, in this study, the PDMS is released from the master by the hand but the applied force should be controlled to prevent detriments to the master.

2.6 Process Optimization

During the PDMS preparation procedure, some variables could be changed. This variation results in some changes in the quality and the operation speed.

Mixing Ratio: It is generally suggested to use 10:1 mixing ratio for the prepolymer and curing agent respectively. There is a considerable change in the characteristics of the cured PDMS due to the variation in the mixing ratio. Sometimes, there is a need for thin layers of PDMS in different applications. In order to achieve these layers, spin coating process can be operated. In an identical spin coating procedure, due to the different mixing ratio, various layer thicknesses are achieved. In order to obtain thinner layers of PDMS, larger amount of curing agent should be added to the material.

One of the most important characteristics of PDMS is the final tensile stress of the cured PDMS. According to the experiments, the tensile stress quantity is in a close relationship with the mixing ratio. By increasing the amount of curing agent, tensile stress quantity is enhanced. According to the experimental results, the highest tensile strength can be achieved in the mixing ratio of 10:1 [58].

2.7 Direct Bonding of Different Materials

In the direct bonding, two layers of the same material or different material can be bonded to each other. A diversity of materials such as glass, silicon, polymers, ceramics and metals can be bonded to each other directly. PDMS is a polymer with low surface energy, which can be bonded to the other layers by the utilization of plasma activation. The density of hydroxyl groups will be increased during the oxidation process and strong molecular bonds will be constructed as a result. Since, the created bonds in this technique is inalterable, it is generally used in microfluidic tools.

2.8 Surface Wettability of PDMS under Oxygen Plasma Treatment

The plasma treated PDMS layer has two significant privileges: Firstly, the plasma treatment makes the PDMS micro channels hydrophilic and provides the opportunity for fluids to fill the channel rapidly.

Secondly, the plasma treated PDMS layers can bond irretrievably to the variety of surfaces including glass, silicon, silicon dioxide, quartz, silicon nitride [40]. Plasma treated PDMS is able to bond to other plasma treated PDMS surface.

Plasma can be produced from variety of sources. Two main methods used in this area are Oxygen Plasma and Corona Plasma. In the Oxygen Plasma System, the gas is excited by the utilization of Radio Frequency (RF) mechanism.

The most important factors, which affect the condensation of ion are intensity of applied electric field, pressure and existing impurities. The real plasma ambient strongly pertains to the plasma chemistry [59].

The oxygen plasma greatly affects the Si atoms. The PDMS layer is oxidized by the transformation of – O Si $(CH_3)_2O$- groups to – $O_nSi\ (OH)_{4-n}$ groups[59], [60].

According to the literature, even if the samples were heated before the plasma treatment there is no improvement in the bonding quality [40]. It becomes clear that high congestion of -OH groups on the PDMS layer can be excited because of oxygen plasma. The main source of -OH formation is mainly from a $–CH_2OH$ composition rather than –SiOH. Therefore, powerful intermolecular bonds will be created [61], [62]. Due to the polarity of silanol groups, they develop the hydrophilic layer and this can be perceived by a pertaining change in the contact angle of deionized water [46]. The created silanol groups can compress with the ones on another layer in a tangent contact. The result of the mentioned reactions is the development of Si-O-Si bonds after the absence of a water molecule. There is no common principle, for adjusting a set of desirable plasma parameter to

achieve an adequate amount of bonding strength. Consequently, the optimization of plasma bonding processes is necessary during the micro fabrication process.

After the plasma treatment, the samples are heated in the pursuit of bonding stabilization. Heating a hydroxylated sample for a long time cause a reduction in the multiplicity of the -OH bands. The decline in this band multiplicity is the consequence of the possible omission of water through -OH group concentration.

Because of the number of different possible configurations for hydrogen bonding in the siloxane structure, a distribution of hydrogen bond strengths is expected, which will broaden the absorption bands considerably.

According to the photochemical disintegration of PDMS, ultraviolet beams constituted -$SiCH_2Si$- or -$SiCH_2CH_2Si$- components based on the radiation wavelength [41].

The measurement of the bonding strength is an important issue in the field of micro fabrication. For this purpose, the contact angle of glass and PDMS layers should be measured. The plasma exposure treatment can be regulated by changing some items such as exposure duration, applied power and chamber pressure. The measured contact angle of glass and PDMS surfaces is in a close relationship with the bonding strength.

2.9 Chemical Treatment of Glass and PDMS

Before the plasma treatment, the glass and PDMS layers should be treated chemically. There are various techniques used for the chemical treatment of the samples. For example, for PDMS-Glass bonding, the glass samples are rinsed in boiling piranha solution (5:1 ratio of concentrated H_2SO_4 and 30% H_2O_2 solution) for 5 minutes and then washed in DI water [40] shown in

Table 2.1. However, the easiest method is to immerse the glass samples in an acetone for about twenty minutes. The next step is to rinse the samples by IPA and DI water and use compressed air to dry the surface [66].

2.10 Optimization of Plasma Treatment

There are three important factors, which affect the bonding strength of PDMS-PDMS and PDMS-Glass samples:

- Chamber Pressure.
- Oxygen Plasma Power.
- Exposure Time.

Chamber Pressure: In the pressure higher than (100 mtorr), there is a slow progress in the bond strength with an increase in the pressure [8]. The plasma becomes isotropic because of the reduction in the free path of the gas molecules. Due to the increment in the pressure, the charged particles shifted toward the substrate and the momentum of the ions will be decreased [67]. The Oxygen molecules with a lower momentum can eliminate the methyl groups from the surface without harming the siloxane component. However, in the lower pressures the contact angle of PDMS increases quicker than glass samples. That is because of the hard structure of the glass samples. Therefore, the higher momentum can cause a change in the chemical structure.

Oxygen Plasma Power: The effect of oxygen plasma power can be studied based on the result investigation of diverse bias levels directed by oxygen plasma power. At low powers, the kinetic energy of ions will also decrease [42]. The reduction of oxygen plasma power and chamber pressure leads to the decline for ions on the surface. In addition, the decline in the electron acceleration results in a decline in the particle congestion. As a result, Si-OH group will be decreased in the surface and the ions prefer to stay at the upper layer without causing much chemical or physical transformation. However, in the higher powers, the Si-O-Si will be damaged.

Time of Exposure: After the plasma exposure, the fragile layer of silica will be created on the surface. According to the literature, to achieve high quality silica layer, lower RF power and shorter treatment times should be applied on the surface of the samples. The longer exposure duration will cause a reorientation of chemical bonds [68].

As a conclusion, in order to convert the hydrophobic feature of PDMS to hydrophilic one, oxygen plasma treatment can be utilized under specific criteria. Utilization of the oxygen plasma with a lower RF power and short exposure duration creates a narrow layer of oxide on the PDMS layer, which leads to the adhesion of two layers.

Two different oxygen plasmas were used during this thesis.

Table 2.1 shows the optimal bonding condition in old plasma tool. Table 2.2 shows the optimal bonding condition in Diener Zepto plasma tool. The optimal results in two different oxygen plasma tools show that lower plasma powers in the range of (20 W) and lower exposure time provide higher bonding strength between PDMS and glass samples.

Table 2.2 shows the optimal condition for the plasma treatment in the Diener Zepto oxygen plasma tool: Oxygen flow rate (26.6 sccm), RF power (20 W) and exposure time (20 s).

Usually, high quality bonding can be achieved under low level RF power and short exposure time. After the plasma treatment, the samples are placed on the heater at the temperature of 110°C for at least 10 minutes. This process enhances the bonding strength. The set of experiments were done for the optimization of PDMS-Glass bonding process. The optimization of plasma treatment process was done on the PDMS layers which are designed for the fabrication of transmitarrays [76].

Table 2.1 Optimization of plasma treatment

Sample #	PDMS layout	Power (W)	Pressure (Torr)	Process Time (s)	With/without piranha cleaning	Bonding quality
1		40	0.8	30	With	Good
2		50	0.7	30	With	Good
3		50	0.7	30	With	Bad
4		50	0.7	30	With	Bad
5		40	0.8	30	With	Good
6		30	0.8	30	With	Good
7		30	0.8	30	With	Good

Table 2.1 (Continued)

8		30	0.9	30	Without	Not good
9		30	0.9	20	Without	Good
10		30	0.7	30	Without	Good
11		30	1	30	Without	Good
12		40	1	30	Without	Good
13		30	0.8	30	Without	Bad
14		30	0.7	30	Without	Bad
15		20	1	20	With	**Very Good**

Table 2.2 Optimization of plasma treatment in Diener Zepto plasma tool

Sample #	PDMS layout	Power (W)	Duration of exposure (s)	Oxygen flow rate (sccm)	Bonding quality
1		80	20	26.6	Bad
2		80	15	26.6	Bad
3		70	20	26.6	Bad
4		70	15	26.6	Bad
5		50	20	26.6	Bad
6		50	15	26.6	Bad
7		40	20	26.6	Bad
8		40	15	26.6	Bad

Table 2.2 (Continued)

9		30	20	26.6	Bad
10		30	15	26.6	Bad
11		20	20	26.6	**Very Good**
12		15	20	26.6	Good
13		15	15	26.6	Good

2.11 Novel method for Bonding of Metallized Glass to PDMS

As mentioned before, PDMS obtains a special place in microfluidics field. It can offer lower cost, easier fabrication techniques, biocompatibility and good thermal and chemical stability. However, there are some restrictions associated with PDMS material. The low surface strength of PDMS creates a difficult condition for a direct metallization required for electrical and electrochemical utilizations. The quality of metal layers directly deposited on the PDMS is inadequate. Instead of using direct metallization approach, another novel method can be used. PDMS can be bonded to the metallized glass pieces using this approach.

In this study, a novel technique is proposed for the bonding of metallized glass samples to the PDMS samples. Irretrievable bonding was accomplished by bringing the two layers in tangency at the room temperature after the plasma treatment.

2.12 Proposed Technique for Bonding of PDMS to Metallized Glass Samples

- ***Preparing the Metallized Glass Samples:*** Before a gold film deposition, a thin layer of Titanium (20nm) must be sputtered on the glass sample to increase the adhesion quality. Then, the gold layer (200nm) is deposited on the glass using sputtering process. Since the plasma treatment cannot provide the possibility of adhesion between PDMS and metallized surfaces, a thin layer of silicon dioxide (50 nm) is deposited on the metal layer using Plasma Enhanced Chemical Vapor Deposition (PECVD) method. Any contamination on the glass slides was removed by immersing them in acetone for 20 minutes. Isopropanol and deionized water are used for washing the chemically treated samples. For the bonding process, oxygen plasma treatment is used.

- ***Plasma Treatment:*** Exposure was done in an inductively coupled high density (ICP) plasma system. Optimal experiment is done at the following case: Oxygen flow rate (26.6 sccm), RF power (20 W) and Exposure time (20 seconds). Usually, high quality bonding can be achieved under low level RF power and short exposure time. After plasma treatment, the samples are placed on the heater at the temperature of 110 $^{\circ}$C for at least 10 minutes. This process enhances the bonding strength.

The mentioned method is applicable in the fabrication of reconfigurable transmit arrays. In this structure, the exact adhesion of the metallized glass to the PDMS layer is required because the gold layer has a special pattern. The detailed explanations are in the following contents.

2.13 Bond Strength Test

The former and easy way for bonding strength measurement, is the utilization of a pointed knife. It is used to disassemble a sample and evaluate the required force. Different kinds of instrument are employed for the measurement of bonding strength in quantitative methods. The most applicable testing methods are peel test [69], blister tests [44], tensile test [70], [71], three-point bending test [72] and razor blade test [73].

Blister Test: The significant properties of the blister test are as follow:

- Extensive diversity of substances can be measured by this method.
- Exact results can be obtained from this testing method.

The master key in this method is to inject a liquid between two layers until the top layer separated in form of a blister. The amount of applied pressure and injected liquid can specify the amount of required work to detach the film. The important issue is to create a blister with desirable form and size. Rectangular blisters grow along the assigned region and represent more benefits in comparison to round ones. An access hole is required in the design to make the injection feasible.

Two techniques are offered for providing access holes in the prepared set. The easier technique is to use drilling method and improvise a small hole before the coating process. The other proposed method is to use electrolytic etching. In addition, Silicone oil and glycerin is favorable in this test structure. The liquid is pumped slowly inside the sample. During the process, the system should be depleted and then pressure can be applied.

However, for getting better results of bonding strength measurement, peeling test was utilized for the bonding strength measurements of PDMS-Glass and PDMS-Metallized glass samples.

Peeling test: One general method to characterize the bonding strength is to use the peeling test, which is usually employed for the measurement of weak adhesions. Because, in the case of strong adhesion, great peel forces may deform the samples. During the process, the bonding strength can be determined by manual peeling of the layers. Here, the principal privileges of qualitative tests like peel test, are simplicity of their execution. The peel energy between the PDMS layer and the glass sample was measured with a 90° peel test using a universal testing machine Instron 5969 MTS shown in Figure 2.7.

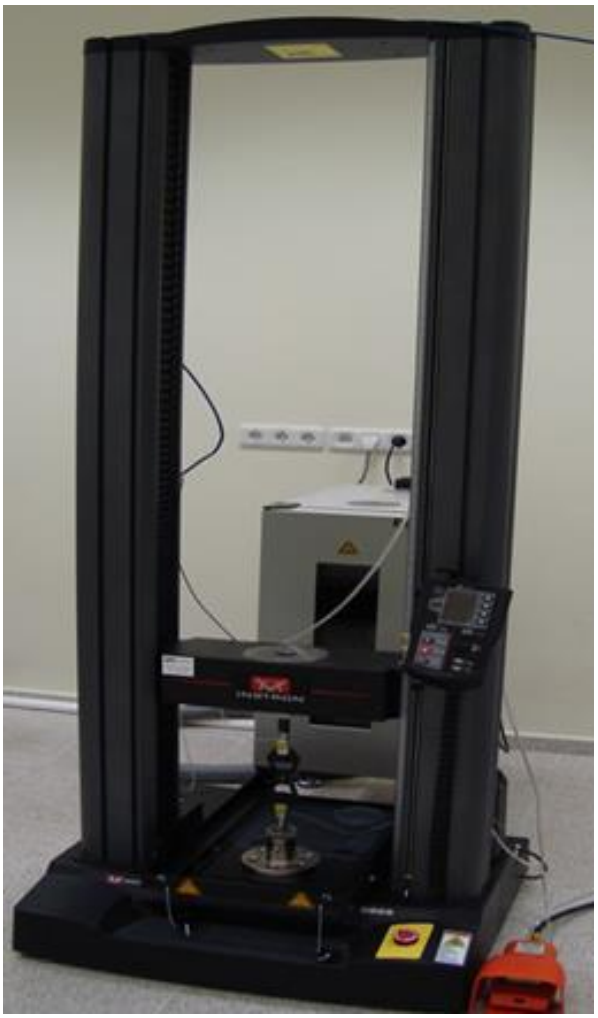


Figure 2.7 Micromechanical tester.

Peel strength is the mean load per unit width involved in the separation of bonded layers where the angle of separation is 90 degrees as illustrated in Figure 2.8. Five specimens were tested for each adhesion study at a peel rate of 10 mm/min.

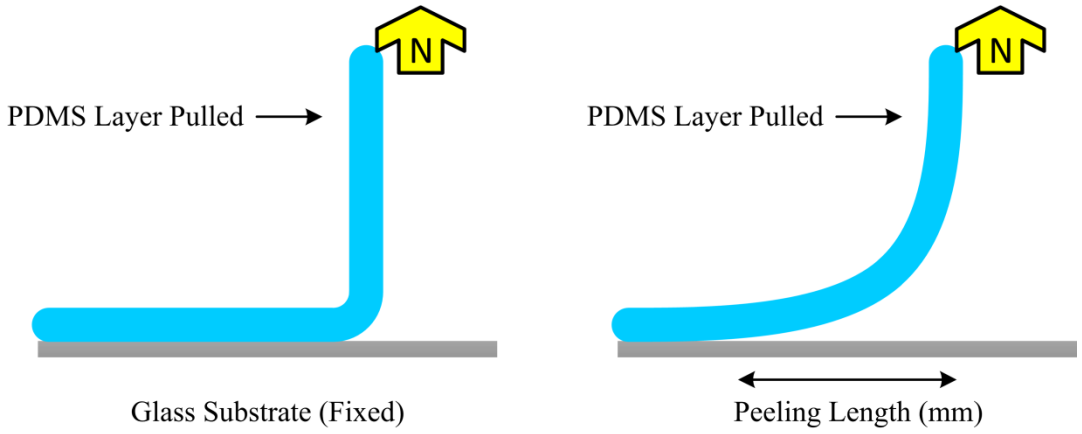


Figure 2.8 Peel test measurement process.

Figure 2.9 demonstrates the samples prepared for the peeling test measurement. The dimensions of the samples are shown in Table 2.3.

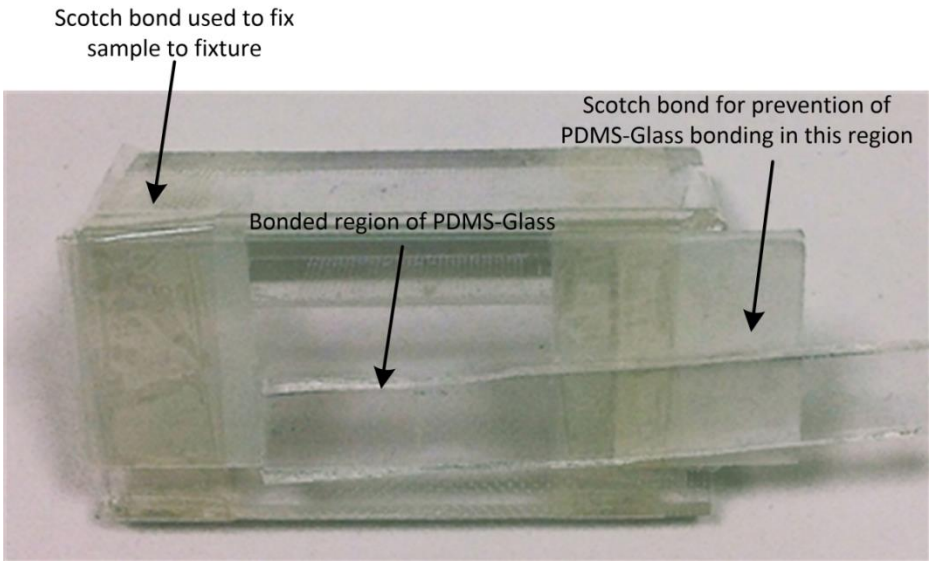


Figure 2.9 Prepared sample for peel test.

The process is initiated by locating the glass part to the bottom gripper of the micromechanical tester as shown in Figure 2.10.

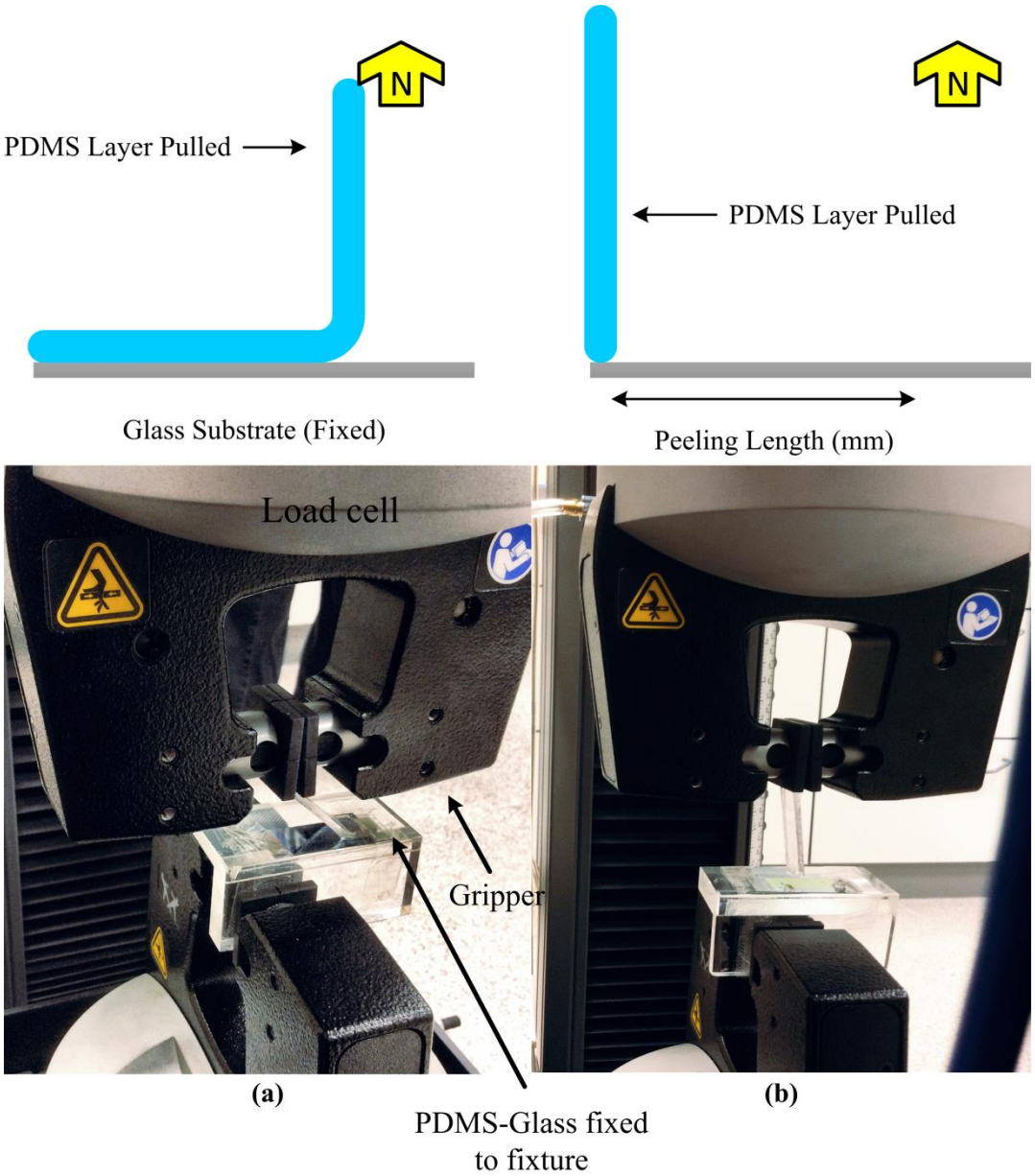


Figure 2.10 (a) The load applies force to rebond the sample, (b) The end of the peeling process.

The PDMS part is placed inside the moving gripper. Some samples have a considerable bonding strength in which, PDMS layers are ruptured before the peeling test as shown in Figure 2.11.

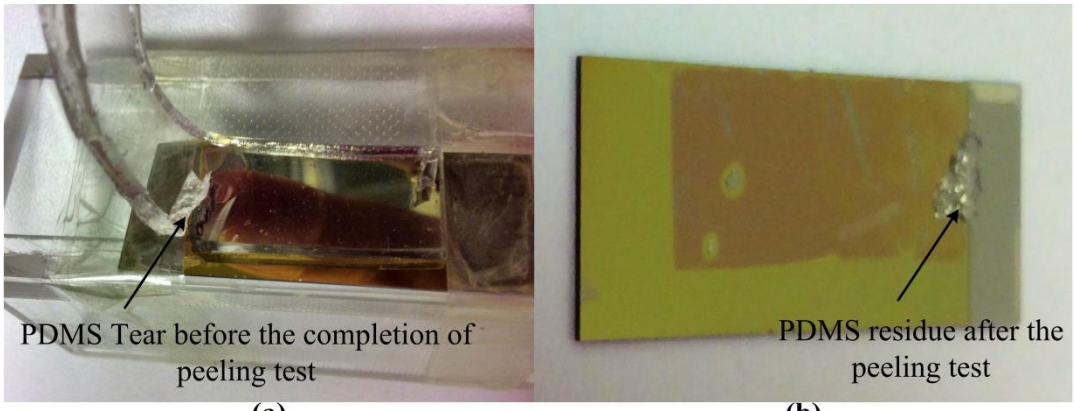


Figure 2.11 (a) PDMS was teared before the completion of the process, (b) Peeling test was done successfully.

During the measurement, 2 kN load cell is connected to the moving gripper. After the beginning of the debonding, the displacement of the moving gripper is saved as the peel length [74]. The measurement plot of force versus the peel length is demonstrated in Figure 2.12. The measurement results shows that PDMS-Metallized glass with silicon dioxide layer has better bonding quality than PDMS-glass layers. Deposition of thicker silicon dioxide layer on metallized glass samples results in better bonding quality.

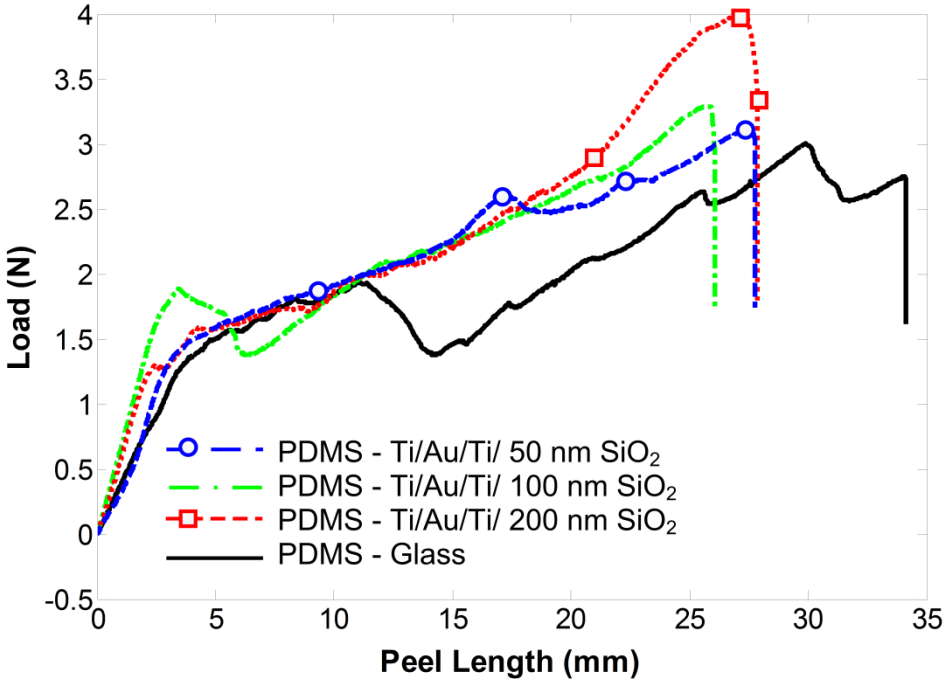


Figure 2.12 Plot of force *vs.* peel length

During the process, the effect of silicon dioxide thickness on the bonding strength is examined. Four categories of samples were provided; each of them included 5 samples (samples with glass/ Ti-Au-Ti/ 50 nm silicon dioxide- PDMS, glass/ Ti-Au-Ti/ 100 nm silicon dioxide- PDMS, Ti-Au-Ti/ 200 nm silicon dioxide- PDMS and samples with glass- PDMS layers). 20 nm/600 nm/20 nm-thick Ti/Au/Ti stack, is sputtered on a glass substrate. Then silicon dioxide (SiO_2) is deposited on the metal layer using Plasma Enhanced Chemical Vapor Deposition (PECVD) method. The metallized glass samples are cleaned by acetone, isopropanol and DI water, respectively. The samples are prepared by using plasma treatment method under the condition as follow: (1) In order to reduce the probability of PDMS layers rupture problems, they are prepared by mixing ratio of 10:1. (2) PDMS and Glass samples are treated under the power of 20 W for 20 s. The dimensions of prepared samples are tabulated in Table 2.3.

Table 2.3 Dimensions of samples measured in the peel test

Specimen label	**Length (mm)**	**Thickness (mm)**	**Width (mm)**	**Area ($mm^{2)}$**
PDMS-Glass-1	3.75	2.79	10.4	29.01
PDMS-Glass-2	3.5	3.1	9.6	33.6
PDMS-Glass-3	2.5	3.13	11.18	27.95
PDMS-Glass-4	2.26	3.1	10.3	32.2
PDMS-Glass-5	2.48	2.8	9.72	27.99
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-1	2.69	2	10.31	20.62
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-2	1.02	3.13	11.06	29.01
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-3	2.47	2.52	9.11	22.95
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-4	2.56	2.92	9.28	27.09
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-5	2.68	2.9	10.73	31.11
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-1	1.97	2.6	11.02	28.65
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-2	3.3	2.89	10.66	30.80
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-3	3	2.7	10.89	32.67
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-4	2.5	3.1	11.01	27.52
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-5	2.71	2.9	10.69	28.96
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-1	5.1	3.01	11.1	33.41
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-2	5.1	3.01	11.1	33.41
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-3	5	2.92	11.99	35.01
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-4	5.2	2.96	11.76	35.21
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-5	5.1	2.94	11.8	35.05

Table 2.4 Peeling test measurement results

Specimen label	**Load at Break (Standard) (N)**	**Tensile stress at Maximum Load (MPa)**
PDMS-Glass-1	2.48	0.10365
PDMS-Glass-2	2.09	0.085
PDMS-Glass-3	1.7	0.05
PDMS-Glass-4	1.8	0.062
PDMS-Glass-5	2.23	0.095
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-1	2.68	0.11202
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-2	2.89	0.12101
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-3	2.47	0.1034
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-4	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-50nm SiO_2-5	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-1	2.70	0.11491
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-2	2.58	0.1098
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-3	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-4	2.82	0.12001
PDMS-Glass-Ti/Au/Ti-100nm SiO_2-5	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-1	2.79	0.11944
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-2	2.7	0.1155
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-3	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-4	PDMS ruptured	PDMS ruptured
PDMS-Glass-Ti/Au/Ti-200nm SiO_2-5	2.88	0.1232

2.14 Conclusion

This chapter provided a brief introduction regarding the PDMS and its fabrication process. Oxygen plasma tool is employed for bonding of PDMS to glass slides. The parameters of oxygen plasma treatment are optimized in order to achieve high quality bonding. Novel method is proposed to solve the challenges exist in direct metallization to PDMS layer. A thin layer of silicon dioxide layer is deposited on the metallized glass samples using PECVD method. With the proposed method, high quality bonding of PDMS to metallized glass samples is provided. Peeling test method is exploited to measure the bonding strength between PDMS and metallized glass samples. In the next chapter, the fabrication of transmitarrays will be discussed.

CHAPTER 3

FABRICATION OF TRANSMITARRAYS

This chapter presents a brief introduction to transmitarrays. Element rotation method is applied in the structure of unit cells in the microfluidic transmitarrays[1]. The fabrication process of the unit cells is also explained in this chapter.

3.1 Introduction to Transmitarrays

There is a high demand for high gain and reconfigurable antennas in various domains such as wireless communication systems, adaptive antenna arrays and satellite communications. Different techniques are applied for the improvement of the wireless communication system. In modern technologies, multi-path transmission lines are utilized between the receivers and transmitters for delivery of the same data. However, the feeding system of each line is different. Variety of modern technologies can be offered based on the type of transmission paths: Space diversity method, polarization diversity system, frequency diversity system, time diversity system and angle diversity system. The method used in transmit arrays, is angle diversity system.

[1] The design of the transmitarray is completed by Dr. Emre Erdil in the frame of his Ph.D. dissertation [30].

- ***Angle diversity system:*** This system localizes signals with variety of angles in a single point. The theory of this method can be achieved by the wave radiation based on the phase shifting function. To arrange phase variation in the systems, different methods are introduced such as:

1. Changing the lengths of the delay lines (transmission line, stub, strip line) is a common method in this technology. However, parasitic radiation is created during this process.
2. Radiators can be embedded inside the unit cells.
3. Phase shifters can generate phase variation.
4. Element rotation method can be employed in the array.
5. MEMS switches, PIN diodes and varactors are employed to create a phase variation. However, there are some disadvantages associated with this method such as lack of continuous tunability, high power consumption, fatigue, contact degradation and dielectric charging.

In the structure of a transmitarray, a feed antenna is utilized for generation of spherical waves illuminating the array. Consequently, the radiation is transferred to the other side as shown in Figure 3.1. The phase of transmit arrays should be adjusted to obtain the radiated waves in an appropriate direction.

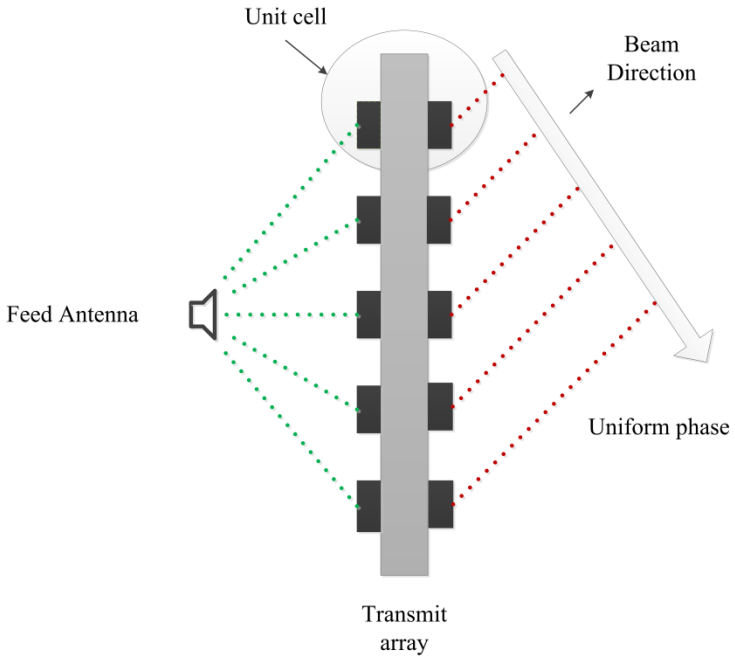
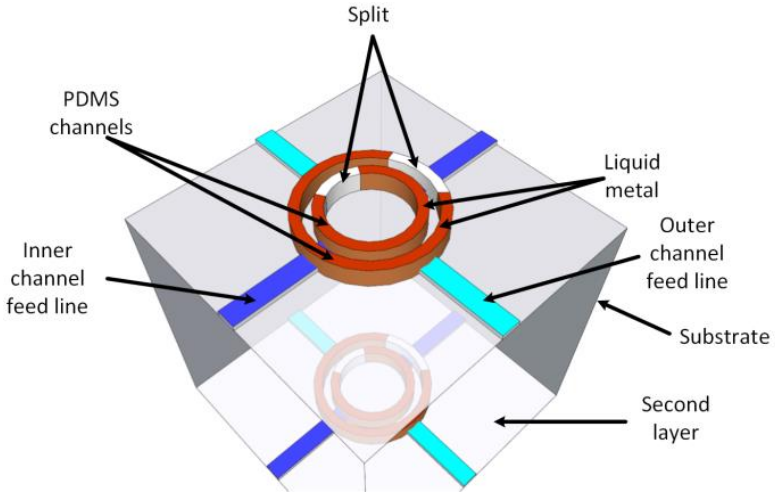
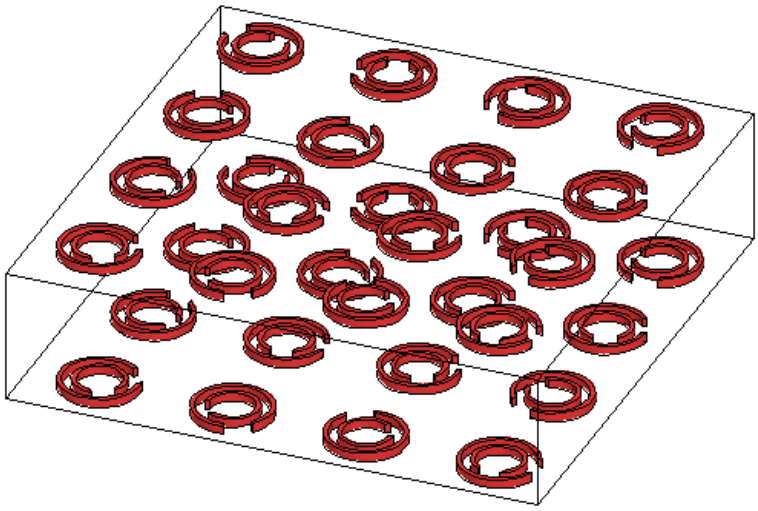


Figure 3.1 Schematic view of Transmit array

Transmitarrays have many applications in variety of areas such as satellite communications and radars. The transmitarray unit cells fabricated in this study, adjust their transmitted phase by employment of microfluidics and the element rotation technique. Figure 3.2 (a) demonstrates a scheme of a microfluidic split ring unit cell structure. Element rotation method is conducted by rotation of the liquid metal inside the microfluidic channel, which offers phase shifting method. Figure 3.2 (b) shows an illustration of a typical transmitarray consisting of microfluidic split ring unit cells.



(a)



(b)

Figure 3.2 (a) Microfluidic split ring unit cell utilized in the element rotation technique, (b) Transmitarray composed of the unit cells in (a) [30].

In this study, rotational phase shift method is used to govern the condition of phases for each element. There are various methods proposed for the element rotation.

- Employing a motor for each element of the array. However, it may cause devastation in the RF efficiency of the transmit arrays.
- Utilization of RF MEMS switches on a split ring structure. However, continuous beam steering is not practical in this method.

A novel method is proposed for the element rotation exertion. In this technique, a split ring resonator is used due to its circular shape. In this design, a liquid metal channel is implemented inside the device by the means of microfluidic methods. This method provides the opportunity to access continuous tuning of the transmitted phase.

In the proposed nested ring-split ring, liquid metal is injected inside the channel, which is created by the bonding of glass substrate to PDMS. Inside the channels, there are two different regions: (1) The liquid metal and (2) The split region, which is due to the existence of air gap. Element rotation is a result of liquid metal position shifting inside the channel as shown in Figure 3.3. Fabrication process of the transmitarray unit cell is demonstrated in the following section.

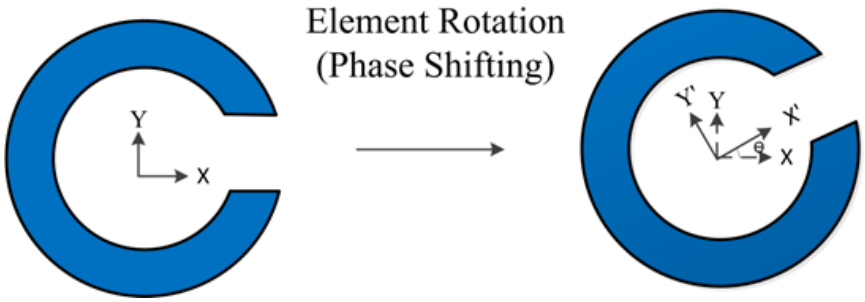


Figure 3.3 Element rotation method, which is employed in unit cells

3.2 Fabrication Method

There are different steps involved in the fabrication of the microfluidic transmit array unit cell. The main part of unit cells are microfluidic channels, which can be created by utilization of soft lithography methods. The microfluidic channels are sandwiched between PDMS and glass layers. During the process, silicon molds are used to form PDMS layer. Silicon molds are fabricated using the DRIE Etching process. The mask layout required for fabrication of silicon mold is shown in Figure 3.4.

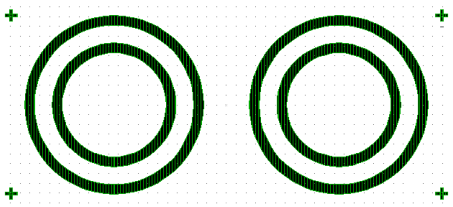


Figure 3.4 Mask layout for preparation of silicon mold

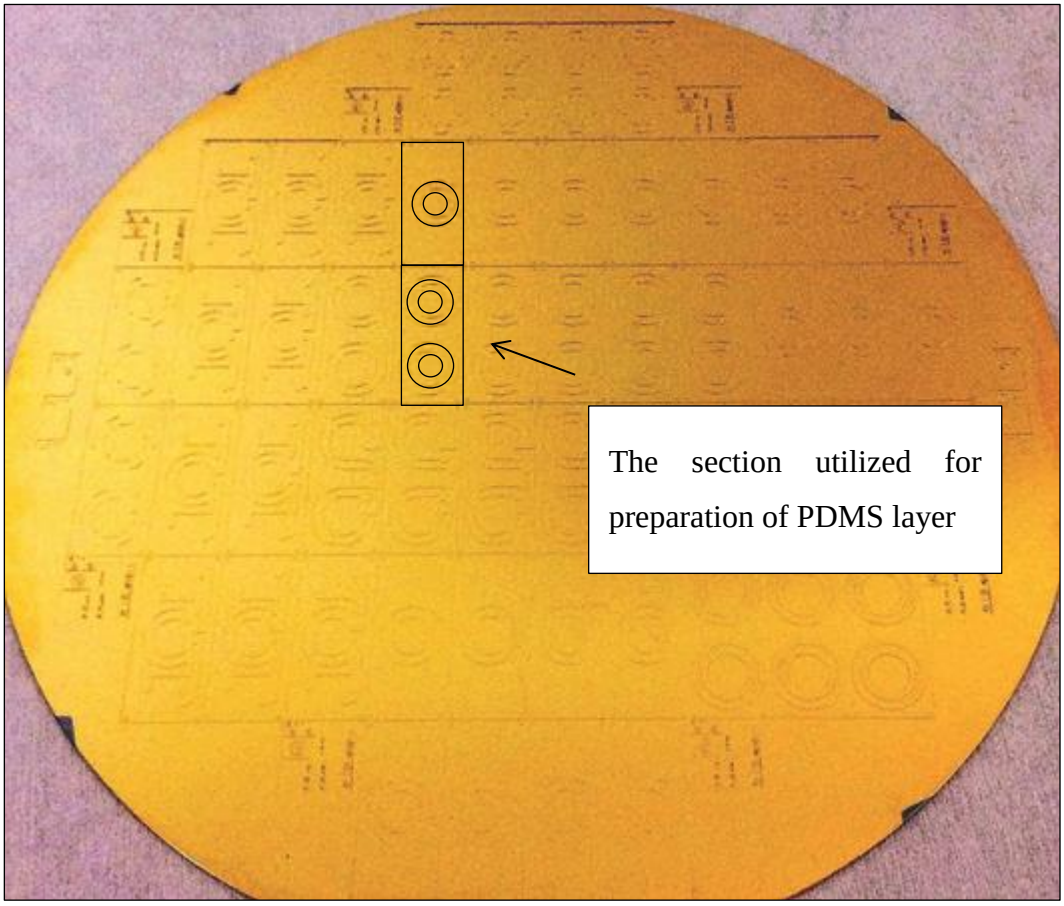


Figure 3.5 Ti/Au layer deposited mold wafer with DRIE depth of 200 μm

The next step in the process is to prepare PDMS. The prepolymer material and the curing agent should be mixed in the ratio of 10:1. The prepared PDMS is poured on the mold wafer and cured at room temperature. There is difficulty in separating the PDMS layer from the silicon layer. There is couple of solutions for this problem: (1) Detergent is spin coated on a silicon mold, (2) A thin layer of Ti/Au is deposited on the silicon mold as shown in Figure 3.5. Both of the stated solutions are applicable in the experiments. However, the second solution is more reliable then the first one.

The main part of the process is related to the bonding of PDMS samples to glass pieces. Glass samples are rinsed with acetone, IPA and deionized water

respectively. The bonding of PDMS-glass layers is done under the oxygen plasma treatment as shown in Figure 3.6. In order to achieve high quality bonding, the parameters should be adjusted as follow:

a. The RF power of the oxygen plasma is adjusted to 20 W.
b. The duration of the plasma treatment is about 20 seconds.
c. The bonded samples are placed on a hot plate at 110°C for 10 minutes.

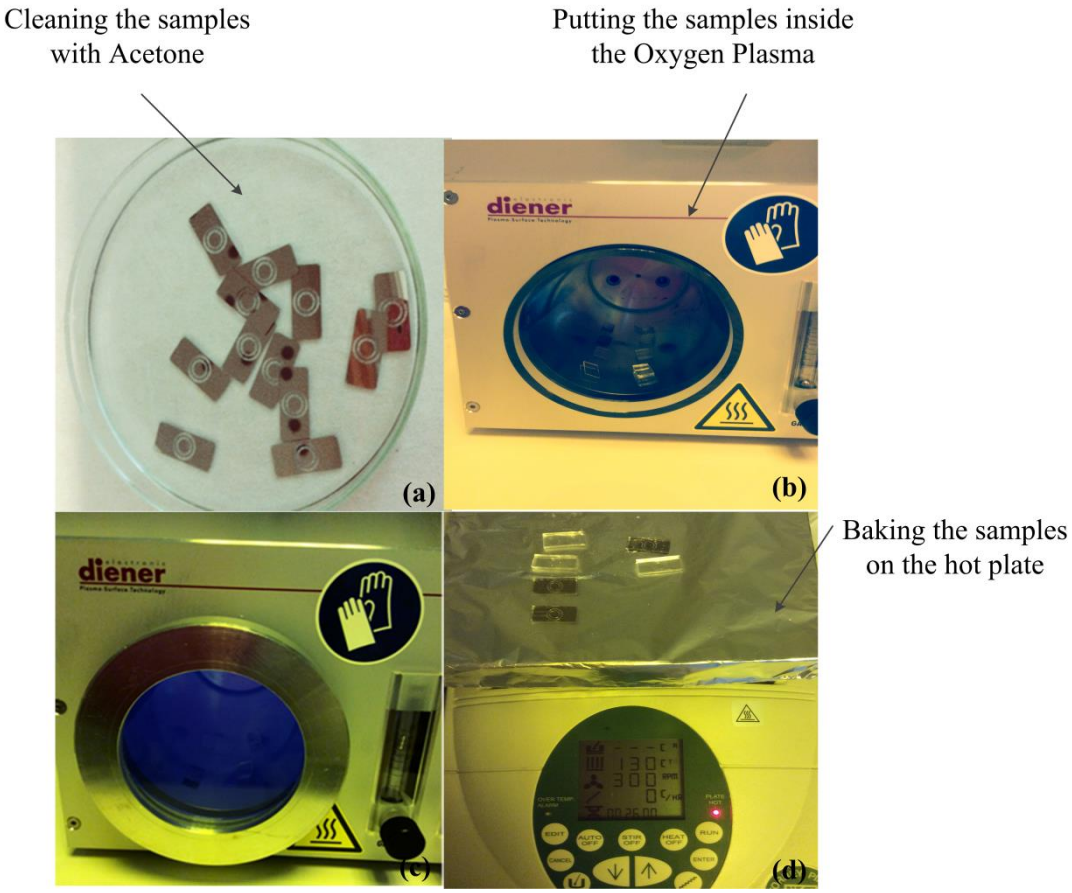


Figure 3.6 (a) Cleaning the glass pieces with Acetone, IPA and DI water , (b) Placing the samples inside the Oxygen Plasma, (c) Plasma treatment of the samples and (d) Placing the samples on the hot plate

The steps of the fabrication process of the microfluidic transmitarray unit cell are demonstrated in Figure 3.7. The microfluidic channels are appeared by pouring the uncured PDMS on a DRIE-etched silicon mold wafer. After PDMS curing, PDMS pieces are bonded to a glass pieces, which are rinsed in aceton, IPA, and DI water respectively. The oxygen plasma treatment is done for a duration of 20 seconds at an RF power of 20 W.

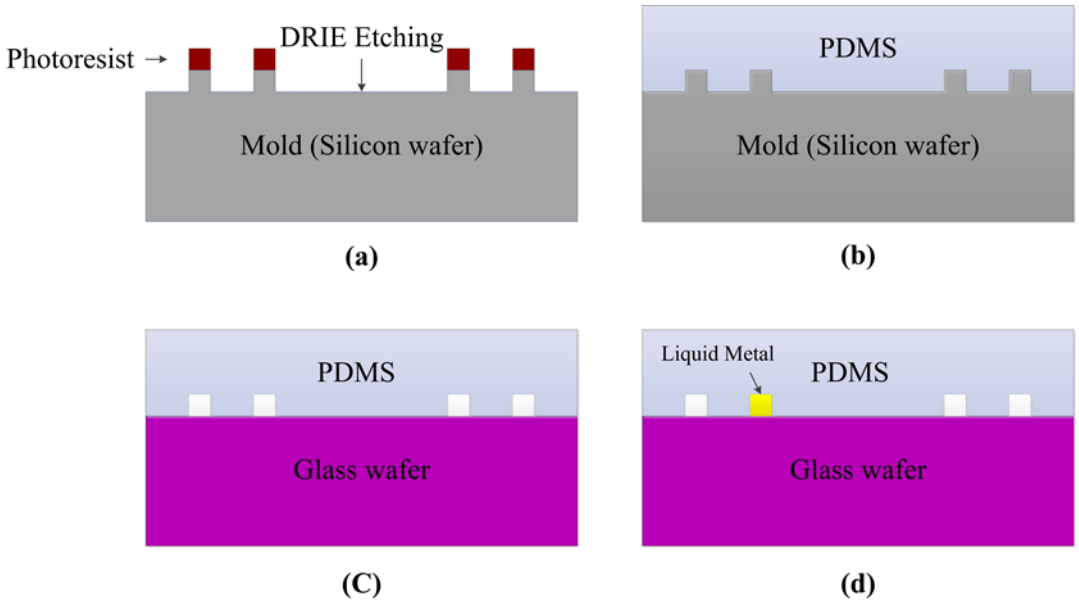


Figure 3.7 Fabrication steps of unit cell transmit array

Figure 3.8 displays the process of liquid metal injection inside the microfluidic channels.

Figure 3.8 Injection of liquid metal inside the microchannels

The final structure of unit cell transmitarray is demonstrated in Figure 3.9. In order to fabricate double layer structure; some glass slides are placed between two single unit cells as shown in Figure 3.10.

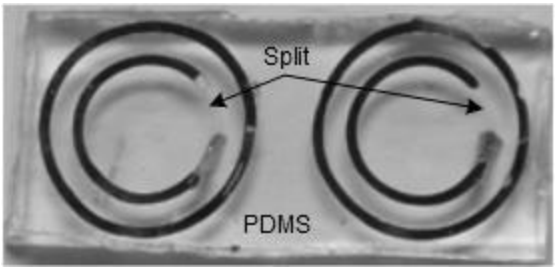


Figure 3.9 Fabricated unit cell, the depth of the channels is 200 μm. The dimensions of are 11.43 mm × 10.16 mm.

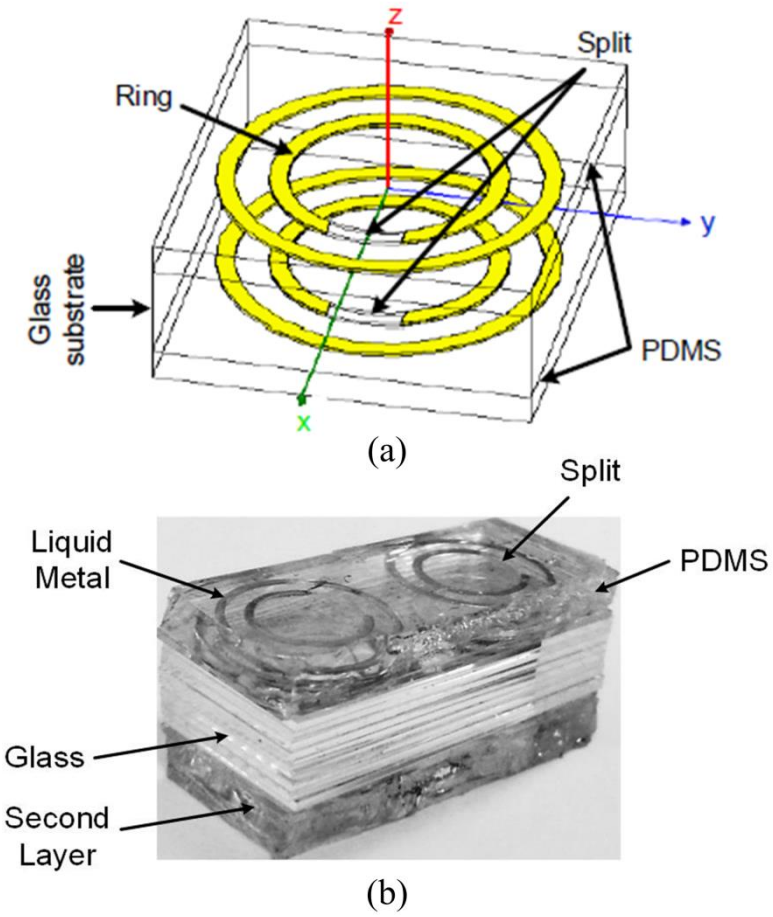


Figure 3.10(a) Schematic view of double layer structure, (b) Fabricated double layer structure [75].

Micropumps can be employed for the rotation of the liquid metal inside the microchannels.

- **Properties of liquid metal:** The liquid metal exploited in the microfluidic channels is Galinstan, which is an alloy of 68.5% Ga, 21.5% In, and 10.0% Sn [76].

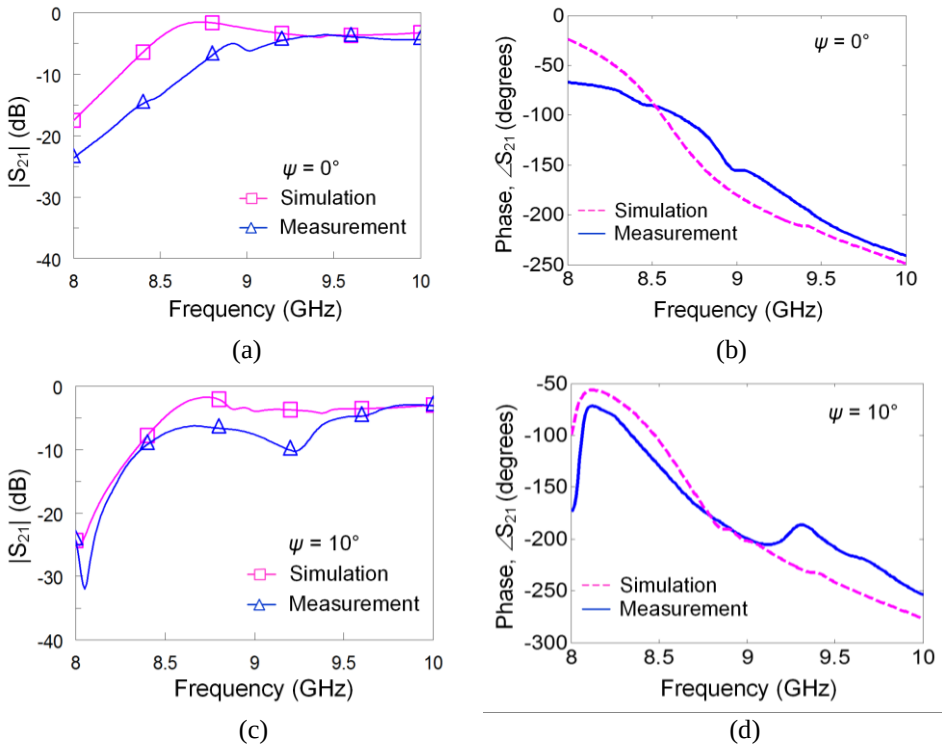
  The special characteristic, which make Galinstan applicable in different areas are as follow:

1) It preserves the liquid state even below 0 ˚C.
2) It is nontoxic.
3) It has low vapor pressure (<10^{-6} Pa at 500 ˚C).

However, the main problem of the Galinstan is that its surface oxidizes immediately in air.

The major privilege of the fabricated unit cell is that there is no requirement for the application of metallic lines, which are indispensible parts of RF MEMS switches and varactors. In the fabricated transmit array unit cell, the location of liquid metal inside the channel can be adjusted by utilization of micro pump.

Figure 3.11 shows the comparison plot between the measurement and simulation results of fabricated transmitarray.



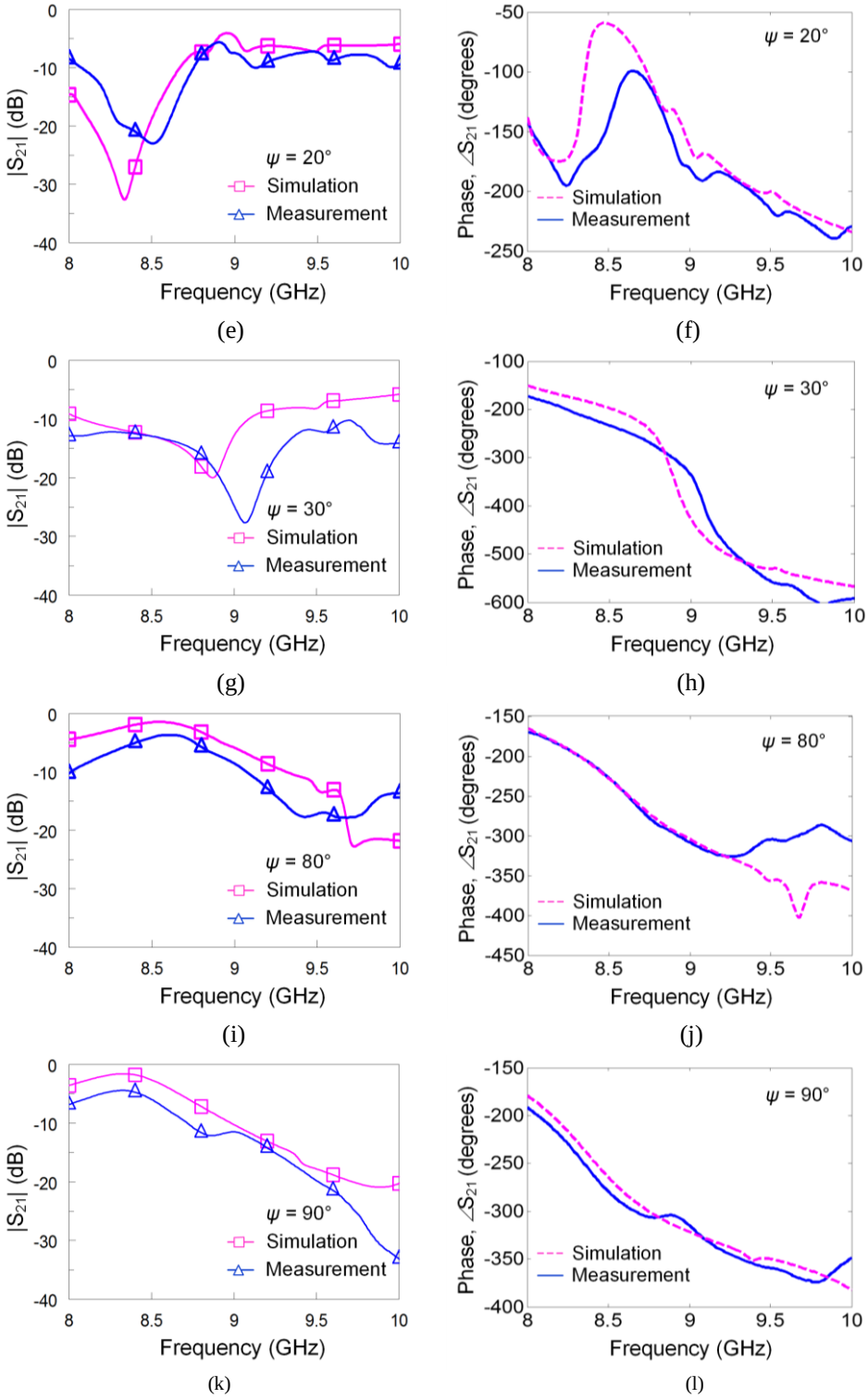


Figure 3.11 (a)-(l) Comparison of the linear polarized measurement and simulation results for the transmission coefficient characteristics and insertion phase of the fabricated double layer transmitarray unit cells rotated at 0° - 10° - 20° - 30° - 80° - 90° [76]

3.3 New design for transmit array unit cell

Some other complementary designs can be employed in the fabrication of unit cells. In the complementary unit cell transmitarray, there is a metal pattern on the glass slices. To create the microchannels, metallized glasses should be bonded to the PDMS samples. However, there are some difficulties in in the fabrication procedure of the proposed unit cell. PDMS cannot adhere to the metal surface easily. Therefore, the surface of the metal is improved.

As mentioned in section 3.2 , the silicon mold is fabricated by applying a DRIE (deep reactive ion etching) PDMS is prepared in the mixing ratio of (1:10) and poured on the silicon mold and cured at room temperature for 24 hours. After peeling off the PDMS layer from the mold wafer PDMS sheets are diced in the same sizes as glass pieces [74].

Figure 3.13 demonstrates a glass substrate covered with 20 nm/600 nm/20 nm-thick Ti/Au/Ti layers by exploitation of photolithography and wet etching processes. Then a thin layer of silicon dioxide is deposited on the metal layer using Plasma Enhanced Chemical Vapor Deposition (PECVD) method.

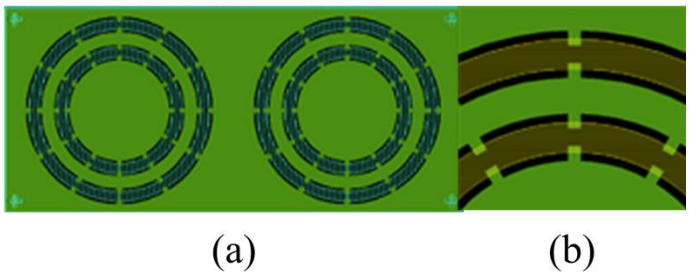


Figure 3.12 (a) Layout drawings of the transmitarray unit cell with metal extensions, (b) close up view of the metal extensions.

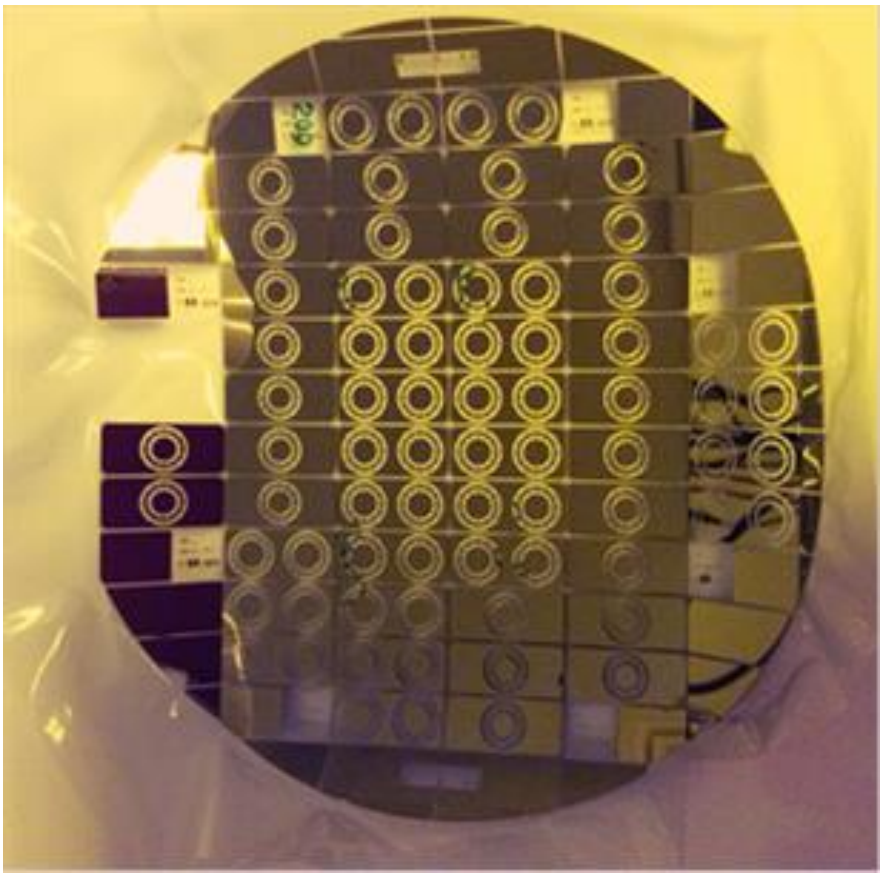


Figure 3.13 Glass samples with metallized pattern

Any contamination on the glass slides can be removed by immersing them in acetone for 30 minutes, followed by isopropanol, and then followed by rinsing in pure water and blow-drying with a N_2 stream. The PDMS slices were rinsed in ethanol and DI water, dried under N_2 stream.

For the bonding process, oxygen plasma treatment is used. Exposure was done in an inductively coupled high-density plasma (ICP) system. Optimal experiment is done in the following case: RF power (20 W), Exposure time (20 s). Usually, high quality bonding can be achieved under low level RF power and short exposure time. The bonded PDMS layer to glass wafer with the patterned antenna metal on it is represented in Figure 3.15.

After plasma treatment, the samples are placed on the heater at the temperature of 110 $^{\circ}$C for at least 10 minutes. This process enhances the bonding strength. The liquid metal droplet is injected into the channel by using injection syringe. The split region is formed after injection.

The circles are appeared as a microfluidic channel inside PDMS. The liquid metal forms the conductive part in the split ring structure. The split region is the vacant area in the channel filled with the liquid metal. The complete process flow is shown in Figure 3.14.

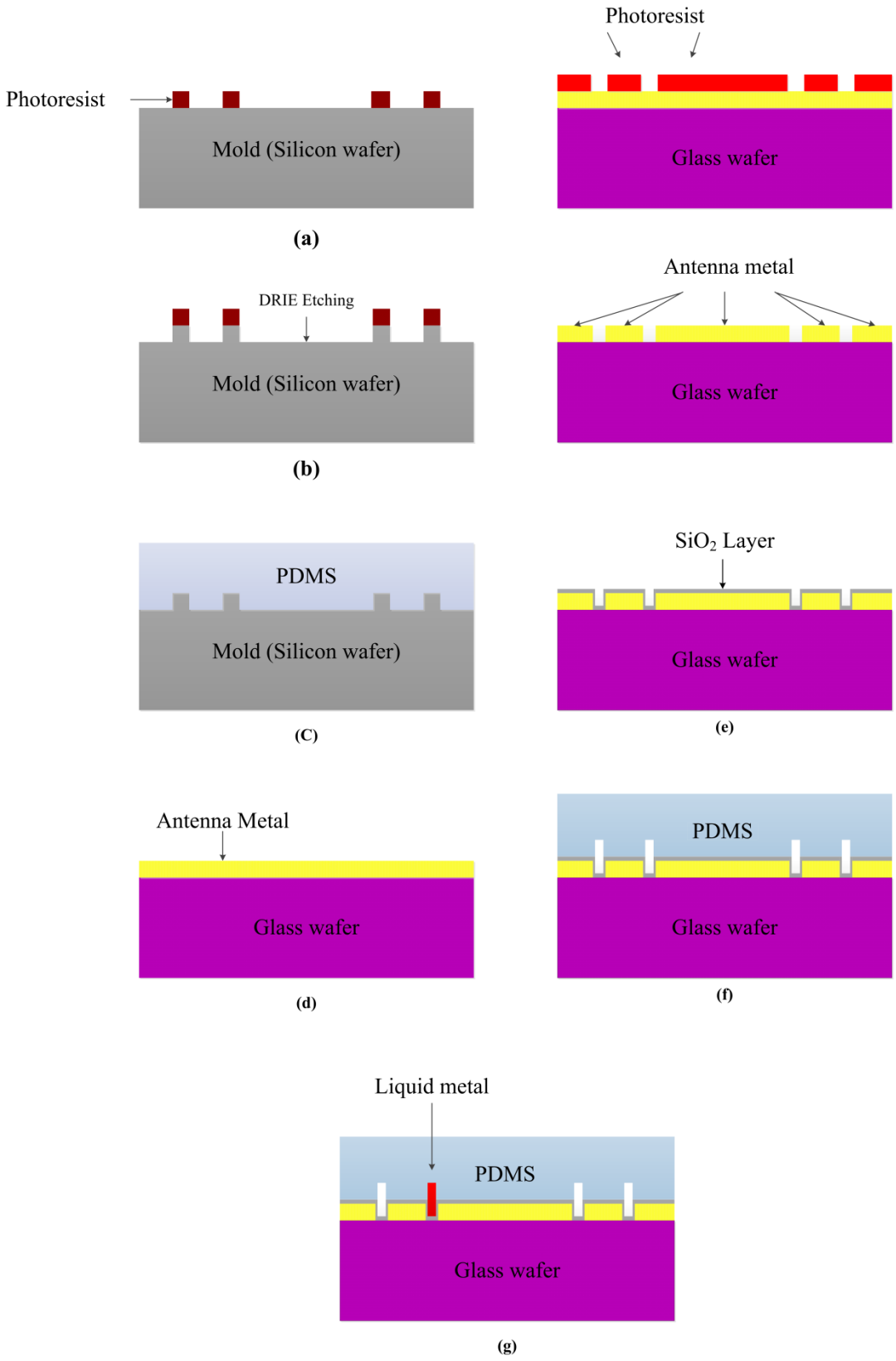


Figure 3.14 Fabrication process flow of metallized pattern unit cell.

The size of the unit cell is 11.43 mm × 10.16 mm, approximately 0.4 λ_0 at 10 GHz. Since the waveguide simulator method [15] is used for the characterization of the fabricated unit cells, this dimension is chosen to ensure that two cascaded unit cells strictly fits into the WR-90 waveguide which has dimension of 22.86 mm × 10.16 mm. The rings are in the form of channels having depth of 0.2 mm. The PDMS layers (ε_r=2.77, tan δ=0.0127) have thicknesses of 1.75 mm.

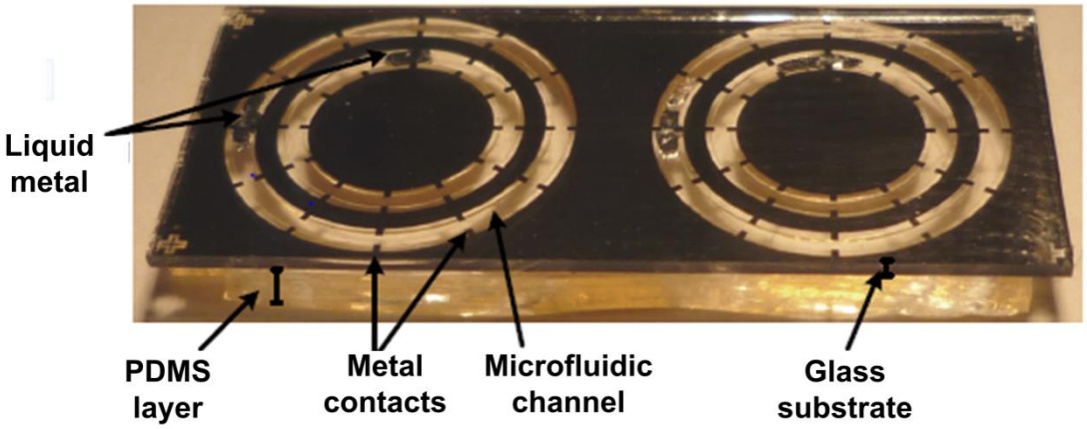


Figure 3.15 Unit cell transmit array with patterned metal.

3.4 Conclusion

This chapter presented a concise introduction to transmitarrays. The advantages of microfluidic based reconfigurable transmitarrays are studied in this chapter The fabrication process of two different microfluidic transmitarray unit cells is also explained in this chapter.

CHAPTER 4

FREQUENCY TUNABLE PATCH ANTENNA WITH LIQUID METAL SWITCHING

The application of liquid metal switch in the structure of frequency-reconfigurable patch antenna is presented in this chapter. The chapter first explains the design procedure involved in creating the dual band antenna. The simulated and measured results of the fabricated antenna are stated in this chapter.

4.1 Introduction to Frequency Reconfigurable Microstrip Patch Antennas

The concept of microstrip antennas was first introduced in the 1950s; however, they received a remarkable attention in the 1970s [78]. Figure 4.1 shows the structure of patch antennas, which consist of very thin metallic strip (patch), dielectric substrate and a ground plane. The strip (patch) is separated from the ground plane by a dielectric layer.

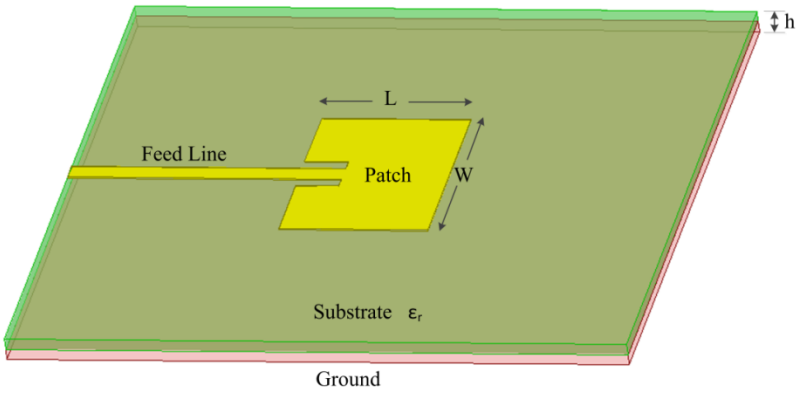


Figure 4.1 Microstrip patch antenna.

The patch antennas can have variety of configurations due to the change in the shape of radiating element. The radiating patch can be square, rectangular, circular and thin strip. The other main challenge of patch antennas is their feeding method. Utilization of microstrip feed line is preferable in this domain because of following reasons:

(1) Ease of fabrication
(2) Simplicity of matching process.

There is a fast progress in the field of communication systems; therefore, the necessity to obtain high-capacity RF systems is not negligible. In the area of wireless communications, integration of different applications has a considerable importance. However, various applications require different range of operation frequency. Variety of reconfiguration methods can be proposed for integration of different applications in a single device.

Variety of switching devices like PIN diodes, GaAs field-effect transistor (FET) switches and RF MEMS switches can be utilized in reconfiguration method. The privileges of RF MEMS switches are their low power consumption, low insertion

loss, reliability and high isolation. However, there are some disadvantages associated with RF MEMS switches such as: slow switching speed, high cost, complex fabrication process, and limited lifetime.

Fluidic systems are one of the best options to be manipulated inside reconfigurable antennas because they can solve the problems associated with RF MEMS switches. The key feature of liquid metal microfluidic switches is the absence of self-actuation and stiction problem. In addition, there is no mechanical fatigue in the microfluidic switches.

Eutectic Gallium Indium (EGaIn) is employed inside the microfluidic channel since it is conductive and non-toxic. In this chapter, the design and fabrication of frequency tunable antenna is explained. Liquid metal channel is used as RF MEMS switch in this design.

4.2 Design Procedure

The microstrip antenna was designed on glass substrate. The structure of antenna in simulation was shown in

Figure 4.2.

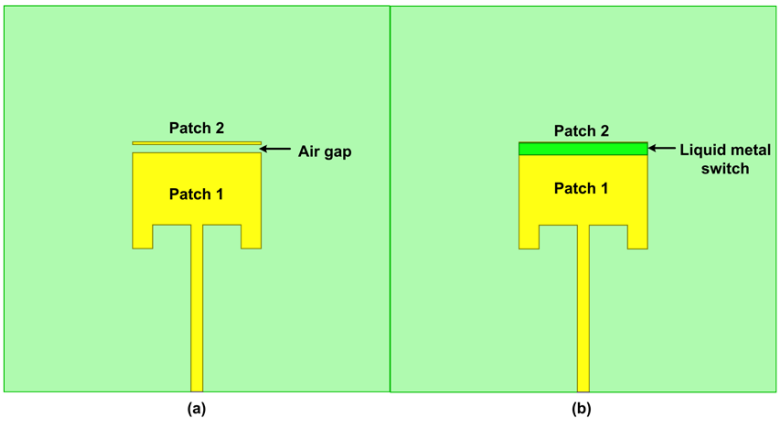


Figure 4.2 The general view of frequency tunable patch antenna.

Figure 4.3 shows the structure of the microfluidic patch antenna, which is constructed on a 500 μm-thick glass substrate (ε_r=4.6, tanδ=0.005). The microfluidic switch is located between the main patch and a parasitic strip parallel to the edge of the patch antenna. Introducing liquid metal into the microchannel electrically connects the main patch and the parasitic strip. Depending on the presence of liquid metal inside the channel, the patch antenna resonates either at f_1 = 10.68 GHz or at f_2 = 9.62 GHz, corresponding to L_1=6.7 mm or L_2=9.5 mm, respectively.

The widths of both patch and parasitic strip are equal to W= 8.96 mm. The inset parameters, Y and Y_0 are tuned to be 2.64 mm and 1.7 mm, respectively, yielding a reflection coefficient better than -15 dB for both of the resonant frequencies.

The 50 Ω microstrip line has a width of W_m= 0.88 mm. The width of the microfluidic channel is L_3=0.8 mm.

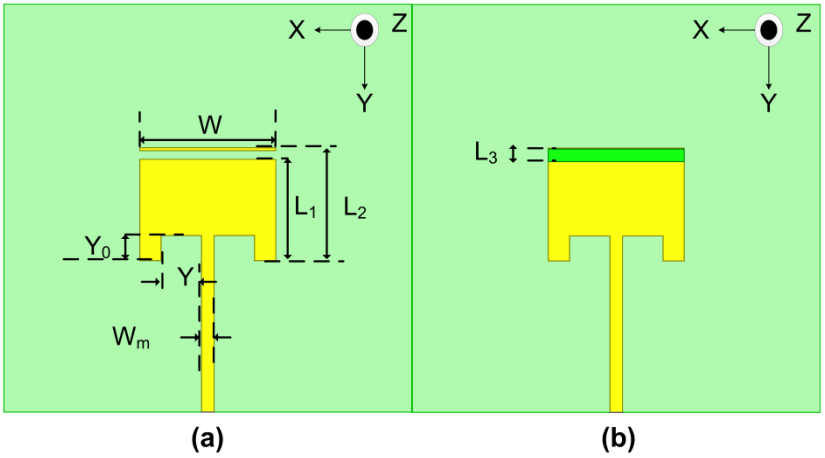


Figure 4.3 Microstrip patch antenna with microfluidic channel as a switch (a) 9.62 GHz Antenna, (b) 10.68 GHz Antenna.

In the following sections, there is an explanation related to the fabrication steps, which is conducted in METU MEMS center. The results related to the HFSS simulation and measurement results are compared to each other.

4.3 Fabrication Process

The antenna structure is fabricated on a glass substrate (ε_r = 4:6, tan δ= 0:005). The microfluidic switch is fabricated using a molded PDMS layer bonded on a glass substrate. The channel inside the PDMS layer is molded using an FR4 master, which is prepared by a LPKF printed circuit board (PCB) fabrication machine [80]. This method offers a low-cost solution for mold preparation. One main challenge in the realization of this microfluidic channel is the low bonding quality of the PDMS to metal layers [81]. In order to alleviate this issue, an intermediate SiO_2 layer, which is known to have good adhesion to PDMS with proper surface treatment [42], is deposited on the metal layer. The thickness of SiO_2 is selected as low as 50 nm in order to maintain a high capacitance between the liquid metal and the antenna metal resulting in a capacitive short circuit. Considering the region where the liquid metal and the antenna metal overlay, the parallel plate capacitance is found to be 0.6 nF giving an impedance of about 0.03 Ω around the frequency of interest (10 GHz).

Figure 4.4 demonstrates the device fabrication process.

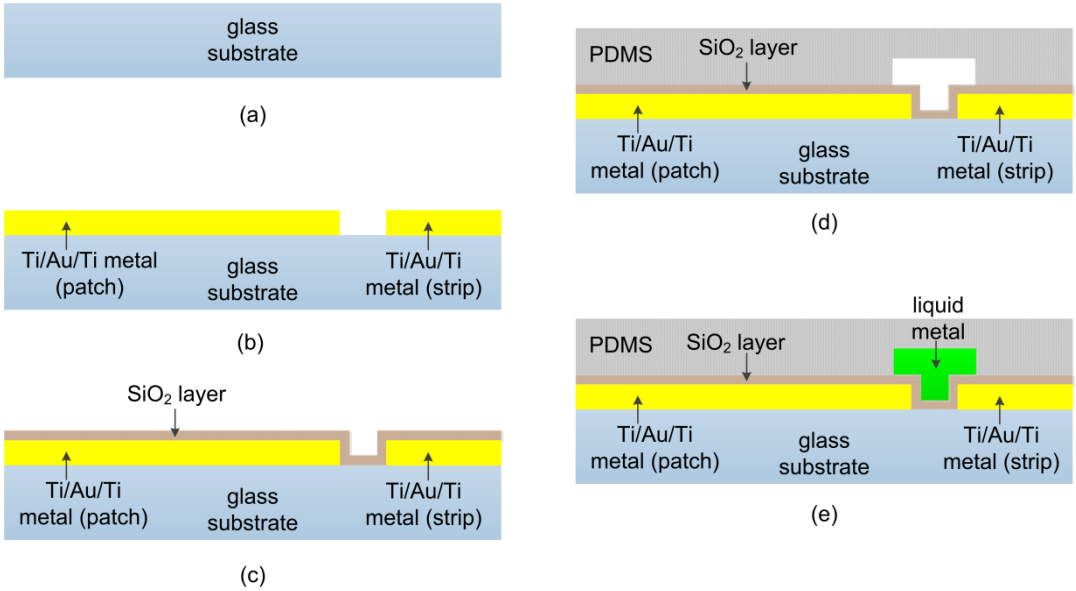


Figure 4.4 Fabrication process of dual frequency tunable patch antenna.

Figure 4.5 illustrates a glass substrate, which is coated with 20 nm/600 nm/20 nm-thick Ti/Au/Ti stack. The stack is patterned by photolithography and wet etching. Then 50 nm-thick silicon dioxide (SiO_2) is deposited on the metal layer using Plasma Enhanced Chemical Vapor Deposition (PECVD) method.

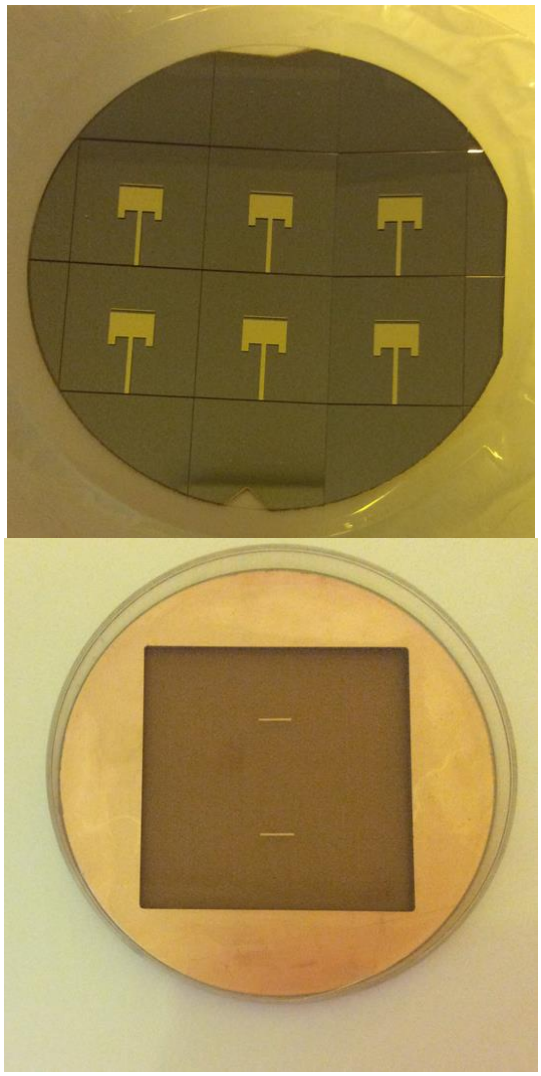


Figure 4.5 (a) Ti-Au-Ti deposited glass substrate, (b) Fabricated mold with LPKF tool for preparation of PDMS channel.

Before the oxygen plasma treatment for PDMS bonding, the antenna printed on the glass substrate is rinsed by immersing in acetone, isopropanol and DI water, respectively as shown in Figure 4.6. The optimal oxygen plasma treatment is done for duration of 20 seconds at RF power of 20 W. After properly aligning and attaching the PDMS layer and the antenna substrates, the stack is heated for 15 minutes at a temperature of 110 $^{\circ}$C on a hot plate.

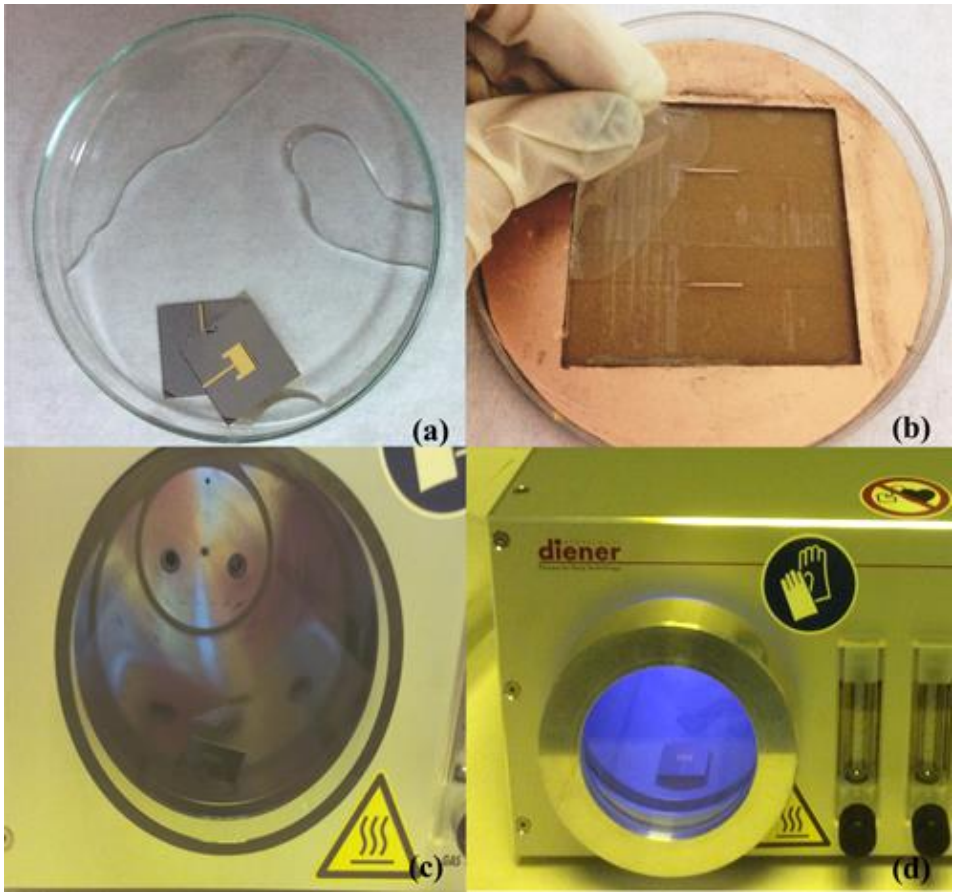


Figure 4.6 (a) Rinsing the patch glass in Acetone, (b) Peeling of the PDMS, (c) Putting the patch substrate and PDMS inside ICP , (d) Oxygen plasma treatment.

The liquid metal droplet is then injected into the microchannel by using an injection syringe as illustrated in Figure 4.7. The final structure of fabricated frequency reconfigurable antenna is shown in Figure 4.8.

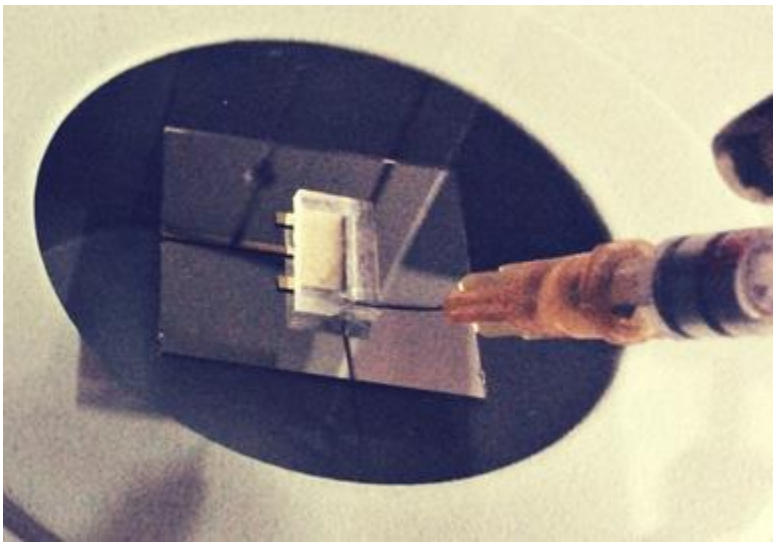


Figure 4.7 Injection of liquid metal inside the channel.

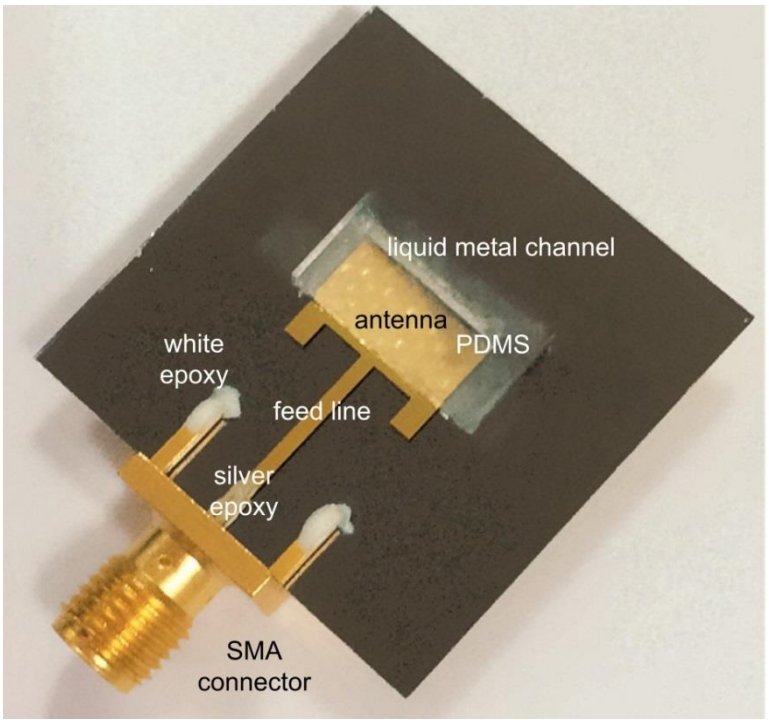


Figure 4.8 Photograph of the fabricated antenna structure.

4.4 Measurements and results

Figure 4.9 and Figure 4.10 illustrates the simulation results for the directivity pattern of patch antenna in two different states. In the first case, the directivity pattern is measured when there is no liquid metal inside the channel. In the second case, the directivity pattern is measured when the liquid metal is injected inside the channel.

Figure 4.9 Directivity Pattern of patch antenna in the case without PDMS (Frequency: 10.68 GHz).

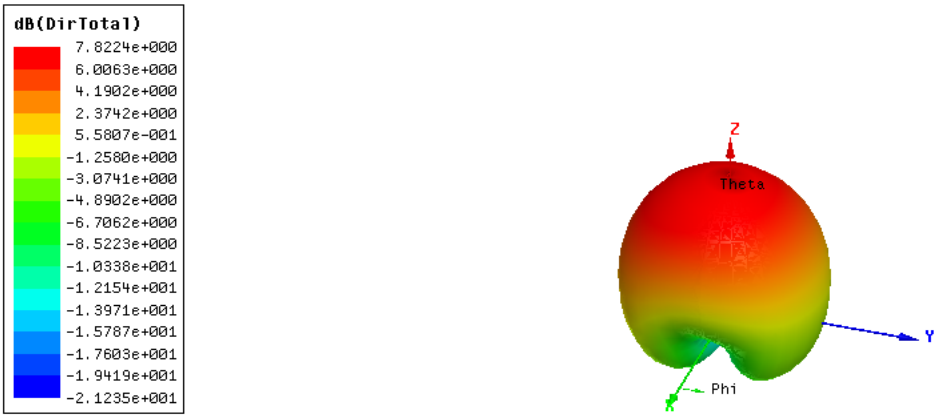


Figure 4.10 Directivity Pattern of patch antenna in the case with Galinstan (Frequency: 9.62 GHz).

Network analyzer shown in Figure 4.11 is employed to measure the reflection coefficient of microfluidic reconfigurable patch antenna. Figure 4.12 illustrates the simulation and measurement results for the reflection coefficient in two different states. In the first state, there is no liquid metal inside the channel and the antenna resonates at 10.68 GHz. In the second state, the liquid metal is injected inside the channel and the resonance frequency is shifted down to 9.62 GHz as the antenna length is increased. There is a good agreement between measured and simulated reflection coefficient characteristics for both of the cases.

Figure 4.11 Measurement of reflection coefficient with network analyzer.

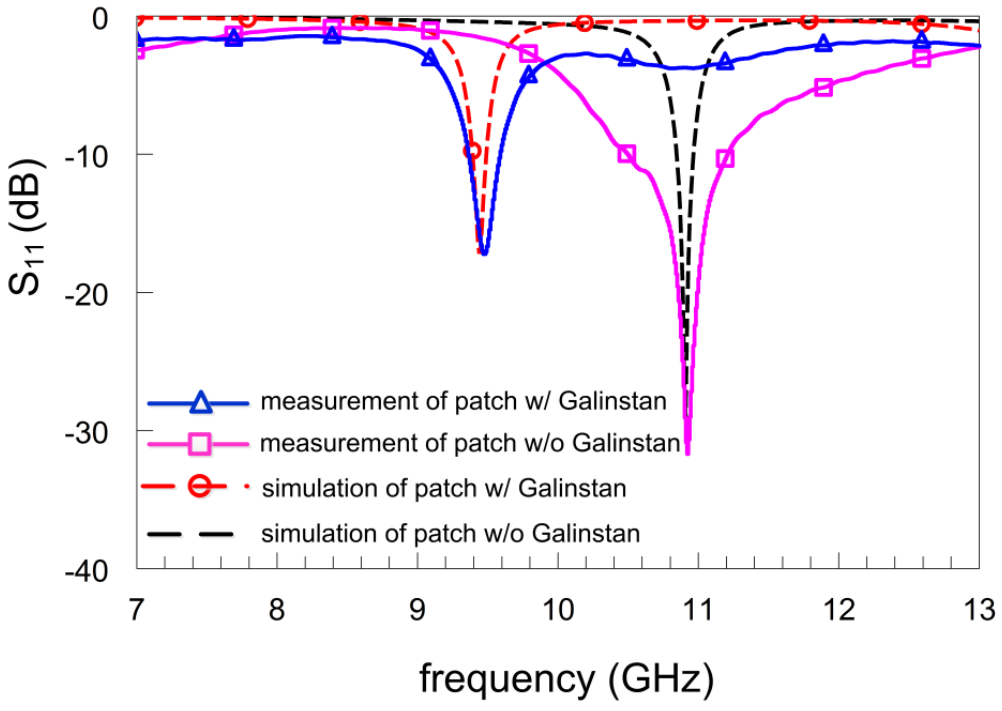


Figure 4.12 Reflection coefficient characteristics for two different states: (a) Patch antenna without Galinstan, (b) Patch Antenna with Galinstan.

Figure 4.13 and Figure 4.14 demonstrate the simulated and measured radiation patterns of the antenna at two frequencies where a good agreement for both E- and H-planes is noted.

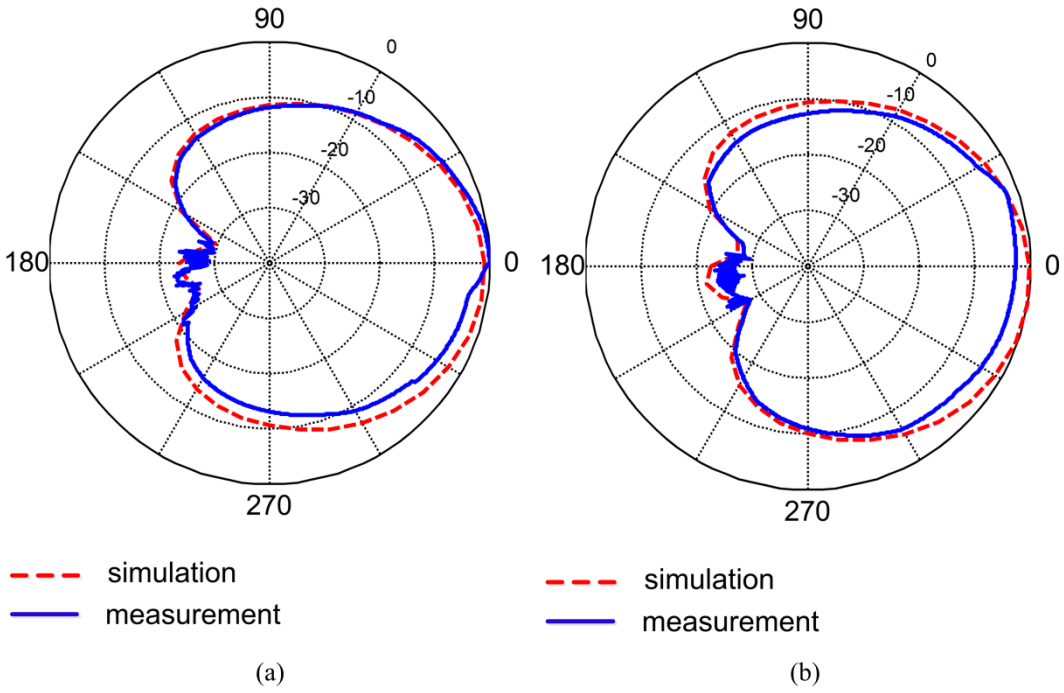


Figure 4.13 (a) Simulated and measured radiation pattern (E-Plane) of the antenna in the case without liquid metal (Frequency: 10.68 GHz), (b) Simulated and measured radiation pattern (E-Plane) of the antenna in the case with liquid metal (Frequency: 9.62 GHz).

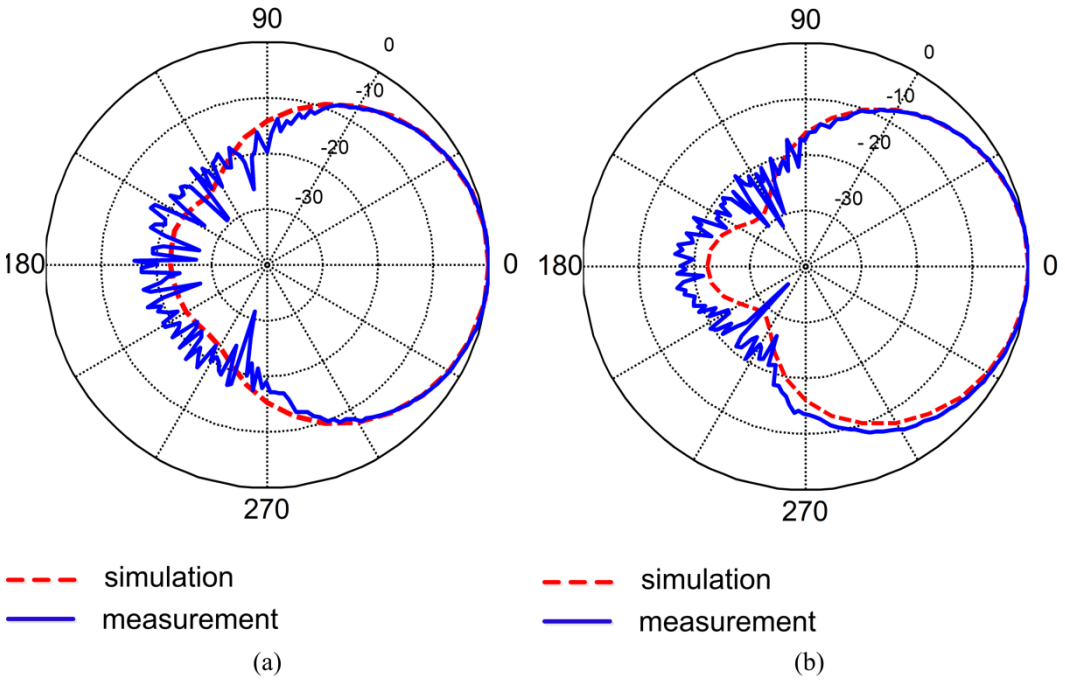


Figure 4.14 (a) Simulated and measured radiation pattern (H-Plane) of the antenna in the case without liquid metal (Frequency: 10.68 GHz), (b) Simulated and measured radiation pattern (H-Plane) of the antenna in the case with liquid metal (Frequency: 9.62 GHz).

4.5 Conclusion

This chapter presented the use of liquid metal as a switching mechanism for a frequency-reconfigurable patch antenna. Liquid metal switching strategy enables a practicable substitute to traditional RF switches in frequency-reconfigurable antennas. The antenna operates in two different frequencies. The measurement and simulation results of the antenna are stated in both frequencies.

CHAPTER 5

CONCLUSION AND FUTURE WORK

5.1 Conclusion

This thesis presents fabrication techniques, design, and measurement results of reconfigurable antennas employing microfluidic technology. Reconfigurable antennas have been extensively investigated in recent years as they offer a solution for the implementation of compact antennas performing different operations using the same structure. However, the proposed technologies used to implement reconfigurable antennas have some limitations. For example, PIN diodes and RF MEMS switches require bias lines that degrade the radiation characteristics of antennas. In addition, RF MEMS devices suffer from the tear and wear of materials due to mechanical operations. Microfluidic switching technology is introduced to solve the issues related with these devices. In this thesis, microfluidic based reconfigurable antennas and transmitarray unit cells are fabricated with new methods.

Conductive sections of microfluidic channels are created by injecting liquid metal inside the PDMS layer. The liquid metal inside the channels, is an alloy of 68.5% Ga, 21.5% In, and 10% Sn. Rotation of the liquid metal inside the rings controls the phase of the transmitted wave.

The proposed fabrication method for the reconfigurable patch antenna is another contribution of this thesis. Soft lithography, which is employed in the construction process of the antenna, is quite straightforward and simple. The mold

which is required for the creation of microfluidic PDMS channel, can be fabricated by PCB Milling equipment. The bonding strength of PDMS to metal surface is improved by employing a PECVD-deposited SiO_2 dielectric layer on top of the metal layer. This study illustrates that PDMS-based microfluidic switching technique is cost effective and easy method utilized in the structure of reconfigurable antennas.

In addition, optimization of parameters related to oxygen plasma tool is accomplished to develop strong structures of PDMS-to-glass microfluidic channels. Tensile test method is employed to measure the bonding strength of the structures.

5.2 Future Work

The concept of microfluidics technology is new and it requires great amount of consideration. The practicable research related to this content can be presented as follows:

- It is a hard process to obtain the desired split inside the unit cell due to the manual injection of the liquid metal. The application of accurate micropumps can solve the existing difficulty.
- In the field of transmitarrays, the rotation of liquid metal inside the microfluidic channel has a great importance. However, the rotation of the liquid metal inside the channel is not achieved up to now. Because, liquid metal leave residue inside the channel. To obtain this goal, a set of materials and devices such as Teflon solution and micropump can be used in the future works.
- The patch antenna presented here, can hardly return to its first state after liquid metal injection. There should be new investigations regarding to the removal of liquid metal after the injection process.

Consequently, employment of microfluidics in antenna technologies represents great chances reducing the restrictions of other methods. The accomplishments in this field will help to the development of new methods in the field of communication and antennas.

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