

SURFACE INTEGRATED MEMBRANE NANOMECHANICAL AND MICROWAVE COPLANAR WAVEGUIDE BASED BIOSENSORS

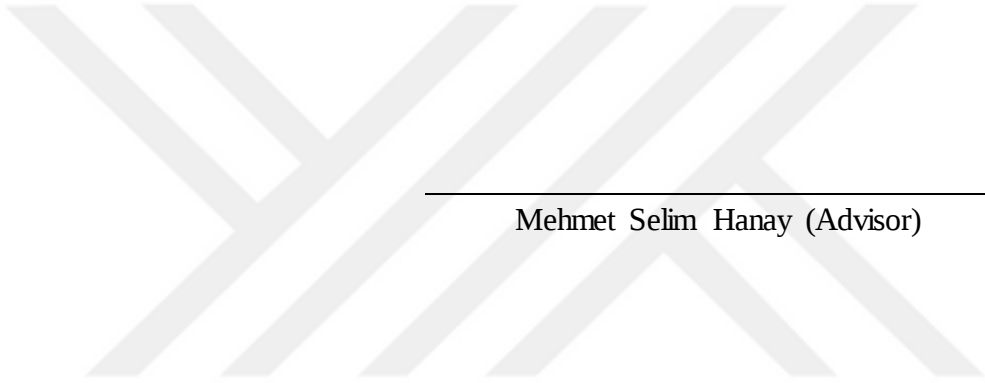
A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF ENGINEERING AND SCIENCE
OF BILKENT UNIVERSITY
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF
MASTER OF SCIENCE
IN
MECHANICAL ENGINEERING

By
Levent Aslanbaş
August 2018

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We certify that we have read this thesis and that in our opinion it is fully adequate,
in scope and in quality, as a thesis for the degree of Master of Science.



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ABSTRACT

SURFACE INTEGRATED MEMBRANE NANOMECHANICAL AND MICROWAVE COPLANAR WAVEGUIDE BASED BIOSENSORS

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M.S. in Mechanical Engineering

Advisor: Mehmet Selim Hanay

August, 2018

Nanoelectromechanical Systems, or NEMS, is the further miniaturized extension of novel devices of 40 years ago, Microelectromechanical Systems or MEMS in short. Nano scale devices first appeared at the dawn of 21st century and they are well established and elaborated by this time to a point which it shapes the technological paradigm of the current decade with various applications such as gene sequencing, improved computers and single molecule detection. The rapid improvement of miniaturization techniques owes a great deal to the hard work of scientists and engineers of previous generation. Fabrication methods which were limited to a small number which disallowed sub-micron features are improved and new methods have been discovered within the previous decades. This has paved the way for creation of very sensitive sensors in NEMS domain.

In this study, a novel biosensor is designed and attempted to be created out of coplanar waveguide resonators which is constructed on top of a nanometers thick membrane and at the sensory region of the resonator a nanopore is proposed to be created. The nanopore is suggested in order to allow nano-particle carrier fluids to pass through the most sensitive region of the resonator, causing a change in its resonant frequency due to electrical property of the nano-particle. The frequency

shift caused by particles is suggested to be used to detect and characterize the particles. The particles in this case are planned to be exosomes which are sub-micron packages with cytoplasmic content, naturally secreted by cells for various reasons. Contents of the exosomes may carry diagnostic information about the cell. Exosomes themselves are still being investigated for their uses and benefits within the context of microbiology which makes the proposed device very crucial for ongoing exosome research effort which is still on the rise.



Keywords: Surface integrated membranes, Silicon nitride membrane, exosome characterization, NEMS, Coplanar Waveguides, microfabrication, nanofabrication. KOH etching of silicon.

ÖZET

YÜZEYE ENTEGRE MEMBRANLI NANOMEKANİK VE MİKRODALGA EŞDÜZLEMSEL DALGA KILAVUZU TEMELLİ BİYOA LGILAYICILAR

Levent Aslanbaş

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Ağustos, 2018

Nanoelektromekanik Sistemler, yani NEMS, 40 yıl öncesinin yenilik getiren cihazları olan Mikroelektromekanik Sistemlerin, yani MEMS'lerin daha da minyatür hale getirilmesinin bir uzantısıdır. Nano ölçekli cihazlar ilk olarak 21. Yüzyılın başlangıcında ortaya çıkmaya başlamışlardı ve şu anda içinde bulunduğumuz onyılın teknolojik paradigmasını gen dizilimi, gelişmiş hesaplama kabiliyetleri ve tekli molekül tespiti gibi yöntemlerle şekillendirecek düzeyde iyi temellendirilmiş ve geliştirilmiştir. Minyatürleştirme tekniklerindeki hızlı ilerleme, önceki nesilden çıkan bilim insanları ve mühendislerin sıkı çalışmalarına çok şey borçludur. Eskiden küçük bir sayıyla sınırlı olan ve mikron altı boyutlarda üretime izin vermeyen üretim yöntemleri, geçtiğimiz on yıllarda daha iyi hale getirilmiş ve yeni yöntemler keşfedilmiştir. Bu sayede çok hassas, NEMS yapılı algılayıcıların yaratılması için yolu açılmıştır.

Bu çalışmada yenilikçi bir biyosensör üretmek için, nanometreler kalınlığında zarsı yüzeylerin üzerine inşa edilen eşdüzlemsel mikrodalga kılavuzu yankılayıcılarının dizaynı ve üretimi denenmiş ve yankılayıcının en hassas noktasına nano boyutta bir delik açılması önerilmiştir. Bu nano boyutlu deliğin önerilme sebebi, nano parçacık taşıyan akışkanların yankılayıcının en hassas

bölgesinden geçmesinin sağlanması ve bu sayede nano parçacıkların elektriksel özelliklerine bağlı olarak yankılayıcının çınlama frekansında bir değişime sebep olması amaçlanmıştır. Bu parçacık kaynaklı frekans kaymalarının, parçacıkların tespiti ve nitelendirilmesi için kullanılması önerilmiştir. Bu durumda, parçacıkların eksosom, yani mikron altı boyuttaki sitoplazmik içerik içeren hücre zarı paketçikleri olmaları planlanmıştır; öyle ki, bu eksosomlar, çeşitli sebeplerden ötürü doğal yollarla hücrelerde oluşturulup dışarı salınmaktadırlar. Eksosomların içerikleri hastalık teşhisi gibi konularda hücre hakkında bilgiler taşıyabilir. Eksosomların kendileri halen işlevleri ve kullanım alanları konusunda mikrobiyolojik bağlamda araştırılmaktayken, bu önerilen cihaz, halen yükselmekte olan bu konunun araştırılması için büyük bir öneme sahip olabilir.

Anahtar kelimeler: *Yüzeye entegre zarlar, Silikon Nitrür zarlar, eksosom niteliklendirme, NEMS, eşdüzlemsel mikrodalga kılavuzları, mikroüretim, nanoüretim, Silikonun KOH aşındırılması.*

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Acknowledgements

First of all, I would like to thank my advisor, Dr. M. Selim Hanay for bringing out my long forgotten passion for science. He is the reason that I am in academia as he invokes enthusiasm and passion for science in every one of his students with his ever-interesting lectures and discussions. One must be glad to even know such a great personality, let alone being advised by him. He has been a candle, a hope in the darkness for me in the days I saw what lies beyond the confinements of our small comfort zone in this beloved country, and what is to come. He gave me the purpose I need in this life as now I see the only way to bring this nation out of the darkness that it succumbed to passes through science and education, and I will work relentlessly to become a candle myself for my future students.

Secondly, I would like to thank all of my former and remaining peers and dear friends in this research environment, namely and foremost; Atakan Arı, Çağatay Karakan, Mehmet Kelleci, Selçuk Oğuz Erbil, Ezgi Orhan and Hande Aydoğmuş.

For creating such a vibrant research environment in this country, I must thank Prof. Dr. Adnan Akay. His presence alone was enough to be inspired for excellence in engineering and science. I also thank UNAM facility employees, especially Semih Bozkurt, Abdullah Kafadenk and Mustafa Güler for their friendly support throughout my fabrication experience here in Bilkent.

I thank my dear beloved family for their continuous support throughout my education. I also thank my dearest friend, my second brother, in this life, Mustafa Kara for his company and support, both personally and also for the scientific and philosophical discussions and brainstorm we had throughout years.

Last but not least by any means, I would like to thank the great leader and visionary, Mustafa Kemal Atatürk as he knew that science would be the only resort with which this nation would rise upon its ashes. A Türk should not need any other source of inspiration when we have him as an example.

Chapter 1

Introduction

Throughout history, there have been few instances which changed the overall life standards of common people. Invention of fire brought light and warmth which brought us as species out of the jungle where we stayed for millions of years. Likewise invention of farming pushed the humans into forming a society in greater numbers, creating the urban life, which was inconceivable to the non-farming population of the past. With the emergence of scientific approach in 18th Century, humankind has experienced yet another paradigm shift of a scale comparable to that of the invention of farming, as it paved the way for industrialization and a new perspective over the nature, through understanding of physics and its practical applications, engineering.

Ever since Sir Isaac Newton and his contemporary scientific community of 18th century started mathematizing the nature, engineering had separated from basic sciences as the two were indistinguishable due to being professed by same few people in the previous ages. Ever since this separation occurred, physics and engineering have been in an eternal out of phase dance as physics feeds engineering through new models and understanding, where engineering takes this understanding and makes it useful for life as well as feeding physics with more capable devices, allowing it to proceed further. This dance is out of phase,

because it is certain that only either produces the greatest effect on any given time. For instance, understanding of calculus, created the physics of 18th century, which led into industrialization and engineering. Decades after that was the discovery and understanding of the electricity which led to yet another golden age of physics, creating the physics of today through decades. This modern physics age taught us about the largest and the smallest that this universe can offer and it only happened within 4 or 5 generations of scientists. There was so much information generated that needed to be put to use, it created a great gap between what we know, and what we did with that knowledge. In that gap, engineering flourished within the decades to come, just as it happened a century ago; except this time, exponential growth of the technology over the years happened so fast that it transformed the world into the state of today, inconceivably different from even the previous decade. For instance, cellular phones were first created roughly 30 years prior and were a luxurious commodity only for half a decade, later to be integrated into the society for the benefit of carrying means of communication wherever one may please. It did not stop there. The innovations kept coming in the following two decades, until the point we could not even comprehend the idea of carrying a phone that was only capable of voice communication. Norms of the society have changed so rapidly it only happened within a time less than one generation.

The huge paradigm shift that we experienced in the 21st century is but only a manifestation of what our recently earned knowledge about the universe is capable of. With the overwhelming abundance of physical understanding, the time of engineering has started once again. This knowledge enabled engineers to delve deeper into the world of small scales, sparking the age of microelectronics and with the invention of transistors [1] which are the backbone of the information age that we live in. Transistor allowed information to be processed extremely faster than what was available before. In time, micro transistor had left its place to nano transistor [2], decreasing the volume of the computers from tens of cubic meters to handheld sizes as well as increasing their computational power millions fold only within five decades [3]. This incredible technological progress that we encounter daily has shown the potential of micro and nano devices. Though the

transistor being the most important part of the developments, it required information itself that needed to be processed. The information that transistors in handheld devices process is often supplied through micro and nano sensors. Sensors such as accelerometers [4], pressure [5] and temperature sensors [6] of Micro-electromechanical (MEMS) constructs receive the input of physical phenomena and convert it into useful information for the integrated devices.

1.1 Nanoelectromechanical Systems (NEMS)

The rapid development of technology that enabled fabrication of small devices have opened a new avenue in the technological advancement. It is now possible to fabricate novel and marvelous constructs in nano scale and test their sensory capabilities as most devices boil down to a signal enhancing plant in a control scheme which takes the initial signal out of the natural phenomena that occur in a well-controlled environment [7]. The output signal of the construct can be used to understand the miniscule changes in its environment in terms of electromagnetic and mechanical phenomena, thus creating a Nanoelectromechanical System (NEMS) based sensor, given that the device has a feature of less than 1 micrometer [8].

NEMS resonators which were first created in 1996 by Roukes and Cleland [9] were the pioneers of a new era in science and engineering. In time, many different applications for various NEMS were found. Sensors for basic physical phenomena such as temperature, pressure and humidity were fabricated and utilized [10]. Thereafter, more intricate devices such as NEMS based mass spectrometry were developed [11].

1.2 Resonant Nanomechanical Sensing

Mass sensors which work depending on the information about change in mechanical resonance frequency due to attachment of mass to the resonators had a well-established physical understanding behind them. The resonance frequency of a resonator depends on effective stiffness - which is similar to the spring constant of a simple mechanical harmonic oscillator that consists of a mass and a spring - as well as the mass that is oscillating in space. Thus, one can deduce the mass of the object attached to the resonator via comparing the frequency with and without the mass, given that the effective stiffness of the system is known [12].

First resonant mode of beam structures were initially used to measure very small masses via resonant sensing techniques [12]. These techniques later evolved into multi resonant mode resonators which were capable of resonating in several resonant modes of the beam simultaneously -by a signal routing method generally referred to as “*downmixing*” [13] [14] - each mode extracting independent bits of information about the analyte mass [15][16]. Due to the location at which the mass is attached to the resonator and the shape of the analyte, well defined resonant mode shapes and frequencies are altered and when measured properly, the change in frequencies could be combined to generate an image of the analyte. This method known as Inertial Imaging allows single protein mass and morphology measurements to be conducted via NEMS resonators [15][16].

For simple beam resonators, the change in resonance frequency Δf could be simply represented as a function of initial frequency $f_{initial}$, effective mass of the structure $M_{initial}$ and the change in mass due to the analyte Δm [17]:

$$\Delta f = -\frac{1}{2} \frac{f_{initial}}{M_{initial}} \Delta m \quad (1.1)$$

Simply put, the resonance shift caused by the addition of mass is directly proportional to the change in resonance frequency. This relation enables real time mass sensing when the resonator is driven in its natural resonance frequency with

a closed loop controller called Phase-Locked Loop (PLL) [18]. This particular controller is used to track the resonance frequency in real time. In principle, the resonator is first characterized for its stock resonant frequencies by an open loop frequency sweep. Then, those frequencies are presented for the PLL controller as initial values for frequency input to the NEMS. Controller probes the NEMS with that input frequency and reads the phase difference between the input and output signals of the NEMS. For each point in time, the frequency and the correspondent phase difference are recorded. According to the magnitude of phase difference, the next frequency input is increased or decreased in a way that the phase difference would be kept at a predetermined fixed value, 0 for acquiring the resonance frequency. Therefore, the controller keeps the resonator resonating precisely at its resonance frequency while recording the values in real time. This enables easily recognizable shifts in frequency due to some particular event, in this case the addition of mass [11].

1.3 Microwave Resonators

Mechanical and electromagnetic resonators are governed by analogous equations. Just as a change in the resonant mass of a mechanical resonator proportionally alters the resonance frequency; changes in the quantity called “*electrical volume*” of an electromagnetic resonator, such as a microstripline microwave resonator, result in a change in resonance frequency with a perfect analogy. The electrical volume is defined as the volume integral of electrical permittivity in the resonant cavity for this analogy to hold [19]. Similar to the initial mass of a mechanical resonator, there is an initial electrical volume of a microwave resonator. For instance, a microstripline resonator which consists of two planar electrodes and a rectangular prism shaped dielectric material in between them, therefore the electrical volume of such resonators are simply the multiplication of the volume of the dielectric prism and its presumably uniform electrical permittivity.

The electrical volume of the resonant region slightly changes along with the resonance frequency of the resonator due to the permittivity of particles in the microfluidic channels that pass through the sensory region of the resonator being different from that of the particle carrier fluid. These particles in a recent study were two different types of human cancer cells and according to the frequency shift in first two resonant modes of the microstripline resonator caused by each of two types of cells were clearly distinguished from one another, effectively establishing a new branch of microfluidic analysis tool and biosensors [19].

The construction of microstripline resonators is comprised of a vertically layered stack of electrodes and a dielectric material in between them. On the contrary, there is also an alternative yet similar microwave resonator class, namely Coplanar Waveguides (CPW). CPW microwave resonators do not require vertical stacking of layers and instead it could be constructed on a single planar surface, which results in a more robust resonator which is essentially nanometers thick, easier to fabricate and with much higher reproducibility [20].

1.4 Mission and Motivation

Knowledge generated through experience of individuals erode out of the research environment as the time passes and individuals who conduct the research change. This knowledge erosion decreases the time efficiency of the research, because when the knowledge disappears upon individual graduation, productivity in many aspects of an ongoing project plummet naturally due to the time consuming nature of the practical learning process which is mostly iterative and often counter intuitive. The experience therefore, has to be converted into a manuscript that could be used by anyone inexperienced, to overcome most of the iterative processes of learning. The details about fabrication of a new branch of microwave devices are represented in this work within a scope of serving the mission of the overall project - which is to create a capable device to characterize and image

nanoparticles in a fluid environment – via easing the adaptation period of the new member of the project.

The main reason for which this manuscript is written is to propagate the practical and fabrication experience collected by one generation of graduate researchers in NemsLab to the next as comprehensively as possible. The experience that is aimed to be conveyed through this manuscript is specifically Micro and Nano scale top-down fabrication as it will explain the details of a specific device fabrication. The device is a Bowtie shaped Coplanar Waveguide Microwave Resonator with a nanoscale electrical hotspot situated on a microns wide, nanometers thick membrane with a nanopore. The end goal of this work is a microwave resonant sensor that could detect nanoparticles in a fluid environment due to the change in resonance frequency created by particle movement through the nanopore located at electrically sensitive zone. Within the scope of biological sensing, it is of paramount importance to increase the sensory capabilities of the present microwave resonators in order to detect and characterize submicron entities such as exosomes and DNA fragments. The successor designs of this device could potentially enable the characterization of nanoscale biological objects and emerge as an alternative imaging technique within years to come. Knowing this alone and expecting the glory of creating a unique and useful device within biotechnological context is the prime source of motivation behind this project.

1.5 Thesis Outline

The main content of this manuscript is the detailed explanation of fabrication techniques used to create a new generation of microwave coplanar waveguide resonators capable of nano-particle sensing as stated in the section “1.4 Mission and Motivation”.

In Chapter 1, an introduction to the content is laid through the scope of scientific and technological advancements that had led to the era of micro and nano engineering, followed by the background information on current applications and capabilities of micro and nano electromechanical systems, mechanical resonators in particular. It is also mentioned that microwave resonators share the common mathematical backbone with nanoelectromechanical resonators. The analogy, which enables similar instrumentation structure to be used for both microwave and nanomechanical resonators, is briefly explained as the focus of the work done does not correspond to the instrumentation and measurement of the devices but rather the fabrication which has been my specialized task within the extent of microwave resonators project.

In Chapter 2, “Design and Fabrication Procedures”, design of the proposed microwave resonator with nano features is explained and fabrication processes that the new design requires are identified. After the design details are conveyed, machines and methods used for fabrication are described in a method and instrument specific detailed format. This chapter serves as a preliminary to the Chapter 3. The fabrication methods which are used to create the devices are analyzed individually, reporting practically relevant information about the fabrication tools. The chapter is constituted as a fabrication guide in an attempt to ease the learning process of the new members of the research group and in order to increase their awareness about capabilities of the methods and machines available in UNAM facilities as well as specific problems encountered in each step of the fabrication.

Chapter 3, namely “Fabrication of Surface Integrated Microwave Coplanar Waveguide Resonators”, is an extended summary of how the methods and machines listed in Chapter 2 were utilized for the fabrication of the mainly focused device, membrane mounted CPW. Whole fabrication process is first summarized to establish the coherence between individually explained fabrication procedures. Separately explained methods are then fused together in a fabrication flow. The fabrication is analyzed in three distinct phases. Also, fabrication of

previous designs which later led to the final design are explained and discussed in minor detail within this chapter.

Chapter 4, “Conclusion, Discussion and Future Prospects” - evidently by the name - concludes the manuscript with the results of the fabrication and the device is presented in its current state which is actually one step short of completion due to an unexpected failure of a fabrication tool which has been unavailable for several months and still is at the time this thesis is submitted. The future prospects for this device and ideas to improve it are also discussed in the end.



Chapter 2

Design and Fabrication Procedures

This chapter starts with an overview of the design process with a discussion about the shortcomings of the previous microwave resonators and potential ways to improve it.

2.1 Fabrication Oriented Design

In order to design an actually manufacturable device, one must know the fabrication process thoroughly in terms of practicality challenges specific to each step of the procedure. Preemptive identification and foresight of possible errors in fabrication is a crucial skill for designing any device of any size. It is a skill best earned through personal experience and often builds up via iteration. However, it is also possible to increase problem identification awareness through second hand knowledge such as content of this manuscript. Micro and nano fabrication experience generated over years enables the fabrication oriented design and creation of this delicate and intricate sensor as it requires an in depth understanding of fabrication methods and technical limitations that they harbor. Designing a system simply through theoretical and analytical approach almost

always fails at the fabrication phase. For this device, the delicacy that comes along with the membrane, on which the sensor region is located, is the main factor that may cause failures during fabrication steps.

Fabrication of a device consists of multiple steps of delicate fabrication procedures. The samples are fabricated in UNAM Cleanroom facility which contains almost all of the most common and universal fabrication tools. These tools enable fabrication of extremely delicate devices and sensors which could be grouped under two main categories: MEMS and NEMS, respectively Micro and Nano Electromechanical Systems. As previously stated, MEMS devices are defined as electro mechanical devices or machines with features smaller than 100 micrometers and similarly NEMS devices have features of 100 or less nanometers. Interestingly, the bowtie shaped Coplanar Waveguide (CPW) resonator on the focus of this manuscript is fabricated with feature sizes varying over five orders of magnitude ranging from 100 nm thick membrane and nanopore to the 3 cm long coplanar waveguide on top of it. Technically, it could be considered as a NEMS due to its small features, however, it has an unorthodox design in terms of scales which is contrary to the commonplace NEMS feature size range, which often extends from 10 nm to 100 μ m at most. For this reason, this device could be considered a NEMS residing on a MEMS.

The resonator itself is primarily distinct in its structure and construction, being a CPW, it is fundamentally different from the previous microstripline resonators that were fabricated largely by manual precision. The greatest disadvantage of the microstripline resonators is that they require a mostly manual multi-layer construction. Sensitivity of such resonators could only be significantly improved via decreasing the thickness of the dielectric layer in between the top and bottom electrodes due to their construction, which is practically impossible due to handling difficulties and restrictions that come along with the biocompatible soft polymer, Polydimethylsiloxane (PDMS) that it is made of. PDMS layer - which contains the micro channels that carry the microbiological samples - is found via personal experience to be extremely difficult to handle when it is thinner than roughly 0.5 mm, due to tearing and electrostatic interactions which cause it to

schrunch up before even attempting to use it. The desired thickness between electrodes, thus evidently could not be achieved with PDMS and even if it was possible to do so, it had very limited room for improvement after the thickness is reduced to the minimum possible value due to channel dimensions. This would present further shortcomings for the future prospects of the development of more capable sensors.

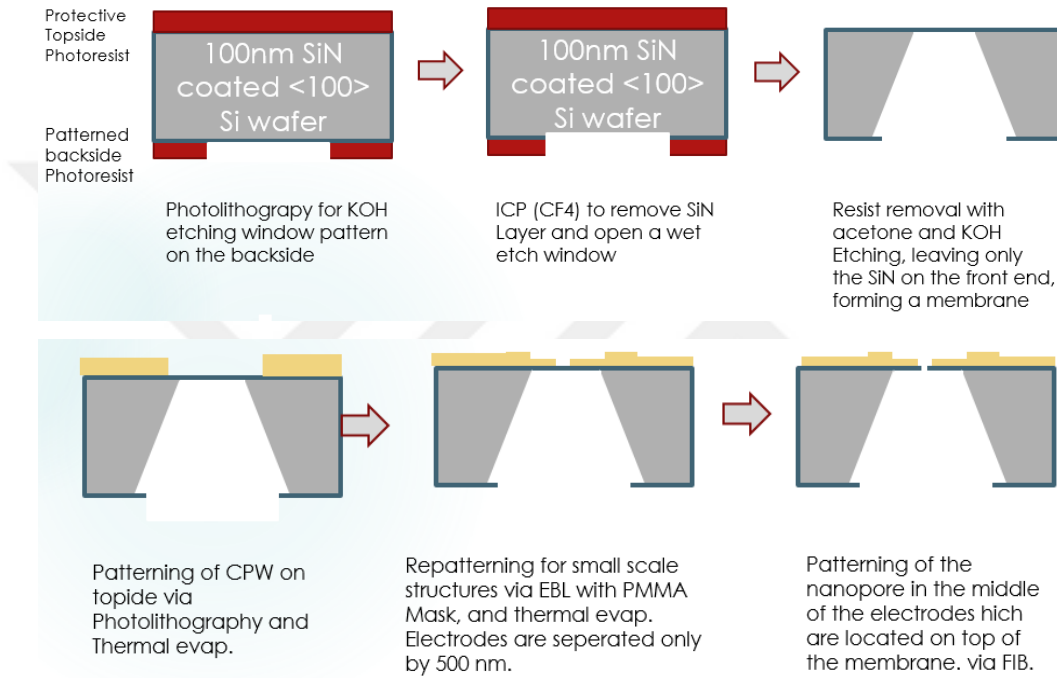


Figure 2.1: A compressed and simplified step-by-step representation of the fabrication designed for the new CPW device in this work.

In order to overcome such limitations, using CPW resonators instead of microstripline resonators is proposed. CPW resonators, evidently by name, are constructed on a single plane such as the smooth surface of a silicon wafer and the sensing capability could be improved by simply shrinking the distance between ground and signal electrodes, which could be achieved through a careful phase of fabrication process optimization. It would be a time consuming yet ultimately possible approach. Moreover, it would greatly reduce the imperfections caused by manual assembly procedure that microstripline resonators are fabricated through, by transferring all of the spatial precision elements of the fabrication to the specialized micro and nano fabrication tools.

Although there had been some previous trials of CPW fabrication with Cu electrodes on Fused Silica glass substrate, the “doubly clamped” structure of the resonator - which was a direct implementation of previous nanomechanical resonator experience of the research group - was later abandoned until the measurement capabilities of the NEMS Lab are enhanced, as we had yet been unable to even locate a reliable resonance frequency, let alone measuring a significant data out of those devices. Now, a circulator circuit which is capable of appropriate routing of input and output signals of the resonator through the same channel is installed to be used for CPW measurements.

The new bowtie CPW resonator at the focus of this manuscript is designed in order to reproduce and improve upon the device represented elsewhere [21], when it is determined that we might be able to fabricate a better version of the device as well as a us having a superior instrumentation than presented there. My design consists of a 500 μm wide signal carrying line, which runs straight between ground electrodes on both sides with a constant dielectric gap of 50 μm , reaching the edge of the wafer where the ground electrode widens with a constant angle on both sides, effectively creating a zone which only allows significant electrical interactions at the end of the line to be confined only to the point where it almost connects to the ground, located at the very tip of the line. This creates a particularly sensitive region between the electrodes [21]. The closer two electrodes are, the higher sensitivity at the tip will be. In order to maximize the sensitivity of resonant frequency to changes in electrical volume which are induced by the particles in an aqueous analyte passing through the gap between the tip and the ground, the distance between the electrodes was shrunk down from its initial distance of 150 μm to 300 nm.

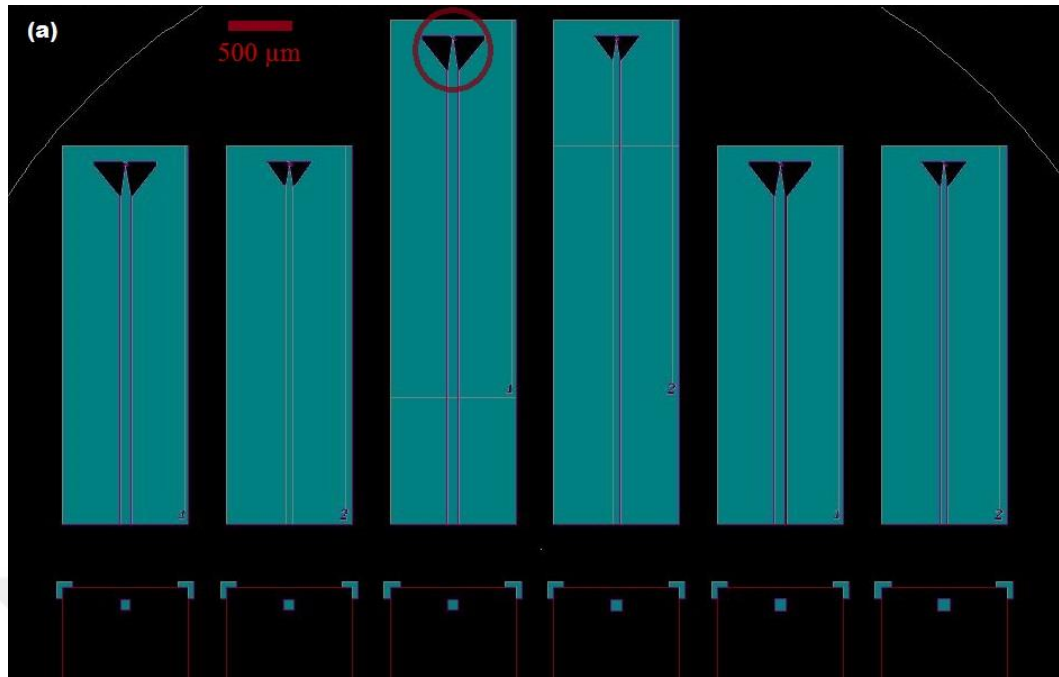


Figure 2.2.a: CPW design along with membrane window patterns printed on a single photomask. Six minorly differentiating iterations of the design are printed together. Red circle represents the zoomed in location for the next figure.

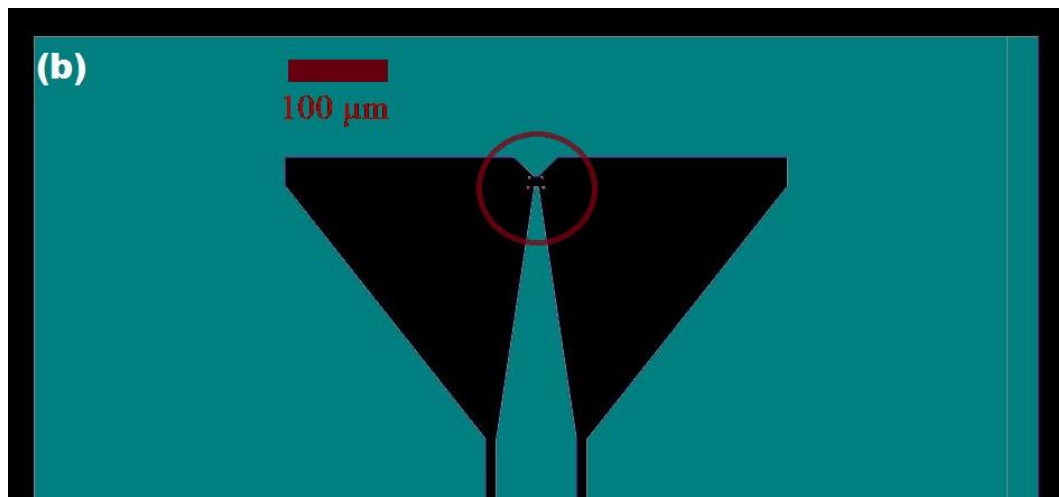


Figure 2.2.b: Micro features are visible at this scale.

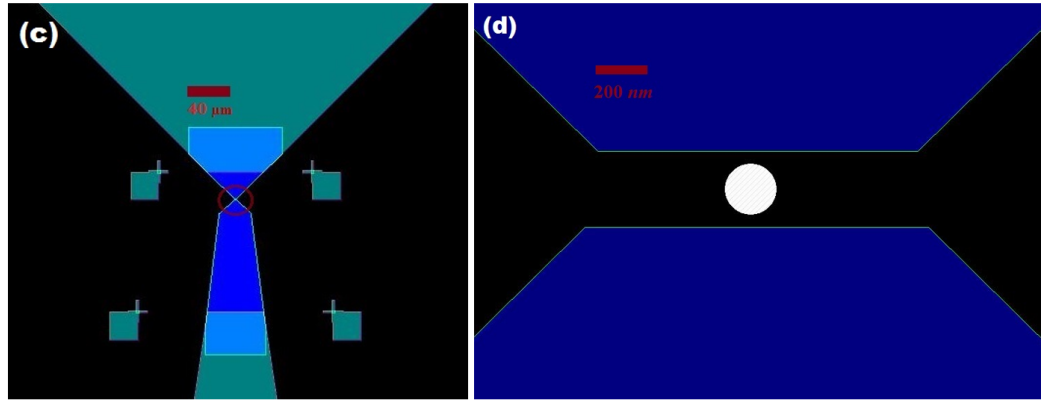


Figure 2.2.c and 2.2.d: Further zoomed in features at the sensor region of the resonator design. Blue represents the E-beam Lithography pattern designed to extend the bowtie structure to 300 nm separation between electrodes. The nanopore designed in the middle of the structure is represented in (d).

2.2 Fabrication Techniques

Fabrication routine regarding the whole device determined in the design phase is later actualized through several consequent processes and machines. Fabrication methods that are required for and covered through this one device fabrication are as listed:

- ☐ Spin Coating
- ☐ Photolithography
- ☐ Inductively Coupled Plasma (ICP)
- ☐ KOH Etching of Silicon
- ☐ Thermal Evaporator
- ☐ E-Beam Evaporator
- ☐ E-Beam Lithography (EBL)
- ☐ Stylus Profilometer
- ☐ Focused Ion Beam - Ion Milling (FIB)

These devices and methods are explained in a scope of working principles and their implementation on fabrication of this particular device. The implementations in this case include tips and tricks about certain devices, collected throughout years of usage, which may either be universally relevant for a certain type of fabrication process or be more specific machine oriented, i.e, thermal evaporator bot holders causing short circuit in a certain arrangement is a device specific problem. Ways to avoid this problem is also discussed in this manuscript in order to convey the information that would otherwise be lost and have to be regained in a time and material consuming process.

2.2.1 Wafer Cleaning

Silicon wafers are cleaned through a series of chemical baths and washing. Appropriately sliced wafers are first put into an acetone bath in order to solve any

greasy and organic residue that litters the surface. The beaker that contains acetone submerged wafers is subjected to ultrasound cleaning for 10 minutes in an attempt to improve the cleaning via mechanically disturbing and dislocating the contaminants alongside with chemical cleaning of acetone. The wafers are then taken out of the bath one at a time, washing each piece with acetone filled wash-bottle. Acetone itself leaves residue upon evaporation, therefore samples are then washed with isopropyl alcohol (IPA) in order to remove any acetone off the surface.

IPA does not leave any residue and upon vaporization, however it is of common practice to complete the wash with deionized water (DI water). I have encountered many different points of view on this issue in academic forums and discussion platforms. My stand point here is that DI water is unnecessary and even counterproductive for it is more likely to contain contaminants than high purity, bottled commercial IPA which is retailed globally and produced in huge batches by reputable chemical companies such as Sigma-Aldrich, whereas DI water is synthesized locally in the UNAM facility, probably with variable perfection.

After the washing is complete whether be it by IPA or DI water, the chip is firmly held with a polymer composite (carbon fiber) tweezer – specifically not with a metal tweezer as it certainly scratches the surface of the wafer – and nitrogen gas is blown over the chip to evaporate or forcefully remove the remaining droplets that were left on the surface.

2.2.2 Spin Coating

Spin coating is the very common technique used for coating various liquid based polymers on top of smooth surfaces such as Silicon wafers or glass microscope slides. Liquid on the surface of the sample is spread out evenly within the continuity of the surface by this method. The principle of operation is the balance between the surface tension of the liquid which tends to clump up the droplet and the forces created due to rotation, which stretch and spread the droplet over the

surface as a function of rotation speed. This naturally ensues, thickness of the liquid film being modulated by rotation speed as well as surface tension of the liquid which is dependent on material specificities such as molecular weight and viscosity. Simply, manufacturers of the commercial coating liquids often supply the “*spin curves*” for each product, defining the thickness with respect to rotation speed and so far all spin curves encountered has been found to be coherent with the thickness measurements.

The mechanism basically consists of a turning chuck which could be adjusted for turning time, angular velocity and acceleration. An external vacuum pump is attached to the device which maintains the vacuum at the tip of the chuck in order to keep the sample stuck while rotating at rotation speeds reaching up to 6000 rpm.

Spin coating is performed for coating photoresist (AZ 4562) and electron beam resist (PMMA) for the content of this project, and are detailed below.

An appropriately sized intermediary vacuum chuck should be placed on the spinner to assure vacuum quality during spinning as the unitless vacuum level parameter displayed on the interface should not be less than 14 while spinning continues. It is advised to keep in mind that spinning vacuum level typically decreases by 1 or 2 units when spinning starts and if it reaches 14, spinning will be prematurely aborted. Therefore, in order to avoid that disruption which leads to tedious recleaning of the wafer, vacuum level should at least be 16 before coating. Sometimes without any apparent user sourced error, level 16 vacuum could not be achieved anyhow. In that case, vacuum requirement could be removed in recipe edit screen at ones own risk of destroying the sample while also limiting the maximum speed to 3000 rpm. Nevertheless, only once have I ever encountered -in hundreds of coatings I have done so far- an occasion that vacuum did not hold and wafer flew off the chuck, so it is usually safe to remove the requirement.

For MicroChemicals brand AZ-4562 photoresist coating [22], operation starts with pre-baking of the previously cleaned samples for 10 minutes at 110°C in

order to remove any humidity off the surface to improve the adhesion between resist and the surface. After the baking, the sample is manually aligned on the vacuum chuck to roughly align the axis of rotation to the center of mass in order to achieve the best results for spinning stability and resist thickness uniformity. The sample, now stuck to the chuck with appropriate vacuum conditions is ready to be coated. The lid is closed and from the hole on top of the lid, using a plastic pipette, HMDS solution which increases adhesiveness of the surface is dripped on top of the stationary chip and a 40 seconds long spinning is started with 4000 rpm steady state speed and 1000 rpm/s acceleration. When the spinning ends, lid is opened and closed again without removing the chip, photoresist is poured onto the surface with a separate pipette, covering as much surface as possible and same spinning recipe is applied once again in order to achieve 6 μm thickness.

After the spinning, a piece of cleaning tissue is dipped into acetone and gently rubbed around the edges of the sample where photoresist is much thicker than optimal. This however should be done very gently in order not to totally remove the photoresist off the surface but only to curb the excesses. It is advised not to break vacuum before rubbing.

The sample is then removed gently and without disturbing the surface, to be placed back on 110°C heater for 5 minutes to evaporate the solvent, forming a solid photoresist layer on the surface. This concludes the spin coating process.

For bilayer PMMA coating which is required for EBL patterning, two separate PMMA models are used: A4 495k and A2 950k (referred as A4 and A2). Before coating, samples are baked on a hot plate at 180°C for 10 minutes to remove moisture. Primarily A4 is coated on the clean surface and A2 is coated over A4. In order to achieve total thickness of 300 nm, 215 nm thick A4 is coated at 2000 rpm for 40 seconds, then the sample is placed on 180°C hot plate for 5 minutes in order to remove the solvent and to heat treat the polymer, forming a solid layer. After the baking, 80 nm thick A2 layer is coated on top of the A4 at 3000 rpm for 40 seconds, followed by post-baking at 180°C for 10 minutes, finalizing the coating process.

It is very important to notice that anisole, the solvent used for PMMA is capable of dissolving plastics, thus using glass pipettes instead of cheaper plastic ones is crucial in order not to contaminate the samples or the whole bottle of PMMA solution. Also, while coating the A2 over A4, the spinning should be started immediately after A2 is dripped on the sample because, anisole in A2 will dissolve the solidified A4, disrupting film quality.

Unlike highly viscous AZ 4562, PMMA solutions are very much like water in terms of fluidic characteristics and do not require an edge bead removal with acetone.

2.2.3 Photolithography

Photolithography is the first of the two lithography processes covered in this study. It is used to transfer microstructure patterns onto the surface of the silicon wafer. In principle, it consists of an ultraviolet light source which shines through the glass substrate of the metal-on-glass photomask to the photoresist coated surface of sample. UV light alters the chemical composition of the photoresist film upon contact, allowing the developer solution AZ 400K to remove the exposed portions, leaving the unexposed regions intact.

Photomask is as a 2.3 mm thick, 5 by 5 inch square fused silica glass, entirely coated with a chrome layer which is opaque to UV light. A photoresist layer is coated on the chrome surface by the manufacturer prior to packaging and they come ready to be patterned with a laser by UNAM Cleanroom engineers. Designed features larger than 10 μm are realibly fashioned by the mask writer, removing the chrome from the surface wherever necessary.

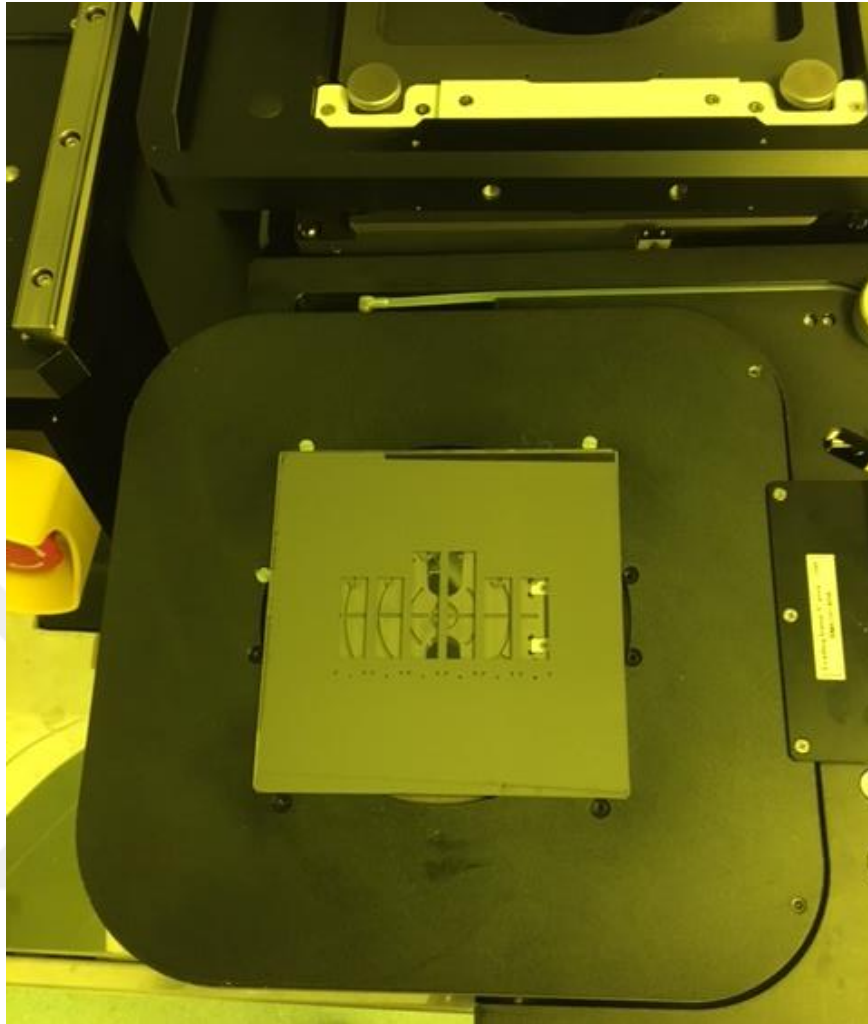


Figure 2.3: The mask placed on the mask holder with three white points aligned to the edges of the mask. The mask holder is installed on top of a tray to serve the mask and samples into the photolithography device, also known as the mask aligner. Cr colored side is facing upwards instead of copper colored side.

This device is probably the most user friendly machine in UNAM because user interface very clearly shows what to do step by step during operation. After the required parameters are chosen, following the prompts of the interface, photomask is first installed to the machine with chrome side facing upwards. The mask is served into the machine on a mask holder placed on the service tray. The mask holder has three points which must contact the edges of photomask to align it to

the correct location. Mask is held in the machine by a vacuum system tht needs to be switched on prior to installation of the mask. Then the photoresist coated samples are placed on the tray and transferred into the machine. Alignment of the pattern to the sample is done with the aid of a built in camera. After the alignment is done, the UV light is applied with constant dose of energy per surface area, specifically 65 mJ/cm^2 is used for $6 \mu\text{m}$ thick AZ 4562. After the exposure, the samples are submerged into AZ 400K developer solution for 60 seconds. UV cured regions of the photoresist are dissolved, exposing the layer beneath the photoresist. This concludes the photolithography process.

UV light that is used in this process is harmful to human eyes and capable of permanently damaging the retina, therefore it is recommended to look away from the machine before pressing “continue” button when alignment is done. The UV light starts immediately after the button is pressed and it happens without a warning.

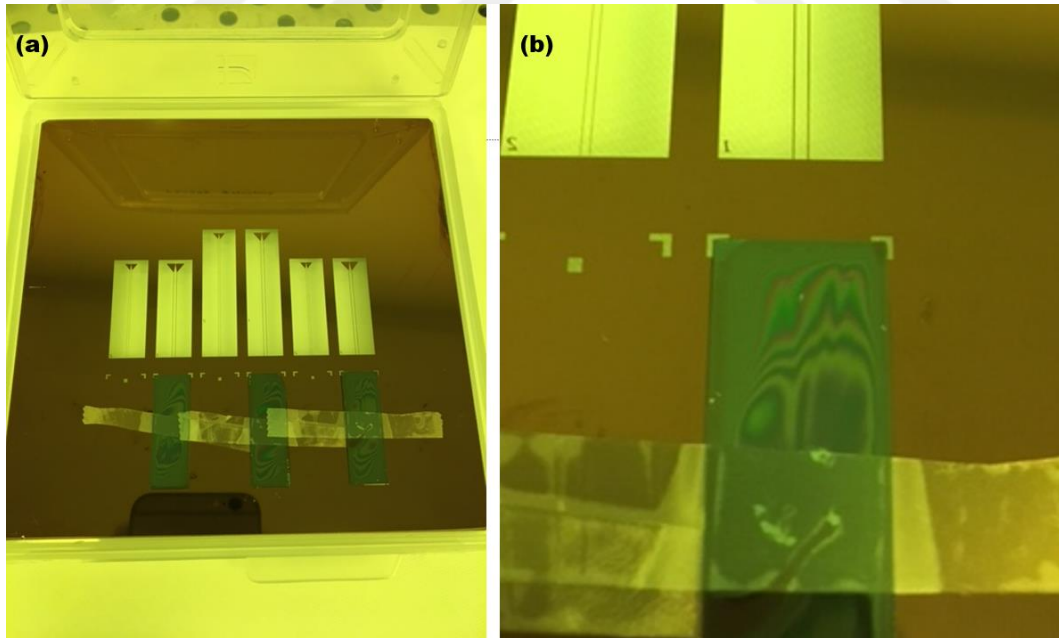


Figure 1.4: The mask used for patterning of membrane creation windows and later the CPW structure is shown in the figure. samples are manually aligned for the alignment markers on the corners in order to center the membrane at the desired location. This is an unorthodox method of alignment however, it results in perfect

precision for such a process and it is insufficient for alignment of CPW to the membranes. This is used for its practicality and significantly quickens the process.

The optimal developer to water volume ratio for preparing the developer solution is 3 to 7, discovered by fellow researcher, Hande Aydoğmuş. To ensure that the concentration does not change abruptly due to chemical agents being depleted with consecutive development of multiple samples, a large volume of developer solution such as 500 ml should be prepared with 150 ml AZ 400K developer and 350 ml DI water.

The sample, held by a polymer tip tweezer, should be submerged into the developer solution and during development, the sample should be gently moved around in the beaker in a stirring motion. This improves the quality of the development because the dissolution of the photoresist into the mixture happens very slowly when it is stationary. This is due to the formation of an obviously visible phase around the chip which distorts the light in a way that hints at its refractive index being different from that of the solution, which indicates that natural dissipation of this separate phase into the solution is not fast enough, thus when left stationary, it effectively blocks the developer from reaching the photoresist.

The development quality could be confirmed by a quick examination with a microscope. If the development is found to be incomplete, the sample could be submerged into the developer to complete the process. This is mentioned especially because it is a common mistake to think that the light of the microscope may expose the photoresist during the examination. It is a mistake because the exposure caused by the visible light of microscope is either totally ineffective due to its frequency or negligible compared to the energy required to expose the photoresist, as it has never resulted with even the slightest indication of overexposure.

2.2.4 Inductively Coupled Plasma Etching

“Etching” is a generic name used for subtractive fabrication techniques that work in principle through selective removal of certain materials by physical or chemical means. Etching often requires patterning and coating of a mask layer that covers the features that are wished to be preserved and exposing the locations where the material is wished to be removed. There are two main categories of etching: wet etching, which is basically submerging the samples in certain aqueous solutions that would chemically react and dissolve the target material; and dry etching which is performed in specialized equipments that can combine certain gases in a confined space creating a controlled environment that reacts selectively with the target material on exposed surfaces.

Inductively Coupled Plasma (ICP) Etching is a very sophisticated dry etching tool that pumps and combine precise amounts of various gases inside a vacuum chamber and ionizes the atoms of the gas mixture by applying a powerful Radio Frequency (RF) electric field with a coil that rips the electrons off the gas atoms in the chamber, creating a plasma of radicals out of the mixture. The highly reactive radical groups created inside the chamber are then directed towards the bottom of the chamber with a platen below the chamber. Samples that require etching is placed at the bottom of the chamber where the radicals are directed towards, which results in unidirectional bombarding of the surface with ions. As a result, anisotropic etching is performed, meaning that the etch rate in a particular direction is forced to be much higher than other directions. The plasma could also be used without giving it a directional bias by disabling the platen, which would result in isotropic etching which has the same etch rate in every direction.

The device is designed to handle 4 inch wafers, therefore, any sample that needs to be etched should be fixated on top of a 4 inch diameter regular glass wafer in order to be served into the chamber. Initially, the plasma chamber itself is cleaned with oxygen plasma for possible leftovers from previous procedures. An empty

glass wafer is used during the cleaning simply because the device could not be operated when the chamber is empty. After the cleaning, the empty wafer is used again for the conditioning of the chamber with the etchants, removing the oxygen that was used for cleaning and rendering the environment ready for the actual etching operation. At this point, the empty wafer is replaced by the glass wafer on which the samples are attached with double sided sticky tape fragments. After the etching, the samples are removed from the glass surface and the process is completed.

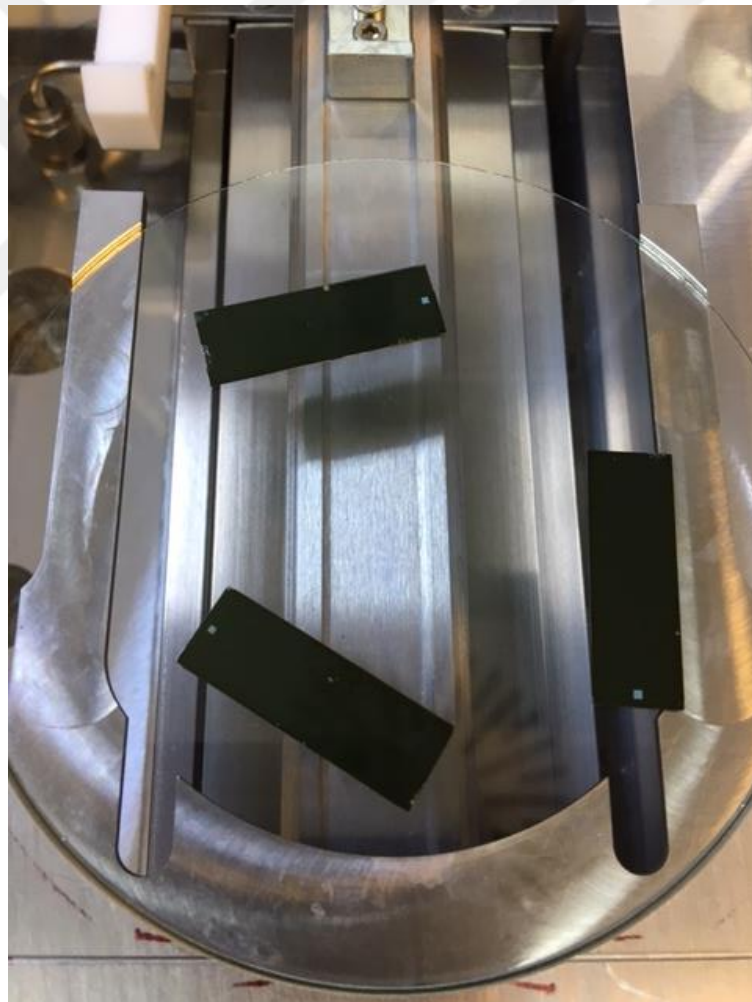


Figure 2.5: The samples attached to a glass wafer with a double sided sticky tape is seen in the photo. Notice the photoresist layer covering all the surface except the square window at the tip, in order to create the membrane seed surface.

Sticky tape fragments must be confined under the samples and should not be directly exposed in order not to contaminate the chamber with unnecessary chemicals that they might produce when exposed to the plasma. It is also recommended to use very small fragments as it becomes very challenging to detach the wafers if the sticky surface is too large, which often results with breaking the wafer while trying to detach it. I use tape fragments with approximately 5 to 10 mm² surface area in order to avoid sticking problems.

For the fabrication of the surface integrated membranes, 100 nm thick silicon nitride (SiN) is dry etched with 20 sccm CF₄ at 5 mTorr pressure and 25 °C temperature, with 400 W coil and 100 W platen power [23], all the way down to the silicon substrate using ICP which will be explained in further detail in Chapter 3. The recipe used for anisotropic etching of SiN was, thankfully, developed by Atakan Bekir Arı [23], a former graduate researcher of the same research group, now alumni, for his own research. Recipe development, especially for ICP, is a tedious and time consuming process even when the required chemicals for the etching is well established through decades. Literature review of such well established processes only presents a starting point for the recipe optimization due to differences between the individual machines and facilities. Therefore, one must generate their optimal etching recipes for the nuances and constructional detail differences specific to the machine that is used.

2.2.5 KOH Etching

KOH etching of silicon is an anisotropic wet etching process which is used to create wedge shaped grooves and membranes [24]. It is a subtractive fabrication method. Although there has been several attempts to develop analytical chemistry models to identify the reaction mechanics, it is interestingly still not well established because the extreme disparity between etch rates of crystal planes has not been explained with a satisfactory model on which there may be a consensus

[25][26]. The most promising model explains the etch rate disparity to be caused by difference between available chemical bond density of crystal planes, however it is still found unsatisfactory to relate etch rate difference of almost hundred folds while maximum variation in bond density among planes is only around 2:1 [26].

The etching process, even though it is not well defined theoretically, in practice is very well established through experimentation. The reaction is actually very simple in terms of inputs and outputs regarding the chemicals involved. Silicon on the surface reacts with Hydroxide ions in the solution. Through a series reduction and oxidation reactions, silicon is dissolved into the water in a water soluble complex of 6 hydroxide groups bound to a silicon atom. Hydrogen gas is also released out of the decomposition of water into hydroxide and hydrogen, where the former being occupied by the silicon [24].

The etch profile is a reversed pyramid shape with side angles of 54.7 degrees from the surface of the wafer. This pyramid shaped etching creates extraordinarily smooth side surfaces because of the etch rate difference between crystal planes of the single crystal silicon being as high as 80 to 100 depending on the etchant KOH solution concentration and temperature [26].

The etch process requires silicon wafer to be masked on both sides with a material that is sufficiently resistant, i.e. very slow or zero etch rate in KOH. For this purpose, SiN is used. A square window is etched away from the surface of the SiN at the bottom side of the wafer. Upon contact with KOH or in fact any other heated caustic alkali solution, the exposed silicon surface at the square window starts to etch away very slowly, taking approximately 30 hours to reach the top side of the 500 μm thick wafer at a rate calculated to be around 270 nm per minute. On the top surface, the 100 nm thick SiN layer acts as a stopping layer for the etching, and effectively becomes a 100 nm thick membrane integral to the surface.

Several different concentrations and temperatures have been tested with accordance to the literature review [27]. In the end it is found out that optimal

concentration is around 35 to 40 by weight percentage as the etch quality was the best at 65 °C. Although it is common to encounter 80 °C in many of the reviewed literature, upon trials, it had consistently caused the SiN to be etched at random points all over the surface, which causes the chip to break apart roughly 6 hours prior to the presumed end of the etching. It causes silicon nitrate being completely removed in the end due to silicon substrate beneath it being etched away almost to the point of total annihilation. The process should be immediately terminated if a major indication of random punctuation of the surface is observed at around 6th hour of etching in order to waste no time and effort because after that point the wafer basically becomes trash, as it is not possible to recover the already obliterated SiN mask.

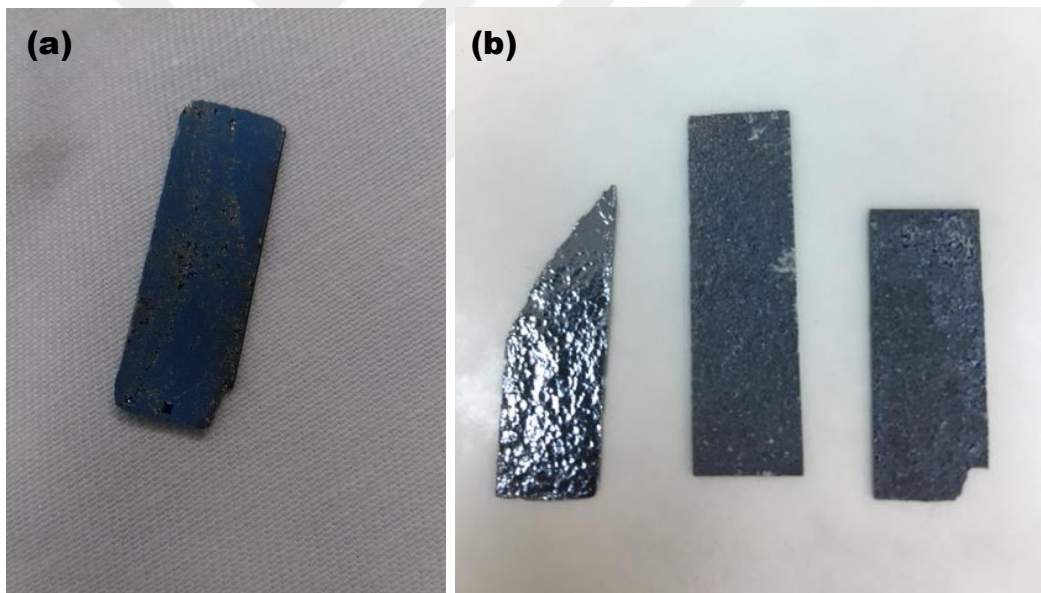


Figure 2.6: Failed KOH etching results. (a) KOH etched away random points on the mask in 12 hours. (b) Membrane continues to etch however, everywhere else also does and leaves the wafer without any SiN mask left on it. 20 Hours of etch results in rough surfaces on the right After 27 hours, the surface is smooth and shiny, seen on the left side.

While preparing the KOH solution, it is advised to be very cautious about mixing the KOH pellets with water. Mixing should be done in small portions at a time. For instance, while preparing 35% KOH solution with 600 ml water, 210 g of

KOH pellets are required and mixing is done by adding roughly one third of the pellets at a time and patiently waiting for the pellets to completely dissolve before adding the next third, and so on. I have made a mistake on my first trial when I added half of the material and upon thinking that it is going to take too long to mix, I have poured the remaining pellets as well. For a few minutes nothing happened and I have agitated the mixture by simply touching the pellets with a stirrer. I was still cautious as I knew the solving KOH in water is an exothermic process, therefore I was wearing the protective apron, mask and gloves, also the protective window of the wet bench was as lowered as possible. I am glad that I was cautious because upon touching the pellets I have probably disturbed an unstable equilibrium in the beaker and all of the pellets which were silently sitting in the bottom of the beaker vanished into the solution in a fraction of a second with a violent release of heat, causing the solution to instantly boil and erupt the interior of the beaker to the ceiling of the wet bench. No harm was done however, if I was not well protected, I would have been seriously injured as the solution is already irritating to skin and when combined with excess heat, it could have easily burnt my face and eyes.

The dissolution is faster when the water is heated to the desired temperature, 65°C, before adding the KOH. This allows mixing to happen almost instantly upon adding the pellets and slowly but continuously adding small amounts is possible without causing a rampage inside the beaker.

Last but not least, using 5 liters of water in a large beaker as a thermal bath and placing the solution in 200 ml beakers with 120 ml bathces (i.e not filling the beaker to the brim), and placing the small beakers, freely floating in the thermal bath, the large quantity of water in the bath acts as a heat sink that dissipates the excess heat from the mixing. It does not even change the temperature of the water tank significantly while mixing. This method also increases the etching stability as the bottom of the small beakers do not touch the heater plate directly, heat flow is uniform from all around the beaker. Also, three separate beakers could be heated in the water bath simultaneously, sustaining the temperature by placing the probe of the heater into the large tank.



Figure 2.7: This photo is taken from inside the thermal bath with 3 KOH etching flasks floating on top of 65°C water. The best results were achieved by this method instead of using a single layer of thermal control, this ensures the temperature is stable throughout the process.

2.2.6 Thermal Evaporation

Thermal evaporation is the primarily utilized physical vapor deposition technique in this study. It is used for coating metals, referred as metallization in micro/nano fabrication jargon. In principle, a boat shaped refractory metal such as tungsten or molybdenum containing the metal is sealed in a vacuum chamber and subjected to very high electric currents, such as 120 Amperes for gold evaporation, in order to

heat the the boat to extremely high temperatures which causes the whitish yellow thermal radiation. The heat melts the metal in the boat and even vaporizes it. The vaporized metal atoms in the vacuum chamber are not subjected to Brownian motion because of very low pressure and they travel freely in the chamber until they hit a surface, where they will stick. This forms a thin layer of the vaporized metal on every surface it could reach via a linear path. The samples are attached to a rotary chuck, 38 cm above the boat and a shutter that blocks beams of metal atoms is used to control the thickness of the metal layer on the samples.

The thickness of the coating is measured with an indirect method, by a mechanical resonator based device called a thickness monitor. The thickness monitor detects the increase in its effective mass due to coated material on top of it, changing its frequency in time [28]. This change is translated to the thickness of the coating by the device software which takes acoustic impedance, and density of the material into consideration along with an arbitrary parameter called the tooling factor. By adjusting the tooling factor according to post-process thickness measurement, the thickness monitor could be adjusted to display the thickness perfectly. This means each material has its own tooling factor, as it is the process optimization parameter specific to each different material. Apart from the thickness monitor, a better way of assuring the thickness is to know how much target metal is required for desired thickness. It is established for the specific thermal evaporator that 0.6 g of gold coats a 100 nm thick layer.

Alternative to the boats, filaments of refractory metals, clad by a metal is also used to heat the metal on its surface and evaporate it without a liquid phase. For instance, Cr is coated with by tungsten core filaments instead of boats due to its high melting and boiling temperature. The thermal evaporator in UNAM Cleanroom is contains three separate bot/filament holders which could coat up to three different materials without breaking the vacuum.

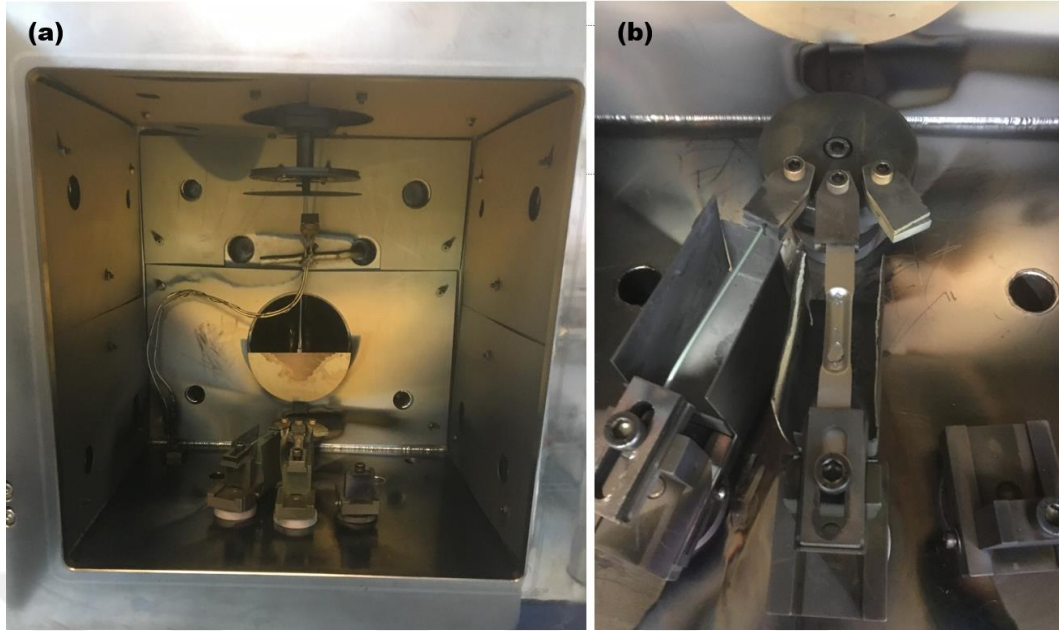


Figure 2.8: Interior of the thermal evaporator vacuum chamber. (a) on top side the rotating chuck for holding the samples is seen right above the thickness monitor. At the bottom of the chamber the target holders are present. (b) a zoomed in photograph of the target holders. A Cr filament with W core is installed on the left side. In the middle there is a boat type heater used to vaporize gold.

Due to the electric current sourced heating in this device, it is very crucial to avoid short circuits due to moveable parts around the boat holders being left in contact before the chamber is subjected to vacuum. If that is the case, both of the boats will be subjected to the electric current while only one is intended to, which causes an undesirable mixture of the two materials to be coated instead of the desired one. For instance, gold electrodes, when plated on Si and SiN substrates, are first coated with a 10 nm thick layer of Cr, which sticks to the surface of the chips better than gold itself. In that case, target holder 1 holds the Cr filament and holder 2 contains the gold boat. There is a barrier on each holder that separates the target materials from each other in order to avoid contaminating each other. These barriers are made of metal and they could be moved by hand. While attaching the boats, these barriers may move and touch each other. It is very important to check if they are in contact before sealing the chamber because it could only be realized when both of the samples are glowing hot. At that point it is too late to recover the damage done, defecting the samples.

The chamber should be left at vacuum for 1 hour after the deposition in order to allow the chamber to cool down slowly, as well as the coated samples to avoid thermal shock. This is especially important if the samples contain membranes or any sort of similar suspended thin structure because thermal effects may damage them easily.

2.2.7 Electron Beam Lithography

E-beam Lithography (EBL) is the backbone of nanofabrication. It is the nano scale equivalent of Photolithography which uses electron beams instead of UV photons to transfer the patterns due to them having much shorter wavelength than that of UV photons, allowing much smaller structures to be patterned, down to tens of nanometers by the device in UNAM.

EBL is a maskless process because the locations that are desired to be patterned are patterned by bending the electron beam via magnetic force due to electrons having charge contrary to the photons.

The device is actually a scanning electron microscope, modified by an external toolkit that enables the patterning to be conducted as well as imaging. The fundamental problem of EBL is that the same electron beam that activates the e-beam resist is also used for imaging, which may cause unwanted exposure while navigating on the surface of the sample in order to find the location at which the patterning will be done.

The polymer poly (methyl methacrylate), abbreviated as PMMA is used as the e-beam resist. PMMA, when exposed to highly energetic electrons of the electron beam, its chemical structure changes, making it vulnerable to the developer solution, namely MIBK. The development time, nominally 45 seconds, should be optimized as well as the dose at which the patterns are exposed to e-beam in order to improve the patterning resolution.

2.2.8 Ion Milling / Focused Ion Beam

Ion milling, also known as Focused Ion Beam, is a very unique etching method that does not require any masking or patterning before the operation. It is evident by its name that it is basically a nano machining tool working similar to a macroscopic scale milling machine. While actual milling is done by turning a cutting drill bit, Ion milling uses electric field accelerated heavy ions beam to perform micro and nano machining. It is capable of drilling holes and removing materials off the surface via bombardment of the surface with heavy element ions, scattering the atoms on the surface purely due to impacts of high kinetic energy particles. It is a kind of physical etching, subtractive by its nature.

The FIB in UNAM is capable of scanning electron microscopy during the milling operation, contrary to the other available nanofabrication tool EBL, allowing in situ analysis of the process. When the desired operation angle is reached, the depth and shape of milling is very simply input to the interface, via drawing the shape on the actual live SEM image. It uses rather heavier elements such as gallium and argon as ion source in order to increase its kinetic impact potential. Unfortunately this device has been broken for more than 4 months at the time this manuscript is written, disallowing its proposed use for this project.

Chapter 3

Device Fabrications

This chapter is dedicated to describe the coherence between individual fabrication techniques as it pivots around the fabrication flow of previous membrane devices, coplanar waveguides and finally focuses on the advanced hybrid of the two, a CPW mounted on a surface integrated membrane.

3.1 Previous Coplanar Waveguide Fabrications

Within the context of microwave imaging project, CPW devices were first designed and fabricated by cumulative effort of NEMS Lab members including myself, two years ago in an attempt to create an alternative microwave resonant sensor to the bulky microstripline resonators which we were focused on back then. CPW as previously explained, have a single layer construction, which has a significant advantage over the microstripline in terms of greatly improved precision due to its systematic approach on fabrication, decreasing the susceptibility to human error.

The device consisted of a soda-line glass microscope slide as substrate and three electrode lines covering the distance along the longitude of the glass from one end to the other. The electrode in the middle is supposed to be the signal carrier whereas the other two are grounds and are symmetrical with respect to the signal line. This device was designed similar to the microstripline resonators that we were fabricating on a daily basis, having two SMA ports on each end, mounted on the device without an intermediary medium such as a separate PCB to mount the device on.

Fabrication starts with cleaning the glass with standard wafer cleaning methods explained in the previous chapter (2.2.1 Wafer Cleaning), basically acetone bath with ultrasound cleaning followed by acetone and IPA wash. Afterwards, AZ-4562 of 6 μm thickness is coated on one surface with spin coating. The photoresist is then patterned by photolithography with the CPW design with minimal effort for alignment because the mask was designed few millimeters excess in length than the glass substrate width. CPW is metallized by Cu electrodes by e-beam evaporator, which is an alternative physical vapor deposition technique to thermal evaporator, however as we have later proceeded to gold electrodes, e-beam evaporation became irrelevant to this thesis, thus not explained in Chapter 2.

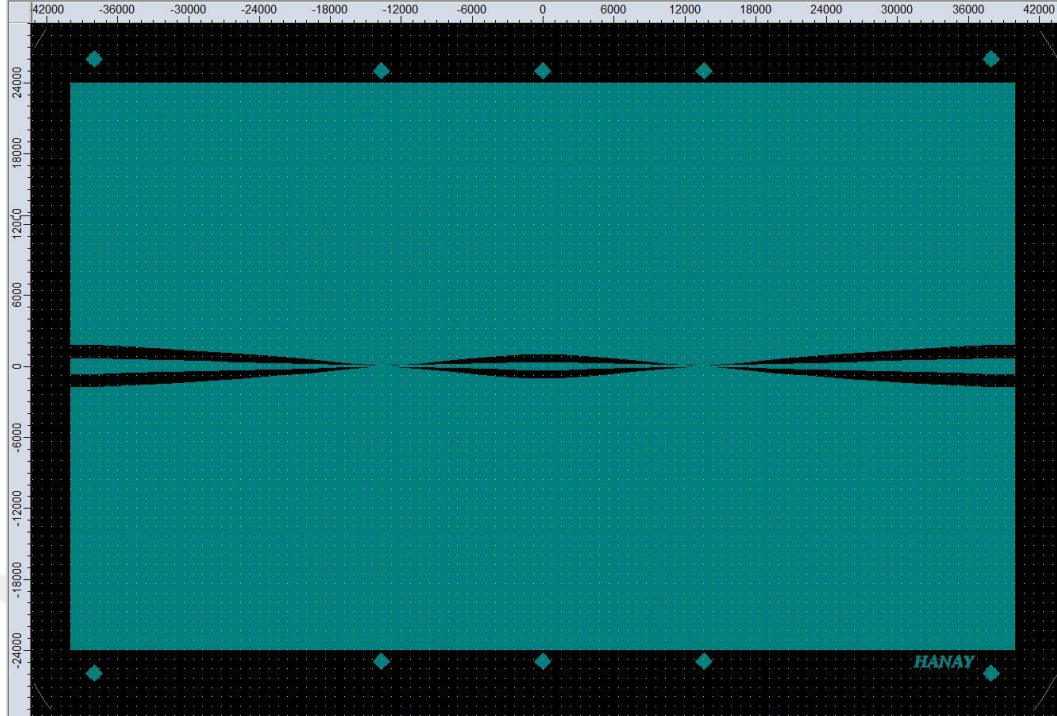


Figure 2.1: Tapered CPW design. Units displayed on the ruler are in micrometers. The locations at which the particles are detected are designed to be tighter in order to increase susceptibility to the changes in frequency

These devices were later found to be useless during that time as we lacked the instruments and know-how on actuation and measurement of such devices. Later during the summer of 2017, Mehmet Kelleci and I have briefly worked on improving the CPW design with a tapered structure what would focus the sensitivity to a single point on the resonator, hoping that it would make it possible to measure the CPW's. We have later understood during spring 2018 that our inability to measure was due to absence of proper instruments. All in all, the new design was also exactly the same in terms of fabrication, only with a different photomask pattern.

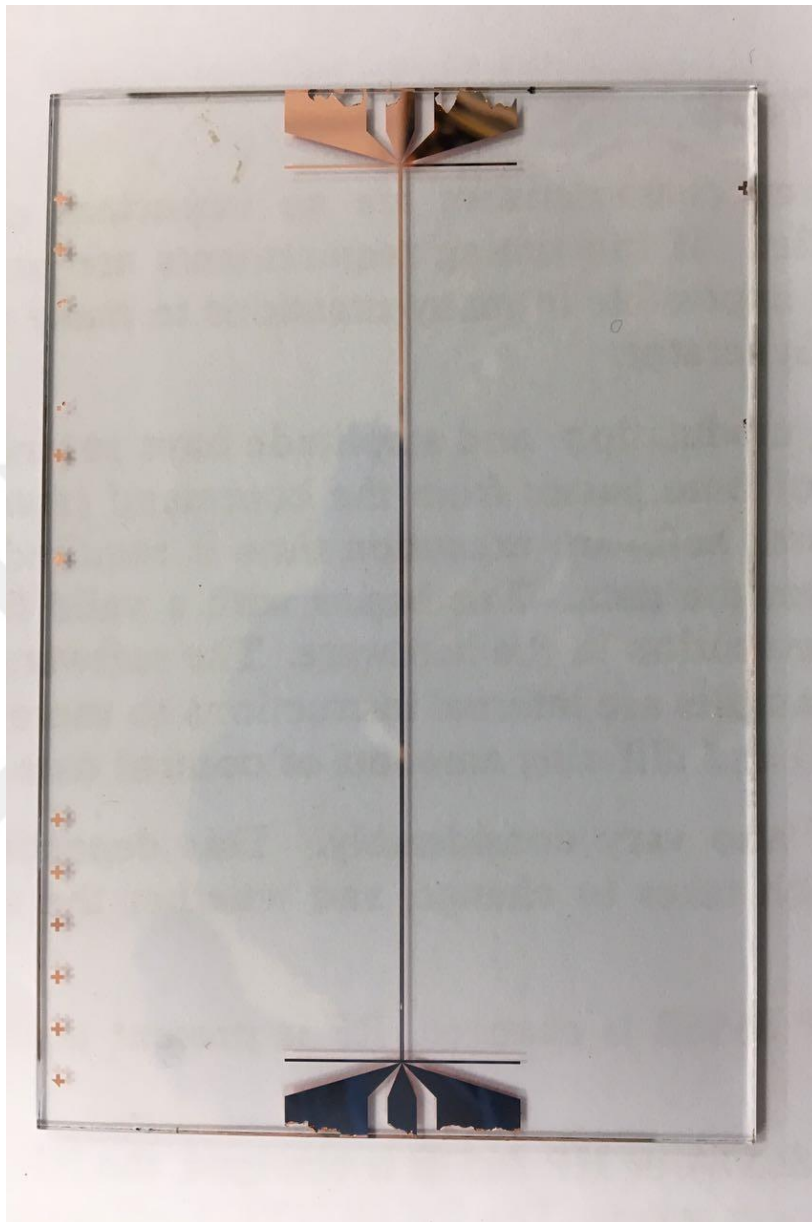


Figure 3.2: One of the previous CPW designs with narrow ground carriers. Fabricated on soda lime glass with copper electrodes. The edges are torn while trying to solder the SMA ports.

3.2 2D Membrane NEMS Resonator Fabrication

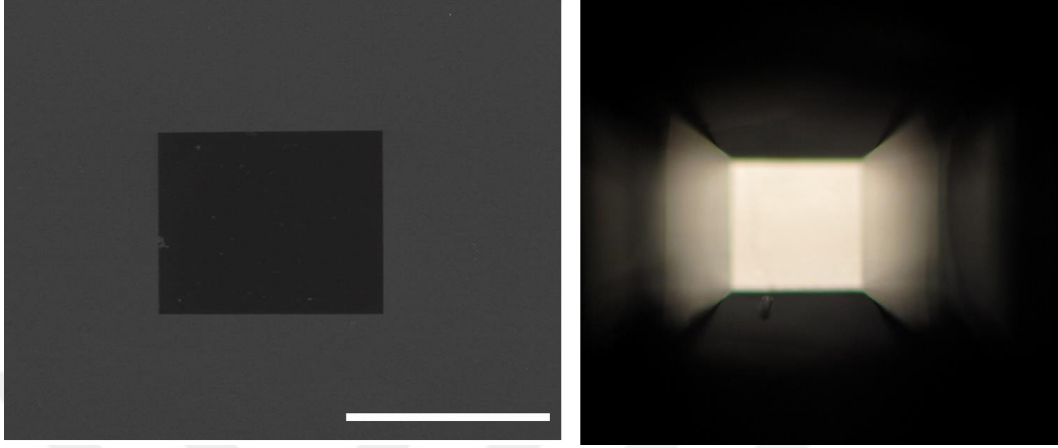


Figure 3.3: A membrane fabricated by Hande Aydoğmuş. On the left, SEM image of the membrane from the top side is seen with scalebar of 40 μm . On the right, a backlit image of the membrane showing the smooth surfaces at the sidewalls.

The first membrane fabrication experience that laid the foundation of the surface integrated membrane microwave CPW resonators started with my involvement in the 2D Membrane Nanomechanical Resonators project. The project was first started by efforts of my colleague Hande Aydoğmuş as she struggled to create membranes out of SiN coated silicon wafers. Although I had been conducting a totally separate research back then, I was curious and became interested in that project as well, and I have spent time and effort in nanofabrication phase while learning the KOH etching from Hande.

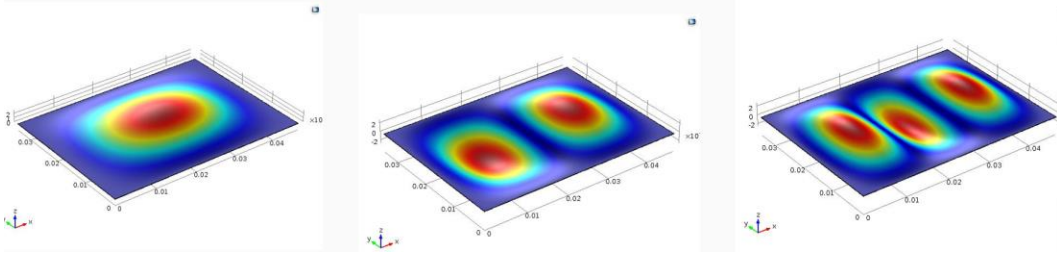


Figure 3.4: COMSOL Multiphysics finite element analysis of the membranes were performed by Hande Aydoğmuş for her project, developing two dimensional nanomechanical resonators.

The device consists of a surface integrated SiN membrane created through spin coating of 6 μm AZ 4562, followed by photolithography of square windows at the bottom side of the ultra low stress SiN coated (100) oriented silicon wafers. Upon photoresist development, creating a square window on the photoresist, the samples are dry etched off the SiN through the Photoresist window with ICP etching with 20 sccm CF_4 at 5 mTorr pressure and 25 $^{\circ}\text{C}$ temperature, with 400 W coil and 100 W platen power for 1 minute [23]. Afterwards, samples are placed in 40% KOH solution at 80 $^{\circ}\text{C}$ and it was purportedly completed in approximately 10 hours, creating a 100 nm thick SiN membrane on the top side of the wafer.

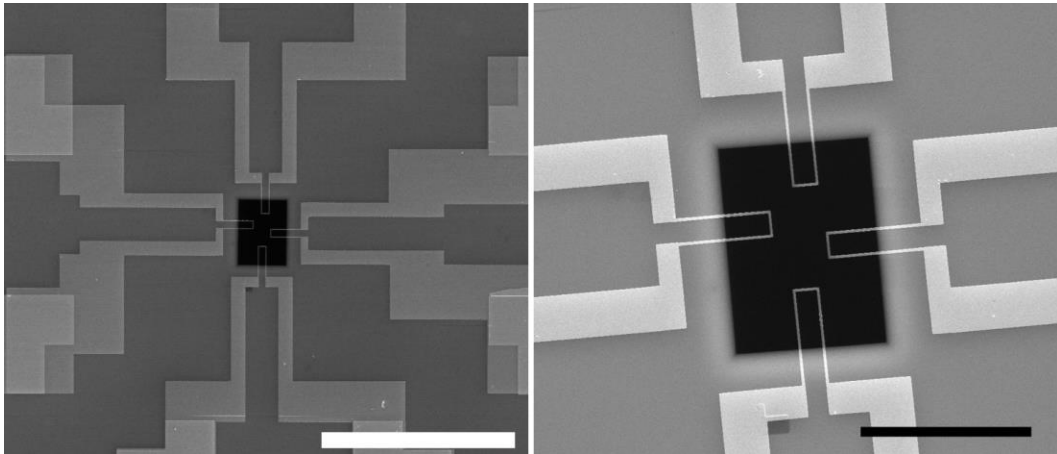


Figure 3.5: Gold electrodes patterned and metallized on top of the membrane. On the left, two stages of the alignment process is seen by the contrast difference on the intersection of the two layers. The scalebar is 100 μm in length On the left a zoomed in version of piezoresistive electrodes are seen with scalebar of 30 μm .

At this point, the membranes were ready to be processed via EBL, therefore the membrane surface is coated with 300 nm PMMA layer. The PMMA is then patterned with EBL in order to create the contact pads with a low resolution yet high speed patterning which is appropriate for crude and large patterns such as contact pads. After the patterning, the PMMA is developed and the contact pads are metallized with 80 nm Au with 10 nm Cr as adhesion layer. The coated samples are then placed in acetone bath in order to remove the PMMA along with the excess metal on top of it, a post metallization process called lift-off. It is crucial not to use ultrasound cleaner which is normally used after lift-off to ensure that the edges of the metal patterns do not have dangling leaflets of excess metal, because ultrasound cleaner certainly damages the membrane without exception regardless of exposure time. After the lift-off, a second EBL patterning is made in order to create piezoresistive gold electrodes on top the membrane. For that purpose once again the chip is spin coated with 300 nm PMMA and this time a slow yet precise patterning is performed as there were much smaller and delicate features. The pattern is aligned to the correct location according to the alignment markers printed on the first EBL process along with the contact pads. After the patterning and development, 40 nm Au is coated, finalizing the 2D NEMS resonator fabrication upon a successful lift-off.

3.3 Fabrication of Surface Integrated Membrane Microwave Coplanar Waveguide Resonator

After the publication of our article “Towards Microwave Imaging of Cells” which reports a series of fabrication and experimental results creating a novel imaging and characterization technique, namely “*Impedance Imaging*”, through distinguishing between types of human cancer cells [19], alternative resonator structures are proposed to improve the shortcomings of microstripline resonators. A bowtie CPW proposed in a recent manuscript [21] by Bhat, A et.al. As I had previous experience on CPW fabrication and design, I was appointed to develop a

better device than which is presented in the mentioned article because we were capable of much more intricate patterning via EBL which was not presented in the original work. I have therefore been influenced by however improved upon their design with the nano scale electrical hotspot instead of their tens of microns wide design.

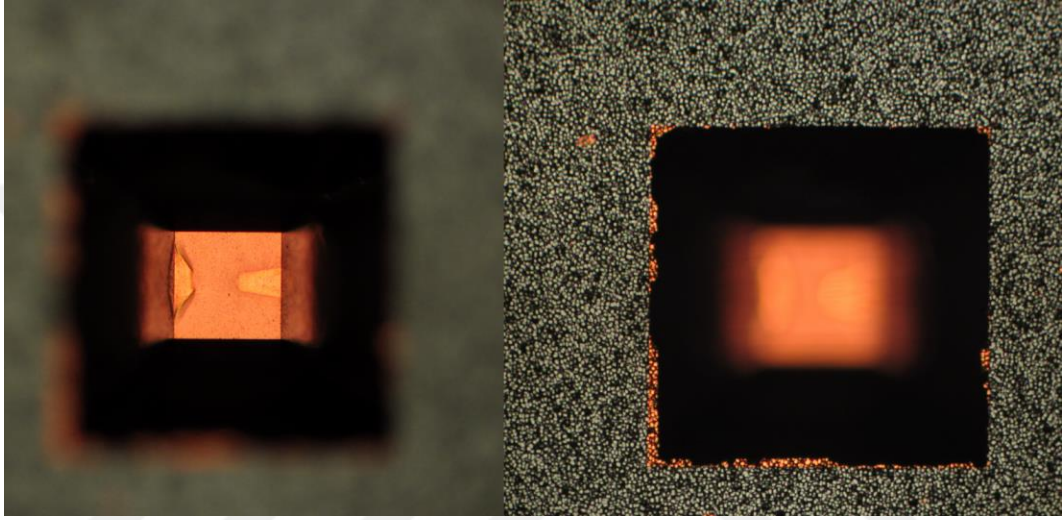


Figure 3.6: Back side of the membrane is shown with two different focus distances. Left side, focusing at the membrane and the structures on top of it, while the right side is focused on the bottom surface of the wafer. The square window is seen to be keeping its form after the etching.

This newly proposed CPW device is built on 200nm ultra low stress Silicon Nitride (Si_3N_4 or SiN for convenience), coated over 0.5 mm thick Silicon (Si) wafer, on both sides. $3 \times 1 \text{ cm}^2$ wafer slices are patterned by Photolithography on the back side to create a square window on 6 μm thick photoresist AZ 4562. SiN exposed by the window is etched away through ICP with the same recipe by Atakan Bekir Arı, previously mentioned. Upon cleaning the wafer of photoresist with acetone, IPA and DI water, samples are placed in 35% KOH aqueous solution, maintained at 65°C double bath for 30 hours. KOH etching of Si substrate through the window on the back side of the wafer causes front side SiN being left with nothing beneath acting as a stopping layer for the etching as it is practically resistant to KOH etching, essentially forming a membrane on the top surface of the wafer.

The second phase starts with Photolithography patterning of CPW resonator structure on the top surface, with alignment for intersecting the membrane with the sensing tip of the resonator. The aligned pattern is then developed later to be thermally metallized by 10nm Cr adhesion layer and 120nm Au, forming the CPW electrodes upon lift-off. Third phase of fabrication is the patterning of nano structures via E-beam Lithography over the micro membrane where tip of the signal electrode reaches to the ground as close as 300 nm, creating a signal hotspot.

At the hotspot between the electrodes which are constructed on top of the 100 nm thick membrane, a hole with diameter of 200 nm is proposed to be drilled by Focused Ion Beam (FIB) to allow a fluid to flow through the hotspot, carrying nanoparticle analytes.

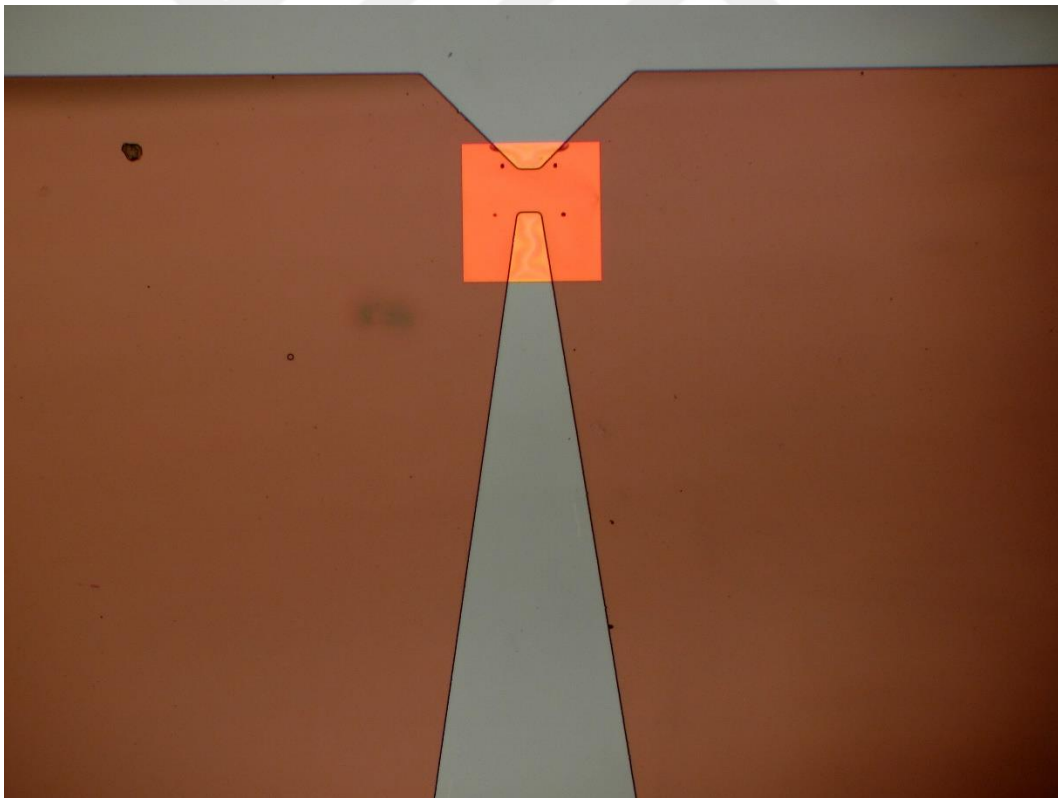


Figure 3.7: Top side of the membrane integrated surface of the wafer with photoresist, patterned for gold deposition. The electrodes intersection point is aligned to be on top of the 100 nanometers thick membrane.

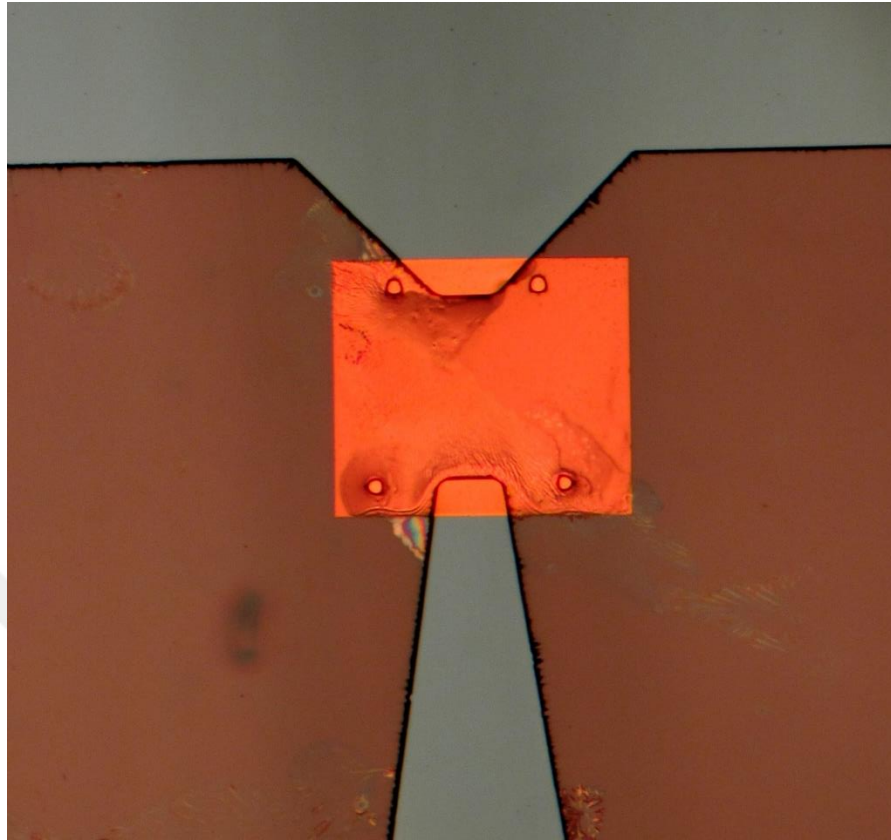


Figure 3.8: Another optical microscope image of the signal hotspot after the photoresist is developed. Notice the small circles around the hotspot; they are alignment markers which were deemed too small for this specific photolithography process.

Due to 4 months long failure of FIB, Ion Milling could not have been performed. Also the EBL has been offline for 2 months starting exactly the day I have reached EBL phase of the fabrication. These machine failures disrupted the project, creating huge time management issues leading to much later-than-expected submission of this thesis with unfortunately incomplete results. This is the grim nature of purely fabrication focused research, which might have also included experiments with tens of nanometers sized biological packages, exosomes in order to create a basis for an exosome analysis tool.

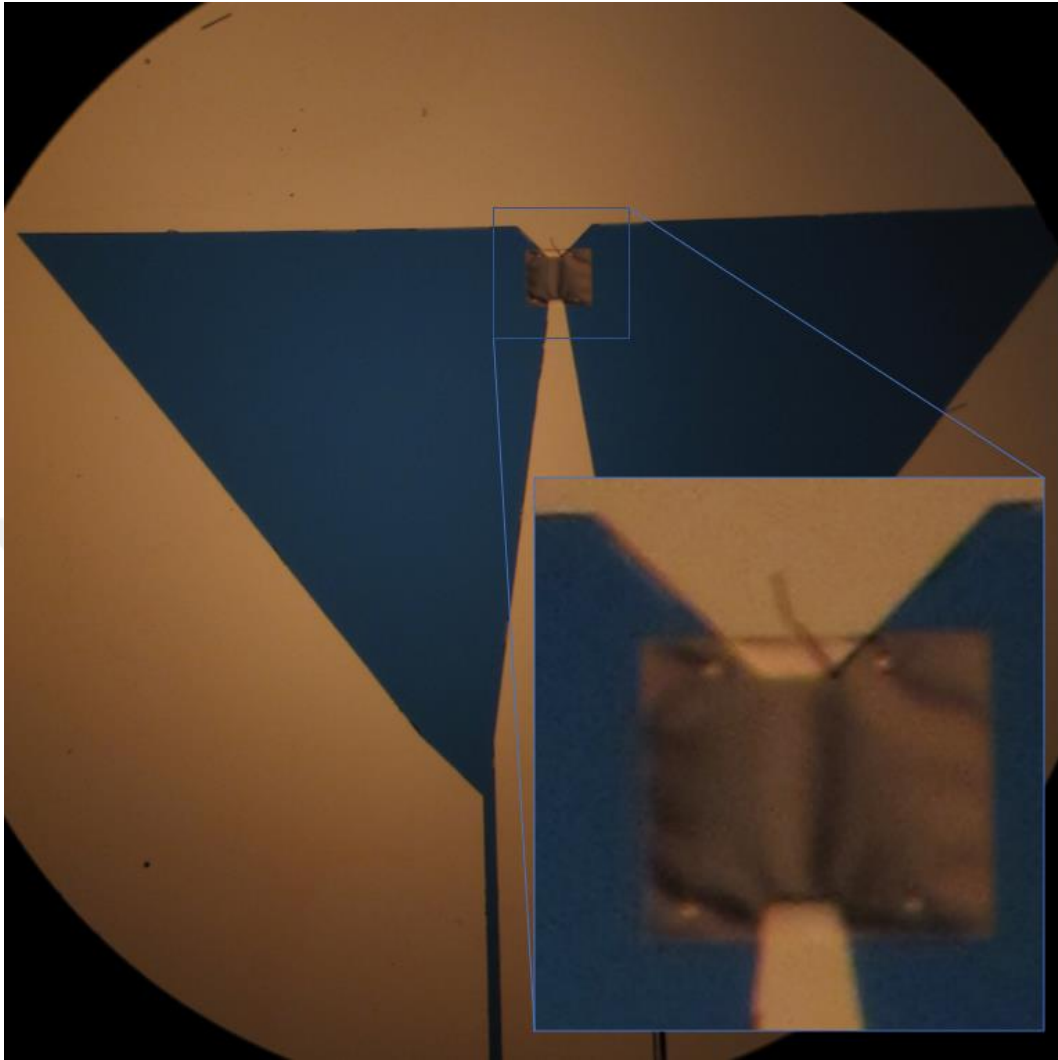


Figure 3.9: The gold electrodes on top of the 100 nm thick, 200 nm wide square membrane are shown in this figure at 5x magnification. The alignment markers are also visible in this specimen even though they are distorted and circular instead of the desired square shape. This appears as a very promising specimen however, in fact the rest of the signal line is in short circuit with the ground electrode because during the lift-off, the residue is left on the edges of the photoresist, and leaving contact after the photoresist is removed. The solution for this problem is normally to bathe the sample in the ultrasound cleaner to, however the membrane is broken instantly when the ultrasound cleaner is applied therefore, the lift-off quality is very difficult to improve.

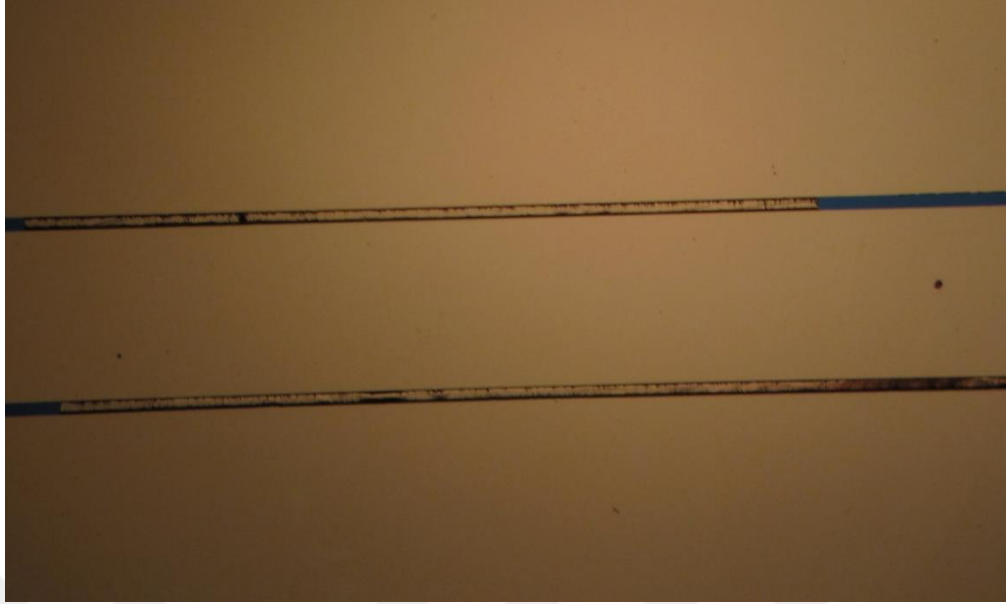


Figure 3.10: The 100 μm wide separation between electrodes is seen in this image. Notice the undesired gold contact between the electrodes. It is standard for Photolithography lift-off, however the usual method used for removing them also destroys the surface integrated membrane, therefore is unavailable for this fabrication.

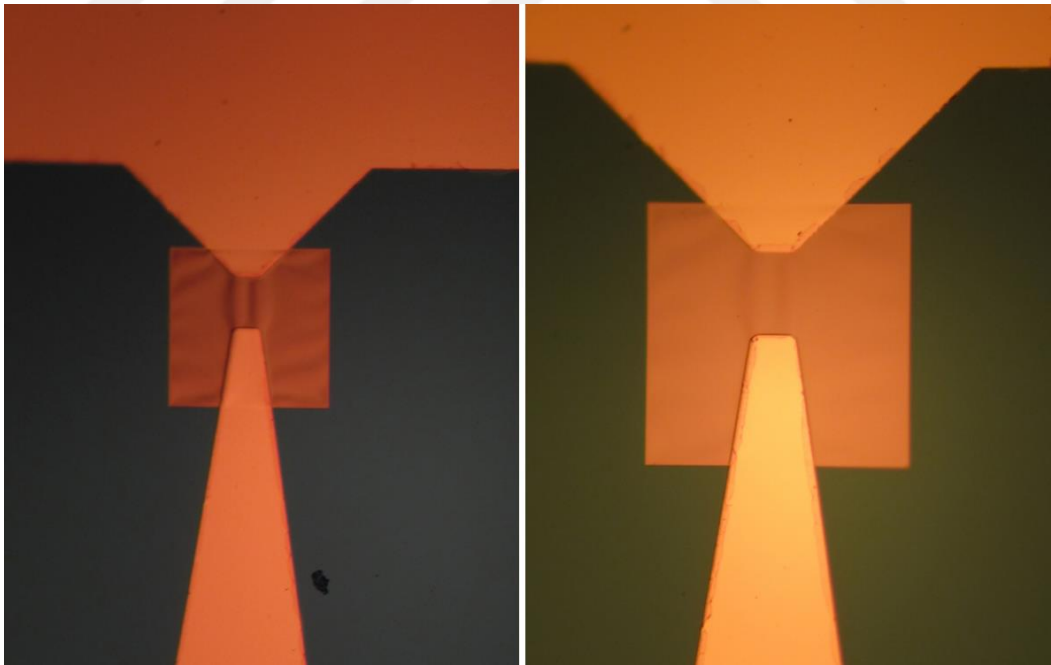


Figure 3.11: Two more of the devices after electrodes are metallized. The one on the left clearly shows the tension on the membrane surface, caused by the cooling of the electrodes after the evaporation.

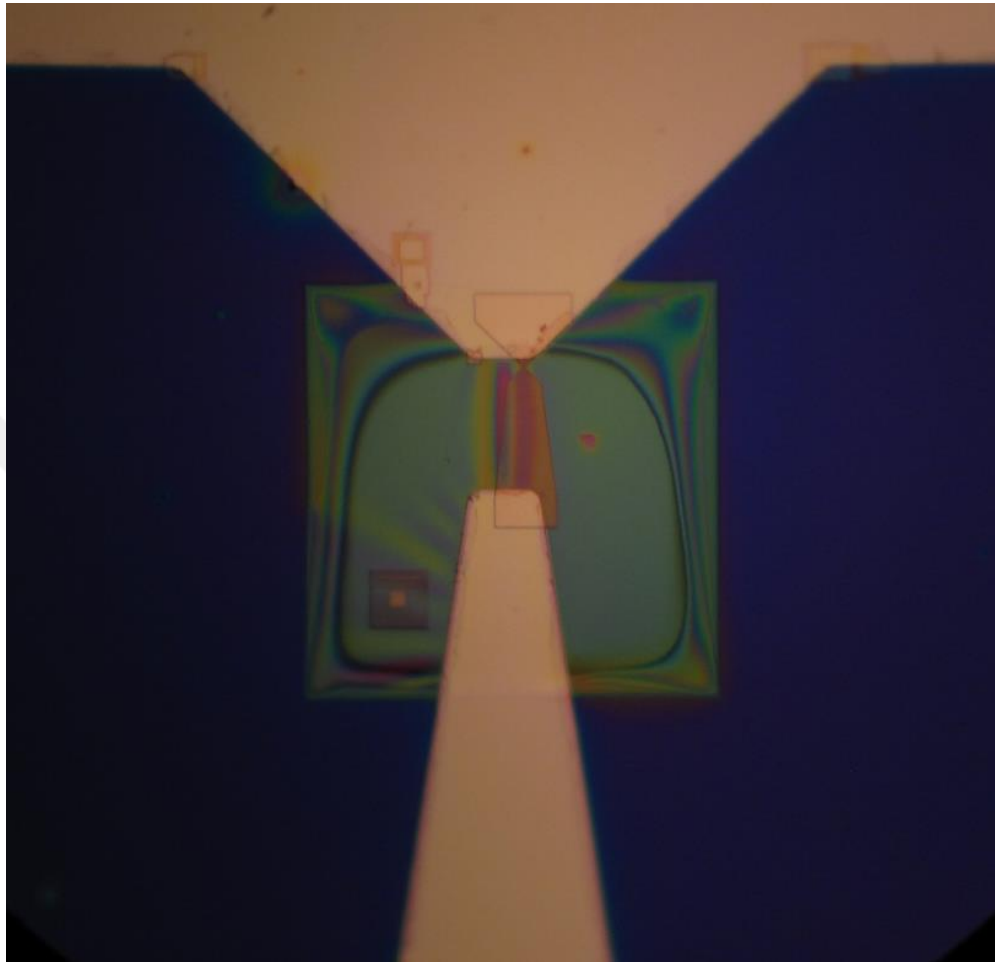


Figure 3.12: Results of the EBL in the sensory region, on top of the membrane. The pattern in this figure was found adequate for metallization, which eventually turned out to be defective and probably either the exposure or development might have been actually inadequate. This is difficult to detect due to the color patterns created by the thin film membrane beneath.

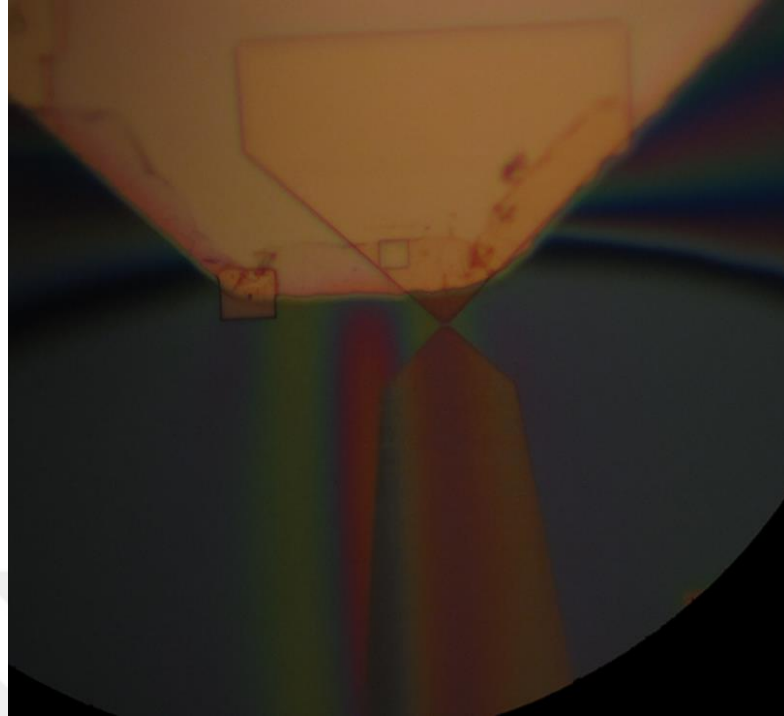


Figure 3.13: A higher magnification of the structure. The color patterns on the patterned locations is attributed to the thin membrane layer beneath however, probably it was due to PMMA leftovers in truth, judging by the results (Figure 3.14)

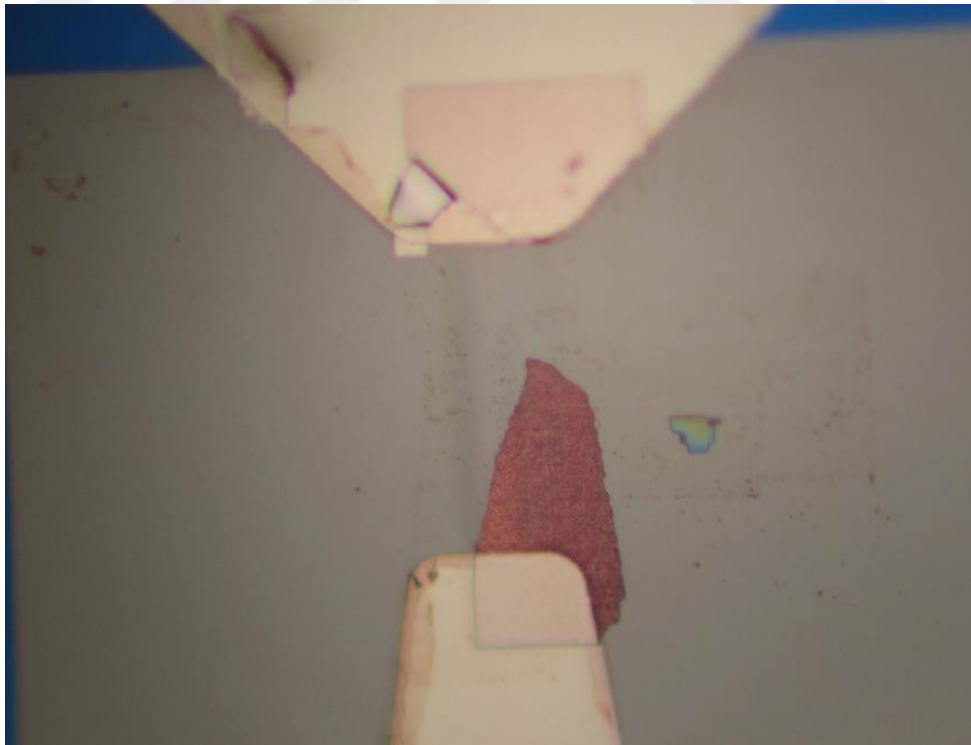


Figure 3.14: The result of the thermal evaporation of gold. The gold on top of the preexisting electrodes was perfectly smooth, however on top of the membrane itself, the gold is crumbled and some portions did not even survive lift-off.

Chapter 4

Conclusion and Future Work

4.1 Conclusion

This project was undertaken in order to design and develop a novel method of nano sensing with microwave CPW resonators. The designed device consisted of feature sizes ranging from 100 nm to 30 μ m – a range that spans five orders of magnitude. This large range brought a yet wider range of fabrication techniques because of the fundamental and technical capabilities of each method being limited to a narrow band of scales. This resulted in a fabrication plan that utilized each method in its own specialized precision band. For instance, fabrication of the CPW electrodes consisted of two types of lithography; photolithography is used in order to fabricate the main body of the CPW on which the tip of the resonator is featured down to 100 μ m, which is then followed by EBL to decrease that gap to 300 nm. This path is selected because a single run of photolithography is fundamentally bound with ultraviolet wavelength of the photons. Diffraction around the small features cause a blurry and imprecise image to be transferred to the surface. Effects of dispersion is detrimental to the precision when the

wavelength of the wave – in this case ultraviolet photons – is comparable to the feature size. In order to overcome this phenomenon, EBL uses beams of accelerated electrons which has sub nanometer wavelengths, allowing nano scale patterning.

Fabrication plan is executed with the first phase that covers fabrication of the membrane. It has been selected to be the first step because it was considered to be practically impossible to perfectly align the membrane to the sensory region due to its etching being performed from the backside. On the other hand, when the membrane is preexistent during CPW patterning, it is very much possible to detect and align the membrane to the desired location.

As planned an ultra-low stress SiN coated wafer is used as the substrate for the device. On the back side of the wafer, spin coated 6 μm thick photoresist AZ 4562 is patterned via photolithography. The pattern which basically is a square window is then dry etched with the photoresist as the etching mask. After the dry etching, the SiN layer in the etching window is removed and the exposed silicon substrate is etched anisotropically via KOH, until a square membrane is formed on the top side of the wafer. In this study it is shown that for this specific membrane fabrication, the optimal etching conditions is 65°C and 40% weight ratio aqueous KOH solution. Two layers of thermal baths is used to sustain the 65°C throughout the hours long process. These conditions result in a slow yet steady etch which takes up to 30 hours while yielding 100% sample survival rate in three batches of six samples.

The microfabrication of CPW followed the membrane phase with photolithography of spin coated 6 μm thick photoresist AZ 4562. The CPW pattern was transferred to the top side of the wafer with alignment of the sensory region to the membrane surface, done manually with optical micro imaging tools of the mask aligner. The photoresist with the patterns is then used for metallization of 100 nm Au electrodes. It has been noted that due to membranes being instantly damaged when ultrasound cleaning is applied, lift-off of centimeters long 100 μm wide gaps between the ground and signal electrodes are

often incomplete. This was an expected challenge of thin membrane being constructed before metallization, however it was the only feasible design for alignment.

In the following EBL process, the nano features have been patterned on the sensory region and through optical imaging, the development of the PMMA coating after the patterning is observed to be adequate for metallization (Figure 3.12 and 3.13). After the metallization was done however, the adequacy of the EBL process was questioned due to the results. The gold plated on top of the membrane was found to be crumbled and even mostly inexistent especially at the ground electrode side. This might also been due to thermal expansion of the membrane during the thermal evaporation, which might cause sagging of the membrane and after the membrane is cooled down to the room temperature. The gold plated on top of it was probably simply crumbled with the residual compressive stress caused by compression of the layer on which it was constructed (Figure 3.14).

Nanofabrication was the last phase of the fabrication chain. EBL process and attempts of nanofabrication has provided a significant knowledge base for nanofabrication on thin membranes. Therefore this thesis is dedicated to transferring practical information to the next generation of researchers, in order to pave the way to new scientific discoveries and engineering achievements in micro and nano sensing field.

4.2 Future Work

The proposed prospect for the devices designed and fabricated in this work was initially planned to characterize exosomes as they could contain all sorts of metabolic products which may be used to determine the activity and health state of the cells that secreted them. Therefore, the proposed device may be capable of

becoming a new diagnosis tool for microbiology or medicine fields in the future of this project.

With that perspective, these new sensors should be thoroughly tested for their sensory capabilities. For this purpose an appropriate instrumentation study should be conducted regarding the circuitry and controller design. There has actually been some minor attempts in terms of actuation and readout components. A circulator is used to deliver the actuation signal while rerouting the reflected signal to a separate channel. If this readout approach with a circulator is to be used however, the limitations and compatibility with the the specific sensor should be addressed as soon as possible in order to develop a robust controller which tracks the resonance frequency in real time. Also for the controller design, PLL structure might not be the only viable option and a closed loop operation which tracks the frequency via minimizing the reflected portion of the signal. If that will be the case, capabilities of reflectivity tracking controllers should be investigated and compared to the PLL and the better option should be determined.

Alternative to simple particle detection, the nanopore when placed on the membranes could be used for DNA sequencing with appropriate modifications to the system. These modifications should consist of a nanofluidic channel system that prepares the DNA strings for sequencing before they reach or when they are within the sensory region. For such an application the requirements and biological limitations should be investigated as it would become a highly interdisciplinary research.

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