Tailored Tunability of Nanoelectromechanical Systems for Mode Coupling and Thermal Studies

A Thesis

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by

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DECLARATION

This thesis is a presentation of my original research work. Wherever contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature, and acknowledgement of collaborative research and discussions.

The work was done under the guidance of Prof. Mandar M. Deshmukh, at the Tata Institute of Fundamental Research, Mumbai.

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In my capacity as supervisor of the candidate’s thesis, I certify that the above statements are true to the best of my knowledge.

Prof. Mandar M. Deshmukh

[Guide’s name and signature]

Date:
To My Parents
The last five years have been truly remarkable and have played a major role in molding me into the person I am today.

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Synopsis

Overview

In this synopsis I summarize the experiments we have done using suspended, electromechanical devices of graphene membranes and InAs nanowires. The experiments involved studying the mechanical vibrations of these nanostructures with resonant frequencies $\sim 100$ MHz. The experiments using graphene involved fabrication of suspended drums with low built-in tension for large electrostatic control. This work focussed on parametric amplification of mechanical motion, and strong coupling between modes. The InAs nanowire resonators, on the other hand, were fabricated in a high tension state with diminished electrostatic tunability. This enabled us to do experiments of localized Joule heating to extract the thermal properties of the nanowire, study the contribution of clamping losses, and non-linear mode coupling in the resonator.

Outline

Nanoelectromechanical systems (NEMS) have brought about tremendous improvements in the field of sensing by pushing the limits of measurement resolution down to the atomic and quantum limits [1, 2, 3, 4, 5, 6]. They have also enabled the study of various fundamental phenomena. In particular, quantum properties of mechanical motion have been generating much interest recently [7, 8, 9, 10, 11].

Experiments in cavity optomechanics [12, 13, 14] have demonstrated the ability to
prepare mechanical systems in their ground state \[7, 8, 9, 15\]. In such experiments a low frequency mechanical oscillator is coupled to an electromagnetic mode of a cavity \[11\]. Figure 1(a) shows a schematic of such experiments where one wall of an electromagnetic cavity is formed by a mechanical oscillator. The mechanical vibrations modulate the resonant mode of the cavity due to the change in length of the cavity. This coupling can be exploited to manipulate the mechanical motion by appropriate pump-probe schemes as shown in figure 1(b)-(c) \[11, 12\]. Since mechanical resonators support a large number of vibrational modes, a natural extension of the optomechanical scheme is to use coupled modes of a mechanical resonator where a high frequency mode plays the role of the cavity \[16, 17\]. There has been considerable interest in exploring coupling among eigenmodes of a mechanical system, demonstrating coherent coupling between low frequency modes \(<10\) MHz) \[16, 17, 18, 19\]. The eventual goal of such experiments is to demonstrate quantum coherent coupling \[20, 21\]. Mechanical resonators using graphene offers advantages for these experiments due to their high mechanical frequencies and large zero-point motion \[22, 23, 24, 25\].

The electrical and mechanical properties of graphene have also made it an ideal candidate in NEMS based sensors. However, the quality factors obtained in graphene resonators have been lagging behind in comparison to other NEMS devices including resonators using carbon nanotubes \[26, 27\]. Parametric amplification of the mechanical response can address this problem. This requires large frequency tunability of the resonator.

In this work, we have fabricated graphene drum resonators \[28, 29, 30\] with low built-in tension yielding large frequency tunability at low temperatures to study:

- parametric amplification of motion
- dynamical strong coupling between mechanical modes by
  - normal mode splitting and
  - non-degenerate parametric amplification

in an all-electrical configuration. We also show that the strong coupling persists for modes with frequencies differing by a factor of 2. Strong coupling between mechanical
Figure 1: Figure taken from reference [14]. (a) Schematic of a mechanical resonator coupled to an optical cavity. (b) Schematic to interpret optomechanical interactions that create amplification (b) and back-action cooling (c) of the mechanical resonator. With the blue detuned pump, phonons are created leading to mechanical amplification, whereas with a red detuned pump, mechanical quanta are removed from the oscillator leading to cooling.
modes with large frequency separation is essential to realize optomechanics experiments using two coupled phonon modes \[7, 15\]. Furthermore, the high frequencies attainable in our system could help realize vacuum squeezed states of mechanical motion at low temperatures \[21\]. This work is detailed in the section “Graphene drums under low tension”.

Another aspect that we have studied in this work relates to the measurement of thermal properties of InAs nanowires using a NEMS geometry. Thermal conductivity and expansion coefficient play an important role in the performance of NEMS devices. Thermal stresses cause frequency shifts due to the non-zero thermal expansion coefficients and reduced thermal conductivity of nanostructures \[31, 32, 33\]. In the work involving InAs nanowire resonators, we have used the sensitivity to thermal stresses to measure the thermal conductivity of the nanowire and study the effect of a negative thermal expansion coefficient. Conventional techniques make use of external thermometry for thermal measurements on individual nanostructures \[34\]. Our method uses the system itself for thermometry by converting any change in temperature into a stress. We make use of localized Joule heating to monitor the frequency shifts with the applied bias voltage. This requires devices with large built-in tension so as to minimize frequency shifts due to change in electrostatics, allowing us to measure frequency shifts solely due to heating. In these experiments, we have also measured the quality factor to study dissipation as a function of the nanowire temperature with local heating while keeping the contact metal electrodes at bath temperature. Microscopic mechanisms of energy loss have been studied extensively in NEMS resonators to improve their performance \[35, 36\]. We show that the dissipation in our resonators show strong dependence on the temperature of the contact electrodes suggesting clamping loss mechanism at the interface of the dissimilar materials. The work on InAs nanowire resonators is discussed in the section “Suspended InAs nanowires under high tension” which presents:

- thermal conductivity measurements of InAs using NEMS geometry
- the role of material dependent clamping losses
- non-linear mode coupling in the resonator

All the experiments described below involve the actuation and detection of vi-
brational modes of the nanostructures. NEMS devices have made use of a variety of methods to transduce mechanical motion including electrical, optical, and magnetomotive techniques [37, 38, 39]. In our experiments we use an electrical transduction scheme where the response is measured directly at the actuating frequency using a lock-in or network analyzer [40]. The suspended material and the gate electrode, together, comprise the plates of a capacitor and the capacitive force between them is used to drive the suspended portion into oscillations. This is realized by applying a dc voltage ($V_{dc} \sim 10 \text{ V}$) along with a small ac voltage ($\tilde{V}_g \sim 1 \text{ mV}$) on the gate electrode. The dc voltage induces a static pull on the suspended nanostructure, inducing a tension, and the ac voltage provides the driving force. The resonant frequencies of the NEMS studied here are in the $\sim 50$-100 MHz regime. Hence, all the devices are fabricated on insulating, sapphire substrates to minimize radio frequency (rf) signal loss due to parasitic capacitances.

Graphene drums under low tension

Low built-in tension in NEMS gives good electrostatic control of the various resonant modes of the system. Previous electromechanical devices of graphene have shown limited tunability of the resonator modes at cryogenic temperatures due to the large tension developed while cooling [29, 30]. This limits their functionality for use in optomechanics experiments in the quantum regime.

Device fabrication and frequency tuning

Here we have fabricated a drum resonator where the graphene is incorporated into the device in the last step of fabrication. Sapphire serves as the substrate to minimize signal loss due to parasitic capacitance. The fabrication involves designing a gate electrode on the sapphire, coating the sapphire substrate with 300 nm SiO$_2$, and further etching the SiO$_2$ ($\sim 3 \mu\text{m}$ diameter) in the desired region to form the drum hollow. Source and drain electrodes are patterned on the SiO$_2$ near the hole, and graphene is transferred over the electrodes and hole region. As the graphene flake is not clamped down by metal electrodes, the flake is able to relax when cooled down
Figure 2: (a) False colored scanning electron microscope image of the graphene drum resonator. The green region shows the suspended part of the graphene. Scale bar is 3 µm. (b) Schematic of the circuit used to actuate and detect the mechanical modes of the drum. (c) Large frequency tunability of the modes with dc gate voltage at 5 K.

to low temperatures and achieve a low tension state. A scanning electron microscope (SEM) image of the drum device is shown in figure 2(a) along with the circuit used to actuate and detect the mechanical motion in figure 2(b). The gate electrodes and the drum act as plates of a capacitor system, so a voltage applied on the gate electrode attracts the graphene flake towards it. As the role of the dc gate voltage is to cause a bending induced tension in the flake, we see that the resonant frequency increases monotonically with applied gate voltage as seen in figure 2(c). Previous devices have not shown such large monotonic change in the resonant frequency of graphene resonators at low temperatures. We use this tunability for the experiments discussed next.

**Parametric amplification**

The large tunability (>1 MHz/V) of the mechanical modes with gate voltage makes parametric pumping possible. Parametric pumping involves modulating the resonator spring constant at twice the resonant frequency. As the gate voltage tunes the tension in the membrane, an additional electrical signal that modulates the tension at twice
the resonant frequency is added onto the gate electrode to parametrically pump the resonator. The amplitude of the pump signal decides the parametric modulation of the tension. Beyond a critical pump amplitude, $V_{pc}$, the resonator enters a regime of self oscillations even in the absence of a driving force as shown in figure 3(a).

For pump amplitudes below $V_{pc}$ we can amplify the motion of the resonator. The phase relationship between the drive and pump signals decides whether the motion is amplified (gain > 1) or deamplified (gain < 1), where the gain of the resonator is defined as the ratio of amplitudes with pump on and off. With our device geometry we are able to demonstrate parametric amplification in multiple modes of the graphene drum resonator as shown in figure 3(b). We observe a gain of nearly 3 in our resonator. Theoretically, the gain goes to infinity when the amplitude of the pump voltage approaches the critical pump voltage. However, we see a stagnation of the gain possibly due to non-linear damping effects present in the graphene membrane. This could be useful in making on-chip amplifiers and allow improvement of graphene based NEMS sensors.

### Strong coupling between mechanical modes

The large tunability of the graphene drum modes further allows us to tune the coupling between various modes as the frequency separation between various modes can
Figure 4: (a) Zoomed in scan of frequency response of the coupled modes as a function of the dc gate voltage showing avoided crossing behavior with no parametric pumping. Red pump experiments are done at the gate voltage indicated by the white dotted line where $V_{g}^{dc} = -36$ V. (b) The red detuned pump is shown alongside the two coupled modes on a frequency axis. (c) Response of mode 1 as a function of the red pump detuning when $V_{p} = 1.5$ V. For nonzero pump amplitude, mode 1 is seen to split in the vicinity of $\omega_{p} \approx \Delta \omega$. Color scale units are $\mu$V. Inset shows the response detected at $\omega_{d} + \omega_{p}$. (d) With an increase in the red detuned pump amplitude, mode 1 is seen to split into two well resolved peaks with a separation given by $2g$. (e) Cooperativity of the modes is seen to be as high as 60 at the largest pump amplitudes. The solid line is a quadratic fit of the data (circles) to the equation $C = \alpha V_{p}^{2}$.

be tuned to cause mode crossings. Figure 4(a) shows the response of two modes that are brought into a region of avoided crossing by changing the DC gate voltage. Here, experiments similar to optomechanics experiments can be used to manipulate the mode coupling. We have demonstrated the dynamic strong coupling between the mechanical modes by pump probe experiments where the pump frequency is either the difference or sum of the individual modes.

First, we look at response of mode 1 when the pump frequency is detuned about the frequency difference (red pump: $\omega_{p} = \Delta \omega$) between the modes [figure 4(b)]. In the presence of a red pump the mode undergoes normal mode splitting as it hybridizes with the second mode as seen in figure 4(c) [16, 17, 44]. This is accompanied by
energy transfer to the second mode as shown in the inset where the response is detected at \( \omega_d + \omega_p \). The coupled modes undergo splitting when the pump induced coupling rate compensates the individual mechanical losses of the modes, and the amount of splitting of mode 1, equal to \( 2g \), is proportional to the pump amplitude [see figure 4(d)]. Here the hybridization of mode 1 and 2 gives rise to two new eigenmodes in the system as seen from the splitting. We have also confirmed the experimentally observed mode splitting by numerically solving the coupled equations of motion of the two modes. The coupling between the modes can be quantified by a figure of merit cooperativity, \( C \), using the frequency splitting \( (2g) \) and the individual dissipation rates \( (\gamma_i) \) of the modes as \( C = \frac{4g^2}{\gamma_1\gamma_2} \). We find a cooperativity as high as 60 [figure 4(e)] [45] between the high frequency mechanical modes. This quantifies the number of cycles of energy transfer that occurs between the coupled modes before it can dissipate to the bath. The parametric tunability of the modes further allows higher order coupling. This was observed experimentally as a mode splitting when \( \omega_p = \Delta \omega/2 \), equivalent to a two-phonon process. This feature was also well captured by the numerical solutions.

While a red detuned pump swaps the energy between modes, further manipulation
of the modes can be done by pumping at the sum of frequencies [blue pump, figure [5(a)-(b)] of the two modes. This can be thought of as an extension to the parametric pumping discussed in the subsection “Parametric amplification”. The response of the mode is seen to become narrower with pump amplitude as shown in figure 5(c). The presence of a blue pump gives rise to a lowered effective dissipation rate that amplifies the mechanical response of the mode. The effective dissipation rate is seen to reduce with increasing pump amplitude and follows a quadratic response [figure 5(d)]. The non-degenerate parametric amplification of these high frequency modes could possibly lead to realization of two mode squeezed states at sufficiently low temperatures [21].

We have also demonstrated normal mode splitting and amplification by pump-probe experiments on modes that have a frequency ratio of $\sim 2$. These experiments are a step towards optomechanics experiments in the quantum regime using NEMS of atomically thin membranes.

**Suspended InAs nanowires under high tension**

In this section I discuss the experiments we have carried out on doubly clamped InAs nanowire resonators of $\sim 100$ nm diameter. The InAs nanowire resonator devices are fabricated on sapphire substrates with the nanowire incorporated into the device from the first step of fabrication. This leads to the nanowire being clamped by metal electrodes that serve the additional purpose of making electrical contact to the nanowire [see figure 6(a) for SEM image of device and circuit used for measuring the mechanical response]. Stresses induced during the fabrication process, and relative expansion of the metal/nanowire/substrate system while cooling to low temperatures lead to a large built-in tension in these nanowire devices. This is usually observed as a natural frequency that is larger than the value predicted from classical beam mechanics. The large built-in tension in the nanowire further causes the resonant frequency to shift negatively with gate voltage due to the capacitive softening effect [figure 6(b)], a feature seen in NEMS devices under high tension. We develop a continuum mechanics model that extracts the built-in tension in the nanowire from the frequency response with gate voltage.
The resonant frequency, $f$, of the beam can be written as:

$$f = \frac{1}{2\pi} \sqrt[1]{\frac{k}{m_{eff}}}$$

where $k$ is the spring constant we obtain from the energetics. For a doubly clamped beam in its fundamental flexural mode, the effective mass is $m_{eff} = 0.397m$, where $m$ is the mass of the beam [46].

From our model we deduce the spring constant, $k$, to be dependent on the gate voltage, $V_g$, as:

$$k = \frac{1024EI}{5L^3} + \frac{512T_0}{105L} + \frac{262144EA}{3675L^3} z_e^2 - \frac{1}{2} C_g'' V_g^2.$$  

where $L$ is the nanowire length, $T_0$ is the built-in tension, $z_e = z_e(V_g)$ is the equilibrium distance, $E$ is the elastic modulus, $I$ is the area moment for cross sectional area $A$, and $C_g$ is the gate capacitance. We have used this expression to fit the experimentally observed tuning of resonant frequency with gate voltage and extract the built-in stress in the nanowire resonator to be $\sim 313$ MPa [see figure 6(b)]. We see that the overall shift in resonant frequency with gate voltage over a range of $\pm 20$ V is small. This is advantageous for the experiments described next.
Figure 7: (a) Experimentally observed variation of resonant frequency of the nanowire with source-drain bias voltage at 16 K bath temperature. The color scale units are in dB. The yellow line is the frequency shift obtained from simulations. The dotted black line shows the expected trend in frequency only due to electrostatics as the bias voltage is changed. (b) Frequency variation with $V_{sd}$ obtained at 20 K bath temperature. The color scale units are in dB. The green line shows the additional stress (right axis) induced in the nanowire due to Joule heating obtained from simulations.

**Measuring thermal conductivity of InAs nanowires**

As the frequency shift with electrostatics is small, we can study the response of the nanowire to localized Joule heating by changing the bias voltage without having electrostatic effects. We use localized Joule heating to estimate the thermal conductivity of the nanowire. This is done by measuring the frequency shift with Joule heating and comparing the observed frequency shifts with an analytical model as well as COMSOL simulations. The heat flow along the length, $L$, of the nanowire of cross-section $A$ is governed by the one dimensional heat equation $A \frac{d}{dx}(\kappa \frac{dT(x)}{dx}) + \dot{w} = 0$ where $T(x)$ is the temperature along the nanowire, $\kappa$ is the thermal conductivity, and $\dot{w}$ is the rate of Joule heating per unit length. Assuming a temperature dependent thermal conductivity of the form $\kappa = bT$ at cryogenic temperatures, we solve the heat equation analytically and get the temperature profile along the nanowire as:

$$T(x) = \sqrt{\frac{AbT_b^2 + L\dot{w}x - \dot{wx}^2}{Ab}} \quad (3)$$

where $x$ takes values between 0 and $L$, and $T_b$ is the bath temperature. This empirical form of $\kappa$ is supported by previous measurements of thermal conductivity of silicon and InAs nanowires that show near linear temperature dependence of thermal conductivity at cryogenic temperatures [47, 48, 49].
To study the frequency shift with Joule heating we need to understand the additional stress that builds up in the nanowire. For a tensile stress $\tau$ on a cylindrical beam of radius $r$, length $L$, and elastic modulus $E$, the natural frequency is given by

$$f_0 = \left(\frac{1}{2\pi} \sqrt{\frac{EI\beta^4}{\rho A}}\right) \sqrt{1 + \frac{0.55\tau A}{\beta^2 EI}}.$$  \hspace{1cm} (4)

where $I = \frac{\pi r^4}{4}$ is the area moment of inertia, $\rho$ is the mass density, $\beta = 4.73/L$ is the mode factor for the fundamental mode, and $A = \pi r^2$ is the cross sectional area. The stress can be written as $\tau = \tau_0 + \Delta\tau_h$ where $\tau_0$ is the initial stress in the nanowire. The additional thermal stress $\Delta\tau_h$ accumulates on the nanowire due to relative expansion and contraction when Joule heated. As the thermal expansion coefficient is strongly temperature dependent we obtain the longitudinal thermal stress by integrating the stress over the length of the nanowire using:

$$\Delta\tau_h = -\frac{E}{L} \int_0^L \left[ \int_{T_b}^{T(x)} \alpha[T(x)]dT \right] dx \hspace{1cm} (5)$$

where $T_b$ is the bath temperature and $\alpha$ is the expansion coefficient obtained from literature [51]. InAs has a highly temperature dependent expansion coefficient with a region of negative expansion around 10-50 K. Due to this we see that the nanowire frequency undergoes a non-monotonic shift with Joule heating as shown in figure 7 [52]. We calculate the shift of resonant frequency using the above model and compare with COMSOL simulations and experimentally observed shifts at four different bath temperatures to extract the value $b = 0.035$ W/mK$^2$ for $T < 60$ K. As the techniques for measuring thermal properties of individual nanostructures are limited, our method shows a way forward for such experiments.

**Material dependent losses in metal clamped resonators**

Our experiment also allows us to elucidate the role of clamping losses in doubly clamped beams. Figure 8(a) shows the quality factor of the nanowire reducing as it is Joule heated. The inverse of the quality factor gives us information about energy losses in the system. As the temperature of the nanowire rises with Joule heating we
can look at the loss as a function of the average nanowire temperature at various bath temperatures [35, 53, 54]. Figure 8 shows that the loss at lower bath temperatures is overall lower even when the average nanowire temperature is equal. This is indicative of material dependent losses at the clamping points [55, 56, 57]. The observation of significant loss at the clamps is further supported by the fact that our nanowire is under high tensile stress as clamping losses are known to be the dominant damping mechanism in doubly clamped beams under high pre-stress [57]. This shines light on the performance limitation of similar NEMS devices with metal clamps at low temperatures.

**Non-linear mode coupling**

We have also looked at the coupling between orthogonal components of the fundamental mechanical mode of the resonator. The nanowire resonator has two closely spaced resonant frequencies that correspond to the fundamental mode vibrating along the direction of the gate and perpendicular to it [see figure 9(a)]. These modes are coupled due to the bending induced tension that arises during vibrations. This is studied by monitoring the response of one mode while driving the second mode with large amplitude. Figure 9(b) shows the increase in frequency of mode 1 when the
resonant mode 2 is approached. As amplitude of mode 2 is large near resonance, the induced tension increases the resonant frequency of mode 1. The behavior of mode 1 is seen to follow the Duffing response of mode 2. This arises due to the presence of terms of the form $\alpha X^3$ and $\beta XY^2$ in the equation of motion where $X,Y$ denote the modal coordinates [58, 59]. We also observe hints of a modified energy dissipation from mode 1 in the coupled regime. Such studies could help improve performance of NEMS sensors and devices by understanding one of the relevant channels for energy loss.

Other projects

During the course of my research I have also been contributing to other projects in the Nanoelectronics lab. In one such project we have studied the photoresponse of WS$_2$ nanotubes and incorporated them into hybrid devices with graphene as contact electrodes. The multi-walled nanotubes have diameter of $\sim$ 100 nm. Scanning photocurrent microscopy can be used to study the local nature of photocurrent generation in individual nanotubes contacted by metal electrodes. This is done using a 532 nm laser modulated by an acousto-optic modulator and lock-in detection of the photocurrent. The spatial profile of the photocurrent along the length of the nanotube is used to extract the minority carrier diffusion length and carrier lifetime in the nanotubes. Further, WS$_2$ nanotube devices with graphene electrodes have been
Figure 10: (a) Optical image of a hybrid device of a WS$_2$ nanotube contacted by graphene electrodes. Scanning photocurrent measurements were carried out on these devices. (b) Photocurrent along the length of the nanotube is shown for three different gate voltages. The dotted line demarcates the position which shows a gate tunable photoresponse. Inset shows a photocurrent map of the hybrid device with the defect region marked by the dotted circle.

Fabricated to study the possibility of enhanced extraction of photo-generated carriers. Figure 10(a) shows an optical image of a graphene-WS$_2$ hybrid device. Figure 10(b) shows the photoresponse along the length of the nanotube in such a hybrid device (inset shows photocurrent map of complete device). We find that certain regions of the nanowire show a gate tunable photoresponse. These regions also show a different Raman spectrum as compared to the rest of the nanotube. This is indicative of a defect in the nanotube that gives rise to a localized electric field aiding photocurrent generation. These hybrid devices could be extended to other semiconductor systems for scanning photocurrent microscopy based defect detection [60].
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Chapter 1

Introduction

The thrust to miniaturize electronic devices has led to many new ideas and inventions in the field of nanotechnology. In particular, the last two decades have seen considerable research interest in nanoelectromechanical systems, referred broadly by the acronym NEMS. NEMS are devices that combine a nanoscale material with mechanical degrees of freedom, and a component that can stimulate or manipulate its motion\[1, 2\]. They originated from a need to outperform micrometer sized electromechanical devices (microelectromechanical systems or MEMS), and today they have dimensions deep in the sub-micrometer regime\[3\]. NEMS have facilitated research on new aspects of fundamental physics while also realizing a variety of technological applications. They are rapidly replacing MEMS in the quest for smaller, improved sensors\[4\].

Today’s NEMS are mostly ‘resonant’ devices, where the nanoscale mechanical element oscillates in one of its fundamental modes of vibration. These can be the flexural\[8, 9\], torsional\[6\], or breathing\[7\] modes of a solid. Figure 1.1 shows these modes seen in various resonators from previous works. The resonant frequency of such devices are generally higher than 1 MHz due to their low mass and can even be designed to be higher than 1 GHz\[10, 11\]. Additionally, the quality factor of NEMS resonators have been recently shown to be as high as $10^8$ at room temperature\[12, 13\]. The high quality factor implies that tiny shifts in the resonant frequency of these devices can be measured. This naturally makes them good sensors of very
Chapter 1. Introduction

Figure 1.1: (a) Scanning electron microscope (SEM) image of a diamond cantilever resonator. Figure adapted from Meesala et al.\textsuperscript{[5]}. (b) SEM image of a torsional carbon nanotube resonator. Figure adapted from Cohen-Karni et al.\textsuperscript{[6]}. Scale bar is 400 nm. (c) SEM image of a micromachined silicon resonator with breathing modes. Figure adapted from Eichenfield et al.\textsuperscript{[7]}. The right panels in (a)-(c) represent the relevant degree of freedom of the device.
small physical quantities that stimulate the resonator. This makes them useful for experiments of fundamental physics as well as for practical applications. They have been used in the detection of a single charge\cite{14}, single spin\cite{15, 16}, as well as for ultra sensitive mass and force sensing\cite{17, 18, 19, 20, 21, 22}. Additionally, the sub-nanometer displacement of these systems can be measured down to the fundamental limits set by quantum mechanics\cite{23}. This has led to many recent studies on the quantum mechanical nature of motion in mechanical systems\cite{24, 25, 26, 27, 28}.

Since the sensitivity of NEMS is inversely related to the dimensions and mass of the active part of the device, researchers have been trying to reduce the size of these devices. The traditional method of fabricating MEMS/NEMS devices relies on etching a material to micrometer/nanometer scale dimensions. This approach is known as surface micromachining\cite{1}. Another approach is to use the advantages of materials that exist or are grown at nanoscale dimensions. The ability to grow quasi one dimensional materials like nanotubes and nanowires, and the advent of layered two dimensional materials has guided this branch of NEMS.

The classic example of a layered two dimensional material is graphene, a one-atom thick sheet of carbon atoms. In 2004, it was shown that atomically thin layers of carbon can be peeled off from a bulk graphite sample to fabricate nanoscale devices\cite{29}. Graphene consists of a honeycomb lattice of $sp^2$ hybridized carbon atoms. It has been shown to have remarkable electrical and mechanical properties\cite{30, 31} including a high in-plane Young’s modulus and large breaking strength\cite{32}. The electrical and mechanical properties of graphene, combined with its low mass, have made it an ideal candidate for NEMS based sensors. NEMS using graphene were first demonstrated in 2007\cite{33}.

Ever since the advent of graphene and graphene based resonators, there has been a push to find alternate materials that could function as two dimensional (2D) electromechanical systems. One such class of materials are the transition metal dichalcogenides (TMDC’s) that are of the form MX$_2$ where M is a transition metal and X is a chalcogen atom. They are graphene-like layered materials that can be peeled down to atomic thicknesses for fabricating nanoscale electronic devices. Such 2D systems, due to their small dimensions and ultra low masses, can be used for ultrasensitive measurements of force, mass, and motion. Many groups have been thus pursuing
such systems for NEMS applications. Graphene based resonators coupled to electromagnetic cavities were recently shown to have a displacement sensitivity of 1.3 fm Hz$^{-1/2}$ and force sensitivity of $\sim$0.4 aN Hz$^{-1/2}$\textsuperscript{34}. There is also currently a research interest in improving the quality factor of these 2D materials based resonators. Although graphene based resonators have been shown to have quality factors as high as $2.2 \times 10^5$ at millikelvin temperatures \textsuperscript{35}, their quality factors have been lagging behind that of resonators of silicon and carbon nanotube based NEMS. Resonators made of WSe\textsubscript{2} were recently shown to have quality factors as high as $\sim 4.7 \times 10^4$ at helium temperature (3.5 K)\textsuperscript{36}.

Nanowires and nanotubes of metallic or semiconducting nature are also widely used as NEMS\textsuperscript{37, 38}. The ability to precisely control the dimensions, stoichiometry, and crystal structure of these materials gives them an added advantage\textsuperscript{39}. Semiconducting indium arsenide (InAs) nanowires are one such example. They are known for their high electron mobility and large spin orbit interaction, which make them useful for nanoelectronics devices. NEMS devices using InAs nanowires have also been demonstrated\textsuperscript{40}.

In our work we performed experiments on NEMS of graphene and InAs nanowires. The graphene devices had a drum resonator geometry, whereas the InAs nanowire resonators were doubly clamped beams. In both the experiments we probed the flexural modes of the resonators. In the NEMS that we study, the stimulus that tunes the resonant frequency of the device is a voltage applied to a gate electrode.
in close proximity to the mechanical element. The mechanical element and the gate, together, form the plates of a capacitor. Therefore, a dc voltage applied on the gate electrode attracts the resonator. This is schematically shown in Figure 1.2.

The tension induced by the bending of the resonator, thus, tunes the resonant frequency of the device. Here, the tunability of the device is inversely related to the initial tension in the device. The graphene drums we study are in a low tension regime whereas the InAs nanowire resonators have a large built-in tension. Therefore, in our experiments with the graphene drums, we see a large tunability of the device with gate voltage ($\sim 1$ MHz/V), and in the experiments involving InAs nanowire resonators we see low tunability ($\sim 10$ kHz/V).

A research field that has developed rapidly over the past decade, and one that is closely related to NEMS research, is cavity optomechanics [42, 43]. In cavity optomechanics, a low frequency mechanical oscillator is coupled to an electromagnetic mode of a cavity [28]. In such experiments, one boundary of the electromagnetic cavity is formed by a mechanical oscillator (see schematic in Figure 1.3(a)). The mechanical vibrations modulate the resonant mode of the cavity due to the change in cavity length. This coupling can be exploited to manipulate the mechanical motion by appropriate pump-probe schemes [28, 44] (Figure 1.3(b)-(c)). Experiments in cavity optomechanics [41, 44, 45] have demonstrated the ability to prepare mechanical oscillators in their quantum ground state [24, 25, 26, 46]. One disadvantage of an optomechanical system is that one has to integrate two distinct physical systems, the mechanical oscillator and the cavity.

A natural extension of the optomechanical scheme is to use coupled modes of a single mechanical resonator. Since mechanical resonators support a large number of vibrational modes, a low frequency mode can play the role of the oscillator and a high frequency mode can play the role of a cavity [47, 48]. There has been considerable interest in exploring coupling between mechanical modes of resonators [49, 47, 48, 50, 51]. One of the eventual goals of such experiments is to study the coupling dynamics in the quantum regime when the resonator is cooled to its ground state [52, 53]. Mechanical resonators using graphene offers advantages for these experiments due to their high mechanical frequencies and large zero-point motion [54, 35, 55, 56].

Our work on coupling the mechanical modes of graphene drums is discussed in
Figure 1.3: Figure adapted from Kippenberg et al. [41]. (a) Schematic of a mechanical resonator coupled to an optical cavity. (b) Schematic to interpret optomechanical interactions that create amplification (b) and back-action cooling (c) of the mechanical resonator. With the blue detuned pump, phonons are created leading to mechanical amplification, whereas with a red detuned pump, mechanical quanta are removed from the oscillator leading to cooling.
The devices that we fabricate are in a low tension regime such that they have large frequency tunability. The large tunability also allows us to tune the tension mediated coupling between different modes of the drum. Similar to optomechanical schemes, we show that dynamical strong coupling, and hybridization can be achieved between mechanical modes under the action of a pump tone at the frequency difference between the modes. We also demonstrate amplification of mechanical motion under the action of a pump tone at the sum of frequencies of the two modes. The tunability is further used to achieve parametric amplification of motion using a $2\omega$ pump.

Another aspect that we have studied in this work relates to the measurement of thermal properties of InAs nanowires using NEMS. The traditional method of measuring thermal conductivity of individual nanostructures is to integrate it into a complex device that has a heater and sensor arrangement (see Figure 1.4). These techniques make use of external thermometry for measuring the temperature change in the system. In our work, we use an alternate approach to measure the thermal conductivity of individual nanowires. Thermal conductivity and expansion coefficient play an important role in the performance of NEMS devices. Thermal stresses cause frequency shifts due to the non-zero thermal expansion coefficients and reduced thermal conductivity of nanostructures.

Our work involving thermal studies on InAs nanowire resonators is discussed in Chapter 6. In these experiments, we have used the frequency shifts of the resonator due to thermal stresses to deduce the increase in temperature and thermal conductivity of the nanowire. Our method uses the system itself for thermometry by converting any change in temperature into a stress. We make use of localized Joule heating to monitor the frequency shifts with the applied bias voltage. Here, we establish how the low tunability of the device helps us to measure the frequency shifts solely due to Joule heating. Microscopic mechanisms of energy loss have also been studied extensively in NEMS resonators to improve their performance. Using our device geometry, we shine light on a possible loss mechanism arising from the structural clamps of the suspended resonator.

This thesis consists of eight chapters. In Chapter 2, we present an overview on the physics of harmonic motion, dynamics of membranes and doubly clamped beams, and mechanisms of energy dissipation in NEMS. Fabrication of the devices used in
Chapter 1. Introduction

Figure 1.4: Typical heater/sensor arrangement used for measuring thermal conductivity of individual nanostructures. Figure adapted from Li et al.[63]. Scale bar of inset is 2 μm.

our work is discussed in Chapter [3] followed by Chapter [4] discussing the measurement schemes that we use. Chapter [5] discusses our experiments on mode coupling in the highly tunable graphene drum resonators. Chapter [6] discusses the thermal studies done on suspended InAs nanowires using electromechanics of the doubly clamped resonators under high tensile stress. In Chapter [7], we give an account of another project involving WS₂ nanotubes and their photoresponse. Finally, Chapter [8] summarizes the results presented in this thesis and provides an outlook for future work.
Chapter 2

Fundamentals of nanoelectromechanical systems

In this chapter we describe the underlying physics of mechanical resonance in NEMS. A suspended structure will undergo oscillations when perturbed. These oscillations occur at a characteristic natural frequency determined by the dimensions, material parameters and built in tension of the suspended structure. The classic example of this is a mass attached to a spring which undergoes oscillations when disturbed, where the frequency of oscillations is decided by the spring stiffness and the mass attached to the spring. When an external force perturbs the system at a frequency that matches its natural frequency it oscillates with maximum amplitude and is said to be in ‘resonance’.

Our experiments study the mechanical resonances in nanoscale, suspended structures involving graphene drums and InAs nanowire resonators. We discuss this physics by looking at the simple model of a harmonic oscillator under the action of a periodic driving force. We discuss extensions to this model by considering effects of non-linear terms in the equation of motion that leads to hysteretic behavior. We then describe the continuum mechanics models that account for the many degrees of freedom in realistic systems. We discuss the two systems studied in our work: circular membranes, and doubly clamped beams under tensile stress. We also discuss some of the mechanisms that lead to energy loss in these nanoscale resonators.
Chapter 2. Fundamentals of nanoelectromechanical systems

2.1 Dynamics of an oscillator

2.1.1 Simple harmonic oscillator

Consider a mass $m$ attached to a massless spring of spring constant $k$. For small displacements, the mass can be approximated to be moving in a harmonic (parabolic) potential. For a displacement, $z$, of the mass from equilibrium, the potential is of the form $U = \frac{1}{2}kz^2$. A restoring force, $F_{\text{res}}$, proportional to the displacement, acts on the mass such that $F_{\text{res}} = -kz$. The equation of motion of the mass can then be written as

$$m \frac{d^2z}{dt^2} + kz = 0.$$  \hfill (2.1)

The motion of the mass is then given by the solution to this differential equation as $z(t) = z_0 \cos(\omega_0 t + \phi)$. We see that the mass oscillates with an amplitude $z_0$ at frequency $\omega_0$ and a phase of motion $\phi$ arising from initial conditions. Here, $\omega_0 = 2\pi f_0 = \sqrt{k/m}$ is the natural frequency of the system. This is the standard description of a frictionless, harmonic oscillator. Next we consider the effect of friction on the motion of the system.

2.1.2 Driven, damped harmonic oscillator

Friction to motion is present in all real life systems. This friction leads to loss in energy from the systems and leads to damped motion. Here we consider a damped oscillator under the influence of a driving force, $F(t)$. The damping, in general, is considered to be an opposing force proportional to the velocity of the mass given by $f_{\text{damping}} = -m\gamma \frac{dz}{dt}$ where $\gamma$ is the damping coefficient. In NEMS, other forms of the damping term are also important.\(^\text{[6]}\). Let the driving force be of amplitude $F_0$ at frequency $\omega$ such that $F(t) = F_0 \cos(\omega t)$. The equation of motion of the system can then be written as

$$m \frac{d^2z}{dt^2} + m\gamma \frac{dz}{dt} + m\omega_0^2 z = F_0 \cos(\omega t).$$  \hfill (2.2)

The steady state solution for this linear, second order differential equation can be
2.1. Dynamics of an oscillator

written in complex variable form as

$$z(t) = \frac{F_0}{m} \frac{e^{i\omega t}}{(\omega_0^2 - \omega^2) + i (\omega \omega_0 / Q)}$$  \hspace{1cm} (2.3)

where $Q$ is the quality factor of the resonator given by $Q = \frac{\omega_0^2}{2 \gamma}$. The quality factor quantifies the damping in the system and is inversely proportional to the energy lost per cycle of oscillation. The amplitude and phase of the response is obtained from the real and imaginary parts of the above solution and can be written as

$$A(\omega) = \frac{F_0}{m} \frac{1}{\sqrt{(\omega_0^2 - \omega^2)^2 + (\omega \omega_0 / Q)^2}},$$  \hspace{1cm} (2.4)

$$\phi(\omega) = \tan^{-1} \left( \frac{\omega \omega_0}{Q (\omega_0^2 - \omega^2)} \right).$$  \hspace{1cm} (2.5)

We see that the amplitude of oscillations is maximized when the driving frequency matches the natural frequency of the oscillator $\omega = \omega_0$ (see Figure 2.1(a)). At this condition the oscillator is said to be in resonance. The amplitude reduces rapidly as the driving frequency is detuned away from the natural frequency. The sharpness of this response is quantified by the quality factor. For a resonator with large damping the quality factor is low and the response is broad (Figure 2.1(b)). We also see that the phase undergoes a shift of $180^\circ$ across the resonance (Figure 2.1(b)).

An underlying assumption in this approach is that the driving force is low and the oscillation amplitude of the resonator is small such that the potential around equilibrium can be approximated by a harmonic potential. However, in physical systems the potential energy cannot be approximated by a harmonic potential for large amplitudes. The presence of an anharmonic potential on the resonator response is discussed next.
Figure 2.1: (a) Amplitude and (b) Phase response of a driven, damped harmonic oscillator. Here the resonant frequency is $\omega_0 = 2\pi 56$ in arbitrary units. We see that the amplitude response is maximum on resonance and the phase undergoes a shift of $180^\circ$ across the resonance.
2.1. Dynamics of an oscillator

2.1.3 Non-linear effects

Duffing oscillator

Until now we have discussed the response of the oscillator to a linear restoring force (harmonic potential). However, when the resonator is driven by a large force, the amplitude of oscillations increases and higher order terms in the potential can no longer be ignored. This leads to a non-linear response of the resonator. In physical systems there can be various origins of these nonlinearities; for example it can arise from the intrinsically nonlinear form of the attractive electrostatic potential in electromechanical systems\[65, 66\]. It can also arise from geometric effects when large oscillation amplitude introduces bending induced nonlinearities in the resonator. Consider a potential of the form

\[ V(z) = \frac{1}{2}kz^2 - \frac{1}{4}\epsilon z^4. \] \hspace{1cm} (2.6)

The equation of motion given in \[2.2\] then becomes

\[ m\frac{d^2 z}{dt^2} + m\gamma \frac{dz}{dt} + m\omega_0^2 z + \epsilon z^3 = F_0 \cos(\omega t). \] \hspace{1cm} (2.7)

This equation of motion is called the Duffing equation. Here, \(\epsilon\) quantifies the non-linearity in the system\[67\].

The solution to the above equation can be obtained using secular perturbation theory\[65\, 67\] and the amplitude, \(A\), and phase, \(\phi\), can be written as

\[ A^2 = \frac{\left( \frac{F_0}{2m\omega_0} \right)^2}{\left( \frac{\omega - \omega_0}{\omega_0} - \frac{3\epsilon A^2}{8m\omega_0^2} \right)^2 + \left( \frac{\gamma}{2\omega_0} \right)^2} \] \hspace{1cm} (2.8)

and

\[ \phi = \tan^{-1}\left( \frac{\gamma/2}{(\omega_0 - \omega) - \frac{3\epsilon A^2}{8\omega_0}} \right). \] \hspace{1cm} (2.9)
Chapter 2. Fundamentals of nanoelectromechanical systems

We see that the presence of a non-zero $\epsilon$ gives the amplitude response multiple real solutions. This is shown in Figure 2.2 (a). For low drive powers we see that the response is that of a harmonic oscillator. For larger drives, the amplitude response starts leaning to the right and for sufficiently high amplitudes it becomes bistable. This implies that the amplitude of oscillations of the resonator now depends on the direction of the frequency sweep. The presence of bistability then gives rise to hysteretic behavior of the resonator for forward and backward frequency sweeps. The direction of leaning is dependent on the sign of the non-linear coefficient $\epsilon$.

The onset of non-linear response is characterized by the amplitude response having an abrupt jump with infinitesimal change in frequency such that the derivative of the driving frequency with respect to amplitude is zero. The amplitude of the critical driving force, $F_c$, that imparts a non-linear response can then be obtained by the equations $\frac{d\omega}{dz} = 0$ and $\frac{d^2\omega}{(dz)^2} = 0$ such that

$$F_c = \left(\frac{4}{3}\right)^{5/4} \frac{m^3}{3\omega_0^3} \sqrt{\epsilon}.$$  \hspace{1cm} (2.10)

Non-linear damping

Another simplification we have made in the above formulation is that of a linear damping term of the form $m\gamma \frac{dz}{dt}$. In general a resonator can have an additional non-linear damping term of the form $\eta \frac{dz^2}{dt}$, where $\eta$ is the coefficient of non-linear damping. We see that this damping term is amplitude dependent and leads to larger dissipation at larger oscillation amplitudes.

$$A^2 = \frac{\left(\frac{F_0}{2m\omega_0^2}\right)^2}{\left(\frac{\omega-\omega_0}{\omega_0} - \frac{3\epsilon A^2}{8m\omega_0^2}\right)^2 + \left(\frac{\gamma}{2\omega_0} + \frac{\eta A^2}{8m\omega_0}\right)^2}$$ \hspace{1cm} (2.11)

The amplitude response of the Duffing resonator with non-linear damping gets modified as shown in equation 2.11. In this picture the resonator can be thought to have an effective damping of $\gamma + \frac{1}{4m} \eta z_0^2$. The effect of $\eta$ is to reduce the magnitude of the resonator when driven to larger amplitudes. The microscopic origin of non-linear damping is not well understood but is known to be present in nanomechanical...
Figure 2.2: (a) Theoretical solutions to the non-linear resonator with a positive non-linear coefficient $\epsilon$. The driving force increases in panel (i) to (iv). Panel (iv) shows the hysteresis for a non-linear resonator in forward and backward frequency sweeps. The red portions of the curve is followed for forward and reverse sweeps, whereas the green (blue) portion is followed only for forward (reverse) sweep. The black portion is an unstable solution that is not traced. (b) Experimentally obtained response of a nanowire resonator with increasing driving force shows the clear jumps due to Duffing behaviour.
systems. The origin of the linear damping term is better understood and will be discussed in Section 2.4.

Until now we have discussed the basics of harmonic motion for systems with a single degree of freedom where the mass is localized in space. However, real systems involve structures that have a spatially distributed mass and have infinite degrees of freedom. For example, a taut string has many vibrational modes with distinct mode shapes. Here we require continuum mechanics models to describe the vibrations of such systems. In the following sections we discuss the continuum mechanics approach to finding the eigenmodes of mechanical systems explored in this work: circular membranes and doubly clamped beams.

2.2 Circular membrane dynamics

The graphene resonators that we study can be considered to be thin plates with a built-in uniform radial tension. The energy for bending thin graphene flakes is negligibly small compared to the energy from the built-in tension that we consider them to be membranes. A schematic of a graphene drum device is shown in Figure 2.3(a). In our devices, the graphene flakes are placed over circular holes on SiO$_2$. The good adhesion of graphene on SiO$_2$ ensures that the membrane is clamped at the edge of the hole. Here we discuss the continuum mechanics model for circular membrane resonators where the thickness is small enough to neglect bending moments.

The equation of motion for transverse vibrations, $w(r, \theta, t)$, of a circular membrane under a uniform radial force per unit length, $T_0$, can be written in polar coordinates as

$$T_0 \left( \frac{\partial^2 w}{\partial r^2} + \frac{1}{r} \frac{\partial w}{\partial r} + \frac{1}{r^2} \frac{\partial^2 w}{\partial \theta^2} \right) = \rho h \frac{\partial^2 w}{\partial t^2}$$

(2.12)

where $h$ is the thickness and $\rho$ is the mass density of the membrane. The boundary
2.2. Circular membrane dynamics

Figure 2.3: (a) Schematic of a circularly clamped membrane drum device. The dotted circle demarcates the clamped drum region. (b) A vibrating membrane of radius $R$ stretched by a uniform radial force per unit length $T_0$ acting along its edge.

conditions for the membrane are

\[
\begin{align*}
  w(R, \theta, t) &= 0 \quad (2.13) \\
  w(r, 0, t) &= w(r, 2\pi, t) \quad (2.14) \\
  \frac{\partial w}{\partial \theta} (r, 0, t) &= \frac{\partial w}{\partial \theta} (r, 2\pi, t) \quad (2.15)
\end{align*}
\]

where the first condition satisfies the clamping along the edge of the membrane (radius $R$) and the last two conditions satisfy the radial symmetry of the system.

We find the eigenmodes of equation 2.12 using a separation of variables approach where $w(r, \theta, t) = U(r)\Theta(\theta)\tau(t)$. This gives us the equations

\[
\begin{align*}
  r^2U'' + rU' + (\lambda^2r^2 - \mu^2)U &= 0 \quad (2.16) \\
  \Theta'' + \mu \Theta &= 0 \quad (2.17) \\
  \frac{\rho h}{T_0}\tau'' + \lambda^2\tau &= 0 \quad (2.18)
\end{align*}
\]

with the boundary conditions

\[
\begin{align*}
  U(R) &= 0 \quad (2.19) \\
  \Theta(\theta) &= \Theta(2\pi + \theta) \quad (2.20) \\
  \Theta'(\theta) &= \Theta'(2\pi + \theta) \quad (2.21)
\end{align*}
\]
where $\lambda$ and $\mu$ are constants.

The boundary conditions immediately give the form of $\Theta(\theta)$ to be

$$\Theta_\mu(\theta) = A_\mu \sin(\mu \theta) + B_\mu \cos(\mu \theta)$$  \hfill (2.22)

where $\mu = 0, 1, \ldots$ and

$$\tau(t) = C_\lambda \sin \left( \sqrt{\frac{T_0}{\rho h}} \lambda t \right) + D_\lambda \cos \left( \sqrt{\frac{T_0}{\rho h}} \lambda t \right).$$  \hfill (2.23)

We see that the system oscillates at a frequency proportional to $\lambda \sqrt{\frac{T_0}{\rho h}}$.

The ordinary differential equation for $U(r)$ is the Bessel's equation of order $\mu$, which has the solution

$$U(r) = c_{1\mu} J_\mu(\lambda r) + c_{2\mu} Y_\mu(\lambda r)$$  \hfill (2.24)

where $J_\mu(\lambda r)$ and $Y_\mu(\lambda r)$ are the Bessel's functions of the first and second kind. Since $Y_\mu(0)$ is infinite, for physical solutions we get $c_{2\mu} = 0$. Using the boundary condition $U(R) = 0$, we get $J_\mu(\lambda R) = 0$ which implies that $\lambda_{\mu,\nu} R = \alpha_{\mu,\nu}$ where $\alpha_{\mu,\nu}$ are the zeroes of the Bessel's function of the first kind with $\nu = 1, 2, \ldots$ for non-trivial solutions.

The general solution for the transverse vibrations of a circular membrane under an axial tension can be thus written as

$$w(r, \theta, t) = AJ_\mu(\lambda_{\mu,\nu} r) \sin(\mu \theta + \theta_1) \cos(\omega_{\mu,\nu} t + \phi)$$  \hfill (2.25)

where $A$, $\theta_1$, and $\phi$ are arbitrary amplitude and phase constants.

Equation 2.25 describes the eigenmodes of a circular membrane vibrating at frequency $\omega_{\mu,\nu}$ given by

$$\omega_{\mu,\nu} = 2\pi f_{\mu,\nu} = \frac{\alpha_{\mu,\nu}}{R} \sqrt{\frac{T_0}{\rho h}}$$  \hfill (2.26)

Figure 2.4 shows the first few vibrational modes of a membrane. We note that the form of the solution given in equations 2.25 and 2.26 gives rise to degenerate frequencies for some of the modes.
Figure 2.4: The first four vibrational modes of a circular membrane. The numbers below each mode denote the zero of the Bessel function ($\alpha_{\mu,\nu}$) corresponding to that mode. Here the second and third modes are doubly degenerate.
2.3 Doubly clamped beam dynamics

In our work we also study suspended InAs nanowire resonators in doubly clamped geometry. These devices can be considered to be doubly clamped beams under tension. Here we discuss the mechanics of such a system where the rigidity also plays a role. First we discuss the case of a beam without residual tension. Consider a homogeneous beam of length $L$ and cross sectional area $A$. The transverse deflections of the beam as a function of the position along its length is denoted by the time dependent function $w(x,t)$. The equation of motion governing the deflections of the beam is then given by the partial differential equation

$$EI \frac{\partial^4 w(x,t)}{\partial x^4} + \rho A \frac{\partial^2 w(x,t)}{\partial t^2} = F(x,t) \quad (2.27)$$

where $E$, $I$, and $\rho$ are the Young’s modulus, second moment of inertia, and the mass density of the beam. $F(x,t)$ denotes a driving force that may be applied on the beam. The equation of motion can be solved using a separation of variables approach to find the eigenvalues (natural frequencies) and the eigenfunctions (mode shapes). We approach the problem by assuming the form of $w(x,t)$ to be

$$w(x,t) = \phi(x)e^{i\omega t} \quad (2.28)$$

where the function $\phi(x)$ represents the mode shape of the beam oscillating at a frequency $\omega$. Using equation [2.27] we obtain the equation for the mode shape as

$$\frac{\partial^4 \phi(x)}{\partial x^4} + k^4 \phi(x) = 0 \quad (2.29)$$
2.3. Doubly clamped beam dynamics

where
\[ k^4 = \rho A \omega^2 / EI. \] (2.30)

The general solution for equation 2.29 will be of the form
\[ \phi(x) = a_1 \cos(kx) + a_2 \sin(kx) + a_3 \cosh(kx) + a_4 \sinh(kx) \] (2.31)

where the constants \( a_i \) are found from the boundary conditions. For a doubly clamped beam, the boundary conditions are given by,
\[ w(0, t) = w(L, t) = 0 \quad \text{and} \quad w(0, t)'(0, t) = w'(L, t) = 0. \] (2.32)

By using the boundary conditions and the form of \( \phi(x) \) we arrive at the characteristic equation given by
\[ \cos(kL) \cosh(kL) = 1. \] (2.34)

Equation 2.34 can be solved numerically and is satisfied for a discrete set of values of \( k \) and we find the eigenvalues from the roots of this equation by putting \( \beta_n = k_n L \). The values of \( \beta_n \) that gives non-trivial solutions are \( \approx 4.730 \) for the fundamental mode and 7.853 for the first harmonic. The eigenvalues of the unstressed beam are thus given by
\[ \omega_n = \frac{\beta_n^2}{L^2} \sqrt{\frac{EI}{\rho A}}. \] (2.35)

In our experiments we see that the resonant frequencies of nanowire resonators are generally higher than the value predicted by equation 2.35. This is due to the presence of residual stress in the nanowire devices. These stresses can arise from the fabrication processes as well as relative expansion of the devices when cooled down to low temperatures. To see the effect of a built in tension on the eigenfrequencies we now consider a beam under tension. Let the beam be subjected to an axial tension, \( T \). The equation of motion given by equation 2.27 now gets modified to
\[ EI \frac{\partial^4 w(x, t)}{\partial x^4} - T \frac{\partial^2 w(x, t)}{\partial x^2} + \rho A \frac{\partial^2 w(x, t)}{\partial t^2} = F(x, t) \] (2.36)
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The characteristic equation now takes the form

\[ \cos(\lambda L) \cosh(\mu L) - \frac{1}{2} \left( \frac{\mu}{\lambda} - \frac{\lambda}{\mu} \right) \sinh(\mu L) \sin(\lambda L) = 1 \] (2.37)

has to be satisfied. Here,

\[ \lambda = k \sqrt{\sqrt{a^2 + 1} - a} \] (2.38)
\[ \mu = k \sqrt{\sqrt{a^2 + 1} + a} \] (2.39)

where \( a = T/2EIk^2 \) with \( k^4 = \rho A \omega^2/EI \).

The mode shape function can be written as

\[ \phi(x) = \cos(\lambda x) - \cosh(\mu x) + \left( \frac{\cos(\lambda L) - \cosh(\mu L)}{\lambda/\mu \sinh(\mu L) - \sin(\lambda L)} \right) \left( \sin(\lambda x) - \frac{\lambda}{\mu} \sinh(\mu x) \right) . \] (2.40)

Once again the characteristic equations is satisfied for multiple values of \( k_n \). The transcendental nature of the characteristic equation, however, prohibits a closed form solution for the eigenvalues. Approximate expressions for the natural frequencies can be obtained by energy considerations and is of the form

\[ \omega_n(T) = \omega_n \sqrt{1 + \gamma_n \frac{T L^2}{12 EI}} \] (2.41)

where \( \omega_n \) are the natural frequencies of the unstressed beam given in equation 2.35. The mode dependent coefficients \( \gamma_n \) are given by

\[ \gamma_n = \frac{12}{L^2} \left( \int_0^L \left( \frac{d\phi_n}{dx} \right)^2 / \int_0^L \left( \frac{d^2\phi_n}{dx^2} \right)^2 \right) \] (2.42)

where \( \phi_n(x) \) are the mode shape functions. This gives the values of \( \gamma_n \) to be \( \approx 0.295 \) for the fundamental mode, and 0.145 for the first harmonic.
2.4 Loss mechanisms

In Section 2.1 we described the response of oscillators with dissipation. We now take a look at the mechanisms of energy loss in mechanical resonators. The energy loss per cycle, or dissipation, in a resonator is characterized by the quality factor, $Q$, where

$$Q^{-1} = \frac{1}{2\pi} \frac{\text{Energy dissipated per cycle}}{\text{Total energy}}.$$  \hspace{1cm} (2.43)

Previously we had discussed the presence of linear and non-linear damping terms in resonators. The microscopic origin of non-linear damping in mechanical resonators is not well understood\[68\]. A better understanding of the linear damping in mechanical systems exists. This form of dissipation can have various origins and can be of two forms, extrinsic and intrinsic. Extrinsic dissipation arises from the interactions of a resonator to its environment and are considered to be engineering constraints, whereas intrinsic dissipation occurs due to various interactions within the resonator\[68\]. Here we discuss some of the common channels of energy loss in a resonator.
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2.4.1 Extrinsic losses

The surrounding environment of a mechanical resonator can play a major role in deciding the dissipation in the system. Here we discuss some common mechanisms of dissipation through interaction with the environment. Some of the common extrinsic loss mechanisms involve viscous damping, and clamping losses.

Air damping

Air damping is a commonly encountered mechanism for energy loss in mechanical systems and it occurs due to friction with the gaseous environment surrounding the resonator. The gas pressure range can be divided into three categories depending on the dominant damping mechanism as intrinsic, molecular, and viscous regimes. In the intrinsic regime the gas pressure is too low to affect the dynamics of the oscillator and air damping is negligible. In the viscous regime the mean free path of the gas molecules is smaller than the size of the gas molecules and the environment can be considered as a continuous viscous fluid. This corresponds to pressures that are greater than or equal to atmospheric pressures.

In the intermediate pressure regime, the molecular regime, the mean free path of the gas molecules are much larger than the size of the molecules. Here gas damping can be understood as the energy lost by the resonator to independent collision events with the surrounding air molecules. The energy loss in this regime is directly related to the pressure of the gas. For an oscillating beam of mass density $\rho$ and resonant frequency $\omega$ the loss due to air damping in the molecular regime can be written as

$$Q^{-1}_{gas} = \frac{Pw}{\rho A \omega_0 v_m}$$  \hspace{1cm} (2.44)

where $P$ is the gas pressure, $A$ and $w$ are the cross sectional area and width of the beam respectively, and $v_m$ is the rms velocity of the gas molecules. We perform our experiments on nanomechanical resonators in the intrinsic regime. The resonators are measured in evacuated chambers with pressure less than $10^{-3}$ mbar such that the effect of air damping is negligibly small.
2.4. Loss mechanisms

Clamping loss

Clamping losses arise due to the strain present at the support structures of the resonator. The resonator can excite acoustic waves into the support structure at the clamping points thus losing energy\cite{79}. Clamping losses are known to be a dominant source of dissipation in flexural modes of various nanoscale resonators as the strain is naturally maximized at the nodes at the supports\cite{68}. This effect is considered to be a temperature independent mechanism where the dimensions of the resonator decide the loss at the clamps. The clamping loss for the fundamental mode of a cantilever of width $w$, thickness $t$, and length $L$ attached to a base of finite thickness was calculated by Photiadis and Judge\cite{80} and estimated to be

$$Q_{clamp}^{-1} \approx 0.31 \frac{w}{L} \left( \frac{t}{L} \right)^4.$$ \hspace{1cm} (2.45)

It is difficult to estimate the exact nature of clamping losses in NEMS. The wavelength of the acoustic wave excited in the substrate can be larger than the substrate thickness and hence even the stage that holds the device becomes part of the substrate\cite{78, 81}. As the damping is dependent on the strain field the built-in stress in the resonator also plays a role in deciding the dissipation at the clamps. It was shown by Villanueva et al. that high stress silicon nitride beams have a dissipation limited by clamping losses\cite{82}.

The dissipation due to the strain field at the supports can also lead to mode dependent dissipation. Different vibrational modes, or harmonics, of a resonator have different strain fields at the supports. The strength of the elastic wave radiated into the supports thus depends strongly on the shape of the vibrational mode\cite{83, 84, 85}.

2.4.2 Intrinsic losses

Here we discuss some of the intrinsic loss mechanisms in mechanical systems. These processes lead to dissipation in the system through interactions within the material. Some of the common intrinsic loss mechanisms involve thermoelastic damping, mechanical defects, and surface losses\cite{68}.
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Thermoelastic damping

The thermal expansion coefficient of a material couples the temperature and strain fields in the material. Strain fields are generated inside a mechanical oscillator as it undergoes vibrations. The presence of a non-zero thermal expansion coefficient then gives rise to temperature differences in a mechanical oscillator due to the strain generated by the mechanical motion. The temperature gradient inside the material thus causes drift of thermal phonons. The diffusive motion of these phonons leads to a dissipation in the system that depends on the thermal time constant of the material, $\tau_{th}$, the expansion coefficient, $\alpha$, and temperature, $T$, of the resonator[66, 86]. The dissipation due to thermoelastic damping is given by

$$Q_{TED}^{-1} = \frac{\alpha^2 T E}{\rho C_p} \frac{\omega \tau_{th}}{1 + (\omega \tau_{th})^2}$$

(2.46)

where $C_p$ is the heat capacity per unit volume, $E$ is the Young’s modulus, and $\omega$ the vibration frequency. The contribution of thermoelastic damping in semiconductor at low temperatures is small and of the order of $1/Q \sim 10^{-6} - 10^{-9}$

Defects

Defects in a solid can act as sources of dissipation in mechanical resonators and are generally studied in the form on two level systems (TLS). In a crystalline solid this could be two states that are available for an ionic defect. The presence of such defect sites is well known in semiconductors like Si and GaAs[61]. Consider two states that have a finite energy difference, $\delta E$. A transition of the defect from the upper to the lower state is accompanied by emission of energy $\delta E$[66]. The relative transitions between the two states is decided by thermodynamics and hence the system also needs energy to populate the upper state. The transition to the upper state can occur by absorbing energy $\delta E$ which might be provided by phonons of frequency $\omega = \delta E/\hbar$. For a mechanical resonator this energy can be provided by the strain field and leads to dissipation in the resonator. Here the rate of energy absorption from the strain field will be proportional to the Boltzmann factor, $e^{-\delta E/k_B T}$ and hence will show a
temperature dependent quality factor. It has been shown by Mohanty et al. that

\[ Q_{\text{TLS}}^{-1} \propto T^{-2}, \quad \delta E \ll k_B T \quad (2.47) \]

\[ Q_{\text{TLS}}^{-1} \propto T^{-1} e^{-2\delta E / k_B T}, \quad \delta E \gg k_B T \quad (2.48) \]

where \( \gamma_0 \) is a coupling constant that relates the TLS and strain field[61].

### Surface losses

Dissipation in mechanical resonators is known to increase with decreasing size as observed from many experiments on nanomechanical systems[2, 87, 88]. Metastable surface defects can arise due to dangling or broken bonds on the surface. The increase in surface area over the volume makes this loss mechanism relevant in nanomechanical systems[61, 82]. Previous experiments have shown improvement in quality factor by using surface treatment and passivation steps[89, 90]. Metallized bilayer structures have also shown an increased dissipation with thickness of deposited metal[85, 91].

Thickness dependent studies of silicon nitride resonators have shown the quality factor to be scaling linearly with the thickness. This is indicative of surface losses being a dominant loss mechanism in these systems[82]. Here the surface roughness (\( \sim 1 \) nm) of the membrane can become a significant fraction of the thickness in thin membranes and could also be an origin of surface loss.

Here we have discussed various loss mechanisms, intrinsic and extrinsic, that contribute to dissipation in a mechanical system. As the total energy dissipated from the resonator is the sum of losses from all sources of dissipation we get the quality factor of the resonator to be

\[ \frac{1}{Q} = \sum \frac{1}{Q_i} \quad (2.49) \]

where \( Q_i \) is the dissipation from the \( i \)-th source.

Some of the common loss mechanisms known to occur in graphene and graphene-like resonators at room temperature are air damping, clamping losses and thermoeelastic losses[92, 93]. Air damping is shown to drastically decrease the quality factor in atomically thin MoS\(_2\) resonators for pressures higher than \( \sim 60 \) mbar. For graphene resonators at room temperature, theory has shown that clamping losses
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contribute $\sim 10^{-6} - 10^{-5}$ to $Q^{-1}$ where as thermoelastic damping contributes $\sim 10^{-7}$. Additionally, Ohmic losses arising from the presence of electronic charges in the graphene-metallic gate electrode system have been argued to be dominant at room temperature with contributions to $Q^{-1}$ as high as $10^{-2}$. At low temperatures, however, clamping losses are considered to be the dominant loss mechanism in graphene resonators.

2.5 Summary

This chapter discussed the basic theory behind resonance phenomena in NEMS. The linear and non-linear aspects of harmonic motion were discussed for simple systems with a single degree of freedom. Further, realistic systems with many degrees of freedom were considered. The continuum mechanics of vibrating membranes and doubly clamped beams under tensile stress was discussed. The vibrational frequencies and shapes for eigenmodes of such systems were presented. We also discussed some of the common loss mechanisms in NEMS.
Chapter 3

Fabrication of nanoscale resonators

In this chapter we will describe the process for fabricating the nanoscale resonators that we use in the later part of this thesis. First, the process for patterning electrodes to contact nanoscale devices using electron beam lithography will be discussed. Then the procedure for fabricating graphene drum resonators will be discussed followed by the fabrication of doubly clamped InAs nanowire resonators. The graphene drums were fabricated in collaboration with Raj Patel and Abhinandan Borah. The nanowire resonators on sapphire were fabricated with the help of Dr. TS Abhilash. Some of the challenges relating to the fabrication processes are also discussed. Additional details on the fabrication process are provided in Appendices A, B, and C of this thesis.

3.1 Nanoscale patterning using electron beam lithography

One of the main tools that is used for fabrication of nanoscale devices is electron beam (e-beam) lithography. E-beam lithography allows us to pattern structures as small as 10 nm, which is useful in making electrical contacts to nanoscale devices. This involves using a SEM that has the ability to precisely scan a narrow beam of electrons in a desired pattern. The substrate that contains the device material is coated with polymers (resists) that are sensitive to high energy electron beams.
EL-9 (copolymer of methyl methacrylate (MMA) and methacrylic acid (MAA) in ethyl lactate (EL) solvent) and poly methyl methacrylate (PMMA) are used as e-beam resists (Figure 3.1(a)). EL-9, a low molecular weight resist, is coated first followed by higher molecular weight polymers (PMMA-495 then PMMA-950). An optical microscope is used to locate the position of the material on the substrate. Then computer aided design (CAD) (Figure 3.1(c)) allows us to create patterns of electrodes for devices. This design is then transferred into the SEM which rasters the beam along the desired pattern. The chip is then placed in a developing solution containing methyl isobutyl ketone (MIBK) diluted in isopropyl alcohol (IPA). The exposed regions of the resist are dissolved in the developer leaving trenches in the desired regions (Figure 3.1(b)). For the case of the positive e-beam resist, used in this work, the role of the e-beam is to break up the resist polymers into smaller chains that are easily dissolved in the developing solution. The differential solubility of these resists in the developer give rise to an undercut shape which prevents the deposition of metal deposition of along the walls of the resists and helps in clean metal liftoff (final step of fabrication to remove excess metal). The chip is then placed in a metal deposition system to cover the entire chip with metal (the height of the metal deposition being smaller than the total resist height). Finally, the chip placed in acetone to liftoff the unwanted metal and resist. This completes the device with metal contacts patterned to our need (Figure 3.1(d)).

Commonly used substrates for device fabrication are highly doped Si with a thin SiO\textsubscript{2} coating. E-beam lithography on such substrates is simple and follows the steps outlined above. However, we are interested in insulating substrates in our experiments to improve signal collection in high frequency electrical applications.\cite{95} The insulating substrate presents a challenge in e-beam lithography as the incident electron beam deposits charges on the substrate. These charges deflect the electron beam and produces a low fidelity reproduction of the desired pattern. Hence, e-beam lithography on insulating substrates is preceded by the deposition of a thin conducting layer (usually evaporated metal) on the substrate that acts as a grounding plane for the incident charges. This layer is etched from the exposed regions prior to metal deposition. The remaining metal layer is also etched after liftoff.
3.1. Nanoscale patterning using electron beam lithography

Figure 3.1: Basic process flow in e-beam lithography. (a)-(b) A substrate is coated with layers of e-beam resists and desired patterns are scanned on the chip using an electron beam. (c) The exposed layers are developed using organic solvents to reveal trenches. The developed region is wider at the bottom due to the differential solubility of different resists in the developer solution. (d) Metal is deposited all over the chip. (d) Lift off in acetone results in metal being left behind only in the desired regions thus forming the pattern.
3.2 Fabrication of graphene drum resonators

Here we describe the process for fabricating electromechanical resonators of graphene drums. The objective was to fabricate sealed graphene drums on an insulating substrate with a local gate underneath the drum for actuating the mechanical motion. The mechanically exfoliated graphene flakes were also incorporated into the device in the final stage of fabrication to ensure that the graphene remains free of chemical contamination.

Fabrication of the graphene drum resonators is quite complex and we discuss the procedure in a few stages. The stages involved are: patterning a local gate electrode on sapphire, deposition of SiO$_2$ and etching a local hole, patterning source and drain electrodes on the SiO$_2$ near the hole, and finally the transfer of a graphene flake to complete the device. Additional details of each process is provided in Appendix C.

We start by using highly resistive c-plane sapphire substrates with resistivity $> 10^{14}$ $\Omega$m. The role of these insulating substrates is to eliminate parasitic capacitances which is detailed in Chapter 4. Figure 3.2 shows schematic of the fabrication steps for the drum resonators. The first part of the fabrication involves patterning the local gate electrode on the sapphire substrate. In order to do e-beam lithography, a thin chromium layer (20 nm) is deposited on the sapphire substrate using thermal evaporation. This ensures that charging effects are not present during the lithography. Here, we chose Cr as the conducting layer because the chemical etchant (Sigma Aldrich 651826) used to etch the metal layer during other parts of the lithography does not affect the substrate, dielectric, and electrode metals that are used during different stages of fabrication. After the Cr layer deposition, resist layers are spin coated and e-beam lithography procedure is followed (as described in Section 3.1) to write a gate electrode pattern in the resists. The Cr layer is then etched from the developed regions of the resists followed by evaporation of titanium/platinum (15 nm/10 nm) to form the gate electrode. The combination of Ti/Pt (as opposed to Cr/Au) is used for the gate electrode as it is stable under reactive ion etching (RIE, used at a later stage in the fabrication process) and does not lead to back sputtering. Lift-off of the excess metal in acetone, followed by removal of the conducting layer of Cr completes the first stage of fabrication.
3.2. Fabrication of graphene drum resonators

Figure 3.2: Steps used for fabricating graphene drum resonators with a local gate on a sapphire substrate (a) A local gate is fabricated on sapphire using e-beam lithography for patterning and metal deposition by evaporation of Ti/Pt. (b) Deposition of SiO$_2$ and e-beam lithography to pattern hole over the local gate electrode. (c) RIE for etching hole in SiO$_2$ after removing Cr in the developed regions. (d) Spin coat resists and evaporate conducting layer of Cr for e-beam lithography step. (e) E-beam lithography followed by etching Cr, developing resists, and deposition of Cr/Au to form source-drain electrodes. (f) Liftoff using acetone and dry transfer of few layer graphene (FLG) flake over the hole and electrodes.
Chapter 3. Fabrication of nanoscale resonators

The next stage involves deposition of SiO\textsubscript{2} and forming a local hole above the gate electrode. The hole in the SiO\textsubscript{2} will act as the hollow for the graphene drum. We use plasma enhanced chemical vapor deposition (PECVD) at 150°C to deposit 300 nm of SiO\textsubscript{2} over the entire chip. Now the SiO\textsubscript{2} has to be etched locally above the gate electrode. An additional region near the outer pad of the gate electrode also has to be etched. This will allow us to make electrical contact to the gate electrode by wirebonding. To make the hole, Cr is thermally evaporated, resist layers are spin-coated and e-beam lithography is done to pattern a circular region above the gate electrode that has to be etched. Then the resist layers are developed and Cr is etched from the developed regions. The micron scale size of the circular region poses the issue of non-uniform etching when the chip is placed in the Cr etchant. This is overcome by using a magnetic stirrer to agitate the Cr etchant when the chip is placed for etching. The Cr is etched by this process over 60 - 90 s and then rinsed in DI water, and subsequently in IPA. The resist layers are then removed using acetone to prevent hard baking during plasma etching in the RIE system. RIE is used to dry etch SiO\textsubscript{2} in the desired region (figure 3.2(c)). The un-etched Cr acts as a mask and protects the surrounding SiO\textsubscript{2} from getting etched. The remaining Cr is etched away to complete the second stage of the fabrication.

The next stage involves the fabrication of source and drain electrodes on the SiO\textsubscript{2} near the hole region. Resist layers are spin coated and 20 nm Cr layer is thermally evaporated on top of the resist stack. E-beam lithography is done to pattern the electrodes and the resist layers are developed after etching away the top Cr layer. The source/drain electrodes are then formed by thermally evaporating chromium (10 nm) and gold (50 nm). Liftoff in acetone completes the fabrication steps and the chip is now ready for the final stage which involves transferring a graphene flake.

We use the scotch tape method\cite{20} to mechanically exfoliate graphene from a bulk piece onto a polydimethylsiloxane (PDMS) film kept on a glass slide. PDMS is a viscoelastic material that will act as a stamp to transfer the graphene flake to the desired location.\cite{96} This technique is an all-dry technique, involving no liquids, and thus facilities the fabrication of suspended devices as the surface tension of liquids can lead to fracturing of nanoscale suspended structures.\cite{96} Thin flakes of graphene are located using an optical microscope. The low visibility of thinner flakes limits the minimum thickness of usable flakes to few nanometers. We use a modified mask...
3.2. Fabrication of graphene drum resonators

Figure 3.3: (a) SEM image of local gate fabricated on sapphire substrate. The PECVD SiO_2 and hole etched in SiO_2 to form the drum cavity is also seen. Optical image of device before (b) and after (c) graphene flake transfer. (d) Zoomed in image of (c). The flake is outlined by a dashed line.

Figure 3.4: (a) A 3D schematic of the final device. The electrode below the two holes represents the local back-gate. Graphene flake is transferred over the smaller hole (marked drum) and the source-drain electrodes. The bigger hole allows wirebonding of the gate electrode. (b) SEM image of a drum resonator just after the transfer step. (The SEM images were taken following the precautionary steps described in the Section 3.2.1) (c) SEM image of a bulged graphene membrane.
aligner system to carry out the step of aligning and transferring the graphene flake on to the device chip. The device chip is kept on a lower stage and the glass slide holding the PDMS is placed on a movable stage above (with the flakes facing the bottom chip). As the PDMS is transparent, we can see through the structure to align a thin flake with the bottom chip. Once aligned, the bottom stage is raised to form contact with the PDMS. Once in contact, the chip is separated slowly from the PDMS which leaves behind the graphene flake on the device. The alignment is done in such a way that the flake touches the source/drain electrodes covering the SiO$_2$ hole. Figure 3.3 shows optical and SEM images of the chip through various steps of the fabrication.

One of the challenges during the stages of device fabrication involved depositing an SiO$_2$ layer that was stable against the metallic etchant used in subsequent steps of fabrication. Previous attempts at fabrication with aluminium as the conducting layer for e-beam lithography were not successful as the etchant used for removing aluminium (1% TMAH) etched away the SiO$_2$ layer. A second challenge to the fabrication was having the graphene layer suspended over the drum without collapsing onto the gate electrode. This was taken into consideration when choosing the diameter of the drums. We limited the drum diameter to less than 4 $\mu$m for ease of transferring the graphene flakes. With the final fabrication recipe we were able to achieve a yield of 67% successful devices.

Figure 3.4 shows a schematic along with SEM images of the final device. We see from the SEM image (Figure 3.4(c)) that the graphene flake has bulged. Graphene is known to be impermeable to many gases\[32\] and traps air underneath the drum during the transfer process. This trapped pocket of air takes a long time to escape. Hence, when the chip is placed in the SEM, the vacuum in the SEM chamber causes a pressure difference across the membrane and causes bulging. The strong adhesion of graphene to the SiO$_2$, and its mechanical strength ensures that the drum is not ruptured by the pressure difference.\[72\] However, we observe that SEM imaging can lead to rupture in these drums under certain circumstances which is discussed next.
3.3. Fabrication of doubly clamped InAs nanowire resonators

Here we discuss the fabrication of doubly clamped InAs nanowire resonators. InAs nanowires grown in Prof. Arnab Bhattacharya’s lab in TIFR are used for this work. The nanowires are grown on a GaAs substrate using metallorganic chemical vapor deposition (MOCVD) process. The nanowires are approximately 100 nm in diameter.
Chapter 3. Fabrication of nanoscale resonators

and 10-20 $\mu$m in length after the growth process and are seen to be densely packed on the growth substrate (see Figure 3.6(a)). The nanowires also have few nm thick native oxide layer on its surface as confirmed from transmission electron microscope (TEM) imaging shown in Figure 3.6(b). We will discuss how the oxide layer has to be removed to make electrical contacts to the nanowire. Additional details of the fabrication steps are provided in Appendix A and B.

A small piece of the growth substrate is kept in a vial with few mL of high purity IPA. Ultrasonication causes the nanowires to break off from the substrate and disperse in the liquid. To fabricate devices we use either sapphire substrates or intrinsic silicon with nitride coating. Either of these substrates facilitate electrical rf measurements at low temperatures as the charge carriers are frozen and parasitic capacitance is reduced. We evaporate 20 nm of aluminium on the sapphire substrates prior to use.

![Figure 3.6: (a) SEM and (b) TEM images of the as grown InAs nanowires. The native oxide layer on the nanowire is clearly visible in the TEM image.](image)

EL-9 is spin coated and baked on the substrate to achieve a 200 nm thick resist layer. This layer will act as the spacer that eventually leads to the nanowire resonator being suspended. Few $\mu$L of the nanowire dispersion is then taken using a micropipette and dropped on the resist coated substrate. After blow drying with $N_2$ we observe many nanowires on the substrate under an optical microscope. Subsequently EL-9, PMMA-495, and PMMA-950 are spin-coated in that order to sandwich the nanowires between resist layers (Figure 3.7(a)).

As the nanowires are randomly positioned and oriented over the entire substrate we use a coordinate system (referred to as markers), patterned using e-beam lithography, to identify the precise location of the nanowires (see Figure 3.7(b)). CAD now allows us to design the electrodes precisely with respect to the marker positions. The
3.3. Fabrication of doubly clamped InAs nanowire resonators

Figure 3.7: (a) The nanowires are sandwiched between layers of e-beam resists. (b) Optical image of markers that were lithographically patterned on the chip. A nanowire between the marker numbers is marked by the arrow. The position of nanowire relative to the marker allows us to precisely design electrodes using CAD. (c) The CAD drawing is loaded into the e-beam lithography system which transfers the patterns onto the resists. The chip is then developed in MIBK:IPA. Metal deposition followed by liftoff completes the fabrication. (d) A tilted SEM image of the completed device.

CAD drawing is transferred onto the resists by e-beam lithography. The chip is then developed in a 1:3 solution of MIBK and IPA. For devices on sapphire this step is followed by an aluminium etch step to remove the metal in the developed regions.

After development, the chip is loaded into a sputtering chamber for metal deposition. Sputtering ensures isotropic metal coverage on the chip depositing metal in and around the nanowire. This leads to the nanowire being clamped by the metal where the metal acts the dual role of contact electrode as well as mechanical supports. Prior to metal deposition we have to remove the native oxide layer on the nanowires. This is done using an in-situ plasma etching system in the sputtering chamber with-
out breaking vacuum. Plasma etching is done with rf power of 25 W for 2 minutes for devices on Si substrates and 45 seconds for devices on sapphire substrates. The plasma treatment also ensures that resist residues are removed from the nanowire surface and helps in forming good Ohmic contacts to the nanowire. We then sputter Cr/Au (50/350 nm) to form electrical contacts as well as the mechanical anchors. The thick metal layer is needed to anchor the ∼100 nm thick nanowires suspended above the substrate. Finally, we carry out liftoff in acetone to remove the excess metal. On sapphire substrates we conclude the fabrication process by removal of the conducting metallic layer by placing the chip in aluminium etchant. This completes the fabrication of doubly clamped InAs nanowire resonators.

3.3.1 Challenges in fabrication on insulating substrates

For devices fabricated on intrinsic silicon we do not have to follow any particular precautions. However, for the devices fabricated on sapphire the insulating nature of the substrate presents some challenges in fabrication. We overcome the challenges in e-beam lithography by using a conducting layer of Al as previously discussed. The plasma etching process prior to metal deposition also requires precaution. The normal duration of plasma etching (2 minutes) used for devices on non-insulating substrates tends to over etch nanowires on insulating substrates. Figure 3.8 shows the effect of a 2 minute etch time for nanowire devices fabricated on sapphire substrates. We see that the nanowire is thinned or completely etched at the contact regions. This is overcome by reducing the plasma etch duration to about 45 seconds in devices with Al conducting layer at the bottom.

3.4 Summary

To summarize, in this chapter we have discussed the fabrication processes for the different types of devices used in our experiments. Basic process flow for patterning nanoscale contacts using electron beam lithography was outlined. We then described in detail the steps for fabricating graphene resonators in a sealed drum geometry. We also provided details on the fabrication of InAs nanowire resonators in doubly
3.4. Summary

(a) (b)

Figure 3.8: SEM images of nanowire devices on sapphire where the nanowire has fallen on the substrate (a) and has thinned near the contact regions (b). Scale bar is 2 µm in (a) and 1 µm in (b).

clammed geometry. We also discussed the challenges in fabrication of resonator devices on insulating substrates.
Chapter 4

Measuring electromechanical resonances in NEMS

In this chapter, we first describe some of the common non-electrical techniques used in actuating and detecting the mechanical motion of nanoscale resonators. Next, a detailed look at the measurement scheme we employ in our experiments is discussed along with typical measurements and procedure for obtaining the resonance parameters.

4.1 Overview of measurement schemes

Many techniques have been used to actuate and detect the mechanical motion of NEMS including electrical, piezoelectric, optical and magnetomotive schemes[2]. As these devices are very small the measurement of small oscillations associated with resonance can be challenging. Here we review some of the common non-capacitive schemes used in measuring mechanical resonance of NEMS. In our work we use electrical (capacitive) actuation and detection of mechanical motion which is discussed in the next section (Section 4.2).
4.1. Overview of measurement schemes

4.1.1 Actuation schemes for NEMS

Piezoelectric actuation

One of the easier ways to drive a NEMS system is to use a piezoelectric material as a vibrating stage\[3\]. The NEMS device is placed on a piezoelectric disk. An oscillating voltage applied to one of its faces causes periodic deformations of the disk. These deformations are transferred to the NEMS device which results in oscillations and resonance when the actuating frequency matches the natural frequency of the nanomechanical system. The availability and ease of use of these piezoelectric disks makes this actuation scheme popular. However, the actuation efficiency becomes heavily attenuated for higher mechanical frequencies and limits the use of this technique in high frequency NEMS applications (>50 MHz). A more efficient way to use piezoelectric actuation is to incorporate a piezoelectric element (like GaAs, PZT etc) on the resonator by microfabrication techniques\[98\]. The strain field which is set up on the piezoelectric material directly actuates the suspended structure\[99, 100\]. This method has been shown to be efficient in actuating structures with resonant frequencies up to hundreds of megahertz\[101\].

Optical actuation

In the optical actuation technique\[102\], a laser with modulated intensity is focussed on the resonator. A laser beam focussed on the resonator can cause heating which gives rise to a thermal stress in the structure. A modulated laser intensity, therefore, leads to a time varying stress on the suspended structure causing the device to vibrate\[103, 104\]. A disadvantage of this technique is the generation of heat and temperature increase in the structure\[105\]. Diffraction limited size limits the use of this technique to smaller structures.

Magnetomotive actuation

The magnetomotive actuation technique\[106, 107\] makes use of the Lorentz force on a current carrying conductor and is generally used for beam like structures. In this
scheme, the device is placed in a static magnetic field perpendicular to the plane of vibration. An AC current is then passed through the device that generates an oscillatory Lorentz force on the beam that causes vibrations. In case of semiconductors, a metallic strip is often deposited on the beam to facilitate the actuation.

**Electrothermal actuation**

The electrothermal actuation technique, similar to the optical technique, relies on a time varying thermal strain acting on the resonator to set it into oscillations. A time varying current is passed through a metallic electrode attached to the surface of the resonator. The resultant Joule heating and thermal expansion of the resonator causes a modulating strain that actuates the resonator\[108\]. This technique can be useful for actuating layered resonator structures where the difference in thermal expansion coefficients causes thermal strain. This technique can also be extended to tune the resonant frequency of resonators by adding a dc current that exerts a constant thermal strain on the system\[60\]. However, use of this technique is limited by the inherent heating of the structure.

### 4.1.2 Detection schemes for NEMS

**Piezoresistive detection**

This scheme requires the inclusion of a piezoresistive material on to the vibrating structure. As the structure vibrates a strain field is generated across the device which gives rise to a time varying resistance change in the piezoresistive material\[109, 110\]. This change in resistance is maximized at resonance and measured to detect the mechanical motion.

**Magnetomotive detection**

This scheme is usually used alongside the magnetomotive actuation scheme. In this technique the vibrating structure is made part of a conducting loop such that a time
4.2. Overview of capacitive actuation and detection

varying electromotive force (EMF) is generated as the structure vibrates. The resultant electromotive force that is generated is subsequently amplified and measured.

Detection using optical and microwave cavities

Conventional use of optical means to detect mechanical motion rely on detecting the intensity variations caused by mechanical oscillations [111]. A probe laser is focussed on the oscillating structure and the reflected beams from the suspended structure and substrate are collected by a photodiode. Interference between the reflected beams strongly depends on the position of the oscillator. Mechanical resonances are then seen as peaks in the output spectrum of the photodiode.

There has been a focussed effort to incorporate mechanical resonators into various types of optical and microwave cavities, where the mechanical element forms part of the cavity [41, 42]. The mechanical arm of the cavity modulates the resonant cavity mode and thus imprints a signature of its motion on the transmission properties of the cavity. These schemes have led to very precise measurements of mechanical displacement [23] and even enabled the recent detection of gravitational waves [112]. Embedding nanomechanical resonators into microfabricated superconducting cavities, where the mechanical motion capacitively couples to the microwave cavity, has also generated much interest recently [113, 44].

In our experiments, the mechanical motion is actuated by capacitive coupling to a gate electrode. The measurement scheme we employ in our devices is discussed next.

4.2 Overview of capacitive actuation and detection

4.2.1 Capacitive actuation

All electrical configurations for actuation and detection of mechanical motion offers advantages in that it is less bulky and useful for low temperature measurements as
opposed to other schemes. In this scheme the actuation is carried out by assuming that the resonator material and a proximal gate electrode form the plates of a parallel plate capacitor\cite{114}. Application of a dc voltage, \( V_{dc}^g \), on the gate electrode causes a static attractive force on the suspended material. An additional signal, \( \tilde{V}_g \), at frequency \( \omega \) is applied to the gate electrode causes a time varying force, \( \tilde{F} \) on the resonator. The total force, \( F_{total} \), on the resonator is given by,

\[
F_{total} = -\frac{1}{2} \frac{dC_g}{dz} (V_{dc}^g + \tilde{V}_g)^2 \\
= -\frac{1}{2} \frac{dC_g'}{dz} (V_{dc}^g)^2 - \frac{dC_g}{dz} (V_{dc}^g \tilde{V}_g) - \frac{dC_g}{dz} (\tilde{V}_g)^2 \\
\approx -\frac{1}{2} C'_g (V_{dc}^g)^2 - C'_g V_{dc}^g \tilde{V}_g
\]

(4.1)

where \( C_g \) and \( z \) is the capacitance and separation between the resonator and gate electrode, \( C'_g = \frac{dC_g}{dz} \), and \( V_{dc}^g, \tilde{V}_g \) are the dc and AC gate voltages respectively. Here, the first term is the static force acting on the resonator and the driving force is seen to be \( \delta F \approx C'_g V_{dc}^g \tilde{V}_g \).

Here we have retained only terms at frequency \( \omega \) as \( \tilde{V}_g \ll V_{dc}^g \). This oscillatory force can actuate the mechanical vibrations of the suspended structure and it enters resonance when the drive frequency matches the mechanical resonance frequency. Typical resonant frequencies of the NEMS we study here are \( \sim 50 \text{ MHz} \).
4.2. Overview of capacitive actuation and detection

4.2.2 Electrical readout

Conventional electrical detection schemes use a frequency mixing technique where the resonance is detected through a low frequency (kHz) downmixed signal. This is done using schemes such as amplitude modulation, or frequency modulation, techniques where one requires a finite transconductance of the semiconducting device\[115, 116, 117]. These techniques make use of the fact that the conductance of the resonator is modulated when it oscillates relative to the gate and the semiconducting resonator itself is used as a mixer to downconvert the mechanical resonance signal to the kHz range. This downconversion is necessary to circumvent issues of parasitic capacitances that lead to low cut-off frequencies of the device. Here, a finite transconductance of the semiconducting device is required for these methods.

Commonly, devices are fabricated on substrates of highly doped Si with a layer of SiO$_2$ where the bottom Si is used as a global back gate for mechanical actuation. However, the presence of a global back gate leads to parasitic capacitances between the gate and source/drain electrodes. The device resistance and parasitic capacitance combine to make the device a low pass filter. The large device resistances ($R_d \sim 20$ kΩ) combined with the parasitic capacitance of $C_p \sim 5$ pF leads to a cut-off frequency ($\frac{1}{2\pi R_d C_p}$) of the order of a MHz. This frequency is smaller than the typical resonant frequencies of our nanoscale resonators. This necessitates the use of downmixing techniques to electrically detect the mechanical motion. Although these techniques are quite effective in characterizing NEMS resonators, mixing the mechanical signal from MHz down to kHz imposes limits of measurement bandwidth. This in turn is a serious disadvantage for real time applications using mechanical resonators (for example high speed sensors). We, therefore, use highly insulating substrates (as described in Chapter 3) with a local gate geometry to minimize effects of parasitics and measure the mechanical resonance directly at radio frequency.

The electrically contacted semiconducting resonator along with the gate electrode together form a field effect transistor (FET). The conductance of the FET channel (here the resonator) is a function of the induced charge carrier density, $q$, where $q = C_g V_g$. Therefore the conductance of the resonator can be modulated by changing either $C_g$ or $V_g$. 
Chapter 4. Measuring electromechanical resonances in NEMS

\[ \tilde{G} = \frac{dG}{dq} \tilde{q} \]

\[ \tilde{q} = \tilde{C}_g V_{dc} + C_g \tilde{V}_g \]  

(4.2)

The capacitance, \( C_g \), depends on the resonator to gate separation. Therefore, at mechanical resonance the oscillation amplitude is large and it modulates the capacitance at the frequency of oscillations. For equilibrium separation, \( z_0 \) between the resonator and gate electrode and time varying oscillations, \( \tilde{z} \), then the instantaneous resonator-gate separation is given by \( z(\omega) = z_0 + \tilde{z} \). So the variation in capacitance when the resonator oscillates is

\[ \tilde{C}_g = \frac{dC_g}{dz} z(\omega) \]  

(4.3)

Therefore the total conductance modulation can be written as

\[ \tilde{G} = \frac{dG}{dq} \left( \frac{dC_g}{dz} z(\omega)V_{dc} + C_g \tilde{V}_g \right) \]  

(4.4)

As \( \tilde{G} \) is directly related to \( z(\omega) \) it shows that the conductance modulation happens at the frequency of oscillations of the resonator and is maximum at resonance. We measure this conductance modulation at the oscillation frequency by directly measuring the rf current as described next.

**Direct readout scheme**

The direct readout scheme employs detection of the mechanical motion at the resonance frequency of the device. This is done by making use of a local gate electrode that provides the actuating force at a frequency near the resonance and detecting the transmitted signal through the resonator. The local gate geometry ensures minimization of parasitic capacitances. Additionally, we employ high resistivity silicon wafers or insulating sapphire wafers as the substrate for the devices to further minimize con-
4.2. Overview of capacitive actuation and detection

The circuit used to measure the mechanical resonance of the NEMS is shown. Bias tees are used to combine or split the rf and dc signals on the device. The amplified rf response transmitted through the resonator is measured using a vector network analyzer (VNA) or lock-in amplifier.

The circuit used to actuate and detect mechanical oscillations of our NEMS is shown in Figure 4.2. A radio frequency (rf) drive of amplitude $\tilde{V}_g$ (from a network analyzer or lock-in amplifier) and a dc voltage $V_{g}^{dc}$ is combined using a bias-tee and applied to the local gate. A dc bias $V_{sd}$ is applied to the source electrode. The drain current is split into its dc and rf components using a second bias-tee and the rf component is amplified and measured. The capacitive coupling between the suspended device and the local gate actuates the mechanical motion of the resonator modulating the rf current through the resonator. The rf current has a capacitive contribution and a resistive contribution. The capacitive component arises from the capacitance modulation where as the resistive component arises from the conductance modulations of the device as it oscillates. The capacitive component can be written as:
Chapter 4. Measuring electromechanical resonances in NEMS

\[ I_{\text{cap}} = \frac{d}{dt}(C_g V_g) = C_g \frac{dV_g}{dt} + V_g \frac{dC_g}{dz} \frac{dz}{dt} \]

\[ = i\omega \left( C_g \tilde{V}_g + C'_g \tilde{V}_g \tilde{z} \right) \quad (4.5) \]

where we have used a harmonic response of oscillation amplitude \( \tilde{z} \) of the resonator to the drive signal. The resistive component can be written as:

\[ I_{\text{res}} = \tilde{G} V_{sd} \]

\[ = \left( \frac{dG}{dV_g} \tilde{V}_g + \frac{dG}{dq} \frac{dz}{dq} \tilde{z} \right) V_{sd} \]

\[ = \left( \tilde{V}_g + V_g \frac{C'_g}{C_g} \tilde{z} \right) \frac{dG}{dV_g} V_{sd} \quad (4.6) \]

where we have used equation [4.4]. Therefore, the total rf current through the resonator is given by [4.8]:

\[ \tilde{I} = j\omega C_{\text{tot}} \tilde{V}_g - j\omega \frac{\tilde{z}}{z_0} C_g V_{gdc} + V_d \frac{dG}{dV_g} \tilde{V}_g - V_d \frac{dG}{dV_g} \frac{\tilde{z}}{z_0} V_g \quad (4.7) \]

where, \( C_{\text{tot}} \) is the total capacitance of the device, \( \omega \) is the driving frequency, and \( C_g \) and \( z_0 \) are the capacitance and separation to the local gate. Here \( \frac{dG}{dV_g} \) is the transconductance of the device.

The first term in equation [4.7] is the overall background signal that is present owing to capacitance between the gate and drain. This term is minimized by use of highly resistive or insulating substrates. This ensures that the mechanical signal can be detected. The second term arises purely due to the mechanical motion of the resonator that modulates the gate capacitance and gives rise to an rf current. The third term has a purely electrical origin. The fourth term again is related to the mechanical motion. As we are working with semiconducting materials the conductance of the device depends on the carrier density. The gate voltage tunes the carrier density in
4.2. Overview of capacitive actuation and detection

Figure 4.3: (a) The dc current through a nanowire resonator is tuned from zero to saturation with applied dc gate voltage. The slope of the curve is directly proportional to the transconductance of the device. The blue circle marks a region of high transconductance whereas the red circle marks a region of low transconductance. (b) The resonance signal measured at the gate voltages marked by the circles in (a).

The resonant response of a nanowire resonator at ±20 V gate voltage showing the effect of a non-zero transconductance. As the magnitude of the gate voltage is same the force acting on the resonator in both cases are equal. However, in regions (blue circle) where the transconductance of the device is high the circuit is more effective in detecting the mechanical motion. Typical resonant response measured in experiments is seen in Figure 4.3(b) as a dip. Here, the signal was detected using a network analyzer where we measure the transmission coefficient of the device $|S_{21}| = 50 \, \Omega \, \tilde{I}/\tilde{V}_g$; where 50 $\Omega$ is the network analyzer’s input impedance. The response can be a peak or a dip depending on the sign of the applied source drain bias voltage which leads to a phase difference between various terms in equation 4.7. The response can be fitted to a Lorentzian with a phase to extract the oscillation amplitude and quality factor.

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4.2.3 Non-Lorentzian line shapes of resonance

The amplitude of motion for a damped harmonic oscillator with a driving force, \( \tilde{F} \), is given by the equation

\[
\tilde{z} = \frac{\tilde{f}}{m} \left( \frac{1}{\omega_0^2 - \omega^2 + j\omega_0\omega/Q} \right)
\]

(4.8)

where, \( m \) and \( \omega_0 = 2\pi f_0 \) is the mass and resonant frequency of nanowire resonator, \( \omega = 2\pi f \) is the driving frequency and \( Q \) the quality factor. For the direct readout scheme used here the driving force is \( \tilde{F} \approx -\frac{C_{bg}}{z_0} V_g V_g \), where \( z_0 \) is the equilibrium separation between the resonator and local gate. This form of the amplitude leads to the oscillation power spectrum having a Lorentzian line shape.

However, as we see from Figure 4.3(b), typical resonant response measured by the direct readout scheme deviates from the ideal Lorentzian response. Electrical measurements of NEMS is usually accompanied by a background signal because of the parasitic capacitance. In our scheme, the phase difference between various terms of the rf current leads to a power spectrum that has a Lorentzian line shape with a phase factor. Following the scheme of [116], we use a function \( L(f) \) with a non-zero phase and background signal to fit all our experimental results unless specified otherwise. The form of the function used to fit the linear amplitude of the measured signal is

\[
L(f) = (A + Bf)e^{i\phi} + \frac{C}{f^2 - f_0^2 + i\omega f_0/Q}
\]

(4.9)

where \( A \) and \( B \) are parameters used for the background signal, \( \phi \) is the phase factor between the background signal and the mechanical term, and \( C \) is a scaling factor for the signal on resonance.

Figure 4.4 shows various line shapes of \( L(f) \). We see that the presence of a background signal naturally leads to complex line shapes for the experimentally observed resonance. We fit the experimentally observed response using the above function to extract the resonance frequency and quality factor of the device. Figure 4.5 shows a
4.2. Overview of capacitive actuation and detection

Figure 4.4: Examples of line shapes of the form given in equation 4.9 with different phase factors using the parameter values $A = 0$, $B = 0.2$, $C = 1$, $f_0 = 10$, and $Q = 100$. The parameter values are specified in arbitrary units and the plots are offset for clarity.
Chapter 4. Measuring electromechanical resonances in NEMS

Figure 4.5: Plot showing experimentally observed resonance response (red circles) along with a fit using equation 4.9 (blue curve). The resonance frequency and quality factor obtained by the fitting procedure is given alongside the plot. The standard error for the resonant frequency is negligibly small.

typical measurement with a fit using equation 4.9. Here, we note that the values of $f_0$ and $Q$ obtained from the fitting procedure are only weakly sensitive to any small changes in the background parameters.

4.2.4 Measurement setup

To reduce effects of viscous drag from the surrounding fluidic environment all measurements were carried out in evacuated chambers. The room temperature measurements were carried out in homemade vacuum chambers with hermetic feedthroughs for electrical connections. The low temperature measurements were carried out in a liquid helium flow cryostat with the device inside a vacuum-can. The vacuum-can was evacuated at room temperature to a pressure of $\sim 10^{-3}$ mbar prior to loading into the cryostat. Although we do not measure the pressure directly near the sample in the cryogenic environment, we assume that the sample is cryo-pumped to pressures well below $10^{-3}$ mbar when cooled to cryogenic temperatures. A low temperature rf amplifier (CITLF1, Microwave Research Group, California Institute of Technology) was setup inside the cryostat vacuum-can near the device to improve signal collection.
4.3 Summary

In this chapter we have discussed some of the common techniques that are used for actuation and detection of mechanical motion in nanoscale resonators. We also discussed the capacitive actuation and electrical readout scheme that we employ in our resonators. We also provide information on typical measurement results and our scheme to calculate the resonant frequency and quality factor. The measurement setups used and challenges were also outlined.
Chapter 5

Highly tunable graphene drums
(low tension regime)

In this chapter we discuss our experiments on graphene drum resonators with low built-in tension at cryogenic temperatures. The low tension makes the resonator frequencies highly tunable using a gate voltage. The electrical tunability of the graphene drum is used to induce coupling between different mechanical modes. We discuss the experiments on dynamical strong coupling of vibrational modes of graphene. The effect of strong coupling on the hybridization and dissipation of mechanical modes is studied using pump-probe schemes. We further discuss the experiments that make use of the high tunability to achieve parametric amplification and self-oscillations in the resonator. We also demonstrate the use of such low tension drums as cryogenic pressure sensors. The results presented here were published in Nature Nanotechnology 11, 747 (2016) and 2D Materials 3, 1 (2016). Raj Patel, Abhinandan Borah, and Dr. R Vijay were collaborators on this work.
5.1 Frequency tuning in an electromechanical drum resonator

In Chapter 2 we had shown how the frequency of a circular membrane is decided by the stress acting on it. This stress can arise during device fabrication and also from the electrostatic force applied from a gate electrode. Figure 5.1(a) shows an SEM image of a graphene drum resonator along with a schematic of the circuit used for actuating and detecting the motion.

Figure 5.1: (a) False colored SEM image of the graphene drum resonator. The region shaded green shows the suspended part of the graphene. Scale bar is 3 µm. (b) Schematic of the circuit used to actuate and detect the mechanical modes of the drum.

Weak radio frequency (rf) signals are applied to the gate to drive the resonator, and the motion is detected using a lock-in measurement of the rf signal transmitted through the graphene flake as described in Chapter 4. The driving rf signal (amplitude: $\tilde{V}_g$, frequency: $\omega_d$) is combined with an appropriate rf pump signal (amplitude: $V_p$, frequency: $\omega_p$) and applied to the gate electrode. The role of the pump signal is explained in the next section. An additional dc voltage is applied using a bias tee on the gate electrode which pulls the drum towards the gate electrode as shown in Figure 5.1(b). This induces a tension in the suspended graphene that provides tunability of various modes of the resonator with dc gate voltage. The gate voltage induced tension in the membrane thus also allows parametric modulation of the resonator frequency.

The graphene drum resonators we fabricate have low built-in tension even at cryogenic temperatures. In our devices, the low tension in the membrane arises from its unclamped geometry. The free expansion of the graphene flake with respect to the
Figure 5.2: Graphene drum electromechanics in the low tension regime. Frequency shift with gate voltage of a graphene drum resonator device at 5 K (a) and another device at 70 K (b). We see that both the devices show considerable, monotonous shift in frequency with absolute value of gate voltage. Panels (c) and (d) show data from literature showing decrease in tunability of clamped graphene resonators when cooled to low temperatures. Presence of a negative dispersion near zero gate voltage is also seen at low temperatures. Panel (c) is adapted from Singh et al. [120] and panel (d) is adapted from Chen et al. [117].
5.1. Frequency tuning in an electromechanical drum resonator

bottom substrate (SiO$_2$ and sapphire) allows for strain relaxation when the device is cooled to low temperatures. As the suspended part of the flake along with the gate electrode forms a parallel plate capacitor, application of a dc voltage on the gate electrode exerts an electrostatic force on the membrane. This leads to bending induced tension in the membrane and subsequently an increase in the resonant frequency as the resonance frequency, $f$, of a membrane under tension $T_0$ is $f \propto \sqrt{T_0}$. The change in frequency with tension is then given by $\frac{df}{dT_0} \propto \frac{1}{2\sqrt{T_0}}$. This implies that a relatively large change in frequency is possible only when $1/\sqrt{T_0}$ is large, i.e. when $T_0$ is small.

Figure 5.2(a)-(b) shows the frequency tunability of two different graphene drum resonators used in our experiments. It is well known from literature\cite{120, 117} that the gate tunability of the fundamental resonance mode of graphene based electromechanical devices decreases drastically at cryogenic temperatures due to an increase in built-in tension. This is also sometimes accompanied by a negative dispersion, or shift, in the resonance frequency near zero gate voltage (see Figure 5.2). The negative dispersion of the frequency near zero gate voltage is seen in many NEMS systems and is understood to be due to the capacitive softening effect. This is primarily seen in devices with large built-in tension. Our devices have large tunability even at low temperatures and does not show signatures of a negative dispersion and are hence in a low tension regime. This is seen from the monotonous increase in frequency with increase in the gate voltage as shown in Figure 5.2(a)-(b) for measurements on two devices at cryogenic temperatures. In comparison, we see from Figure 5.2(c)-(d) that the tunability of similar devices studied previously (figures adapted from references \cite{120} and \cite{117} respectively) reduces at low temperatures.

The frequency dispersion with gate voltage can be understood by a simple model that incorporates the energy due to built-in strain, deformation induced strain, and electrostatic energy of the gate-membrane capacitor system\cite{121, 119}. The spring constant, $k$, of the system can be determined from the second derivative of the energy (with respect to displacement). For a fully clamped resonator of radius $R$, and thickness $t$, it has been shown that the spring constant is given by\cite{119}

$$k = \frac{2\pi Et\epsilon_0}{1 - \nu^2} + \frac{8\pi Et}{(1 - \nu^2)R^2\sigma_e^2} - \frac{1}{2} C'_g V_g^2$$

where $E$ is the Young’s modulus, $\nu$ is the Poisson ratio, $\epsilon_0$ is the built-in strain,
Chapter 5. Highly tunable graphene drums (low tension regime)

\[ C'' = \frac{d^2C_g}{dz^2} \] with gate capacitance \( C_g \), and \( V_g \) is the applied dc gate voltage. Here \( z_e \) is the static displacement of the center of the drum and is a function of the applied gate voltage (as shown in Figure 5.1(b)). The frequency of the resonator is then given by \( f = \frac{1}{2\pi} \sqrt{\frac{k}{m_{\text{eff}}}} \) where \( m_{\text{eff}} \) is the effective mass of the mechanical mode. The effective mass is mode shape dependent and can be different from the material mass due to presence of residue or adsorbates on the drum. The built-in strain of the drum resonators can be obtained by using \( \epsilon_0 \) and \( m_{\text{eff}} \) as parameters to fit the experimentally obtained frequency dispersion using equation 5.1. Using values of \( E = 1 \text{ TPa}, \nu = 0.165, R = 1.75 \mu \text{m}, \) and \( t = 3.5 \text{ nm} \), we estimate a built in strain of \( \sim 6 \times 10^{-4} \) for device 1 at 5 K which is lower than the built-in strain observed in previous graphene based devices at cryogenic temperatures [120, 117, 121].

For a membrane with low built-in tension the gate voltage induced static deflection can be significant and induce a stress that varies spatially over the membrane. This inhomogeneous, deflection-induced tension causes frequency crossing of different modes [122] and couples various flexural modes of the resonator. The coupling strength between the modes can be made large in graphene due to the high elastic modulus and large static deflection. Next we discuss the experiments on coupling mechanical modes of graphene drums.

5.2 Dynamical strong coupling between mechanical modes in graphene drums

There has been a considerable interest in exploring coupling between mechanical modes with the eventual goal of studying coupling in the quantum regime [52, 53]. Here it is useful to have a system that has mechanical modes of high frequency and tunable coupling strength between the modes. Previous studies on mechanical mode coupling have demonstrated coherent energy exchange between low frequency (\( \leq 10 \text{ MHz} \)) mechanical modes [47, 50, 48, 51]. The graphene drums that we study have high mechanical frequencies (\( \sim 100 \text{ MHz} \)). Figure 5.3 shows the response of our drum resonator (device 1) as a function of the applied gate voltage. We see that our graphene drum resonator has a family of modes with large tunability. In the following discussions we study the coupling between various modes of this resonator.
5.2. Dynamical strong coupling between mechanical modes in graphene drums

![Graph showing frequency tunability of modes of a graphene drum resonator.](image)

Figure 5.3: (a) Large frequency tunability of modes of a graphene drum resonator. The modes near 95 MHz show an avoided crossing (outlined by the black dotted lines). (b) Zoomed in scan of frequency response of the coupled modes as a function of the dc gate voltage showing avoided crossing behavior with no parametric pumping. Red pump experiments are done at the gate voltage indicated by the white dotted line where $V_g^{dc} = -36$ V.

The dashed rectangle in Figure 5.3(a) marks the region where two modes have a frequency crossing. These modes are of nearly 100 MHz frequency and are coupled. In the following text we will discuss the response of these modes to dynamical tuning of the intermodal coupling strength. We carry out experiments using a pump-probe scheme where a driving signal (probe) is used to detect the response of a mechanical mode under the action of a pump signal that modulates the mode coupling. These experiments are similar to optomechanics experiments where a low frequency oscillator is coupled to a high frequency cavity so as to realize cooling or amplification of the mechanical oscillator\[43\].

Figure 5.3(b) shows a zoomed in measurement of the coupled modes showing an avoided crossing. The avoided crossing exhibited is characteristic of strong coupling.
between the modes. We see that the modes have a frequency detuning which is controlled by the gate voltage and have a separation of \( \sim 2 \text{ MHz} \) at \( V_g^{dc} = -35 \text{ V} \).

As the gate voltage tunes the coupling between the modes we can apply rf signals on the gate electrode that dynamically tune the intermodal coupling strength (this is the pump signal). We can study the system by pumping at the frequency difference of the two mechanical modes (red pump) or at the sum of the frequencies of the two modes (blue pump). The red pump experiments allow for coherent energy exchange between the modes and can hybridize the system whereas the blue pump experiments allow us to modify the dissipation of the modes to the environment. The equations of motion of such a system and the response to red/blue pump schemes are discussed next.

### 5.2.1 Equations of motion for coupled modes

As seen from Figure 5.3(b), the gate voltage modifies the frequencies and coupling between the mechanical modes. We consider our system to be two coupled modes with voltage controlled linear detuning. The dynamics of our system can then be described using the equations of motion for two coupled vibrational modes given by Okamoto et al.\[48\] as:

\[
\ddot{x} + \gamma_1 \dot{x} + (\omega_1^2 + \Gamma_1 \cos(\omega_p t))x + \Lambda \cos(\omega_p t)y = F_1 \cos(\omega_d t + \phi) \tag{5.2}
\]

\[
\ddot{y} + \gamma_2 \dot{y} + (\omega_2^2 + \Gamma_2 \cos(\omega_p t))y + \Lambda \cos(\omega_p t)x = F_2 \cos(\omega_d t + \phi) \tag{5.3}
\]

where \( x \) and \( y \) are the displacements of the two modes, \( \Gamma_i \) are the parametric drives that modulate the stiffness of the modes, \( \Lambda \) is the coefficient of mode coupling, and \( F_i \) are the forces at drive frequency \( \omega_d \). The terms involving \( \Lambda \) are responsible for transferring energy between the two modes, whereas the terms having \( \Gamma_i \) tunes the frequencies of the modes. Here the mass of the two modes have been set to unity for simplicity. The equations (5.2)-(5.3) are used to solve the dynamics of the system numerically. Further details about individual terms in the equations of motion and their dependence on the pump voltage amplitude are discussed in Section 5.2.4. We now discuss the experiments on the coupled modes with the red pump scheme.
5.2. Dynamical strong coupling between mechanical modes in graphene drums

5.2.2 Normal mode splitting

Figure 5.4: Strong coupling between electromechanical modes. (a) The red detuned pump is shown alongside the two coupled modes on a frequency axis. Response of mode 1 as a function of the red pump detuning when (b) $V_p = 0$ V and (c) $V_p = 1.5$ V. For nonzero pump amplitude, mode 1 is seen to split in the vicinity of $\omega_p \approx \Delta \omega$. Color scale units are $\mu$V. Inset of (c) shows the response detected at $\omega_d + \omega_p$. Non-zero response indicates energy transfer to the second mode when $\omega_d + \omega_p \approx \omega_2$.

Figure 5.3(b) shows the electrostatic tunability with gate voltage of the two strongly coupled modes (as seen from the avoided crossing). We refer to the lower (higher) frequency mode as mode 1 (2) from now on. The separation between mode 1 and 2 decreases with decreasing $|V_{dc}^g|$ achieving perfect tuning at $V_{dc}^g = -35$ V. The dynamics of the system can be studied under the influence of a red, $\omega_p \sim (\omega_2 - \omega_1) = \Delta \omega$, or blue, $\omega_p \sim (\omega_1 + \omega_2)$, detuned pump signal that parametrically modulates the coupling and frequencies of the two modes. In a later section we show strong coupling between modes separated by a frequency ratio of $\sim 2$, however, we now focus on the dynamics of the two, nearby modes in the presence of a red detuned pump.

By applying $V_{dc}^g = -36$ V on the gate electrode, we tune the modes to $\omega_1 = 2\pi \times 94.65$ MHz and $\omega_2 = 2\pi \times 96.94$ MHz such that the frequency difference is
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Figure 5.5: Normal mode splitting and large cooperativity. (a),(b) With an increase in the red detuned pump amplitude, mode 1 is seen to split into two well resolved peaks with a separation given by $2g$. Constant offset has been added to the response at various pump amplitudes in (a). Dotted lines are guides to the eye. Color scale units are $\mu$V. (c) Cooperativity of the modes is seen to be as high as 60 at the largest pump amplitudes. The solid line is a quadratic fit of the data (circles) to the equation $C = \alpha V_p^2$. 
5.2. Dynamical strong coupling between mechanical modes in graphene drums

\[ \Delta \omega = 2\pi \times 2.29 \text{ MHz} \]. This is schematically shown in Figure 5.4(a) as a spectrum along with the red pump signal. We measure the response of mode 1 as a function of pump frequency at various pump amplitudes. Here, the response of the mode is detected with a weak driving signal at a frequency \( \omega_d \). Figure 5.4(b) shows the response of mode 1 as a function of the pump frequency at \( V_p = 0 \text{ V} \). This serves as a control experiment to show that at zero pump power mode 1 remains unperturbed. When the pump strength is increased to 1.5 V, mode 1 is seen to split into two when the pump frequency approaches \( \Delta \omega \) (Figure 5.4(c)). This splitting signifies a regime of strong coupling where mixing between the modes gives rise to new eigenmodes in the system [47, 48, 105]. In the strong coupling regime the two initial modes interact such that there is coherent energy transfer between them. This is demonstrated by the measurements given in the inset of Figure 5.4(c) where the response is simultaneously demodulated at \( \omega_d + \omega_p \) with no driving signal at \( \omega_2 \). As there is no driving signal at frequency \( \omega_2 \) we expect zero response from the second mode. However, energy transfer to the second mode is observed as a non-zero signal in the region where \( \omega_d + \omega_p \approx \omega_2 \).

The amount of splitting characterizes the strength of the inter-modal coupling \( g \) and it can be controlled with the amplitude of the pump signal applied on the gate electrode. Figure 5.5(a) shows the response of mode 1 when pumped at a fixed frequency of \( \Delta \omega = 2\pi \times 2.29 \text{ MHz} \) at coarse intervals of pump strength. As the pump voltage is increased, the resonance peak of mode 1 can be seen to split into two distinct peaks. This peak splitting is equivalent to an optomechanically induced transparency where coupling between a mechanical oscillator and an optical cavity gives rise to a controllable transparency region for an optical beam incident on the oscillator [47, 123]. The coupling coefficient in the equations of motion, \( \Lambda \), is proportional to the pump amplitude \( V_p \), therefore the experimentally observed splitting increases linearly (Figure 5.5(a)) with pump amplitude. Figure 5.5(b) shows a colour plot of the mode splitting at fine intervals of the pump amplitude. As the pump is increased, the split in mode 1 increases showing two well separated peaks with a separation given by the coupling rate equal to \( 2g \). The region of splitting is characterized by the coupling rate of the modes becoming larger than their individual dissipation rates (\( \gamma_i \)). This means that there is coherent energy exchange between the two modes at a rate faster than the rate at which they lose energy to the environment. At the highest pump amplitude
of $V_p = 1.5 \text{ V}$ the splitting ($2g \approx 2\pi \times 450 \text{ kHz}$) exceeds both $\gamma_1 \approx 2\pi \times 64 \text{ kHz}$ and $\gamma_2 \approx 2\pi \times 51 \text{ kHz}$. This region of strong coupling can be quantified by a figure of merit cooperativity defined as $C = \frac{4g^2}{\gamma_1 \gamma_2}$ (which is, by definition, greater than unity in the strong coupling regime).

Figure 5.5(c) shows the cooperativity between the two modes as a function of the pump amplitude and is seen to be as high as 60. This indicates that many cycles of energy transfer can be achieved between the modes before dissipation to the bath. We see that the cooperativity follows a quadratic trend with pump amplitude, which is indicative of the $V_p$ dependence of $\Lambda$. Here we have discussed red pump experiments where the modes hybridize in the presence of a red pump that provides energy at the frequency difference of the two modes. Next we discuss higher order coupling in the presence of a red pump detuned away from $\Delta \omega$.

### 5.2.3 Higher order coupling

Previously we discussed the dynamical strong coupling of modes when the pump was detuned to the frequency difference of the two modes, $\omega_p \approx \Delta \omega$. This results in hybridization and splitting of the mode and comes from the terms involving $\Lambda$ in the equations of motion. In addition, the terms involving $\Gamma_i$ lead to higher order coupling between the modes where a second order coupling is characterized by $\Gamma_i \times \Lambda$. In Figure 5.6(a), the response of mode 1 over a large range of pump frequency shows the emergence of an additional splitting when $\omega_p \approx \Delta \omega/2$. From the equation of motion we see that the spring constant term of mode 1 involves $\Gamma_1$. As $\Gamma_1 \propto V_p$ (see Section 5.2.4), this term parametrically tunes the frequency of the mode in the presence of a pump. When the pump strength is sufficiently large, the parametric modulation term and the mode coupling coefficients combine to cause energy transfer from mode 1 when $\omega_p \approx \Delta \omega/2$. In an equivalent quantum mechanical picture this can be thought of as energy exchange between mode 1 to mode 2 via a two phonon process (schematically shown in Figure 5.6(c)).

The energy exchange between the two modes via the second order coupling is further shown in Figure 5.7(a)-(b). In the region of mode splitting, we also observe a non-zero response from mode 2 (Figure 5.7(b)) even though there is no driving
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Figure 5.6: (a), Mode 1 response is probed as a function of the pump detuning over a larger frequency range with $V_p = 1.5$ V. Apart from normal mode splitting at $\omega_p \approx \Delta \omega$, an additional splitting at $\omega_p \approx \frac{\Delta \omega}{2}$ can be seen, suggesting the onset of higher order inter-modal coupling. (b),(c) Schematics to explain the normal mode splitting and avoided crossing due to higher order coupling. The coupled modes undergo mode splitting in the presence of a red detuned pump when the coupling rate compensates the individual mechanical losses. The coupled modes further interact near $\omega_p = \frac{\Delta \omega}{2}$ due to the second order coupling.

signal at frequencies around $\omega_2$. Here the signal is obtained by demodulating the resonator response at a frequency of $\omega_1 + 2\omega_p$. As both $\Lambda$ and $\Gamma_i$ are proportional to the pump amplitude the coupling rate corresponding to the second order coupling is proportional to $V_p^2$. Figure 5.7(c)-(d) shows the experimentally obtained first order and second order splitting when the pump frequency is fixed to $\Delta \omega$ and $\Delta \omega/2$ respectively. The linear and quadratic nature of the splitting for the two cases is clearly seen. The lines in Figure 5.7(c)-(d) are fits for the $n$-th order splitting by an equation of the form $y(V_p) = c_n V_p^n$ with $c_n$ as the fitting parameter for $n = 1, 2$. The low visibility of the split modes at large pump powers is possibly related to the visibility of the initial modes. However, this aspect requires further study. Another aspect that could be further probed is the presence of higher order coupling for $n = 3, 4$ etc. The equations of motion then suggests additional mode splitting when $\omega_p = \frac{\Delta \omega}{3}, \frac{\Delta \omega}{4}$. However, the coupling efficiency decreases at higher orders and large pump amplitudes.
are necessary to study these features.

The experimentally observed splitting in the presence of the red pump can be better understood by taking a closer look at the equations of motion. Next we discuss the relation between the applied signals and the terms in the coupled equations of motion. Subsequently the simulated response of these equations are compared with experimental results.

5.2.4 Comparison with numerical solutions

Here we describe the terms in the equations of motion in more detail and look at how these equations compare with the experimentally observed results. The terms appearing in the equations of motion can be obtained from the gate voltage tunability of the modes shown in Figure 5.3(b). The frequency separation of $\sim 2 \text{ MHz}$ at the perfect tuning point of $V_g = -35 \text{ V}$ is proportional to the coupling constant between the two modes.

Figure 5.8(a) shows dispersion of the two modes with gate voltage along with some of the relevant parameters related to the terms in the equations of motion. Following the analysis of Okamoto et al.[48], we see that the terms $\Gamma_i$ and $\Lambda$ used in the equations of motion are given by

$$
\Gamma_1 = \frac{\Gamma}{2} \left( 1 + \frac{\Omega_1 \delta \Omega}{\sqrt{c^2 + \Omega_1^2 \delta \Omega^2}} \right) 
$$

$$
\Gamma_2 = \frac{\Gamma}{2} \left( 1 - \frac{\Omega_1 \delta \Omega}{\sqrt{c^2 + \Omega_1^2 \delta \Omega^2}} \right) 
$$

$$
\Lambda = \frac{\Gamma c}{2\sqrt{c^2 + \Omega_1^2 \delta \Omega^2}} 
$$

where $\Omega_i$ are the eigenfrequencies of the two modes when their coupling is turned off (green dotted lines in Figure 5.8(a)) and $\delta \Omega = \Omega_2 - \Omega_1$, $\Gamma = a V_p = \Omega_1 \times \delta \Omega / \delta V \times V_p$ is the tunability of the modes, and $c = (\omega_a^2 - \omega_b^2)/2$ is the coupling constant ($\omega_a$ and $\omega_b$ are the measured resonant frequencies along the black dotted line in Figure 5.8(a)). For the red pump experiments done at -36 V, $\Omega_1 = 2\pi \times 95.0 \text{ MHz}$, $\Omega_2 = 2\pi \times 96.2$
5.2. Dynamical strong coupling between mechanical modes in graphene drums

Figure 5.7: Second order strong coupling and dependence on pump amplitude. (a) Mode 1 response shows splitting with the pump frequency approaching $\Delta \omega/2$ (b) Simultaneous response detected at the second mode (at $\omega_1 + 2\omega_p$). Mode 1 response with the pump amplitude for (c) $\omega_p = \Delta \omega$, and (d) $\omega_p = \Delta \omega/2$. The extracted coupling rate is shown in (e) with linear and quadratic fits shown by red and green lines respectively.
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Figure 5.8: Numerical solutions of the equations of motion. (a) Avoided crossing of the two modes with gate voltage. Yellow dotted lines show the eigenfrequencies ($\Omega_1, \Omega_2$) of the modes. (b) Simulated response of mode 1 as a function of the red pump amplitude. (c) The simulated response of mode 1 also closely resembles the experimental data showing first and second order coupling. Frequency dependent background was added to the simulated response.
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MHz, \( \omega_a = 2\pi \times 96.2 \text{ MHz} \), \( \omega_b = 2\pi \times 94.1 \text{ MHz} \), and the voltage controlled frequency detuning is \( \delta \Omega / \delta V = 2\pi \times 1.25 \text{ MHz} \).

Using the above given form of the terms, the coupled equations (5.2)-(5.3) are solved numerically in Mathematica. We solve for the steady state amplitude of mode 1 at each drive frequency and pump amplitude and form a colour plot to compare it to the experimental results. In order to follow the experimental conditions, the driving term for mode 1 is kept non-zero and in the linear regime, and driving term for mode 2 is kept zero. The frequencies and quality factors are set from experimentally measured response of mode 1 and 2. Figure 5.8(b) shows the simulated normal mode splitting with red pumping. The simulated response is seen to match well with the experimental results shown in Figure 5.5(b). We see that mode 1 splits in the presence of the pump signal and the mode separation increases linearly with pump amplitude. Similarly, we perform the simulation as a function of the drive and pump frequencies. Figure 5.8(c) shows the simulated response for the strong coupling experiments over extended range of the pump frequency. We see that the simulated response matches very well with the first order and second order splitting observed in the experiment shown in Figure 5.5(a).

Hitherto, we have discussed the response of the coupled modes under the action of a red detuned pump signal. In the following section, we discuss experiments with a blue detuned pump.

5.2.5 Non-degenerate parametric amplification

In previous sections, we saw that the red pump transfers energy between the two modes and causes hybridization and mode splitting when the coupling rate exceeded the rate of energy loss from the modes. In contrast, the nature of a blue detuned pump is to modify the dissipation of the modes which leads to amplification. Experiments involving a blue detuned pump are now discussed where the pump frequency is \( \omega_p \approx \omega_1 + \omega_2 \).

Figure 5.9(a) shows the frequency spectrum of the modes along with the blue pump. Care has to be taken when doing the blue pump experiments since there could be other mechanical modes near \( \omega_1 + \omega_2 \). The excitation of a third mode with
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Figure 5.9: Amplification of motion using blue pump. (a), Schematic showing spectrum of the two modes along with the blue pump. (b), The action of the blue pump is understood as a non-degenerate parametric amplification of the mechanical modes. Here $\omega_1 = 2\pi \times 94.90$ MHz and $\omega_2 = 2\pi \times 102.03$ MHz. (c), Response of mode 1 as a function of the blue pump amplitude at $V_p^{dc} = 44$ V. Color scale units are $\mu$V. The response is seen to narrow with increasing pump amplitude indicative of amplification of the mode. (d), The effective dissipation rate of the mode is seen to decrease with the pump amplitude.

In optomechanical systems, a blue detuned pump laser is used to amplify the motional amplitude of a mechanical resonator. This scenario is equivalent to an enhanced Stokes scattering of the pump photon in the cavity which increases the population of the low frequency mechanical mode to achieve amplification. The pump populates the mechanical mode faster than the rate of relaxation from the mode such that the effective dissipation rate is reduced.\[42] In our experiment with the drum resonator, the coupling between the two modes mediates this down conversion process. This process is schematically shown in Figure 5.9(b). The blue pump signal is fixed.

large amplitude could lead to complex dynamics in the system. Here we have carried out the experiments at $V_p^{dc} = 44$ V with mode 1: $\omega_1 = 2\pi \times 94.90$ MHz and mode 2: $\omega_2 = 2\pi \times 102.03$ MHz. We choose this detuning of the modes in order to avoid direct excitation of a mechanical mode near 190 MHz by the pump signal.
5.2. Dynamical strong coupling between mechanical modes in graphene drums

at a frequency of $\omega_p = 2\pi \times 196.93$ MHz. Figure 5.9(c) shows the response of mode 1 as a function of the pump amplitude. We see that the width of the mechanical response reduces with increasing pump amplitude. Figure 4d plots the linewidth of mode 1 with increasing blue detuned pump strength.

The narrowing of linewidth is understood as a non-degenerate parametric amplification of the resonator. The down converted pump populates both the mechanical modes thereby achieving amplification. From analogy to optomechanical systems, we use cavity-optomechanics theory and write down the relation between the effective dissipation rate and the pump amplitude as $\gamma(V_p) = \gamma_1(1 - \beta V_p^2)$ where $\gamma_1$ is the initial dissipation rate and $\beta$ is related to the intermodal coupling strength[42].

We fit our experimental results with $\beta$ as the fitting parameter. Using an initial dissipation rate of $\gamma_1 \sim 2\pi \times 89$ kHz we see that the experiment matches well to the theoretical response. We see that the effective dissipation rate is tuned by a factor of 4 at the highest pump amplitude before approaching the region of instability. The region of instability is characterized by the effective dissipation becoming negative at large pump amplitudes. However, we note that the effective dissipation rate could not be reduced below 10 kHz in our device. At low dissipation rates the amplitude of the resonator is large and we expect non-linear phenomena to play a role. This was observed as complex lineshapes and the emergence of additional peaks in the mechanical response of the resonator. Additionally, non-linear damping of the resonator at large motional amplitude presents another challenge in reducing the effective dissipation further. This aspect is further discussed in Section 5.3.

The blue pump experiments can be considered to be an extension to the standard case of parametric amplification of a single mode with a $2\omega$ pump[124]. In our system the gate voltage induced tension can be used to modulate the mode at twice its resonant frequency and amplify the mechanical response. These experiments using a $2\omega$ pump are discussed in Section 5.3.

5.2.6 Coupling between well separated modes

So far we have shown that modes that are separated in frequency by $\sim1$ MHz can be strongly coupled. However, to demonstrate the ability of a mechanical system to
Figure 5.10: (a) The modes of our drum resonator are shown as a function of the dc gate voltage. The black circles mark the well separated modes on which coupling experiments were done. (b) The highest mode we observe is also seen to have good tunability with the gate voltage. The arrows mark the position of the mode to aid visibility. (c) Schematic showing the spectrum of modes near 47 and 92 MHz along with the red sideband. (d) Splitting of the lower mode is seen with increasing the red pump amplitude at a frequency of $\Delta \omega \approx 44.7$ MHz. The response beyond pump amplitude of 0.3 V is not well understood. (e) Cooperativity of the well separated modes is seen to increase to $\sim 10$ for pump amplitude up to 0.3 V.
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match an optomechanical system one needs to show that modes that are well separated can also be coupled in a similar manner. Coupling between various modes of the drum can be used to cool a low frequency mode using a higher frequency 'cavity' using sideband cooling schemes. Here we discuss the dynamical strong coupling experiments on modes separated by a factor of \( \sim 2 \) in frequency.

Our drum resonator has a family of tunable mechanical modes as shown in Figure 5.10(a) where the highest mode that we detect has a frequency \( f \sim 240 \text{ MHz} \). Figure 5.10(c)-(e) shows the red pump experiments for mechanical modes near 47 MHz and 91 MHz at a gate voltage of \( V_g = 34 \text{ V} \) with a red pump frequency of \( \Delta \omega \approx 44.7 \text{ MHz} \). Increasing mode splitting of the mode near 47 MHz is seen with pump amplitude with highest cooperativity of \( \sim 10 \). The new emergent modes are seen to disappear beyond a pump amplitude of 0.3 V. This is an aspect which is not fully understood and requires further study.

Similarly, non-degenerate parametric amplification of the well separated mechanical modes is achieved using blue pump experiments. Figure 5.11 shows the narrowing of the line response of the mode at 47 MHz along with the decreasing effective dissipation rate with blue pump amplitude with an initial dissipation rate of \( \gamma_1 = 2\pi \times 68 \text{ kHz} \).

The coupling between modes that showed an avoided crossing near 95 MHz was relatively straightforward to capture using the equations of motion provided in 5.2–5.3. Such a simplified description of mode coupling for the well separated modes is difficult. However, to shine light on the nature of coupling between these various modes, we now discuss the origin of mode coupling in graphene drum resonators.

5.2.7 Origin of modal coupling in graphene drums

The nature of intermodal coupling can be complex and can arise from several sources like geometric non-linearities, and asymmetries in the system including folds, wrinkles, and mass loading. The generalized formulation for modal coupling in electromechanical resonators of graphene drums was shown by Eriksson et al.\cite{122}. The equations of motion of any set of coupled modes can be written as\cite{122}.
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Figure 5.11: (a) Schematic for amplification experiments showing the spectrum of modes near 47 and 92 MHz along with the blue sideband. (b) Response of the 47 MHz mode to a blue pump of frequency \( \omega_p = 2\pi \times 139.28 \) MHz shows a narrowing of the mode which signifies an effective dissipation rate that decrease with the pump amplitude as shown in (c).

\[
\frac{\partial^2 x_i}{\partial \tau^2} + \Lambda_i x_i + \sum_{j=1}^{\infty} \sum_{k \geq j}^{\infty} Q_{jk}^i x_j x_k + \sum_{j=1}^{\infty} \sum_{k \geq j}^{\infty} \sum_{l \geq k}^{\infty} C_{jkl}^i x_j x_k x_l = f_i(\tau) \quad (5.7)
\]

where \( x_i \) is the displacement coordinate of the i-th mode and \( Q_{jk}^i \) and \( C_{jkl}^i \) are coupling coefficients that give rise to intermodal coupling.

Asymmetries can give rise to complex response of the system and is ignored in the proceeding discussion. Here we focus on the geometric effects that lead to non-linear modal coupling. Consider a drum of radius \( R \) and thickness \( t \) with an initial tension \( T_0 \). We consider the drum to be an electromechanical resonator with a gate electrode at a distance \( d \). A dc voltage applied on the gate electrode attracts the membrane towards the gate due to the electrostatic attractive force giving a static deflection to the center of the membrane, \( z_e \). The deflection of the membrane induces an additional stress with a spatially varying inhomogeneous component that can result in coupling between different modes of the resonator. The geometric non-linear coupling arising out of deflection induced stresses in the membrane can therefore give rise to mode
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Figure 5.12: Tunability of the coupling strength (prefactor of the coefficients $Q_{jk}^i$) as a function of the applied dc gate voltage. The static deflection induced tension modifies the coupling coefficients by orders of magnitude allowing us to parametrically tune the mode coupling.

Specific coupling coefficients, $Q_{jk}^i$ and $C_{jkl}^i$. Since these coefficients arise due to tension induced by the electrostatics it naturally leads to tunability of the coupling coefficients with applied gate voltage.

For modes 1 and 2 described in the previous sections, terms of the form $Q_{23}^1$ and $Q_{13}^2$ (or in general terms having $Q_{2j}^i$, and $Q_{1i}^2$) are non-zero and can provide a linear coupling. This gives a set of linearly coupled equations of motion, similar to the model we have used to explain our observations in the previous sections. The exact form of the coupling coefficients $Q_{jk}^i$ depends on the particular modes that are being coupled. However, the mode dependent parts can be shown to have a magnitude of $\sim 10$. In addition, the coupling coefficients have a mode independent prefactor which is related to the static part of the stress due to electrostatic tuning. We now show how this prefactor of $Q_{jk}^i$ varies with the gate voltage.

The mode coupling prefactor is given by $\frac{Et}{TR} \frac{z_e}{1 - \nu}$, where $E$ is the elastic modulus and $T$ is the total stress on the membrane. Here $T$ includes the built-in stress, $T_0$, and a gate voltage dependent part where the total stress is related to the static center deflection by $T = T_0(1 + z_e^2G^2/4)$ where $G$ is a geometric factor given by $G = 1/R(Et/T_0)^{1/2}\sqrt{(3 - \nu)/(1 - \nu)}$.

Figure 5.12 shows how the prefactor of coupling coefficients can be tuned with
the gate voltage (here we have used parameters for device 1 as given in Section 5.1). The strain in the device was estimated by fitting the gate voltage tunability of the fundamental resonant mode and was found to be $\sim 0.06\%$. Following the analysis of Eriksson et al., the center static deflection was calculated to be $\sim 20$ nm at $V_{dc} = 40$ V. For such deflections, the presence of inhomogeneous terms in the deflection induced stress can lead to frequency crossings in the resonator. For the strong coupling experiments discussed previously we have performed the measurements at gate voltages in the range of 35-45 V. In this regime the static deflection of the drum is large enough to cause large intermodal coupling strength and frequency crossings, which were aspects that we explored in our experiments.

Until now we have discussed mode coupling experiments in graphene drums that made use of large frequency tunability in the system. In the following section we discuss how the tunability can be used to parametrically excite the resonator to achieve amplification and self-oscillations.

### 5.3 Parametric amplification of mechanical motion

The tunability of frequency with gate voltage further allows us to parametrically modulate the spring constant of the drum resonator and realize amplification. There has been considerable interest in parametric resonators as they can act as ‘on chip’ mechanical amplifiers. The simplest example of a parametric resonator is that of a child on a swing. Here a standing child takes a ‘dip’ at the extreme points of the motion, thereby modulating the mass distribution at twice the resonant frequency and achieving amplification of motion. Here the equation of motion of the resonator of mass $m$ can be expressed as

$$m \frac{d^2 z}{dt^2} + m \left( \frac{\omega_0}{Q} \frac{dz}{dt} + [k_0 + k_p(t)] \right) x = F(t)$$

where $z$ is the resonator displacement, $\omega_0$ and $k_0$ are the resonant frequency and unperturbed spring constant of the resonator respectively, $Q$ is the quality factor, $F(t)$ is the driving force, and $k_p(t)$ is the modulated spring constant. This equation is known as the damped Mathieu equation when the spring constant is modulated.
5.3. Parametric amplification of mechanical motion

harmonically such that $k_p(t) = \Delta k \sin(\omega_p t)$ where $\omega_p$ is the pump frequency.

In our drum resonator we see that the frequency of the lower mode increases with magnitude of the gate voltage with a nearly constant slope of $\sim 1$ MHz/V at higher voltages. The monotonic increase in frequency with gate voltage is an indication of low built-in tension in the membrane. When the dc voltage is increased the membrane is pulled closer to the gate electrode due to electrostatic attraction and introduces a bending induced tension that increases the resonant frequency. We exploit this tunability to parametrically modulate the spring constant of the resonator. This is achieved by applying a pump signal at $V_p \sin(\omega_p t)$ on the gate electrode. The dc ($V_{dc}^g$) and pump voltages cause a modulation of the spring constant given by

$$k_p(t) = \Delta k \sin(\omega_p t) = \frac{d^2C}{dz^2} V_{dc}^g V_p \sin(\omega_p t)$$

(5.9)

For pump strengths exceeding a threshold value the resonator undergoes self-oscillations, whereas the mechanical amplitude is amplified for pump strengths below the critical pump amplitude. Next we discuss self oscillations and parametric amplification observed in our graphene drum resonators.

5.3.1 Self oscillations

Self sustained oscillations, or self oscillations occur when a resonator undergoes oscillations even in the absence of a driving force. In electromechanical resonators, self oscillations occur when the parametric pump voltage is larger than a threshold value such that the resonator is in a regime of instability. For our electromechanical resonator this threshold pump value is denoted by a critical pump amplitude ($V_{pc}$).

To find the critical pump amplitude we detect the response of the resonator with increasing pump amplitude while keeping the driving force set to zero.

Figure 5.13 shows the response of the first mode measured at frequency $\omega_d$ as a function of the pump amplitude when the driving force at $\omega_d$ is zero ($V_g = 0$ mV). Here the pump frequency is being swept at a frequency of $2\omega_d$. As the driving force is kept zero, there is zero response from the resonator at frequency $\omega_d$ for low pump amplitudes. However, at larger pump amplitudes we measure a non-zero...
response from the resonator. In this condition the graphene drum enters a regime of self-oscillations beyond a critical pump amplitude ($V_{pc}$) of 248 mV at a resonance frequency of $\omega_0 = 2\pi \times 51.67$ MHz. The tongue shaped response is characteristic of instability in parametrically driven systems.[125] The critical modulation of the spring constant makes the resonator unstable whereby any noise or fluctuations in the system drives it into oscillations. Next we discuss the response of the resonator when parametrically excited below the threshold for self oscillations.

### 5.3.2 Response below critical pump amplitudes

We now consider the response of the resonator for pump strengths below the critical pump amplitude ($V_p < V_{pc}$). Let the driving force acting on the resonator be denoted by $F(t) = F_0 \cos(\omega_0 t + \phi)$ and let the pump be fixed at twice the resonant frequency such that there is a well defined phase difference, $\phi$, between the drive and pump signals. We assume that the steady state solution of the resonator is of the form $a(t) = A \exp(j\omega_0 t)$ where $A$ is the complex amplitude of motion. The parametric resonator equation (equation 5.8) can then be solved in the high quality factor limit. The amplitude response is given by[124]
5.3. Parametric amplification of mechanical motion

\[ A = F_0 \frac{Q \omega_0}{k_0} \left[ \frac{\cos(\phi)}{1 + Q \Delta k/2k_0} + j \frac{\sin(\phi)}{1 - Q \Delta k/2k_0} \right]. \]  

(5.10)

We see that the amplitude of motion is a function of the pump strength as well as the phase difference between the drive and pump signals. We can quantify the amplitude of motion in the presence of the pump using the gain, \( G(\phi) \), defined as

\[ G(\phi) = \frac{|A|_{\text{pump on}}}{|A|_{\text{pump off}}}. \]  

(5.11)

Figure 5.14: 2\( \omega \) pumping induced amplification and self oscillations. (b) Influence of pump voltage at 2\( \omega_0 \) shown as a function of the phase difference between the pump and drive signals. The drive signal amplitude is \( \sim 1 \text{ mV} \).

Following the analysis of Rugar et al.\cite{124} we see that the gain of the amplifier can be written as

\[ G(\phi) = \left[ \frac{\cos^2(\phi)}{(1 + V_p/V_{pc})^2} + \frac{\sin^2(\phi)}{(1 - V_p/V_{pc})^2} \right]^{1/2} \]  

(5.12)

where \( V_{pc} = 2k_0/QV_g d^2C/dz^2 \). This implies that the amplitude of motion increases with pump amplitude for the phase difference \( \phi = \pi/2 \) and approaches infinity for \( V_p \to V_{pc} \). This is the threshold for self oscillations as seen earlier. For the phase \( \phi = 0 \) we see that the gain reduces below one and the motion is deamplified.
Chapter 5. Highly tunable graphene drums (low tension regime)

In our experiments we realize the above conditions by applying a small tone at $\omega_0$ to drive the system at its resonance. The pump is then fixed at the frequency $2\omega_0$. Figure 5.14 shows the response of the drum resonator at resonance as a function of the phase, $\phi$, for increasing pump amplitudes. With the pump on we observe a periodic modulation of the otherwise flat signal with phase. The response with pump is seen to go above and below the response at zero pump. This is characteristic of systems with parametric amplification where the response is amplified for certain values of phase difference and de-amplified for others. To quantify the gain in our resonator we need to measure the amplitude of motion with and without the pump signal. However, the response measured here is not directly proportional to the amplitude of motion. This is because of the electrical background that is measured along with the resonance signal.

From Chapter 4, we see that the rf current $\tilde{I}$ we measure for $V_{sd} = 0$ V in our scheme of transduction is related to the amplitude of oscillations $z$ by:

$$\tilde{I} = i\omega_d C_p \tilde{V}_g - i\omega_d \frac{z}{d} C_g V^{dc}$$

(5.13)

where $C_p$ is the parasitic capacitance between the gate and drain and $C_g$ is the capacitance of the gate at distance $d$ from the membrane. To acquire the response proportional to the amplitude of motion the first term has to be nullified. For this we measure the response at $\omega_d$ keeping $V^{dc} = 0$ V. This background is then vectorially subtracted from the measured signal to give only the second term, proportional to the amplitude of motion. Figure 5.15 shows the response of the resonator before and after background subtraction. We see that the background has been nullified and we regain the normal Lorentzian response of a driven resonator. The background subtracted response of the resonator is used to quantify the gain in our parametric resonator.

Figure 5.16(a) and (b) shows the gain of the resonator as a function of the phase and pump amplitude by using the background subtracted signals. The gain is seen to be periodically modulated with the phase with the modulation strength increasing with pump amplitude. We see that the phase for maximum deamplification is not zero. This could be due to phase differences in the $\omega_0$ and $2\omega_0$ signals arising from our experimental setup. The phase response of the resonator is fitted using equation...
5.3. Parametric amplification of mechanical motion

Figure 5.15: Response of the fundamental mode before and after subtraction of the background signal. The background signal arises from parasitic capacitances and can lead to non-Lorentzian lineshapes of the resonance.

From Figure 5.16(b) we observe that the experimentally attained gain deviates from the prediction from equation 5.12. For higher pump powers the gain does not follow the predicted value and stagnates to a maximum of around 3. This deviation from the theoretically predicted behaviour could be due to non-linear damping in graphene and is discussed next.

Non-linear damping

The presence of non-linear terms in the equation of motion of NEMS systems was discussed in Chapter 2. These terms can cause saturation in a parametric amplifier since the dissipation depends on the amplitude of motion.\[64, 126\]

Previous studies have shown that non-linear damping plays a role in resonators made of carbon nanotubes and graphene.\[64\] We also see the effects of non-linear
Figure 5.16: (a) Gain of the resonator as a function of the phase difference between drive and pump signals along with a fit using equation 5.12. (b) A colour plot showing the gain of the resonator as a function of the pump amplitude and phase difference. (c) Gain at two different phase differences extracted from (b) and shown along with a theoretical fit. Stagnation of the experimental gain is seen.
damping in our graphene drum resonator device. Figure 5.17 shows the quality factor of the resonator as a function of the rf drive voltage. Here the drive strength is kept low to avoid broadening due to the Duffing response. We observe a decrease in quality factor with increasing drive. As the amplitude of motion, is directly proportional to the drive voltage we infer that the resonator is more lossy at larger amplitude of oscillations. This supports the observed stagnation of gain in our parametric pumping experiments.

5.4 Pressure sensing with graphene drums

Until now we have discussed experiments that made use of large frequency tunability for studies of a fundamental nature on graphene based resonators. To further demonstrate the use of these devices we now discuss experiments on cryogenic pressure sensing using graphene drums.

The following results are obtained from device 2 at a temperature of 70 K with measurements being carried out in the absence of a low temperature rf amplifier. So in order to boost the measured signal we apply a source-drain bias voltage of $V_{sd}^{dc} = -300$ mV as discussed in Chapter 4. We study the shift in frequency of the drum device when a known quantity of helium gas is introduced above the device. The role of the gas molecules is to exert a pressure on the drum such that it aids the capacitive pull from the gate by shifting the equilibrium position of the drum downwards. Figure
Figure 5.18: (a) Measured resonance response of device 2 as a function of $V_g$ with -10 dBm drive power in presence of helium (red dots) and in vacuum (green dots) at 70 K. The red and green lines are fits obtained from the procedure outlined in the text. (b) Change in resonance frequency measured with time in presence of helium with pressure 14 Torr (from 0 to 12 minutes) and in vacuum after 12 minutes (background subtracted, response in dB).

5.18(a) shows the frequency dispersion of device 2 in vacuum and in the presence of 14 torr of helium gas. The reference gas pressure was measured using a Baratron gauge (MKS) placed at the top of the cryostat. The gas aids the capacitive pull by the gate, thus increasing the tension and resonant frequency of the drum at a given gate voltage.

For use as a sensor we estimate the pressure using a simple model that captures the modified frequency dispersion in the presence of the gas. As discussed in the section on tunability of the graphene drums equation 5.1 gives the effective spring constant of our electromechanical drum resonator when no external gases are present. In the presence of a gas we calculate the contribution of pressure, $P$, to the total energy of the electromechanical system and find the new effective spring constant, $k_{eff} = k_{eff}(V_g, P)\[119\]$.

The total energy in presence of helium molecules consists of the stored elastic energy ($E_{elastic}$) in the membrane, electrostatic energy ($E_{electrostatic}$) and the energy due to the force exerted by gas pressure ($E_{pressure}$). Therefore, the total energy of the
5.4. Pressure sensing with graphene drums

device in the presence of a gas at pressure $P$ can be expressed as,

$$E_{\text{total}} = \frac{\pi E t}{1 - \nu^2} \int_0^R \left[ \varepsilon_0 + \frac{1}{2} \xi'(r)^2 \right] rdr - \frac{1}{2} C_g V_g^2 - \frac{1}{2} \int_0^R \int_0^{2\pi} \xi(r) r dr d\theta. \quad (5.14)$$

Here $\xi(r)$ is the shape of the drum under a uniform load and is given by $\xi(r) = z(1 - \frac{r^2}{R^2})$, where $z$ is the static center deflection of the membrane, and $r$ is the distance of any point on the membrane from the center. Here, the pressure on the membrane is uniform throughout the surface. At equilibrium the drum attains an equilibrium static deflection $z_e$ at the center. We minimize the total energy at $z_e$ to obtain an equation that allows us to solve for the static deflection for an applied gate voltage.

$$\frac{8\pi E t}{3(1 - \nu^2)R^2} z_e^3 + \left( \frac{2\pi E t \Gamma_0}{(1 - \nu^2)} - \frac{1}{2} C_g V_g^2 \right) z_e - \left( \frac{1}{2} P \pi R^2 + \frac{1}{2} C_g V_g^2 \right) = 0 \quad (5.15)$$

The real solution of the cubic equation (Equation 5.15) gives $z_e$ as a function of the initial strain, gate voltage and gas pressure. We assume the drum to have an initial droop of $z_0$ such that $z_e \rightarrow z_e + z_0$. As the membrane is not held down by metal electrodes, relative expansion and contraction of the sapphire, SiO$_2$ and graphene can lead to sagging of the membrane upon cooling. Then the spring constant of the system, $k_{eff}$, can be written as

$$k_{eff} = \frac{\partial^2 (E_{\text{elastic}} + E_{\text{electrostatic}} + E_{\text{pressure}})}{\partial z^2} \bigg|_{z_e}. \quad (5.16)$$

Although the spring constant is not explicitly a function of $P$, it depends on the pressure through $z_e$. We use this $k_{eff}$ in the frequency expression given by,

$$f = \frac{1}{2\pi} \sqrt{\frac{k_{eff}}{m_{eff}}}$$

where $m_{eff}$ is the effective mass of the multilayer graphene membrane. This model is valid both in vacuum ($P = 0$ Torr) and in the presence of a gas ($P \neq 0$) and can be used to find the pressure by fitting the observed frequency dispersion with gate voltage. We first fit our frequency dispersion data in vacuum using the above model with $P = 0$ Torr (Figure 5.18(a) green curve) using initial strain and effective mass as parameters, which yields the value of initial strain as 0.03\%, initial droop, $z_0$, as 6 nm and effective mass as 2.2 times the actual mass of the
multilayer graphene membrane. The strain is seen to be lower than previous studies on graphene based resonators at cryogenic temperatures and shows that our device is under low tension \[119, 95\]. The increased effective mass arises due to adsorption of gases, and residue on the membrane during fabrication, and can also vary with mode shape. \[127, 120\] The dimensions of device 2 used in the calculation are drum radius \(R = 1.62 \times 10^{-6} \text{ m}\), thickness \(t = 7.5 \text{ nm}\), and initially suspended at a height of 300 nm above the substrate. Subsequently, \(P\) was used as the only free parameter to fit the frequency response with gate voltage in presence of gas. The value of \(P\) obtained from fitting was 15.7 Torr, in close agreement to the reference value measured by a reference gauge.

Additionally, the response of the mode at a fixed gate voltage was monitored as a function of time when the pressure was again reduced to zero. Figure 5.18(b) shows the resonance frequency shift (seen as a jump) when the helium gas was pumped out. The system was pumped out at the 12 minute mark (marked by the arrow in Figure 5.18(b)) at which point the resonance frequency drops from \(\sim 47\) to \(46\) MHz. Although graphene is a near impermeable material, it does allow for gas diffusion along the edges over long time periods \[32\]. We ensured that the timescale in which our experiments were performed was small enough not to allow diffusion of helium molecules across the drum \[32\]. This shows that our device can be used for pressure sensing by monitoring the frequency shift occurring from pressure changes. As a frequency shift of 1 MHz is observed for a pressure of 14 Torr and as the quality factor of this device is near 500 at 70 K, this translates to a pressure sensing resolution of 1 Torr in these devices.

### 5.5 Summary

To summarize, in this chapter we have discussed some experiments on graphene drum resonators with large frequency tunability. The device architecture gives rise to a low tension state and large tunability of the drum resonator even at low temperatures. Experiments similar to optomechanical systems were performed using coupled mechanical modes. We discussed the dynamical strong coupling of mechanical modes which was realized by pump-probe schemes. Hybridization and mode splitting in
5.5. Summary

the presence of a red pump, and amplification in the presence of a blue detuned pump were discussed. Additionally, the experiments involving a $2\omega$ pump were discussed that allowed us to achieve parametric amplification and self-oscillations in the graphene drums. Finally, a potential use of these drum resonators as pressure sensors at cryogenic temperatures was discussed.
Chapter 6

InAs nanowire resonators with low tunability (high tension regime)

In this chapter we discuss experiments on doubly clamped InAs nanowire resonators under high tensile stress with low frequency tunability. We discuss how the thermal properties of individual nanowires can be measured from their mechanical response to thermal stress. This is achieved by monitoring the frequency shifts of the fundamental mode of resonance when the semiconductor is Joule heated. The nature of the experimentally observed frequency shifts with Joule heating is attributed to the thermal expansion coefficient and thermal conductivity of InAs. We also discuss the effects of clamping loss on the observed temperature dependence of dissipation in our devices. Further, we look at mode-mode coupling between perpendicular components of the fundamental vibrational mode. The main results of this chapter were published in Nano Letters 15, 11 (2015). Raj Patel, Abhinandan Borah, Carina B Maliakkal, and Dr. TS Abhilash were collaborators on this work.
6.1 Measuring thermal conductivity using electromechanics

There are few methods to measure the thermal conductivity of individual nanoscale materials, including the direct use of Fourier’s law, the $3\omega$ method, and the time domain reflectance measurements. They have helped immensely in understanding thermal conductivity of various systems. However, there is a need for measuring thermal conductivity of wide variety of nanostructures in a suspended beam, or drum geometry. In subsequent discussions we will describe how our method uses the system itself for thermometry. Using this method, any change in temperature profile across the nanostructure is transduced into a stress that changes the mechanical frequency of the suspended material.

As discussed in Chapter 3, the devices we use here involve InAs nanowires in a suspended field effect transistor geometry fabricated on insulating substrates. The nanowires are suspended 200 nm above the substrate by metallic contact electrodes. A schematic of the circuit used along with an SEM image of a device is shown in Figure 6.1. The circuit shown here allows us to heat the nanowire as well as measure its resonance frequency. A source-drain bias voltage is applied to one of the contacts of the nanowire to pass a dc current and Joule-heat the nanowire. The dc and rf currents through the nanowire are simultaneously measured. A local gate electrode is used to actuate the mechanical vibrations of the nanowire. As InAs is a semiconductor the gate also allows us to tune the charge carrier density in the nanowire. The dc current flowing through the nanowire can thus be tuned by a voltage on the gate electrode.

In the experiments discussed in the following section, we are interested in studying the mechanical response of the nanowire to applied thermal stresses. We first discuss the experiments on Joule heating and study its effect on the temperature across the suspended nanowire device. Subsequently, we discuss the effects of thermal stress on the mechanics of the nanowire resonator to relate the resonant frequency shifts to the thermal conductivity of the material.
6.1.1 Joule heating and temperature increase in the nanowire

In the Joule heating, or ohmic heating method, a dc current, $I$, passing through a material of resistance, $R$, releases heat into the material with power $I^2R$. The resistance of the material is related to its charge carrier density which is tunable by electrostatic doping. In the presence of a dc gate voltage, $V_{dc}^g$, the nanowire is doped by an amount $C_g V_{dc}^g$ where $C_g$ is the capacitance between the gate and semiconductor. This modifies the charge transport through the semiconductor since its conductance is directly proportional to the charge carrier density. Figure 6.2(a) shows the gating curve of the InAs nanowire device used in our study. We see that the current through the nanowire saturates at large, positive gate voltages and goes to zero at large, negative gate voltages. This is indicative of an n-type character of the nanowires. The InAs nanowires we use are unintentionally n-type doped during the growth process.

Figure 6.2(b) shows current-voltage response of the nanowire at a fixed gate voltage of $V_g = -20$ V. Here the current is measured through a bias tee on the low temperature rf amplifier that provides an additional series resistance of 20 kΩ. The current passing through the nanowire is seen to be of the order of few microamperes.
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Figure 6.2: (a) Variation with gate voltage of the dc current through the nanowire for an applied source-drain bias voltage of $V_{sd} = -0.3$ V. (b) I-V curve of the nanowire for a fixed gate voltage of $V_{g} = -20$ V. Both the measurements were carried out at 13 K.

The slight asymmetry of the IV response is due to the effective shift in the potential of the nanowire with respect to the gate voltage as $V_{sd}$ is changed. The presence of a dc current leads to Joule heating in the nanowire that causes an increase in temperature. The temperature rise in the material depends on the thermal conductivity of the nanowire.

The thermal conductivity of nanoscale semiconductors is known to be much lower than the bulk material due to the presence of defects and surface scattering. The InAs nanowires used in our study are known to have many structural defects like twin defects that degrades their thermal conductivity. The as-grown nanowires have many structural defects as seen from the high resolution TEM images in Figure 6.3(a)-(b). The defects are seen to increase the surface roughness of the nanowires and are mostly twins and polytypes occurring perpendicular to the growth axis of the nanowire. The increased phonon scattering from these random defects lowers the thermal conductivity of the nanowires as compared to its bulk value.

The low thermal conductivity of the nanowire implies that the heat generated from Joule heating does not escape the system quickly. This causes a considerable amount of heat to accumulate in the nanowire. Here we make the assumption that the clamping points of the suspended nanowire are pinned to the bath temperature through
Chapter 6. InAs nanowire resonators with low tunability (high tension regime)

Figure 6.3: (a)-(b) TEM images of the InAs nanowires showing structural defects including twin defects and polytypes.

the gold electrodes. This assumption is supported by the following observations: (i) the cross sectional area and width of the gold electrodes is much larger than that of the nanowire, (ii) as the as-grown nanowires are approximately 10 \( \mu \text{m} \) long, and as the suspended part of the nanowire is only few micrometers, a considerable portion (> 1 \( \mu \text{m} \)) of the nanowire is mechanically held inside the gold contacts, and (iii) the electrical and thermal conductivities of the metal electrodes are orders of magnitude larger than that of the nanowire. Therefore, for typical currents (see Figure 6.2(b)) passing through the nanowire, temperature rise at the metallic contacts is negligible. Consequently the temperature along the length of the nanowire increases.

To quantify the temperature increase in the suspended nanowire we look at the heat flow through the device. The steady state heat flow along the length \( L \) of a nanowire of cross sectional area \( A \) and thermal conductivity \( \kappa \) is governed by the one dimensional heat equation given by:

\[
A \frac{d}{dx}(\kappa \frac{dT}{dx}) + \dot{w} = 0
\]  

(6.1)

where \( \dot{w} = I^2R/L \) is the rate of Joule heating per unit length of the nanowire. Here we assume the entire electrical power to be conducted along the length of the nanowire. Other contributions that could remove heat from the system are losses due to convection and radiation. The contribution of these mechanisms are shown to be negligible in Section 6.1.2.

As the temperature increase in the nanowire can be large, we have to account for
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Figure 6.4: (a) Temperature profile along the length of the nanowire for a bias of $V_{sd} = -0.4$ V. (b) Maximum temperature in the nanowire shown as a function of the applied bias voltage. The temperature rise in the nanowire can be many times the bath temperature due to low thermal conductivity of the nanowire. Both the plots are for a bath temperature of 13 K.

the temperature dependence of the thermal conductivity. The temperature dependent thermal conductivity, $\kappa$, is assumed to have the temperature dependent form $\kappa = bT$ in the temperature range 10-60 K, where $b$ is a parameter that will be determined from resonance measurements of the suspended device. The thermal properties, including the thermal conductivity, of individual nanowires are known to have significant device-to-device variation\[134\]. Previous thermal conductivity measurements on silicon and InAs nanowires have also shown near linear temperature dependence of thermal conductivity at cryogenic temperatures\[131, 134, 133\]. This supports the empirical form of the thermal conductivity used here.

Using the temperature dependent form of $\kappa$, equation 6.1 can be solved analytically to obtain the temperature profile along the nanowire. The temperature profile along the nanowire can be then written as:

$$T(x) = \sqrt{\frac{AbT_b^2 + L\dot{w}x - \dot{w}x^2}{Ab}}$$  \hspace{1cm} (6.2)

where $x$ takes values between 0 and L. Here we have assumed thermal equilibrium of the clamping points to the bath temperature, $T_b$, using the boundary conditions $T(x) = T_b$ for $x = 0, L$.

Although the analytic model provides physical insight on the temperature increase
in the nanowire, we also performed detailed finite element method (FEM) simulations for a more comprehensive understanding of the heating in our device. This will be discussed in Section 6.1.5. Figure 6.4(a) shows the temperature profile along the nanowire for an applied bias voltage of $-400$ mV at a bath temperature of $T_b = 13$ K. Here the value of $b$ used is $0.035$ W/mK. The procedure for obtaining $b$ involves studying the mechanical frequencies of the suspended nanowire as it is Joule heated and is discussed in Sections 6.1.4 and 6.1.5. From Figure 6.4(b) we see that the temperature in the nanowire is many times the bath temperature. As the value of $\kappa$ is low, few microamperes of current can drastically increase the temperature in the nanowire and change the mechanical stress state of the device. This presents an inherent limit to carrying out electrical measurements on suspended devices of materials with low thermal conductivity at cryogenic temperatures.

We now discuss the dominant contribution of conductive heat transfer in the nanowire device.

6.1.2 Contribution of other heat loss mechanisms

In the preceding discussion we assumed that the entire electric power was conducted as heat through the nanowire. Other contributions that could remove heat from the system are convective and radiative heat losses. Here we discuss how we minimize the effects of convective heat loss to the environment, and the negligible contribution of radiation loss from the nanoscale structure.

Heat loss through convection occurs due to the transfer of energy to the fluidic environment surrounding the nanowire. Here the gas pressure around the device becomes important. A complete theory of convective heat loss of a nanoscale structure to its fluidic surroundings does not exist. However, convective losses are known to be significant only at sufficiently high pressures where the gaseous environment is in a fluidic regime [129, 135]. Thus effects of convection can be eliminated by doing the measurements at high vacuum. To reduce convective heat transfer the device is pumped out to roughly $10^{-3}$ mbar prior to cooling down. As the device is cooled in the cryostat, pressure near the device further lowers due to the condensation of any remaining gas molecules. Cryo-pumping at low temperatures lowers the pressure to
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high vacuum (typically \( \sim 10^{-7} \) mbar) and hence we minimize convective heat loss. Although an accurate description of heat exchange between gases and nanoscale surfaces is missing, the contribution of heat loss to air has been shown to be less than 10% in similar nanowire systems for pressures \(<1\) mbar even at room temperature\[135\]. As the thermal conductivity of most gases is significantly lower at lower temperatures and as our experiments are done at high vacuum we rule out heat loss through convection.

The contribution to radiation heat loss can be calculated using the Stefan-Boltzmann law\[129\]. For a suspended cylindrical rod of length \( L \) and radius \( r \), having emissivity \( \epsilon \), the radiative heat loss per unit length is given by the Stefan-Boltzmann law as \( \dot{w}_R(x) = 2\pi r\epsilon \sigma (T^4(x) - T^4_b) \). Here \( T(x) \) is the temperature along the radiator, \( T_b \) is the bath temperature, and \( \sigma = 5.67 \times 10^{-8} \) W/m\(^2\)K\(^4\) is the Stefan-Boltzmann constant. We note that the total radiative heat loss is proportional to the surface area of the suspended nanowire. We also see that in order to maximize the contribution of radiation loss, the temperature along the nanowire, \( T(x) \), has to be high.

Here we take \( T(x) \) to be of the form given by equation 6.2 and assume that the nanowire is a perfect emitter with \( \epsilon = 1 \). The average radiative loss per unit length of the nanowire is then given by \( \langle \dot{w}_R(x) \rangle = \frac{1}{L} \int_0^L \dot{w}_R(x) dx = \frac{1}{L} \int_0^L 2\pi r\epsilon \sigma (T^4(x) - T^4_b) dx \). At 13 K bath temperature, for a current of 4 \( \mu \)A at bias of 0.5 V we calculate the average radiation loss per unit length to be \( \langle \dot{w}_R(x) \rangle = 2 \times 10^{-7} \) W/m, where we have used the form of the temperature profile, \( T(x) \), given by equation 6.2. The rate of Joule heating per unit length is, however, \( \dot{w} = VI/L = 0.625 \) W/m. Therefore the Joule heating power is roughly 6 orders of magnitude higher than rate of energy loss due to radiation. In general, we note that the radiative heat loss from nanoscale structures at cryogenic temperatures is very low due to the small surface area and \( T^4 \) dependence in the Stefan-Boltzmann law. Hence, we assume conduction to be the only mechanism of heat transfer when we Joule heat the suspended nanowires.
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Figure 6.5: (a) Fundamental mode of the nanowire resonator obtained from FEM simulations using COMSOL (red/blue represents large/small displacement). The nanowire has two perpendicular fundamental modes. The one we study oscillates in the plane of the device. (b) Response of the nanowire resonator measured at $V_{dc} = -20$ V at two different source-drain bias voltages at 13 K.

6.1.3 InAs nanowire resonators for studying thermal conductivity

In the following sections we discuss the method of obtaining the factor $b$ in the expression for thermal conductivity by studying the relation between thermal stress due to Joule heating and mechanical frequency shifts of the suspended nanowire. Prior to that we discuss the electromechanical properties of these resonators.

Figure 6.5(a) shows the fundamental mode shape of vibrations of the nanowire resonator obtained from FEM simulations using COMSOL. The nanowire is assumed to be a beam of circular cross section with fixed end faces. The fundamental mode of the resonator has two perpendicular components. The mode that we consider in this study is the mode that oscillates in the plane of the device. Figure 6.5(b) shows the response of this mode for two applied bias voltages at 13 K bath temperature. We see that both the frequency as well as quality factor of the resonator changes with applied bias voltage. In subsequent sections we try to understand the origin of these frequency shifts and the modified dissipation.

As discussed in Chapter 2, the mechanical frequency is tunable electrostatically using a gate electrode. Figure 6.6(a) shows the frequency shift of the nanowire resonator with applied dc gate voltage. We see that the frequency of the mode shifts by
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Figure 6.6: (a) The frequency dispersion with gate voltage of the fundamental mode of a nanowire resonator measured at 5 K. The negative dispersion is indicative of the built-in tension in the nanowire being high. The colour-scale plot units are in dB. (b) Frequency of the nanowire shown as a function of the bath temperature. The frequency is seen to be higher at low temperatures.

approximately 200 kHz over a gate voltage range of 20 V. At a gate voltage of around 20 V the shift in frequency is almost linear with a slope of 12 kHz/V. We also note that the nature of the dispersion is negative (decreasing frequency with increasing magnitude of gate voltage) throughout the whole range of the measurement.

This is indicative of large built-in tension in the nanowire. As discussed in Chapter 2 the fundamental frequency of an unstressed doubly clamped cylindrical beam of radius \( r \), length \( L \), elastic modulus \( E \), inertia moment \( I = \frac{\pi r^4}{4} \), and density \( \rho \) is given by \( f_0 = \frac{1}{2\pi} \sqrt{\frac{EI\beta^4}{\rho A}} \), where \( \beta = 4.73/L \) is the mode factor for the fundamental mode and \( A = \pi r^2 \) is the cross sectional area. Using the values \( r = 60 \text{ nm}, L = 3.2 \text{ \mu m}, E = 97 \times 10^9 \text{ Pa}, \) and \( \rho = 5670 \text{ kg/m}^3 \) we get \( f_0 = 43 \text{ MHz} \). However, we see that the resonant frequency we observe at low temperatures is \( \sim 59 \text{ MHz} \). This implies that the beam is under tensile stress that causes an increase in the resonant frequency.

This stress arises from fabrication processes and thermal expansion of the substrate, electrodes, and nanowire while cooling to low temperatures. Figure 6.6(b) shows the change in the nanowire frequency as the bath temperature is increased from 5 K to 80 K. We see that the nanowire frequency is higher at lower temperatures of the cryostat. This supports the conjecture that cooling the device also contributes
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to the large tensile stress on the nanowire. The initial stress in the nanowire can be estimated from the frequency dispersion observed with gate voltage in Figure 6.6(a).

We now derive an expression for the electrostatic tunability of resonant frequency of a beam under tension.

**Theoretical model for frequency tunability with gate voltage**

Here we present a theoretical model that describes the gate voltage dependence of resonance frequency of a beam under initial tension. We extend the model by Chen et. al to account for the large flexural rigidity of the beam[119]. Consider a doubly clamped, suspended rod of length \( L \) and radius \( r \) under the influence of an electrostatic force from a gate electrode. Due to the capacitance, \( C_g \), the effect of a gate voltage is to attract the nanowire towards the gate electrode giving it a shape \( \epsilon(x) \) where \( 0 < x < L \).

The elastic energy of the bent tube is given by[136]

\[
U_e[\epsilon(x)] = \int_0^L \frac{dx}{2} \left\{ EI\epsilon''^2(x) + \left[ T_0 + \frac{EA}{2L} \int_0^L \epsilon'^2(x) dx \right] \epsilon'^2(x) \right\}
\] (6.3)

where \( E, I, \) and \( A \) are the elastic modulus, inertia moment (\( = \pi r^4/4 \)), and cross sectional area (\( = \pi r^2 \)) of the rod. Here \( T_0 \) is the residual tension in the beam.

For a voltage \( V_g \) applied on the gate electrode we write down the electrostatic energy as \( U_{es} = -\frac{1}{2} C_g V_g^2 \). To account for the effects of fringing fields we make the approximation that the width of the gate electrode is equal to the suspended nanowire length thus providing a uniform electrostatic load on the beam.

The shape of the bent rod is modeled as a beam under uniform load and is given by[136]

\[
\epsilon(x) = \frac{16}{L^4} \left( L^2 x^2 - 2Lx^3 + x^4 \right)
\] (6.4)

where \( z \) is the center displacement of the beam and the factor \( \frac{16}{L^4} \) is to normalize the displacement to \( z \) at the center of the beam. Then equation 6.3 becomes:

\[
U_e = \frac{65536EA}{11025L^3} z^4 + \left( \frac{512EI}{5L^3} + \frac{256T_0}{105L} \right) z^2
\] (6.5)
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![Figure 6.7: (a) Schematic showing the bending of the nanowire due to the electrostatic force. (b) The red circles show the measured resonant frequency of the nanowire with applied dc gate voltage. The blue line is a fit obtained with initial tension as a parameter using our continuum mechanics model.](image)

To account for the change in capacitance while bending we write $C_g(z) = C_0 + C'_g z + \frac{1}{2} C''_g z^2$ where $C_0$ is the capacitance at zero deflection and $C'_g = dC_g/dz$ and $C''_g = d^2C_g/dz^2$.

Let the equilibrium deflection at the center of the beam be $z_e$ for an applied $V_g$ (see schematic in Figure 6.7(a)). Then at equilibrium we minimize the total energy to get the equation:

$$\frac{262144EA}{11025L^3} z_e^3 + \left(\frac{1024EI}{5L^3} + \frac{512T_0}{105L} - \frac{1}{2} C''_g V_g^2\right) z_e - \frac{1}{2} C'_g V_g^2 = 0 \quad (6.6)$$

This is a cubic equation in $z_e$ that can be solved to find the equilibrium center displacement of the beam. The real solution of equation 6.6 gives the static center deflection of the nanowire for an applied gate voltage.

The resonant frequency of the beam is given by:

$$f = \frac{1}{2\pi} \sqrt{\frac{k}{m_{\text{eff}}}} \quad (6.7)$$

where $k$ is the spring constant. For a doubly clamped beam in its fundamental flexural mode, the effective mass, $m_{\text{eff}} = 0.367m$ where $m$ is the mass of the beam [127].
The spring constant is obtained from the energetics and given by:

\[
k = \frac{\partial^2 (U_e + U_{es})}{\partial z^2} \bigg|_{z=z_e} = 1024EI \frac{1}{5L^3} + \frac{512T_0}{105L} + \frac{262144EA}{3675L^3}z_e^2 - \frac{1}{2} C_g' V_g^2.
\]  

(6.8)

Here, the first two terms are of comparable magnitude when the in-built tension is large compared to \(EI/L^2\) whereas the last two terms coming from the static deflection and gate voltage are an order of magnitude smaller. We use equations (6.7)-(6.8) to fit the experimentally observed frequency dispersion with gate voltage with the initial tension as the parameter. Figure (6.7) shows the measured frequency shift with gate voltage at 5 K and the fitting obtained from the above discussion with \(T_0\) as the fitting parameter where \(T_0/A = 306\) MPa.

We see that the nanowire is initially under a tensile stress of \(\sim 0.3\) GPa at low temperatures. This corresponds to a strain of \(\approx 0.3\%\). This high value of strain is responsible for the low tunability in this device. We now discuss the change in this stress state as the resonator is subjected to a thermal stress.

### 6.1.4 Frequency shift with Joule heating

In previous sections we have shown that the nanowire is heated to many times the bath temperature with Joule heating and we also saw that the built-in stress in the nanowire is high and of the order of 0.3 GPa. When the nanowire is Joule heated, an additional stress builds up in the nanowire due to the presence of a non-zero thermal expansion coefficient \(\alpha\). Here the additional thermal stress acts only on the suspended nanowire, and the substrate and electrodes do not contribute with any relative expansion as we have made the assumption that the clamping points are pinned to the bath temperature. In Chapter [2] we had shown that the resonant frequency of a beam is a function of the tension, therefore any change in stress state causes a shift in its resonant frequency.

Figure (6.8) shows the shift in resonant frequency of the fundamental mode of our nanowire resonator at a bath temperature of 13 K. For comparison we show measurements at room temperature from a different nanowire resonator device in Figure (6.8 b). The measurement at cryogenic temperature shows that at low bias voltages
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Figure 6.8: (a) Frequency shift of the nanowire resonator device with bias voltage at low temperature ($T_b = 13$ K). Colour scale units are in dB. Non-monotonic frequency shift is observed. (b) Room temperature measurement of frequency shift with bias voltage from another nanowire resonator device showing a monotonic, negative frequency shift. The calibrated response from the network analyzer is plotted with the colour scale units in dB. The nature of the frequency shift is related to the sign of thermal expansion coefficient of InAs where the tensile stress reduces and the resonant frequency decreases for a positive expansion coefficient.

The resonator frequency increases and at larger bias voltages the frequency decreases. The room temperature measurement, however, shows a monotonic decrease in the nanowire frequency. This monotonic decrease has been observed in multiple nanowire resonator devices. Previous reports in literature have also shown the decrease in resonant frequency of NEMS devices with Joule heating [137, 138, 139]. This is attributed to the change in stress state arising from the positive thermal expansion coefficient of the materials.

Figure 6.9(a)-(b) further shows the frequency shift with Joule heating at cryogenic temperatures in our device. The observed non-monotonicity in the frequency shift arises from the non-monotonic dependence of linear thermal expansion coefficient of InAs on temperature. InAs is known to have a negative thermal expansion at in the temperature range 10-50 K (shown in Figure 6.9(c)) [140]. This feature is also observed in other materials like silicon that have a lattice of tetrahedrally bonded atoms [141, 142]. Although a complete theory for negative expansion in these materials does not exist, it is considered to arise due to the flexibility of the tetrahedral network [143].
Chapter 6. InAs nanowire resonators with low tunability (high tension regime)

When the nanowire resonator is Joule heated, the temperature in the middle of the nanowire increases. Segments of the resonator, therefore, experience a temperature gradient that causes a thermal stress that depends on the sign of the thermal expansion coefficient. The temperature dependence of the thermal expansion coefficient, therefore, causes the nanowire to expand and contract along its length which causes the non-monotonic frequency shifts observed in our experiments.

We now quantify the observed frequency shifts by studying the nature of the stresses acting on our nanowire resonator. Although we have suggested Joule heating to be the cause of the observed non-monotonic frequency shifts, another mechanism that could lead to such a behaviour is the change in electrostatic environment as we change the bias voltage. In the experiments shown in Figure 6.8(a) and Figure 6.9(a)-(b) the gate voltage is fixed at $V_{dc}^g = -20$ V. However, the potential difference between the gate electrode and the nanowire changes by 1 V when the source-drain bias voltage is changed from -0.5 V to 0.5 V. This could also lead to a shift in resonant frequency since our nanowire resonator is an electromechanical system where the resonant frequency is tunable by a changing gate voltage.

Figure 6.9(d) shows the frequency tunability with gate voltage of our device at 5 K bath temperature. We see that the overall tunability of the resonant frequency with gate voltage is only $\sim 200$ kHz for a change in gate voltage of 20 V. At larger $V_{dc}^g$ the shift in frequency is almost linear with a slope of 12 kHz/V (marked by the black line at -20 V in Figure 6.9(d)). We assume this gate voltage tunability to hold even for $T_b = 16$ K since the resonant frequencies are nearly the same at a gate voltage of -20 V (see Figure 6.9(a),(d)). Therefore, we expect a monotonic, linear shift of 12 kHz in resonant frequency as the bias voltage changes from -0.5 to 0.5 V. This shift due to the electrostatics is marked by the black, dotted line in Figure 6.9(a) and is seen to be small. Hence the non-monotonic response of the nanowire frequency is predominantly from Joule heating.

This tells us why it is desirable to have low tunability for carrying out studies of Joule heating using electromechanical resonators. In the previous chapter we saw graphene drum resonators with tunability of $\sim 1$ MHz/V. Such large tunability causes large frequency shifts when the source-drain bias voltage is changed to pass a current through the device. Therefore, any frequency shift occurring due to heating effects
Figure 6.9: Experimentally observed resonant frequency shifts of the nanowire with $V_{sd}$ at (a) 16 K and (b) 20 K bath temperature. The color scale units are in dB. The dc current through the nanowire gives rise to Joule heating and a thermal stress in the nanowire. The yellow line in (a) is the frequency shift obtained from simulations (see Section 6.1.5). The black dotted line shows the expected trend in frequency solely due to electrostatics as $V_{sd}$ is changed. The green line in (b) shows the additional thermal stress (right axis) induced in the nanowire due to Joule heating and is obtained from simulations (see discussion in Section 6.1.5). (c) Temperature dependence of linear thermal expansion coefficient of InAs obtained from reference [140]. Figure adapted from reference [140]. (d) Tunability of resonant frequency with dc gate voltage for $V_{sd} = -300$ mV at 5 K. The overall change in resonant frequency over 20 V of gate voltage is only $\sim 200$ kHz. The small black line represents the change in electrostatics in the experiments in (a) and (b) when $V_{sd}$ is changed.
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will be masked by the frequency shifts due to electrostatics. Our low tunability device thus allows us to study the thermal properties of the nanowire using Joule heating.

Since the observed frequency shift is closely related to the Joule heating we can use the nanowire as a thermometer to measure its temperature. This allows us to measure the thermal properties of the nanowire by studying the frequency shifts with Joule heating. In order to find the thermal conductivity of the nanowire we performed FEM simulations to relate the observed frequency shifts to a physical temperature of the nanowire. Next we present details on the simulations that tell us how the frequency shifts are related to the physical temperature of the nanowire.

### 6.1.5 Simulated frequency shift with Joule heating

We performed FEM simulations using COMSOL Multiphysics to investigate the effect of Joule heating on the temperature and resonant frequency of the nanowire. The simulations allow us to calculate the temperature increase in the nanowire, and the amount of additional stress in the nanowire due to thermal expansion from Joule heating.

In the COMSOL simulations, the nanowire was modeled as a suspended, cylindrical beam with an initial built-in stress of \( \approx 0.3 \text{ GPa} \). The initial stress was chosen such that the resonant frequency at a particular bath temperature matches the experimentally observed frequency at zero source-drain bias voltage. This tensile stress was specified under the linear elastic material properties of the material. A physics-controlled normal meshing scheme was chosen for the simulations and a fixed constraint was specified for the cylinder faces. The dimensions of the nanowire were obtained from SEM images.

Bulk values of mass density (5670 kg/m\(^3\)), Poisson ratio (0.35), and Young’s modulus (97 GPa) were provided as material parameters [144]. The dc current measured from experiments and the expansion coefficient of InAs was used as input for the simulations and the thermal conductivity of the structure was chosen to be of the form \( \kappa = b \beta T \). One end of the cylindrical beam was fixed at bias voltage, \( V_{sd} \), while the other was grounded, and both were fixed to the bath temperature, \( T_b \). We used the Joule heating and thermal expansion components of COMSOL Multiphysics to...
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calculate the longitudinal stress due to Joule heating. A parametric sweep of the source-drain bias voltage was performed where a stationary study along with eigen-frequency study of the beam was performed at each bias voltage. This gave us the temperature profile, thermal stress and eigenfrequency of the resonator as a function of the source-drain bias voltage.

Figure 6.10: (a) Comparison of maximum temperature in the nanowire obtained analytically and from simulations at 13 K bath temperature. (b) Additional thermal stress due to Joule heating the nanowire at a bath temperature of 13 K. The analytic calculation of the thermal stress is given in Section 6.1.6.

Figure 6.10(a) shows the maximum temperature of the nanowire as a function of $V_{sd}$ obtained from simulations by using $b = 0.035$ W/mK$^2$. It is seen to match well with the analytical form of temperature increase given in equation 6.2.

The eigenfrequency simulations are performed at all bath temperatures and compared with the experimentally observed frequency shifts. Figure 6.9(a) shows the simulated change in resonant frequency of the nanowire with applied $V_{sd}$ at $T_b = 16$ K. Similarly, we carried out simulations for different values of $b$ and the simulated frequency shift with bias voltage was compared with the experimentally observed shift at bath temperatures of 13, 20, and 30 K. At a particular bath temperature, the difference between simulated and experimental frequencies for each bias voltage was analyzed by the least square method. The value of $b$ that minimized this error for all bath temperatures was obtained to be $b = 0.035 \pm 0.010$ W/mK$^2$ for this particular device. The assumption of the temperature dependence of thermal conductivity and the value of $b$ provided above is only valid in the temperature range of $\sim 10$-60 K. In this temperature range, we see that the value of thermal conductivity obtained here
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is much lower than the bulk value and is close to previously reported value of $\kappa$ for InAs nanowires [134, 133].

Therefore, by studying the frequency shifts to controlled Joule heating and by comparing it with simulated results we have been able to measure the thermal conductivity of the nanowire. In the following discussion we present a physical model that captures the physics of frequency shifts arising from Joule heating.

6.1.6 Continuum mechanics model for frequency shift with Joule heating

To get a better understanding of the experiments described in the previous sections we develop a physical model that relates the frequency of the resonator to its physical temperature. From Chapter 2 we saw that for a tensile stress $\tau$ acting on a suspended beam, the natural frequency is given by [75]:

$$ f_0 = \left( \frac{1}{2\pi} \sqrt{\frac{EI\beta^4}{\rho A}} \right) \sqrt{1 + \frac{0.55\tau A}{\beta^2 EI}}. \quad (6.9) $$

In our nanowire resonator, the stress can be written as $\tau = \tau_0 + \Delta\tau_h$ where $\tau_0$ is the initial stress in the nanowire which was shown to be $\approx 0.3$ GPa. The additional thermal stress, $\Delta\tau_h$, accumulates on the nanowire due to relative expansion and contraction when Joule heated.

Consider an infinitesimal segment of the nanowire at position $x, 0 < x < L$ and temperature $T(x)$. As the thermal expansion coefficient is strongly temperature dependent, the stress due to thermal expansion of the segment is given by

$$ \frac{E}{L} \int_{T_h}^{T(x)} \alpha[T(x)]dT. \quad (6.10) $$

We then obtain the total contribution of the thermal stress by integrating the
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stress from the infinitesimal segment over the entire length of the nanowire using:

\[
\Delta \tau_h = -\frac{E}{L} \int_0^L \left[ \int_{T_b}^{T(x)} \alpha [T(x)]dT \right] dx
\]

(6.11)

where \( T_b \) is the bath temperature and \( \alpha \) is the temperature dependent expansion coefficient\[140\]. For \( T(x) \) we use the analytic form derived in equation 6.2. Here the overall negative sign comes from the net strain in the system being zero. Since the nanowire is clamped at its endpoints, there is no overall change in length and, therefore, no net strain.

The analytic form of the thermal stress and eigenfrequency given here matches well with the FEM simulations. Figure 6.10(b) shows the comparison between thermal stress obtained from simulations and analytic form given in equation 6.11. We see that our physical model matches the simulations well, and gives a good description of the mechanics of our system when Joule heated.

6.1.7 Data from second device

We also measured the thermal conductivity of a second nanowire resonator device that was fabricated on a sapphire substrate by performing similar experiments of Joule heating. Figure 6.11 shows the electromechanical response of this device to an applied gate voltage. The frequency of the resonator is seen to be \( \sim 50 \) MHz at low temperatures, which is higher than the value predicted by the equation for frequency of an unstressed beam. For length of 3.5 \( \mu m \) and diameter of 100 nm, the unstressed frequency of the nanowire is \( \approx 30 \) MHz. The higher frequency observed at low temperature is again an indication of the large built-in tension in the nanowire.

The observed frequency shift with gate voltage is analyzed using the theoretical model we have derived in Section 6.1.3. Equation 6.8 is used to fit the experimentally observed dispersion to ascertain the built-in stress in the nanowire. The fitting is shown in Figure 6.11(b) with the parameter of initial stress, \( \tau_0 = 379 \) MPa. This corresponds to a high strain of \( \sim 0.4\% \) in the nanowire. The large strain state is further reflected in the low electromechanical tunability and negative dispersion of the fundamental mode even at large gate voltages as shown in Figure 6.11(a).
The low electromechanical tunability of the device allows us to study the frequency shifts due to Joule heating. We carried out Joule heating experiments on the second nanowire resonator device and Figure 6.12 shows the frequency shift with $V_{sd}$ obtained for this device at cryogenic temperatures. We see a similar trend as that of the first device where the resonant frequency shows a non-monotonic response. The form of thermal conductivity was again assumed to be of the form $\kappa = bT$. We perform simulations as detailed in Section 6.1.5 to relate the observed frequency shift to the temperature profile along the nanowire. By simulations at bath temperatures of 13, 20, and 30 K, and minimizing the difference in frequency shifts to the experimentally observed results we obtain the thermal conductivity parameter to be $b = 0.12 \pm 0.02$ W/mK$^2$ where $\kappa = bT$ with $T$ in units of kelvin and in the range of $10 - 60$ K.

Thus far we have discussed how controlled heating and monitoring the resonant frequency allows us to measure the thermal conductivity of an individual nanostructure. As we had seen earlier, the dissipation, or quality factor of our devices is also modified when the device is Joule heated. Our device geometry further allows us to address the question of how dissipation in the suspended structure is modified when only the temperature of the nanowire is changed.
6.2 Studying dissipation using Joule heating

As discussed in Chapter 2, the energy loss in a resonator can be quantified by the quality factor $Q$ where the loss can be due to various factors such as thermoelastic dissipation, surface losses, viscous damping, and clamping losses\cite{86, 61, 88}. The effect of temperature on the dissipation of mechanical resonators has been previously studied on various systems\cite{145, 61, 62, 68}. Conventionally, temperature dependent studies on dissipation are done by heating the entire sample including the bottom substrate\cite{62}. Our system allows us to address the question of how the losses of a system change if the local temperature of the clamps is varied while the average temperature of the suspended nanowire is held constant. From the Joule heating experiments described above we can find the relation of dissipation to the average temperature of the nanowire at various bath temperatures.

To study the effect of Joule heating on the dissipation of the device, we fit the device response to a Lorentzian with a background following the discussion in Chapter 4 and extract the quality factor. The quality factor is studied as a function of the average temperature of the nanowire. Figure 6.13(a) plots the variation in quality factor of the first device as a function of the applied source-drain bias voltage at a bath temperature of 16 K. The quality factor was obtained from the response shown

Figure 6.12: Data from device 2. Frequency shift with Joule heating at a bath temperature of (a) 13 K and 20 K (b) 20 K along with the respective simulation results shown by the yellow curve in each panel. Both the experiments were carried out at a gate voltage of 40 V.
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in Figure 6.9(a). We see that Q decreases with increasing $|V_{sd}|$.

We had seen that for an applied bias voltage, the temperature of the resonator attains a gradient where the maximum temperature is many times the bath temperature. The dissipation in nanomechanical systems is known to increase with increasing temperature due to effects like defect activation. This explains the decrease in Q factor seen in Figure 6.13(a). To quantify the temperature of the nanowire we define the average temperature of the nanowire as

$$T_{nw} = \frac{1}{L} \int_{0}^{L} T(x)dx$$  \hspace{1cm} (6.12)

where $T(x)$ is the temperature profile attained at each bias voltage. The average nanowire temperature, $T_{nw}$, can also be obtained from simulations. To further elucidate the role of Joule heating on the dissipation in our system we study the experimentally observed loss, $(1/Q)$, as a function of $T_{nw}$ at different bath temperatures.

Figure 6.13(b) shows the variation of loss in the nanowire as a function of the average temperature of the nanowire at different bath temperatures. Here the average nanowire temperature was obtained from simulations and is also well described by equation 6.12. We see that the loss increases with increasing average temperature of the nanowire ($T_{nw}$). The variation of $1/Q$ with $T_{nw}$ is seen to follow a power law dependence ($T_{nw}^{0.1}$) at lower bath temperatures. Similar power law temperature dependence has been observed in systems of GaAs, Si, and diamond[61, 62]. Although the origin of this complex temperature dependence is not fully understood, theory suggests the role of defects such as impurities and grain-boundaries in the nanomechanical resonator[63]. In addition, we observe that the loss at 13 K bath temperature is always lower than the loss at 30 K even when the average temperature of the nanowire is the same.

We find similar results when studying the losses in the second device. Figure 6.14 shows the variation of loss with average nanowire temperature in the second device. We again see that the loss follows a power law dependence on the nanowire temperature and the loss is always seen to be lower at lower bath temperatures. The
6.2. Studying dissipation using Joule heating

Figure 6.13: (a) Measured quality factor of the resonator as a function of applied bias voltage at 16 K bath temperature. The quality factor reduces due to increase of the average nanowire temperature with Joule heating. (b) Inverse of the resonator quality factor (loss) is plotted as a function of the average nanowire temperature at various bath temperatures (log-log plot). The trend is seen to be same at lower bath temperatures with the exception of a positive offset (dotted lines) with increasing bath temperature. The black line is a plot of the power law relation $T_{nw}^{0.1}$. The average nanowire temperature is obtained from simulations.
observed offset in dissipation is possibly due to loss at the clamping points and is discussed next.

6.2.1 Discussion on role of clamping points in dissipation

From the previous discussion we saw how the dissipation in our nanowire resonators depends strongly on the bath temperature even when the average nanowire temperature was the same. Our experiments suggest the role of a potential loss mechanism in these resonators relating to the loss at the clamping points. From the offset between the loss at various bath temperatures seen in Figure 6.13(b) and Figure 6.14 it is clear that the dissipation in our resonator is determined by the bath temperature with a dissipation less than $10^{-4}$.

This could be an indication of losses occurring at the clamping points since the only part of the nanowire resonator at equilibrium with the bath temperature (when Joule heated) are the metal clamps. As discussed in Chapter 2, clamping losses in NEMS arise when part of the vibrational energy is dissipated as an acoustic wave into the support structures including the substrate. Previous studies on clamping losses
6.2. Studying dissipation using Joule heating

have focussed on single crystal NEMS structures where the supports and the resonator are all fabricated from the same material\[79\]. In such systems, the clamping losses arise purely from the geometry of the device and is considered to be temperature independent. In our devices the vibrating material is different from the clamping material, and we believe an additional loss mechanism contributes due to the metallic support structures. As the vibrating nanowire excites acoustic waves in the metallic contacts, we expect the temperature dependent loss of gold to play a role in the observed dissipation.

Further, clamping losses are known to be the dominant mechanism for dissipation in doubly clamped beams with a large built-in stress\[78\]. This further supports our observation that there is significant loss at the clamps since our nanowire devices are under high tensile stress. Here a large stress is quantified by the tension being much larger than the flexural rigidity of the resonator such that the beam mimics the mechanics of a stretched string. This condition is satisfied when \( \tau_0 \gg \frac{EI}{AL^2} \).

We had shown that the built-in stress in the first device was approximately 306 MPa at low temperatures. This is significantly larger than \( \frac{EI}{AL^2} \approx 9 \text{ MPa} \). Similarly, the built-in stress of the second device was approximately 379 MPa at low temperatures, much larger than \( \frac{EI}{AL^2} \approx 5 \text{ MPa} \).

This supports the significance of clamping losses in our devices at low temperatures. The temperature dependent offset observed in the dissipation could be due to the losses arising from the gold clamps. Previous studies of internal friction in thin films of gold have shown temperature dependent losses of the order of \( 10^{-4} \) at low temperatures\[145\]. This matches well with the observed offset in dissipation seen in our devices.

Other possible candidates for the observed dissipation, like internal defects described in Chapter 2, are ruled out since the average temperature of our resonant structures is held the same\[61\]. The role of viscous damping in the observed offset at different temperatures is also ruled out as the sample is efficiently cryopumped to low pressures (less than 0.01 Pa) in the temperature range of our experiments\[78\]. The effect of viscous damping for gas pressures below 0.01 Pa is known to be negligible \[61\]. Furthermore, the low quality factor seen in our system rules out the role of thermoelastic dissipation\[78\], since thermoelastic dissipation in systems of similar
dimensions is known to be orders of magnitude lower than the dissipation we observe of $2 - 5 \times 10^{-4}$.[86]

One factor that could change the dissipation with bath temperature is the change in residual stress itself since it is known that the built-in stress can be used to tune the quality factor in NEMS systems.[146, 78] However, the change in $\tau_0$ in both the devices at different bath temperatures is less than 2%, as seen from the change in frequency at zero source-drain voltage, whereas the Q factor change is of the order of 30% (see Figure 6.13(b)).

Our argument for a different type of clamping loss mechanism provided above, can be proved by further studies on resonators that use various clamping metals with differing internal friction. Our work also suggests the possibility of increasing the quality factor in similar NEMS devices by using low loss metals at the support structures. This concludes the sections discussing the measurements involving Joule heating on individual nanostructures. We have shown how the thermal stress acting on an electromechanical resonator can be used to measure the thermal conductivity and dissipation of the system. In the following section we discuss ongoing experiments on coupling between the perpendicular flexural modes of the nanowire resonators.

6.3 Mode coupling experiments

As mentioned earlier, the fundamental flexural mode of the nanowire resonator devices consists of two perpendicular components: an in-plane mode and an out-of-plane mode. In a perfectly symmetric, unstressed beam these two modes are degenerate. However, in our nanowire devices we see that this degeneracy is lifted and the two perpendicular flexural modes are no longer at the same frequency. The lifting of the degeneracy can occur do to structural asymmetry, residual tension, or mass loading on the nanowire. Here we discuss some of the ongoing work on coupling between these perpendicular modes of a nanowire resonator.

The experiments discussed here were performed at 5 K. Figure 6.15(a) shows the response of the two modes at a gate voltage of -20 V. The images next to the resonance peaks show FEM simulations for the mode shapes. Here we refer to the
Figure 6.15: (a) Response of the nanowire resonator to a harmonic probe signal at a fixed gate voltage of -20 V. Two modes of the resonator are seen with a separation of \( \sim 1.4 \) MHz. (b) Dispersion of the two modes with the dc gate voltage. The nature of the dispersion and resonant response can be used to identify the direction of each mode. The calibrated response of the device is plotted with the colour-scale units in dB. Dotted lines show the dispersion of the modes over the whole range of the gate voltage and are guides to the eye.
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![Graph showing Duffing behaviour of two modes](image)

Figure 6.16: Duffing behaviour of the two modes is seen as a vertical jump in the response when a forward sweep of the frequency is carried out at a large driving voltage of $\sim 0.3$ V. Here $V_{dc}^g = -20$ V.

lower/higher frequency mode as mode 1/mode 2. The direction of the vibrational mode is ascertained from the gate voltage response of the device. Figure 6.15(b) shows the dispersion of the resonant modes with the dc gate voltage. Here both the modes are of the fundamental mode shape of the nanowire. Mode 1 shows larger response and negative dispersion with gate voltage. As the negative dispersion is understood to be of capacitive origin, we reason that mode 1 is affected by the gate capacitance and, hence, oscillates in the plane of the substrate (the in-plane mode). Mode 2 is, therefore, the out-of-plane vibrations of the nanowire and is seen to have a small dispersion with gate voltage.

In Chapter 2, we had seen that the response of an oscillator displays non-linear effects when driven at large amplitudes. We see such non-linear effects in our nanowire resonator device as well. Figure 6.16 shows the response of the two modes when driven by a large $rf$ signal. Here the frequency is swept in the forward direction. The leaning of the resonant frequency and subsequent, sudden jump in the response is characteristic of the Duffing response in these devices. This arises due to the presence of terms like $\alpha x^3$ in the equation of motion. The direction of the frequency leaning observed in our resonator suggests that $\alpha$ is a positive quantity.

Using the expression for amplitude of a driven damped harmonic oscillator given in Chapter 4, we find the amplitude of our resonator at large driving voltages to be $\sim 10$ nm. Here the parameters used are $f = 59.5$ MHz, $Q = 3100$, $V_{dc}^g = -20$ V,
6.3. Mode coupling experiments

\( V_g = 0.3 \) V. The nanowire has a length of 3.2 \( \mu \)m, radius of 60 nm, and has a gate separation of 300 nm. We see that the amplitude of oscillation of \( \sim 10 \) nm is large and comparable to the physical dimensions of the nanowire.

From the equation for the elastic energy of the beam (equation 6.3) we had seen that the total tension in a beam depends on the residual tension, as well as the tension developed due to static deformation of the beam. The deformation induced tension for a suspended resonator also becomes important when the amplitude of oscillations is comparable to the physical dimensions of the system. The deformation induced tension can be written as

\[
\frac{EA}{2L} \int_0^L \left( \frac{\partial \epsilon}{\partial x} \right)^2 dx.
\] (6.13)

where \( E \) is the Young’s modulus, \( A \) and \( L \) are the cross sectional area and length of the beam respectively, and \( \epsilon \) is the flexural mode shape. Since our nanowire resonator has two perpendicular flexural modes, the mode shape can be written in terms of the in-plane, \( u(x) \), and out-of-plane, \( v(x) \), components. Then the governing differential equations of motion for each mode shape will include a term of the form

\[
\left[ \frac{EA}{2L} \int_0^L \left( \left( \frac{\partial u(x)}{\partial x} \right)^2 + \left( \frac{\partial v(x)}{\partial x} \right)^2 \right) dx \right] \frac{\partial^2 w}{\partial x^2}.
\] (6.14)

where \( w \) is either \( u(x) \) or \( v(x) \).

This naturally gives rise to a coupling between the two modes of the resonator. For large amplitude of motion of a particular mode, the deformation induced tension can modify the spring constant of another mode in the resonator, thereby coupling them. To study this in our system, we perform experiments where the first mode is harmonically probed in the linear regime while the second mode is pumped to large oscillation amplitudes.

The response of mode 1 is probed with a small driving signal while the frequency of a large, pump signal near mode 2 is swept in the forward direction. Figure 6.17 shows the response of mode 1 at two powers of the pump signal where the blue coloured regions represent the resonance of mode 1. In the left panel of Figure 6.17, we see...
Figure 6.17: Experimentally obtained response of mode 1 is plotted as a function of the probe and pump signals. The pump signal power is (left panel) 0 dBm and (right panel) 10 dBm where the frequency is swept forward in both cases. The colour scale On/Off represents mode 1 being on/off resonance. Mode 1 is driven with -30 dBm power.

mode 1 starting to deviate from its frequency when the pump frequency is in the vicinity of mode 2. At larger pump powers (right panel of Figure 6.17), we see that mode 1 frequency increases and shifts by many linewidths over a larger frequency range of the pump signal around mode 2.

This represents the modification of the spring constant of mode 1 by the large oscillation amplitude of mode 2. As mode 2 is driven with a larger force, it enters into a Duffing regime and increases the tension in the system by deforming the nanowire. The bistable nature of the Duffing response of mode 2 is clearly seen on the response of mode 1 as a sudden jump in resonant frequency. This shows that mode 1 and mode 2 are coupled via the deformation induced tension, where the coupling is tunable via the oscillation amplitude of a mode.

The effect of Duffing behavior on the coupling between the modes is further seen by studying the response of mode 1 to the sweep direction of the pump frequency. Hysteretic response is a common feature of Duffing behaviour in mechanical systems when the sweep direction of frequency is changed. This is reflected in the mode coupling as a hysteresis in the spring constant tuning of mode 1 when the sweep direction of pump signal is reversed (see the panels in Figure 6.18).

We write down a simplified form of the equations of motion for the coupled modes
using their modal amplitudes, $u_a$ and $v_a$, as

\begin{align}
\ddot{u}_a + \gamma \dot{u}_a + \omega_a^2 u + \alpha u_a^3 + \beta v_a^2 u &= f_u(t) \\
\ddot{v}_a + \gamma \dot{v}_a + \omega_v^2 v + \alpha v_a^3 + \beta u_a^2 v &= f_v(t).
\end{align}

(6.15)  (6.16)

Here the terms $\alpha u_a^3$ and $\alpha v_a^3$ represent the Duffing character of the modes and are responsible for the hysteretic behaviour. Forces of the form $\beta v_a^2 u_a$ and $\beta u_a^2 v_a$ naturally arise from the deformation induced tension in the beam. These terms capture the mode-mode coupling where the factor $\beta$ is related to the mode shape functions. Preliminary investigations have shown the numerical solutions of these equations to have a response similar to the experimentally observed results. Figure 6.19 shows the numerical solutions of the above equations by setting $\alpha = \beta = 1.95 \times 10^{34}$ kg m$^{-2}$s$^{-2}$ with mass of unity. Further investigation is necessary to fully quantify the nature of mode-mode coupling in our electromechanical resonator.
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6.4 Summary

To recap, in this chapter we discussed experiments on doubly clamped, InAs nanowire resonators. The main result that was presented involved studying the thermal properties of the nanowire using electromechanics in the high tension regime. We presented a model that captures the frequency shift of the device with gate voltage to estimate the large built-in stress in the nanowires.

The low tunability of these devices, due to the large built-in stress, allowed us to carry out Joule heating studies. The thermal stresses that were generated in the nanowire due to Joule heating, translated into frequency shifts of the resonator. This, along with simulations, were used to find the temperature increase in the device and measure the thermal conductivity of individual nanowires. Further, our experiments allowed us to study the dissipation in the device at various bath temperatures. We found evidence for a damping mechanism arising from the metallic clamping points that showed a lower dissipation at lower temperatures.

Finally, we presented results from ongoing work on coupling between two perpendicular flexural modes of the resonator. The effect of large oscillation amplitudes on the spring constant of a mode was explored. We saw that the large oscillation amplitude of a mode increases the tension in the system due to structural deformation and increased the frequency of the second, coupled mode. The Duffing response of one mode was also seen to directly influence the frequency of the second mode.
Chapter 7

Other projects

Until now we have discussed the main experiments of mode coupling and Joule heating in electromechanical systems using graphene and InAs nanowires. In this chapter we discuss some of the other experiments that were done during the course of this work. We now discuss the photocurrent studies done on WS$_2$ nanotube devices. The work described here was done in collaboration with Mr. Gobinath Jegannathan and published in Applied Physics Letters 105, 22 (2014). Other persons who contributed to this work are Sameer Grover, Pratiksha Dongare, Rudheer Bapat, Bhagyashree Chalke and SC Purandare.

7.1 Photocurrent studies on WS$_2$ nanotube hybrid devices

The superior optical and electronic properties of graphene and transition metal dichalcogenides (TMDCs) have fueled research on heterostructure devices where different materials are combined for the best optoelectronic response. TMDCs are layered materials, similar to graphite, where subsequent layers are held together by the van der Waals’ interaction. They are known to have good photoresponse due to their semiconducting nature and presence of van Hove singularities in the electronic density of states. This, combined with the exceptional mobility of
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Figure 7.1: TEM image of a WS\textsubscript{2} nanotube with capped (left) and open (right) ends. Some of the nanotubes have a central hollow region (shown by the double sided arrow). The TEM imaging was done by Rudheer Bapat, Bhagyashree Chalke, and SC Purandare.

graphene, has led to various heterostructure devices that show good optoelectronic response\cite{150,152,153,154,155,156}. These devices have incorporated graphene with thin films of a TMDC to build devices with good efficiency for photocurrent generation.

Similar to allotropes of carbon, TMDCs also form tubular and cage like structures\cite{157,158}. Therefore, an alternative for optoelectronic applications is to use nanotubes of TMDCs. The use of nanotubes is advantageous since they can have enhanced light absorption by forming optical cavities\cite{159,160}. Silicon and carbon nanotubes have been shown to be promising materials for solar-cell applications\cite{161,162}. Similarly, TMDC nanotubes could facilitate large scale integration of on-chip optoelectronic elements.

In the following sections we discuss experiments on multi-walled WS\textsubscript{2} nanotubes\cite{157,163} bought from NanoMaterials Ltd. (http://www.apnano.com/). These nanotubes are $\sim 10$ $\mu$m in length and $\sim 100$ nm in diameter and have capped, or uncapped ends\cite{157}. Figure 7.1 shows TEM images of the nanotubes. The multi-walled nature of the nanotubes is clearly seen.

The nanotubes are further characterized by Raman spectroscopy (using a confocal Raman imaging system from WITec). The nanotubes were dispersed on an Si/SiO\textsubscript{2} chip and a 532 nm wavelength laser was used for excitation. Figure 7.2(a) shows
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Figure 7.2: (a) Raman spectrum obtained from the middle of an individual WS$_2$ nanotube using 532 nm laser. Characteristic Raman peak of silicon substrate is seen near 520 cm$^{-1}$. Peaks marked with a star and circle are characteristic Raman peaks of WS$_2$ nanotube. (b) A spatial map of Raman intensity around the 351 cm$^{-1}$ (star) and 417 cm$^{-1}$ (circle) peaks clearly shows the location of the nanotube. The scan area is a square of length 14.7 µm.

the Raman spectrum from the middle of a nanotube. We see two Raman peaks of the WS$_2$ nanotubes near 351 cm$^{-1}$ and 417 cm$^{-1}$ that closely resemble the $E_{2g}$ and $A_{1g}$ modes[160, 164] seen in bulk crystals and nanoparticles of WS$_2$. In the bulk crystals, these correspond to the out-of-plane and in-plane vibrations of the sulfur atoms respectively. We further generate a spatial map of the Raman intensity (shown in Figure 7.2(b)) by using the WITec system. The spatial maps show some non-uniformity along the length of the nanotube possibly due to the presence of structural defects like cracks. These are further probed by scanning photocurrent microscopy experiments in Section 7.1.2.

In the following sections, we discuss the photoresponse of WS$_2$ nanotube devices in field-effect transistor (FET) geometry with gold electrodes, and the photoresponse properties of hybrid devices of WS$_2$ nanotubes with graphene contacts.

7.1.1 Photoresponse of individual WS$_2$ nanotubes

Here we discuss the results of photocurrent measurements done on WS$_2$ nanotube devices contacted by gold electrodes. To study the photoresponse we fabricated two probe devices of these nanotubes. The nanotubes were drop coated on a 300 nm
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Figure 7.3: (a) SEM image of a WS$_2$ nanotube (NT) device contacted with gold electrodes. (Inset) Schematic diagram of the device in field effect transistor geometry. (b) I-V characteristics of a WS$_2$ nanotube device measured in ambient conditions at 300 K while applying continuous illumination from a 532 nm laser over the entire device area (laser power = 10 $\mu$W, gate voltage = 0 V).

SiO$_2$/Si$^{++}$ chip and standard electron beam lithography techniques were used for device fabrication. Gold was sputtered to form metal contacts on the nanotubes. Figure 7.3(a) shows an SEM image of a device along with a schematic for the FET geometry.

We performed experiments where the current through the device was measured in response to constant illumination from a laser source. Figure 7.3(b) shows the I-V response of a WS$_2$ nanotube device measured under ambient conditions with and without laser illumination. We see that the device shows non-linear current voltage characteristics. This is due to the formation of Schottky-barrier contacts originating from the band offsets of WS$_2$ and gold[165]. The current through the device at a particular bias voltage was seen to be higher when illuminated by a 532 nm laser. Photon absorption in the semiconductor leads to generation of charge carriers. The excess current in the presence of light is the photocurrent and is seen to be $\sim$ 30 nA in the device for an applied bias of 1 V.

To measure the current generated purely due to the photoresponse of the nanotubes we employed a lock-in technique. The laser was modulated using an acoustooptic modulator, and the photocurrent was measured using a lock-in amplifier locked to the modulation frequency. Figure 7.4(a) shows the response of a nanotube device
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Figure 7.4: (a) Photocurrent response of a nanotube device plotted as a function of incident power for two laser wavelengths. These measurements were performed in ambient with a bias and gate voltage of 0 V. The modulated laser spot was held near the metal contact to the nanotube. (b) Photocurrent response of a nanotube device plotted as a function of modulation frequency of the laser using 532 nm laser and bias voltage of -1 V. The dark current was $\sim 25$ pA. The lines are only a visual guide.

We see that the photocurrent increases linearly with the incident laser power. This is because the number of photocarriers generated per unit area is proportional to the absorbed optical power\cite{166}. Figure 7.4(b) further shows the dependence of the photocurrent on the modulation frequency of the laser. We see that the photoresponse is strongly diminished for a modulation frequency higher than $\sim 3$ kHz, which gives a response time of approximately 0.1 ms in our device, comparable to the projected value for WS$_2$ nanotubes\cite{167}.

We further use scanning photocurrent microscopy\cite{168, 169, 170, 171} to understand the spatial nature of photocurrent generation in our devices. Figure 7.5 shows a schematic of the setup used for these measurements. The device was loaded on a scanning piezo stage and the photocurrent was measured at various points of the device to create a map of the photoresponse. During such scans it is important to know the actual location of photocarrier generation to avoid errors due to piezo drifts.

The light reflected from the device was simultaneously measured using a photodetector connected to a second lock-in amplifier (see schematic in Figure 7.5). The reflected light is more intense when the beam falls on the metal contacts. Therefore, the reflection measurement can be used to identify the regions in the photocurrent maps.
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Figure 7.5: Image shows a schematic of the setup used for scanning photocurrent spectroscopy measurements.

Figure 7.6: Photocurrent response of a WS$_2$ nanotube measured in ambient as a function of the source-drain bias voltage with $V_g = 0$ V at 300 K. Laser of 532 nm wavelength at 1 $\mu$W of power was used. The applied bias shifts the band bending resulting in a reversal of the photocurrent. The inset in each image illustrates tilting of the bands caused by the applied bias voltage. The arrows denote the direction of current flow in the device.
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Figure 7.7: Photocurrent response of the WS$_2$ nanotube measured in Figure 7.6 at $V_{sd} = 0$ V.

Figure 7.6 presents the photocurrent maps of a WS$_2$ nanotube device as a function of the applied source-drain bias voltage. The electrode positions are outlined using an overlaid map of the reflected signal. For $V_{sd} = 0$ V (middle panel), we see that the photocurrent is localized near the nanotube-metal contact regions. This is known to be an effect of electric field present at the contact regions due to band bending\cite{156, 172, 173, 169}. Earlier we had shown the nature of electrical contact with the nanotube to have a Schottky character such that band bending at the metal-semiconductor interface led to barrier formation. The band bending gives rise to an electric field that accelerates the photogenerated charge carriers which is measured as a photocurrent. The photocurrent profile along the length of the nanotube can be extracted from these maps. This is shown in Figure 7.7. The photocurrent is seen to decay into the nanotube away from the Schottky-barrier regions because of carrier diffusion in the nanotube.

An exponential decay function can be fitted to the photocurrent profile to extract information about the minority carrier diffusion length, $L_D$, in the nanotube\cite{172, 174}. The photocurrent profile inside the nanotube is extracted and fit to an equation of the form $ae^{(-x-x_0)/L_d}$, where $a$ is the current amplitude, $x_0$ is the position offset and $L_d$ is the minority carrier diffusion length. The diffusion length was found to be $316 \pm 34$ nm from the fit. The diffusion length is related to the minority charge carrier lifetime, $\tau$, and mobility, $\mu$, by the relation $L_D = \sqrt{k_B T e/\mu \tau}$, where $k_B$ is the Boltzmann constant, $T$ is the temperature and $e$ is the electronic charge. Using the value of hole mobility in
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WS$_2$ reported in literature[175], we find the carrier lifetime to be $\tau = 0.6$ ns at room temperature. This value is comparable to the carrier lifetimes reported in literature for other TMDC systems[176, 177, 166].

From Figure 7.6 we also see that the photoresponse exhibits a shift in the photocurrent toward one of the electrodes with changing sign of the source-drain bias voltage. The applied bias voltage changes the Schottky-barrier heights of the electrodes asymmetrically, evoking a photoresponse from either end of the nanotube[178, 179]. This is schematically represented by the inset in each panel of Figure 7.6. We also generated photocurrent maps as a function of the gate voltage. However, the photoresponse was seen to be unaffected by the gate voltage.

In this section we have discussed the photoresponse of WS$_2$ nanotubes contacted by gold electrodes. In the following section we discuss experiments where graphene was used to make electrical contact to the WS$_2$ nanotubes.

7.1.2 Photoresponse of WS$_2$ nanotubes contacted by graphene electrodes

Here we discuss experiments on the second device geometry explored in our work where the nanotubes were contacted by graphene electrodes. One of the motivations for using graphene electrodes was to be able to modulate the density of carriers in the graphene and modify the Schottky barrier height[180]. The other motivation was to study the spatial homogeneity of the observed photoresponse. We used these devices to understand the role of defects in modifying the optoelectronic properties of the nanotubes[181].

The fabrication of these devices involves electron beam lithography to fabricate a two probe device out of exfoliated graphene, followed by selective oxygen plasma etching to remove the middle portion of the graphene device leaving behind a slit. A dry transfer process is used to place a WS$_2$ nanotube across the slit in the graphene. Figure 7.8 shows a schematic of the fabrication process. An optical image of a WS$_2$ nanotube device with graphene electrodes is shown in Figure 7.9(a). The I-V characteristic of the device presented in Figure 7.9(b) shows that the electrical behavior is
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Figure 7.8: Schematic shows the fabrication steps for the slit devices.

non-linear and similar to that of a two probe WS$_2$ nanotube device\cite{182} with metal contacts.

Figure 7.9(c) shows a reflection map of the hybrid device obtained using the measurement setup shown in Figure 7.5. The reflected light provides information regarding the absorption of light by the material\cite{183, 184}. Assuming a thin film response, the fraction of light absorption by the nanotube can be calculated to be $(1 - e^{-\alpha d}) \sim 63\%$ where the thickness is approximated by the diameter of the nanotube $d = 100$ nm. Here $\alpha$ is the absorption coefficient\cite{185} equal to $10^5$ cm$^{-1}$. Comparing the reflection from the substrate and the nanotube in the reflection map we estimate the relative absorption as $(R_s - R_{NT})/R_s$ where $R_s$ and $R_{NT}$ are the measured reflection from the 300 nm SiO$_2$/Si substrate and the WS$_2$ nanotube respectively. The absorption in our nanotubes is estimated to be $\sim 52 - 58\%$. This is in good comparison to the value of absorption calculated from the thin film response.

The external quantum efficiency, $\eta$, gives the fraction of charge carriers generated for every photon of a given energy absorbed by the material. It is calculated as $\eta = \frac{heP\lambda}{eP\lambda}$ where $e$ is the electronic charge, $h$ is Planck’s constant, $c$ is the speed of light, $P$ is the incident laser power, $\lambda$ is the wavelength used, and $I$ is the photocurrent generated. As the laser power is spread over the area of the beam spot ($\sim 1$ $\mu$m
diameter), only the region of the laser spot overlapping with the nanotube contributes to photocarrier generation. We calculate the efficiency by multiplying with a factor accounting for the area overlap of a 1 µm beam spot with a 100 nm diameter nanotube $(\frac{\text{spot area}}{\text{nanotube area inside spot}} = \frac{\pi \times 0.5 \mu m \times 0.5 \mu m}{1 \mu m \times 100 n m} = 7.9)$. For measurements with $\lambda = 532$ nm, $P = 1$ µW, and $I = 600$ pA, the calculated efficiency for a hybrid device is $\sim 1\%$ with a corresponding responsivity of $\sim 4.7$ mA/W.

To study the effect of an electric field we performed experiments on these hybrid devices by generating photocurrent maps at different gate voltages. Figure 7.10(a) shows the photoresponse of another graphene contacted WS$_2$ nanotube device as a function of the gate voltage with 0 V bias voltage. We observe an inhomogeneous photoresponse along the length of the nanotube at zero bias voltage. At 0 V $V_g$ we see that photocurrent is generated near the slit region[151] as well as at the nanotube ends. Photocurrent generation near the nanotube ends hints at the presence of band bending, possibly due to the capped end seen from the TEM images.

Defects in the nanotube could also modify the local electronic structure of the nanotube, and so we expect photocurrent generation where there is additional band bending along the length of the nanotube. This is seen as the appearance of an additional photocurrent spot with increasing gate voltage[186]. Figure 7.10(b) shows the photocurrent along the length of the nanotube obtained from the maps in Fig-

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**Figure 7.9:** (a) Optical image of a WS$_2$ nanotube contacted by graphene electrodes. The slit gap is 1 µm. Scale bar is 2 µm. (b) I-V characteristics of the device depicted in (a) measured under ambient conditions at 300 K using continuous illumination from a 532 nm laser over the entire device area (laser power = 10 µW, gate voltage = 0 V.) (c) Reflection map of the device in (a) shows the absorption by the nanotube. Color-scale is the measured reflection in arbitrary units. Scale bar is 3 µm.
7.1. **Photocurrent studies on WS\textsubscript{2} nanotube hybrid devices**

Figure 7.10: (a) Reflection (top left) and photocurrent maps of a second slit device (slit gap = 500 nm) obtained at various gate voltages in ambient conditions at 300 K ($V_{sd} = 0$ V, laser: 532 nm at 1 $\mu$W power). The photoresponse is observed only from regions where the nanotube overlaps with the graphene electrode. (b) Photocurrent along the line joining points X and O obtained from (a). The dotted circle indicates the defect region tuned by the gate voltage. (c) Illustration of the local band bending caused by the presence of a defect region on the nanotube. Two segments (red and blue circles) along a single defect region are drawn to illustrate the change in interfacial electric field. Thus, a gate tunable region of the interfacial electric field is created between the nanotube and the graphene contact.
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Figure 7.11: (a) Optical image of the device presented in Figure 7.11. The locations of normal photocurrent (A) and a defect site (B) are marked in the image. (b) Raman spectrum at the site marked A (red curve) and the site marked B (blue curve) in the image on the left for laser excitation at 532 nm. The spectrum at site B exhibits a shift in the characteristic Raman peak of the WS₂ nanotube, possibly because of the formation of a crack acting as a defect site.

In our experiments, photocarriers generated in the WS₂ nanotubes are extracted by the graphene layer just below the nanotube. The interfacial electric field between a defect in the WS₂ nanotube and the graphene results in the transfer of charge carriers to the graphene layer. The variation of the interfacial electric field across the span of the defect then causes the photocurrent to show a local peak. However, there was no change in the sign of the photocurrent in our experiments because the sign of the interfacial electric field between the WS₂ and the graphene did not change. Similar spots of large photoresponse are also seen in literature from an analogous device structure using thin films of WS₂ contacted by graphene[150]. We believe the origin of the inhomogeneous photocurrent in those experiments to be similar to our device.

The modification of the workfunction of graphene with gate voltage could be one possible mechanism for the observed photoresponse[188], as it would result in
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a change in the interfacial electric field. However, we see that not all spots in the spatial photoresponse map are modified as a function of gate voltage and only a particular region has a tunable photoresponse. The absence of a uniform modulation of photoresponse suggests the role of a localized source in the nanotube itself.

There are reports in literature[189] that show enhanced photoluminescence in cracked regions of MoS$_2$ monolayers caused by the adsorption of oxygen in the sulfur vacancy regions. This was accompanied by a blue-shift of the $A_{1g}$ mode in the Raman spectrum of the cracked regions. Figure 7.11 shows the Raman spectra of our hybrid WS$_2$ nanotube device from the normal region and the region of gate tunable photoresponse. We observe a shift of $\sim 2$ cm$^{-1}$ in the $A_{1g}$ peak of the nanotube at the defect site of the device. These defects could be cracks developed in the nanotube due to the ultrasonication of the nanotube solution or due to the transfer process during device fabrication. Although this supports the role of the defects in the enhanced photoresponse observed in our graphene-WS$_2$ devices, the resolution of our technique is limited by the spot size of the incident laser. Therefore it does not provide any direct insight regarding the microscopic nature of the defects. The presence of localized defects in carbon nanotubes[186, 187, 190] has been studied extensively in the past, and similar defects are likely to exist in inorganic TMDC nanotubes as well.

We have also seen a similar photoresponse in another hybrid device. Figure 7.12 shows the data obtained from the WS$_2$ slit device shown in Figure 7.9. The photocurrent maps and response along the nanotube length show the presence of a region of photoresponse that is tunable with gate voltage. This region is seen to have a shift in its Raman spectrum from other regions in the nanotube. This again supports the role of defect regions in the tunable photoresponse seen in our hybrid devices.

7.2 Summary

To summarize, we performed photoresponse studies on WS$_2$ nanotube devices with gold and graphene as contact electrodes. We generated photocurrent maps of the devices to study the local nature of photocurrent generation. The Schottky barrier near the contact regions of the nanotubes were seen to give rise to the photoresponse in the metal contacted nanotubes. We further observed spatially inhomogeneous photore-
Chapter 7. Other projects

Figure 7.12: (a) Photocurrent map of the slit device at zero gate voltage obtained using 532 nm laser in ambient conditions (bias voltage = 1 V, laser power = 4 µW). Photoresponse is seen only from the region of the nanotube overlapping with graphene. The dotted circle (in blue) shows the local defect region which is tuned by gate voltage. (b) Variation in the photocurrent determined from the map shown in (a) across the line joining points X and O. (c) Optical image of the hybrid device with corresponding Raman spectrum (right) for the points 'a' and 'b' showing the defect site 'b' showing a shift in the characteristic WS$_2$ Raman peak.
sponse in the nanotube contacted by graphene electrodes and demonstrated a method of detecting defects in WS\textsubscript{2} nanotubes using scanning photocurrent microscopy.
Chapter 8

Summary and outlook

In this thesis, we have described the experiments on two main systems: the electromechanics of graphene drums in the low tension regime and doubly clamped InAs nanowires in the high tension regime. The tunability of these systems were seen to be related to the built-in tension. The high tunability of the graphene drums allowed us to parametrically modulate the frequencies and tune the coupling between the mechanical modes. The low tunability of the InAs nanowire resonators allowed us to do Joule heating studies to probe the thermal properties of the system. Here, we summarize the results discussed in previous chapters and present an outlook for future experiments.

In Chapter 5, we discussed the experiments on graphene drums that were highly tunable at low temperatures. The main results of this chapter were published as “Dynamical strong coupling and parametric amplification of mechanical modes in graphene drums”, John P Mathew et al., Nature Nanotechnology 11, 747 (2016). The low tension in the membranes allowed us to tune the fundamental mode frequency over a large range. This allowed us to parametrically modulate the spring constant of the mode. When the spring constant was modulated using a pump signal at twice the resonant frequency we achieved parametric amplification as well as deamplification of the mechanical motion. At a particular phase difference between the pump and drive signals the amplitude of the resonator was amplified by a factor of $\sim 3$. The amplitude gain achieved deviated from the theoretically predicted value.
at large pump amplitudes. The gain was shown to be limited by the presence of non-linear dissipation in the graphene drum.

We also discussed the experiments on coupling mechanical modes of the drum. The low tension in the system allowed us to deform the membrane to such an extent that the presence of inhomogeneous stress terms became important. The coupling strength between various modes is proportional to the deflection induced tension and therefore tunable with the gate voltage. With increasing gate voltage, two modes were seen to exhibit an avoided crossing, characteristic of a strong coupling between them. We performed pump-probe experiments where the coupling between the modes was parametrically modulated.

For one set of experiments the pump frequency was fixed to the frequency difference of the two modes (red pump). This allowed us to control the rate of energy transfer between the modes and achieve hybridized mechanical states. In this state, the coupled modes transferred energy faster than their dissipation rates. In the presence of the red pump the coupling led to a mode splitting, where the frequency separation between the new modes was linearly proportional to the pump amplitude. We showed that we could achieve a cooperativity as high as 60 between the mechanical modes of frequency $\sim 100$ MHz. The tunability of the modes further led to higher order mode splitting when the pump frequency was detuned to half the frequency difference of the modes. The mode splitting was seen to be proportional to the square of the pump amplitude. This response was also well captured by the numerical solutions of the equations of motion for the coupled modes.

An effect similar to parametric amplification was also observed when the pump was detuned to the sum of the frequencies of the two modes (blue pump). The blue pump provided control over the dissipation rates of the individual modes. The coupling between the modes led to down conversion of the pump signal which populated the mechanical modes and amplified the motion. The dissipation was seen to quadratically decrease with the pump voltage. This showed that we could use the coupling to manipulate the response of individual modes in the system. One of the reasons for studying mode coupling was to show that the coupled mechanical modes mimic an optomechanical system where a low frequency mode plays the role of oscillator and high frequency mode is the cavity. Such an analogy works better when we can...
Chapter 8. Summary and outlook

manipulate well separated mechanical modes using the pump probe schemes. For this, we demonstrated dynamical strong coupling between mechanical modes of the drum that were separated in frequency by a factor of \( \sim 2 \). Similar physics of mode splitting and non-degenerate parametric amplification was observed in these modes as well.

Studying dissipation in carbon based resonators is a promising field of research. Although non-linear dissipation has been observed\(^[64]\) in graphene and carbon nanotubes, we do not yet have a complete understanding on the origin of this phenomenon. Thermal fluctuation induced energy exchange between coupled modes was theoretically shown\(^[191]\) to increase the dissipation in carbon nanotube resonators. This could also play a major role in the low dissipation observed in graphene based sensors at room temperature. Therefore, a systematic study of the dissipation and precise control over the coupling of various modes in these systems could prove useful in designing better NEMS based sensors for the future.

In Chapter 6 we discussed experiments on doubly clamped InAs nanowire resonators. The main results of this chapter were published as “Nanoscale electromechanics for measuring thermal conductivity, expansion and interfacial losses” John P Mathew et al., *Nano Letters* 15, 11 (2015). These devices were seen to have a high built-in tension and showed low tunability with gate voltage. This implied that a small change in electrostatics would not cause large frequency shifts in the device. This allowed us to do Joule heating experiments on the suspended devices to study their thermal properties. A dc current gave rise to a large temperature increase in the nanowires due to their low thermal conductivity and induced an additional thermal stress on the nanowire. The mechanical frequency shifts with source-drain bias voltage was measured at low temperatures and the non-monotonic shift of the resonant frequency was attributed to the temperature dependence of the thermal expansion coefficient of InAs. We performed simulations of our system and correlated the observed frequency shifts to the temperature increase and thermal stress in the nanowires. We assumed the thermal conductivity to be linearly dependent on the temperature and have the form \( \kappa = bT \) in the temperature range of 10-60 K. By comparing the experimentally observed frequency shifts to the simulations at various bath temperatures we were able to obtain the value of the constant \( b \) for the nanowires. Thus, by converting the temperature changes in the nanowire to a mechanical frequency shift we
were able to measure the thermal conductivity of individual nanowires.

We then studied the change in dissipation in the nanowires with Joule heating. The quality factor of the device was seen to decrease when the nanowire was Joule heated. We showed that the dissipation in the device increases with increasing average temperature of the nanowire. However, the dissipation was seen to be lower for a lower bath temperature even when the average temperature of the nanowire was held constant. This gave us insight on the dissipation mechanism arising from the contact regions of the device. We attributed the offset seen in the dissipation with bath temperature to the temperature dependent losses of the gold contacts of the device. Similar metal contacted NEMS devices fabricated with low loss metal supports could be explored in the future to study this in detail. It would be interesting to see if the room temperature quality factors of NEMS device can be improved by this method.

Ongoing work includes studying the coupling between perpendicular modes of the InAs nanowire resonators. The tension induced in the nanowire due to large oscillation amplitude of one mode can tune the frequency of the second mode. The effect of the Duffing character of one mode on the response of the second mode is an interesting topic for further studies. This was also briefly discussed in Chapter 6.

Finally, in Chapter 7 we presented one of the other projects that was explored during the course of this work. This work was published as “Light matter interaction in WS\(_2\) nanotube-graphene hybrid devices” John P Mathew et al., Applied Physics Letters 105, 22 (2014). We discussed how the Schottky barrier present at the contact regions of WS\(_2\) nanotube devices gave rise to photocurrent generation. We further used graphene contacts to study the local nature of photoresponse along the length of the nanotubes and showed that defects in the nanotube gave rise to gate tunable photoresponse.
Appendix A: Fabrication details for InAs nanowire resonator on non-insulating substrates

Resist coating

Prior to coating the e-beam resists the sample is cleaned in acetone and isopropyl alcohol. The chip is then blow dried using nitrogen gas.

Resist spin speeds and baking times for suspended nanowire devices

<table>
<thead>
<tr>
<th>Resist</th>
<th>Speed (rpm)</th>
<th>Time (s)</th>
<th>Temperature (°C)</th>
<th>Time (minutes)</th>
<th>Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EL-9</td>
<td>10,000</td>
<td>60</td>
<td>178</td>
<td>7</td>
<td>200</td>
</tr>
<tr>
<td>PMMA-495 A4</td>
<td>4,500</td>
<td>45</td>
<td>180</td>
<td>2</td>
<td>250</td>
</tr>
<tr>
<td>PMMA-950 A2</td>
<td>2,000</td>
<td>45</td>
<td>180</td>
<td>2</td>
<td>250</td>
</tr>
</tbody>
</table>

The following resist developing times are used after the e-beam lithography step in all our devices.

E-beam resist developing times followed in this work

<table>
<thead>
<tr>
<th>Developer</th>
<th>Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIBK-IPA (1:3)</td>
<td>90</td>
</tr>
<tr>
<td>Stopper</td>
<td>Time (s)</td>
</tr>
<tr>
<td>IPA</td>
<td>60</td>
</tr>
</tbody>
</table>

Chip is blow dried using nitrogen gas after the developing process.
Metal deposition details - sputtering

Metal deposition is carried out by sputtering the target metal in the presence of a plasma. Sputtering is carried out after pumping the chamber to low $10^{-7}$ mbar pressure. Prior to metallization we carry out plasma etching to remove resist residues and the native oxide on the nanowire. The details for rf plasma etching is given below.

Parameters for plasma etching

<table>
<thead>
<tr>
<th>Gas</th>
<th>Pressure (mbar)</th>
<th>rf power (W)</th>
<th>Time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Argon</td>
<td>$2.5 \times 10^{-2}$</td>
<td>25</td>
<td>2</td>
</tr>
</tbody>
</table>

Metal deposition is carried out by dc magnetron sputtering. The standard recipe involves depositing $\sim 400$ nm of metal. This ensures that the nanowire is clamped by the metal and held suspended above the substrate. The usual metal of choice for suspended InAs nanowires is 350 nm of gold with a thin layer (50 nm) of chromium for improved adhesion. Below we list some of the details of sputtering various targets.

Parameters for various sputtering targets

<table>
<thead>
<tr>
<th>Metal</th>
<th>Pressure (mbar)</th>
<th>Voltage (kV)</th>
<th>Current (A)</th>
<th>Rate (nm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium</td>
<td>$7.5 \times 10^{-3}$</td>
<td>0.28</td>
<td>0.15</td>
<td>20</td>
</tr>
<tr>
<td>Gold</td>
<td>$7.5 \times 10^{-3}$</td>
<td>0.38</td>
<td>0.15</td>
<td>85</td>
</tr>
<tr>
<td>Terfenol-D</td>
<td>$7.5 \times 10^{-3}$</td>
<td>0.29</td>
<td>0.22</td>
<td>20</td>
</tr>
<tr>
<td>ITO</td>
<td>$8 \times 10^{-3}$</td>
<td>0.32</td>
<td>0.17</td>
<td>50</td>
</tr>
<tr>
<td>MoRe</td>
<td>$8 \times 10^{-3}$</td>
<td>0.36</td>
<td>0.10</td>
<td>30</td>
</tr>
</tbody>
</table>

The above recipe can be used for devices on Si$^{++}$/SiO$_2$ as well as on intrinsic silicon.
Appendix B: Fabrication details for InAs nanowire resonator on insulating substrates

We use c-plane sapphire substrates as the insulating substrate. Cleaving sapphire can be challenging as it does not break as easily as silicon based substrates. We use a diamond scribe to scratch a sapphire wafer into a rectangle of roughly 1 cm². The chip is then broken from the wafer by hand. Gloves are worn to ensure the chip is not contaminated. The chip is then cleaned in acetone and isopropyl alcohol followed by blow drying using nitrogen gas.

As the substrate is insulating we provide a conducting path for performing e-beam lithography by evaporating aluminium on the sapphire chip. This is followed by the standard resist coating recipe provide in Appendix A. After developing the chip, the Al below the developed regions is etched using the time duration given in the table.

<table>
<thead>
<tr>
<th>Metal</th>
<th>thickness (nm)</th>
<th>Etchant</th>
<th>Etch time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>20</td>
<td>1% TMAH</td>
<td>1</td>
</tr>
</tbody>
</table>

After etching, the chip is dipped in DI water for 2 minutes and then rinsed in IPA, and blow dried using nitrogen gas.

The other precaution while carrying out fabrication on sapphire is to reduce the time of *in situ* plasma etching prior to metal deposition. The parameters used for plasma etching are given below.

<table>
<thead>
<tr>
<th>Gas</th>
<th>Flow (sccm)</th>
<th>Pressure (mbar)</th>
<th>rf power (W)</th>
<th>Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Argon</td>
<td>60</td>
<td>$2.5\times10^{-2}$</td>
<td>25</td>
<td>40</td>
</tr>
</tbody>
</table>

After plasma etching and sputtering, the chip is placed in acetone for liftoff. The excess metal is normally removed in few minutes. The chip is then rinsed in IPA (1 minute) and DI water (2 minutes) and placed in a petri dish containing TMAH for a minute to remove the remaining aluminium.
Appendix C: Fabrication details for drum resonators

Fabricating gate electrode on sapphire

The substrate used for drum resonators is c-plane sapphire. A small chip (7 mm x 7 mm) is cleaned in IPA and dried. A 20 nm thick conducting metal layer of chromium is used prior to e-beam lithography to fabricate the gate electrode.

These parameters are used for all conducting layers of chromium used. The conducting metal layer is followed by spin coating e-beam resists. The two layer resist recipe given in the table below is followed.

Two layer e-beam resist recipe used in the drum device fabrication

<table>
<thead>
<tr>
<th>Resist</th>
<th>Speed (rpm)</th>
<th>Time (s)</th>
<th>Temperature (°C)</th>
<th>Time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EL-9</td>
<td>3,200</td>
<td>45</td>
<td>175</td>
<td>7</td>
</tr>
<tr>
<td>PMMA-950 A2</td>
<td>2,000</td>
<td>45</td>
<td>175</td>
<td>2</td>
</tr>
</tbody>
</table>

Lithography is then performed to write the markers and gate electrode at the center of the chip. The gate electrode is kept inside the same write-field as that of the markers for ease and is written in the same step. After lithography the chip is developed using the recipe given in Appendix A. The chromium in the developed regions are then etched. As the gate electrode is small the etchant could have hindrance in reaching the metal at the bottom. Therefore, the chip is placed in a small beaker with the etchant for one minute and a magnetic stirrer is spun at a low rate for uniform etching. Details of the chromium layer are provided in the table below.

Conducting layer of Cr used on sapphire

<table>
<thead>
<tr>
<th>Metal</th>
<th>thickness (nm)</th>
<th>Etchant</th>
<th>Etch time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>20</td>
<td>Sigma-Aldrich 651826</td>
<td>0.5-3 (see text)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After etching, the chip is dipped in DI water for 45 s and then rinsed in IPA, and blow dried using nitrogen</td>
<td></td>
</tr>
</tbody>
</table>

The gate is formed by evaporating 15 nm of titanium and 10 nm of platinum. The chip is then placed in acetone for 30 minutes for liftoff of excess metal. Then the remaining chromium is etched in a beaker containing the etchant for one minute.
using a magnetic stirrer. The marker pattern and the local gate are now defined in metal on the sapphire substrate.

**Depositing SiO₂ and etching drum cavity**

Multiple chips with the local gate patterned are loaded together for SiO₂ deposition. The chips are cleaned with IPA and blow dried using nitrogen prior to loading in the PECVD chamber. 300 nm of SiO₂ is deposited at 150°C. The PECVD recipe involves a cleaning step using O₂ plasma prior to the SiO₂ deposition.

The hole is patterned using e-beam lithography for which 20 nm of chromium is deposited on the SiO₂. E-beam resist is coated following the two layer resist recipe prescribed above. The hole and a second hole over the outer pad of the gate electrode is drawn in the same CAD file as that of the gate electrode. As the marker pattern is underneath 300 nm of SiO₂ and 20 nm of chromium, its visibility becomes low in the SEM. Therefore, e-beam lithography is done with the help of the SE-2 mode of the detector for ease of locating the marker pattern. The SE-2 mode offers better contrast of the sub-surface features and helps in locating the marker pattern. After lithography, the resist is developed and the chromium is removed from the developed regions. Once again, the Cr etching is carried out in a beaker with magnetic stirring. After drying, the chip is placed in acetone for half an hour to completely remove the resist layers.

RIE is done to etch the hole. The Cr layer acts as a mask and protects the SiO₂ layer. The parameters for etching the SiO₂ to form the drum cavity are provided in the table below.

<table>
<thead>
<tr>
<th>Gases (flow in sccm)</th>
<th>rf power (W)</th>
<th>Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHF₃ (98), O₂ (2)</td>
<td>150</td>
<td>1000</td>
</tr>
</tbody>
</table>

After the RIE step, the chip is placed in chromium etchant for three minutes to completely remove any traces of chromium. Subsequently, the chip is placed in DI water, IPA, and blow dried. The chip now has a local gate electrode, 300 nm of SiO₂, and holes in the SiO₂ over the central gate region and outer pad of the gate electrode.
Patterning source/drain electrodes above the SiO$_2$

The two layer resist recipe is followed to cover the chip now with e-beam resists. The conducting metal layer of chromium is then evaporated above the resist layers. The drawing for the source-drain electrodes is also made in the same CAD file as the gate electrode and hole. For e-beam lithography the SE-2 mode is used for ease of visibility of the markers. After lithography, the Cr layer is etched by placing in the etchant for 30 seconds followed by rinsing and drying. The resist layers are now developed using the standard procedure. The dried chip is loaded into the evaporator for depositing 10 nm of chromium and 50 nm of gold.

The metal deposited chip is placed in acetone for 15 minutes for final liftoff of excess metal. Once the chip is rinsed in IPA and dried using nitrogen gas the fabrication process is complete. Any layered material can now be exfoliated and placed over the source/drain electrodes and hole region using the PDMS transfer technique to complete the device.
Appendix D: Details of low temperature, \textit{rf} amplifier

The low temperature \textit{rf} amplifier we use (CITLF1 from Caltech) is able to provide $\sim 45$ dB of gain at cryogenic temperatures. Figure A1 shows an image of the amplifier with the connection ports marked. The amplifier, along with its wiring, is kept fixed in the low temperature \textit{rf} insert. The connections on top of the \textit{rf} insert corresponding to the amplifier are:

Pin 1: amplifier power
Pin 9: dc output from amplifier bias tee
Pin 10: amplified \textit{rf} output.

Figure A1(b) shows the gain of the amplifier measured inside the insert (kept outside the cryostat at room temperature in ambient) with a bias of 5 V and current of 19.3 mA (power was applied from Pin 1 of the insert using a Keithley sourcemeter). The same biasing point (5 V, $\sim 19$ mA) can also be used at cryogenic conditions down to a temperature of 5 K. However, the amplifier can contribute to heating when placed in the cryostat at lower temperatures. Therefore, for temperatures below 30 K a large flow of helium must be used to achieve thermal equilibrium in the cryostat.
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