

TOWARDS MODE SHAPE INDEPENDENT NANOELECTROMECHANICAL MASS SPECTROMETRY UNDER ATMOSPHERIC CONDITIONS

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Towards mode shape independent nanoelectromechanical mass spectrometry under atmospheric conditions

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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

TOWARDS MODE SHAPE INDEPENDENT NANOELECTROMECHANICAL MASS SPECTROMETRY UNDER ATMOSPHERIC CONDITIONS

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Nanoelectromechanical systems are being utilized in mass sensing applications owing to their small footprints and high mass sensitivities, targeting masses that are unreachable by most other techniques (>10 MDa) while also being able to measure masses in the kDa range. Recently, these sensors were deployed under atmospheric conditions by integrating a polymeric focusing lens, which increased capture efficiencies and decreased the system cost, both of which have been significant challenges in the NEMS-MS field. However, when deployed under atmospheric conditions, the displacement profiles of these sensors become dependent on the environment due to dissipative effects, unlike symmetric displacement profiles that follow the mode shapes in vacuum. Since the frequency shift by particle landing depends on the landing position, the corrections to the governing equations make the analysis more accurate, especially when the quality factor of the sensor is low. Therefore, devices that enable single mode mass sensing under atmospheric conditions remove the need for corrections and enable mode shape independent mass sensing. Here, we propose devices with uniform regions in their mode shapes, which removes the position dependency when calculating the mass of a landing particle. The uniform mode shapes are analyzed and validated by simulations and experiments using 200 nm fluorescent polystyrene nanoparticles whilst considering the landing positions of the particles and frequency shifts. After the uniform mode shape validation, we conducted 40 nm gold nanoparticle mass sensing experiments. Therefore, the proposed devices allowed us to perform mode shape independent mass sensing under atmospheric conditions.

Keywords: NEMS, Nanomechanical Mass Sensing, Atmospheric Pressure Mass Spectrometry, Mechanical Mode Shape, Single Mode Mass Sensing.

ÖZET

ATMOSFER KOŞULLARI ALTINDA MOD ŞEKLİNDEN BAĞIMSIZ NANOELEKTROMEKANİK KÜTLE SPEKTROMETRİSİNE DOĞRU

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Nanoelektromekanik sistemler, küçük ayak izleri ve üstün kütle hassasiyetleri sayesinde kütle algılama uygulamalarında kullanılmaktadırlar ve diğer birçok teknikle ulaşılamayan (>10 MDa) kütleleri hedeflerken, kDa aralığındaki kütleleri de ölçebilirler. Son zamanlarda, bu sensörler, her ikisi de NEMS-MS alanında önemli zorluklar olan yakalama verimliliklerini artıran ve sistem maliyetini azaltan bir polimerik odaklama merceği entegre edilerek atmosfer koşulları altında konuşlandırıldı. Bununla birlikte, atmosfer koşulları altında konuşlandırıldıklarında, vakumdaki mod şekillerini izleyen simetrik yer değiştirme profillerinin aksine, bu sensörlerin yer değiştirme profilleri, enerji tüketen etkiler nedeniyle ortama bağımlı hale gelir. Parçacık inişiyle frekans kayması iniş konumuna bağlı olduğundan, ilgili denklemlerdeki düzeltmeler, özellikle sensörün kalite faktörü düşük olduğunda analizi daha doğru hale getirir. Bu nedenle, atmosfer koşulları altında tek mod kütle algılaması sağlayan cihazlar, düzeltme ihtiyacını ortadan kaldırır ve mod şeklinden bağımsız kütle algılama sağlar. Burada, bir iniş parçacığının kütlesini hesaplarken konum bağımlılığını ortadan kaldıran, mod şekillerinde tekdüze bölgelere sahip cihazlar öneriyoruz. Tekdüze mod şekilleri, parçacıkların iniş konumları ve frekans kaymaları dikkate alınarak 200 nm floresan polistiren nanoparçacıklar kullanılarak deneylerle ve simülasyonlarla analiz edildi ve doğrulandı. Mod şeklinin tekdüzeliğini doğruladıktan sonra, 40 nm altın nanoparçacık kütle algılama deneyleri yapıldı. Bu nedenle, önerilen cihazlar atmosfer koşulları altında mod şeklinden bağımsız kütle algılaması yapmamızı sağladı.

Anahtar sözcükler: NEMS, Nanomekanik Kütle Algılama, Atmosferik Basıncı Kütle Spektrometresi, Mekanik Mod Şekli, Tek Mod Kütle Algılama.

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Chapter 1

Introduction

This thesis introduces mode shape independent mass sensing under atmospheric conditions by nanoelectromechanical systems (NEMS) using a nanoelectromechanical device architecture called paddle NEMS devices. Paddle NEMS devices eliminate the mode shape dependency in mass sensing applications, unlike other architectures such as doubly-clamped beams and cantilevers. The motivation behind this architecture is presented in Chapter 1. Device design, simulations, and fabrication processes are discussed in Chapter 2. Then, the experimental details and the theory behind the paddle NEMS devices are discussed in Chapter 3. After building the theory, a uniform mode shape validation and mass sensing experiment using a single paddle NEMS device and 200 nm fluorescent polystyrene nanoparticles (F-PSNP) is conducted. Next, the uniform mode shape validation experiments for double paddle NEMS devices using 200 nm F-PSNP and mass sensing experiments using 40 nm gold nanoparticles (GNP) are conducted, and the experimental results are presented in Chapter 4. Lastly, the conclusion is made, and possible future works are discussed in Chapter 5.

1.1 Nanoelectromechanical Mass Spectrometry

Nanoelectromechanical mass spectrometry (NEMS-MS) is a powerful technique for the detection of analytes ranging from kilo-Daltons (kDa) to Giga-Daltons (GDa) [1, 4–12]. The ability to measure such a broad range makes this technique a potent alternative to conventional mass spectrometry (MS) systems since commercial MS systems are not capable of detecting particles that are heavier than 100 MDa due to large mass-to-charge ratios (m/z) [13–15]. Therefore, NEMS-MS fills a critical technological gap by successfully performing measurements in this range. This ability makes NEMS-MS a great candidate for the characterization of heavy particles (*i.e.* >10 MDa) such as engineered metallic, ceramic, polymeric and biological particles including but not limited to viruses which cannot be targeted by conventional mass spectrometry systems. Therefore, it is important to develop the NEMS-MS further to make it more versatile and increase the variety of the device architectures for engineered and biological nanoparticle detection and characterization, especially under atmospheric conditions.

1.2 Atmospheric Pressure Mass Spectrometry by Nanoelectromechanical Systems

Our recent study enabled the NEMS-MS system to work entirely under atmospheric conditions while resolving problems associated with the small size of the NEMS sensors. The NEMS devices have at least one sub-micron dimension and therefore have a rather small surface area. While this feature enables ultra-sensitive mass measurements [5], it results in low particle capture efficiencies [1]. Prior to atmospheric pressure NEMS-MS, several techniques were deployed to increase the capture efficiencies such as ion optics and aerodynamic lensing via differential vacuum systems [8, 9, 16]. These techniques successfully deliver the target analytes to the capture area of the devices and increase the capture efficiencies to some degree; however, it also increases the overall cost and size of the whole system.

On the other hand, atmospheric pressure NEMS-MS approach deployed the whole system entirely under atmospheric conditions owing to the integration of the polymeric lensing structure on top of the chip inspired by approaches in other fields such as micro 3D printing and nanopatterning [17, 18]. The polymeric lensing structure plays a crucial role since ion optics do not work unless deployed inside vacuum systems [19, 20]. Unlike other lensing techniques, the integrated polymeric lens facilitates the focusing towards the active area by getting charged by the incoming ions. This integration has not only solved one of the major problems of NEMS-MS systems, *i.e.* bulky vacuum elements but also enabled to achieve record capture efficiencies compared to the previous state-of-the-art [1]. The overall system can be seen in Figure 1.1 [1].

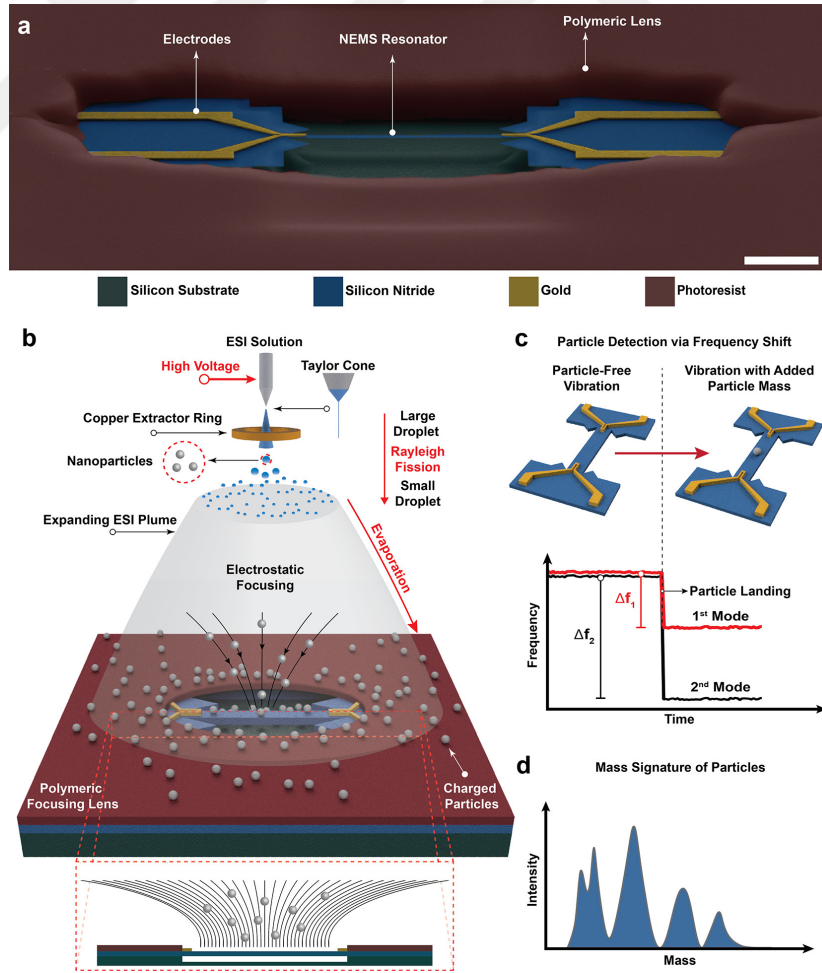


Figure 1.1: Overall atmospheric pressure NEMS-MS system. Taken from Figure 1 of reference [1].

The integration of the polymeric lens focuses the particles towards the sensing area of the NEMS device while being operated entirely under atmospheric conditions. The comparison between the lensing vs. non lensing experiments can be seen in Figure 1.2 [1]. It can be seen that the polymeric lens significantly increased the number of events per unit time compared to the non lensing case.

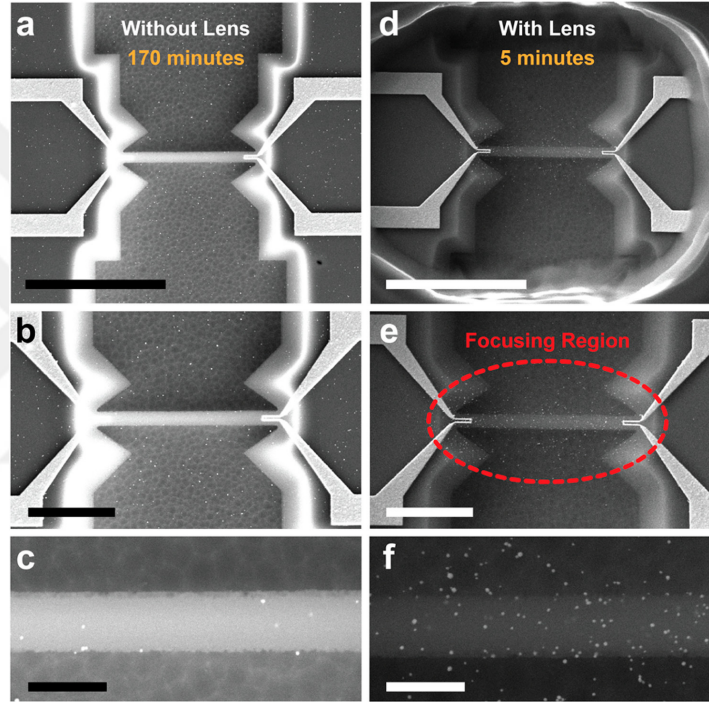


Figure 1.2: Lensing vs. non lensing experiments. Taken from Figure 3 of reference [1].

In the atmospheric pressure NEMS-MS work, after validating the system in terms of analyte focusing, nanoparticle measurements took place. These measurements consist of 20 nm and 40 nm gold nanoparticles (GNP) and 100 nm fluorescent polystyrene nanoparticles (F-PSNP). The mass and diameter histograms can be seen in Figure 1.3 [1].

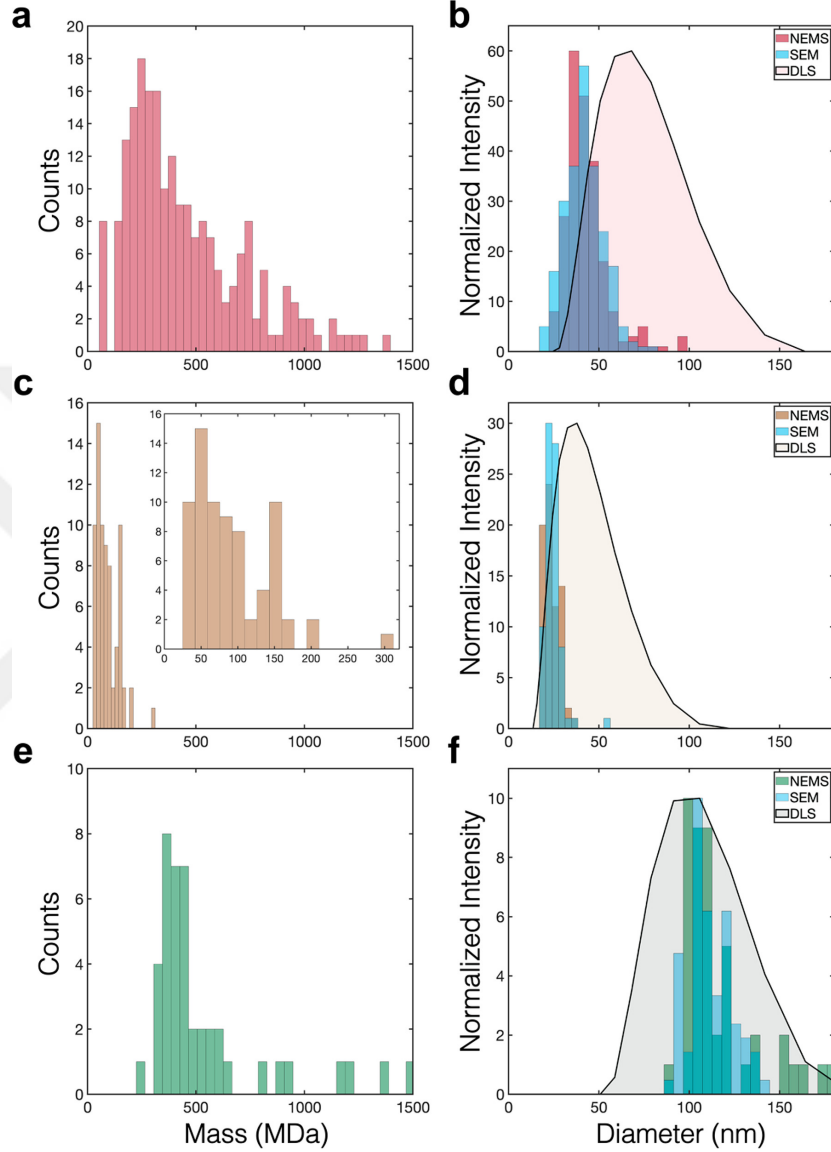


Figure 1.3: Mass and diameter histograms of atmospheric pressure NEMS-MS measurements. Taken from Figure 5 of reference [1].

Considering Figures 1.1, 1.2 and 1.3, we can conclude that the atmospheric pressure NEMS-MS is a powerful technique for the characterization of particles in the MDa range. Therefore, the development of the atmospheric pressure NEMS-MS not only solved major problems associated with NEMS-MS systems but also opened doors to new improvements in the NEMS-MS field.

1.3 Motivation

The motivation behind this thesis comes from the governing equations used in the mass calculations in the case of atmospheric pressure NEMS-MS. In doubly-clamped beams or cantilevers, one needs to track at least the first two out-of-plane mechanical modes, assuming the beam has an out-of-plane motion and the particle as a point particle, in order to calculate the landing position and the mass of the particle in the case of a landing event [6, 21, 22]. The first two out-of-plane modes of a doubly-clamped beam resonator, the square of the mode shapes, and the normalized linear mass density plots can be seen in Figure 1.4.

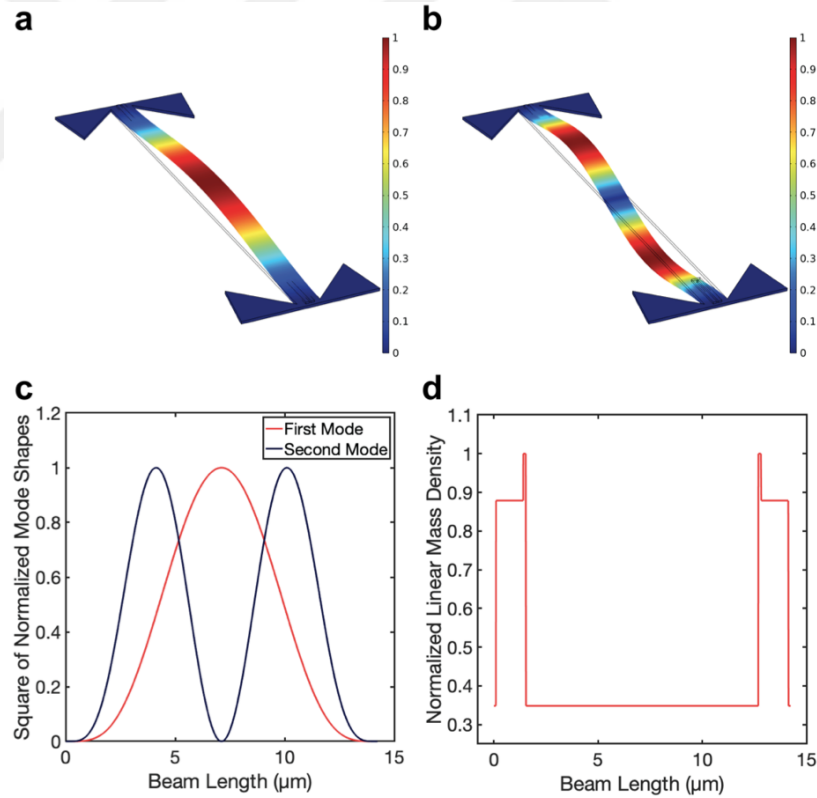


Figure 1.4: First two out-of-plane mechanical modes of the doubly-clamped beam resonator along with the square of the mode shapes and normalized linear mass density. Taken from the Supporting Figure S8 of reference [1].

There are two unknowns when a particle lands on the resonator (*i.e.* doubly-clamped beam or cantilever). These unknowns are the landing position and the mass of the particle. In order to solve for these unknowns, we need to construct

two equations, and these equations can be derived using two different mode shape functions (*i.e.* first and second out-of-plane mechanical mode shapes). Using these mode shape functions, we can use the following equation to solve for the particle's landing position and mass [6, 23].

$$\Delta f_n = -\frac{f_n}{\alpha_n M_{total}} \Delta m \phi_n(a)^2 \quad (1.1)$$

Where Δf_n is the frequency shift at a landing event, f_n is the frequency of the mechanical mode just before the landing event, M_{total} is the total resonator mass, Δm is the added particle mass and ϕ_n is the normalized mode shape function of the relevant mode. n denotes the mode number, and a denotes the normalized landing position that ranges from 0 to 1. Finally, we define the constant α_n as:

$$\alpha_n = 2 \int_{a=0}^{a=1} \phi_n(a)^2 da \quad (1.2)$$

The constant α_n allows us to calculate the effective mass of each mode as follows:

$$M_{eff}^{(n)} = \frac{\alpha_n}{2} M_{total} \quad (1.3)$$

The effective mass calculation accounts for the mode shape and, therefore, the relevant displacements on the beam. After introducing the parameters, we can observe that there are two unknowns in the case of a landing event as discussed previously (*i.e.* the landing position a and the mass of the particle Δm).

Since the mode shapes are symmetric with respect to the middle position of the beam (see Figure 1.4), one can find the position and the mass of the particle using the derived system of equations and the frequency shifts in the first two out-of-plane mechanical modes. This symmetry also results in two solutions for the system of equations; however, these two solutions yield the same Δm value

but two different positions that are symmetric with respect to the center of the device.

A recent study showed that the displacement profiles of these devices follow the mode shapes when the device operates at low dissipation, which means the device is under vacuum [2]. Therefore, under vacuum, the theory for mass calculations holds without any further corrections due to the symmetric mode shapes, hence symmetric displacement profiles. However, due to the actuation and the read-out electrodes, clamping regions and the dissipation, the displacement profiles become skewed (*i.e.* the displacement magnitude decreases towards the detection electrode), and the symmetry of the displacement profiles with respect to the center position breaks. The measured displacements on the device under air can be seen in Figure 1.5.

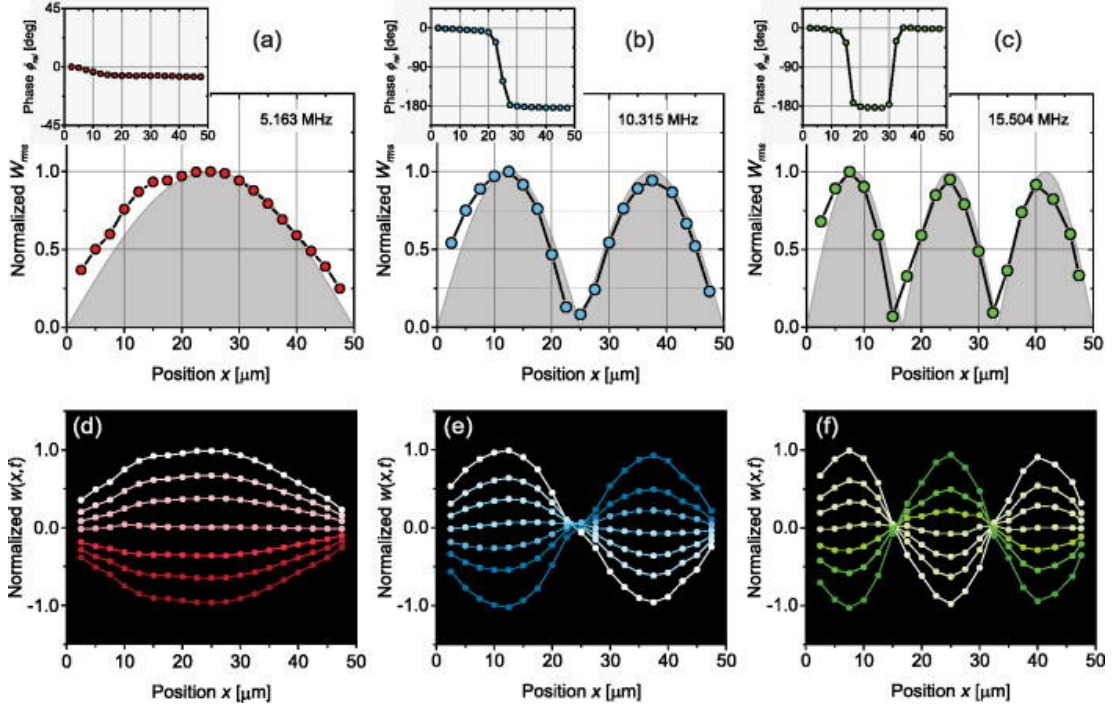


Figure 1.5: First three out-of-plane mechanical displacement profiles at resonance of a doubly-clamped beam resonator under air. Taken from reference [2].

The asymmetric displacement profiles result in two different solutions when we derive the system of equations using Equation 1.1 by replacing the mode shape function with the asymmetric displacement profile. Therefore we cannot

calculate the mass of a landing particle as accurately as in vacuum since the same frequency shifts in the first two out-of-plane mechanical modes will correspond to two different mass values. Also, the suspended silicon nitride near the clamping region causes uncertainties when a particle lands on these regions. Since these regions are suspended due to the isotropic etching of silicon, they move with the NEMS beam, and any particle landing on these regions results in frequency shifts. However, it is almost impossible to resolve the mass and the landing position of such particle landing events and therefore these regions results in uncertainties in the mass sensing experiments.

In the atmospheric pressure NEMS-MS work, the collected particle events resulted in accurate mass and position values, however in reference [2] the discussion on the quality factors take place. As the quality factor of the NEMS device decreases, the alteration on the displacement profiles increases. Therefore, it is important to develop mode shape independent mass sensors for the applications that take place with devices that have low quality factors. Here, we propose a device architecture, *i.e.* paddle NEMS devices, where the device features a region with a uniform mode shape. This uniform mode shape allows us to perform mode shape independent mass measurements since the particles that land on this region create frequency shifts that are not functions of the landing position. Also, it is important to note that, if wanted, the paddle NEMS devices can be deployed under both vacuum and air.

Atmospheric pressure NEMS-MS work showed that operating the system under atmospheric conditions is much more beneficial compared to operating the system under vacuum. However, it is also shown that the displacement profiles are not symmetric anymore when the NEMS device is operated in a dissipative environment (*i.e.* under atmospheric conditions) [2]. Therefore it is important to develop devices that are able to relate the mass change on the beam to the device mass and the frequency shift only, without any dependence on the mode shapes. Paddle NEMS devices remove the dependency of the mode shape thanks to the uniform platform displacement and therefore enable us to perform mass sensing using only one mechanical mode.

It is important to note that the paddle NEMS devices make inertial imaging applications much more difficult due to the uniform mode shape while enabling single mode mass sensing [24,25]. In the literature, one instance of a single mode mass sensing targeting a couple of MDa analytes was shown in the field of optomechanical sensing, which takes place inside vacuum, unlike atmospheric pressure paddle NEMS devices [12]. Also, similar geometries with paddle NEMS devices were investigated for the torsional and translational motion and were used in nanomechanical infrared thermal sensing and nanomechanical electrometer applications where they utilized the torsional modes of the resonators [26–28].

1.4 Thesis Outline

Chapter 1 starts with an introduction to the NEMS-MS field, discussing the major challenges and the demonstrated solution in a recent publication, *atmospheric pressure NEMS-MS*, in reference [1], in which I am an equal contribution co-first author. After introducing the atmospheric pressure NEMS-MS system, the motivation behind this thesis, *i.e.* paddle NEMS devices, is discussed by referring to a recent publication that discusses the alteration in the displacement profiles of doubly-clamped beam devices. Chapter 1 ends the discussion on how the alteration in the displacement profiles build the foundation of the mode shape independent mass sensing idea using paddle NEMS devices.

Chapter 2 starts with the design of the paddle NEMS devices and how they differ from the doubly-clamped NEMS devices. Next, the discussion on the mode shapes using simulations takes place. At this point, the mode shapes are compared with the doubly-clamped NEMS devices used in the literature before. The simulations showing the uniform mode shapes are presented. Finally, the fabrication of the paddle NEMS devices is discussed.

Chapter 3 starts with an explanation of actuation and detection techniques utilized for the paddle NEMS devices. The electronic circuit used in the experiments and the open sweeps of a double paddle NEMS device is presented. Next,

the analyte sample preparation and analyte delivery system, *i.e.* electrospray ionization setup, are discussed. Finally, the simulations of the paddle NEMS devices, mode shape characterization, and the theory behind the mass calculations are discussed.

Chapter 4 presents a uniform mode shape validation and mass sensing experiment using a single paddle NEMS device and 200 nm fluorescent polystyrene nanoparticles. After the single paddle NEMS device experiment, we used devices that feature two paddle NEMS devices side-by-side for mode shape validation and mass sensing experiments using 200 nm fluorescent polystyrene nanoparticles and 40 nm gold nanoparticles, respectively. The results from each experiment are discussed.

Chapter 5 concludes this thesis by referring to the relevant results and discussing possible future works.

Chapter 2

Design, Simulation, and Fabrication of Paddle NEMS Devices

2.1 Device Design

The design of the devices is one of the most important steps of NEMS-MS. Since the theory behind the mass calculations yields expressions that are functions of the total device mass, one can increase or decrease the mass sensitivity of the device and the targeted mass range by changing the device architecture. It was shown in the literature that even atomic scale mass values can be targeted [5,29–31]. Also, the mechanical mode shapes strongly depend on the device design, where we can control the overall trend of the mode shapes, such as uniformity. The frequency stability of the devices also depends on the designs and the fabrication flow. The frequency stability increases when the fabrication of the device undergoes without any problem and when the substrate is clean. Therefore, the design process and the simulations before the fabrication and finally developing a clean and neat fabrication flow is very important.

2.1.1 Design of Doubly-Clamped Beam NEMS Devices

Doubly-clamped beam devices consist of two main parts, which are the mechanical beam and the actuation/detection electrodes. The mechanical beam structures are fabricated mainly from thin silicon nitride films and generally placed on top of silicon substrates. The electrodes are commonly fabricated using gold and are placed on top of the mechanical doubly-clamped beams in the case of thermoelastic actuation and piezoresistive detection [32]. One can see the SEM images of these devices in Figures 1.1 and 1.2.

The doubly-clamped beam devices that I have designed in our group are typically 10 μm long, 0.8 μm wide, and 100 nm thick. The fabrication flow of these devices is very similar to paddle NEMS device fabrication flow and can be seen in Section 2.3. More detailed fabrication discussions can be seen in theses in references [33, 34]. A typical mask design of a doubly-clamped beam can be seen in Figure 2.1.

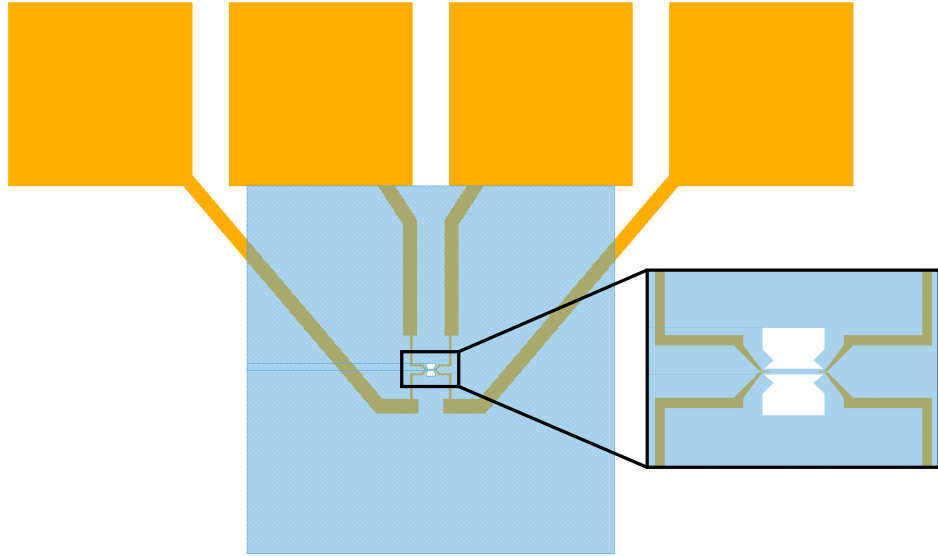


Figure 2.1: The mask design of a doubly-clamped beam NEMS device.

The displacement profiles of these devices are very similar to the ones in Section 1.3 in Figure 1.4; therefore would result in asymmetry when operated under atmospheric conditions. This asymmetry would then create a variation in the

mass values as discussed in Section 1.3. Therefore, the need for devices that has uniformity in their mode shapes (*i.e.* frequency shift is mode shape independent) arises.

2.1.2 Design of Paddle NEMS Devices

In this thesis, there are two different layouts of paddle NEMS devices that are used in the mass sensing experiments. The first layout features a single paddle NEMS device, whereas the second one features two devices side-by-side. For completeness, the second layout, where there are two devices, will be discussed in this section. These two layouts only differ in the number of devices placed on the same chip. Another reason to use the double paddle NEMS device architecture in this section is that it features two different designs on each device, where one of the devices features a hole in the middle of the platform, whereas the other does not. This way, the discussion on the double paddle NEMS device (see Figure 2.2) layout covers both designs.

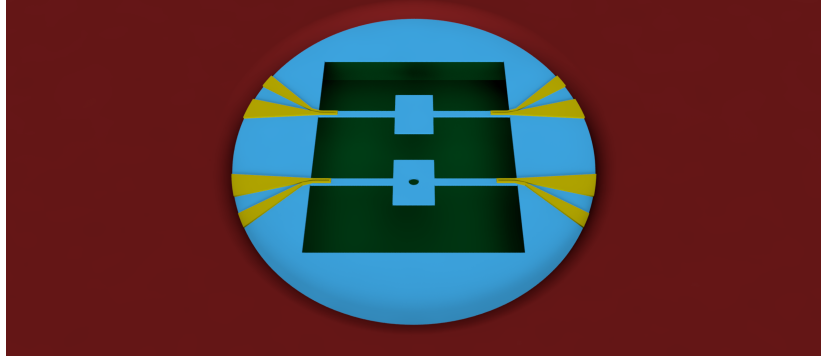


Figure 2.2: CAD render of the double paddle NEMS device.

Since the need for mode shape independence arises in the case of atmospheric pressure mass sensing due to the asymmetric displacement profiles (even more in other environments, such as liquids), we designed devices that have rather large platforms in the middle of the structure. This platform both makes the mode shape uniform and increases the surface area of the uniform displacement region (see Section 2.2). The CAD render of the double paddle NEMS device can be seen in Figure 2.2.

The mask design of the device can be seen in Figure 2.3. The layers in the GDS file are exactly the same as the ones in the doubly-clamped beam case, and a detailed discussion can be seen in reference [34]. Therefore, besides the additional platform, every aspect of the paddle NEMS device, in terms of mask design, is the same with doubly-clamped beam devices (*i.e.* bonding pads, electrodes, etc.). In the mask design, one can see that there are two devices side-by-side (*i.e.* the device that will be used in 40 nm GNP experiments), one of which has an additional hole in the middle of the platform.

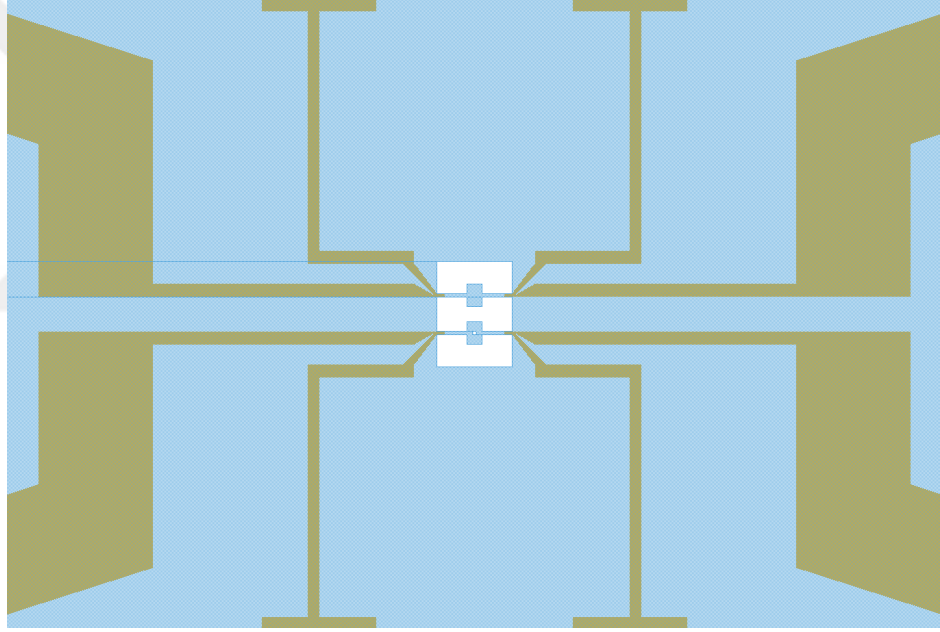


Figure 2.3: Mask design of the double paddle NEMS device.

2.2 Mode Shape Simulations

After the initial designs of the paddle NEMS devices, we continued with the finite element analysis (FEA) simulations. In this section, both a doubly-clamped beam and paddle NEMS device mode shapes and simulations will be discussed.

2.2.1 Doubly-Clamped Beam NEMS Device Simulations

In the case of doubly-clamped beam resonators, we need at least two out-of-plane mode shapes in order to solve for the landing position and the mass of a landing particle [6]. Therefore, in the simulation part, one needs to observe the first two out-of-plane mechanical mode shapes. The mode shapes and the simulations for a doubly-clamped beam device can be seen in Figure 1.4. For a detailed discussion on doubly-clamped beam simulations, see reference [1].

2.2.2 Paddle NEMS Device Simulations

Unlike the doubly-clamped beams, we need the first out-of-plane mechanical mode shape of the paddle devices to be uniform in the middle platform in order to perform mode shape independent mass sensing. The first out-of-plane mode shape in our paddle NEMS device design can be seen in Figure 2.4. We observed this mode shape independent from the silicon nitride film stress, and the frequency in the simulation is matched with the open sweep values later on in order to get an accurate normalized mode shape function for further calculations.

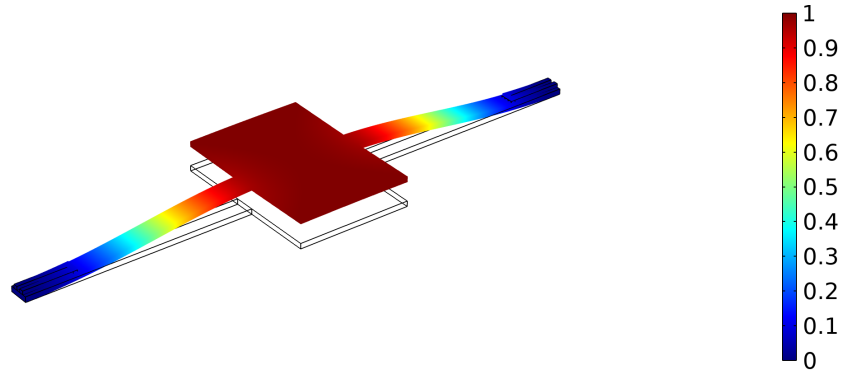


Figure 2.4: The first mode shape of the paddle NEMS device.

One can see from Figure 2.4 that the mode shape on the middle platform is almost completely uniform (for a detailed analysis, see Section 3.5). Also, it

is important to note that the simulations are done for both designs (*i.e.* with and without a hole on the platform); therefore, the simulations cover both single and double paddle NEMS device layouts since some of the double paddle NEMS device layouts feature both designs.

After verifying the mode shape with simulations, we continued with the mode shape of another device which has a hole in the middle of the platform, in order to decrease the mass of the device. The idea behind the mass decrease is to increase the mass sensitivity (*i.e.* decrease the mass resolution). This device also resulted in a uniform platform region in terms of displacement, and the comparison between the one with the hole and the one without the hole can be seen in Figure 2.5a. Also, the difference in the amplitudes are plotted in Figure 2.5b. Each device can be seen in the render in Figure 2.2.

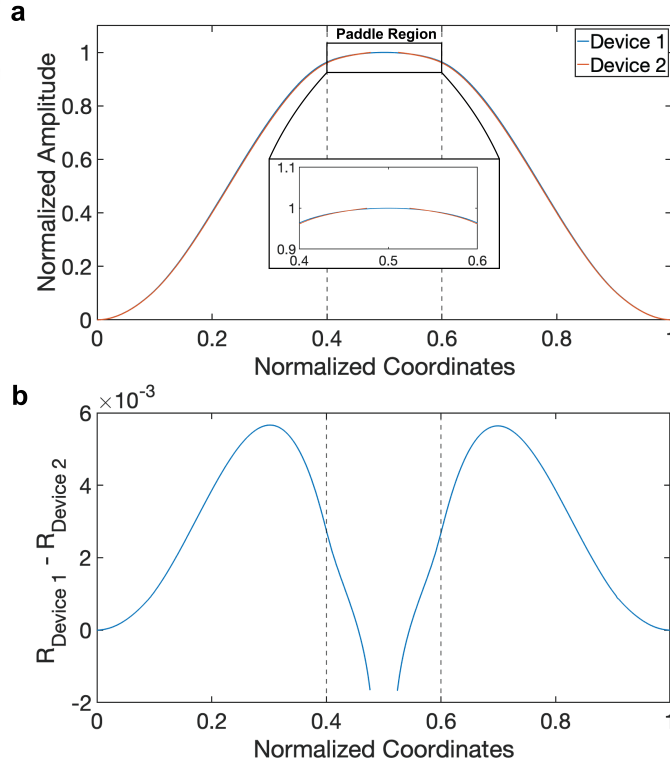


Figure 2.5: (a) The comparison of the mode shapes between the devices with hole and without hole. Device 1 denotes the device that has no hole, and device 2 denotes the device that has a hole. Inset shows the amplitudes of device 1 and device 2 in the paddle region. (b) Shows the difference in the amplitudes of device 1 and device 2.

One can see that the difference in the displacement is small between the two, and therefore, the device with the middle hole can also be used for single mode mass sensing applications, *i.e.* the presence of hole does not degrade uniformity. These simulations formed the basis of the idea and proved that these devices indeed have uniform mode shape sections, which will relate the frequency shift directly to the device mass and the added particle mass without any dependence on the mode shape.

Also, it is important to compare the paddle NEMS device with the doubly-clamped beam resonator. The comparison for their fundamental-mode mode shapes can be seen in Figure 2.6.

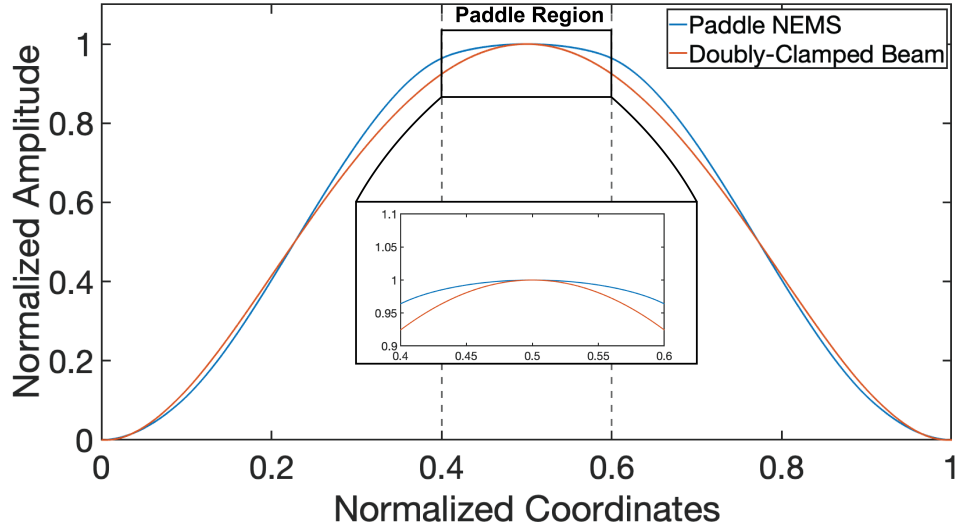


Figure 2.6: The comparison of the normalized mode shapes between a doubly-clamped beam and a paddle NEMS device. Inset shows the amplitude of the doubly-clamped beam and the paddle NEMS device in paddle region.

In Figure 2.6, we can see that the middle portion of the paddle NEMS device is uniform compared to the doubly-clamped beam mode shape. It is also important to note that this uniform region of the device has almost 65% of the device surface area. Therefore, we have a uniform region, and the capture area of this region is much larger compared to the middle portion of the doubly-clamped beam. For detailed numbers, see Section 3.5.

2.3 Fabrication of Paddle NEMS Devices

After demonstrating that the proposed paddle NEMS device architecture has a uniform mode shape on the middle platform, we continued with the fabrication of the devices. In this section, again, the double paddle NEMS device layout is featured since it includes both two designs where the devices differ with a hole in the middle of the platform—this way, the fabrication for both designs is presented.

The fabrication starts with the wafers purchased from University Wafer (Part No: 1917) and classified as low-stress for the thin silicon nitride film. The specified silicon nitride film stress by the vendor (University Wafer) is < 200 MPa. The purchased wafers have LPCVD 100 nm stoichiometric silicon nitride films on top of doped 500 μm thick silicon substrates. The fabrication flow is explained in detail in references [1, 33–35]. However, for the sake of the completeness of this thesis, the relevant steps are discussed briefly.

The process starts with electron beam lithography for the actuation/detection electrodes [32, 36, 37]. The electron beam lithography steps including the first gold deposition step took place at Sabanci University Nanotechnology Research and Application Center (SUNUM). The bare wafer can be seen in Figure A.1a. Also, it is important to note that details on small fabrication steps such as dicing and protective photoresist layer for back etching of Si_3N_4 are omitted since they do not have significance on the overall fabrication process.

- On the wafer that is shown in Figure A.1a, a bilayer Poly (methyl methacrylate) (PMMA) was coated to be exposed using EBL. Since this step is done in collaboration with SUNUM, the details are omitted. The coated wafer can be seen in Figure A.1b.
- After coating the wafer with PMMA, the resistances and bonding pads are exposed and developed. The developed PMMA can be seen in Figure A.2a. The bonding pads are omitted in the figures for simplicity and space.

- After developing the PMMA, the chip is coated with a 5 nm chromium adhesion layer and 70 nm gold using a thermal evaporator. This step defines the gold regions, including the resistances and the bonding pads. The substrate after coating can be seen in Figure A.2b.
- After the chromium and gold coating, the bilayer PMMA is removed inside an acetone bath. The substrate after the first lift-off step can be seen in Figure A.3a.
- After defining the gold regions, another EBL step takes place in order to define the hard mask to protect the silicon nitride film and to define the mechanical structure. The substrate after the second bilayer PMMA coating can be seen in Figure A.3b.
- As in the case of the first EBL step, the bilayer PMMA is developed after the exposure and is ready for 40 nm copper hard mask coating. The substrate after the second development step can be seen in Figure A.4a.
- After the development step for the second EBL process, the substrate is coated with 40 nm copper using a thermal evaporator. This 40 nm copper works as a hard mask in the upcoming inductively coupled plasma (ICP) etching steps. The 40 nm copper coated substrate can be seen in Figure A.4b.
- As in the case of the previous lift-off step, the second bilayer PMMA was removed inside an acetone bath. The substrate after the lift-off can be seen in Figure A.5a.
- After defining the hard mask using 40 nm copper for the mechanical structures, we continued with the dry etching steps of silicon nitride and silicon. Since the paddle devices are wider than doubly-clamped beam structures, the only difference in the fabrication steps comes in the dry etching step. The dry etching is facilitated using ICP etching, and the parameters can be seen in Table 2.1.

The dry etching step using ICP starts with anisotropic Si_3N_4 etching. The exposing silicon nitride (*i.e.* near the mechanical structures) is etched in

Table 2.1: Inductively Coupled Plasma Etching Parameters

Recipe	Silicon Nitride (Si_3N_4)	Silicon (Si)
Etchant Gas	CF4	SF6
Etchant Gas Flow Rate (sccm)	20	15
Passivation Gas	CHF3	CHF3
Passivation Gas Flow Rate (sccm)	3	2
Coil Power (Watt)	400	150
Platen Power (Watt)	100	30
Chamber Pressure (mTorr)	5	10
Etching Duration (s)	50	450

this step. The substrate after Si_3N_4 etching can be seen in Figure A.5b.

- After etching the Si_3N_4 , the process continues with the isotropic etching of silicon underneath the mechanical structures. The device suspends in this step. The suspended device after the silicon etching step can be seen in Figure A.6a.
- After completing both Si_3N_4 and silicon etching, the protective 40 nm copper hard mask can now be removed using wet etching. The etchant used in this step is MicroChemicals TechniEtch Al80. The device after the hard mask removal can now be seen in Figure A.6b.
- The copper removal step finishes the first main part of the fabrication. Now that the device can be used, however, it is important to integrate the polymeric focusing lens to increase the capture efficiency [1]. Therefore, the device is now coated with a thick positive photoresist layer (AZ 4533) for the photolithography step. The coated device can be seen in Figure A.7a.
- Next, the photoresist is exposed to UV light using a mask aligner after aligning the device to a photomask. The photomask contains openings at the active region of the device, bonding pads, and alignment markers at the corners of the substrate. The detailed discussion and parameters for this step can be seen in the reference [34]; however, for the sake of completeness, the parameters for both photolithography and development steps can also be seen in Table 2.2 (* Normally the substrate thickness is 500 μm for the devices; however, we use a carrier wafer for the photoresist coating

step which is also 500 μm thick. Therefore the total thickness including the carrier wafer is 1mm.). The developed structure can be seen in Figure A.7b.

Table 2.2: Photolithography and Development Step Parameters

Photolithography	
Parameters	Value
Dose (Constant) (mJ/cm^3)	270
Seperation (μm)	80
Proximity (μm)	80
Substrate Thickness* (mm)	1
Resist Thickness (μm)	3.5
Development	
Developer	AZ 400K
Stopper	DI water
Developer Solution	1:4 - AZ 400K:DI water
Develop Time (s)	90
Stopper Time (s)	10-15

- The integration of the photoresist layer (*i.e.* the polymeric focusing lens) finalizes the fabrication. An SEM image of the double paddle NEMS device can be seen in Figure 2.7.
- After the fabrication, the device is placed on a custom printed circuit board (PCB) for outside electrical connections. These connections are established with wire bonding using aluminum wedge bonds. This step is performed in collaboration with METU MEMS Center. The device on a PCB after wire bonding can be seen in Figure A.8.

Also, an SEM image of a single paddle NEMS device at the end of the fabrication can be seen in Figure 2.8

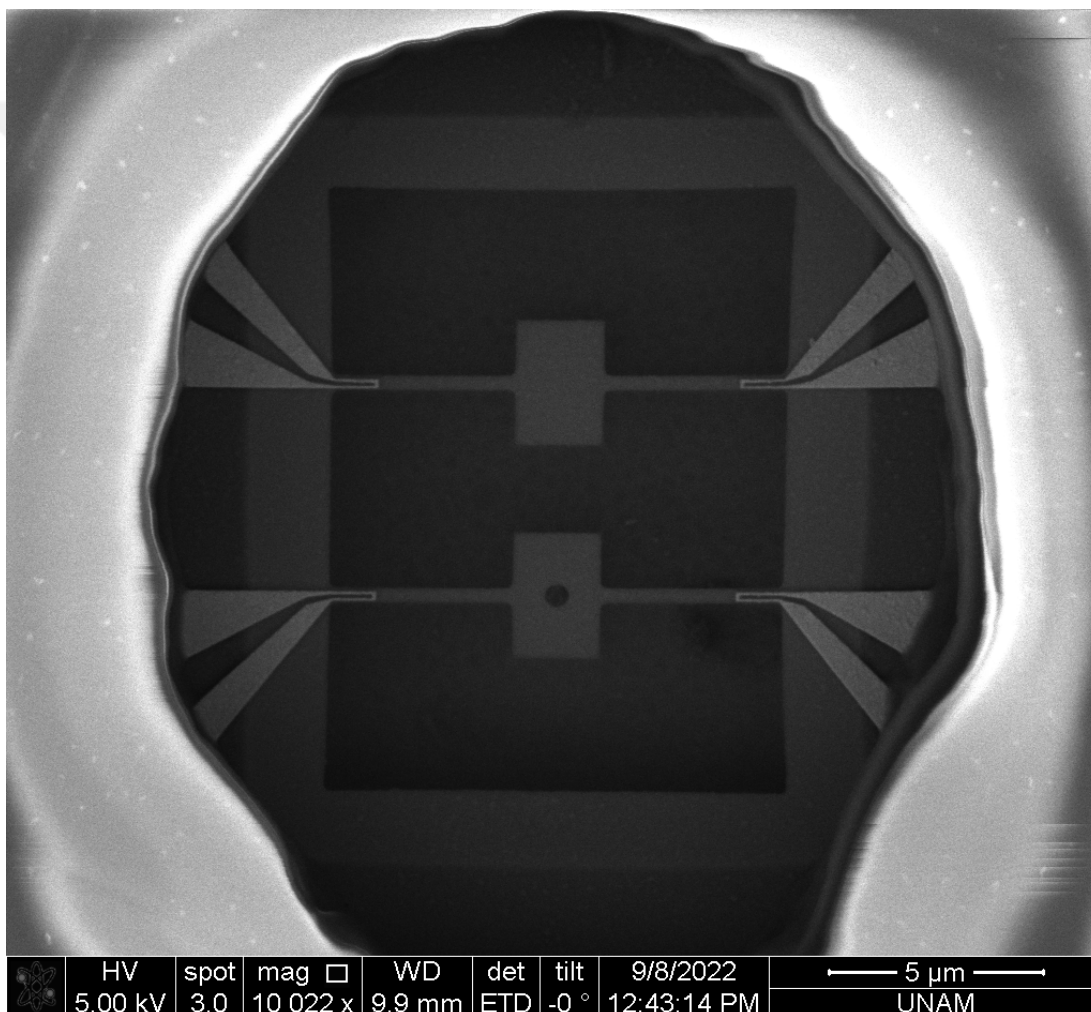


Figure 2.7: SEM image of the double paddle NEMS device at the end of fabrication. This device is used in 40 nm GNP mass sensing experiments in Section 4.2.2.

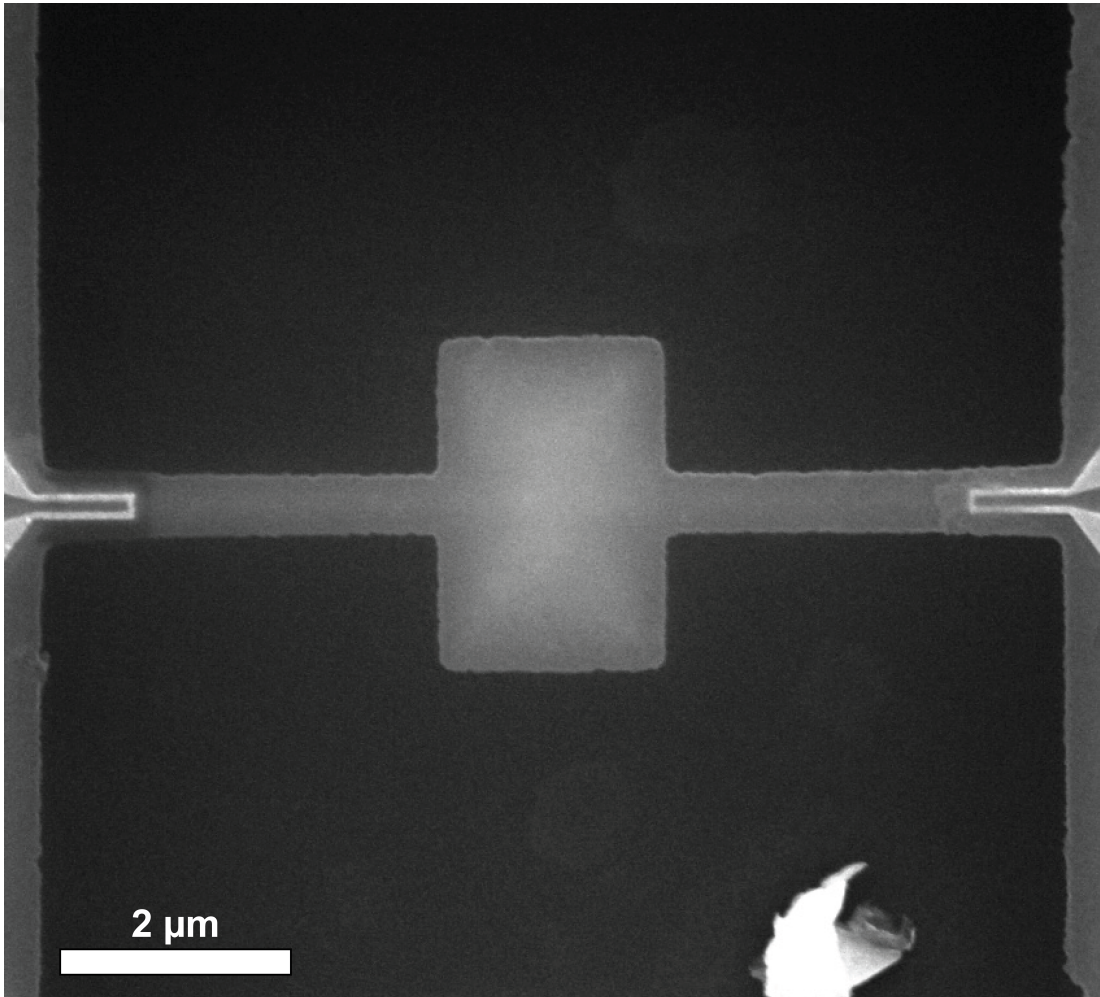


Figure 2.8: SEM image of the single paddle NEMS device at the end of fabrication. This device is used in the single paddle NEMS device experiments in Section 4.1.

Chapter 3

Experimentation and Theory

3.1 Thermoelastic Actuation and Piezoresistive Detection

The idea behind the actuation and the detection of paddle NEMS devices are the same as doubly-clamped beam devices that utilize thermoelastic actuation and piezoresistive detection [32, 38]. Thermoelastic actuation relies on the difference in the thermal expansion coefficients of two materials, one on top of the other. In our case, this is satisfied while using the device layer as silicon nitride and the electrode layer as gold. The reason behind the need for different thermal expansion coefficients is that the heating due to the applied AC voltage to the resistances results in thermal expansion on both device and electrode layer. This expansion on both device and electrode layers results in different amounts of elongation, which then develops stress. This stress, as a result of the AC voltage, causes the beam to resonate. It is also important to note that the heating on the resistances scale with the square of the applied voltage amplitude (*i.e.* V^2), and therefore the frequency of the beam oscillation (ω) occurs at twice of the applied AC voltage frequency ($\omega/2$). In order to match the actuation and the oscillation frequency of the NEMS devices, one needs to supply a DC voltage along with

the AC voltage to the NEMS actuation resistance. For the sake of simplicity, this method is not mentioned in this thesis since it was not deployed but can be seen in detail in reference [3]. For these two thermoelastic actuation schemes, see Figure 3.1 taken from reference [3].

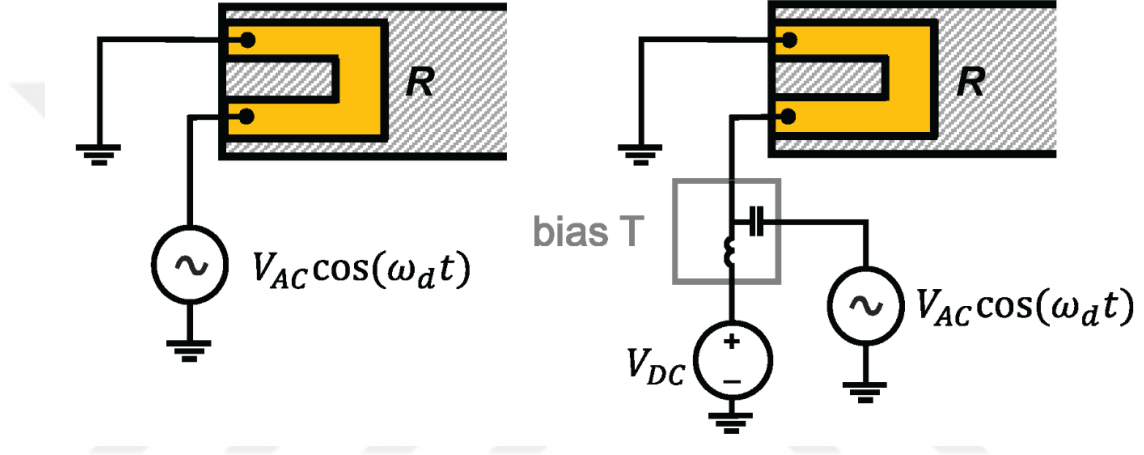


Figure 3.1: ω and $\omega/2$ thermoelastic actuation schemes. Taken from the reference [3].

The detection scheme is done with the opposite electrode using a technique called piezoresistive detection. Piezoresistive detection is a scheme to detect the resistance change that occurs on one of the NEMS resistances due to the mechanical motion of the NEMS device. In order to detect this resistance change, we used a bias tee and supplied the DC port with a constant voltage. Therefore, we can measure any change in the resistance while the NEMS device moves. Also, it is important to note that the resistance change is maximum at resonance due to maximum displacement. Since the resistance change depends on the actuation and hence depends on the displacement of the beam, the resistance changes at the frequency of the NEMS oscillation. Also, it is important to mention that the Lock-in-Amplifier input impedance is $50 \, \Omega$, and we design the resistances on the NEMS devices to be $50 \, \Omega$ for impedance matching with the used RF instruments. The majority of this resistance is at the loop (*i.e.* the suspended region of the gold placed on the suspended mechanical device), which can be seen on the doubly-clamped beam device or double paddle NEMS device circuits in Section 3.2.

3.2 Open Sweep Measurements & Phase-Locked-Loop

The open sweep measurements take place after the end of the fabrication process in order to determine the resonance frequencies of the paddle NEMS devices. In these measurements, we used an HF2LI Lock-in-Amplifier (Zurich Instruments) and custom circuitry containing a bias tee and a low-noise amplifier. In the case of a doubly-clamped beam and a single paddle device, there is only one input and one output to the NEMS resonator. The circuit schematic that is used for both can be seen in Figure 3.2.

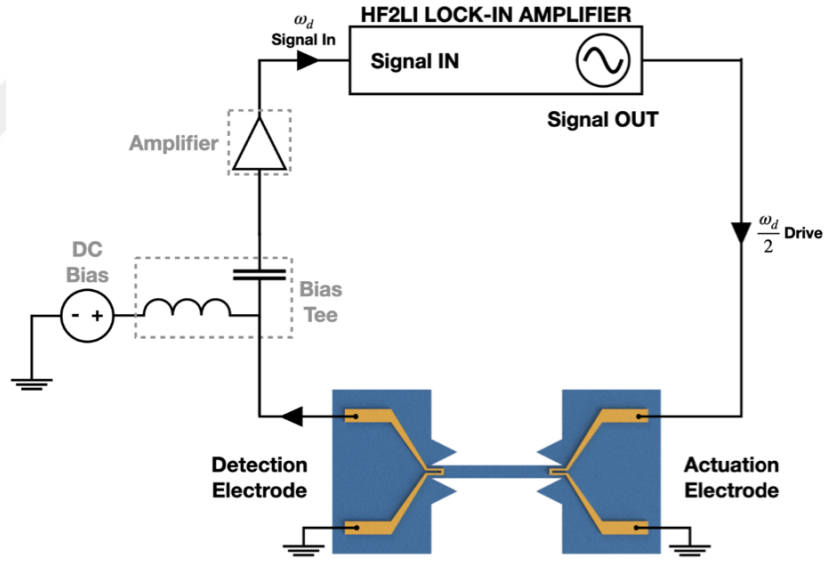


Figure 3.2: The circuitry that is used in the detection and actuation of doubly-clamped beams and single paddle NEMS devices. Taken from the reference [1].

Since there is only one output and one input, the two mode frequencies are given as a linear combination of the two, and the read-out frequencies are demodulated at the second harmonics of the output frequencies that are supplied to the NEMS device for actuation. This feature of the HF2LI Lock-in-Amplifier allowed us to perform two mode phase-locked-loop (PLL) in the case of doubly-clamped beam NEMS devices and therefore enables us to track the first two out-of-plane resonance frequencies. As expected we used only one PLL in the case of single paddle NEMS devices using the same circuit. After taking the open sweeps, we

determine the corresponding phase value at resonance and set it up as the set-point for PLL. PLL on both modes allows us to track the resonance frequencies and determine the frequency shifts when a particle lands on the structure. The circuitry and the detection scheme are the same for the single paddle NEMS devices since they feature two resistances that are used for the thermoelastic actuation and piezoresistive detection of the device. However, there are some differences in the case of double paddle NEMS devices (see Figure 3.3), and these differences are discussed below.

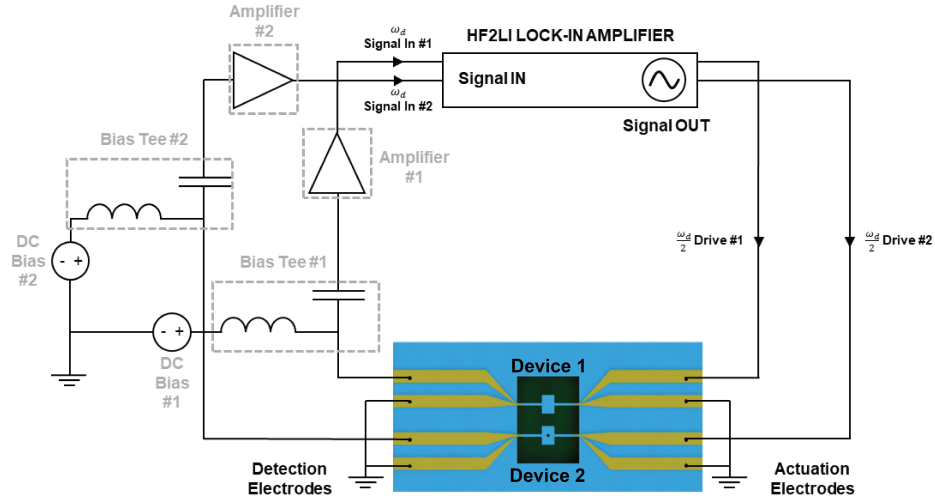


Figure 3.3: The circuitry that is used in the detection and actuation of double paddle NEMS devices.

The architecture of the double paddle NEMS requires us to have two inputs and two outputs, both separately addressing one electrode on each device. Therefore, we need to construct the circuit in Figure 3.2 for each of the paddle NEMS devices on the PCB. The schematic of the circuitry used in the double paddle NEMS experiments can be seen in Figure 3.3. The PLL configuration is basically the same for the double paddle NEMS devices; however, in the double paddle NEMS case, we need to address the demodulators and the signal outputs to different ports of the HF2LI Lock-in-Amplifier. Since there are two paddle NEMS devices on the same chip side-by-side, for this section, from now on, the device without the hole will be referred to as device 1, and the device with the hole will

be referred to as device 2.

After the discussion on the fundamentals of the circuitry and the PLL, it is important to discuss the open-loop sweeps of the devices. Since the single paddle NEMS devices are fundamentally the same as the doubly-clamped beam devices in terms of the measurement scheme, a double paddle NEMS device that features both a device with a hole and a device without a hole will be used in this section. The simultaneous open sweeps for two side-by-side resonators (*i.e.* Device 1 & Device 2) on the same chip can be seen in Figure 3.4.

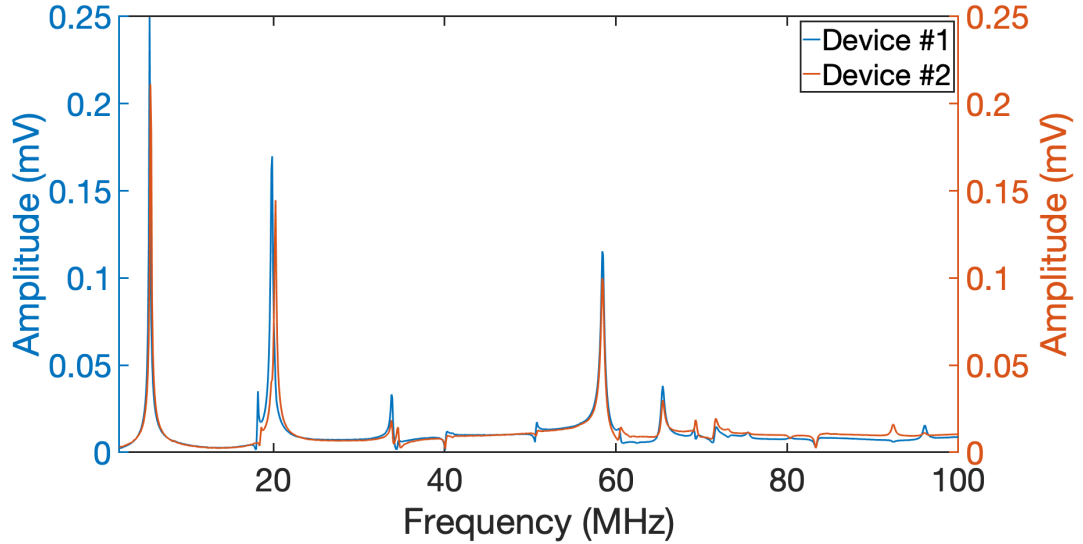


Figure 3.4: Simultaneous open sweeps of device 1 (no hole) and device 2 (with hole) from 2 MHz to 100 MHz.

One can see the frequency spectrum in Figure 3.4. In Section 2.2.2, it was mentioned that the first resonance mode is the mode of interest where the platform on the middle of the device has a uniform out-of-plane displacement without any dependence on the silicon nitride thin film stress (*i.e.* changing the silicon nitride thin film stress does not change the mode shape in the simulations and the first mode is always the mode where the middle platform has a uniform displacement). Therefore, another simultaneous sweep around their first resonance frequencies was taken and can be seen in Figure 3.5. Also, it is important to mention that after the open sweeps, the simulations for each device are reiterated for different stress values on the silicon nitride thin film. This step is important to simulate

accurate mode shapes and to collect accurate displacement values.

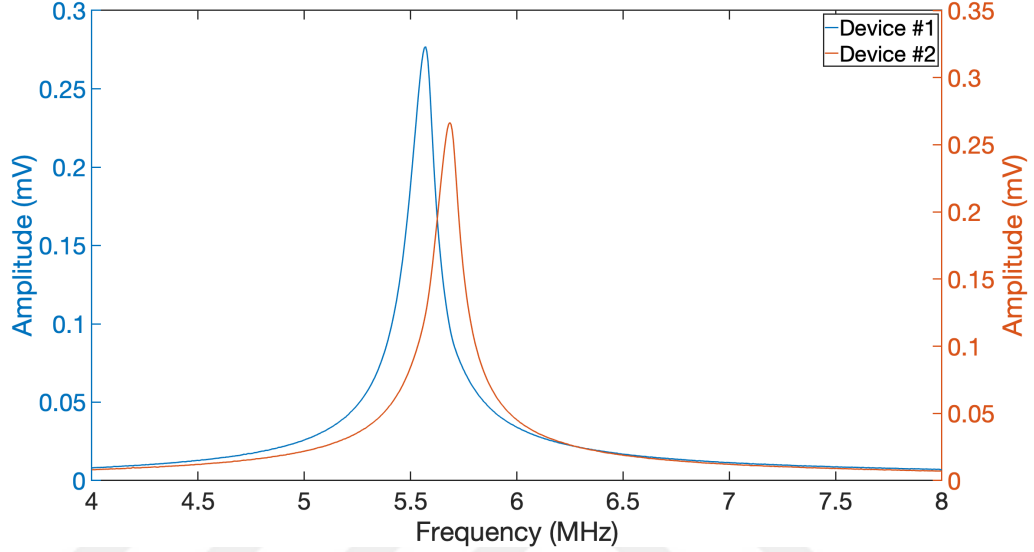


Figure 3.5: Simultaneous open sweeps of device 1 (no hole) and device 2 (with hole) around their first resonance frequencies.

The resonance frequencies came as 5.57 MHz for device 1 and 5.68 MHz for device 2. Therefore the stress values on simulations are adjusted so that the eigenfrequencies of the system will be exactly the same as the experimental resonance frequencies. In Section 2.2, the mode shapes of the paddle NEMS devices that are matched with the experimental frequencies can be seen.

It is important to calculate the frequency stability before each experiment, and these calculations for individual experiments can be seen in Chapter 4. By using the double paddle NEMS architecture, we covered both designs, where one of them features a hole in the middle of the platform.

3.3 Nanoparticle Solution Preperation

The nanoparticle solution preparation takes place just before introducing the nanoparticles to the ESI system in Section 3.4. The particles are purchased from vendors (*i.e.* Nanopartz & ThermoScientific) and diluted using an ESI

compatible buffer. The buffer that we used is an 80 mM ammonium acetate buffer. The addition of ammonium acetate is essential since the ESI solution has to be conductive to facilitate the electrospray. One other addition to the final ESI concentration is the 10% methanol by volume. The addition of methanol enhances the evaporation in the ESI process. After preparing the ESI compatible 80 mM ammonium acetate buffer, we continued with the ESI sample preparation using the purchased 200 nm fluorescent polystyrene nanoparticles (F-PSNP) (Thermo Scientific Fluoro-Max Green Fluorescent Polymer Microspheres G200) and 40 nm gold nanoparticles (GNP) (Nanopartz A11-40).

For the preparation of the 200 nm F-PSNP solution, we diluted the stock solution using double-filtered (using 220 nm syringe filters) 80 mM ammonium acetate buffer and HPLC methanol. The vendor supplies the Equation 3.1 for the calculation of the particle concentration. It is important to note that the stock solution contains 1% solids by mass.

$$\text{number of particles/mL} = \frac{6\rho_f}{\pi D_p^3(\rho_f + \frac{\rho_b}{C} - \rho_b)} \quad (3.1)$$

In the equation, C denotes the particle concentration by percent mass, D_p denotes the particle diameter, ρ_f and ρ_b denotes the density of suspension (*i.e.* water), and the density of the nanoparticles, respectively. Using Equation 3.1 we find the concentration as 2.25×10^{12} particles/mL. The final diluted solution contains 10 μ L stock solution, 890 μ L 80mM ammonium acetate, and finally 100 μ L HPLC methanol. Considering the dilution amount, the final concentration used in the experiments is 2.25×10^{10} particles/mL.

The preparation of the 40 nm GNP solution was the same as the 200 nm F-PSNP solution. The only difference this time is that the purchased solution had a previously calculated concentration value which is 8.53×10^{10} particles/mL supplied by the vendor. This time the final solution contains 100 μ L stock 40 nm GNP solution, 800 μ L 80 mM ammonium acetate, and finally 100 μ L HPLC methanol. Therefore, the final used concentration is 8.53×10^9 particles/mL.

3.4 Electrospray Ionization and Analyte Delivery

A custom made electrospray ionization (ESI) setup is used for the efficient delivery of the analytes. The electrospray ionization facilitates the delivery of the nanoparticles to the active area of the paddle NEMS devices. The ESI setup is the same as the one that was used in the atmospheric pressure NEMS-MS work [1]. The schematic of the system can be seen in Figure 3.6, which is taken from the reference [1].

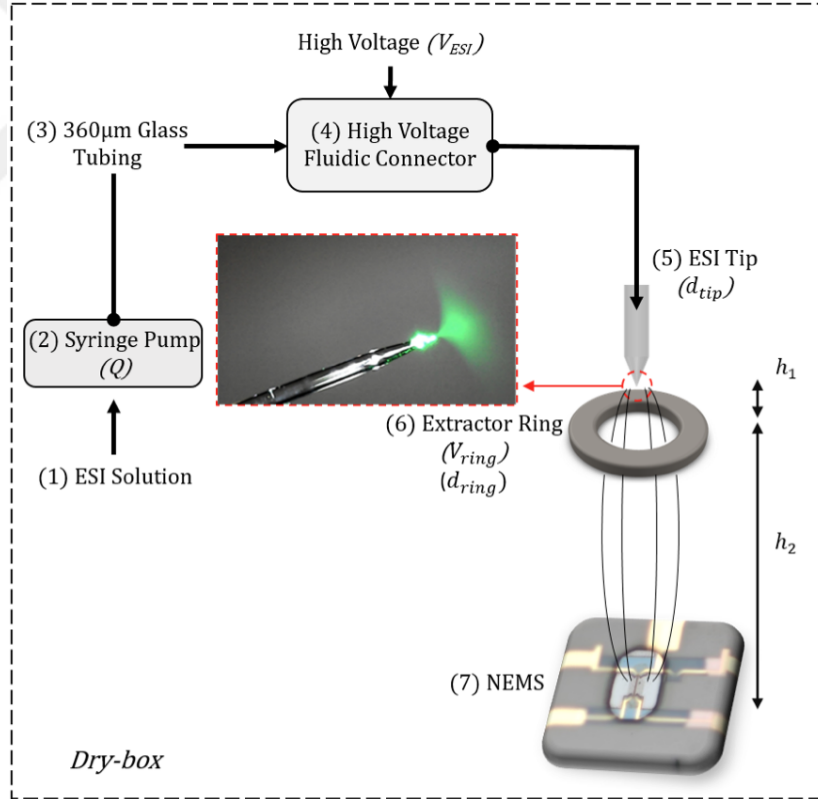


Figure 3.6: Schematic of the ESI setup used in the experiments. Taken from reference [1].

In Figure 3.6, one can see the separate parts of the ESI system. (1) shows the ESI solution that contains the nanoparticles, (2) shows the syringe pump that facilitates the flow to the ESI tip (5). (3) shows the microfluidic tubing

between the high-voltage connector (4) and the syringe pump (2). The high-voltage connector is supplied with 5.5 kV to facilitate the electrospray. (6) shows the extractor ring that is placed between the NEMS device (7) and the ESI tip (5). The NEMS device placed on this figure is a doubly-clamped beam device. The whole system is also placed inside a dry box. The inset in Figure 3.6 shows the ESI tip under operation. The experimental parameters can be seen in Table 3.1. For a more detailed discussion on the ESI setup, see reference [1].

Table 3.1: Electrospray Ionization Parameters

Flow Rate (nL/min)	500
ESI Voltage (kV)	5.50
Ring Voltage (kV)	1.3
Ring Diameter (mm)	15
Tip Diameter (μm)	10-30

The ESI process also helps with the focusing of the nanoparticles to the device surface by charging up the polymeric focusing layer. Therefore, the ESI setup is one of the most important parts of the atmospheric pressure NEMS-MS technique and hence is used in the paddle NEMS device experiments.

3.5 Simulations and Theory Behind The Mass Measurements

In Figure 3.7, the render of the device (SEM image can be seen in Figure 2.7) that will be used in this section can be seen. The reason, again, is that this device features two different paddle NEMS designs that are used in this thesis. This device will also be used in the double paddle NEMS 40 nm GNP experiments and is used in this section in order to explain the background calculations before discussing the experiments. Device 1 corresponds to the device without a hole, and device 2 corresponds to the device with a hole.

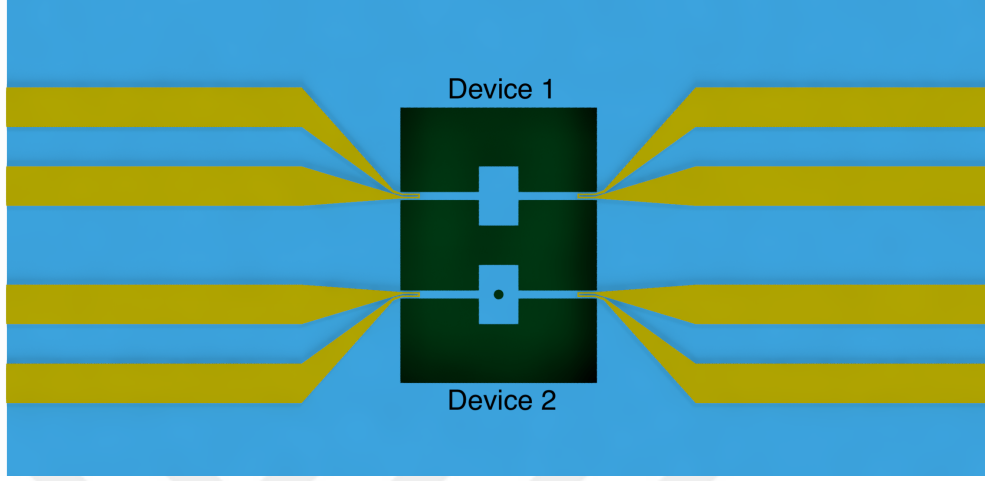


Figure 3.7: The render of the double paddle NEMS device without the polymeric focusing lens.

After discussing the experimentation part and the motivation behind the paddle NEMS devices, it is important to establish the theory behind the mass measurements and related calculations to be used in the data analysis. The main motivation discussed in Chapter 1 was relating the particle mass to the mass of the device and the frequency shifts without any dependence on the mode shapes at a landing event. Therefore, one of the most important parameters is the effective mass of the device. If we were to recall the Equation 1.3 for the calculation of the effective mass:

$$M_{eff}^{(n)} = \frac{\alpha_n}{2} M_{total} \quad (3.2)$$

The M_{total} value used in Equation 3.2 addresses only the beam mass, but not the additional masses on top of the structure such as the actuation/detection electrodes and the platform, thus we need to construct a new relationship to find the effective mass of the device.

The largest contribution to the mass comes from the middle platform. Also, the electrodes are made out of gold which has a density larger than the silicon nitride and they have a relatively significant contribution to the mass of the device. Therefore, it is important to account for the masses of additional structures on

the NEMS device such as the platform and the resistances. In order to do so, we need to relate the effective mass calculation to the mass density function of the device that accounts for the additional structures. Before calculating the M_{eff} , we relate the total mass of the device to the mass density function $\rho(\mathbf{r})$:

$$M_{total} = \sum_n \int_{\Omega} \rho_n(\mathbf{r}) dV \quad (3.3)$$

The function $\rho_n(\mathbf{r})$ is the mass density function of the n^{th} material where $\mathbf{r}$ is the three dimensional position vector and therefore the mass density function can be denoted as $\rho_n(x, y, z)$ in cartesian coordinates. Therefore the relation in Equation 3.3 can be written as:

$$M_{total} = \sum_n \int_z \int_y \int_x \rho_n(x, y, z) dx dy dz \quad (3.4)$$

In order to account for the mode shape of the structure, where the motion is out-of-plane (*i.e.* along the x-axis), in the effective mass calculation we introduce the linear mass density function:

$$\lambda(x) = \sum_n \int_z \int_y \rho_n(x, y, z) dy dz \quad (3.5)$$

Also, by introducing the linear mass density function $\lambda(x)$, now we can calculate the total mass of the device as:

$$M_{total} = \int_x \lambda(x) dx \quad (3.6)$$

The linear mass density function $\lambda(x)$, will decrease the integral dimension from 3 to 1 in the case of paddle NEMS devices since the platform will be taken into account as if it is a separate material and will have a different n value while calculating the integral in Equation 3.5. Next, we need to consider the mode

shape in the effective mass calculations. Therefore, we introduce the following equation for the calculation of the effective mass M_{eff} :

$$M_{eff} = \int_x \lambda(x) \phi(x)^2 dx \quad (3.7)$$

Since we know that the integration domain is from 0 to the device length L , Equations 3.6 and 3.7 can be written as:

$$M_{total} = \int_{x=0}^{x=L} \lambda(x) dx \quad (3.8)$$

$$M_{eff} = \int_{x=0}^{x=L} \lambda(x) \phi(x)^2 dx \quad (3.9)$$

Relating the effective and total mass of the paddle NEMS devices to a 1D integral that takes the mode shape and the mass of the additional structures into account will make the calculations much easier compared to using the integral in Equation 3.3. Also, the 1D integrals that we will be using for the calculation of the effective mass and the total mass yield accurate results since the mode shape on the platform is uniform.

Since the M_{eff} value is a function of both $\lambda(x)$ and $\phi(x)$ we need to find these functions. In order to find the mode shape of the device, we reiterated the simulations after taking the open sweeps and the SEM images for each fabricated device. The reason behind the open sweeps is to find the exact stress value on the thin silicon nitride film, which has a contribution to the mode shape of the device. SEM images are taken in order to measure the exact device dimensions since the dimensions are often not the same with the mask designs due to nano/microfabrication processes and are important for mode shape simulations [39].

After matching the experimental resonance frequencies with the simulated ones, we analyzed the displacement values on the platform of the paddle NEMS devices. For the discussion below, the device in Figure 2.7 is used.

First, we simulated device 1 (*i.e.* one without a hole in the middle) to match the first mode eigenfrequency value that is solved with the experimental first mode resonance frequency. In open sweeps, the first mode frequency comes at 5.57065 MHz, and after adjusting the stress values to match the frequency, the simulation yields a frequency of 5.5712 MHz. In Figure 3.8, the mode shape of device 1 can be seen from the top of the device.

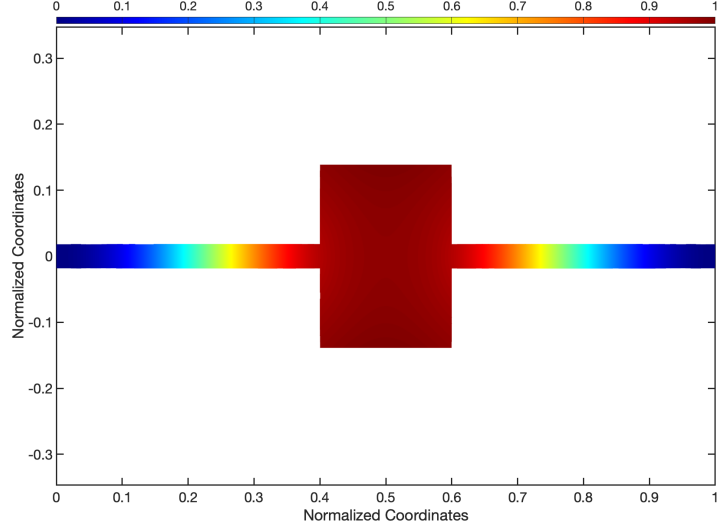


Figure 3.8: 2D mode shape plot for device 1.

Also, in Figure 3.8, the uniform mode shape on the platform of the device can be seen. This uniform region's surface area is more dominant compared to the supporting beam regions. In Table 3.2, some statistics for device 1 can be seen.

Table 3.2: Device 1 simulation statistics for middle platform

Maximum Normalized Displacement (A.U.)	1
Minimum Normalized Displacement (A.U.)	0.9481
Average Normalized Displacement (A.U.)	0.9811
Maximum Percent Difference	5.19%
Standard Deviation (A.U.)	9.79×10^{-3}
Percent Standard Deviation	0.9974%

The percent standard deviation of 0.9974% shows that the mode shape is uniform, even the percent difference between the points that have the maximum displacement and minimum displacement is low with a difference of 5.19%. This

analysis is done by exporting the displacement values from the simulations, and the number of data points for the displacement values on the platform is over 6.1 million.

After showing the uniformity on device 1, next, we analyzed device 2. This time the device has a hole in the middle of the platform, which reduces the mass and increases the resonance frequency of the paddle NEMS device. The 2D mode shape plot can be seen in Figure 3.9.

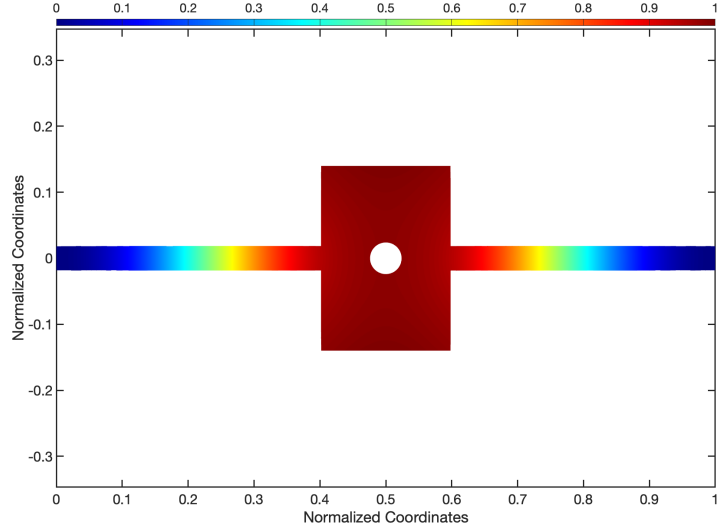


Figure 3.9: 2D mode shape plot for device 2.

In this plot, the mode shape of device 2 is very similar to the mode shape of device 1. Therefore, one can argue that the platform has a uniform displacement at resonance. The statistics for device 2 can be seen in Table 3.3.

Table 3.3: Device 2 simulation statistics for middle platform

Maximum Normalized Displacement (A.U.)	1
Minimum Normalized Displacement (A.U.)	0.9465
Average Normalized Displacement (A.U.)	0.9806
Maximum Percent Difference	5.35%
Standard Deviation (A.U.)	10.4×10^{-3}
Percent Standard Deviation	1.06%

The hole in the middle of the platform reduced the uniformity and increased

the maximum percent difference (*i.e.* percent difference between maximum displacement and minimum displacement) when compared to the case that has no hole, however the percent standard deviation value of 1.06% is still low enough to consider the mode shape as uniform on the platform. The mode shapes of these two devices can be seen in Figure 2.5, however, it is also important to plot the 1D-projected mode shape functions of the paddle NEMS devices at this point. The mode shape functions can also be seen in Figure 3.10.

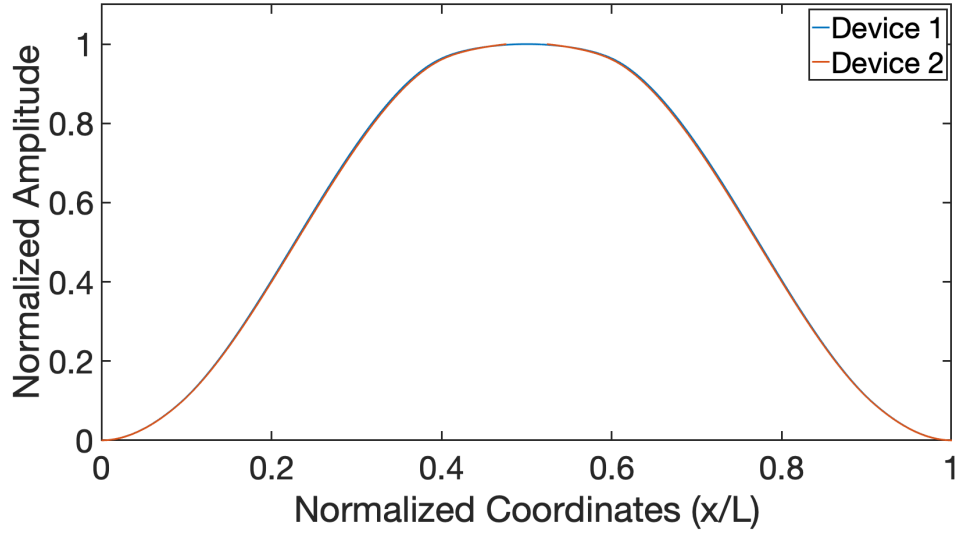


Figure 3.10: 1D mode shape functions of device 1 and device 2.

After validating the uniform mode shapes from simulations, we next calculate the effective mass values that will be used in the data analysis part. Since the mode shapes on the platforms are uniform with percent standard deviations around 1%, we will utilize the 1D linear mass density function $\lambda(x)$. This assumption leads to a higher linear mass density value on the platform positions since the total mass of the platform is much higher compared to the supporting beams. The normalized linear mass density plots for both devices can be seen in Figure 3.11. The normalization is done with respect to the maximum value of device 1 to highlight the difference created by the middle hole. Since the lengths of the devices are also different, the x-axis is normalized with respect to the relevant device length L . The decrease in mass due to the hole is distributed to the whole

platform and can be seen in the normalized linear mass density plot. This distribution is done considering the percent standard deviation of the displacement on the platform which is low with a 1.06%.

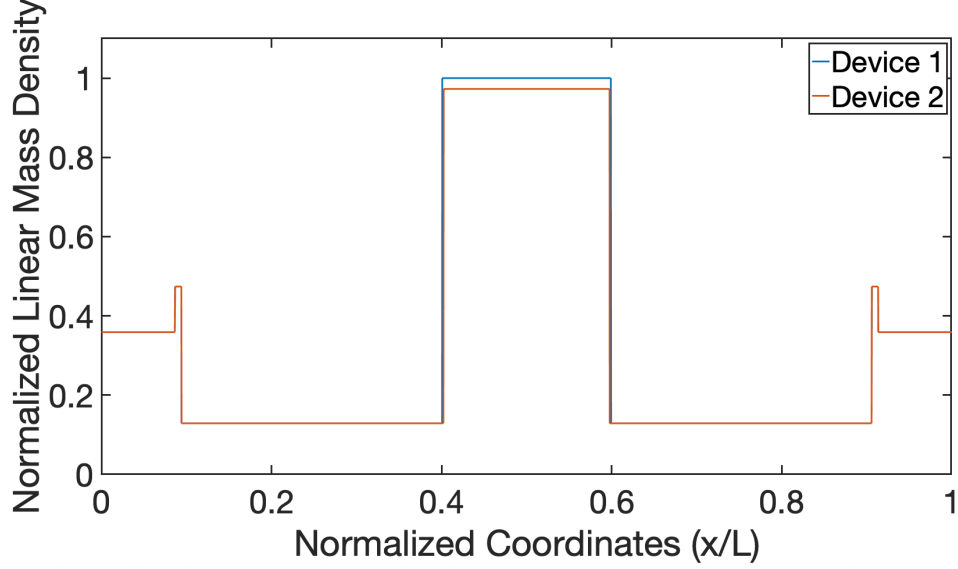


Figure 3.11: Normalized linear mass density plots of device 1 and device 2.

After building the 1D mass calculation analogy, we need to recall Equation 3.8 and Equation 3.9 which are:

$$M_{total} = \int_{x=0}^{x=L} \lambda(x) dx \quad (3.10)$$

$$M_{eff} = \int_{x=0}^{x=L} \lambda(x) \phi(x)^2 dx \quad (3.11)$$

In Equations 3.10 and 3.11, the function $\lambda(x)$ is the linear mass density function of the paddle NEMS device. In order to highlight the mass contributions of the middle platform and the resistances, we introduce a normalization constant λ_0 :

$$\lambda_0 \equiv \iint_{\text{supporting beam}} \rho_n(x, y, z) dy dz \quad (3.12)$$

The integral in Equation 3.12 is evaluated at a point where the only material is silicon nitride and has a uniform cross section (*i.e.* supporting beams). Therefore, in a paddle NEMS device case, the positions of the supporting beams, which have no resistances on top have a value of unity. In Figure 3.12, the linear mass density functions can be seen that is normalized with respect to a λ_0 that corresponds to the middle of the supporting beams.

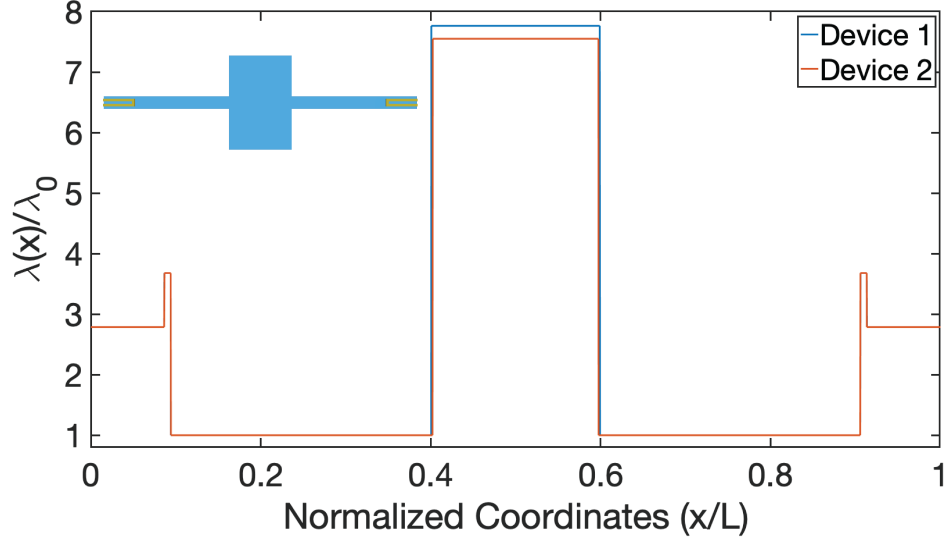


Figure 3.12: Normalized linear mass density functions with respect to λ_0 for device 1 and device 2. Inset shows the CAD render of device 1.

If we calculate the total mass of the devices based on measured dimensions using SEM, without using the mode shape and the linear mass density functions (*i.e.* $Volume \times Density$), we would get the following values for device 1 (one without hole) and device 2 (one with the hole): $M_{total, device\ 1, SEM} = 3457.9$ fg and $M_{total, device\ 2, SEM} = 3369.4$ fg.

Next, we will utilize the linear mass density functions in Figure 3.12 in order to calculate the total mass values using Equation 3.10. As previously discussed, the linear mass density function $\lambda(x)$ accounts for the additional structures and materials on the device. Therefore, if we were to analyze a doubly-clamped beam without any additional structures on top, the normalization with respect to λ_0 (in this case $\lambda_0 \equiv \iint_{clamp} \rho_n(x, y, z) dy dz$), the $\lambda(x)/\lambda_0$ plot would yield a value of unity at every point along the length of the device.

Using Equation 3.10, the total mass values are calculated as (LMD: Linear Mass Density): $M_{total, Device\ 1, LMD} = 3457.9$ fg and $M_{total, Device\ 2, LMD} = 3369.4$ fg. The mass values that are calculated using SEM dimensions and the linear mass density functions using Equation 3.10 values yield the same M_{total} values. Therefore, our linear mass density approach in order to account for the platform mass using a 1D integral is justified.

After calculating the M_{total} values, we next calculate the M_{eff} values for both device 1 and device 2. In order to calculate the M_{eff} values, we need to use Equation 3.11 with $\phi(x)$ and $\lambda(x)$ functions where normalized versions with respect to the device lengths are plotted in Figures 3.10 and 3.12, respectively. Since the hole in the case of device 2 creates a discontinuity, we simulated the same device without a hole with the same parameters and used the resulting mode shape function. The effective mass values for each device come as: $M_{eff, Device\ 1} = 2243.7$ fg and $M_{eff, Device\ 2} = 2157.8$ fg.

After showing that the mode shape is uniform on the platform and calculating the effective mass of the devices before each experiment, we need to form the equation that will allow us to calculate the Δm values for each landing particle. In order to do so, we need to recall Equation 1.1:

$$\Delta f_n = -\frac{f_n}{\alpha_n M_{total}} \Delta m \phi_n(a)^2 \quad (3.13)$$

We know that $\alpha_n M_{total} = 2M_{eff}$ and for the platform $\phi(a) = 1$ (in Equation 3.11 $\phi(x) = 1$ for each position on the platform). Therefore Equation 3.13 becomes:

$$\Delta f_n = -\frac{f_n}{2M_{eff}} \Delta m \quad (3.14)$$

Since $n = 1$ for the first mode, the Equation 3.14 can also be written as:

$$\Delta m = -2M_{eff} \frac{\Delta f}{f} \quad (3.15)$$

Therefore using Equation 3.15, if a particle lands on the platform of the device, we can relate the frequency shifts to the mass of the particles directly using only M_{eff} , and f values, which are already known prior to the frequency shift event without any dependence on the mode shape.

The calculations done above demonstrate and justify the theory of paddle NEMS devices along with the governing equation (*i.e.* Equation 3.15), and calculates the effective mass values for device 1 and device 2, which will all be used in Chapter 4 to generate the mass and diameter histograms.

Chapter 4

Mode Shape Uniformity Validation and Nanoparticle Measurement Experiments

4.1 Single Paddle NEMS Device Experiments

After showing that the mode shape is uniform on the platform by simulations, we continued to experimentally prove that by using 200 nm fluorescent polystyrene nanoparticles and single paddle NEMS devices. The solution preparation for 200 nm F-PSNP can be seen in Section 3.3. The reason that we used fluorescent particles is that they are easy to locate on the device under a fluorescent microscope after the experiments. The device we used here can be seen in Figure 2.8, and features a single paddle NEMS device.

The open sweep of the single paddle NEMS device used in 200 nm F-PSNP experiments can be seen below in Figure 4.1. Only the fundamental mode is shown since it is the mode of interest and will be used in the experiments.

The phase and amplitude plots can be seen in Figure 4.1. After setting up

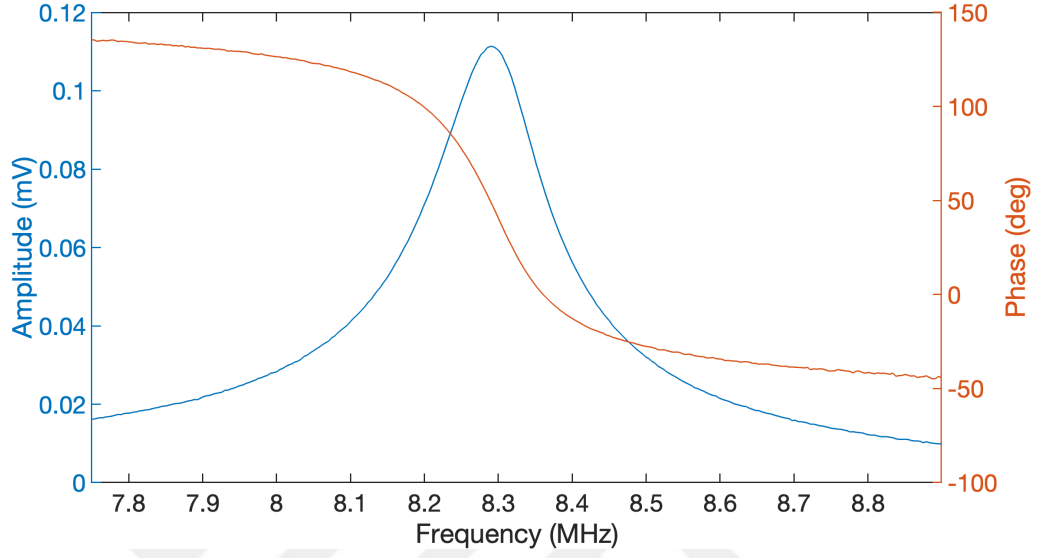


Figure 4.1: Open sweep of the single paddle NEMS device used in 200 nm F-PSNP experiments.

the PLL, we continued with the Allan Deviation calculations. The PLL time scale is 140 ms for all of the experiments that are presented in this thesis, and therefore, the reported Allan Deviation values are taken at 140 ms time scale. The values that correspond to the lower time scales are not valid and hence are inconsequential [40]. The Allan Deviation plot can be seen below in Figure 4.2.

The Allan Deviation, σ_{Allan} , is calculated as 2.75×10^{-6} at 140 ms. After calculating the Allan Deviation, we calculated the minimum detectable mass using the explicit formula in Equation 4.1 in order to compare it to the doubly-clamped beam devices used in the literature before and to see the measurable mass range.

$$\delta m_{min} = \sqrt{2} \times 2M_{eff} \left\langle \frac{\delta f}{f_{0 min}} \right\rangle \quad (4.1)$$

δm_{min} is the minimum detectable mass (*i.e.* mass resolution) and $\left\langle \frac{\delta f}{f_{0 min}} \right\rangle$ is the calculated frequency stability, *i.e.* the Allan Deviation. Since the Allan Deviation is calculated as 2.75×10^{-6} and the M_{eff} for this device is 2572.4 fg, the minimum detectable mass is calculated as 20 ag, which is 12 MDa. This value is only 4

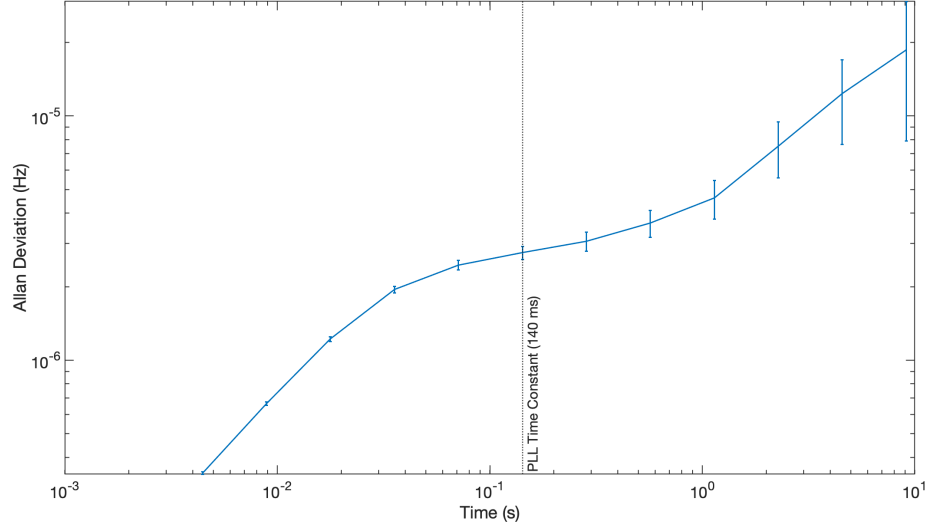


Figure 4.2: Allan Deviation plot for the single paddle NEMS device that is used in 200 nm F-PSNP experiments.

times worse and hence comparable with the mass resolution of a doubly-clamped beam NEMS device that is operated under atmospheric conditions [1]. Also, in the 200 nm F-PSNP experiment, the mass of the target particles is ≈ 2.67 GDa, which is well above the minimum detectable mass. It is important to note that this minimum detectable mass value is only valid for the particles that land on the middle platform since the minimum detectable mass calculation is done with respect to the Equation 3.15.

After calculating the Allan Deviation and the minimum detectable mass, we started the experiment by spraying 200 nm F-PSNP to the single paddle NEMS device. A section of the PLL data showing 6 landing events can be seen below in Figure 4.3.

Single paddle NEMS device used in this experiment captured 29 landing events and the resulting mass histogram using Equation 3.15 can be seen in Figure 4.4a.

We observed a peak at the expected mass of 200 nm F-PSNP in the mass histogram. Also, the left tail of the peak is more crowded compared to right and we attribute this to the landing events on the supporting beams. Any particle

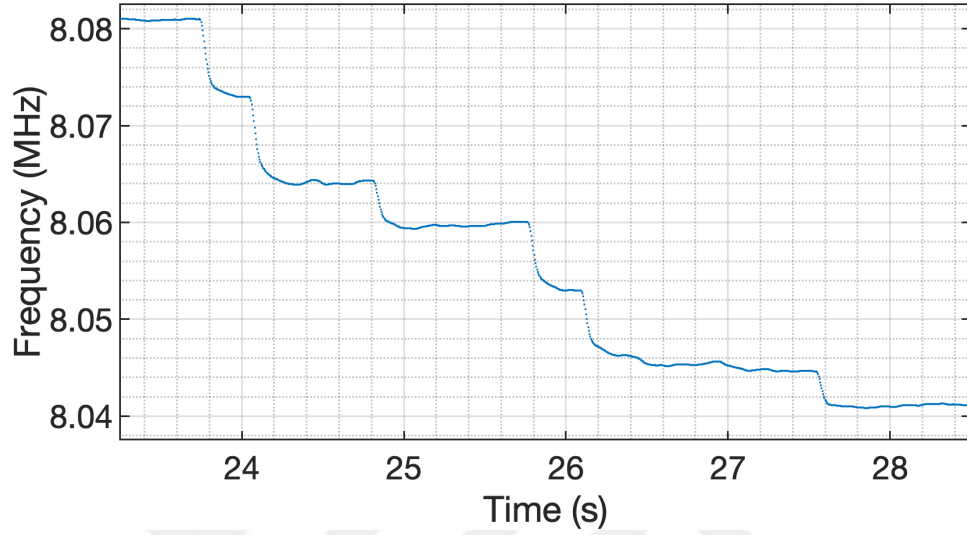


Figure 4.3: A section of the PLL data showing 6 200 nm fluorescent polystyrene nanoparticle landing events.

landing on the supporting beams would result in a lower frequency shift than expected due to the lower displacement values (*i.e.* mode shape). Next, we calculated the corresponding particle diameters using the density of the polystyrene (1.06 g/cm^3) and assuming the particles as perfect spheres ($m = \rho_{PS} \times \frac{4}{3}\pi(\frac{d}{2})^3$). The resulting diameter histogram can be seen in Figure 4.4b.

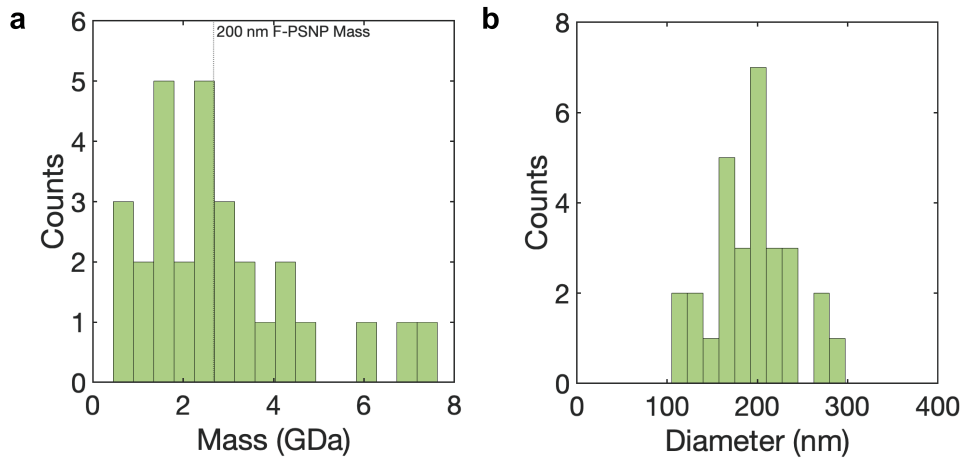


Figure 4.4: (a) Mass and (b) diameter histograms of the single paddle NEMS 200 nm F-PSNP experiment.

The diameter histogram resulted in an average of 194.9 nm, which is very close

to the expected value that is 200 nm. The reason that the average value is less than the expected diameter is the landing events on the supporting beams since as discussed previously they result in lower frequency shifts hence lower diameter values. Also, the peak in the diameter histogram includes 7 landing events, and the 2 bars on the left and the right of the peak includes 3 events each. Therefore, we observed a total of 13 events that resulted in diameter values close to 200 nm.

It is also important to compare the mass and diameter histograms to the fluorescent image of the device after the experiment. Both brightfield and fluorescent images of the device after the experiment can be seen in Figure 4.5.

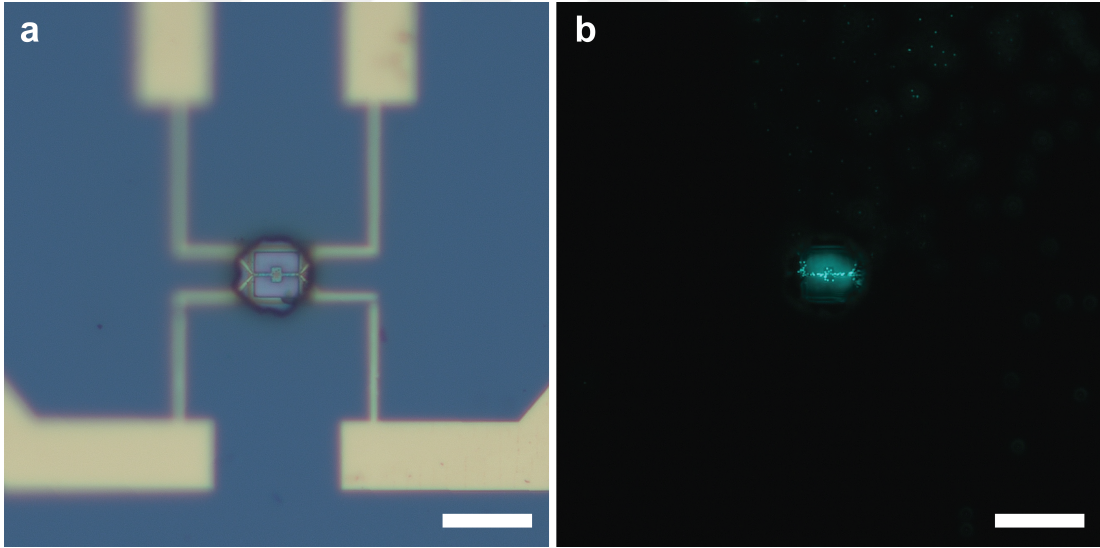


Figure 4.5: (a) Brightfield image of the single paddle NEMS device after the 200 nm F-PNSP experiment. (b) Fluorescent image of the device after the experiment. Scale bar is 20 μm for both images.

We observed exactly 13 particles on the platform, suggesting that the 13 events we observed on the diameter histogram are coming from the particles that landed on the platform. Therefore, the frequency shifts due to the landing events on the platform yielded accurate measurements using Equation 3.15. As a result, the single paddle NEMS device experiment using 200 nm F-PSNP both validated

the uniform mode shape on the platform considering the mass measurements and fluorescent images, and resulted in accurate measurements with an average diameter value of 194.9 nm.

4.2 Double Paddle NEMS Device Experiments

In this section a double paddle NEMS architecture is deployed. The doubly-clamped beam resonators require us to track two out-of-plane mechanical modes in order to resolve the landing position and the mass of the particles, whereas the paddle NEMS devices require us to track only one. Therefore, with the same number of PLL, we can track two different paddle NEMS devices simultaneously and this feature of the paddle NEMS is used in this section for 200 nm F-PSNP experiments and 40 nm GNP experiments.

4.2.1 200 nm Fluorescent Polystyrene Nanoparticle Experiments for Uniform Mode Shape Validation of Double Paddle NEMS Devices

Here, we utilize two devices side-by-side since we can track two devices simultaneously with the equal number of PLL as doubly-clamped beam devices. Therefore, it is easier to deploy two devices at the same time with paddle NEMS devices and this feature is utilized in this section.

We used a device that features two paddle NEMS devices side-by-side both of which has a hole in the middle of the platform. In this architecture we utilized only one of the devices to perform mode shape uniformity validation experiments before continuing with 40 nm GNP experiments where we utilized both two devices simultaneously. The open sweep around the first mode resonance frequency of the device used in this section can be seen below in Figure 4.6.

From the open sweep in Figure 4.6, we find the resonance frequency of the

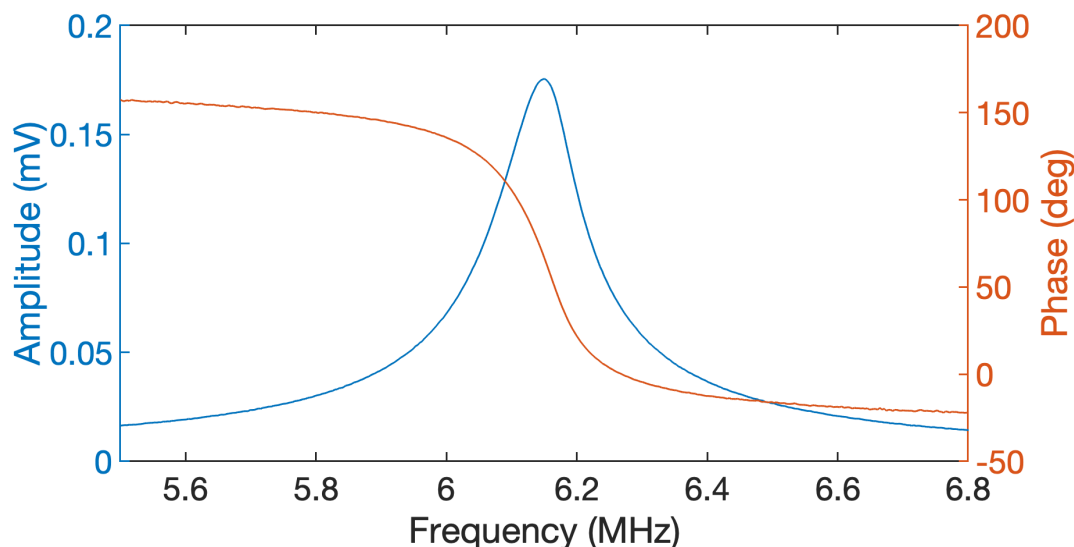


Figure 4.6: Open sweep of the device used in double paddle NEMS 200 nm F-PSNP experiments.

device and the corresponding phase value at resonance. After finding these values we set up the PLL and as in the case of the single paddle NEMS devices, we calculated the frequency stability, *i.e.* Allan Deviation, of the device. The Allan Deviation plot can be seen below in Figure 4.7.

As discussed previously the PLL time constant for all the experiments are 140 ms and the corresponding Allan Deviation for the device used in this section is 4.03×10^{-6} . This Allan Deviation then used for the calculation of the minimum detectable mass which is 22.7 ag (13.7 MDa). The minimum detectable mass is comparable with the doubly-clamped beam devices that is operated under atmospheric conditions. Also, it is important to note that the target mass of the 200 nm F-PSNP particles is ≈ 2.67 GDa which is again well above the minimum detectable mass.

After calculating the Allan Deviation and the minimum detectable mass, we started the ESI experiments using 200 nm F-PNSP. As previously discussed, only one device was used in these experiments to validate the uniform mode shape on the platform. We conducted 3 consecutive experiments, and a total of 8 landing events were observed. The frequency shifts as a result of these landing events can

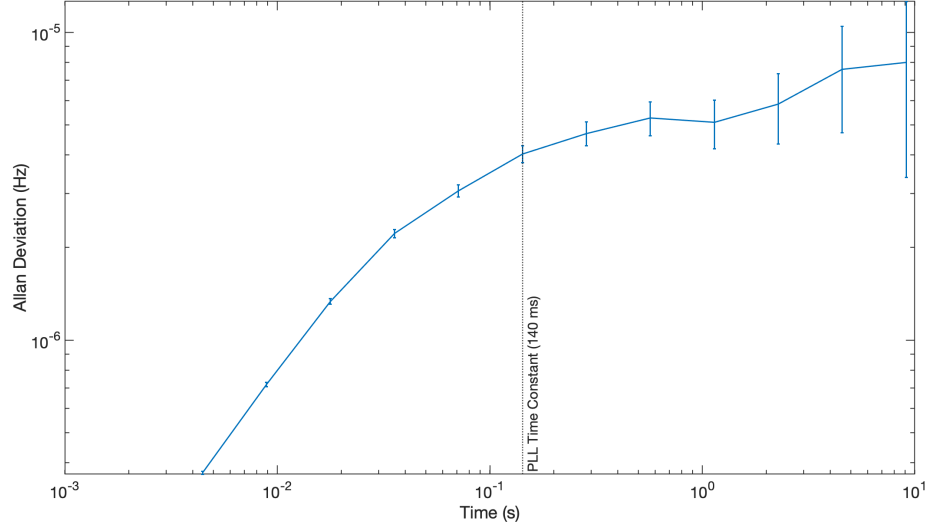


Figure 4.7: Allan Deviation plot for the device that is used in double paddle NEMS 200 nm F-PSNP experiments.

be seen in the PLL data in Figure 4.8.

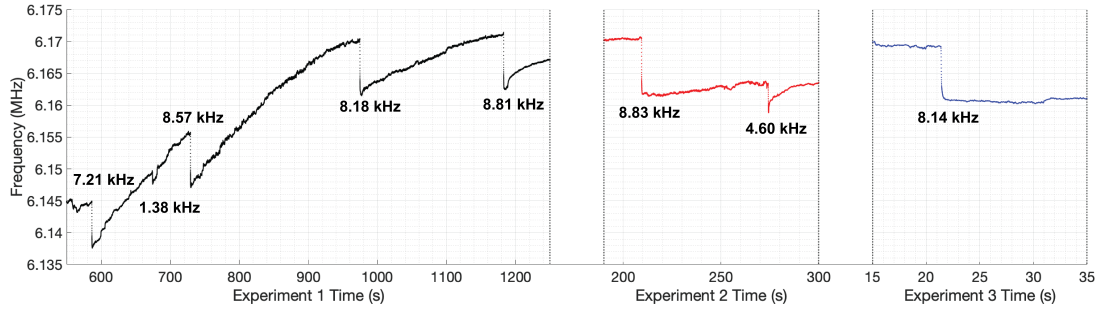


Figure 4.8: PLL data of the device that is used in double paddle NEMS 200 nm F-PSNP experiments. 8 landing events can be seen in 3 consecutive experiments.

From the 8 landing events, 6 of them are very close to each other in terms of the induced frequency shifts. Therefore, this similarity is promising to validate the uniformity of the mode shape on the platform. Thus, we continued with taking a fluorescent image of the device after the third experiment to find the particle positions. The fluorescent image of the device after the last experiment can be seen in Figure 4.9.

In the fluorescent image in Figure 4.9, one can observe that there are 8 particles

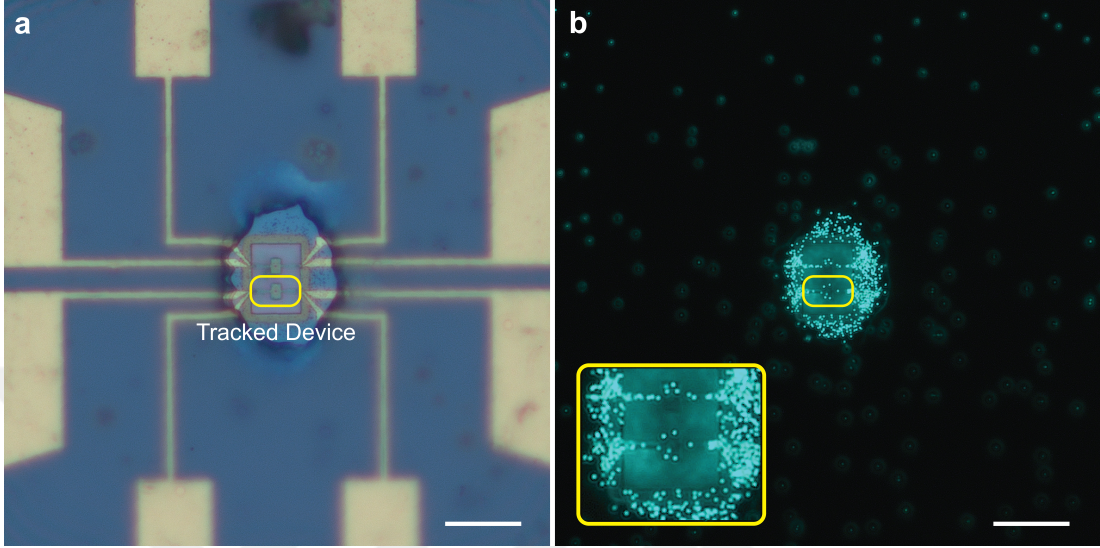


Figure 4.9: (a) Microscope image of the device used in double paddle NEMS 200 nm F-PSNP experiments. Scale bar is 15 μm . (b) Fluorescent image of the device after the third experiment. Scale bar is 15 μm .

on top of the tracked device, 6 of them on the platform, one of which is at the edge. The remaining two are observed on the supporting beams. Unlike the single paddle NEMS device experiment, these two events proved that any particle landing on the supporting beams results in lower frequency shifts than expected. Also, the particle positions that are gathered from the fluorescent image correlate with the PLL data that was shown in Figure 4.8. In Table 4.1, the 6 similar frequency shifts and a detailed analysis of these frequency shifts can be seen.

Table 4.1: Detailed analysis of 6 similar frequency shifts in double paddle NEMS 200 nm F-PSNP experiments.

Event Number	1	2	3	4	5	6
Frequency Shifts (Hz)	7212	8567	8176	8808	8831	8140
Calculated Diameter (nm)	203.34	215.22	211.73	217.05	217.24	211.43
Mean (Hz)	8288.8					
Standard Deviation (Hz)	606					
Coefficient of Variation	7.31%					

The coefficient of variation is calculated as 7.31%, which is low if we consider the polydispersity of the PSNP particles. The polydispersity of the 200 nm F-PSNP is reported as $<10\%$, which correlates with the PLL data and the

frequency shifts. Other sources that result in this percent standard deviation can be the differences in the displacement on the platform, which was discussed in Chapter 3. Overall, this device resulted in a narrower frequency shift distribution when compared to the single paddle NEMS device.

Also, it is important to investigate the outlier particle (#1) in Table 4.1, which created a lower frequency shift compared to the other 5. We attribute this to the particle that was observed at the edge of the platform. If we were to omit the frequency shift that was induced by this particle while calculating the coefficient of variation, it comes as 3.92%. This value validates the uniformity of the mode shape even further by experimental means.

The discussion that took place in Chapter 3 on the uniformity of the platform mode shape based on the simulations is further proven by the 200 nm F-PSNP experiments for the case of double paddle NEMS devices. The frequency shifts that were observed on the PLL data correlate with the fluorescent images of the device after the last experiment. We observed 8 particles on the fluorescent image and 8 frequency shifts on the PLL data, and the analysis on Table 4.1 proves that the mode shape is uniform on the platform since the induced frequency shifts are very similar to each other. The experimentally proven uniform mode shape on the platform for double paddle NEMS devices enabled us to continue to perform mass sensing experiments using double paddle NEMS architecture and 40 nm gold nanoparticles.

4.2.2 40 nm Gold Nanoparticle Mass Sensing Experiments Using Double Paddle NEMS Devices

The validation of the uniform mode shape for paddle NEMS devices in Chapter 3 by simulations and in Section 4.2.1 for double paddle NEMS devices by the experiments allowed us to continue further with the mass sensing experiments using 40 nm GNPs and a double paddle NEMS device. In these experiments, the solution that was discussed in Section 3.3 containing 40 nm GNP was used.

Before starting the actual experiment, we took the open sweeps of the devices, which can also be seen in Section 3.2, in Figure 3.4, and in Figure 3.5. We used the device that was discussed in Chapter 3 in these experiments. Therefore, the details of this device are not discussed one more time in this section.

The chip that was used in 40 nm GNP experiments contains two paddle NEMS devices and can be seen in Figure 2.7. After setting up the PLL for both devices, we calculated the Allan Deviation and the minimum detectable mass for each device. The Allan Deviation plots can be seen in Figure 4.10. The devices that are used in this section are referred to as device 1 (without hole) and device 2 (with hole) and can be seen in Figure 3.7 as a Render and in Figure 2.7 as an SEM image.

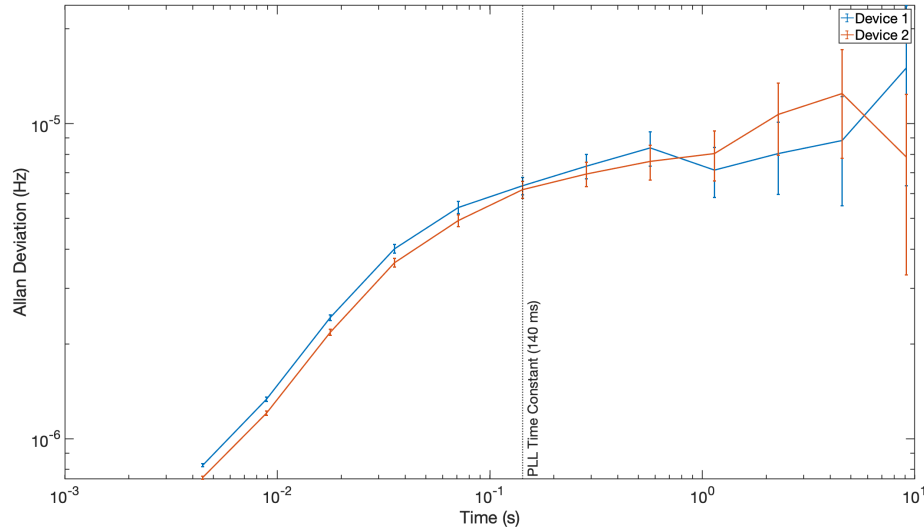


Figure 4.10: Allan Deviation plots for the devices (*i.e.* device 1 and device 2) that are used in 40 nm GNP experiments.

The Allan Deviations are calculated as 6.35×10^{-6} and 6.18×10^{-6} for device 1 and device 2, respectively. After calculating the Allan Deviations, we continued with the calculation of minimum detectable mass for each device using Equation 4.1. The minimum detectable masses are 40.3 ag (≈ 24.3 MDa) and 37.7 ag (≈ 22.7 MDa) for device 1 and device 2, respectively. Again these values are comparable to doubly-clamped beam devices that were used in the literature [1], however are higher when compared to the other devices used in the previous

sections.

Next, we started the experiment by starting the electrospray that contains 40 nm GNPs to the NEMS device. In the PLL data, we observed a total of 181 events from both devices. Device 1 collected 139 events, whereas device 2 collected 42 events. The simultaneous PLL data can be seen in Figure 4.11. In this PLL data, we observed that a frequency shift on one device does not create any frequency shift on the other one. Therefore, they don't have any effect on each other in the case of a landing event.

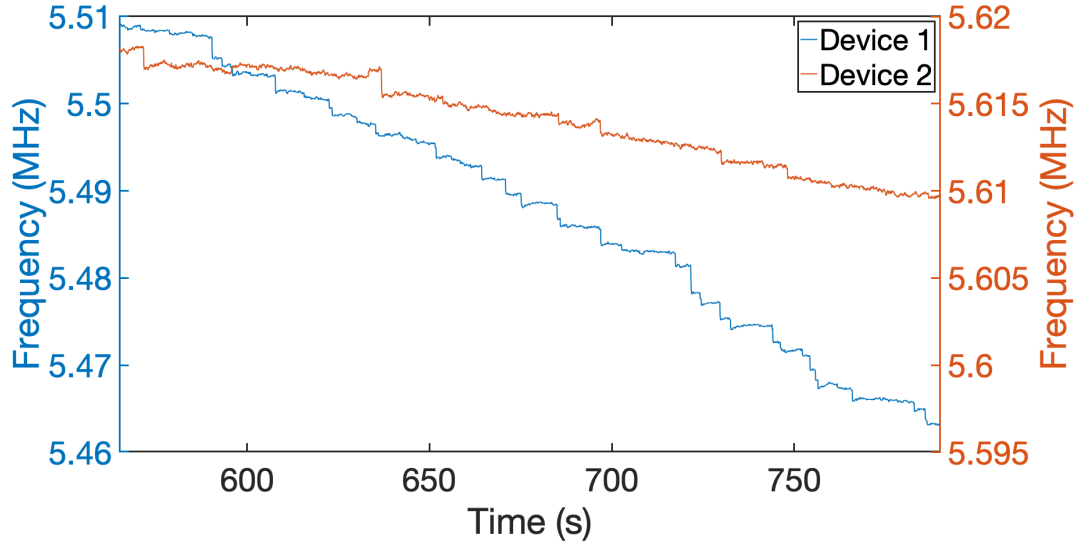


Figure 4.11: Simultaneous PLL data of device 1 and device 2 in 40 nm GNP spray experiment.

After collecting the frequency shifts from the PLL data, we calculated the particle mass values as discussed in Section 3.5 in Equation 3.15.

Since we calculated the effective mass of each device (see Section 3.5) and collected the frequency shifts and initial frequency values from the PLL data (see Figure 4.11), the only unknown left is the Δm values for each landing event. Using Equation 3.15 we calculated the Δm values, and the constructed mass histogram from both device 1 and device 2 can be seen below in Figure 4.12a. Also, it is important to note that beam mass is updated after each landing event with the mass of each landing particle.

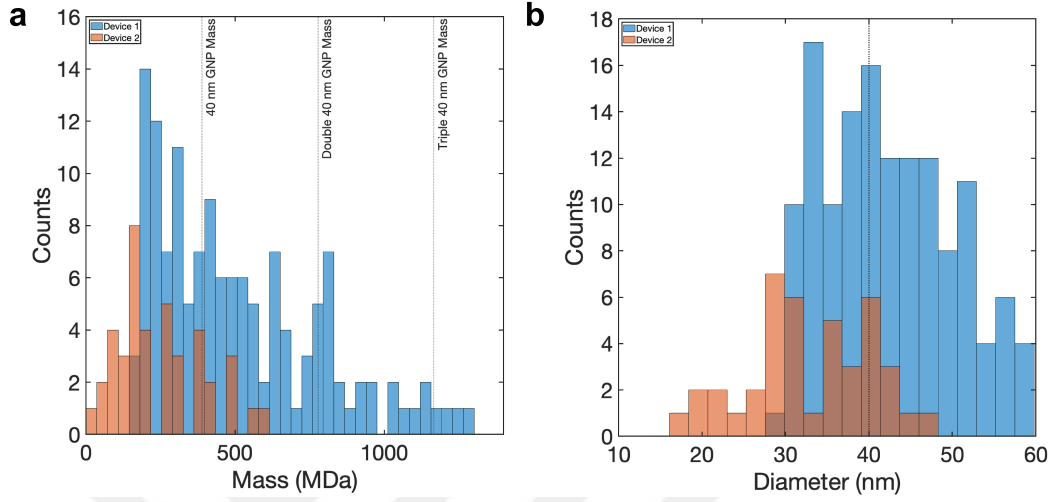


Figure 4.12: 40 nm GNP (a) mass, and (b) diameter histograms for both device 1 and device 2.

In the mass histogram that comes from device 1, one can see that there is an emerging peak on the right of the expected 40 nm GNP mass. Also, again, in the device 1 mass histogram, there are almost 15 events in the vicinity of double 40 nm GNP mass. However, due to the small number of events that device 2 collected, there are not many events around the mass of the 40 nm GNP in the mass histogram of device 2. Nevertheless, there are 6 events that are close to the 40 nm GNP mass. Also, we attribute the lower end of the mass histograms to the particles that landed on the supporting beams, as previously shown in 200 nm F-PSNP experiments. Since the displacement values (*i.e.* mode shape) in supporting beams are lower when compared to the platform, any landing particle is expected to induce a lower frequency shift than expected. Lastly, it is important to note that the bin widths in both device 1 and device 2 mass histograms are chosen to be two times device 1 minimum detectable mass since it is larger compared to device 2 minimum detectable mass.

After calculating the mass values for the landing events for device 1 and device 2, we calculated corresponding diameter values using the density of gold (*i.e.* 19.27 g/cm^3) and assuming the particles are spherical ($m = \rho_{\text{Au}} \times \frac{4}{3}\pi(\frac{d}{2})^3$). The diameter histograms can be seen in Figure 4.12b.

In the diameter histogram for device 1, there is a peak at 40 nm, and the average value, considering all of the events, comes as 42.65 nm. However, due to the low number of events and the events that are coming from the supporting beams, the constructed diameter histogram for device 2 has a lower average which is 32.96 nm. Nevertheless, there is an observed peak at 40 nm in the device 2 diameter histogram. It is also important to note that the bin widths in diameter histograms are chosen to be the deviation that 2 times the mass resolution would create around the expected diameter.

After the mode shape validation experiments using 200 nm F-PSNP and both single and double paddle NEMS devices, the 40 nm GNP mass sensing experiments showed that the mode shape independent mass sensing is possible thanks to the uniform mode shape on the platform in paddle NEMS devices. Therefore, using atmospheric pressure NEMS-MS system and paddle NEMS devices, the mass sensing can now be performed independent of the device mode shapes with the remaining limitation being particles landing on supporting beams.

Chapter 5

Conclusion and Future Work

Atmospheric pressure NEMS-MS demonstrated the characterization of nanoparticles successfully under atmospheric conditions while increasing the capture efficiencies and decreasing overall system cost. Therefore, the NEMS-MS field is more accessible now than before, thanks to the ability to operate entirely under atmospheric conditions. However, it is shown that deploying the doubly-clamped NEMS devices under atmospheric conditions breaks the symmetry in their displacement leading to some uncertainties in the governing equations for mass sensing applications. Here, we proposed a method and a device architecture to resolve this uncertainty instead of adding corrections to the aforementioned governing equations. Paddle NEMS devices removed this uncertainty by enabling mode shape independent mass sensing under atmospheric conditions. The simulations done to investigate the uniformity in the paddle NEMS device mode shapes showed that the displacement on the middle platform is uniform, therefore, in the case of a particle landing, the frequency shift is not a function of the landing position anymore. We can now relate the mass of a landing particle, without considering the landing position on the platform, to the mass of the platform and the frequency shift.

After validating the mode shapes by simulations, 200 nm fluorescent polystyrene nanoparticle experiment was performed using single paddle NEMS

devices for the experimental validation. In this experiment, the diameter histogram resulted an average diameter value of 194.9 nm with 13 events in the vicinity of 200 nm. Also, this 13 particles were observed on the platform using the fluorescent image of the device after the experiment. The correlation between the diameter histogram and the fluorescent microscope image validated the uniform mode shape of the platform and enabled us to perform mode shape independent mass sensing experiments under atmospheric conditions.

Next, we utilized double paddle NEMS devices featuring two resonators side-by-side. First we conducted uniform mode shape validation experiments using 200 nm fluorescent polystyrene nanoparticles. In these experiments a total of 8 landing events were observed, and out of these 8 events, 6 of them were observed on the platform. This visual inspection correlates with the PLL data, where 6 out of 8 landing events resulted in similar frequency shifts. This experimental validation on the mode shapes for double paddle NEMS devices enabled us to continue with mass sensing experiments. In mass sensing experiments, we used 40 nm gold nanoparticles while performing PLL on two devices on the same chip. One of these devices collected 139 events, and we observed a peak at the expected mass value for 40 nm gold nanoparticles. Also, these 139 events yield an average diameter value of 42.65 nm. Therefore, in this thesis, the mode shape independent mass sensing under atmospheric conditions was demonstrated successfully considering both single and double paddle NEMS architectures.

Paddle NEMS devices not only enable single mode mass sensing but open doors for an alternative, possibly more accessible, integration of arrayed NEMS devices. When the NEMS devices are designed as arrays, a sequential read-out scheme is used since it is almost impossible to address each device simultaneously [41]. Here, by showing that single mode mass sensing is possible under atmospheric conditions, the amount of addressing gold lines and electronics decreases in the case of an arrayed paddle NEMS application and thus makes the design and applications of arrayed NEMS architectures easier.

Another important thing to discuss is the alteration in the displacement profiles of paddle NEMS devices. Since it was shown that the displacement profiles alter

in atmospheric conditions in the case of doubly-clamped beam devices, paddle NEMS devices are also prone to this alteration. However, the quality factors we observed in our devices were close to 60, suggesting that the problem is rather minimal. Also, other device architectures can be used, such as actuation on two sides of the device and detection from the other two sides. In this configuration, the actuation on both sides might prevent the alteration in the displacement profiles since the actuation electrodes will be equally distanced from the middle position of the device. SEM image of such a device architecture can be seen in Figure 5.1.

Lastly, it is important to eliminate the particle focusing towards the supporting beams, since any particle landing results in lower frequency shift than expected thus creates uncertainty in the measurements. This drawback of paddle NEMS devices integrated with polymeric focusing lenses can be eliminated with narrower focusing windows which requires a high resolution photolithography with a thick photoresist. Another option can be using electron beam lithography to make a narrower window which will focus the particles only to the middle portion of the device (*i.e.* the platform). Also, 3D focusing lens structures can be deployed using tools such as two photon lithography.

To conclude, in this thesis, single mode mass sensing was demonstrated using paddle NEMS devices where these devices have a uniform region in their mode shapes. This demonstration opens doors to new applications and eliminates the mode shape dependency in mass sensing applications.

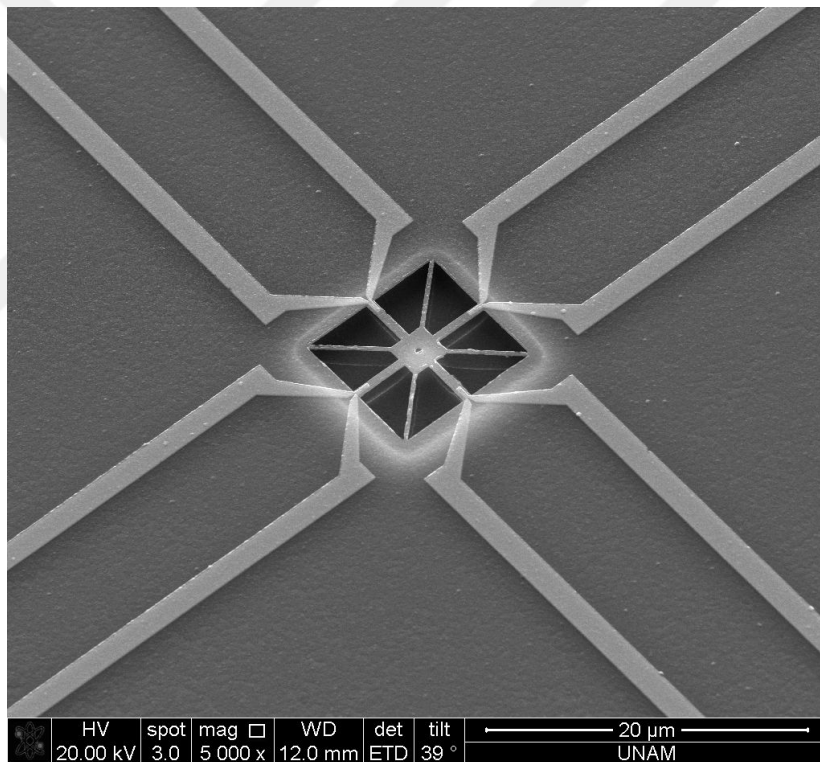


Figure 5.1: Proposed device to eliminate the alteration in the displacement profile of paddle NEMS devices.

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Appendix A

Micro/Nanofabrication Process

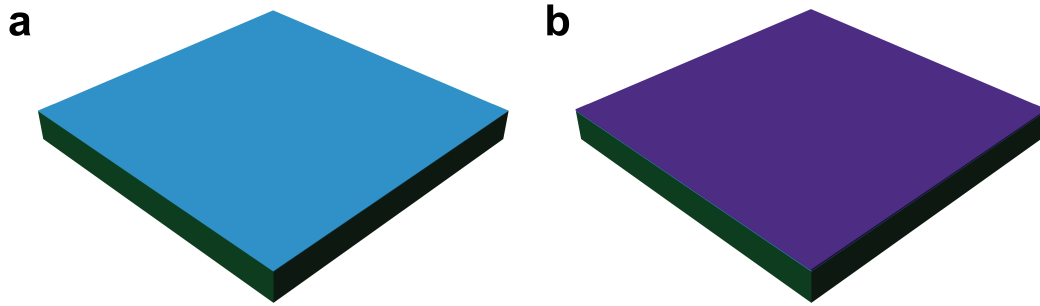


Figure A.1: (a) The bare silicon nitride on silicon wafer before any process. (b) Bilayer PMMA coated silicon nitride on the silicon wafer.

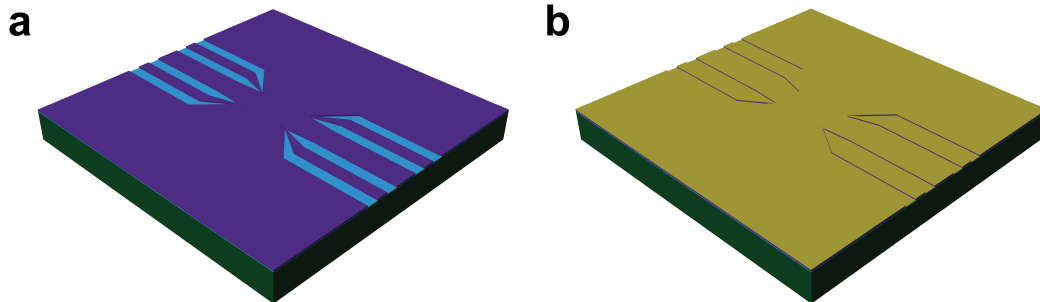


Figure A.2: (a) The wafer after exposing and developing the bilayer PMMA. (b) The wafer after the deposition of 5 nm chromium adhesion layer and 70 nm gold.

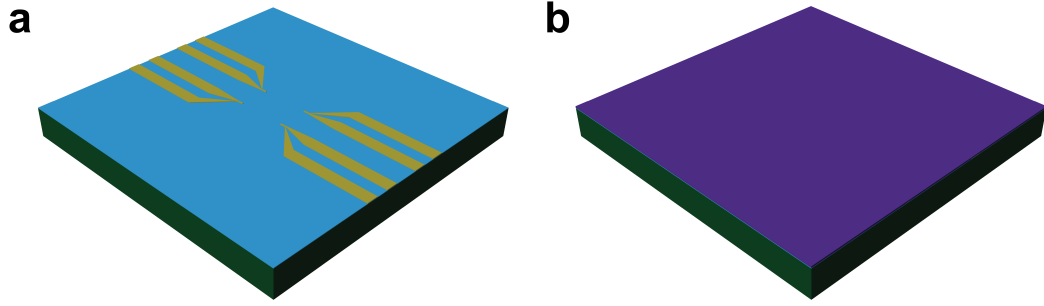


Figure A.3: (a) Substrate after the first lift-off step. Gold resistances can be seen on top of the silicon nitride film. (b) The substrate after the second bilayer PMMA coating.

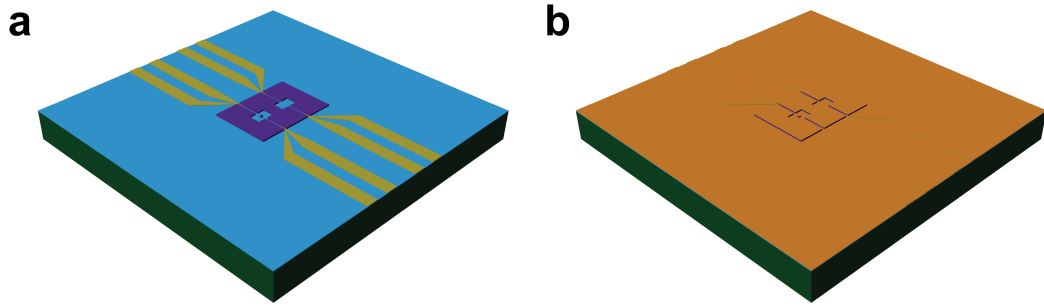


Figure A.4: (a) The substrate after the development of the second bilayer PMMA. After this step, the substrate is ready for copper coating. (b) The substrate after 40 nm copper hard mask coating.

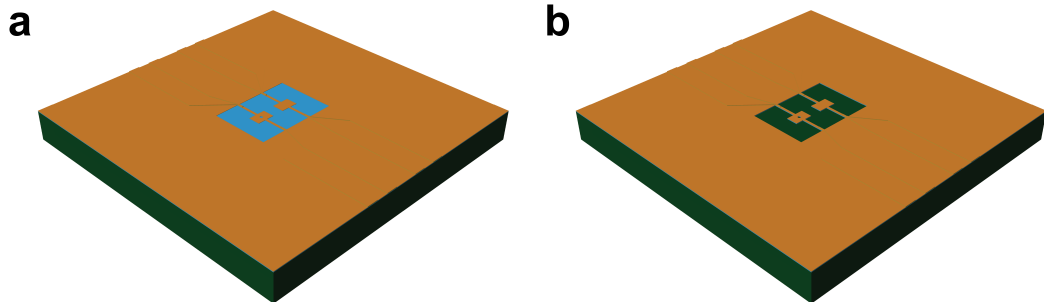


Figure A.5: (a) The substrate after the second lift-off step. It is now ready for the dry etching steps. (b) The substrate after anisotropic Si_3N_4 etching step. The underlying silicon can be seen in the non-protected region (*i.e.* next to the mechanical structures).

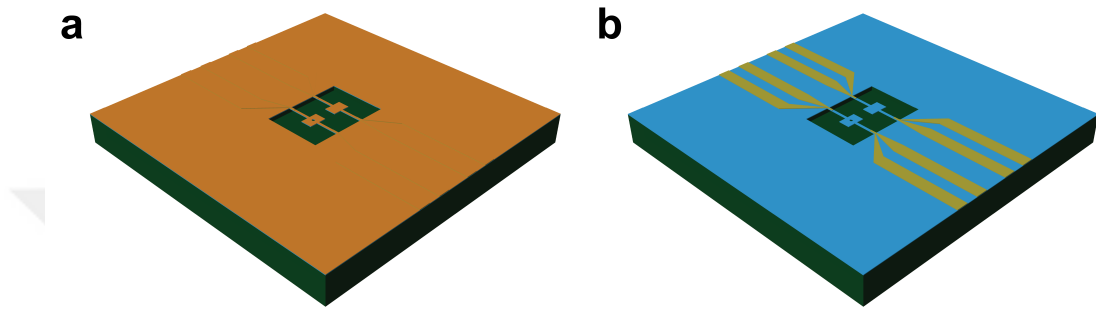


Figure A.6: (a) The suspended structure can be seen after the isotropic silicon etching step. (b) The ready to be used device except for the polymeric focusing lens.

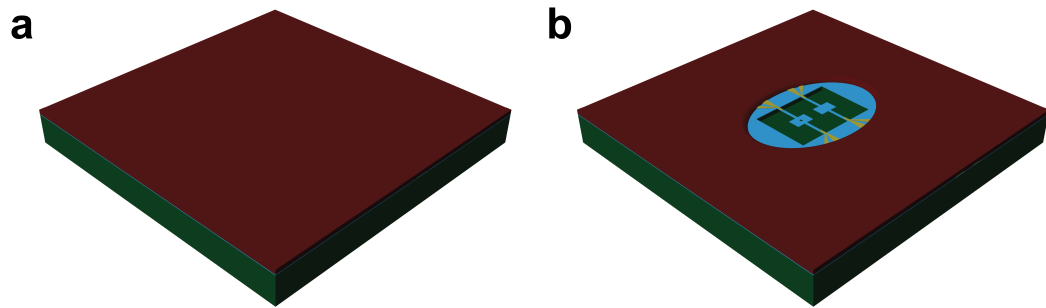


Figure A.7: (a) Thick photoresist coated device. (b) The paddle NEMS device at the end of fabrication.

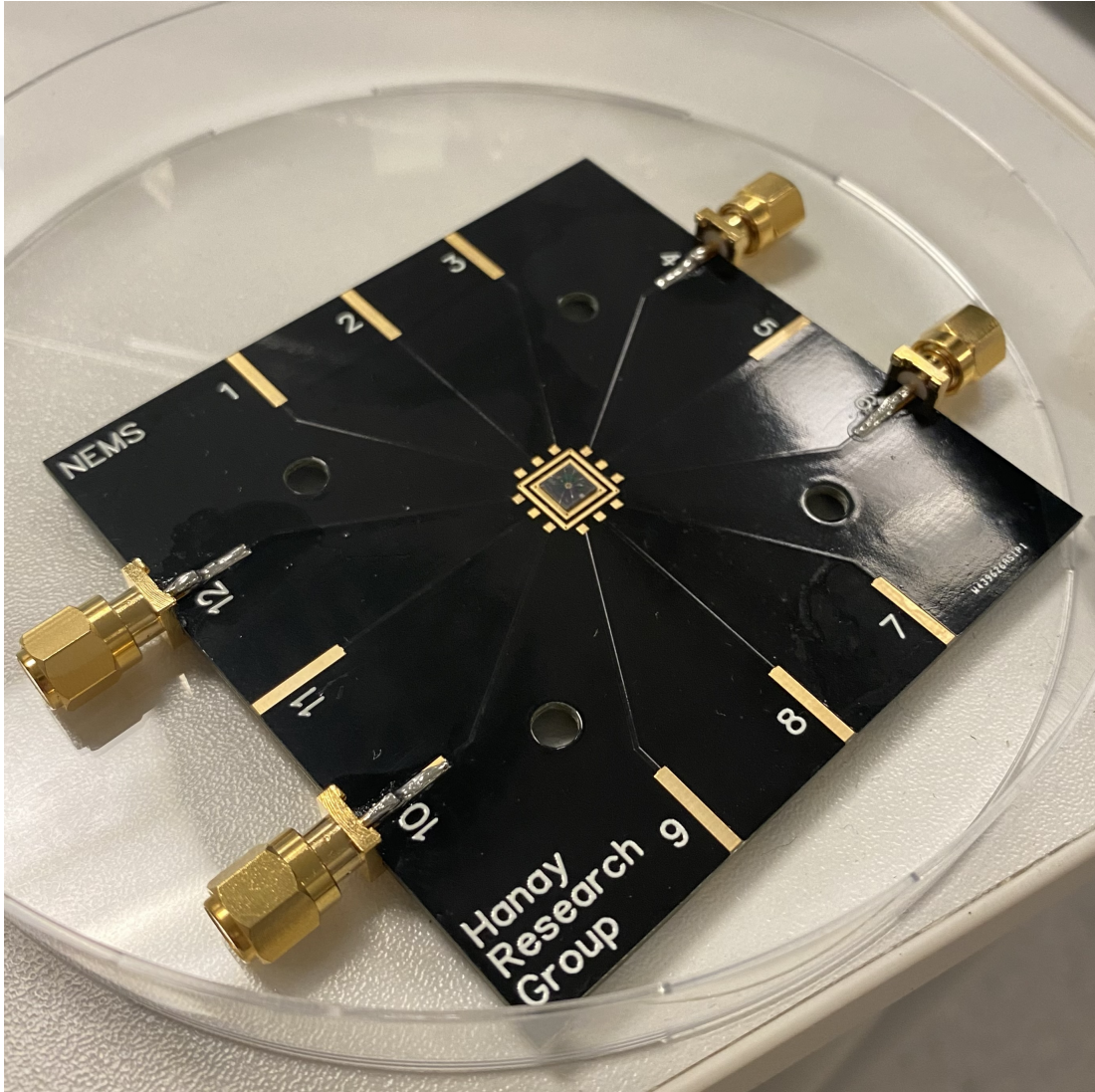


Figure A.8: Double paddle NEMS device on a PCB after wire bonding step. This device in the picture is ready to be used in the experiments.