

**Microfabricated Silicon Nano-Electromechanical
Resonators with Widely Tunable Resonant Properties for
Gas Sensing Application**

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**Microfabricated Silicon Nano-Electromechanical
Resonators with Widely Tunable Resonant Properties for
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Abstract

Over the past few years, the demand for sophisticated gas detection and monitoring has grown, driven by increasing concerns for human health and medical purposes. Nano-electromechanical system (NEMS) resonators have emerged as high-performance gas sensors due to their ultralow mass, allowing them to monitor parts-per-trillion-level gas variations. Key metrics such as responsivity and detection limit, significantly influenced by the resonator's properties like mass, resonance frequency, and nonlinearity, are crucial for their sensing performance. Tunable NEMS resonators, which allow adjustments of resonance frequency and nonlinearity post-fabrication, present a promising opportunity to enhance sensing performance such as increasing responsivity and decreasing detection limit. However, integrating atomically thin materials in NEMS resonator devices demands complex and specialized fabrication processes, which, combined with the lack of wafer-scale single-crystal-quality base materials, restricts their ubiquitous application. Thus, achieving comparable electrostatic tunability in conventional monolithically microfabricated resonator devices, especially from silicon (Si), which offers wafer-scale, inexpensive, single-crystal-quality material and scalable monolithic fabrication process, is highly desired but challenging. The main goal of this research is to develop a monolithic microfabrication process for creating ultrathin Si NEMS resonators with ultrawide tunability in resonant properties for gas sensing application.

At first, by carefully designing the dimensions and employing a scalable microfabrication process involving advanced electron-beam-lithography and optimized deep reactive-ion etching, ultrathin Si NEMS resonators (width ~ 10 nm, length ~ 100 μm , and height ~ 7.5 μm) are monolithically fabricated. The largest aspect ratio can reach around 15000. The characterization of fabricated devices highlighted their stress-dominated dynamic behaviour. By gradually decreasing our device's width from 300 nm to 20 nm, we demonstrate a gradual transition from beam-like to string-like dynamic behaviour with thinning of width.

Then, the ultrawide resonance frequency tunability in the developed ultrathin Si NEMS resonators is experimentally demonstrated and theoretically investigated. By applying DC gate voltages between the actuation electrode and nanoresonator structure, ultrawide electrostatic tuning ($\sim 70\%$) of (initial) resonance-frequency ($\sim 7\% \text{ V}^{-1}$) at room temperature is observed in a fabricated

ultrathin Si NEMS (width ~ 40 nm, length ~ 200 μm) resonator device. Such wide tunability is comparable to atomically thin resonators, such as Graphene and MoS₂-based resonators. An approximate electromechanical model is established to explain the tuning mechanism and explore the tuning behaviours under various conditions. Furthermore, the temperature-induced resonance frequency tuning and temperature dependence of electrostatic tunability are investigated by varying the device's substrate temperature.

According to the experimental resonance response results of fabricated Si NEMS resonators under the nonlinear regime, the transition from linear to nonlinear regimes is identified as increasing driving voltage. By manipulating the DC gate voltage, even the cancellation of nonlinearity can be approximately reached when flipping the sign of nonlinearity. The linear dynamic range of the device can be broadened around this cancellation point, reaching around 86 dB based on the estimation of one device with a width of 100 nm and a length of 100 nm. The study also demonstrated the activation of internal resonance without requiring special structural designs, showing the versatility of Si NEMS resonators in nonlinear regime applications, including advanced signal processing and energy harvesting.

The high surface-to-volume ratio and the low mass of the Si NEMS resonators significantly enhanced their responsivity to gas molecules, demonstrated by the experimental detection of CO₂ with a high responsivity of 0.019 Hz/ppm. The study also explored methods to improve sensing performance through electrostatic tuning, allowing real-time adjustments to the resonator's responsivity and detection limit. This work highlights the potential for ultrathin Si NEMS resonators to benefit various sensing applications, offering high responsivity, operational flexibility, and potential for integration into various industrial and environmental monitoring systems.

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Chapter 1 Introduction

1.1 Nano-electromechanical system (NEMS) resonator

1.1.1 Introduction to NEMS

Rapid progress of micro / nano device technology in recent decades has dramatically influenced human life and social progress. Continued growth in this area combined with the ongoing progress in information- and bio-technology is expected to lead us to a technologically superior society and human-centric development. Since the advent of 'Internet of things' (IoT) technology, the need for new electronic devices and sophisticated sensors has been steadily increasing. One area in this realm of building novel devices that has seen a tremendous progress over the past few decades is known as the microelectromechanical systems (MEMS).¹ MEMS are devices that derive functionality from the mechanical motion of integrated micrometer-scale object(s) and they are exceptionally efficient and reliable at sensing various physical quantities such as acceleration, pressure, rotation, etc.² Low-cost, high power-efficiency and small-size of MEMS has made their presence ubiquitous, especially in communications, automotive, and aerospace industries.

Over the years, miniaturization has been a critical focus for integrated circuits (ICs) to stay consistent with Moore's Law. Similarly, the MEMS industry also seeks size reduction and performance enhancement through the pursuit of miniaturization by creating nanometer-scale components. Nanoelectromechanical systems (NEMS) represent an impressive advancement over MEMS by shrinking mechanical and electrical components to nanometer dimensions.³ This size reduction brings with it many novel effects, such as ultrahigh resonance frequency, higher responsivity, molecular-level detection resolution for biomolecules sensing, reduced power consumption, and stronger interactions with various physical quantities like light, heat and electricity. NEMS technology has opened up new opportunities in various domains, from precise sensing and identification of biological or chemical molecules, signal processing, to advanced wireless communication.⁴ NEMS devices' compact dimensions enable the integration of multiple functions onto one chip for creating complex systems with versatile capabilities.

A series of applications of NEMS technology can be realized by the creation of a class of devices known as NEMS resonators. NEMS resonators operate through the principle of resonant vibration of an integrated mechanical nanostructure; thus, they typically operate at high frequencies and are

sensitive to minute perturbations to their mass, mechanical stiffness or environment, - making them suitable for ultra-sensitive sensing applications.⁵ NEMS resonators can also exhibit wide resonance-frequency tunability, low energy consumption, and complex nonlinear behaviours.⁶ These exceptional performance characteristics, combined with nanomaterial properties and factors arising from scaling down the device dimensions, create a powerful platform for detecting and analyzing various physical, chemical and biological phenomena. For instance, adding even minute amounts of substances such as gas molecules or biomolecules to their surface can cause detectable frequency shifts in NEMS resonators, making identification even of extremely small quantities of substances possible. Li et al. demonstrate the fabrication and operation of self-sensing NEMS cantilevers resonator with very high frequency resonances, achieving thermomechanical-noise-limited displacement responsivity of $39 \text{ fm/Hz}^{1/2}$ and mass resolution less than 1 attogram in ambient conditions.⁷ Ollier et al. present the design, fabrication, and implementation of NEMS gas sensors in a breakthrough multigas analysis system, achieving a frequency stability with an Allan deviation of less than 5×10^{-7} in air and demonstrating the capability to analyze complex gas mixtures with typical detection limits in the ppm to ppb range.⁸ Morelli et al. present a novel multi-layered graphene gas sensor functionalized with gold nanoparticles, demonstrating both signal saturation during analyte exposure and an inverted current response, with enhanced responsivity to 1 ppm of NO_2 at room temperature.⁹ NEMS resonator can also be used to perform real-time inertial imaging of individual analytes adsorbing by monitoring the frequency shifts in multiple vibrational modes.¹⁰ Besides sensing the mass or gas molecules, the NEMS resonator can also detect the other physical quantities, applied as a magnetic sensor or force sensor. Zheng and Xu integrated a resonant graphene membrane with a conventional fiber sensing system to create a compact fiber magnetic sensor, achieving a device size of $14.9 \text{ }\mu\text{m}$ and a responsivity of 110 kHz/T .¹¹ Lee et al. developed a novel nanomechanical beam structure integrated with a photonic crystal waveguide filter, creating an optical NEMS-based force sensor capable of detecting vertical deformations as small as 20-25 nm and strains as low as 0.004%, demonstrating high responsivity to applied forces through shifts in resonant wavelength.¹²

1.1.2 Structures of NEMS resonator devices

NEMS resonators have various structures designed for different applications and performance requirements. These resonators can be broadly classified into several categories based on their structural constraints: beam¹³ / string type¹⁴ (Fig.1-1a, b), center-supported disk type¹⁵ (Fig.1-1 c) and suspended membrane type¹⁶ (Fig.1-1 d). Researchers often modify these basic structures for distinct advantages to meet specific practical needs.

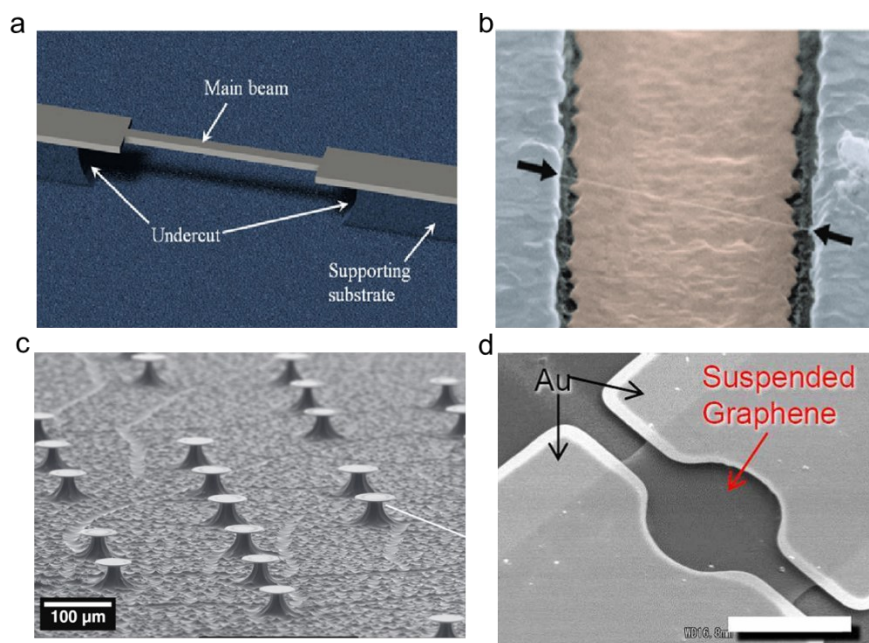


Figure 1-1 Common structures of NEMS resonators. a. Double-clamped Si NEMS resonator.¹³ b. 1D CNT resonator.¹⁴ c. Disk-type NEMS resonators.¹⁵ d. Membrane-type NEMS resonator.¹⁶

Beam-type resonators are among the most common NEMS structures. These typically consist of a nanoscale beam, which can be a cantilevered, clamped-clamped, or free-free, capable of vibrating at specific resonant frequencies determined by their dimensions (length, width, and height) and material properties (mass-density, Young's modulus), etc. With their larger working area and stable, simple geometric structure, single-ended fixed cantilever beam resonators are widely used in sensing applications. For example, Kosaka et al. used single-ended fixed cantilever beam resonators to detect cancer biomarkers in serum with a detection limit of 10^{-16} g mL⁻¹.¹⁷ These cantilevers are also excellent for studying cutting-edge physical phenomena such as laser cooling, cavity optomechanics, and quantum noise squeezing. Clamped-clamped beams, fixed at both ends, offer higher stiffness and frequency than cantilever beams, making them advantageous in applications requiring high resonance frequencies and stability, such as RF signal processing and high-

performance sensing. In 2012, Mahboob et al. achieved sideband cooling on double-ended fixed cantilever beams by applying parametric excitation signals,¹⁸ while Antonio et al. realized internal resonance by achieving a frequency ratio of 1:3 between modes through electric field tuning, stabilizing the mechanical resonator frequencies.¹⁹

The suspended membrane-type mechanical resonator is another key structure, with its edges anchored to the base while the middle part of the membrane can vibrate freely. These resonators are highly sensitive to pressure changes and are suitable for pressure-sensing applications. The high internal stress generated during fabrication gives membrane resonators extremely high-quality factors, up to 10^7 .²⁰

Centre-supported disk resonators have a constraint point at the center, allowing the margin to vibrate freely. These structures are commonly used in inertial sensors, such as gyroscopes.²¹ Disk resonators can support multiple vibrational modes, including radial and tangential modes, and are known for their high-quality factors and ability to achieve higher frequencies. The paired orthogonal modes of disk-type micro-mechanical resonators are particularly useful for detecting Coriolis forces, making them valuable for ultra-high precision mass detection²² and optical-mechanical coupling with optical waveguides.²³

In addition to these main types, other mechanically resonant structures exist without specific shapes, such as carbon nanotube resonators made from one-dimensional materials and graphene resonators made from two-dimensional materials.²⁴ Multi-layered and heterostructure resonators combine multiple layers of different materials, such as metals, semiconductors, and insulators, to enhance performance.²⁵ The integration of diverse materials allows for tuning of mechanical, electrical, and thermal properties, increasing their attention.

1.1.3 Material and fabrication

NEMS resonators' materials and fabrication processes significantly impact their resonant properties and their performance in practical applications. The fabrication techniques for NEMS resonators can also be categorized into two types: top-down and bottom-up, as shown in Fig. 1-2.²⁶

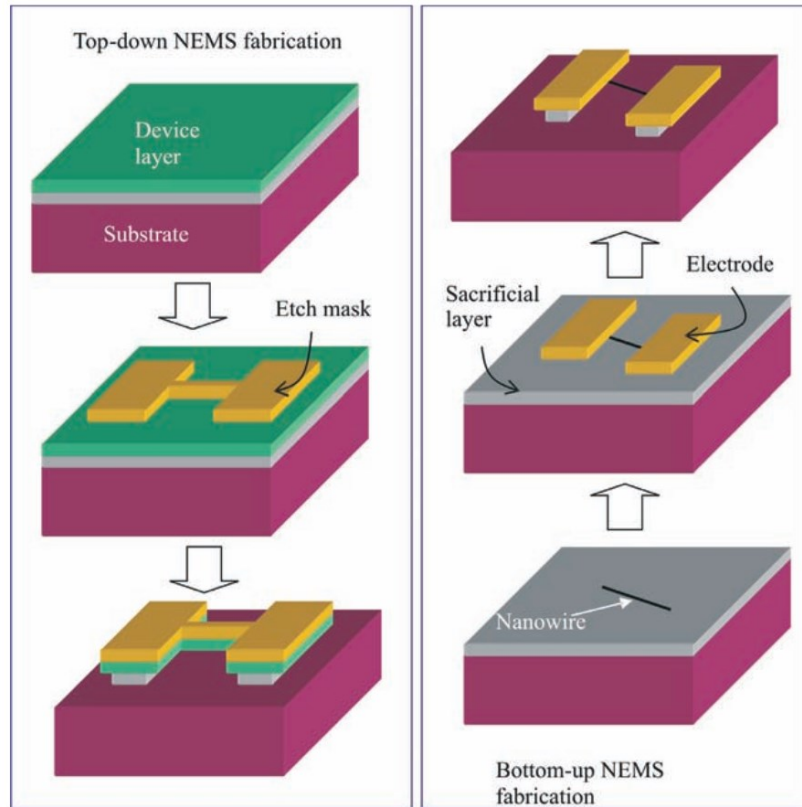


Figure 1-2 Schematic process flow diagrams for top-down and bottom-up NEMS fabrication processes²⁶.

Top-down fabrication method

Top-down fabrication techniques, derived from traditional semiconductor processing methods, involve the precise removal of material from a larger structure.²⁷ A prominent example is silicon-based resonators fabricated from silicon (Si) and silicon-on-insulator (SOI) wafers. Silicon-based resonators, known for their high intrinsic quality factor, offer excellent frequency stability, repeatability, and compatibility with CMOS processes. The fabrication process employs photolithography and reactive ion etching (RIE) to enable selective material removal for ideal resonator structures, facilitating the production of simple microstructures such as membranes, square mass blocks, clamped beams, and cantilever beams.

The frequently used microfabrication techniques used for NEMS resonators through a ‘top-down’ approach include:²⁸

Photolithography: A fundamental process in microfabrication that involves transferring a geometric pattern from a photomask to a light-sensitive chemical photoresist on the substrate. The pattern created by photolithography defines the regions where material deposition or etching will

occur. It is crucial to define the fine features of NEMS devices.

Electron Beam Lithography (EBL): EBL is used to create extremely fine ($\sim$ a few nanometers) patterns with high precision, essential for fabricating NEMS resonators with nanoscale dimensions. Unlike photolithography, EBL uses a focused beam of electrons to directly write patterns onto the resist, allowing for higher resolution.

Chemical Vapor Deposition (CVD): CVD is a process used to deposit thin films of materials onto the substrate. In NEMS fabrication, materials such as silicon, silicon carbide, or various metal oxides are deposited using CVD. This method ensures uniform and high-quality films, which are necessary for the resonant properties of NEMS devices.

Etching: Both dry etching (such as reactive ion etching) and wet etching are used to remove specific material areas from the substrate to create the desired NEMS structures. Dry etching provides better anisotropy and precision, which is critical for maintaining the integrity of the nanoscale features.

Bottom-up fabrication method

Bottom-up fabrication techniques build structures atom-by-atom or molecule-by-molecule, often resulting in materials with superior properties.²⁹ Carbon-based NEMS resonators, such as CNT and graphene, are fabricated using CVD methods. These materials provide excellent mechanical strength and electrical properties at nanoscale dimensions. For instance, the team led by Fei Wei at Tsinghua University has achieved millimetre-scale CNT resonators by enhancing catalyst activity and developing a non-contact acoustic resonance test system to study the fatigue behaviour of single CNT, demonstrating excellent fatigue resistance under continuous stretching.³⁰ An essential step in the bottom-up approach is the transfer process, where the fabricated nanostructures are carefully moved from the growth substrate to the substrate prepared for device.³¹ It often involves techniques like dry or wet transfer methods, which aim to maintain the integrity and properties of the nanoscale structures during relocation. Achieving precise alignment during the transfer is challenging, and there is a risk of damaging the desired nanostructures, resulting in the defects or degraded performance. Additionally, the transfer process can introduce contamination or residual stresses, which may affect the overall properties of the NEMS resonators.

Material selection and fabrication techniques are crucial in defining the performance, reliability, and integration capability of NEMS resonators. Material properties combined with fabrication techniques ultimately determine their suitability for intended applications.

1.1.4 Actuation and detection

Actuation and detection are critical aspects of NEMS resonators, determining their functionality and application performance. The choice of actuation or detection methods will affect the resonator's responsivity, resolution, and efficiency in sensing applications.³²

Table 1-1 The concluded advantages and drawbacks of common MEMS resonator's actuation and detection methods

Actuation	Advantages	Drawbacks
Electrostatic	Simple, minimal additional components, easy integration	Pull-in, parasitic capacitance, poor electromagnetic interference resistance, weaker forces
Electromagnetic	Contactless operation	Integration challenges, heat dissipation issues
Piezoelectric	High efficiency, low power consumption, rapid response	Thermal expansion mismatches
Thermal	Straightforward	Slower response times, higher power consumption, thermal stress issues
Detection	Advantages	Drawbacks
capacitive	Highly sensitive, compact designs	Parasitic capacitances, signal crosstalk
piezoelectric,	Direct electrical readouts	Fabrication complexity, environmental responsivity,
piezoresistive	Straightforward, integratable	Temperature-sensitive, complex fabrication
optical	Sub-nanometer resolution, non-contact methods	complex optical setups, integration challenges

Actuation involves applying the external force to induce mechanical motion in a resonator. Electrostatic actuation is one of the most common techniques, using a voltage between the resonator and nearby electrode to generate electrostatic force that drives it into motion. Electrostatic actuation is popular due to its simplicity, minimal additional equipment requirements and easy integration.

Unfortunately, it may suffer from pull-in instability, parasitic capacitance and poor electromagnetic interference resistance, limiting its usage. Other actuation methods include electromagnetic, piezoelectric and thermal. Their advantages and drawbacks³³ are concluded in Table 1-1.

Detection involves measuring the mechanical response of a resonator to external stimuli. One popular approach, capacitive detection, measures changes in capacitance between the resonator and nearby electrode as it moves. This highly sensitive method can be combined with electrostatic actuation for compact designs; however, precise control over the electrode-resonator gap may be needed as parasitic capacitances could limit resolution and cause signal crosstalk issues. Other detection methods like piezoelectric, piezoresistive, and optical detection methods offer unique advantages and challenges for resonator motion detection. Their advantages and drawbacks are also concluded in Table 1-1.

The aforementioned methods are commonly used for MEMS resonators. NEMS resonators' small vibration amplitude and difficult signal extraction pose challenges for detection. Electrical detection methods are compatible with electronic applications and can directly integrate with silicon-based integrated circuits. However, the nanoscale size of these devices makes their weak mechanical resonance signals easily masked by parasitic signals from large-scale electrodes, complicating signal extraction. Typically, a combination of advanced signal processing techniques and noise reduction strategies is employed to enhance the detection of these weak signals. For example, differential measurement setups and lock-in amplifiers can filter out noise and isolate the desired signal.³⁴

1.1.5 Resonant vibration

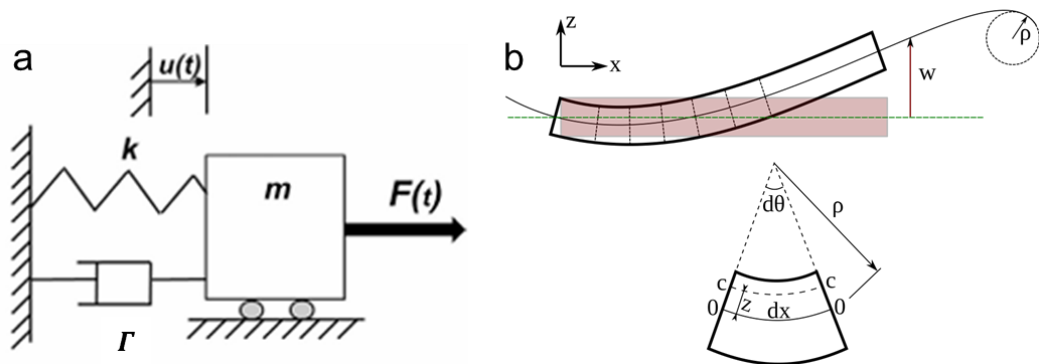


Figure 1-3 a. Mass-spring-dashpot system equivalent model. b. Bending of an Euler-Bernoulli beam. Each cross-section of the beam is at 90 degrees to the neutral axis.

NEMS resonators' resonance vibration can be effectively modelled using the mass-spring-

dashpot system (Fig. 1-3a), a foundational concept in mechanics.³³ This equivalent discrete model can describe continuous mechanical systems that approximately obey Hooke's law and Newton's laws, such as a stressed string or a drumhead. The actual motion of such a system can be described by the linear differential equation:

$$F(t) = m_{\text{eff}} \frac{d^2 u}{dt^2} + 2m_{\text{eff}} \Gamma \frac{du}{dt} + k_0 u \quad 1-1$$

where $F(t)$ is the time-varying force, m_{eff} is the effective mass, u is the displacement from the equilibrium position, Γ is the energy dissipation coefficient, and k_0 is the spring constant. This differential equation is known as the damped driven harmonic oscillator equation. When the system is driven by a simple periodic force, $F(t)$ can be expressed as:

$$F(t) = F_0 \cos(\omega t) \quad 1-2$$

where F_0 is the amplitude, and ω is the angular frequency of the driving force. Under this periodic driving force, the steady-state displacement of the system is:

$$u(t) = u_0(\omega) \cos(\omega t - \phi(\omega)) \quad 1-3$$

Solving the differential equation yields the amplitude $u_0(\omega)$ and the phase $\phi(\omega)$ as functions of the frequency:

$$u_0(\omega) = \frac{F_0/m_{\text{eff}}}{\sqrt{(\omega_0^2 - \omega^2)^2 + (\omega\omega_0/Q)^2}} \quad 1-4$$

$$\tan(\phi) = \frac{\omega\omega_0/Q}{\omega_0^2 - \omega^2} \quad 1-5$$

where the parameter $\omega_0 = \sqrt{k_0/m_{\text{eff}}}$ represents the resonant angular frequency of the resonator in the specific mode. Q is the quality factor, defined a ratio of the energy stored in the resonator to the dissipation energy per cycle by damping. In a lightly damped resonant system (where the energy lost per vibration period is small), the quality factor can be approximated as:

$$Q = \frac{\omega_0}{2\Gamma} \quad 1-6$$

Physically, the resonant frequency assesses the stiffness and mass of the system, while the quality factor evaluates the energy dissipation of the system.

This study focuses on the beam-type structure of the NEMS resonator. The Euler-Bernoulli beam theory (Fig. 1-3b) is a foundational approach that provides a deeper understanding of its physical essence.³⁵ It describes the bending behavior of slender beams under the assumptions of linear elasticity, small deflections, and the beam's cross-sectional dimensions are small compared to its

length. The equation of motion for free-vibration of the beam according to this theory is given by:

$$\rho A \frac{\partial^2 u(x, t)}{\partial t^2} + EI \frac{\partial^4 u(x, t)}{\partial x^4} = 0 \quad 1-7$$

where ρ the mass density, A is the cross-sectional area, E is Young's modulus, I is the area moment of inertia, x is the spatial coordinate along the length of the beam, t is the time variable and $u(x, t)$ is the transverse displacement. The solution to this differential equation involves finding the normal modes of vibration, which are functions of both position and time. When considering NEMS resonators, especially for the ultrathin film/beam, the effect of microfabrication process-related tensile stress (σ) on the eigenfrequency cannot be neglected. The Euler-Bernoulli equation for such prestressed NEMS resonators includes additional terms to account for this tensile force, which can be described by:

$$\rho A \frac{\partial^2 u(x, t)}{\partial t^2} + EI \frac{\partial^4 u(x, t)}{\partial x^4} - \sigma A \frac{\partial^2 u(x, t)}{\partial x^2} = 0 \quad 1-8$$

By solving the equation (1-8), the eigenfrequency Ω_n can be achieved as:

$$\Omega_n = \beta_n^2 \sqrt{\frac{EI}{\rho A}} \sqrt{1 + \frac{\sigma A}{EI \beta_n^2}} \quad 1-9$$

Where β_n is the wavenumber which should be determined to stratify the boundary conditions. Equation (1-9) shows that the eigenfrequency is basically the eigenfrequency of an unstressed beam multiplied by a term which increases with stress. And considering β_n is choose as

$$\beta_n = \frac{n\pi}{L} \quad 1-10$$

Where L is the length of the beam, when $\frac{\sigma A}{EI \beta_n^2} \gg 1$, the Ω_n could reduce to

$$\Omega_n = \frac{n\pi}{L} \sqrt{\frac{\sigma}{\rho}} \quad 1-11$$

Which becomes the eigenfrequency of a string. For a NEMS resonator, which typically has both ends clamped with non-negligible rigidity, the greater the tensile stress, the more its dynamic behaviour resembles that of a string.

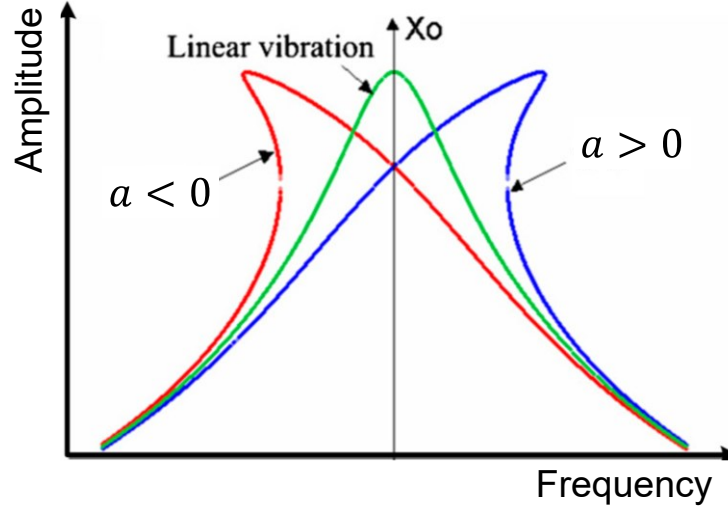


Figure 1-4 The softening ($\alpha < 0$) and hardening ($\alpha > 0$) type nonlinearity.³⁸

While the simple harmonic oscillator model provides a foundation, NEMS resonators often exhibit nonlinear behaviour due to their small dimensions, which makes geometric nonlinearity have a significant effect at relatively smaller amplitudes of vibration.³⁶ This nonlinearity can significantly impact their performance and must be considered in their analysis and design. Nonlinear behaviour in NEMS resonators can arise from several sources, such as geometric nonlinearity, where the amplitude of vibration increases and the assumptions of linear restoring force no longer hold, leading to nonlinear restoring forces; material nonlinearity, where at high strain levels, the stress-strain relationship of the material deviates from linearity; and electrostatic forces, where in capacitive NEMS resonators, the electrostatic forces between the electrodes are inherently nonlinear.

The equation of motion for a nonlinear oscillator can be established based on equation (1-1) as:

$$F(t) = m_{\text{eff}} \frac{d^2 u}{dt^2} + 2m_{\text{eff}} \Gamma \frac{du}{dt} + k_0 u + \alpha u^3 \quad 1-12$$

where α is the coefficient of the cubic nonlinear term. Nonlinear behaviour in NEMS resonators is of great interest due to its significant implications for various applications.³⁷ Unlike linear systems, where the resonance frequency remains constant regardless of the amplitude, nonlinear systems exhibit amplitude-frequency dependence. The resonance frequency (frequency corresponding to maximum attainable amplitude) changes with the amplitude. The sign of the α plays a crucial role in determining the type of nonlinearity the system exhibits, as shown in Fig.1-4. When $\alpha > 0$, the system shows hardening nonlinearity, where the system's restoring force increases

more rapidly than in a linear system as the amplitude increases. This results in a resonance frequency that increases with increasing amplitude, manifesting as a rightward tilt of the resonance peak in the frequency response curve. Conversely, when $\alpha < 0$, the system exhibits softening nonlinearity, where the system's restoring force grows more slowly compared to a linear system as the amplitude increases. This results in a resonance frequency that decreases with increasing amplitude, showing a leftward tilt of the resonance peak.³⁸ Nonlinear systems often display bifurcation and jump phenomena, where multiple stable states exist for any given frequency, leading to sudden transitions between them as the driving force or frequency changes, creating opportunities for switching and memory applications. Harnessing such jumps can enhance sensor performance by providing operating modes or states depending on environmental conditions.

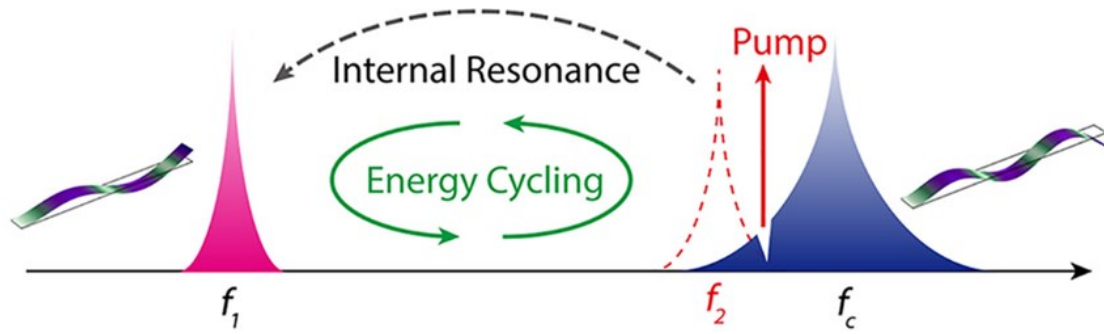


Figure 1-5 Schematic of the internal resonance system, the energy is cycled between two modals³⁹

NEMS resonators can exhibit another important nonlinear behavior called internal resonance (Fig. 1-5).³⁹ This phenomenon occurs when two or more vibrational modes interact strongly due to nonlinear coupling, leading to energy transfer between modes that results in complex dynamic behavior. If the frequency of one mode is an integer multiple or simple fraction of another mode's frequency, internal resonance could occur and cause significant variations to both its amplitude and stability of vibration. Understanding and exploiting internal resonance of NEMS resonators is especially useful for applications requiring increased responsivity or specific frequency responses, such as mass sensing, frequency mixing, or signal amplification.

1.1.6 Tuning resonant properties

The efficient and often unprecedented performance of NEMS resonators in various applications is attributed to their small mass and rich dynamic behavior. The ability to tune the core resonant properties of these NEMS resonators, such as their resonance frequency and nonlinearity, plays a vital role in enhancing their functionality.⁶

A common strategy for tuning the resonance frequency of NEMS resonators involves employing various physical mechanisms, such as mechanical stress,^{40,41} electrostatic force,⁴² and thermal and electrothermal effects, to manipulate the axial strain on the NEMS resonator.^{43,44} The natural frequencies of NEMS resonators are strongly influenced by axial stress, unlike their micrometer-scale counterparts, where natural frequency is predominantly governed by the elastic stiffness determined by the material and geometry.⁶ Therefore, post-fabrication mechanisms capable of manipulating axial strain on the NEMS resonator provide easy access to resonance frequency tuning.

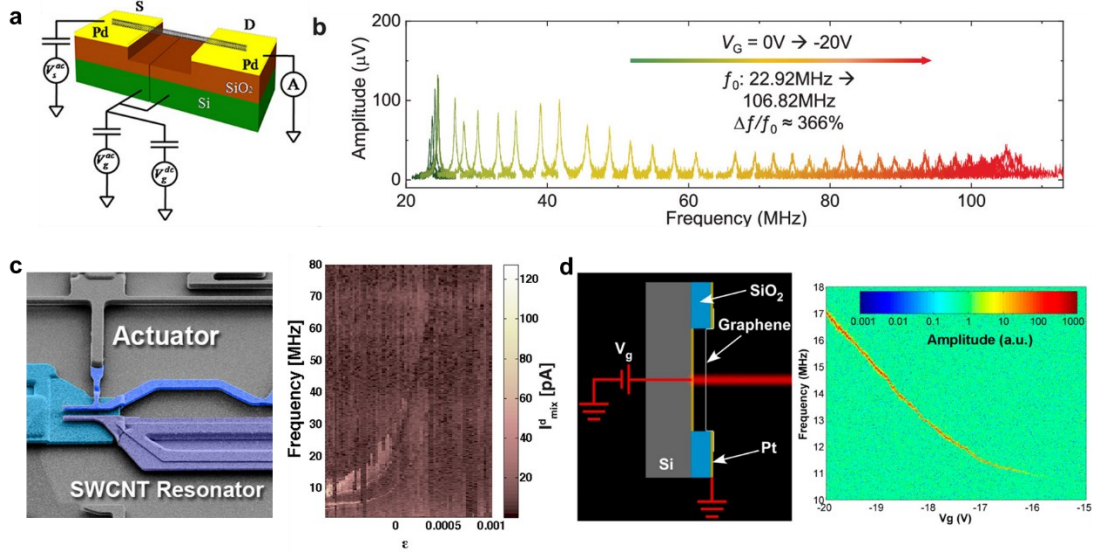


Figure 1-6 Resonance frequency tuning in NEMS resonators. a. schematic of a CNT NEMS resonator's axial strain tuned by a piezoelectric substrate.⁴⁰ b. ultrawide frequency tuning range reported in a heterostructure NEMS resonator.⁶⁶ c. using a MEMS electrothermal actuator to apply in-plane tension for strain modification.⁴¹ d. electrostatic force applied to tune the resonance frequency.⁴²

For instance, Ning et al. demonstrated tuning the resonance frequency of carbon nanotube NEMS resonators using a piezoelectric substrate to exert axial strain, achieving a 20 times frequency adjustment.⁴⁰ Similarly, Truax et al. reported a mechanical strain modification strategy using a MEMS electrothermal actuator to apply in-plane tension to a carbon nanotube resonator, resulting in an increase in resonance frequency from 10 MHz to 60 MHz.⁴¹ Additionally, the application of electrostatic force has been shown to effectively tune the resonance frequency of graphene NEMS resonators by up to 58% by varying a DC gate voltage between the resonator and a nearby gate electrode.⁴² Other studies have shown that the resonance frequencies of MoS₂ and graphene resonators can be tuned by varying thermal pulses or temperatures.

Table 1-2 List of electrostatic tuning of resonance frequency by gate-voltage at room temperature
in some atomically thin and other NEMS resonators

Reference	NEMS resonator Material	Frequency tuning range (T_R)	Voltage (V)	Average frequency tuning efficiency (T_e) (%V ⁻¹)
[44]	Graphene	157%	10	15.7
[45]	Graphene	140.7%	8	17.6
[46]	Graphene	58.3%	16	3.6
[47]	Graphene	58.7%	7	8.4
[42]	Graphene	51.9%	10	5.2
[48]	Graphene	28%	5	5.6
[49]	Graphene	14%	1.3	10.8
[50]	Graphene	12.9%	6	2.16
[51]	MoS ₂	5.35%	5	1.1
[52]	MoS ₂	154%	30	5.1
[53]	MoS ₂	153.3%	30	5.1
[54]	MoS ₂	84.3%	30	2.8
[55]	MoS ₂	25.5%	20	1.3
[66]	Graphene/MoS ₂ Heterostructure	366.4%	20	18.3
[56]	Graphene/MoS ₂ Heterostructure	20%	10	2
[57]	MoSe ₂	12.2%	40	0.3
[58]	MoTe ₂	12.3%	16	0.8
[59]	WSe ₂	231%	10	23.1
[60]	Black P	163%	30	5.4
[61]	CNT	116%	8	14.5
[62]	SnO ₂ NW	1.5%	5	0.3
[63]	SiC beam	6%	30	0.2
[64]	Si beam	2.3%	4	0.6
[65]	Si beam	0.8%	20	0.04

Ultrawide tunability of resonance frequency is predominantly achieved in atomically thin material-based resonators, which are notably sensitive to strain variations due to their extreme aspect ratio, leading to negligible elastic stiffness. Resonance frequency tuning ranges exceeding 100% have been reported using purely electrostatic forces at room temperature in graphene, black phosphorus, and MoS₂ resonators. The table below summarizes the electrostatic tuning of resonance

frequency by gate voltage at room temperature in some atomically thin and other nanoresonators. Notably, atomically thin material-based resonators, especially graphene and its heterostructures, demonstrate superior performance in both tuning range and efficiency compared to more traditional silicon-based resonators. As shown in Fig. 1-6b, the ultrawide electrostatic tuning range of frequency around 366.4% is reported in a Graphene/MoS₂ heterostructure NEMS resonator.⁶⁶

Nonlinearity in NEMS resonators can also be tuned by adjusting the gate voltage. As shown in Fig. 1-6b, the cubic nonlinearity (a) of a bilayer MoS₂ resonator has been shown to be manipulated between hardening and softening types and even nullified by changing the gate voltage.⁶⁷

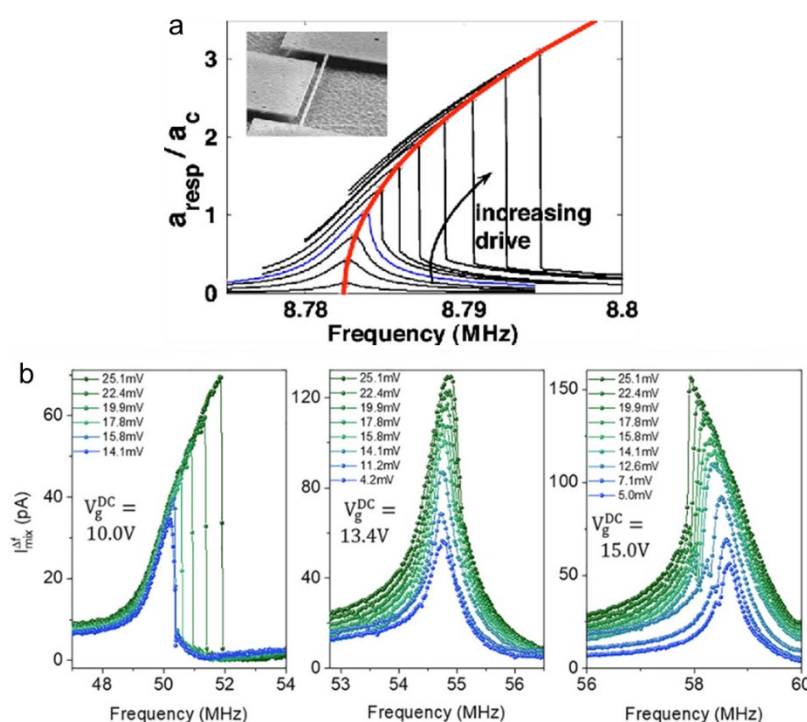


Figure 1-7 Tuning of nonlinearity in NEMS resonator. a. Gate voltage-induced nonlinearity tuning reported in SiC NEMS resonator.⁶³ b. Tuning nonlinearity of a MoS₂ NEMS resonator between hardening and softening types and even nullified by changing gate voltage.⁶⁷

Achieving practical means to attain significant resonance frequency and nonlinearity tunability at the application stage of NEMS resonators promises potential benefits for numerous practical applications. These include improving NEMS resonator-based sensor responsivity, developing filters with adjustable bandwidths, mode-matching in inertial sensors, enhancing the efficiency of mechanical energy harvesting devices, impedance matching in nanoelectronic circuits, broadening the linear dynamic range, and enhancing frequency stability in timing devices.⁶⁸

1.2 NEMS resonator for gas sensing

With the rapid development of science, technology, and the national economy, the risk of increasing environmental problems have become more prominent. Quick, accurate detection of harmful gases in the air is increasingly important, highlighting the role of gas sensors in environmental and health monitoring. In 1967, Nathanson et al. used semiconductor technology to fabricate silicon-based cantilever beam resonators, initially used for sensing applications.⁶⁹ The emergence of this cantilever beam resonator prompted a wave of research on gas sensors among scientists worldwide.

1.2.1 Sensing mechanism and metrics

NEMS resonators detect gases through a mechanism primarily based on mass loading. When gas molecules adsorb onto the surface of a NEMS resonator, they add mass to the system, causing a shift in the resonance frequency. This shift is proportional to the amount of gas adsorbed, allowing for the quantification of gas concentration. Fig. 1-8 describes the gas sensing mechanism with a nanobeam type NEMS resonator.³³

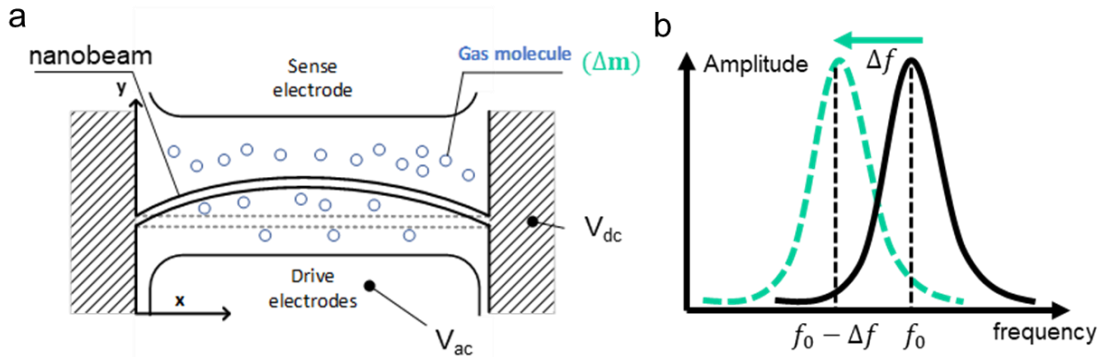


Figure 1-8 The sensing mechanism explained by a nanobeam type NEMS resonator

Several metrics are used to evaluate the sensing performance of NEMS resonator gas sensors:

Responsivity: Responsivity can be defined as the ratio of change in resonance frequency per unit mass change, estimated as $-\omega_0/2m_{eff}$.⁷⁰ Achieving high responsivity is crucial for detecting small mass shifts and can often be improved by decreasing the resonator's mass and increasing the resonance frequency.

Detection resolution/limit: This refers to the minimum mass or gas concentration that can be reliably identified. It is influenced by the noise characteristics of the measurement system and the

responsivity of the resonator (i.e. mass resonance frequency and nonlinearity of resonator).⁷⁰

Selectivity: This refers to a sensor's ability of the sensor to distinguish between various kinds of molecules. Selectivity can be increased by coating resonator surfaces with specific materials that more easily attach themselves to target molecules.

Response Time: The response time of sensors when responding to changes in mass or gas concentration. Fast response times are essential for real-time monitoring applications.

Stability: Stability refers to a sensor's ability to maintain consistent performance over time and in various environmental conditions. Due to minimal mechanical losses during vibration, silicon beam-type structures generally offer high quality factors, repeatability, and stability with consistent performance over time.

1.2.2 Sensing performance enhancement

Enhancing the performance of NEMS resonator sensors involves several strategies to improve responsivity, selectivity, and stability while reducing the detection resolution and response time. These strategies include:

Material Selection and Surface Functionalization:

Choosing resonator materials with high Q-factors, such as silicon carbide or diamond, can significantly enhance stability. Surface functionalization with selective coatings or molecular recognition elements can improve selectivity and reduce the limit of detection. The sensitive layer's material and thickness on the resonator surface are crucial for targeting different gases effectively. For example, Mirza et al. designed a CMOS MEMS cantilever resonant gas sensor for carbon dioxide detection, testing the responsivity of the sensor with a 5 μm polymer layer.⁷¹ Dorsey et al. used thermal evaporation deposition to obtain a polymer sensitive layer on a semiconductor cantilever, optimizing the polymer layer to improve responsivity and stability.⁷²

Geometric Optimization:

Designing resonators with higher aspect ratios and lower masses can enhance responsivity. For instance, reducing the thickness of the resonator while maintaining its structural integrity can lead to higher frequency shifts for a given mass change. This is the reason why NEMS resonator with ultrathin material exhibits ultrahigh responsivity and becomes good candidate for gas sensing application.³³

Multiplexing and Array Configurations:

Employing arrays of NEMS resonators with different functionalisations or integrating multiple target-sensitive resonators into a single sensor can enable simultaneous detection of multiple target molecules, enhancing the overall sensing capability. For instance, Potyrailo et al. designed a gas sensor with dual sensitive layers for simultaneous detection of two target gases, significantly enhancing sensor integration.⁷³ Similarly, Hagleitner et al. integrated three different detection principle-based chemical microsensors on a single chip, significantly improving overall sensor performance by allowing control and monitoring of sensor functions, as well as on-chip signal amplification and adjustment.⁷⁴

Advanced Sensing Modes and Excitation Methods:

Utilizing nonlinear driving methods and higher-order mode excitation can significantly enhance sensor performance. Venstra et al. demonstrated that the frequency shift caused by adsorption and desorption under nonlinear drive increased threefold compared to conventional modes, significantly enhancing sensor performance.⁷⁵

In conclusion, the performance enhancement of NEMS resonator-based gas sensors can be divided into two main categories. The first category focuses on optimizing the structure and resonant properties of the resonator itself. This involves striving for a smaller mass and higher resonance frequency, which in turn leads to greater responsivity and a lower detection limit. By refining the resonator's geometry, such as minimizing its size, the surface area is increased, enhancing mass loading and improving the sensor's responsivity.

The second category centers on material optimization.³³ Choosing the appropriate materials for the resonator can significantly improve its stability. High mechanical quality factor materials like silicon carbide or diamond help reduce energy losses, thus maintaining high responsivity over longer periods. Additionally, enhancing selectivity is achieved by coating the resonator with specific chemical or biological sensitive layers.

1.3 Motivation

Over the past few years, the demand for gas detection and monitoring has been growing, driven by the growing concerns of human health and medical purposes. NEMS resonators can perform as high-performance gas sensors because of their ultrasmall mass. It offers an opportunity to monitor the parts-per-trillion level variation of the gases in human breath by working as an electronic nose.

As mentioned in section 1.2.1, the main metrics of the resonator gas sensors are responsivity and detection limit, while they are highly affected by the resonator's mass, resonance frequency and nonlinearity. Tunable NEMS resonators, which allow for the adjustment of resonance frequency and nonlinearity during the operation of resonators, offer a promising opportunity to improve sensing performance further in the post-fabrication stage. The prominent wide tunability in resonant properties is usually achieved in atomically-thin materials-based resonators, such as graphene, MoS₂, CNT, WSe₂, etc. as indicated in Table 1-2. Unfortunately, integrating atomically thin materials in NEMS resonator devices requires a complex and specialized fabrication process (section 1.1.3), which, combined with a lack of wafer-scale single-crystal-quality base materials, restricts the ubiquitous application of tunable NEMS resonators. Besides, complex and non-standardized fabrication routines also bring atomically-thin resonators unpredictable dynamic behaviours and reduce resonance quality factors. Thus, achieving comparable electrostatic tunability in conventional monolithically microfabricated resonator devices, especially from Si, which offers wafer-scale, inexpensive, single-crystal-quality material and scalable monolithic fabrication process is desired but considered challenging.

This research aims to develop ultrathin Silicon NEMS resonators with ultrawide tunability in resonant properties for gas sensing applications. Specifically, it can be divided into 4 specific goals:

- 1) To develop a scalable and monolithic fabrication process for creating Si NEMS resonators with ultrathin width and high aspect ratio using standard microfabrication tools and to build the necessary experimental setup for conducting a thorough characterization of the resonance properties of the fabricated ultrathin Si NEMS resonators under various conditions to comprehensively study the dynamic behaviours
- 2) By building an effective characterization set up, to experimentally demonstrate wide resonance frequency tunability in the fabricated ultrathin Si NEMS resonators and to build adequate theoretical models to elucidate the underlying tuning mechanism.
- 3) To achieve wide tunability in the nonlinear behavior of the ultrathin Si NEMS resonators.
- 4) Examining gas sensing ability of ultrathin Si NEMS resonators and exploring the role of tunability to enhance sensing performance.

1.4 Thesis outline

- (1) In chapter 1, an overview of NEMS resonators' basic structures, actuation/detection methods, fabrication technologies, resonant properties and the tunability in resonance frequency and nonlinearity is introduced. Motivated by the goal of developing Si NEMS resonator with widely tunable resonant properties for gas sensing application, the sensing mechanism of resonator gas sensors, performance metrics such as responsivity and detection limit, and their enhancement strategies are also introduced.
- (2) Chapter 2 focuses on the fabrication and characterization of ultrathin Si NEMS resonators. The details of fabrication process and actuation/detection system are provided. The device with smallest width of 20 nm and maximum aspect ratio (length/width) of 15000 is successfully fabricated. The typical resonance response of the fabricated devices is discussed to reveal the dynamic characteristics of the ultrathin Si NEMS resonators.
- (3) Chapter 3 experimentally demonstrates the ultrawide tunability in resonance frequency achieved in our devices. By applying DC gate voltages between the actuation electrode and NEMS resonator structure, ultrawide electrostatic tuning ($\sim 70\%$) of initial resonance-frequency ($\sim 7\% \text{ V}^{-1}$) at room temperature is observed in a fabricated ultrathin Si NEMS (width $\sim 40 \text{ nm}$, length $\sim 200 \text{ }\mu\text{m}$) resonator device. An approximate electromechanical model is established to explain the tuning mechanism and explore the tuning behaviours under various conditions.
- (4) In Chapter 4, the nonlinearity tuning observed in our devices is presented. The approximate cancellation of nonlinearity, achievable by flipping the sign of nonlinearity, is discussed. The broad linear dynamic range of approximately 86 dB achieved in a device with a width of 100 nm and a length of 100 μm around this cancellation point is also examined. Furthermore, the internal resonance observed in our devices is presented.
- (5) After experimentally examine the CO_2 gas sensing ability of our deices, Chapter 5 proposes a gas sensing performance enhancement strategy based on the ultrawide tunability in resonant properties demonstrated in our devices.
- (6) Conclusions are presented in chapter 6 together with recommendations for future works.

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Chapter 2 Development of Fabrication and Characterization Methods of

Ultrathin Silicon NEMS Resonators

2.1 Introduction

In recent years, ultrathin NEMS resonators have garnered considerable interest due to their numerous advantages and potential applications in various fields.¹ These resonators, characterized by their nanometer-scale widths, offer exceptional responsivity and high-frequency operation, making them ideal for applications in sensing, signal processing, quantum computing, etc.² Their ultra-low mass and enhanced mechanical properties enable precise detection of minute perturbations in physical, chemical, and biological domain, which is crucial for developing advanced sensors and actuators.¹

Traditionally, ultrathin NEMS resonators developed from atomically thin two-dimensional (2D) materials such as graphene,³ molybdenum disulfide (MoS_2),⁴ hexagonal boron nitride (h-BN),⁵ etc. have been widely studied. These materials possess exceptional electrical, mechanical, and thermal properties, making them suitable for high-performance NEMS devices. The typical fabrication process for 2D resonators involves mechanical exfoliation or chemical vapor deposition (CVD) to obtain monolayer or few-layer films, followed by transfer techniques to place these films onto substrates.⁶ Photolithography and electron-beam lithography are then used to define the resonator structures. However, the fabrication of 2D material resonators comes with several challenges. The transfer process can introduce contaminants, wrinkles, and cracks, affecting the quality and performance of the resonators. Additionally, achieving uniform and large-area coverage with 2D materials remains challenging, limiting their scalability and integration into commercial devices.⁷ These limitations highlight the need for approaches that can improve the performance and functionality of monolithically fabricated NEMS resonators.

Monolithic fabrication of NEMS resonators, where the entire device is built from a single substrate without transfer steps, presents a promising solution. This approach can potentially offer better structural integrity, scalability, and compatibility with existing semiconductor manufacturing processes.⁸ However, monolithic fabrication of ultrathin NEMS resonators is challenging due to the rigorous requirements for precision and control in nanoscale patterning and etching processes.⁹

Achieving the desired device dimensions and properties while avoiding damage and contamination requires advanced fabrication techniques and careful process optimization. Silicon-based NEMS resonators provide a compelling alternative to 2D material resonators. Silicon (Si), with its well-established fabrication techniques and compatibility with integrated circuits, offers several advantages.⁹ Si resonators can be fabricated using silicon-on-insulator (SOI) wafers, allowing for precise control over device dimensions and properties. The use of conventional Si microfabrication methods, such as, photolithography, electron-beam lithography, and deep-reactive-ion-etching (DRIE) can potentially enable the creation of high-quality, ultrathin Si resonators with excellent electromechanical performance.¹⁰

The purpose of this chapter is to establish the fabrication and characterization processes of ultrathin Si NEMS resonators. We begin by describing the structural design of the resonators, followed by a step-by-step account of the fabrication process using SOI wafers. Challenges encountered during fabrication and the solutions implemented to address these issues are discussed. Subsequently, we present a method for the characterization of the resonant properties of the fabricated resonators, which will allow us to explore their resonance frequency, quality factor, and dynamic behaviors under various conditions described in this thesis.

2.2 Device structure and fabrication

2.2.1 Structure of ultrathin Si NEMS resonator

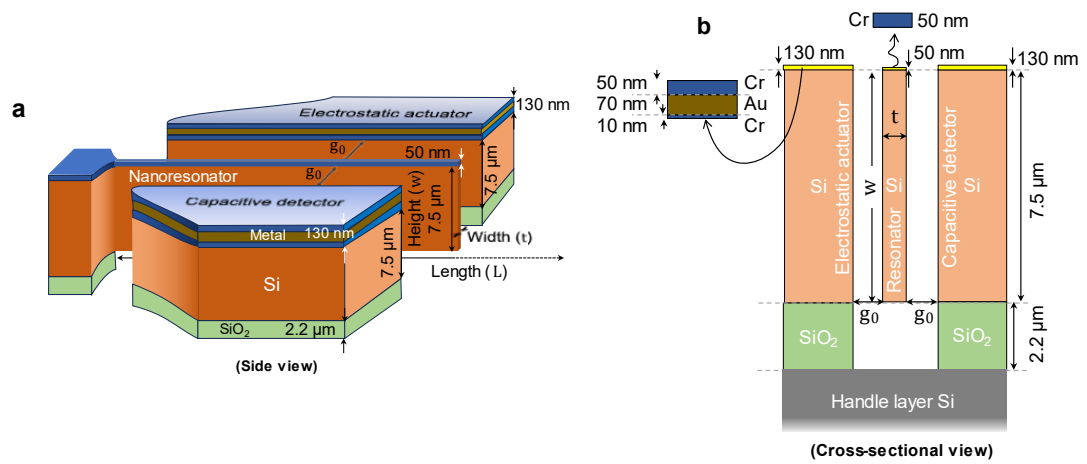


Figure 2-1 Schematic illustration of a Si NEMS resonator device structure from (a) side view, (b) cross-sectional view.

The ultrathin Si NEMS resonator devices consist of (a) a mechanically vibrating nanostructure (which will be referred as nanoresonator), (b) an actuation electrode (a side-gate) for inducing electrostatic actuation, and (c) a capacitive detector electrode (another side-gate) for capacitively detecting the nanomechanical motion of the nanoresonator. The material of Si nanostructure and electrodes is n-type single-crystal Si (details given later). A schematic illustration of the device structure as seen from the side-view is given in Figure 2-1 a. Across devices, we vary the width (t) of the resonators from 20 – 300 nm and the length of the nanoresonators (L) from 100 – 300 μm , while all resonators share similar heights (w) of ~ 7.5 μm . We also vary the gap-size between the side-gate electrodes and the nanoresonator (g_0) between 1 – 3 μm . Metal thin films are deposited on top of all the electrodes (130 nm Cr/Au/Cr) and the nanoresonator structure (50 nm Cr) for making electrical connections between the device and external electronic components. The relative position of device components and heights of each material layer is outlined in a schematic illustration of the cross-sectional view of the device in Figure 2-1 b.

2.2.2 Fabrication process

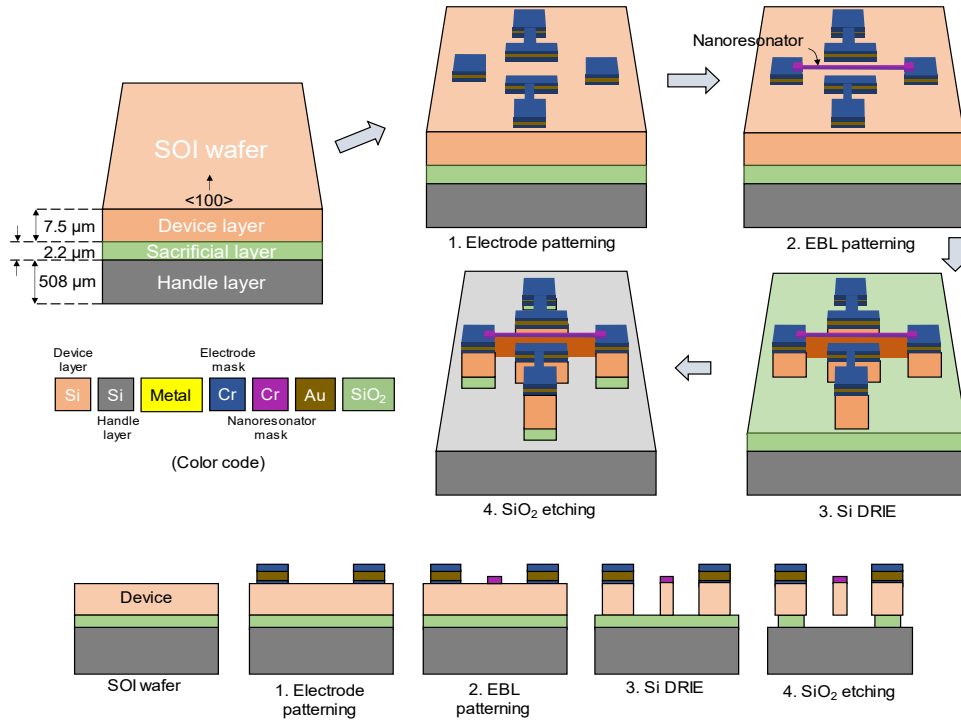


Figure 2-2 Schematic illustration of the device fabrication process. Device fabrication steps: devices are fabricated from SOI wafers in our cleanrooms by metal electrode patterning through photolithography, nanoresonator patterning by electron-beam lithography, and deep-reactive-ion-etching (DRIE) of device layer, and etching of sacrificial SiO₂ layer by vapor HF.

The ultrathin Si NEMS resonator devices presented in this work are fabricated from 4 inch (dia.) silicon-on-insulator (SOI) wafers purchased from IceMOS Technology. SOI wafers consist of a n-type (dopant, As; doping concentration $2.86 \times 10^{19} \text{ cm}^{-3} - 3.09 \times 10^{19} \text{ cm}^{-3}$) single-crystal (100) Si device layer of $7.5 \pm 0.5 \text{ }\mu\text{m}$ thickness and (manufacturer specified) resistivity in the range of $0.0022 - 0.0023 \text{ }\Omega\text{-cm}$, a n-type (dopant, P) handle layer of $508 \pm 5 \text{ }\mu\text{m}$ thickness made of high-resistivity ($5000 - 10000 \text{ }\Omega\text{-cm}$) Si, and a sacrificial SiO_2 layer of $2.2 \pm 0.1 \text{ }\mu\text{m}$ thickness. Thin, native oxide layer ($\sim 1 \text{ nm}$) on the device layer and the thick oxide layer ($\sim 2 \text{ }\mu\text{m}$) on the backside of the handle layer are first removed by submerging the wafers in buffered hydrofluoric acid (bHF). The purpose of removing the native oxide on the device layer is to improve adhesion of the metal-films subsequently deposited as an electrode material, whereas the primary motivation for removing the thick oxide-layer on the backside of the handle layer is to reduce H_2O vapor production during sacrificial layer etching, thus, reducing the tendency of sticking of the Si nanoresonators on the nearby gate-electrodes. Subsequently, SOI wafers are cleaned by acetone, IPA, O_2 -plasma, and finally by Piranha solution. NEMS resonator devices consisting of a double-clamped (c-c) nanoresonator and side-gate electrodes on each-side separated by a micro-scale gap (used for actuation and sensing of vibration) are fabricated in our cleanrooms by the following steps shown in Figure 2-2:

Step 1. Patterning of metal electrodes, and device areas other than the nanoresonator

Metal electrodes are patterned by UV lithography process (Union Optical, PEM-800) using a chromium photomask created by direct laser writing (Heidelberg Instruments, DWL2000) and an image reversal photoresist (AZ5214E). Then, Cr/Au/Cr thin films (thickness 10 nm/ 70 nm/ 50 nm, respectively) are deposited by electron-beam deposition (while the bottom Cr film improves adhesion between Si and Au film, the top Cr film acts as mask material during the subsequent deep-reactive-ion-etching process). Finally, the photoresist is removed by n-methyl-2-pyrrolidone (NMP) solution. Details of the process recipe are provided below:

Table 2-1 Process recipe for metal layer deposition

Spin coating	Dehydration of wafers	150° C, 5 min on hotplate
	Hexamethyldisilazane (HMDS) coating	3000 rpm, 60 s on spin-coater
	AZ5214E coating	4000 rpm, 30 s on spin-coater
	Pre-bake	110° C, 1 min on hotplate
First UV exposure	Exposure with photomask	8.6 mJ/cm ²
	Reverse baking	117° C, 2 min on hotplate
Second UV exposure	Flood exposure without a photomask	365 mJ/cm ²
Development	NMD-3 (Tetramethylammonium Hydroxide (TMAH), 2.38%)	36 s
	Deionized (DI) water	90 s + 30 s
Metal deposition	Electron-beam evaporation	Cr (10 nm), Au (70 nm), Cr (50 nm)
Lift off	NMP	80° C, shaker (20 rpm)

Step 2. Nanoresonator patterning by electron-beam lithography

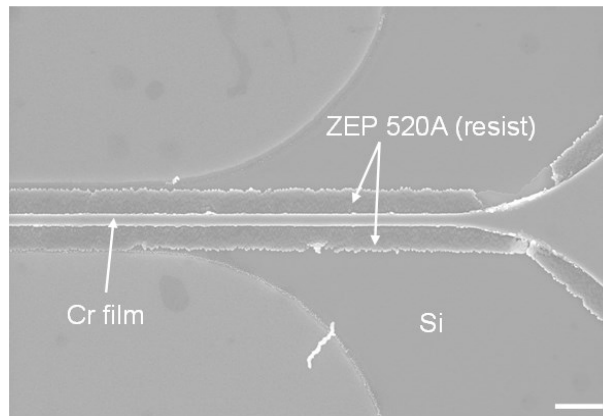


Figure 2-3 Scanning electron microscopy (SEM) image of a device under fabrication after nanoresonator patterning (scale bar = 1 μ m). Cr film (50 nm) is deposited on the sample after electron-beam lithography process using ZEP 520A resist. Following a subsequent Liftoff process using NMP solution, a Cr strip that defines the nanoresonator structure is achieved between the two gate-electrodes. ZEP 520A residue observed around the edges of the Cr strip is removed by piranha cleaning.

Nanoresonator structures are patterned by electron-beam lithography (EBL). SOI wafers with metal electrodes and alignment marks patterned on them in the previous step are first cleaned with

piranha solution. A high-resolution positive electron-beam resist (ZEP 520A) of ~ 300 nm thickness is spin-coated on the samples. Electron-beam exposure is done by electron-beam lithography system (Elionix, ELS-F125HS) using global-alignment marks ('Reg-2' marks) that were patterned in the previous step. Samples are developed by ZED-N50 developer after electron-beam exposure. 50 nm thick Cr film is evaporated on the sample by an electron-beam evaporation system and liftoff is carried out by submerging the samples in hot NMP solution. As a result of this process, Cr masks for nanoresonator are patterned on the device layer. Usually, resist residue remains around the edges of the Cr film (Figure 2-3), which is removed by piranha solution. Alternatively, the resist residue can be removed by ultrasonication for 1 – 5 minutes in NMP solution, but this process damages the Cr film as it leads to delamination or rugged edges, thus not recommended. Details of the process recipe are given below:

Table 2-2 Process recipe for nanoresonator patterning

Spin coating	Dehydration of wafers	180° C, 3 min on hotplate
	ZEP 520A coating	5000 rpm, 80 s on spin-coater
	Pre-bake	180° C, 3 min on hotplate
EBL exposure	Elionix, ELS-F125HS	200 keV, 20 pA, dose time: 0.024 ms
Development	ZED-N50	165 s
Metal deposition	Electron-beam evaporation	Cr (50 nm)
Liftoff	NMP	80° C, shaker (20 rpm)

Step 3. Deep-reactive-ion-etching (DRIE) of the device layer

Si device layer is etched by deep-reactive-ion-etching (Samco, RIE-800iPB-KU) using the Cr films deposited in previous steps as mask material. The process successfully etched ~ 7.5 μm depth of Si device layer for 20 – 300 nm wide and 100 – 300 μm long strips of Cr mask and generated almost vertical sidewalls. Following the DRIE process, the devices were decontaminated by O₂ plasma (50 W, 5 min $\times$ 4 times). Caution should be taken to avoid over-etching of the device layer during the DRIE process. Although for microscale structures slight over-etching usually does not

cause a discernable difference in etched structures, it was found in case of our nanoresonators that over-etching causes detachment of the nanoresonators from their SiO₂ base. This detachment prematurely releases the nanoresonators by removing the bottom part of the nanoresonators.

Micro-masking effect is a common phenomenon observed during the DRIE process, particularly when using metal hard masks such as Cr.¹¹ This effect results in the formation of undesired high-aspect-ratio features, such as needle-like Si structures (Si whiskers), which can significantly impact the fidelity and functionality of the etched patterns. This phenomenon was observed in our initial fabrication, as shown in Figure 2-4 (a). The micro-masking effect occurs when small particles or debris from the hard mask redeposit onto the Si surface during the etching process. These particles act as micro-masks, shielding the Si beneath them from being etched while the surrounding Si continues to be etched away. Several key factors contribute to this effect:

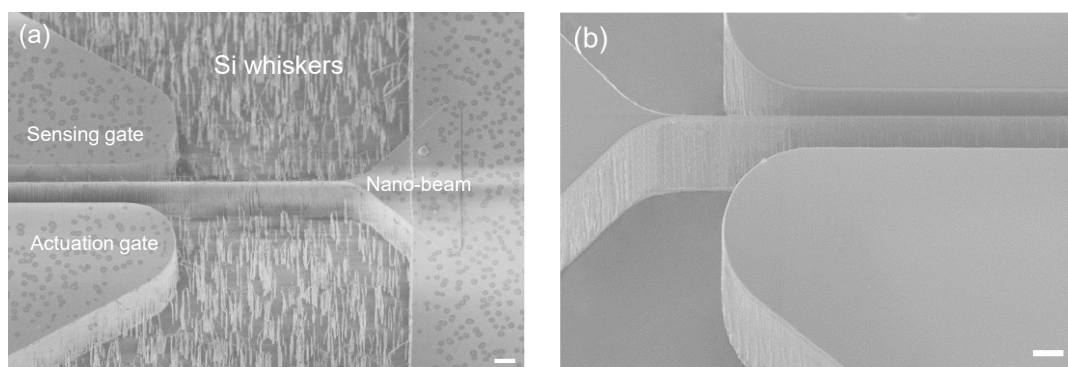


Figure 2-4 Scanning electron microscopy images showing (a) micro-masking problem (undesired needle-like Si nanostructures appearing in around the device areas) encountered after the Deep-RIE process. (b) micro masking problem has been solved by optimizing the DRIE parameters. Scale bar = 2 μm .

Hard Mask Erosion: Erosion of the hard mask by plasma can cause particles to redeposit onto the Si surface, creating localized areas that are protected from etching.

Particle Contamination: Particles present in the chamber can settle on the Si surface and act as micro-masks.

Plasma Chemistry Byproducts: Byproducts from the plasma chemistry during the Deep-RIE process can also redeposit onto the Si surface, contributing to the micro-masking effect.

To minimize the impact of the micro-masking effect during Deep-RIE, maintaining a clean etching chamber and fine-tuning the etching parameters—such as gas composition, pressure, power,

and etching time—can help achieve a more uniform etch rate and reduce the formation of micro-masks. By maintaining a clean Deep-RIE chamber and optimizing the parameters, the micro-masking problem was greatly improved in our fabrication. The improved results are shown in Figure 2-4(b), where it is clear that the needle-like structures have been dramatically mitigated around the Si nanobeam. Discuss recipe modification of DRIE process that solved micromasking issue.

Step 4. Sacrificial layer etching by vapor HF

Si nanoresonators are released by etching the underlying SiO₂ film by vapor HF (Sumitomo Precision Products, MLT-SLE-Ox). The vapor HF etching process mitigates the risk of stiction in released movable components and prevents harm to fragile nanostructures by using a mixture of vapor HF and ethanol vapor for etching SiO₂. Unlike microbeam resonators, we found that nanoresonators presented in this work are more susceptible to sticking (onto one of the side-gates) and damage during this process. We suspected that H₂O vapor released as a byproduct of this etching process might be responsible for this problem. Therefore, we took extra cautions to minimize the rate and amount of H₂O vapor release, by reducing the HF concentration, introducing more evacuation steps in the etching cycle, and by removing the SiO₂ layer from the backside of handle-layer Si before starting the fabrication process. In addition, samples were divided into multiple batches based on their width and etched separately for just enough time to release the widest beam of the batch.

2.2.3 Choice of device parameters

A few points regarding the consequences of the choices made in device design parameters and fabrication process adopted for this study should be highlighted. It is worth noting that the device dimensions adopted here are noticeably different from atomically thin resonators as well as conventional nanoresonators that implement similar actuation and detection schemes. For example, the gap between the resonator and the actuation/ detection electrodes (g_0) is $\sim 1\ \mu\text{m}$, which is an order of magnitude larger than typical nanoresonators.⁹ Adopting a larger value for g_0 could be detrimental to achieving efficient actuation and detection of nanomechanical motion (because it reduces the capacitance between the nanoresonator and actuation/ detection electrodes), however, this choice is intentional in our case despite having sufficient patterning ability to adopt a much smaller g_0 (the motivation behind this choice will be discussed in Chapter 3). We adequately

compensate for the choice of larger g_0 by increasing the plate area ($w \times L$) of the gate capacitors: firstly, by choosing a thick device layer (which makes $w \sim 7.5 \mu\text{m}$) and patterning long nanoresonators ($L \sim 100 \mu\text{m}$). Considering the ultrathin width of the nanoresonators that we intended for this study, we had to optimize and take extra caution in fabrications steps 2 – 4 (especially for the DRIE step) details of which are given above. It should also be noted that from a fabrication point of view, choice of g_0 is also related to the choice of device layer thickness (which defines, the dimension w of the nanoresonator, and the thicknesses of the actuator and detector), because for the optimum DRIE parameters (to attain ultrathin nanostructures), sufficient plasma penetration near the bottom of the DRIE trench would require a certain minimum trench width (defined by g_0). It should also be noted that the effect of the electrostatic actuation force on the resonator is also significantly influenced by g_0 . Thus, the device dimensions should be carefully considered to achieve specific performance goals, to mitigate fabrication challenges, and to ensure sufficient detection responsivity.

2.2.4 Result of device fabrication

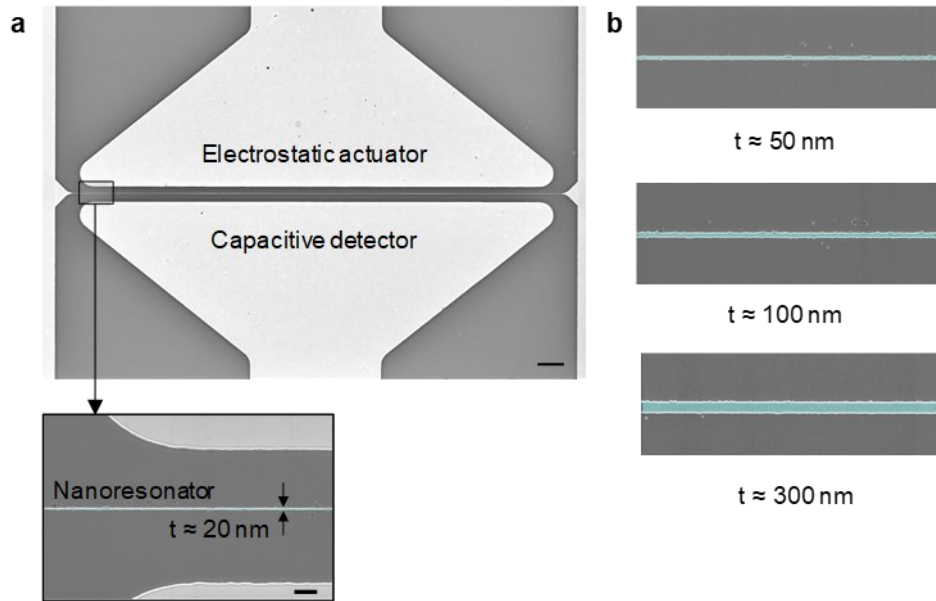


Figure 2-5 Fabricated ultrathin NEMS resonator devices. (a) Scanning electron micrograph of a Si nanoresonator device (taken before releasing the nanoresonator, scale-bar, $10 \mu\text{m}$); $\sim 130 \text{ nm}$ (Cr/Au/Cr) metal films are deposited on the electrode areas and a $\sim 50 \text{ nm}$ (Cr) metal film is present on the top surface of the nanoresonator; inset shows a (false colored) close-up of the $t \sim 20 \text{ nm}$, $L \sim 200 \mu\text{m}$ nanoresonator, the scale-bar is $1 \mu\text{m}$. (b) False-colored scanning electron micrographs of nanoresonators of $t \sim 50 \text{ nm}$, 100 nm , 300 nm , respectively.

Following the fabrication process described in section 2.2.2, Si nanoresonators are successfully fabricated. Across devices, nanoresonator dimensions vary in length (L) (100 – 300 μm) and width (t) (20 – 300 nm) (Figure 2-5); gap-size (g_0) between a side-gate and the static nanoresonator varies (1 – 3 μm); while all devices share identical height (w) (~ 7.5 μm). Actuation and detection electrodes (height ~ 7.5 μm) are situated parallel to the nanoresonator in the same plane at g_0 distances. The maximum aspect ratio (length/width) achieved among our fabricated devices reaches 15000.

2.3 Actuation and read-out methods

2.3.1 Actuation and detection circuit

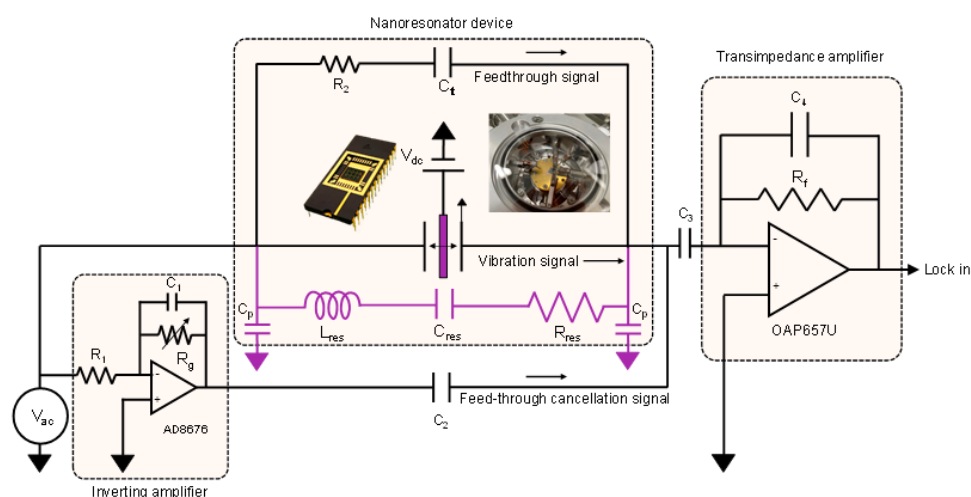


Figure 2-6 Schematic diagram of the vibration amplitude detection circuit. When operated in the linear regime, nanoresonator devices can be represented as a linear LCR circuit (purple lines) with a feedthrough capacitance. Actuation ac signal is simultaneously passed through an inverting amplifier with tunable gain and used for cancelling the feedthrough ac signal in the range of frequency sweep. The resulting signal is finally passed through a transimpedance amplifier to convert it to a voltage signal whose amplitude is then detected by a lock-in amplifier.

To characterize the resonant properties of the fabricated ultrathin nanoresonators one of the anchors of the nanoresonator, a dc bias voltage (V_{dc}) is applied (Agilent, E3647A) via a thin-film metal electrode. An ac voltage of amplitude, V_{ac} , and frequency, f , is applied on the actuation electrode from the output terminal of the lock-in amplifier. This exerts a periodic actuation force ($\sim \frac{C_g^0}{g_0} V_{dc} V_{ac} \cos 2\pi f t$) on the nanoresonator. As the nanoresonator oscillates, the capacitance between the nanoresonator and the detection electrode fluctuates periodically. The periodic change

in capacitance generates a corresponding periodic current signal, which is amplified and converted to a voltage signal through a transimpedance amplifier (TIA) and then analyzed by a lock-in amplifier (Zurich Instruments, MFLI 5MHz) as shown in Figure 2-6.

NEMS resonator device is represented by an LCR circuit as shown (in purple) in Figure 2-6, with a parallel feedthrough branch represented by the feedthrough capacitor (C_{ft}) and parasitic capacitances (C_p). In frequency sweeps, the feedthrough signal via C_{ft} causes a linear background at the output, which can potentially obscure the signal produced by the motion of the nanoresonator. To cancel this background signal, a feedthrough cancellation sub-circuit is implemented. Here, the input ac actuation signal is inverted by an inverting amplifier and added to the output of the resonator. To control this cancellation, the gain of the inverting amplifier is adjusted by R_g .

2.3.2 Capacitive detection efficiency

Having sufficient detection efficiency for detecting the nanomechanical motion of the resonator is crucial for realizing the several practical applications of NEMS resonators. The current actuation and detection scheme of our NEMS resonator is based on similar approaches established in the past for ‘all electronic’ capacitive detection method in MEMS/ NEMS resonators.¹² For example, Truitt et al.¹³ demonstrated that nanomechanical displacement responsivity comparable to optical interferometry and magnetomotive detection methods is possible with capacitive readout technique by implementing methods to efficiently coupling a nanoresonator with macro-scale components. Miller et al.¹⁴ achieved 99.8% thermomechanical-noise-limited resolution in displacement responsivity in MEMS resonator with capacitive scheme with similar size g_0 .

The detection capability of the device relies on the gate capacitance(s) (C_g^0) and the gap-size between resonator and actuation / detection electrodes (g_0). In general, maximizing C_g^0/g_0 ratio is beneficial for detection, but the choice depends on device’s design, operational principle, and limitations arising from the device fabrication process as we discussed. In our typical resonator $\frac{C_g^0}{g_0} \sim 3.3 \text{ aF/nm}$, which is similar to many atomically thin resonators and microfabricated resonators. However, in practical applications, managing noise, efficient coupling between components (impedance mismatches), and electrical losses can play a significant role in ensuring high detection efficiency and responsivity. Taking the TIA’s noise as a reference point, which in most ideal condition (only considering the thermal noise from the gain-resistor, R_f at room temperature), is

expected to produce an approximate noise amplitude spectral density $\sim 100 \text{ nV/Hz}^{1/2}$ in the 100 kHz – 400 kHz frequency range (estimated by LTspice software, by Analog Devices, Inc., USA), the ideal displacement responsivity in our typical device is $\sim 10^{-12} \text{ m/Hz}^{1/2}$ (at $V_{dc} = 10 \text{ V}$). However, this estimation ignores the parasitic input current noise to the TIA, which can form a significant part of the noise from the TIA; it also ignores the noise from other equipment; thus, the $10^{-12} \text{ m/Hz}^{1/2}$ value could be a significant overestimation (upper limit) of the intrinsic displacement responsivity of our current system. There is also indication of significant parasitic losses. The motional resistance of the nanoresonators is typically in the $\sim 100 \text{ k}\Omega$ order (around $V_{dc} = 10 \text{ V}$) indicating a mismatch with the lock-in. Thus, although with our current detection system with the lock-in amplifier, we are able to drive and detect NEMS resonators' frequency response at moderate ac ($\sim 1 \text{ mV}$) and dc ($\sim 10 \text{ V}$) voltages and observe their dynamic behaviors, there is scope to further optimize and enhance the detection responsivity and efficiency of the measurement system, such as by using (a) a more sophisticated TIA design¹⁴ could be considered to achieve a higher S/N ratio in the amplified signal, (b) coupling between the device and the external circuit components could be made more efficient.¹³ These improvements will be beneficial in various practical applications such as, gas sensing, inertial sensing, signal/ information processing, usually a standalone, portable detection system is needed with less sophisticated (compared to lock-in) equipment.

2.4 Characterization of resonant properties

2.4.1 Resonance frequency

Dynamic-behavior studies of fabricated ultrathin Si NEMS resonator devices are conducted inside vacuum-chambers under various pressure (P) ($10^{-1} - 10^{-4} \text{ Pa}$) and temperature (T) ($90 - 300 \text{ K}$) conditions. To vibrate a Si nanoresonator, a dc-voltage (V_{dc}) and an ac-voltage (V_{ac}) of frequency (f) are applied between the nanoresonator and the actuation electrode ($V_{ac} \ll V_{dc}$). Consequently, the nanoresonator is subjected to static and harmonic forcing. Resulting vibration amplitude ($A(f)$) of the nanoresonator is detected capacitively using the current (I_c) generated at the capacitive detector by a lock-in amplifier after converting it to a proportional voltage signal ($R_f I_c$) via a transimpedance amplifier (TIA). By sweeping f around the resonance-frequency (f_0) of the fundamental in-plane flexural-mode vibration of the nanoresonator, a resonance-peak is

obtained, which is used to identify f_0 and to estimate the Q-factor. A resonance-peak acquired from a fabricated Si NEMS resonator of $t \sim 100$ nm, and $L \sim 200$ μm at $P \sim 1.3 \times 10^{-4}$ Pa, and $T = 95$ K is shown in Figure 2-7. $f_0 \sim 226.83$ kHz and Q-factor ~ 9000 is estimated by fitting a Lorentzian function to the experimental data.^{12,15}

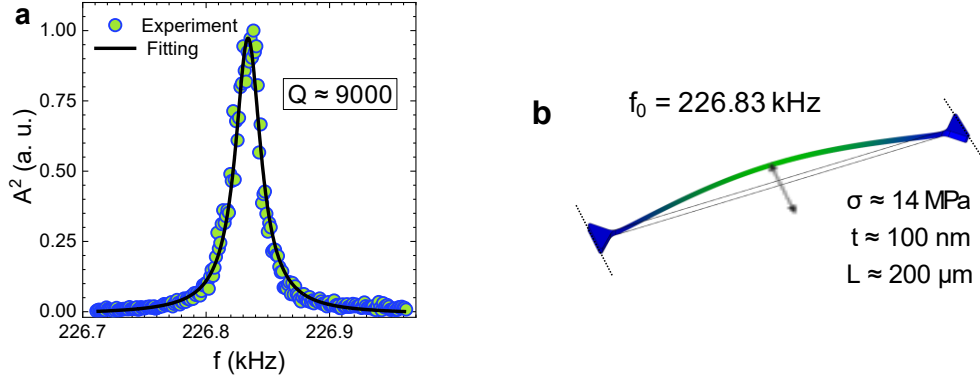


Figure 2-7 (a) A resonance-peak acquired at $P \sim 1.3 \times 10^{-4}$ Pa, $T = 95$ K from a nanoresonator of $t \sim 100$ nm, $L \sim 200$ μm , $w \sim 7.5$ μm . (b) Finite-element simulation of the first in-plane flexural-mode vibration of the nanoresonator described in d with an axial-tensile-stress of ~ 14 MPa.

Natural-frequency (f_{EB}) of slender elastic-structures are evaluated by the conventional Euler-Bernoulli beam model, which, for the case in Figure 2-7a predicts, $f_{\text{EB}} = \frac{\beta_1^2}{2\pi L^2} \sqrt{\frac{EI}{\rho A_{\text{cs}}}} \sim 26$ kHz, where, $E = 170$ GPa, $\rho = 2330$ kgm^{-3} are Young's modulus and density of Si, respectively; $A_{\text{cs}} = wt$, $I = \frac{wt^3}{12}$ are cross-sectional area and second-moment of A_{cs} , respectively; and, $\beta_1 \approx 4.73$. In Figure 2-7a, f_0 is significantly higher than f_{EB} ($\frac{f_0}{f_{\text{EB}}} \approx 9$); $\frac{f_0}{f_{\text{EB}}} > 1$ is observed in all tested devices. Divergence between f_0 and f_{EB} is attributed to an axial-tensile-stress (σ) introduced in the c-c nanoresonators during fabrication and measurement processes. $\sigma \sim 14$ MPa accounts for the f_0 observed in Figure 2-7a as shown in the numerical-simulation result in Figure 2-7b. As nanoresonators' width are narrowed, contribution of elastic-bending-stiffness ($\propto EI$) to their overall mechanical-stiffness (and thus to f_0) diminishes, while the influence of σ on f_0 increases, a phenomenon widely reported in atomically thin resonators^{16,17} and other ultrathin resonators.^{18,19} In σ -dominated regime, where $\frac{f_0}{f_{\text{EB}}} \gg 1$ (~ 10), a nanoresonator essentially behaves like a string. Typical sources of σ are, (a) a native-stress (σ_0 , at room-temperature) introduced during fabrication, mechanism of which can be hard to pinpoint and control, and (b)

electrostatic-force used for actuation, which can be precisely controlled. In current devices, typical values of σ_0 are estimated to be 10 – 40 MPa.

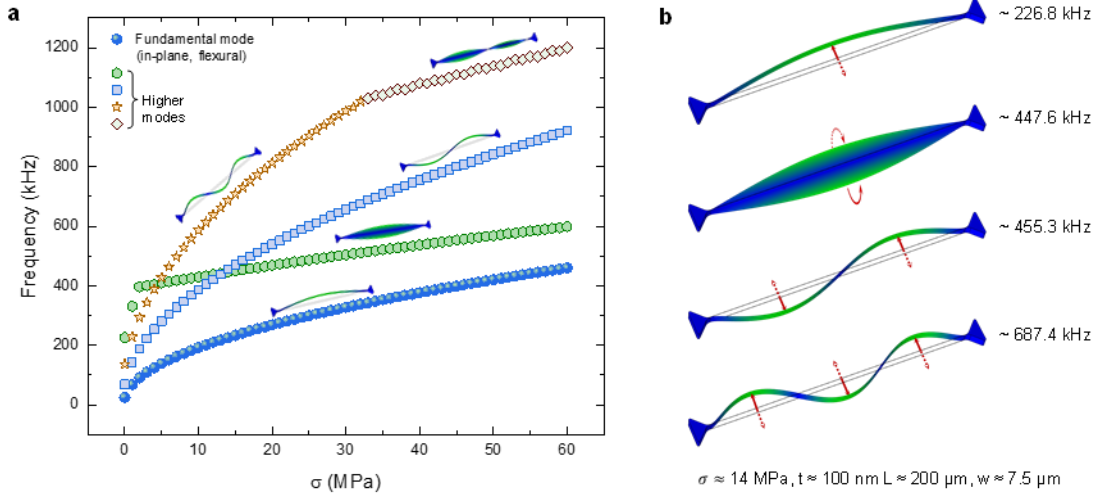


Figure 2-8 Numerically simulated vibrational modes and frequencies of a Si nanoresonator. (a) Mode-shapes and frequencies of a Si nanoresonator corresponding to various axial-tensile-stresses. Nanoresonator dimensions are: $L = 200$ μ m, $w = 7.5$ μ m, and $t = 100$ nm. (b) Four vibrational modes in ascending order of resonance-frequencies for the nanoresonator resonator discussed in Figure 2-7.

Vibration modes of the ultrathin Si NEMS resonator with axial-tensile-stress are further investigated by finite element simulations via Comsol Multiphysics. The lowest (in frequency) four modes of free-vibration and their corresponding frequencies for a nanoresonator of $L = 200$ μ m, $w = 7.5$ μ m, and $t = 100$ nm are shown in Figure 2-7a for $\sigma = 0$ to 60 MPa. The dimensions of this resonator are identical to the one whose resonance peak is shown in Figure 2-7a. It is observed in the simulation results shown in Figure 2-8a that the lowest four modes can potentially include in-plane flexural / torsional vibrations with the in-plane flexural fundamental (1st) mode having the lowest frequency. Modes are mostly spread out in frequency space, and the lowest mode's frequency is significantly separated from higher modes. In our experiments, care was taken to ensure that we measure the lowest peak in frequency. It is worth mentioning that in our capacitive detection scheme, the torsional modes, and the even-numbered (2nd, 4th, ...) in-plane flexural modes are unlikely to be detected. Therefore, we conclude, all the resonance peaks (f_0) reported in this work correspond to in-plane fundamental flexural mode of the nanoresonators. At $\sigma \approx 14$ MPa, simulated frequency

closely matches with the measured resonance frequency in Figure 2-7a. In Figure 2-8b, we show the three nearest modes of vibration and their corresponding frequencies.

2.4.2 The investigation of stress-dominated string-like dynamic behaviour of resonator

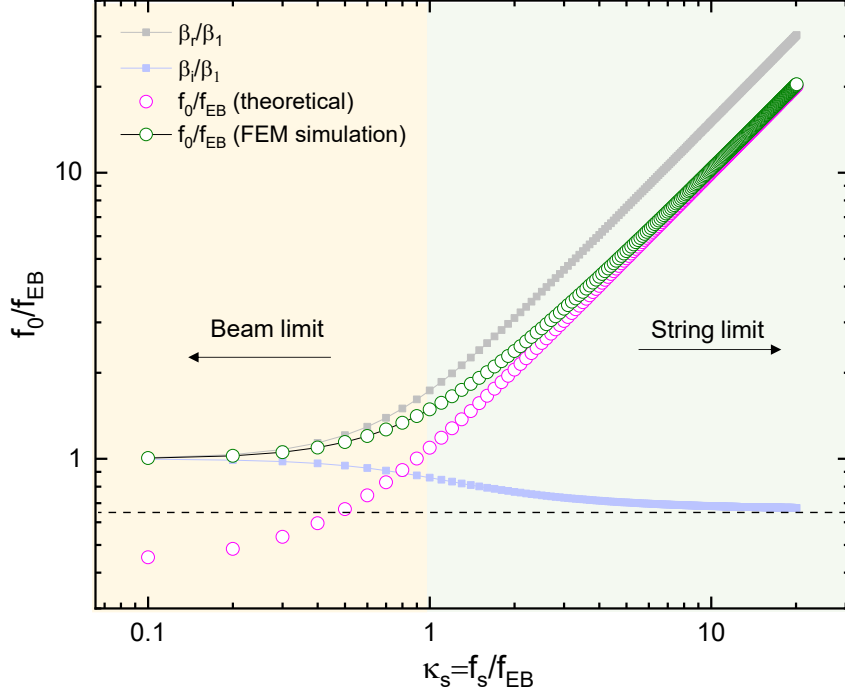


Figure 2-9 Natural frequency of nanoresonator. Horizontal axis indicated the ratio of expected natural frequency if it were to determine only by native stress (f_s) divided by expected natural frequency if it were to determine only by flexural stiffness (f_{EB}). In vertical axis, we plot the overall natural frequency (f_0) divided by f_{EB} .

As we gradually reduce the width of ultrathin Si NEMS resonators, a trend in resonance-frequency and frequency tunability is experimentally observed. We examine this trend from the perspective of $\frac{f_0}{f_{EB}}$ ratio to indicate the transition from beamlike to stringlike dynamic behavior in previous section. Here, we investigate the expected natural frequencies of our Si nanoresonators when treated as continuum mechanical structures to evaluate the significance of $\frac{f_0}{f_{EB}}$ in determining the role of native stress versus elastic-stiffness.

Small amplitude vibration of our nanoresonators resonators can be modelled as a double-clamped, stressed Euler-Bernoulli beam of length L , width t , and height w , which is governed by the following equation of motion¹²:

$$\rho A_{cs} \frac{\partial^2}{\partial \tau^2} y(x, \tau) = -EI \frac{\partial^4}{\partial x^4} y(x, \tau) + T_s \frac{\partial^2}{\partial x^2} y(x, \tau) \quad 2-1$$

where, $y(x, \tau)$ is the displacement of the stressed-beam at position ' x ' and at time ' τ '; T_s is the axial tension. For a tension-free Euler-Bernoulli beam (i.e., when $T_s = 0$), equation (2-1) predicts a natural frequency,

$$f_{EB} = \frac{\beta_1^2}{2\pi L^2} \sqrt{\frac{EI}{\rho A_{cs}}} \quad 2-2$$

where mode constant (β_1) is defined as, $\beta_1 \approx 4.73$. On the other hand, for a string under tension with no flexural stiffness (i.e., when $EI = 0$), natural frequency evaluated by equation (2-1),

$$f_s = \frac{1}{2L} \sqrt{\frac{T_s}{\rho A_{cs}}} \quad 2-3$$

In the intermediate regime, where neither the tension nor the flexural rigidity can be neglected, an approximate closed form formula¹² for natural frequency (f_0) of fundamental flexural-mode can be derived by assuming a solution consisting of string mode-shape function ($\varphi_s(x)$), $y(x, t) = \varphi_s(x) \cos 2\pi f \tau$, where $\varphi_s(x) = C \sin \frac{\pi x}{L}$, and simply supported boundary condition, $\varphi_s(0) = \varphi_s(L) = \frac{\partial^2 \varphi_s(x)}{\partial x^2} \big|_{x=0} = \frac{\partial^2 \varphi_s(x)}{\partial x^2} \big|_{x=L} = 0$. The approximate formula for natural frequency (f_0) thus achieved can be expressed by following equation¹²,

$$f_0 \approx \frac{\pi}{2L^2} \sqrt{\frac{EI}{\rho A_{cs}}} \sqrt{1 + \frac{T_s L^2}{\pi^2 EI}} \quad 2-4$$

Equation (2-4) implies that the change in the natural frequency of a Euler-Bernoulli beam due to tension (T_s) will follow,

$$\frac{f_0}{f_{EB}} \approx \frac{\pi^2}{\beta_1^2} \sqrt{1 + \frac{T_s L^2}{\pi^2 EI}} \quad 2-5$$

However, the aforementioned assumptions about the mode-shape function and boundary conditions are valid only at relatively high-tension regime ($\frac{f_0}{f_{EB}} \gg 1$). In low tension regime, the accuracy of the formula in equation (2-4) can be low. Therefore, we explore what sort of relationship of $\frac{f_0}{f_{EB}}$ is anticipated for our nanoresonators.

Natural frequency of the fundamental mode f_0 of the stressed beam can be calculated by inserting a function, $y(x, \tau) = \varphi(x) \cos(\omega_0 \tau)$ into equation (2-1), where $\omega_0 = 2\pi f_0$. Equation (2-1) takes the form,

$$\frac{\partial^4 \varphi}{\partial x^4} - 2a_s \frac{\partial^2 \varphi}{\partial x^2} - b_s^2 \omega_0^2 \varphi = 0 \quad 2-6$$

where $a_s = T/(2EI)$, $b_s^2 = \rho A_{cs}/(EI)$. Inserting a trial solution of the form, $\varphi(x) = C e^{\beta x}$ into equation (2-6), yields a solution for the mode-shape function,

$$\varphi(x) = c_1 \cosh \beta_r x + c_2 \sinh \beta_r x + c_3 \cos \beta_i x + c_4 \sin \beta_i x \quad 2-7$$

where c_1, c_2, c_3, c_4 are arbitrary constants to be determined by utilizing the boundary conditions at $x = 0$ and at $x = L$; and $\beta_r = \left[(a_s^2 + b_s^2 \omega_0^2)^{\frac{1}{2}} + a_s \right]^{\frac{1}{2}}$; and $\beta_i = \left[(a_s^2 + b_s^2 \omega_0^2)^{\frac{1}{2}} - a_s \right]^{\frac{1}{2}}$, which implies,

$$\omega_0 = \frac{\beta_r \beta_i}{b} \quad 2-8$$

$$\beta_r^2 - \beta_i^2 = 2a_s \quad 2-9$$

Plugging in double-clamped beam boundary conditions, $\varphi(0) = \varphi(L) = \frac{d\varphi(x)}{dx}|_0 = \frac{d\varphi(x)}{dx}|_L = 0$ into the mode-shape function in equation (2-6), gives the set of linear equations:

$$\begin{pmatrix} 1 & 0 & 1 & 0 \\ 0 & \beta_r & 0 & \beta_i \\ \cosh \beta_r L & \sinh \beta_r L & \cos \beta_i L & \sin \beta_i L \\ \beta_r \sinh \beta_r L & \beta_r \cosh \beta_r L & -\beta_i \sin \beta_i L & \beta_i \cos \beta_i L \end{pmatrix} \begin{pmatrix} c_1 \\ c_2 \\ c_3 \\ c_4 \end{pmatrix} = 0 \quad 2-10$$

For nontrivial solution, setting the determinant of the coefficient matrix on the left to zero:

$$\frac{(\beta_r L)^2 - (\beta_i L)^2}{2(\beta_r L)(\beta_i L)} \sinh(\beta_r L) \sin(\beta_i L) + (1 - \cosh(\beta_r L) \cos(\beta_i L)) = 0 \quad 2-11$$

Rewriting equation (2-9) in the following form:

$$(\beta_r L)^2 - (\beta_i L)^2 = \alpha_s^2 \quad 2-12$$

where, $\alpha_s = \sqrt{2a_s}L = \sqrt{\frac{T_s L^2}{EI}}$ is an unitless quantity, which can be expressed in terms of the ratio (κ_s) of resonance frequency (f_s) of a simply supported string without flexural rigidity to the frequency of a tension-free double-clamped beam (f_{EB}):

$$\alpha_s = \sqrt{\frac{TL^2}{EI}} \approx 7.12 \left(\frac{f_s}{f_{EB}} \right) = 7.12 \kappa_s \quad 2-13$$

It is worth mentioning that the limit $T_s \rightarrow 0$, i.e., $\kappa_s \rightarrow 0$, indicate the tension-free double-clamped Euler-Bernoulli beam, namely, $\beta_r = \beta_i = \beta_1 \approx 4.73$; $0 < \kappa_s \ll 1$ indicates a dominant beamlike behavior; at $\kappa_s = 1$, if acted independently, stress as well as flexural rigidity will lead to the same natural frequency for fundamental mode, i.e., $f_s = f_{EB}$; and $\kappa \gg 1$ indicate a dominant

stringlike behavior. In the following figure, we sweep κ_s from $\kappa = 0.1$ to 20.1 , and numerically solve equations (2-9) to evaluate $X = \beta_r L$, and $Y = \beta_i L$. In Figure 2-9, we plot $\frac{f_0}{f_{EB}} = \frac{\omega_0}{\omega_{EB}} = \frac{\beta_r \beta_i}{\beta_1^2}$ as a function of κ_s .

In order to numerically calculate a stressed beam's resonance-frequency of fundamental mode, 'Prestress-eigenfrequency' simulations are conducted with $L = 200 \text{ } \mu\text{m}$, $w = 7.5 \text{ } \mu\text{m}$, and $t = 100 \text{ nm}$ (dimensions similar to the one whose data is shown in Figure 2-7a) using Comsol Multiphysics. Both ends of the beam are set to 'fixed' (no displacement) to mimic 'double clamped' boundary condition. In this FEM simulation, the effect of stress on resonance frequency is evaluated by introducing a tensile 'pre-stress' parameter (σ_0) in the simulation; values of the σ_0 parameter is swept from $10^3 - 10^8 \text{ Pa}$ and respective natural frequencies ($f_0(\sigma_0)$) are noted. One simulation is conducted at $\sigma_0 = 0 \text{ Pa}$ to evaluate the frequency corresponding to stress-free Euler-Bernoulli beam. To simulate corresponding natural frequency of a nanoresonator under tension, elastic modulus of the beam is reduced to 100 MPa ($\sim 0.06\%$ of Si) to set the flexural rigidity ($\sim EI$) close to zero. The aforementioned simulation is then repeated by sweeping σ_0 and natural frequencies of string mode (f_s) are calculated. Simulation results are plotted in Figure 2-9, along with the values calculated by the analytical method described above and using the formula in equation (2-4). Results are in agreement with each other, except at small values of κ_s (i.e., at very low stress).

In short, a transition from beamlike to stringlike behavior can be identified by measuring $\frac{f_0}{f_{EB}}$ of our ultrathin Si nanoresonators.

2.4.3 Quality factor and dissipation mechanism

Quality-factor (Q), a common performance parameter of nanoresonators, is relevant for many practical applications. In sensing applications, usually a higher quality-factor enhances the performance of the nanoresonator device. A quality-factor of $10^3 - 10^4$ is observed in our nanoresonator devices. Q did not increase appreciably even at high vacuum of $P \sim 1 \times 10^{-4} \text{ Pa}$, and down to $T \sim 90 \text{ K}$ (compared to the values we observed at room temperature and in low vacuum ($P \sim 10^{-1} \text{ Pa}$) conditions, e.g., in the measurement of device shown in figure 2-7, where Q factor values are typically $\sim 10^3$), although a weak upward trend (within the same order of magnitude) is observed with lowering of temperature (Figure 2-10a). The Q -values and the $Q(T)$ trend

observed in our devices is similar to a previous report by Tao et al. ,²⁰ for Si resonators of comparable dimensions.

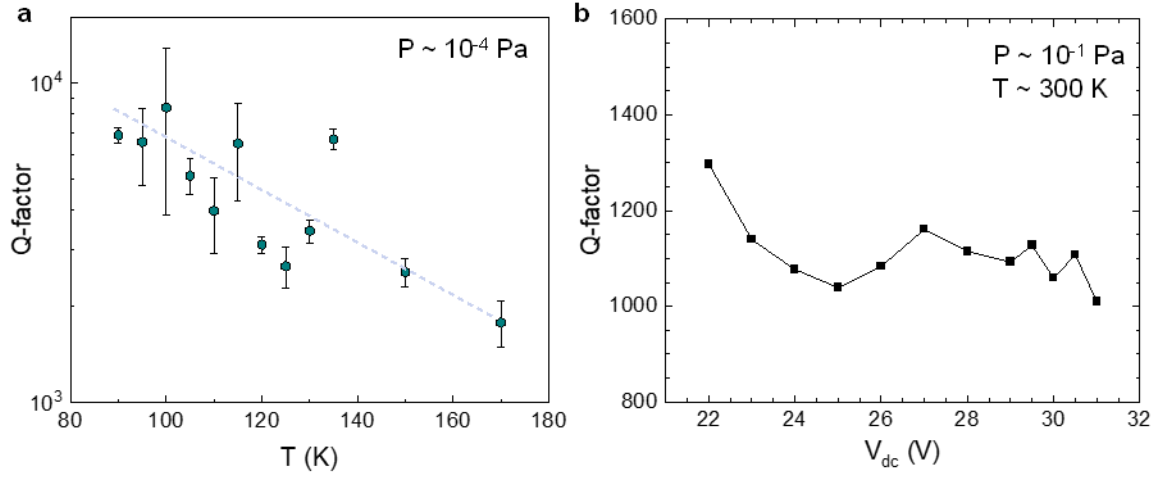


Figure 2-10 Quality factor variation with temperature and V_{dc} . (a) Quality-factor of Si nanoresonator measured at different temperatures ($P \sim 10^{-4}$ Pa). This data is collected from the same nanoresonator whose data is shown in Figure 2-7a. Datapoints represent the averages, and the error-bars indicate the standard deviations of three consecutive measurements conducted at the same temperature (vacuum condition slightly varied from measurement to measurement). A weak increasing trend (indicated by the dashed line) with reducing T between 170 K and 90 K is observed, but the measured values are lower than what would be expected for air damping, thermoelastic damping, or anchor loss. (b) Q -factor variation with V_{dc} at room temperature and at $P \sim 10^{-1}$ Pa pressure.

Q value depends on various damping mechanisms. Common damping mechanisms for micro / nano resonators are air damping, thermoelastic damping (TED), anchor loss, etc. The overall quality factor of the resonator, $\frac{1}{Q} = \sum \frac{1}{Q_i} = \frac{1}{Q_{air}} + \frac{1}{Q_{TED}} + \frac{1}{Q_{anchor}} + \dots$, where Q_i indicate expected quality-factor corresponding to the 'i'-th loss mechanism if it were to act alone. Therefore, the overall Q value is always lower than that of the most dominant loss mechanism.

We theoretically¹² and by FEM simulation estimated the expected Q factors considering the aforementioned losses individually. The expected Q factors are listed below:

Table 2-3 Expected Q -factors associated with common damping mechanisms

Q_{air} (Squeeze-film)	3.7×10^6
Q_{TED}	8.4×10^{11}
Q_{anchor}	4.2×10^9

From the Q values shown in Table 2-3, we conclude that none of the aforementioned mechanisms are responsible for the dominant loss. The weak upward trend with decreasing temperature is also not sufficiently explained by these losses. In ultrathin NEMS resonators, where the surface to volume ratio is quite large, a type of intrinsic loss mechanism called ‘surface-loss’ can become dominant²¹. For our ultrathin Si NEMS resonators, roles of contaminant residues from DRIE process, native oxide layer formation on the surface after vapor HF process, and surface-adsorbed molecules needs to be investigated to definitively conclude whether surface-loss is indeed the dominant mechanism for energy dissipation. Further investigation is underway to identify the dominant loss, and potentially enhance the Q factor of our ultrathin Si NEMS resonators.

We have observed some variation of the Q -factor with the dc gate-voltage (V_{dc}) in our nanoresonators. The observation from device measured in Figure 2-7a is shown in the Figure 2-10b. The $Q(V_{dc})$ trends in our devices are typically non-monotonic (and sensitive to device parameters and experimental conditions), similar to observations made in atomically thin resonators. In electrostatically tuned ultrathin resonators such as in atomically thin resonators, multiple influencing factors, such as, variation of dissipation dilution factor²² due to the variation of axial strain by V_{dc} (electrostatic force), changes in electrical losses (e.g., loaded- Q effect) with variations in V_{dc} , variation in nanoresonator’s frequency with V_{dc} due to the tuning effect, etc. can potentially influence the behavior of the $Q(V_{dc})$ trends. Therefore, further experimental, and theoretical investigations are needed to pinpoint a mechanism and to determine optimum conditions for $Q(V_{dc})$ tuning in our devices.

2.5 Discussion and conclusion

In this chapter, we have provided a detailed presentation of the fabrication and characterization of ultrathin Si NEMS resonators. By carefully designing the dimensions and employing a scalable microfabrication process involving advanced electron-beam-lithography and optimized deep reactive-ion etching, ultrathin Si NEMS resonators (width ~ 10 nm, length ~ 100 μm , and height ~ 7.5 μm) are monolithically fabricated. The largest aspect ratio can reach around 15000. The characterization of fabricated NEMS resonators realized by a customized detection circuit and lock-in amplifier highlighted their exceptional mechanical properties, including high resonance frequencies and stress-dominated dynamic behaviour. By gradually decreasing our device’s width

from 300 nm to 20 nm, we demonstrate a gradual transition from beamlike to string-like dynamic behaviour indicated by increased stress-dominated resonance frequency with thinning of width.

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Chapter 3 Ultrawide Tunability of Resonance Frequency in Ultrathin Silicon

NEMS Resonator

3.1 Introduction

The unparalleled performance of nanoresonators in their wide-ranging applications is fundamentally derived from a mechanically vibrating nanostructure's resonant vibration at a high frequency and its rich dynamic-behavior, which are primarily determined by its geometry and material adopted at the device fabrication stage. However, an ability to widely tune the resonance-frequency (f_0) and the dynamic-behavior of a nanoresonator at the post-fabrication (application) stage can enable us to promptly adjust the nanoresonator's vital operational parameters to meet a wide range of requirements on-the-spot. Bringing such flexibility will be precious for realizing or aiding applications, such as, voltage-controlled oscillators,¹ multiband filtering, impedance-matching and mode-matching² in nanoelectronics, mode-matching in inertial sensors,³ enhancing frequency-stability⁴ in timing devices, highly efficient mechanical energy harvesting devices,⁵ sensors with a broad and tunable linear-dynamic-range⁶ for ultrasensitive chemical, biological sensing, and other novel applications of nanoresonators.

To achieve significant room temperature tunability, it is advantageous to build the vibrating mechanical-elements of nanoresonator devices using an ultrathin material, such as, carbon-nanotube (CNT), graphene, MoS₂,⁷ WSe₂,⁸ Mxene,⁹ *etc.* Dynamic-behavior of these atomically thin resonators is governed by stress, which makes their behavior akin to strings. Tuning this 'native' stress (σ_0) post-fabrication by applying an electrostatic-force via a dc gate-voltage (V_{dc}) (gate-tuning) offers ultrawide tunability in f_0 , nonlinearity,^{10,11,12} quality-factor¹³ (Q -factor), *etc.* Unfortunately, integrating atomically thin materials in nanoresonator devices require a complex and specialized fabrication process, which combined with a lack of wafer-scale single-crystal-quality base-materials restrict the ubiquitous application of tunable nanoresonators.¹⁴ Achieving comparable electrostatic tunability in conventional monolithically microfabricated resonator devices, especially from Si, which offers wafer-scale, inexpensive, single-crystal-quality material and scalable monolithic fabrication process is desired but considered challenging. Moreover, microfabricated tunable nanoresonators can allow easier integration of additional device

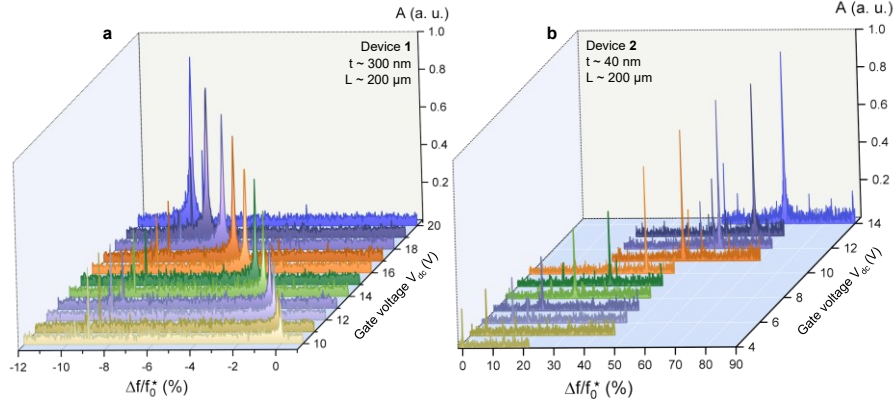


Figure 3-1 Electrostatic tuning of resonance-frequency (f_0) of Si nanoresonators by gate-voltage (V_{dc}) at room temperature. (a) Downward tuning of f_0 of $\sim 9\%$ is observed in a $t \sim 300$ nm Si nanoresonator ($L \sim 200$ μm, $w \sim 7.5$ μm, $g_0 \sim 2$ μm, $P \sim 10^{-1}$ Pa, $T \sim 300$ K) by V_{dc} (estimated Q -factors are in 1100 – 2300 range). (b) Upward tuning of f_0 of $\sim 70\%$ is observed in a $t \sim 40$ nm Si nanoresonator ($L \sim 200$ μm, $w \sim 7.5$ μm, $g_0 \sim 2$ μm, $P \sim 10^{-1}$ Pa, $T \sim 300$ K) by V_{dc} (estimated Q -factor are in 1000 – 1700 range). In a and b, f_0^* represents the resonance-frequency measured at the lowest V_{dc} in each case, and $\Delta f = f - f_0^*$.

components for new functionality while offering highly efficient on-chip detection techniques.^{15, 16} In the previous chapter, we successfully fabricated ultrathin silicon (Si) NEMS resonators and demonstrated their dynamic behavior, which closely resembles that of string-like resonators. These results indicate the potential of these Si NEMS resonator devices could also achieve wide tunability in dynamic properties. In this chapter, we explore the tunability of resonance frequency in ultrathin Si NEMS resonators, focusing on the experimental results and theoretical investigation of frequency tuning through electrostatic and thermal means. We present both experimental results and theoretical models to study the frequency tuning behaviours of these resonators under varied conditions.

3.2 Gate voltage induced electrostatic resonance frequency tuning

As stated in the previous chapter, the dynamic behaviours our ultrathin Si NEMS resonator is governed by the axial tensile stress (σ). The typical sources of σ are, (a) a native-stress (σ_0 , at room-temperature) introduced during fabrication, mechanism of which can be hard to pinpoint and control, and (b) electrostatic-force used for actuation, which can be precisely controlled. In current devices, typical values of σ_0 are estimated to be 10 – 40 MPa. Beside σ_0 , f_0 depends on other factors that control the stress-distribution on the nanoresonator. For example, variations in V_{dc} , T , can significantly shift f_0 in either upward or downward direction via specific electro-thermo-

mechanical mechanisms, which form the basis for wide tunability of f_0 in current nanoresonators as well as in atomically thin nanoresonators.

3.2.1 Experimental results of resonance frequency tuning

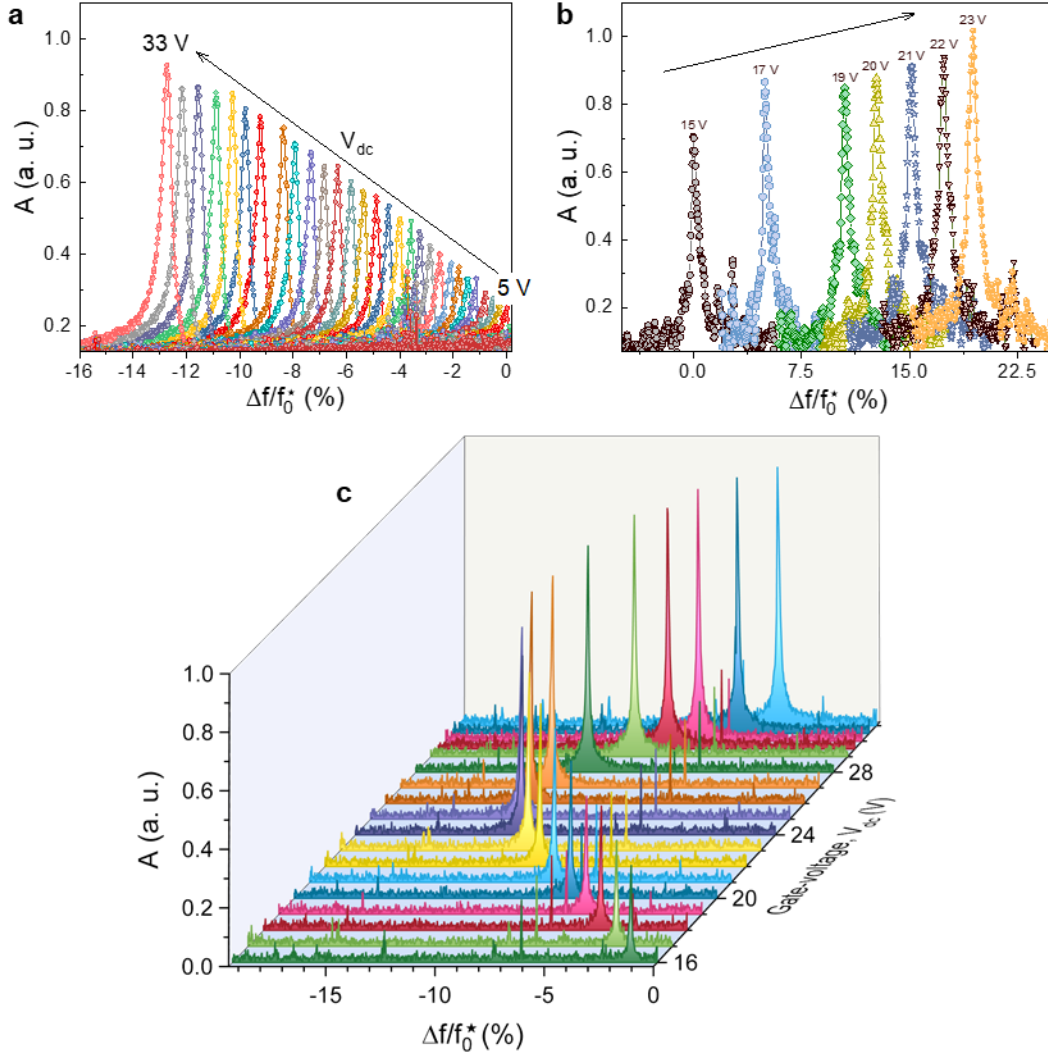


Figure 3-2 Demonstration of resonance-frequency tuning by gate-voltage in additional Si nanoresonator resonator devices. a, downward frequency tuning in a device with dimensions, $t \sim 250$ nm, $L \sim 200$ μ m, $w \sim 7.5$ μ m, and $g_0 \sim 2.0$ μ m. Acquisition of data started at $V_{dc} = 5$ V and stopped at $V_{dc} = 33$ V, $f_0^* \sim 399.1$ kHz. b, Upward frequency tuning in a device with dimensions, $t \sim 40$ nm, $L \sim 100$ μ m, $w \sim 7.5$ μ m, and $g_0 \sim 2.0$ μ m. Acquisition of data started at $V_{dc} = 15$ V and stopped at $V_{dc} = 23$ V, $f_0^* \sim 315.0$ kHz. c, Mixed (nonmonotonic) tuning of resonance frequency observed in a device (Device 3) with dimensions, $t \sim 100$ nm, $L \sim 100$ μ m, $w \sim 7.5$ μ m, and $g_0 \sim 2.0$ μ m. Acquisition of data started at $V_{dc} = 16$ V and stopped at $V_{dc} = 31$ V, $f_0^* \sim 355.7$ kHz.

Electrostatic tuning of f_0 by V_{dc} of $\sim 10\%$ or more with tuning-efficiency $> 1\% V^{-1}$ is frequently reported in atomically thin CNT,¹⁷ graphene,¹ MoS₂,⁶ WSe₂,⁸ NEMS resonators at room temperature (tunability typically reduces at low temperature), which makes them excellent and unique candidates for developing many nanoelectronics applications.¹⁸ Demonstration of such wide and efficient electrostatic tuning of resonance-frequency in microfabricated resonators is lacking and considered challenging. In our Si nanoresonator devices, we have observed $\sim 70\%$ upward and $\sim 10\%$ downward electrostatic tuning-range of f_0 by V_{dc} at room temperature. We estimate the f_0 tuning-range (T_R) and the average tuning-efficiency (T_e) by calculating, $T_R = \left| \frac{\Delta f_0}{f_0^*} \right| \%$ and $T_e = \left| \frac{1}{\Delta V_{dc}} \frac{\Delta f_0}{f_0^*} \right| \% V^{-1}$, where f_0^* is the f_0 measured at the lowest V_{dc} and Δf_0 is the total shift in f_0 (relative to f_0^*) for a V_{dc} change of ΔV_{dc} . For a device with $t \sim 300$ nm nanoresonator (Device 1), a downward $T_R \sim 9.4\%$ is achieved (Figure 3-1a) by varying V_{dc} from 10 – 20 V, indicating an average downward $T_e \sim 1\% V^{-1}$. Upward $T_R \sim 70\%$ (Figure 3-1b) with an average $T_e \sim 7\% V^{-1}$ is achieved by varying V_{dc} from 4 – 14 V in another device containing a nanoresonator of $t \sim 40$ nm (Device 2), which is the narrowest in the series of devices we tested. Room temperature electrostatic tunability obtained in our Si nanoresonator ($T_R \sim 70\%$ and $T_e \sim 7\% V^{-1}$) are higher than typical values reported in nanowire/ nanobeam resonators ($T_R \sim 0.8 - 6\%$ and $T_e \sim 0.04 - 0.6 \% V^{-1}$)¹⁹ and comparable to various reports on atomically thin resonators ($T_R \sim 10 - 300\%$, $T_e \sim 1 - 20 \% V^{-1}$).¹ Further downward ($T_R \sim 13\%$), upward ($T_R \sim 19\%$), and mixed (nonmonotonic) f_0 tuning results obtained from additional Si nanoresonator devices are presented in Figure 3-2.

3.2.2 Comparison with similar work

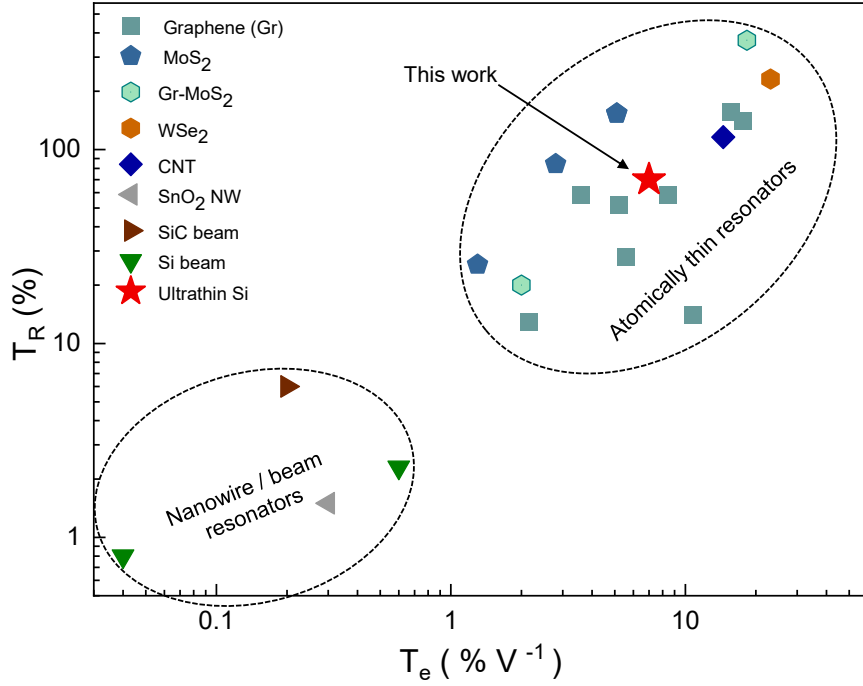


Figure 3-3 Comparison of resonance-frequency tuning-range (T_R) and tuning-efficiency (T_e) between this work and related works. T_R and T_e achieved by electrostatic tuning via gate-voltage at room temperature for atomically thin resonators and nanowire / beam resonators from some of the references in Table 1-2 are plotted.

In Table. 1-2, we provide a list of electrostatic resonance-frequency tuning-range (T_R) and tuning-efficiency (T_e) by gate-voltage at room temperature reported in atomically thin resonators and other nanowire / beam resonators. Values of T_R , T_e achieved in the present work are shown in Figure 3-3 to compare with the reported values. Apparently, the tunability achieved in the work is on par with the most of atomically thin resonators.

3.3 Theoretical investigation of frequency tunability

To gain a comprehensive understanding of how the resonance frequency of ultrathin Si NEMS resonators can be tuned by applying a DC gate voltage, we present an approximated theoretical model in this section. This investigation looks into the electromechanical interactions that influence the dynamic behavior of these resonators, providing understandings into both the mechanisms and the practical implications of frequency tuning.

3.3.1 Electromechanical approximate model for resonance frequency tuning

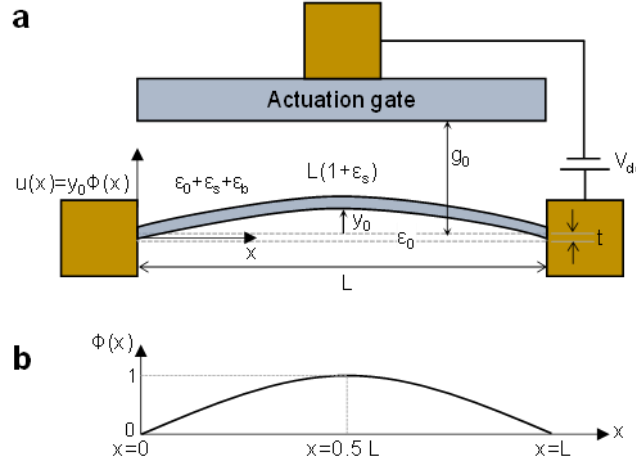


Figure 3-4 Schematic diagram of the electromechanical model of the nanoresonator resonators. a, Deflection, $u(x, V_{dc}) = y_0(V_{dc})\phi(x)$ of the nanoresonator under gate-voltage (V_{dc}); y_0 is the deflection of the string center at V_{dc} . b, $\phi(x)$ defines the deflection shape of the string with the following constraints and symmetry, $\phi(0) = \phi(L) = 0$, $\phi(\frac{L}{2}) = 1$, and $\phi(\frac{L}{2} - x) = \phi(\frac{L}{2} + x)$.

Here, we present a theoretical model to elaborate the resonance-frequency tuning (f_0) by dc gate-voltage (V_{dc}) as shown in Fig. 3-1 and Fig. 3-2. In the absence of V_{dc} , a nanoresonator's resonance frequency (f_0) is determined by its geometrical shape, dimensions, clamping conditions, material properties, and the native stress (σ_0). V_{dc} influences f_0 via electromechanical forces, which results in two competing effects vis a vis resultant resonance-frequency: (a) a static loading of the nanoresonator that geometrically stretches its length and thus hardens the linear stiffness (Fig. 3-4a), and (b) a counter-restoring force which softens the linear stiffness. We follow along the strategy presented in ref ⁶ and ref ²⁰ to develop an electromechanical potential model to explain the observations regarding V_{dc} induced f_0 tuning reported in this work. Usually, to model the electromechanical potential, an appropriate deflection shape ($u(x)$) of the nanoresonator is assumed in the beginning of the deduction. However, here we keep the deduction more general by not assuming an explicit function for $u(x)$ until later.

When a V_{dc} is applied, the nanoresonator experiences a deflection ($u(x, V_{dc})$) towards the actuation electrode. Considering the symmetry of the device design, we assume the maximum deflection (y_0) happens at the nanoresonator's center (i.e., at $x = L/2$). $y_0(V_{dc})$ is determined by the new equilibrium point where the attractive electrostatic force is balanced by the mechanical

restoring force of the nanoresonator. The deflection shape of the nanoresonator $u(x, V_{dc})$ is then expressed by $u(x, V_{dc}) = y_0(V_{dc})\phi(x)$, where $\phi(x)$ defines the shape of the deflection (Fig. 3-4b). $\phi(x)$ must satisfy the boundary conditions, $\phi(0) = \phi(L) = 0$, and $\phi\left(\frac{L}{2}\right) = 1$ to satisfy the deflection condition at the midpoint, $u\left(\frac{L}{2}, V_{dc}\right) = y_0(V_{dc})$. In addition, it is reasonable to assume $\phi(x)$ to be symmetric about an axis perpendicular to the axis of the stationary nanoresonator and passing through $x = L/2$, i.e., $\phi\left(\frac{L}{2} - x\right) = \phi\left(\frac{L}{2} + x\right)$. At this stage, we don't commit to any explicit mathematical form for $\phi(x)$.

The NEMS resonator, in the configuration shown in Fig. 3-5a, experiences three types of mechanical strains, (a) a uniform native axial-tensile-strain ($\varepsilon_0 = \sigma_0/E$), which we assume remains unchanged, (b) an additional tensile-strain (ε_s) due to the stretching of the nanoresonator, which we assume to be uniform and directed only along the nanoresonator axis, and (c) an elastic strain distribution (ε_b) along nanoresonator axis due to the bending of the nanoresonator (considering the fact our Si nanoresonators have a nonzero flexural stiffness, especially for wider nanoresonators). We assume, $y_0(V_{dc})$ is small enough, so that the total strain (ε_T) at a point inside the nanoresonator is given by linear superposition, i.e., $\varepsilon_T = \varepsilon_0 + \varepsilon_s + \varepsilon_b$. Thus, the total mechanical potential energy (U_m) of the nanoresonator can be evaluated as, $U_m = \frac{E}{2} \iiint \varepsilon_T^2 dv'$, where the volume integration is performed on the entire volume (v') of the nanoresonator. ε_0 is estimated from native stress as $\varepsilon_0(x', y', z'; V_{dc}) = \frac{\sigma_0}{E}$; $\varepsilon_s(x', y', z'; V_{dc}) \approx \frac{1}{2L} \int_0^L \left(\frac{\partial u}{\partial x}\right)^2 dx$; and $\varepsilon_b(x', y', z'; V_{dc}) \approx \left(\frac{\partial^2 u}{\partial x^2}\right) y'$, where the primed coordinates (x', y', z') represents nanoresonator's internal coordinates along length, width ($y' = 0$, at the neutral axis), and height direction (we assume, the internal axes system remains roughly parallel to the external coordinates, which is valid only for small deflection). Total mechanical potential energy (U_m) of the nanoresonator for a deflection y of the nanoresonator-center can be expressed as:

$$U_m = \left(\frac{EA_{cs}L\varepsilon_0^2}{2}\right) + \left(\frac{J_2EI}{2L^3} + \frac{J_1EA_{cs}\varepsilon_0}{2L}\right)y^2 + \left(\frac{J_1^2EA_{cs}}{8L^3}\right)y^4 \quad 3-1$$

where, A_{cs} is cross-sectional area, ρ and E are the density and the elastic modulus of the material of the nanoresonator, respectively. Two dimensionless constants in the above expression, $J_1 = L \int_0^L \left(\frac{d\phi(x)}{dx}\right)^2 dx$, and $J_2 = L^3 \int_0^L \left(\frac{d^2\phi(x)}{dx^2}\right)^2 dx$ depend on the choice of $\phi(x)$.

The electrostatic potential energy (U_e) of the nanoresonator can be expressed as:

$$U_e = -\frac{\zeta}{2} V_{dc}^2 \int_0^L \frac{\epsilon_0 w dx}{g_0 - y(x, V_{dc})} \quad 3-2$$

$$\approx -\left(\frac{\zeta}{2} C_g^0 V_{dc}^2\right) \left[1 + o_1 \left(\frac{y}{g_0}\right) + \frac{o_2}{2} \left(\frac{y}{g_0}\right)^2 + \frac{o_3}{3} \left(\frac{y}{g_0}\right)^3 + \frac{o_4}{4} \left(\frac{y}{g_0}\right)^4\right]$$

with a capacitance correction-factor, $\zeta \approx 1$, permittivity of free-space, ϵ_0 , static-capacitance,

$$C_g^0 = \frac{\epsilon_0 L w}{g_0}, \text{ and dimensionless constants, } o_1 = \frac{1}{L} \int_0^L \phi(x) dx, \quad o_2 = \frac{2}{L} \int_0^L (\phi(x))^2 dx, \quad o_3 = \frac{3}{L} \int_0^L (\phi(x))^3 dx, \text{ and } o_4 = \frac{4}{L} \int_0^L (\phi(x))^4 dx \text{ are to be derived from } \phi(x).$$

Introducing, $U_0 = \frac{E}{2} \epsilon_0^2 L A_{cs}$, normalized deflection of the nanoresonator-center, $\bar{y} = \frac{y}{g_0}$, and, dimensionless constants, $a = \frac{J_1}{2\epsilon_0} \left(\frac{g_0}{L}\right)^2$, $b = \frac{1}{12\epsilon_0} \left(\frac{J_2}{J_1}\right) \left(\frac{t}{L}\right)^2$, and a dimensionless voltage parameter, $\bar{v} = \frac{\zeta C_g^0 V_{dc}^2}{4a^2 U_0}$, the mechanical and the electrostatic potential energies can be expressed as:

$$U_m(\bar{y}) = U_0 [1 + 2a(b+1)\bar{y}^2 + a^2\bar{y}^4] \quad 3-3$$

$$U_e(\bar{y}) \approx -(4a^2 U_0 \bar{v}) \left[1 + o_1 \bar{y} + \frac{o_2}{2} \bar{y}^2 + \frac{o_3}{3} \bar{y}^3 + \frac{o_4}{4} \bar{y}^4\right] \quad 3-4$$

Finally, we normalize $U_m(\bar{y})$ and $U_e(\bar{y})$ by $4a^2 U_0$ to express dimensionless mechanical ($\bar{U}_m(\bar{y})$) and electrostatic ($\bar{U}_e(\bar{y})$) potential energies by the following expressions:

$$\bar{U}_m(\bar{y}) = \left[\frac{1}{4a^2} + \frac{b+1}{2a} \bar{y}^2 + \frac{1}{4} \bar{y}^4\right] \quad 3-5$$

$$\bar{U}_e(\bar{y}) \approx -\bar{v} \left[1 + o_1 \bar{y} + \frac{o_2}{2} \bar{y}^2 + \frac{o_3}{3} \bar{y}^3 + \frac{o_4}{4} \bar{y}^4\right] \quad 3-6$$

At the normalized equilibrium position of the nanoresonator-center ($\bar{y}_0 = \frac{y_0}{g_0}$), $\left[\frac{d(\bar{U}_m + \bar{U}_e)}{d\bar{y}}\right]_{\bar{y}_0} = 0$, which implies,

$$\bar{y}_0^3 + \left(\frac{b+1}{a}\right) \bar{y}_0 - \bar{v} [o_1 + o_2 \bar{y}_0 + o_3 \bar{y}_0^2 + o_4 \bar{y}_0^3] = 0 \quad 3-7$$

Rearranging terms,

$$(1 - o_4 \bar{v}) \bar{y}_0^3 - (o_3 \bar{v}) \bar{y}_0^2 + \left(\frac{b+1}{a} - o_2 \bar{v}\right) \bar{y}_0 - o_1 \bar{v} = 0 \quad 3-8$$

Since, we are interested in solutions for $\bar{y}_0 < 1$ (i.e., before the nanoresonator touches the gate-electrode), an approximate solution, valid only at small ($\bar{y}_0 \ll 1$),

$$\bar{y}_0 = \frac{o_1 \bar{v}}{\left(\frac{b+1}{a} - o_2 \bar{v}\right)} \approx o_1 \chi \bar{v} (1 + o_2 \chi \bar{v}) \quad 3-9$$

where, $\chi = \frac{a}{b+1}$, and assuming $o_2\chi\bar{v} \ll 1$ is valid. Dimensionless linear stiffness of the nanoresonator can then be calculated as,

$$\bar{k} = \left[\frac{d^2(\bar{U}_m + \bar{U}_e)}{d\bar{y}^2} \right]_{\bar{y}_0} = 3\bar{y}_0^2 + \left(\frac{b+1}{a} \right) - \bar{v}[o_2 + 2o_3\bar{y}_0 + 3o_4\bar{y}_0^2] \quad 3-10$$

$$\bar{k} = (3 - 3o_4\bar{v})\bar{y}_0^2 - (2o_3\bar{v})\bar{y}_0 + \left(\frac{b+1}{a} - o_2\bar{v} \right) \quad 3-11$$

Considering terms only up to second order in $\bar{v}$,

$$\bar{k} \approx \frac{b+1}{a} - o_2\bar{v} + o_1\chi(3o_1\chi - 2o_3)\bar{v}^2 \quad 3-12$$

The condition for upward tuning, i.e., $\frac{d\bar{k}}{d\bar{v}} \geq 0$ is satisfied for,

$$\bar{v} \geq \frac{o_2}{2o_1\chi(3o_1\chi - 2o_3)} \quad 3-13$$

Converting the expressions back to quantities with physical units,

$$k(V_{dc}) \approx \left(\frac{J_2}{12} \frac{Ewt^3}{L^3} + J_1 \frac{Ewt\epsilon_0}{L} \right) - \left(\frac{\zeta o_2}{2} \frac{\epsilon_0 Lw}{g_0^3} \right) V_{dc}^2 + \left(\frac{3o_1^2\zeta^2}{8} \frac{\epsilon_0^2 Lw}{g_0^4 Et\epsilon_0^2} \right) V_{dc}^4 \quad 3-14$$

Resonance-frequency, $f_0(V_{dc}) = \frac{1}{2\pi} \sqrt{\frac{k(V_{dc})}{m_{eff}}}$, with $m_{eff} = \frac{1}{2}\rho Lwt$ is thus,

$$f_0(V_{dc}) = \frac{1}{2\pi} \sqrt{\left(J_2 \frac{Et^2}{6\rho L^4} + J_1 \frac{2E\epsilon_0}{\rho L^2} \right) - \left(\frac{o_2\zeta\epsilon_0}{\rho t g_0^3} \right) V_{dc}^2 + \left(\frac{3o_1^2\zeta^2\epsilon_0^2}{4\rho g_0^4 Et^2\epsilon_0^2} \right) V_{dc}^4} \quad 3-15$$

and the condition for upward frequency tuning,

$$V_{dc} \geq \sqrt{\frac{2o_2g_0Et}{3o_1^2\zeta\epsilon_0}} \epsilon_0 \quad 3-16$$

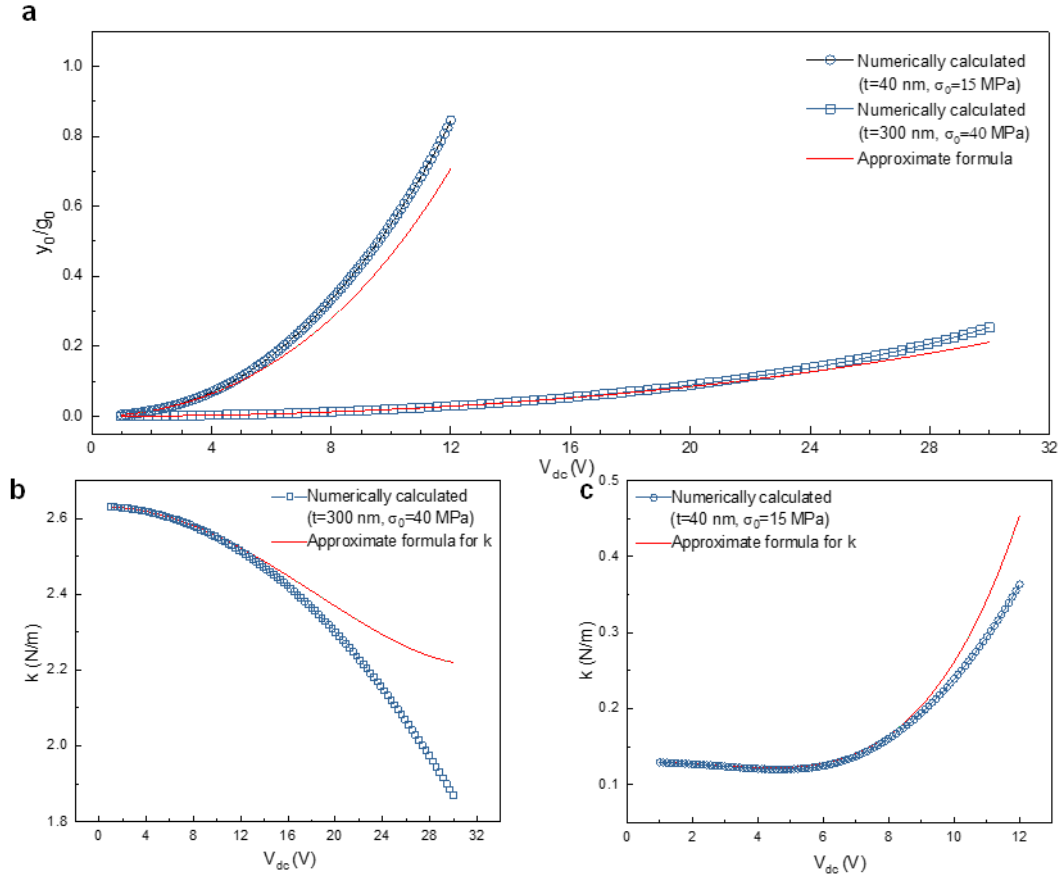


Figure 3-5 Theoretical calculation of linear stiffness (k) variation of Si nanoresonators by V_{dc} . a, Deflection of string-center with V_{dc} calculated for a nanoresonator resonator device of $L = 200$ μm , $w = 7.5$ μm , $g_0 = 2$ μm for different t and σ_0 . b-c, $k(V_{dc})$ plots for the aforementioned nanoresonators. Blue circles pertain to calculations where y_0 is numerically estimated by solving the full cubic equation (3-8), while the red lines correspond to plots of the approximate closed form formulas presented in equations (3-9 and 3-14).

It should be noted that the closed form formula for $\bar{y}_0(V_{dc})$, $k(V_{dc})$, $f(V_{dc})$ and the condition for upward-tuning of resonance frequency are only valid for small deflection (Fig. 3-6). For fitting our frequency tuning data, we calculated $\bar{y}_0$ by numerically solving the full cubic equation (3-8) and plugging in numerically calculated $\bar{y}_0$ in the expression for k . In Fig. 3-5a, divergence of $\bar{y}_0(V_{dc})$ obtained by numerically solving the full cubic equation (3-8) vis a vis from the approximate closed form expression in equation (3-9) can be observed at higher V_{dc} . Similarly, in Fig. 3-5b and Fig. 3-5c, difference in $k(V_{dc})$ obtained from full numerical solution versus the approximate closed form formula for k in equation (3-14) can be seen. Nevertheless, the closed form formulas might be helpful in building an intuitive understanding of the V_{dc} dependent

behavior of f_0 .

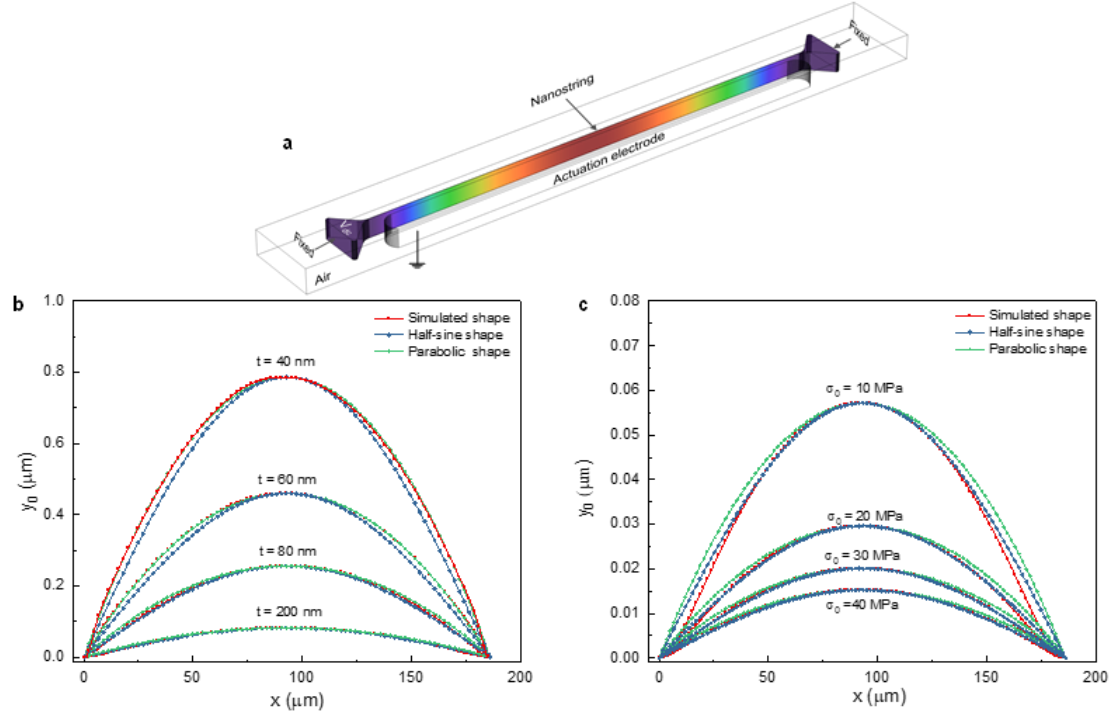


Figure 3-6 Deflection of nanoresonators estimated by finite element simulation. a, Simulation setup, b-c, Deflection, $u(x, V_{dc}) = y_0(V_{dc})\phi(x)$ of the nanoresonator for various parameters (Table 3-2). In b, σ_0 is fixed at 30 MPa, while in c, t is fixed at 200 nm. $L = 200 \mu\text{m}$, $w = 7.5 \mu\text{m}$, $g_0 = 2 \mu\text{m}$ in all cases.

We set up a finite element simulation to investigate the expected deflection shape of our nanoresonators. After importing the geometry of the devices in Comsol, we used ‘Multiphysics’ configuration by integrating ‘Structural mechanics’, ‘AC/DC’ modules to perform electromechanical simulation (Fig. 3-6a). Deflection shapes of nanoresonators from simulation ($u_{sim}(x, V_{dc})$) for various t , V_{dc} , and σ_0 are extracted (Fig. 3-6b and Fig. 3-6c). After normalizing the $u_{sim}(x, V_{dc})$ by its corresponding value at $x = \frac{L}{2}$, we get simulated deflection shape function, $\phi_{sim}(x)$, from which we estimate the values of J_1 , J_2 , σ_1 , σ_2 , σ_3 , σ_4 . For comparison, we also plot (Fig. 3-7b and Fig. 3-7c) and evaluate the values of the shape-dependent constants for (a) a half-sine shape, $\phi_1(x) = \sin\left(\frac{\pi x}{L}\right)$, and (b) a parabolic function, $\phi_2(x) = \frac{4}{L^2}x(L-x)$. Comparison of values of J_1 , J_2 , σ_1 , σ_2 , σ_3 , σ_4 are shown in Table 3-1.

Table 3-1 Comparison of J_1 , J_2 , o_1 , o_2 , o_3 , o_4 calculated from ϕ_{sim} , ϕ_1 , ϕ_2

t (nm)		V_{dc} (V)	σ_0 (MPa)	o_1	$o_2/2$	$o_3/3$	$o_4/4$	J_1	J_2
Calculated from simulation Results (ϕ_{sim})									
40		7	30	0.6651	0.5315	0.4552	0.4045	5.5697	3189.08
60		7	30	0.6669	0.5344	0.4583	0.4076	5.5772	2681.36
80		7	30	0.6447	0.5139	0.4400	0.3909	5.2362	883.41
100		7	30	0.6380	0.5064	0.4321	0.3829	5.1643	750.51
120		7	30	0.6382	0.5072	0.4332	0.3841	5.1851	702.22
140		7	30	0.6377	0.5076	0.4340	0.3852	5.2046	648.36
160		7	30	0.6347	0.5045	0.4310	0.3822	5.1845	599.48
180		7	30	0.6369	0.5084	0.4356	0.3871	5.2630	576.23
200		7	30	0.6305	0.5013	0.4286	0.3804	5.1770	526.22
200		10	10	0.6146	0.4877	0.4173	0.3708	5.1419	385.86
200		10	20	0.6284	0.5008	0.4292	0.3816	5.2041	504.84
200		10	30	0.6351	0.5100	0.4347	0.3867	5.2320	524.11
200		10	40	0.6393	0.5107	0.4380	0.3896	5.2493	734.72
Calculated from $\phi_1(x) = \sin\left(\frac{\pi x}{L}\right)$									
-		-	-	0.6366	0.500	0.4244	0.375	4.9348	48.704
Calculated from $\phi_2(x) = \frac{4}{L^2}x(L-x)$									
-		-	-	0.6667	0.5333	0.4571	0.4063	5.3333	64.00

It is observed in Fig. 3-6 and in the values listed in Table 3-1 that the deflection shapes are typically closer to $\phi_2(x)$ for $t \leq 100$ nm nanoresonators; however, for wider nanoresonators, deflection shapes resemble more to $\phi_1(x)$ across a wide range of values for σ_0 . Therefore, for theoretical calculation of $f_0(V_{dc})$, we adopt $\phi_1(x)$ for $t \geq 100$ nm nanoresonators, while $\phi_2(x)$ is adopted for $t \leq 100$ nm nanoresonators.

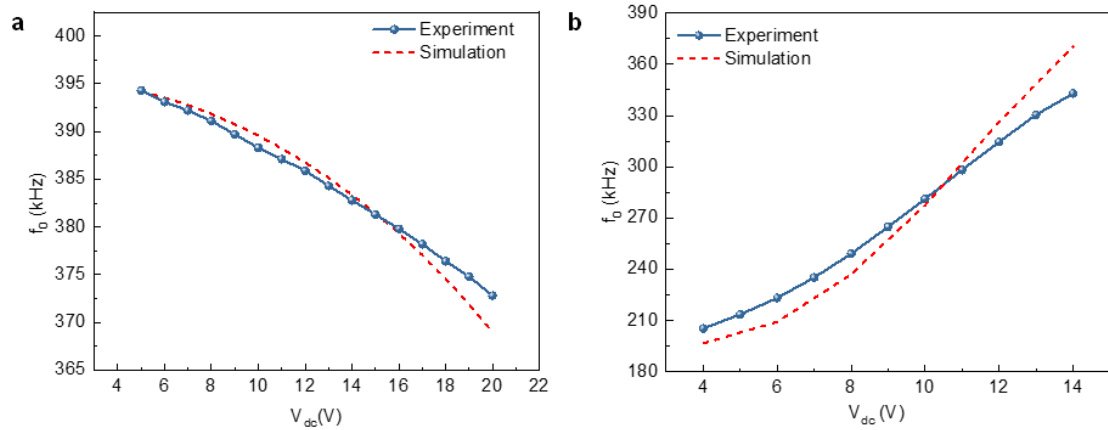


Figure 3-7 V_{dc} induced resonance frequency tuning calculated by finite element simulation. Comparison of experimental and simulation results for nanoresonator resonators of $\sim 200 \mu\text{m}$ length and respective widths of a, 250 nm, and b, 40 nm.

3.3.2 Numerical analysis of resonance frequency tuning

We also estimate resonance-frequency tuning with V_{dc} by finite element analysis by incorporating eigenfrequency study with the coupled electromechanical study shown in Fig. 3-8. The experimental data in Fig. 3-7a corresponds to a 250 nm width, 200 μm long nanoresonator (data shown in Fig. 3-2a), while in Fig. 3-7b, the experimental data is acquired from a 40 nm wide, 200 μm long nanoresonator (data shown in Fig. 3-1b).

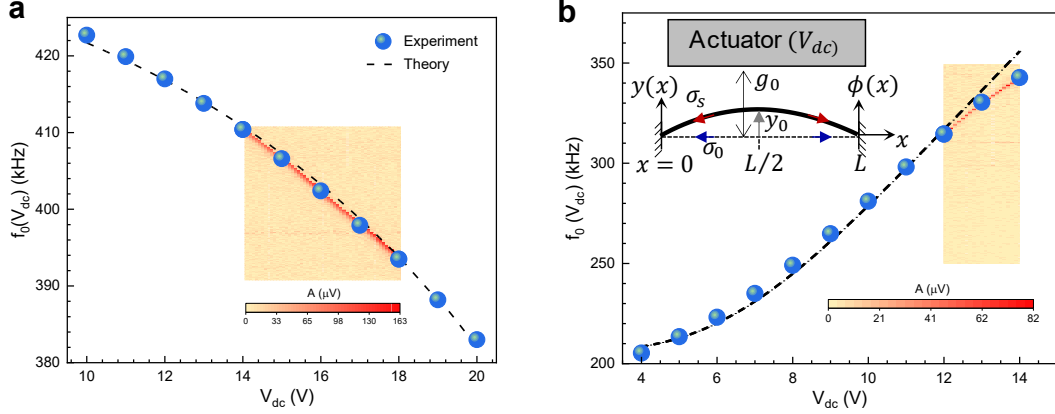


Figure 3-8 $f_0 - V_{dc}$ plots acquired from Device 1 ($t \sim 300$ nm, $l \sim 200$ μm) (a) and Device 2 ($t \sim 40$ nm, $l \sim 200$ μm) (b), respectively, and fitted with the theoretical model built from electromechanical potential calculation based on the deflection of the nanoresonators by V_{dc} as shown in the inset of b; colormaps represent nanoresonators' response and shifts of f_0 under fine variations of V_{dc} , A is the amplitude.

The numerical analysis of resonance frequency tuning has been conducted based on the electromechanical model developed by potential-energy formalism²⁰ to interpret the frequency-tuning behavior and compare with the experimental results. When V_{dc} is applied, the nanoresonator's midpoint ($x = \frac{L}{2}$) shifts to a new equilibrium-position ($y = y_0(V_{dc})$) towards the actuation electrode where the attractive electrostatic force is balanced by the mechanical restoring force. Resulting deflection increases the native axial-tensile-strain ($\varepsilon_0 = \sigma_0/E$) via stretching and bending as well as modifies the electrostatic effect.²¹ We estimate $f_0(V_{dc})$ by numerically calculating the stiffness of the nanoresonator at $y_0(V_{dc})$ using the potential model. For small-deflection ($y_0 \ll L$), a closed-form approximate formula of $f_0(V_{dc})$, is given in equation (3-15).

From equation (3-15), the upward-tuning of resonance-frequency ($\frac{df_0}{dV_{dc}} > 0$) starts at $|V_{dc}| \geq$

$\sqrt{\frac{2\sigma_2 g_0 E t}{3\sigma_1^2 \zeta \epsilon_0}} \epsilon_0$. Thus, the smaller t of the nanoresonator in device 2 compared to device 1 enables upward-tuning of f_0 at a lower V_{dc} despite their other parameters being comparable. To determine J_i and o_j , a suitable $\phi(x)$ must be adopted. We conducted electromechanical simulation on devices with different dimensions using finite-element method, extracted their deflection shapes and numerically calculated J_i and o_j in previous section. Comparing the numerically calculated values against those attained from two candidate functions, $\phi_1(x) = \sin\left(\frac{\pi x}{L}\right)$, and $\phi_2(x) = \frac{4}{L^2}x(L-x)$, we found $\phi_1(x)$ is appropriate for nanoresonators with $t > 100$ nm, while $\phi_2(x)$ suits better for $t \leq 100$ nm. Using $\phi_1(x)$ and $\phi_2(x)$, $f_0 - V_{dc}$ plots in Fig. 3-8a and Fig. 3-8b are theoretically fitted (fitting with finite element simulation results is shown in section 3.3.1). Natural frequencies ($f_0(V_{dc} = 0)$) of nanoresonators are calculated by extrapolating the theoretical fitting to $V_{dc} = 0$.

3.4 Temperature dependence of resonance frequency tunability

3.4.1 Temperature induced resonance frequency tuning

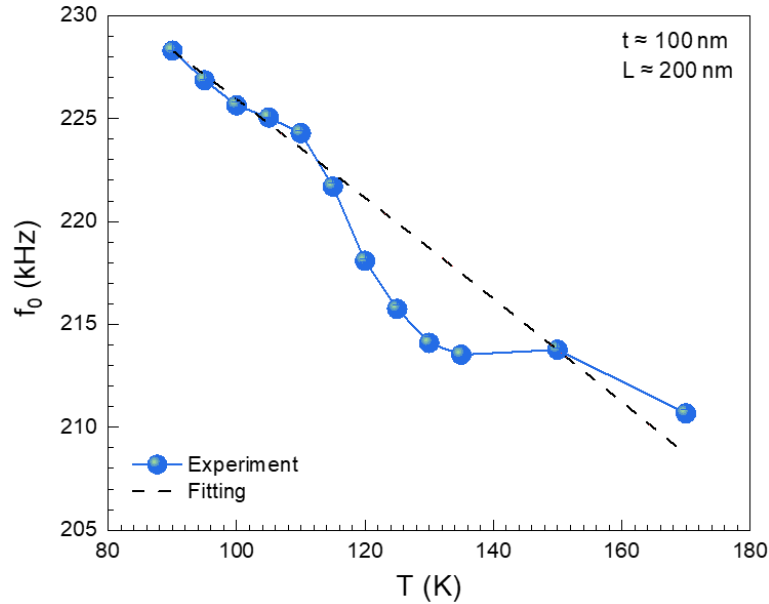


Figure 3-9 Resonance-frequency (f_0) tuning by as a function of device temperature (T) in a ultrathin Si NEMS resonator device. The experimental data is fitted with the theoretically expected frequency shift assuming a thermal expansion coefficient difference ($\Delta\alpha_{th}$) $\sim 2.2 \times 10^{-6} \text{ K}^{-1}$ between the nanoresonator and the substrate. Nanoresonator dimensions are as follows $t \sim 100$ nm, $L \sim 200$ μm , $w \sim 7.5$ μm .

To investigate resonance-frequency tuning by temperature, sample stage is cooled to a specific temperature (T) and $f_0(T)$ is measured by conducting a frequency sweep. A stable stage temperature in our measurement system is achieved by liquid N₂ and an integrated heating system. A variation of 20 kHz in resonance-frequency is observed for a change of 80 K in stage temperature for the nanoresonator sample whose resonance peak is depicted in Fig. 2-7a. The frequency variation with temperature is shown in Fig. 3-9. The average value of temperature tuning efficiency (% change in resonance frequency per Kelvin), also known as the temperature coefficient of frequency (TCF) is about 0.1 %K⁻¹, which is significantly lower than typical values (0.26 – 0.85 %K⁻¹) observed in atomically thin resonators.⁹ This is expected in our device, since the temperature tuning relies on the difference in thermal expansion coefficients ($\Delta\alpha_{th}$) between the material of the resonator and that of the substrate²². This is because the strain on the resonator at temperature T can be expressed as, $\varepsilon(T) = \varepsilon_0(T_0) + \Delta\alpha_{th}(T - T_0)$, where ε_0 is the strain at a reference temperature T_0 . Although, in the present case, the substrate (handle-layer) and the nanoresonator both are made of Si, an effective $\Delta\alpha_{th}$ exists, perhaps, due to the thin ($\sim 2.2 \mu\text{m}$) intermediate SiO₂ layer, the metal thin-films on the device layer, and the difference in crystalline orientation between the device and handle layer Si. $\Delta\alpha_{th}$ is treated as a fitting parameter for fitting the $f_0(T)$ data (Fig. 3-9) with the theoretical formula²² $f_0(T) = \frac{\pi}{2L^2} \sqrt{\frac{EI}{\rho A_{cs}}} \sqrt{1 + \frac{\varepsilon(T)L^2}{\pi^2 I}}$. In the fitting process, the initial strain (ε_0) is adjusted to match the experimental data obtained at the reference temperature, $T_0 = 90 \text{ K}$, then the best fit for the experimental result is achieved for $\Delta\alpha_{th} = 2.2 \times 10^{-6} \text{ K}^{-1}$. The relatively lower value of TCF observed in our nanoresonators could be beneficial for applications intended at room temperature condition, because a lower TCF should cause a lesser drift of the resonance frequency due to temperature fluctuation.

3.4.2 Temperature effect on electrostatic tuning

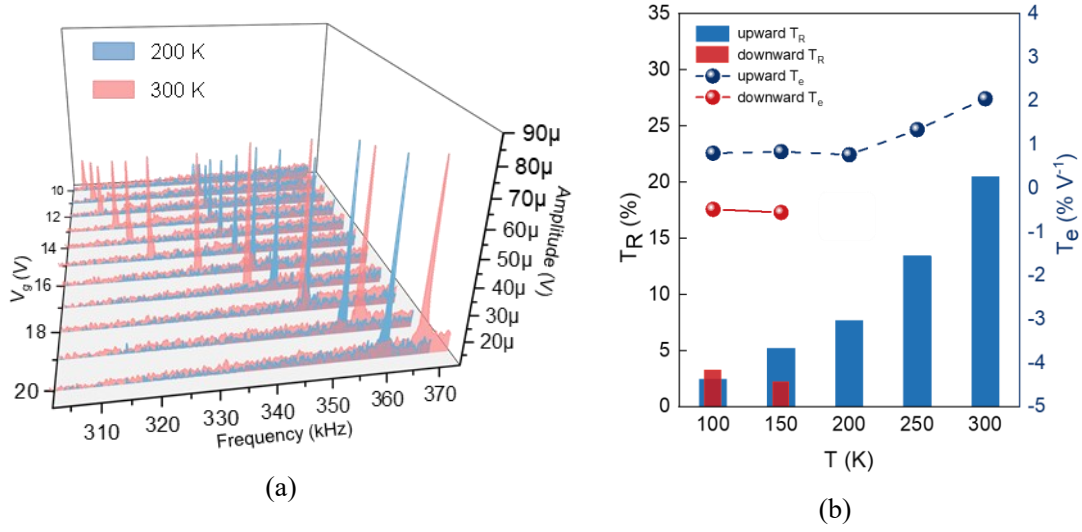


Figure 3-10 (a) The experimental results of resonance response detected from a device with dimensions of 200 μ m in length and 80 nm in width as gate voltage (V_g) varies from 10 V to 20 V under substrate temperatures of 300 K and 200 K, respectively. (b) The measured resonance frequency tuning range (T_R) (bar chart), alongside the tuning efficiency (T_e) (dot-line chart), where the T_e is calculated by T_R divided by the applied voltage range. The upward tuning condition and downward condition are separately presented.

Further, a nanoresonator device with 80 nm width and 200 μ m length is chosen for the investigation of the electrostatic resonance frequency tuning behavior under varying temperatures within the vacuum chamber. The substrate temperature in the vacuum chamber is reduced from room temperature (~ 300 K) down to 100 K in the intervals of 50 K. At each temperature, the nanoresonator's frequency responses are measured in its linear response regime with a constant AC drive voltage of 1 mV, while the V_g is varied from 10 V to 20 V. Fig. 3-10a shows the detected frequency response at 300 K and 200 K. In both temperature conditions, an increase in resonance frequency is observed with increasing V_g , indicating an upward electrostatic frequency tunability. This upward tuning in the resonance frequency with increasing V_g is attributed to a static deflection of the nanoresonator due to the applied electrostatic force, which increases the axial strain of the nanoresonator. The significant role of the axial strain on the resonance frequency is appreciable even at the lowest V_g (10 V) since the measured frequency (f_0) is ~ 17.3 times (in 300 K condition) and 19.1 times (in 200 K condition) larger than what would be expected from an equivalent tension-free Euler-Bernoulli beam.²³ In the plot of Fig. 3-10a, notably, at 200 K, a higher f_0 at V_g of 10 V and a

lower f_0 at 20 V are recorded compared to the observation made at 300 K, indicating an increased initial resonance frequency (f_0 at $V_g = 10$ V) and a narrower electrostatic tuning range and efficiency. To quantify such measurements, we define the tuning range for both upward tuning trend and downward tuning trend, where the upward tuning range $T_{R-upward} = (f_u^{max} - f_0^{min})/f_0^{min} \%$, where f_0^{min} , and f_u^{max} are respectively the minimum and the upward tuned maximum resonance frequencies measured in the V_g range of 10 – 20 V. Similarly, the downward tuning range $T_{R-downward} = (f_d^{max} - f_0^{min})/f_d^{max} \%$, where f_d^{max} is the maximum resonance frequencies during downward tuning. We also define a local tuning efficiency (T_e) for both tuning trends which is the ratio between the upward ($T_{R-upward}$) or downward tuning range ($T_{R-downward}$) and the voltages required for such tuning. In Fig. 3-10a, the measured T_R achieved diminished from 20.5% at 300 K to 7.5% at 200 K. The bar plot in 3-10b summarizes the measured resonance frequency tuning range and the circle-dash line plot shows the corresponding tuning efficiency T_e as V_g is varied between 10 V and 20 V across different substrate temperatures. Note that the frequency tuning trend can be non-monotonic, viz. the minimum frequency is observed at an intermediate value of V_g . In such a scenario, with increasing V_g , a part of the tuning data will show a downward frequency tuning trend, while the other part will show an upward frequency tuning trend. In the plot of Fig. 3-10b, the downward trends are indicated by red bars in the plot, while the upward frequency trends are shown in blue bars. A consistent decrease in T_R is observed with the reduction of temperature below 300 K. The downward tuning trend is observed in temperature conditions of 150 K and 100 K. The upward T_e decreases from the condition of 300 K to 200 K, and becomes relatively stable in the range from 200 K to 100 K. Meanwhile, downward T_e shows a certain increase when the temperature conditions change from 150 K to 100 K.

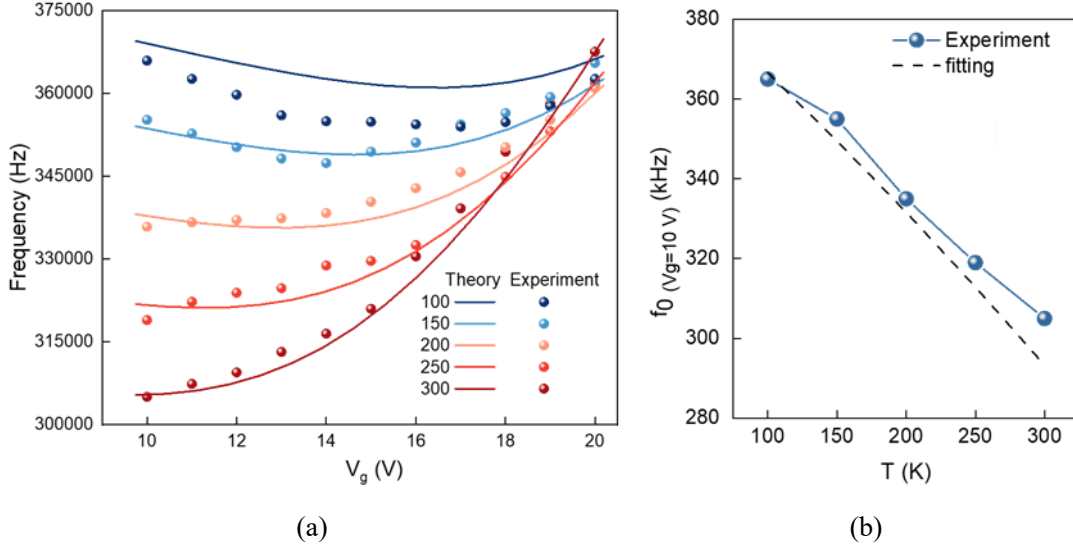


Figure 3-11 (a) The estimated resonance frequency tuned by gate voltage during the device's temperature decreases from 300 K to 100 K based on the approximate tuning model with the experimental measured results. (b) The experimental frequency data at $V_g = 10$ V fitted with the theoretically expected frequency value.

The results of electrostatic tuning of resonance frequency conducted at various temperature conditions are shown in the plot of Fig. 3-11a. Beside the reduction in T_R as mentioned above, a non-monotonic trend in tuning emerges at lower temperature. The downward trend in frequency tuning signifies a dominant role of the electrostatic softening effect²⁴ over the deflection induced stretching of the nanoresonator, leading to an overall downward tuning of the resonance frequency. The competition between these two mechanisms can be understood by an approximate electromechanical model of the nanoresonator, which can approximately predict the resonance frequency,

$$f_0(V_{dc}, T) = \frac{1}{2\pi} \sqrt{\left(J_2 \frac{Et^2}{6\rho L^4} + J_1 \frac{2E\varepsilon(T)}{\rho L^2} \right) - \left(\frac{o_2 \zeta \epsilon_0}{\rho t g_0^3} \right) V_{dc}^2 + \left(\frac{3o_1^2 \zeta^2 \epsilon_0^2}{4\rho g_0^4 Et^2 \varepsilon^2(T)} \right) V_{dc}^4} \quad 3-17$$

Where, $\varepsilon(T)$ is the initial axial tensile strain at $V_g = 0$ V and at a given temperature (T), The first term under the square-root symbol in equation (3-17) represents the initial resonance frequency without the effect of V_g , predominantly influenced by $\varepsilon(T)$; the second term indicates the decrease in f_0 , which is contributed by the electrostatic softening effect; and the final term indicates the stretching effect. The actual frequency tuning trend is determined by the competition between these last two terms, while the initial frequency increases with an increase in $\varepsilon(T)$.

In Fig. 3-11b, $f_0(V_g = 10 \text{ V})$ measured at different temperatures are plotted. We fit this trend using

equation (3-17) to extract the value of $\Delta\alpha_{th} \sim 1.3 \times 10^{-6} \text{ K}^{-1}$, and $\varepsilon_0(300 \text{ K}) \sim 23 \text{ MPa}$. It is worth noting that the increase in $f_0(V_g = 10 \text{ V})$ with decreasing temperature suggests an increase in $\varepsilon(T)$ in the resonator. This explains the variation in the tuning trend (Fig. 3-11a), which goes from an upward trend at higher temperatures ($T = 300, 250, 200 \text{ K}$) to non-monotonic at lower temperatures ($T = 150, 100 \text{ K}$).

The results shown in Fig. 3-10 and 3-11 show the temperature dependence of the electrostatic tunability of resonance frequency in an ultrathin Si nanoresonator between 100 K and 300 K, where a strong relationship between temperature and tunability is observed. The results not only indicate a significant decrease in tuning range and efficiency but also show a more complex behavior in terms of the trend of the frequency tuning, suggesting that the stage temperature control can be an effective method to control the electrostatic tunability. Thus, a dual tuning strategy of the nanoresonator by V_{dc} and T can provide further opportunities in terms of on-the-spot adjustment of the nanoresonator's frequency as demanded in specific applications.

3.4.3 Tunable temperature coefficients of frequency

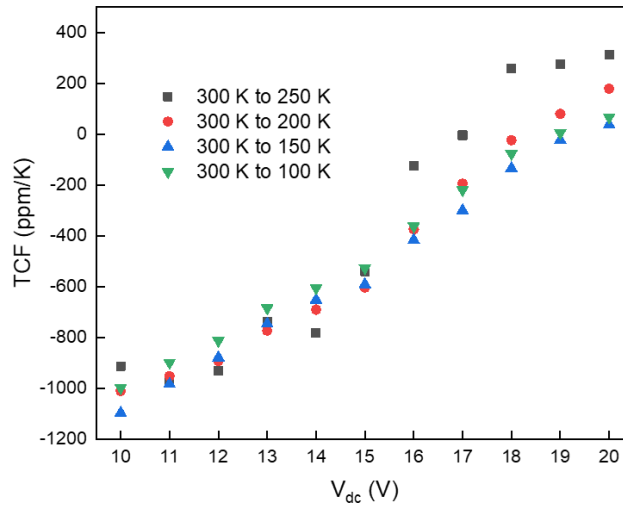


Figure 3-12 The measured temperature coefficients of frequency (TCF) calculated at different temperature variation ranges changes with the increase of V_{dc} .

Another noteworthy observation from Fig.3-12 is the span of resonance frequencies across the temperature range (100 – 300 K) shrinks with increasing V_{dc} . The resonance frequency span indicates the temperature fluctuation-induced variability in resonance frequency (thermal drift) of the device during operation, which is a critical factor in the design and performance of resonator

devices used in telecommunications, navigation, and timekeeping.²⁵ A wider frequency span suggests higher responsivity to temperature fluctuations, whereas a smaller span indicates better temperature stability. It is important to note that the higher temperature responsivity of the resonance frequency of the device is not always undesirable; in applications such as resonator-based temperature sensors, it can be advantageous. Compared to MEMS resonators, NEMS resonators typically exhibit higher responsivity to temperature fluctuation because they are more susceptible to strain change, making them well-suited for temperature sensing but less desirable in applications where frequency stability is important (such as in timing devices). The temperature coefficients of frequency (TCF) defined as the relative resonance frequency shift per degree Kelvin of temperature change is commonly used for evaluating such temperature responsivity. In recent reports, the TCF of conventional NEMS resonators, such as MoS₂ resonator and h-BN resonator, are around -3960 ppm/K²⁶ and -2850 ppm/K²⁷, respectively. For Graphene resonators, this value is much higher, which can reach around -8600 ppm/K. In contrast, the conventional MEMS resonator exhibits a typical TCF in the range of -20 to -100 ppm/K.^{28, 29}

The measured device's TCF at different V_{dc} is estimated by calculating the resonance frequency shift per degree Kelvin as the temperature decreased from 300 K to 250 K, 200 K, 150 K and 100 K, as shown in Fig. 3-12. Initially, the TCF exhibits negative values and it increases as V_{dc} increases, and it subsequently becomes positive values. The lowest observed absolute TCF value is approximately 3.6 ppm/K at V_{dc} of 17 V, comparable to conventional MEMS resonators, while the initial TCF with V_{dc} =10 V was around -1096 ppm/K, aligning with typical NEMS resonators. Both the values and the sign of TCF can be tuned by adjusting V_{dc} which indicates the direction of frequency shift relative to the temperature variation can also be tuned: A positive TCF implies that the frequency increases with rising temperature, whereas a negative TCF suggests a decrease in frequency as temperature increases. These results demonstrate that the proposed gate voltage-controlled method effectively adjusts the resonators' temperature responsivity, enabling the dual functions of temperature sensing and temperature stability within the same device.

3.5 Conclusion

In this chapter, we demonstrate wide electrostatic tuning of resonance-frequency of fabricated ultrathin Si NEMS resonator devices, monolithically fabricated from conventional SOI wafers using a scalable microfabrication process. The resonance-frequency tuning-range ($\sim 70\%$) and tuning-efficiency ($\sim 7\% \text{ V}^{-1}$) achieved in our devices are comparable to values reported for atomically thin nanoresonators at room temperature. Theoretical modeling, based on an electromechanical potential framework, explained the observed gate voltage induced frequency tuning by considering the interplay of electrostatic forces and mechanical restoring forces. Numerical analysis aligned well with experimental data, validating our theoretical approach and offering deeper insights into the mechanisms governing resonance frequency tuning. The study also examined the temperature dependence of resonance frequency tunability. It was observed that lower temperatures result in a reduced tuning range and efficiency, with tuning trends shifting from upward to non-monotonic at very low temperatures. This behavior was attributed to the competing effects of electrostatic softening and deflection-induced stretching, influenced by the initial axial tensile strain and thermal expansion differences.

The ability to achieve ultrawide tunability in microfabricated Si resonator devices will facilitate rapid commercialization of ultrathin, tunable nanoresonators for many applications. The potential of such wide frequency tunability on improving resonator sensing performance will be further explored in Chapter 5.

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Chapter 4 Nonlinear Behaviors of Ultrathin Silicon NEMS Resonator

4.1 Introduction

Nonlinearity in the dynamic behavior of nanomechanical resonators plays a vital role in many applications,¹ such as, determining the linear dynamic range of resonant sensors,² ensuring frequency and amplitude stability,³ designing mechanical filters and frequency mixers,⁴ and achieving strong coupling for quantum information processing.⁵ Additionally, NEMS resonators also open a new practical window for studying the systems' nonlinear dynamics since downsizing the resonators makes the resonance response easily transit from the linear to nonlinear regime. With increasing amplitude of vibration, nanoresonators' dynamic-behavior shifts to nonlinear-regime⁶ discernable by the emergence of jump and hysteresis phenomenon in $A(f) - f$ plots. Typically, a cubic nonlinear-stiffness-coefficient (α) is considered along with the linear-stiffness (k) of nanoresonators to account for these effects. Magnitude of α controls the strength of nonlinearity while its sign controls the qualitative behavior of the nanoresonator in the nonlinear regime. Ability to tune α by V_{dc} is beneficial for many practical applications of nanoresonators. For example, a broad linear-dynamic-range, achieved by reducing α , is advantageous for achieving high signal-to-noise ratio.

The complex and abundant nonlinear dynamic phenomena can lead to both challenges and opportunities for superior applications. For instance, exploiting the bifurcation point in the hysteretic frequency response to achieve broadband resonance⁷ and improve the resonator's responsivity^{8,9}, developing logical gate through bistability of forced vibrations¹⁰, realizing vibration energy harvesting¹¹ and frequency stabilization¹² by nonlinear mode coupling^{13,14}. Notably, the intermodal coupling strength between different modes in the nonlinear regime will intensify if internal resonance (IR) occurs.

This chapter explores the nonlinear behaviors of ultrathin silicon NEMS resonators, focusing on their dynamic responses under various driving conditions. We investigate the transition from linear to nonlinear regimes, the tuning of nonlinear coefficients, linear dynamic range and the activation of internal resonance. Our experimental observations and theoretical analyses reveal the complex

dynamics of these resonators, highlighting their potential for high-performance applications in sensing, signal processing, and energy harvesting.

4.2 Nonlinear response and dynamic range

4.2.1 Experimental results of nonlinear response

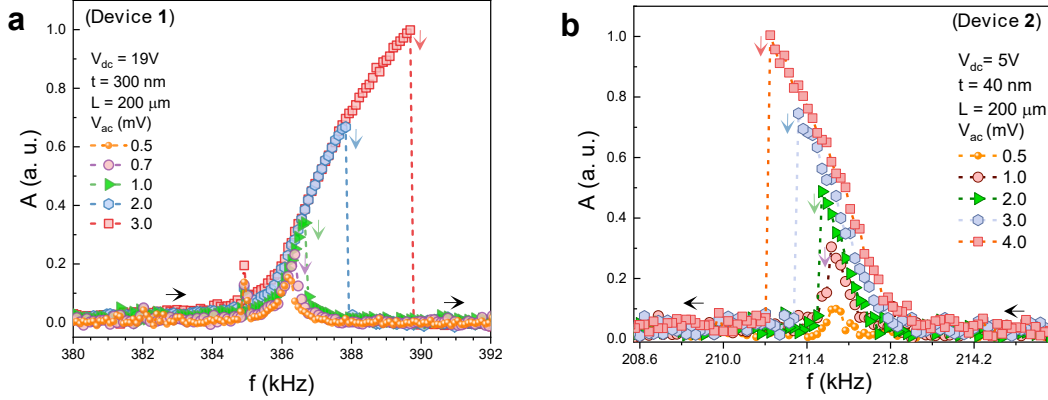


Figure 4-1 Tuning of nonlinear behavior of ultrathin Si NEMS resonator devices. (a) Hardening-type ($\alpha > 0$) cubic-nonlinear behavior observed in a $t \sim 300$ nm nanoresonator device ($L \sim 200$ μ m, $w \sim 7.5$ μ m, $g_0 \sim 2$ μ m) at $V_{dc} = 19$ V as V_{ac} is varied. (b) In a $t \sim 40$ nm nanoresonator device ($L \sim 200$ μ m, $w \sim 7.5$ μ m, $g_0 \sim 2$ μ m) softening-type ($\alpha < 0$) (electrostatic) cubic-nonlinear behavior is observed to dominate at $V_{dc} = 5$ V with varied V_{ac} . All measurements are done at room temperature, Q -factor are estimated to be around 1000.

In the Chapter 3.2, the linear resonance response of Device 1 and 2 have been thoroughly analyzed, demonstrating their capability to achieve significant frequency tuning through the application of varying DC gate voltages. Here, the frequency response of Device 1 and 2 in the nonlinear regime are shown in Fig. 3-1a and Fig. 3-1b, respectively. In both cases, $V_{dc} = 19$ V and 5 V, respectively, are kept fixed, while V_{ac} is varied for each of the multiple frequency-sweeps shown. Nonlinear-behavior in the frequency-response is observed in both cases as V_{ac} is increased, however, in Device 1 ($t \sim 300$ nm) a hardening-type nonlinearity persisted at $V_{dc} = 19$ V, while in Device 2 ($t \sim 40$ nm) a softening-type nonlinearity is observed at $V_{dc} = 5$ V. The observed nonlinear behavior can be attributed to the interplay between geometric nonlinearity and electrostatic nonlinearity in the devices. In electromechanical resonators like our NEMS devices, geometric nonlinearity arises from the stretching of the resonator, which typically results in a hardening-type nonlinearity (positive α). On the other hand, the nonlinearity of the electrostatic

field tends to contribute a softening-type nonlinearity (negative α). The overall nonlinear response of the device is determined by the relative strengths of these two contributions. For instance, in Device 1, the hardening-type nonlinearity dominates due to the significant geometric stretching at $V_{dc}=19$ V. Conversely, in Device 2, the electrostatic softening effect is more pronounced at $V_{dc}=5$ V, leading to a softening-type nonlinearity.

4.2.2 Nonlinearity fitting and analysis

Building on the experimental observations of nonlinear response in Device 1 and Device 2, it is crucial to comprehensively understand the different types of nonlinearities observed between them. In this section, we will systematically analyze the nonlinear response curves by fitting them to theoretical models, thereby extracting key parameters that characterize the nonlinear dynamics of the devices.

Nonlinearity appears in the dynamic-behavior of nanoresonators at large amplitudes. In the present case, cubic nonlinearity in the stiffness of the nanoresonators manifests in the frequency response curves as characteristic ‘jumps’ (as shown in Figure 3-1) as the resonator’s vibrational displacement (y) is increased by exerting higher actuation forces (here, y represents the dynamic displacement of the nanoresonator-center as opposed to the static deflection represented by the same symbol in section 3.3.1). The cubic nonlinearity term introduces a nonlinear force term of the form $-\alpha y^3$ in the equation of motion, where the cubic nonlinear stiffness coefficient, α can potentially be positive (thus effectively hardening the stiffness at large amplitude), i.e., ‘hardening’ type, or negative (which softens the effective stiffness at large amplitude), i.e., softening type. In addition, nonlinearity in damping may also appear, where the effective damping increases at large y . However, we will show, in the present case, the effect of nonlinear damping is negligible.

The equation of motion corresponding to a lumped element mass-spring-damper model of a nanoresonator of effective mass, m_{eff} , linear stiffness, k , linear damping coefficient, Γ , cubic nonlinear stiffness, α , and (for the time being) assuming a nonlinear damping coefficient, η (for a nonlinear damping force of the form, $-\eta y^2 \dot{y}$), can be written as:

$$m_{\text{eff}} \ddot{y} + (\Gamma + \eta y^2) \dot{y} + ky + \alpha y^3 = F_0 \cos \omega t \quad 4-1$$

where, F_0 is the amplitude of a harmonic driving force of angular frequency, ω . Following Lifshitz and Cross¹⁵, the steady-state amplitude response of the resonator ($A(\omega)$) for periodic solutions can be expressed as:

$$A^2 = \frac{\left(\frac{F_0}{2m_{\text{eff}}\omega_0^2}\right)^2}{\left(\frac{\omega - \omega_0}{\omega_0} - \frac{3}{8}\frac{\alpha}{m_{\text{eff}}\omega_0^2}A^2\right)^2 + \left(\frac{1}{2Q} + \frac{1}{8}\frac{\eta}{m_{\text{eff}}\omega_0}A^2\right)^2} \quad 4-2$$

where, ω_0 is the natural angular frequency of the nanoresonator, and $Q = \frac{m_{\text{eff}}\omega_0}{r}$ is the Q-factor corresponding to linear damping of the nanoresonator. A typical plot of $A(\omega)$ looks like a ‘tilted’ Lorentzian peak, where the peak position shifts away from ω_0 , towards a higher frequency for $\alpha > 0$ or a lower frequency for $\alpha < 0$. Beyond a critical amplitude, $A(\omega)$ admits three real solutions in an intermediate regime of ω , two of them, the highest and the lowest, correspond to stable solutions, and in-between lies an unstable branch of solutions. In experiments, if ω is swept along the largest amplitude branch, the response amplitude, $A(\omega)$ reaches a peak-value, $A_{\text{max}}(\omega_{\text{max}})$, typically, at the end of that branch (i.e., at the peak of the tilted Lorentzian) at $\omega = \omega_{\text{max}}$, wherein it jumps down to the lowest amplitude branch (Fig. 3a). To analyze the nonlinear response curve, it is beneficial to cast equation (4-2) in the following form:⁵

$$\frac{\omega - \omega_0}{\omega_0} = \frac{3}{8}\frac{\alpha}{m_{\text{eff}}\omega_0^2}A^2 \pm \sqrt{\left(\frac{F_0}{2m_{\text{eff}}\omega_0^2}\frac{1}{A}\right)^2 - \left(\frac{1}{2Q} + \frac{1}{8}\frac{\eta}{m_{\text{eff}}\omega_0}A^2\right)^2} \quad 4-3$$

Bistability in $A(\omega)$ corresponds to the regime where the quantity under the square root in equation (4-3) is positive. The term before it is responsible for forming the so-called backbone of the nonlinear curves. $A_{\text{max}}(\omega_{\text{max}})$ arises for the value of A, where the quantity under the square root becomes zero. Thus, the condition for Peak-amplitude ($A = A_{\text{max}}$), is attained when,

$$\left(\frac{F_0}{2m_{\text{eff}}\omega_0^2}\frac{1}{A_{\text{max}}}\right)^2 - \left(\frac{1}{2Q} + \frac{1}{8}\frac{\eta}{m_{\text{eff}}\omega_0}A_{\text{max}}^2\right)^2 = 0 \quad 4-4$$

Reorganization of terms leads to the relation,

$$\frac{F_0Q}{k}\frac{1}{A_{\text{max}}} - 1 = \frac{1}{4}\frac{Q\eta\omega_0}{k}A_{\text{max}}^2 \quad 4-5$$

It should be noted from equation (4-5), in the absence of nonlinear damping (i.e., when $\eta = 0$), A_{max} is identical to the expected amplitude for an equivalent linear oscillator (even though the Lorentzian shape could be bent, depending on the value of α), i.e., $A_{max} = \frac{F_0 Q}{k}$, and $\frac{A_{max}}{F_0}$ ratio remains unchanged with increasing F_0 . For $\eta > 0$, $\frac{A_{max}}{F_0}$ ratio would decrease with increasing F_0 . Thus, observation of a linear variation in A_{max} versus F_0 plot in the experimental data would indicate negligible nonlinear damping⁶. In our experiment, F_0 is varied by varying the amplitude of the ac actuation voltage, i.e., $F_0 = s_1 V_{ac}$ (keeping V_{dc} fixed), while the proxy for amplitude of vibration, A , is the amplitude of the transduced electrical voltage (V_m), where, $A = s_2 V_m$ (s_1 and s_2 are constants of proportionality). A_{max} corresponds to the value of V_m at peak (V_{max}), $A_{max} = s_2 V_{max}$. Thus, in terms of experimental parameters, equation (4-5) can be written as,

$$\frac{s_1 V_{ac} Q}{k} \frac{1}{s_2 V_{max}} - 1 = \frac{1}{4} \frac{Q \eta \omega_0}{k} s_2^2 V_{max}^2 \quad 4-6$$

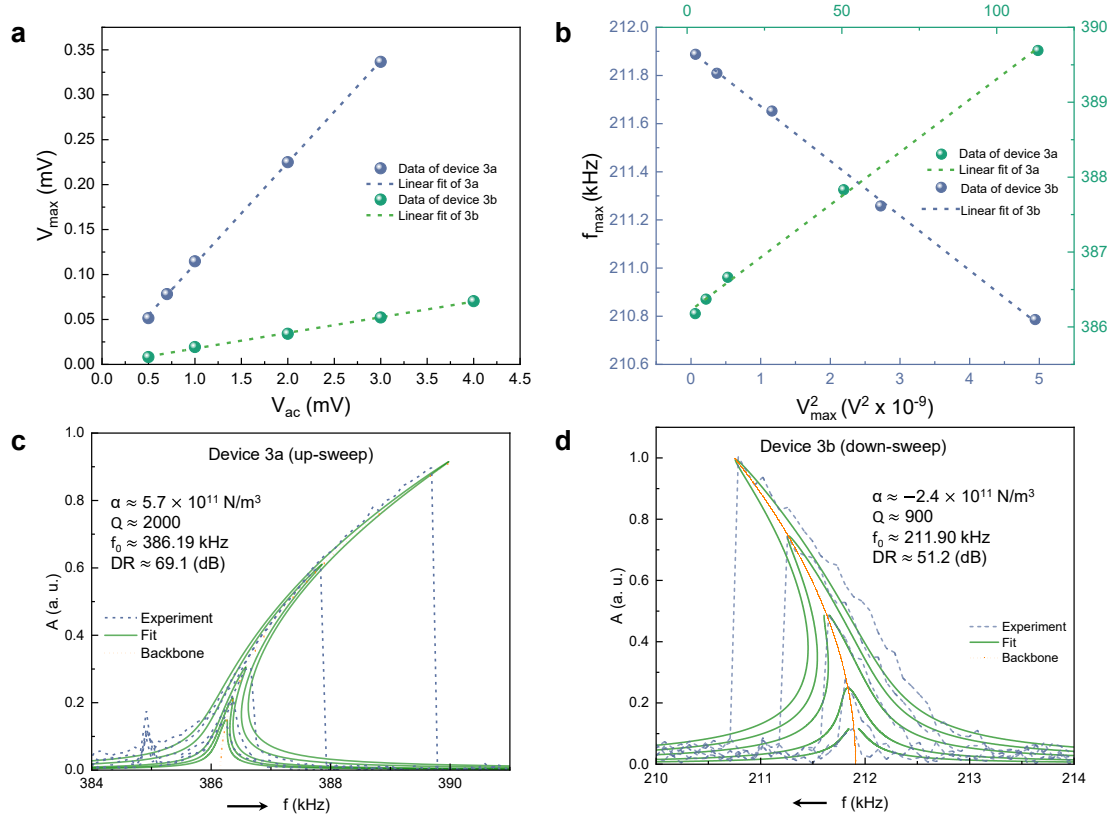


Figure 4-2 Analysis of nonlinear behavior of the devices shown in Fig. 4-1a and Fig. 4-1b. a, Peak-amplitude (amplitude at jump), V_{max} versus ac actuation voltage, V_{ac} . b, Jump-frequency, f_{max} versus V_{max}^2 . c-d Fitting of nonlinear resonance curves.

In Fig. 4-2a, we plot V_{max} versus V_{ac} for Device 1 (forward-sweep data shown Fig. 4-1a), and for Device 2 (reverse-sweep data shown in Fig. 4-1b), where linear relationships are observed (indicated by the linear fitting lines). Therefore, we conclude that $\eta = 0$ would be a reasonable assumption for the experiments presented in this work. Under this assumption,

$$\frac{V_{max}}{V_{ac}} = \frac{s_1 Q}{k} \frac{1}{s_2} = \left(\frac{s_1}{s_2}\right) \frac{Q}{k} \quad 4-7$$

Subsequently, from equation (4-3),

$$\frac{\omega_{max} - \omega_0}{\omega_0} = \frac{3}{8} \frac{\alpha}{m_{eff} \omega_0^2} A_{max}^2 \quad 4-8$$

In terms of experimental parameters, the above equation is written in the following way:

$$f_{max} = f_0 + \frac{3}{8} \frac{\alpha}{k} f_0 s_2^2 V_{max}^2 \quad 4-9$$

where, $f_0 = \frac{\omega_0}{2\pi}$ and $f_{max} = \frac{\omega_{max}}{2\pi}$. Plots of f_{max} versus V_{max}^2 yield linear plots as shown in Fig. 4-2b. f_0 is the intercept of the linear fits in Fig. 3-8b, from which corresponding, $k = 4\pi^2 m_{eff} f_0^2$ are evaluated. Making reasonable estimations for Q from the frequency response in the linear regime and assuming $s_1 \approx \frac{\epsilon_0 L W}{g_0^2} V_{dc}$, respective values of s_2 are calculated from the slopes of the linear fits in Fig. 4-2a. Finally, the nonlinear coefficient (α), of the nanoresonators are extracted from the slopes of the linear fits in Fig. 4-2b. With the extracted values of the fitting parameters (values shown in Fig. 4-2a-b), the nonlinear response curves of Device 1 and Device 2 are fitted, as shown in Fig. 4-2c and Fig. 4-2d, respectively. The fitting curves appear to agree well with the experimental data. Evidently, for Device 1, hardening type ($\alpha > 0$) nonlinearity is observed, while for Device 2, softening type nonlinearity is observed ($\alpha < 0$). In electromechanical resonators, such as our nanoresonators, the hardening-type nonlinearity is caused by geometric nonlinearity due to the stretching of the resonator, while nonlinearity of the electrostatic field contributes to a softening-type nonlinearity. The actual value and sign of the cubic coefficient are determined by the relative strength of these two types of nonlinearities.

4.2.3 Tuning and cancellation of nonlinearity

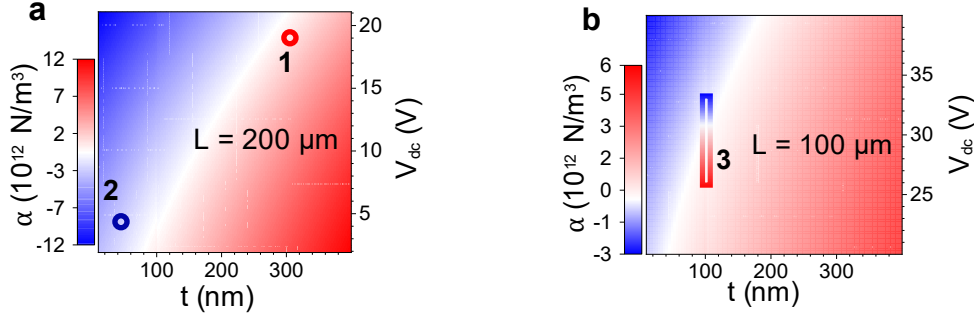


Figure 4-3 Colormaps representing variation of α with t and V_{dc} for (a), Devices 1, 2, (b) Device 3. Colormaps are generated using $\phi_2(x)$ for $t \leq 100 \text{ nm}$ and $\phi_1(x)$ for $100 \text{ nm} < t \leq 300 \text{ nm}$.

To quantitatively analyze the data illustrated in Figure. 4-1, we extend the potential energy model presented in section 3.3.1, to derive an approximate expression for α , since $\alpha = \left[\frac{d^4(U_m + U_e)}{dy^4} \right]_{y_0}$ implies,

$$\alpha \approx \frac{3J_1^2 E w t}{L^3} - \frac{3\zeta o_4 \epsilon_0 L w}{g_0^5} V_{dc}^2 \quad 4-10$$

The first term in α represents a hardening-type nonlinearity, while the second term (which depends on the dc gate-voltage, V_{dc}) represents a softening-type nonlinearity. Equation (4-10) indicate that α can be tuned by V_{dc} , the extent of which can be especially significant for narrower and longer nanoresonators. Equation (4-10) suggests that the cubic nonlinearity coefficient, α , can be tuned by V_{dc} in the same device, and at $V_{dc} \approx \frac{J_1}{L^2} \sqrt{\frac{E t g_0^5}{\zeta o_4 \epsilon_0}}$, α can be effectively cancelled. Further increase in V_{dc} will make α go from positive to negative. We show the variation of $\alpha(t, V_{dc})$ in the colormap of Fig. 4-3 using equation (4-10), which elucidates the behavior described in Fig. 4-1. Equations (4-10) and results from devices 1 and 2 suggest that nonlinear behavior of our nanoresonators can be significantly tuned by V_{dc} . We demonstrate this in a device (Device 3) with $t \sim 100 \text{ nm}$, $L \sim 100 \mu\text{m}$, and $g_0 \sim 2 \mu\text{m}$, where the nonlinear-response is gradually flipped from hardening-type ($\alpha > 0$) to softening-type ($\alpha < 0$) by varying V_{dc} .

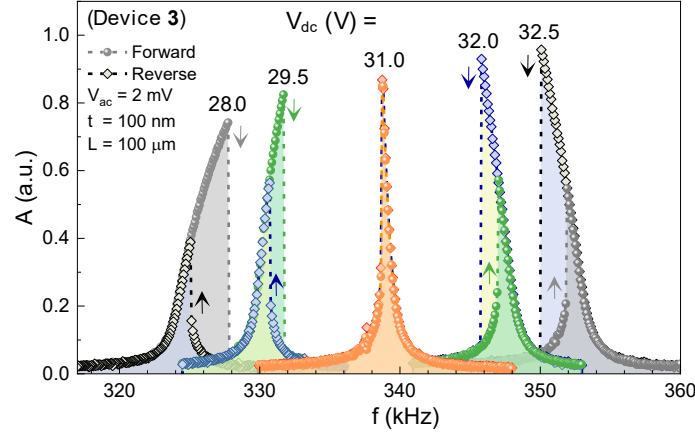


Figure 4-4 Tuning of α by V_{dc} shown in Device 3 (frequency tuning for this device is shown in Fig. 3-2c in section 3.2.1); α is gradually changed from $\alpha > 0$ to $\alpha \approx 0$ to $\alpha < 0$. All measurements are done at room temperature, Q -factor are estimated to be around 1000.

At an intermediate $V_{dc} \approx 31$ V, α is effectively cancelled and linear-response is restored, as indicated by the disappearance of jump and hysteresis in the frequency-sweep (Fig. 4-4). At this point, the linearity of the device is limited by higher-order nonlinearities (otherwise shadowed by the stronger-effect of cubic-nonlinearity), when the vibration amplitude is further increased. Gate-voltage induced cancellation of α (and $\alpha > 0$ to $\alpha < 0$ transition) is shown previously in Graphene and MoS₂ resonators and a partial cancellation is reported in a Si microresonator.¹⁶ For Device 3 described here, this transition is observed at $V_{dc} \approx 31$ V. In Fig. 4-5, this transition is further investigated, where the effect of higher order nonlinearities could be observed around the V_{dc} corresponding to the cancellation of α . Since α is the dominant source of nonlinearity in the system, the linear dynamic range of the nanoresonator significantly broadens near the α cancellation point.

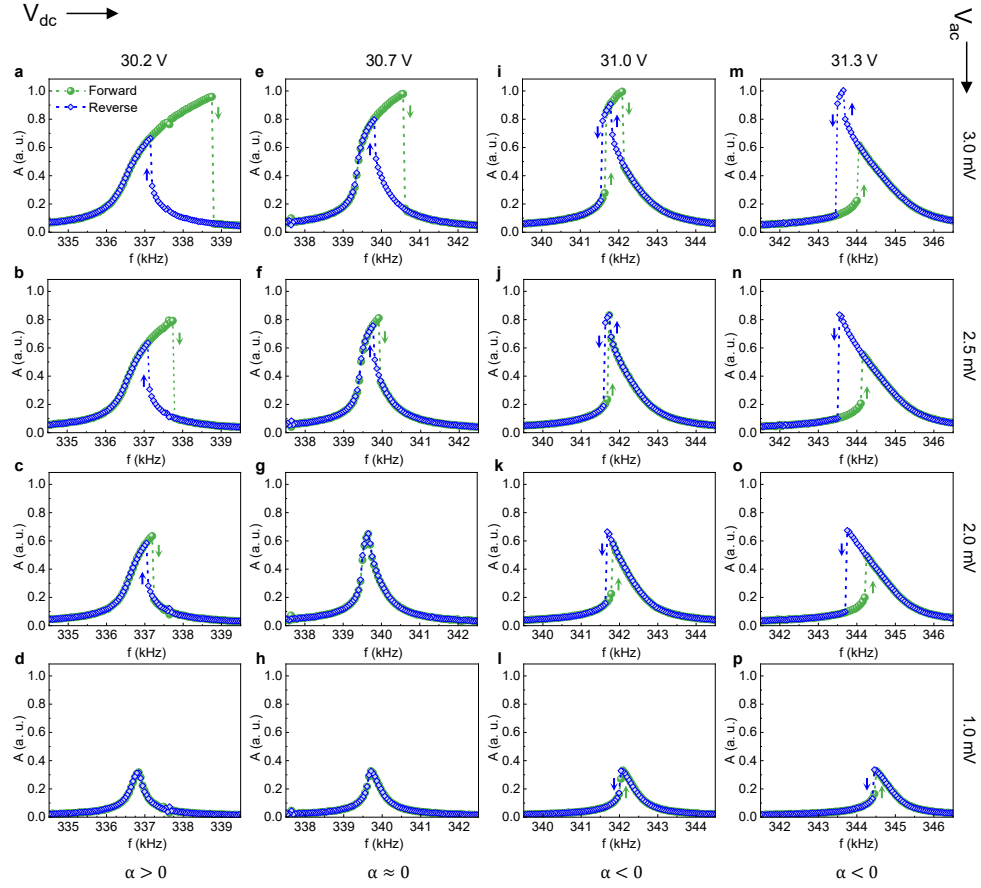


Figure 4-5 Nonlinear response of Device 3 around the cubic-nonlinearity cancellation condition. V_{dc} is kept fixed for plots on each column, while V_{ac} is varied and both forward and reverse frequency sweeps are conducted. Effect of higher-order nonlinearity, likely a hardening-type quintic nonlinearity, becomes apparent by the emergence of mixed nonlinear response (simultaneous hardening and softening type response); see especially the sub-figure for $V_{dc} = 31$ V, $V_{ac} = 3$ mV. Since, the higher order nonlinearity appears to be of hardening-type (for the V_{dc} values presented here), its effect becomes prominent at a V_{dc} value where the cubic-nonlinearity coefficient is slightly below zero, and V_{ac} is large.

4.2.4 Linear dynamic range

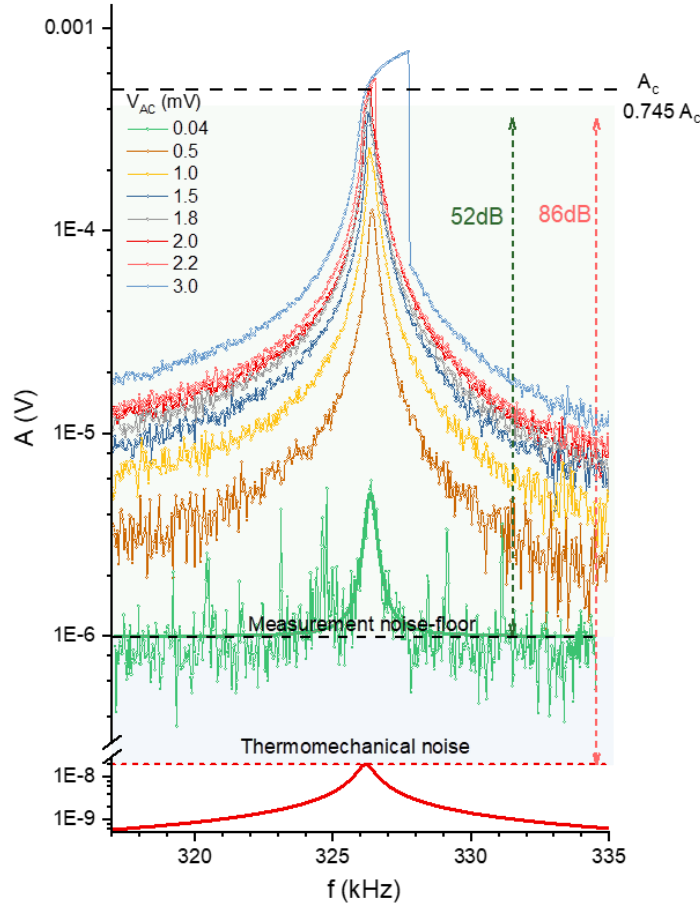


Figure 4-6 Estimation of linear dynamic-range (DR) of Device 3 close to the cubic-nonlinearity (α) cancellation condition ($V_{dc} = 28.2$ V). Subsequent measurements represent the frequency response for increasing V_{ac} . Intrinsic DR estimated in reference to theoretically expected thermomechanical noise-floor is about 86 dB, while the measured DR estimated in reference to measurement noise-floor is about 52 dB.

A direct consequence of cancelling the cubic-nonlinearity is realized via the broadening of the linear-dynamic-range (DR) of nanoresonators, which is a measure of the range of linear-response with respect to the thermomechanical noise-floor. A broad DR indicates the ability to detect weak signals while handling strong signals without distortion or saturation and is beneficial for many applications, such as, sensing, filtering, signal processing, *etc.*

The upper limit of the DR (U_{DR}) is the linear signal ceiling usually defined as the 1 dB compression point of the critical amplitude (A_c) of the nanoresonator,¹⁷ which is $\sim 0.745 A_c$. As a

fundamental noise sources in nanoresonator devices, thermomechanical noise arising from thermal vibration of the nanoresonator is considered to be the intrinsic noise-floor of the nanoresonator device, thus, constitutes the lower limit of the intrinsic DR (L_{DR}). The dynamic ratio ($r_D = U_{DR}/L_{DR}$) is the ratio of this upper and the lower limit, then linear dynamic range can be calculated as $DR = 20\log_{10} r_D$.

As shown in Fig. 4-6, the critical amplitude in the case of device 3 is experimentally extracted by carefully increasing the V_{ac} during measurement. Then the 1 dB compression point of the critical amplitude is calculated and used as U_{DR} for calculating the r_D . Since, in our current detection system, the thermomechanical noise-floor cannot be experimentally detected, L_{DR} is estimated by theoretically calculating the nanoresonator's displacement amplitude density spectra due to thermomechanical motion (assuming the experimentally measured Q -factor, resonance-frequency, and $T = 300$ K), which is then converted to a corresponding voltage output expected from our capacitive detector and measurement circuit (assuming ideal, noise-free condition) using a bandwidth value equal to the bandwidth used for collecting the experimental data. The peak value of this voltage signal is then considered as L_{DR} . The estimated intrinsic linear DR is about ~ 86 dB.

In order to compare this DR value with that of devices that operate significantly away from the α cancellation point (such as in device 1, and 2), we use the measurement shown in Fig. 4-1. r_D in these cases are estimated based on fitting the multiple frequency runs shown in Fig. 4-1, and using a standard formula² $r_D = 0.327 \sqrt{\frac{m_{\text{eff}}^2 \omega_0^5}{k_B T Q^2 \Delta f |\alpha|}}$ where, k_B is the Boltzmann's constant, Δf , T are respectively the measurement bandwidth¹⁸ (used in Fig. 4-1) and temperature (300 K); α , f_0 , Q are fitted cubic nonlinearity, resonance frequency and quality factor, m_{eff} is the effective mass of the nanoresonator, and $\omega_0 = 2\pi f_0$. The estimated intrinsic linear DR for device 1 and device 2 are ~ 69.1 dB, and ~ 51.2 dB, respectively. It is observed that the DR is highly enhanced for the case of device 3. This is because it was operated around the point where α is almost cancelled by V_{dc} , while device 1 and device 2 were not operated near the cancellation point.

In this regard, it might be relevant to mention experimental detection of thermomechanical noise has been achieved in other resonators, thus, a direct and more accurate estimation of the intrinsic DR is experimentally possible. However, noise in the measurement system often elevates the

measurement noise-floor significantly above the thermomechanical noise-floor, such as in the present case. Thus, it may be relevant to define a measured DR by assuming the measurement noise-floor as the lower limit for DR. For Device 3, the measured DR is ~ 52 . Also, it is worth mention that both α and ω_0 are widely tunable by V_{dc} , the intrinsic DR in our Si nanoresonator devices can also be tuned by choosing a suitable pair of (t, V_{dc}) .

4.3 Nonlinear modal interaction

Nonlinear modal interactions in ultrathin Si NEMS resonators are crucial for understanding their dynamic behavior under various operating conditions. In these resonators, different vibrational modes can interact nonlinearly, leading to complex phenomena such as energy transfer between modes, frequency mixing, and internal resonance.¹⁹ These interactions can significantly influence the performance of the resonators, particularly in applications requiring precise control of their dynamic response.²⁰ In this section, we will present the internal resonance behavior observed in our developed resonator devices, along with a brief theoretical introduction to energy transfer and internal resonance.

4.3.1 Energy transfer and internal resonance

When a NEMS resonator is driven at high amplitudes, the energy can transfer between different vibrational modes due to nonlinear interactions. This energy transfer can manifest as shifts in resonance frequencies and changes in the amplitude response of the modes. The strength and nature of these interactions depend on factors such as the geometry of the resonator, material properties, and the type and amplitude of the driving force.²¹

The primary mechanism behind these interactions is the nonlinear coupling terms in the equations of motion of the resonator. For instance, considering two modes with natural frequencies ω_1 and ω_2 , When the resonator is excited near ω_1 , the nonlinear coupling can induce a response at ω_2 , effectively transferring energy between the modes. This phenomenon is often observed as frequency shifts and changes in the amplitude response in the frequency spectrum of the resonator.

Internal resonance (IR) is a specific type of nonlinear interaction where the natural frequencies of two or more modes are commensurate, meaning they are integer multiples of each other (e.g.,

$\omega_2=2\omega_1$). This condition leads to a strong coupling between the modes, resulting in significant energy exchange and complex dynamic behavior.²²

To understand and predict internal resonance in NEMS resonators, theoretical modeling is essential. The nonlinear dynamics of the system can be described using coupled differential equations that incorporate nonlinear stiffness and damping terms.²³ These equations incorporate nonlinear stiffness and damping terms that account for the energy exchange between the modes. To model 1:2 IR, we consider two coupled modes with natural frequencies ω_1 and $\omega_2=2\omega_1$. The motion equations of mode x and y can be written as:

$$\ddot{x} + \frac{\Gamma_1}{m_1} \dot{x} + \frac{k_1}{m_1} x + \frac{a_1}{m_1} x^3 + \frac{g_c}{m_1} xy = \frac{F_0}{m_1} \cos \omega t \quad 4-11$$

$$\ddot{y} + \frac{\Gamma_2}{m_1} \dot{y} + \frac{k_2}{m_1} y + \frac{a_2}{m_1} y^3 + \frac{0.5 * g_c}{m_1} x^2 = 0 \quad 4-12$$

Where x , y are displacements of the two modes, Γ_1 and Γ_2 are damping coefficients of the two modes, k_1 , k_2 , a_1 , a_2 are linear and nonlinear stiffness coefficients of the two modes, g_c is the coupling coefficient.

Studying IR allows for the precise control of dynamic responses, which is crucial in applications such as sensing, signal processing, and energy harvesting. Internal resonance can be used to achieve higher responsivity in sensors, improve the efficiency of energy harvesters, and enable advanced signal processing techniques by exploiting the strong mode coupling and energy transfer. In conventional MEMS resonators, achieving internal resonance typically requires the design of special structures or applying external load to ensure the commensurability of mode frequencies. This often involves intricate fabrication techniques and careful parameter selection to realize the desired resonant conditions.²⁴

For the ultrathin Si NEMS resonators developed in this work, both the nonlinearity and the resonance frequencies can be widely tuned by controlling DC gate voltage V_{dc} . Such tunability simplifies the process of achieving IR and enhances the flexibility and functionality of the resonators, since the activation of IR is mainly depended on the resonator's resonance frequency and nonlinearity based on the differential equations of (4-11) and (4-12).

4.3.2 Experimental observations of internal resonance

Experimental evidence of internal resonance (IR) activation typically includes key observations. At lower driving voltages, the peak frequency increases with driving strength as expected for a duffing oscillator, but at higher driving voltages, the peak (drop) frequency remains constant, this saturation of amplitude is one evidence of energy being effectively transferred from the driven mode to the undriven mode due to IR. When driving voltages are further increased, a sudden jump in the amplitude of the driven mode will be observed at certain drive frequency, in which the amplitude jumps from a lower value to a significantly higher value. This jump occurs when the system surpasses a critical energy threshold, triggering the internal resonance, which is another evidence indicating the onset of IR.

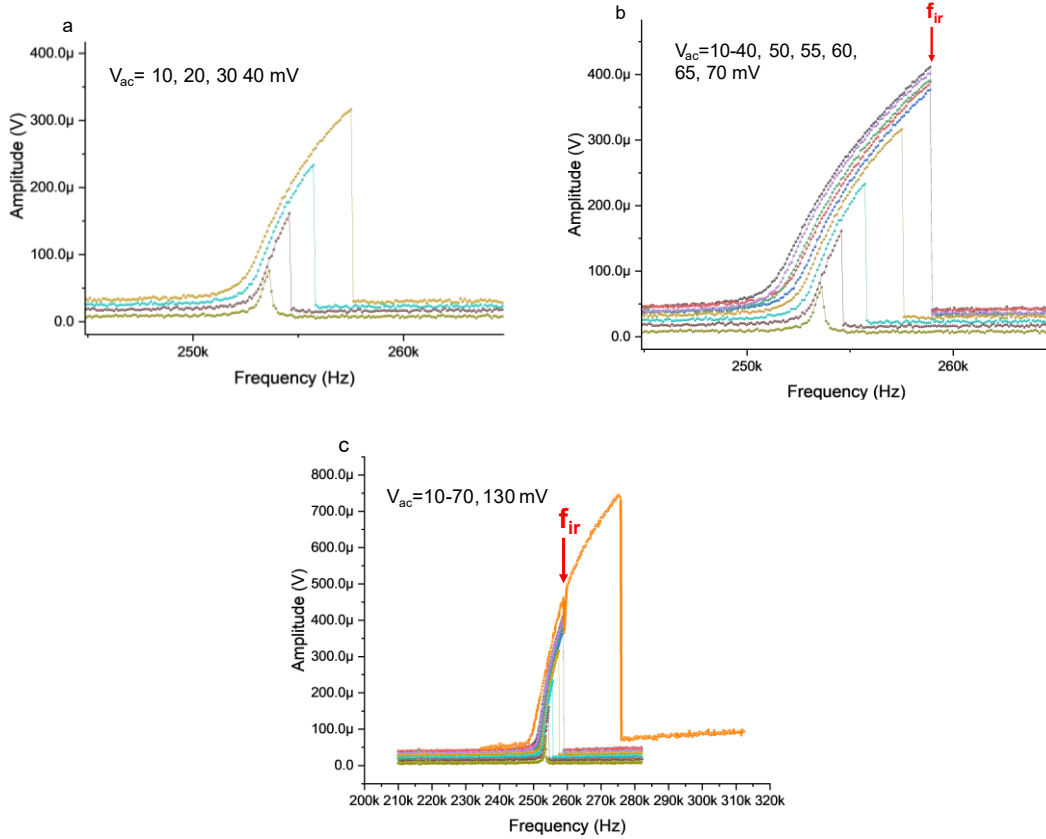


Figure 4-7 The observation of IR activation with increasing driving voltage V_{ac} in $t \sim 150$ nm NEMS resonator device ($L \sim 200 \mu\text{m}$, $w \sim 7.5 \mu\text{m}$, $g_0 \sim 2 \mu\text{m}$) at $V_{dc} = 15$ V as V_{ac} is varied. a, the IR is not activated at low V_{ac} (10 - 40 mV). b, the IR is activated when V_{ac} increases to 50 mV, evidenced by the locking of the drop frequency (f_{ir}) and saturation of the peak amplitude. c, when V_{ac} is further increased to 130 mV, the drop frequency is unlocked and replaced by a sharp dip at frequency f_{ir} where the internal resonance activated.

Through controlling the gate voltage V_{dc} applied, the 1:2 IR can be easily activated in our fabricated ultrathin Si NEMS resonator devices. As depicted in Fig. 4-7, the observation of IR activation process is shown in one of our devices. Initially, at low V_{ac} levels (10 - 40 mV), IR is not activated, and the device exhibits typical nonlinear amplitude response without coupling, as shown in Figure 4-7a. When V_{ac} increases to 50 mV (Figure 4-7b), IR is activated, evidenced by the locking of the drop frequency (f_{ir}) and the saturation of the peak amplitude, indicating strong intermodal energy transfer and stabilization of the oscillation frequency. As V_{ac} is further increased to 130 mV (Figure 4-7c), the drop frequency unlocks and is replaced by a sharp dip at f_{ir} , where the internal resonance is fully activated. This sharp dip signifies significant energy transfer between modes, validating the IR activation. The collective phenomena, including the initial absence of IR, the subsequent locking and saturation of the drop frequency, and the final sharp dip observed at higher V_{ac} , demonstrate the IR activation has been successfully observed in our devices with conventional double clamped structures. The ability to activate IR without needing special structural designs, showcases the versatility and practicality of our ultrathin Si NEMS resonators, making them suitable for a wide range of high-performance applications, such as sensing in nonlinear regime, nonlinear signal processing and enhancing energy harvesting efficiency.

4.4 Discussion and conclusion

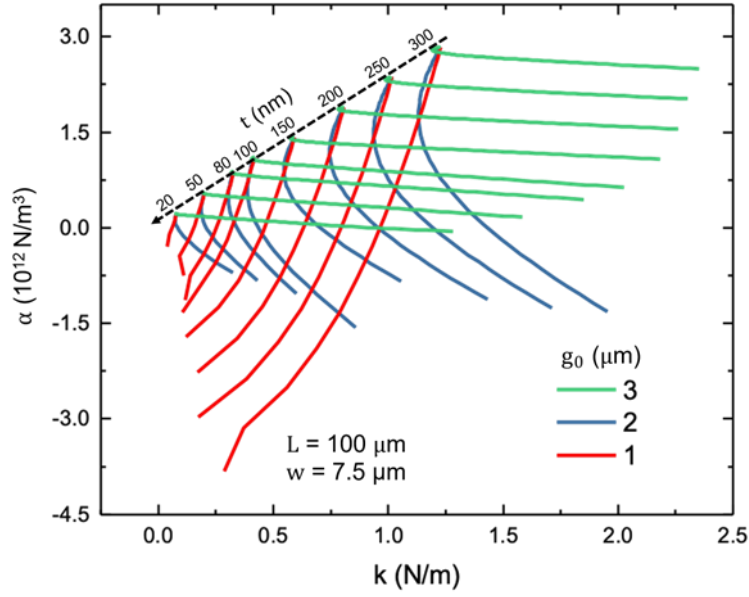


Figure 4-8 Variation of $k(V_{dc})$ and $\alpha(V_{dc})$ for devices with different dimensions. V_{dc} starts at 0.1 V at the top of the plot and ends either at 100 V or when the nanoresonator crashes into the actuation electrode, whichever happens earlier. Note: $\frac{dk(V_{dc})}{dV_{dc}}$ and $\frac{d\alpha(V_{dc})}{dV_{dc}}$ varies nonlinearly with V_{dc} , thus rates of changes can be very slow in the lower V_{dc} and much faster at higher V_{dc} . Also, actual starting point in measurement will effectively start at a V_{dc} value where the detection signal reaches the minimum detection limit of the measurement system, so a significant portion of the initial part of the graph may not be accessible in applications, depending on the measurement systems.

Finally, it should be noted that the frequency and nonlinearity tuning are tied via the same tuning parameter (V_{dc}), i.e., the frequency and the nonlinearity cannot be tuned independently in the same device. Therefore, to target specific goals in terms of frequency and nonlinearity tuning, device dimensions should be carefully considered at the outset. To explore this point, we numerically plotted the variation of the linear ($k(V_{dc})$) and nonlinear ($\alpha(V_{dc})$) stiffnesses with V_{dc} in the (k, α) plane for various device parameters in Fig. 4-8. V_{dc} starts at 0.1 V at the top of the plot and ends either at 100 V or when the nanoresonator crashes into the actuation electrode, whichever happens earlier. We observe that specific (k, α) values within limitation can be attained by tuning, for suitable device parameters. Also, the intersection points in the plot are useful, because they indicate that similar combinations of (k, α) can be achieved by different combinations of design parameters (at

different V_{dc}); each combination allowing different tuning trend (rate of change) for k and α . It is however challenging to decouple k and α tuning in the same device. We speculate whether by introducing additional electromechanical degrees of freedom to the device, such as, (a) via the handle layer (to apply an additional out of plane electrostatic force), (b) by splitting the actuation gate and placing the parts at different g_0 , (c) introducing the ability to vary the g_0 by connecting the side-gate to an additional on-chip electrostatic/ thermal actuator, etc. can be useful in this regard. However, further theoretical investigations are necessary to substantiate the effectiveness of these ideas.

Overall, we explored the nonlinear behaviors of ultrathin Si NEMS resonators in this chapter, focusing on their dynamic responses under various driving conditions. We demonstrated the ability to tune nonlinearity by adjusting the DC gate voltage, allowing precise control over the device's dynamic range. Besides, our experimental observations showed the activation of internal resonance (IR) in our devices without requiring special structural designs. The ease of achieving and controlling IR encourages further research and innovation in application of Si NEMS resonator device in nonlinear region.

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Chapter 5 Sensing Application of Ultrathin Silicon NEMS Resonator

5.1 Introduction

NEMS resonators have gained significant attention for their potential applications in various sensing technologies due to their high responsivity, small size, and tunable properties. These resonators operate on the principle of detecting shifts in resonance frequency or amplitude in response to external stimuli, making them ideal for applications in gas sensing, temperature sensing, biological detection, and more. Two critical metrics commonly used to evaluate the resonator's mass sensing performance are responsivity ($\mathcal{R}$) and detection resolution/limit (δM).¹ The former (rate of frequency shift with adsorbed mass) refers to the sensor's ability to detect minute variations of mass, while the latter represents the smallest detectable mass change. The lower the δM , the smaller the mass changes that can be reliably detected. Assuming the added mass is evenly distributed upon the resonators, the expression of $\mathcal{R}$ and δM are given as follows ²:

$$\mathcal{R} = -\frac{\omega_0}{2M_{\text{eff}}} \quad 5-1$$

$$\delta M \approx \left(\frac{M_{\text{eff}}}{Q}\right) \cdot 10^{-DR/20} \quad 5-2$$

where, ω_0 , M_{eff} represent the resonance frequency and effective mass of the resonator, respectively, and Q is the quality factor of the resonator. The linear dynamic range (DR) indicates the range over which the device can respond linearly to input signals. This range is constrained by resonators' nonlinearity (α) and the measurement noise floor.³ Considering the thermomechanical noise as the intrinsic noise floor and using the expression of DR given by Molina et al.,⁴ Equation (5-4) can be rewritten as a theoretical limit for mass-detection,

$$\delta M \approx 3.06 \sqrt{\frac{k_B T \Delta f |\alpha|}{\omega_0^5}} \quad 5-3$$

Here, k_B is the Boltzmann constant, Δf , T are the measurement bandwidth³ and temperature, respectively.

According to equation (5-1) and equation (5-2), it is apparent that reducing the resonator's mass

is an effective way to improve responsivity and reach a smaller mass detection limit. For instance, Lassagne et al.⁵ enhance a carbon nanotube's responsivity to 11 Hz/yg by downsizing its diameter to 1 nm. Also, equation (5-1) and equation (5-3) suggest that achieving higher resonance frequencies can be potentially beneficial to enhance the responsivity and detection limit of sensing. This can be accomplished either through higher-order resonance modes⁶ or by selecting different resonator materials.⁷ In this regard, modifying the resonator's material or structure has also been adopted to enhance sensing performance. This includes strategies like adjusting the anchor structure for quasi-one-dimensional oscillation with high-quality factors,⁸ heating buckled micro-beam resonators for highly selective detection,⁹ expanding analyte adsorption area using porous nanostructures,¹⁰ and surface functionalization with specific sensing materials,¹¹ *etc.* These approaches primarily focus on the pre-application stages; once a device is fabricated, opportunities for further improvement or control over responsivity and detection limit are limited. This highlights the need for a method that can enhance sensing performance and enables control over responsivity and detection limit during the operational phase. Tunable micro-/nanomechanical resonators,¹² which allow for the adjustment of resonance frequency and nonlinearity during operation of resonators,^{13,14,15,16} offer a promising opportunity to improve sensing performance in post-fabrication stage. In terms of nonlinearity tuning, the cubic nonlinearity of a bilayer MoS₂ resonator is shown to be manipulated between hardening and softening types, and even nullified by changing gate voltage.¹⁷ Such wide tunability in frequency and nonlinearity offer potential advantages for not only enhancing mass responsivity but also tuning detection resolution, which can be beneficial for realizing a wide linear-range-detection of different target masses on the same device.¹⁸

Developed ultrathin silicon (Si) NEMS resonators have also shown great promise in gas/mass sensing applications due to their low mass and high surface-to-volume ratio, which enhances their responsivity and interaction with gas molecules. The following sections will introduce the gas sensing mechanisms and sensing estimation techniques at first, and then present specific results related to CO₂ gas detection, and explore the mass sensing performance enhancement through gate tuning at last.

5.2 Ultrathin Si NEMS resonator applied as gas sensor

5.2.1 Gas sensing mechanism and estimation

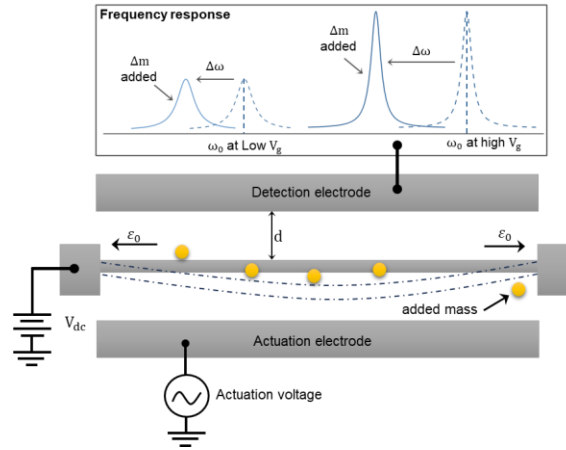


Figure 5-1 Schematic diagram of a resonator mass sensor with electrostatic actuation and capacitive read-out. The improvement of sensing performance by tuning gate voltage is also indicated in this plot.

The gas sensing mechanism of ultrathin Si NEMS resonators relies on the adsorption of gas molecules onto the surface of the resonator, which induces a change in the mass and surface properties of the resonator. This mass change leads to a shift in the resonance frequency, which can be measured to estimate the concentration of the gas.¹⁹ As an example, Figure 5-1 shows a schematic diagram of a resonator mass sensor employing electrostatic actuation and capacitive read-out. A periodic actuation force, generated when an AC voltage is applied to the actuation electrode along with a DC gate voltage (V_{dc}) applied between the resonator and electrode, induces the resonator to vibrate. The capacitance fluctuation due to the oscillation of the resonator leads to a current signal that can be optionally detected using a lock-in amplifier after being converted to a voltage signal by the transimpedance amplifier.²⁰ The initially detected resonance frequency serves as a baseline, which shifts to a lower frequency when target masses attach to the resonator. Mass sensing is realized by evaluating this frequency shift relative to the baseline.

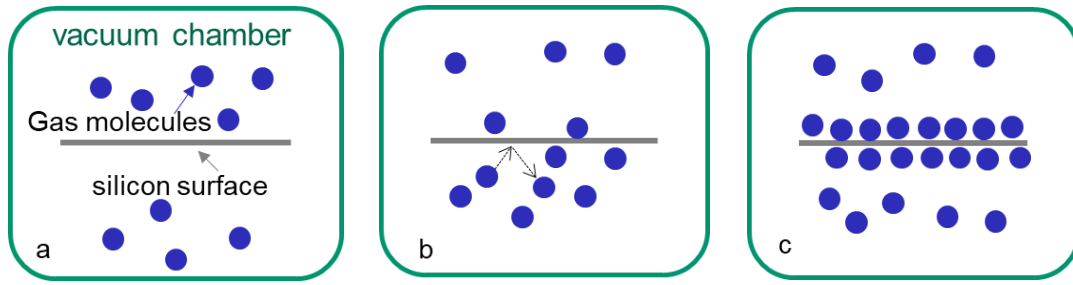


Figure 5-2 The process of gas molecules adsorption on Si resonator surface. a, Gas is introduced into the chamber and gas molecules begin to diffuse in the chamber. b, When molecules contact the Si surface, they may adhere (physisorption or chemisorption) depending on the surface characteristics and molecular energy dynamics. c, Over time, the surface reaches a saturation point where no additional adsorption occurs, an equilibrium is reached where the rate of adsorption equals the rate of desorption

Figure 5-2 states the process about how the gas molecules interact with and adsorb on the Si resonator surface. Initially, the Si resonator's surface is exposed to a vacuum environment within the chamber, ensuring a clean baseline for gas adsorption experiments. Upon introducing gas into the chamber, molecules diffuse and interact with the Si surface through physisorption, involving weak van der Waals forces, or chemisorption, forming stronger chemical bonds. The likelihood of a molecule adhering to the surface, known as the sticking probability (S), varies based on surface treatments and the intrinsic properties of both the Si and the gas molecules. Since we didn't apply any surface treatment or coat any sensitive layer on our device, the S should be lower than 0.001 during the estimation. The impingement rate (J), defined as the flux of molecules arriving at the surface per unit area per unit time, can be calculated using the formula,

$$J = P \sqrt{\frac{2\pi m}{k_B T}} \quad 5-4$$

where P is the gas pressure, m is the molecular mass, k_B is the Boltzmann constant, and T is the absolute temperature. As molecules adhere to the Si surface, adsorption reaches an equilibrium where the rates of adsorption and desorption are equal, saturating the surface. The total number of adsorbed molecules (N) is determined by the product of the sticking probability, impingement rate, surface area, and exposure time. This process results in a mass change (Δm) of the resonator, calculated as

$$\Delta m = S \cdot N \cdot m_{\text{gas}}$$

5-5

where m_{gas} is the mass of a single gas molecule. The additional mass shifts the resonator's frequency, a key parameter for detecting and quantifying gas presence. Thus, comprehensively understanding gas adsorption mechanisms and modeling these interactions is crucial for estimating and optimizing the Si NEMS gas sensors' performance.

5.2.2 Experimental results of CO₂ gas detection

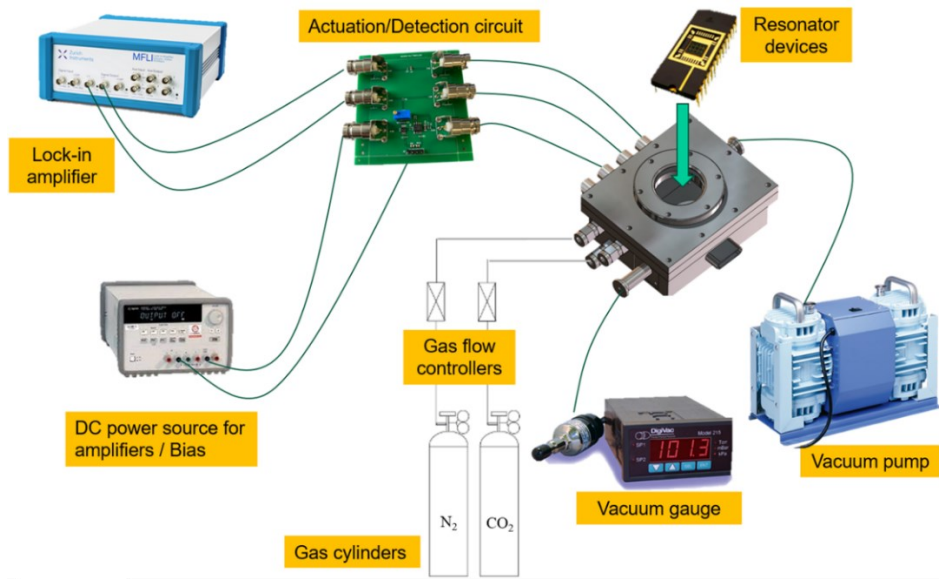


Figure 5-3 The experimental gas sensing test platform for ultrathin Si NEMS resonator devices

To experimentally test the gas sensing performance of fabricated ultrathin Si NEMS resonator devices, a test platform has been developed as shown in Fig. 5-4. The platform consists of a customized vacuum chamber equipped with ports for introducing gases and electrical connections to the resonator. The resonator device is mounted inside the chamber. The setup includes two gas cylinders of CO₂ as detected gas and N₂ as background gas; a vacuum pump to evacuate the chamber to a low pressure, typically around 10 Pa, ensuring an initial clean environment for the tests. The experimental procedure begins with detecting the initial resonance frequency of the resonator under vacuum conditions. Following this baseline measurement, gas flow controllers introduce the target gas into the chamber at a controlled rate, allowing precise regulation of gas concentration and exposure time. As gas molecules adsorb onto the resonator's surface, the resulting mass change leads to a shift in the resonance frequency, which is continuously processed and monitored through a

designed detection circuit board and a lock-in amplifier.

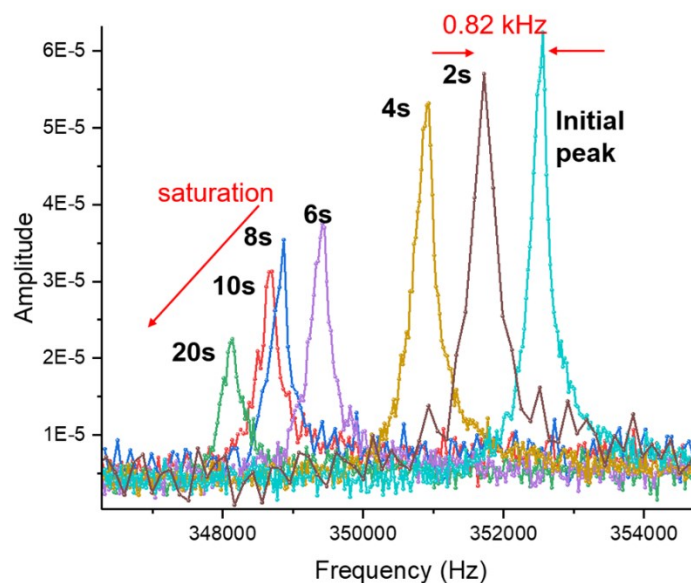


Figure 5-4 The detected resonance frequency shift of a Si NEMS resonator device ($w \sim 100$ nm, $L \sim 200$ μ m) during the introduction of CO_2 gas as pulses of 2 seconds each at a rate of 0.1 L/min.

Fig. 5-4 shows the CO_2 gas sensing experimental results conducted on a Si NEMS resonator device with width of 100 nm and length of 200 μ m. In the beginning of experiment, the initial resonance peak around frequency around 352.56 kHz was detected without introducing CO_2 gas under vacuum environment. Then CO_2 gas was introduced into the vacuum chamber in a controlled manner, with pulses of 2 seconds each at a rate of 0.1 L/min. The change in the resonator's mass, as calculated from the frequency shift of 0.82 kHz, was approximately 1.59 pg for first 2-second pulse of CO_2 introduction. As more CO_2 gas was introduced, the quality factor of the resonator decreased, indicating increased damping due to gas adsorption and chamber pressure increases.

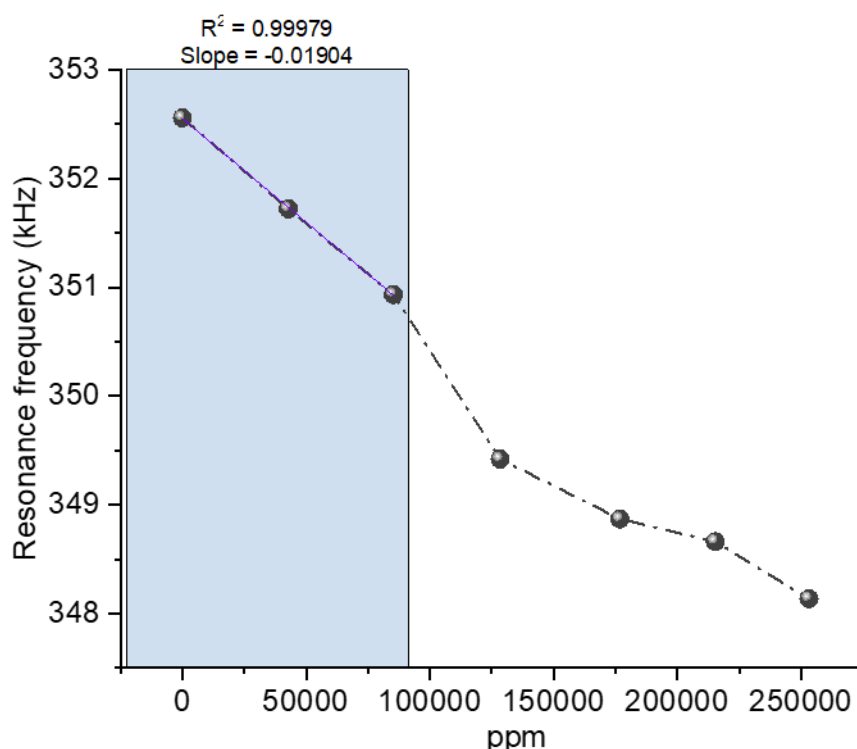


Figure 5-5 The detected resonance frequency with change of CO₂ concentration in ppm. This graph illustrates the linear relationship between the resonance frequency shift and the concentration of CO₂ in ppm. The frequency shift data was obtained by introducing CO₂ gas in controlled pulses at a rate of 0.1 L/min with 2 s. The responsivity of the ultrathin Si nanoresonator can be approximately estimated as the slope of the linear part in the plot, which is around 0.019 Hz/ppm

The detected resonance frequencies change with the varied CO₂ concentration is shown in Fig.5-6. The gas sensing responsivity measured to be approximately 0.019 Hz/ppm based on the linear part in the plot. Table 5-1 compares this responsivity with other resonator-based CO₂ gas sensing results reported in the literature. The comparative analysis revealed that the responsivity of the fabricated Si NEMS resonator device, without any surface coating, is close to or even exceeds that of MEMS resonators that utilize sensitive layer coatings. For instance, the responsivity of the MEMS cantilever resonator coated with tetrahydroxy-eethylenediamine (THEED) was found to be 0.016 Hz/ppm, while another MEMS cantilever coated with linear polyethylenimine achieved a responsivity of 0.0035 Hz/ppm. Compared to these, the uncoated NEMS resonator device here achieved a responsivity of ~0.019 Hz/ppm. This comparative analysis shows the advantages of using proposed ultrathin Si NEMS resonators in gas sensing, offering high responsivity and eliminating the need for complex surface coatings. The high responsivity and simplicity of the ultrathin Si nanoresonator make it a promising candidate for various gas sensing applications, potentially

improving the performance and reducing the cost and complexity of gas sensor devices. Furthermore, it is anticipated that by incorporating sensitive coatings or surface treatments, the responsivity of proposed NEMS resonator-based gas sensor could be further enhanced, realizing ultrasensitive performance in nano-scale devices.

Table 5-1 Comparative resonator-based CO₂ gas responsivity analysis

Gas type	Device type	Dimension (length, width, height)	Responsivity	Coating or surface treatment	Ref
CO ₂	Ultrathin Si NEMS resonator	200 μm , 100 nm, 7.5 μm	~ 0.019 Hz/ppm	No	This work
	MEMS resonator	1000 μm , 324 μm , 45 μm	0.0318 Hz/ppm	coated with Polyallylamine (PEO)	[21]
	MEMS resonator	Not given	0.12 Hz/ppm	coated with poly(ethylene oxide) (PEO) and poly(ethyleneimine) (PEI)	[22]
	MEMS resonator	220 μm , 22 μm , 40 μm	0.0035 Hz / ppm	coated with Linear Polyethylenimine	[23]
	MEMS resonator	1000 μm , 300 μm , 45 μm	0.016 Hz / ppm	coated with sensitive polymer	[24]

5.3 The application of tunable NEMS resonator on enhancing sensing performance

5.3.1 Sensing performance enhancement mechanism

The prospective improvement of mass-sensing performance by taking advantage of tunable resonators has been suggested in chapter 3. To substantiate and enrich this perspective, we theoretically explore the potential of utilizing the wide tunability offered by the ultrathin nanomechanical resonators for enhancing performance in mass-sensing applications in this section. Specifically, the improvement model of responsivity and detection limit is developed, and then the tuning of resonator's dynamic behaviour and its effect on resonator's sensing performance are numerically estimated. We aim to investigate an improvement strategy for responsivity and detection resolution by utilizing the wide tunability in resonance frequency and nonlinearity during

the application stage. Furthermore, the factors influencing the improvement in these parameters are also analyzed for optimal utilization of this strategy.

In previous chapter, the effective tuning of ω_0 and α in Si NEMS resonators can be achieved through electrostatic means, where altering the gate voltage modifies the axial tension in the resonator by controlling its deflection. This tuning mechanism is especially pronounced in atomically-thin resonators, such as graphene,²⁵ MoS₂,²⁶ and carbon nanotube (CNT)²⁷ resonators, whose dynamic behavior is dominated by built-in tension due to their extreme aspect ratio,²⁸ making them highly responsive to change in V_g . To substantiate our proposed sensing performance enhancement strategy from an universally applicable perspective, the following analysis is based on a conventional MoS₂ resonator.

Following the tuning strategies outlined in ref ²⁹ and ref ³⁰ the electromechanical potential model is utilized to explain the electrostatic tuning behavior of ω_0 and α . The equations for mechanical energy and electrostatic energy of the resonator under electrostatic force are first established. The resonator's static displacement to a new equilibrium position under the influence of the electrostatic force is then determined by finding the local minima of the total energy. Taking the second order differentiation of the total potential energy at the equilibrium position, the approximated expression of linear stiffness $k(V_g)$ can be obtained. In ref. [29], Lee et al. developed the $k(V_g)$ tuning model and demonstrated electrical tunability and broad linear dynamic range in MoS₂ circular drumhead resonators. Here, we would like to derive an approximate nonlinearity tuning model $\alpha(V_g)$ based on their work and theoretically explore the enhancement of $\mathcal{R}$ and δM by tuning $k(V_g)$ and $\alpha(V_g)$. The dependence of k on V_g can be approximated as,

$$k(V_g) \approx \frac{\pi\epsilon_0^2}{8(1-\nu^2)E_Y t \epsilon_0^2 d^4} r^2 V_g^4 + 4.9E_Y t \epsilon_0 - \frac{\epsilon_0 \pi r^2}{3d^3} V_g^2 \quad 5-6$$

where ϵ_0 is vacuum permittivity, r is the resonator's radius, ϵ_0 is initial strain, ν is Poisson's ratio, E_Y is Young's modulus, t is the thickness of the resonator, and d is the vacuum gap distance between the resonator and electrode. Consequently, ω_0 can be expressed as

$$\omega_0(V_g) \approx \sqrt{\frac{\frac{\pi\epsilon_0^2}{8(1-v^2)E_Y t \epsilon_0^2} \frac{r^2}{d^4} V_g^4 + 4.9E_Y t \epsilon_0 - \frac{\epsilon_0 \pi r^2}{3d^3} V_g^2}{M_{\text{eff}}}} \quad 5-7$$

Similarly, the approximate cubic nonlinear stiffness, $\alpha(V_g)$ is the fourth order differentiation of the total potential energy at equilibrium, which is

$$\alpha(V_g) \approx \frac{8\pi E_Y t}{(1-v^2)r^2} - \frac{12\epsilon_0 \pi r^2}{5d^5} V_g^2 \quad 5-8$$

5.3.2 Numerical estimation of tunability in resonance properties

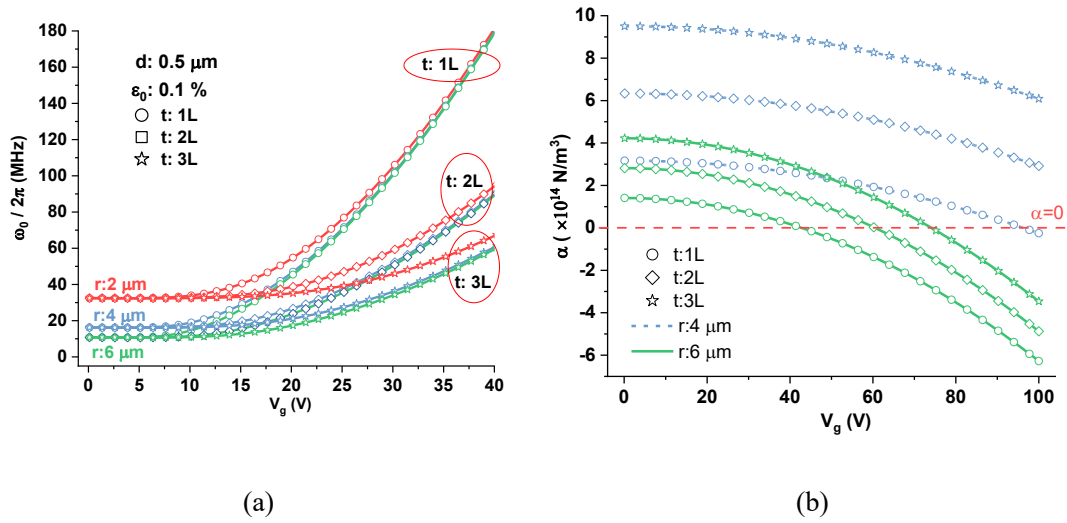


Figure 5-6 A theoretical plot of Electrostatic tuning of (a) resonance frequency and (b) cubic nonlinearity with varying the radius r (2 μm , 4 μm , 6 μm) and layers L (thickness t of 1L, 2L, 3L are 0.7 nm, 1.4 nm, 2.1 nm). The vacuum gap distance ($d = 0.5 \mu\text{m}$) and initial strain (0.1%), values are adopted from ref ²⁹.

It is worth mentioning that the proposed sensing performance improving strategy is not limited to any specific device, since the wide tunability of $k(V_g)$ and $\alpha(V_g)$ have been reported in various resonator devices.^{19,24,26} Since the developed $k(V_g)$ and $\alpha(V_g)$ tuning models are based on the MoS₂ drumhead resonator presented by Lee et al.²⁹, we would like to take such circular drumhead structure as a case study. Based on equation (5-7) and (5-8), the electrostatic tuning of ω_0 and α with varying dimensions are estimated in Fig. 5-7(a) and Fig. 5-7(b), respectively. The initial ω_0 without tuning ($V_g = 0$) is primarily affected by r for a given initial strain, while with the increase of V_g , the ω_0 converges across various t (different layer numbers). This suggests that larger r

values and lower t values result in more efficient ω_0 tuning. The initial value and tuning efficiency of ω_0 are also significantly affected by ε_0 , which is set as 0.1% in this study. Generally, a higher ε_0 corresponds to a larger initial ω_0 but lower tuning efficiency.

Concerning the tuning of nonlinearity, as shown in Fig. 5-7(b), both the initial value and the tuning range of α are mainly determined by resonator's dimensions. A larger r facilitates higher tuning efficiency, while a lower t can potentially yield lower α values when r is fixed. Notably, α consistently decreases with increasing V_g , approaching zero at certain point. This indicates a wider linear DR and the potential for achieving a smaller δM around this point.

5.3.3 The improvement of sensing performance through electrostatic tuning

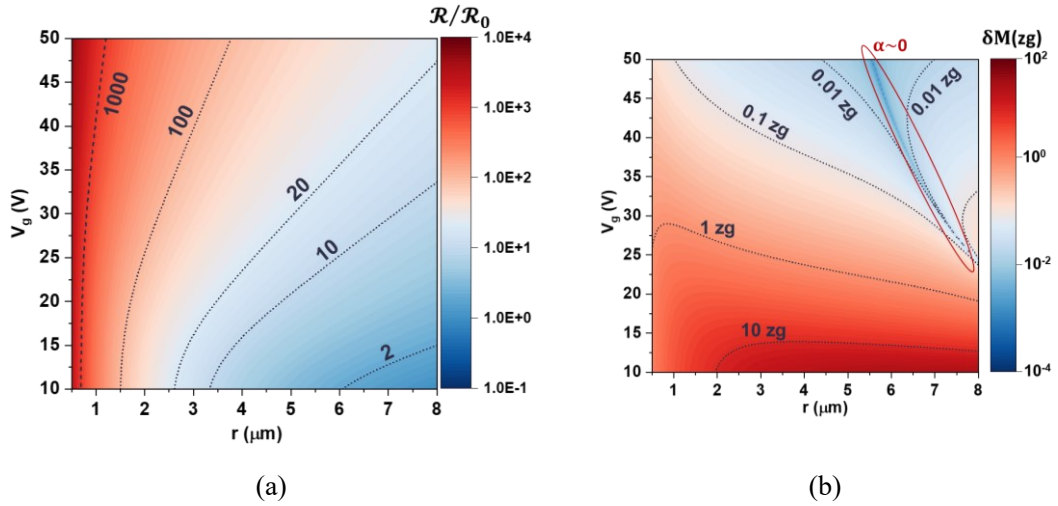


Figure 5-7 (a) The responsivity improvement ratio $\mathcal{R}/\mathcal{R}_0$ ($\mathcal{R}_0$ denotes the lowest value within the given dimension and voltage range) changes with r and V_g . (b) δM changes with different r and V_g . A red circle highlights a dark blue area in the colormap, indicating the case of α close to 0. All the results are estimated with $t \sim 0.7$ nm (monolayer MoS_2), $T \sim 300$ K, $\Delta f \sim 1$ Hz.

The previous analysis reveals that higher ω_0 , lower α and M_{eff} are benefited by a lower t . Based on this, we assume the lowest feasible t (0.7nm) for subsequent analyses of $\mathcal{R}$ and δM . As suggested in Fig. 5-8(a), significant improvements in $\mathcal{R}$ are attainable through both electrostatic tuning and downsizing the resonator. The lowest $\mathcal{R}$ is observed at the minimum V_g and maximum r , with $\mathcal{R}$ enhancing as r decreases and V_g increases. However, this improvement is less pronounced in resonators with smaller r . For instance, an increase in V_g from 10 V to 50 V improves the $\mathcal{R}/\mathcal{R}_0$ by 22.2 times for an 8 μm resonator, but only 2.3 times for a 1 μm resonator.

However, the relationship between r , V_g and δM , as shown in Fig. 5-8 (b), is more complex. Here, δM initially decreases and then increases with V_g for a given r . This result is attributed to α approaching zero in some cases, leading to extremely low δM values (indicated by the dark blue part in this plot), and then shifting to negative values of α , which increase δM when V_g is further increased. It can be observed that larger r makes it easier to achieve extremely low δM values and a wider tuning range of δM . Since tunable δM is valuable for realizing selective detection resolution, it is meaningful to consider using relatively larger r resonators for sensing applications where $\mathcal{R}$ already satisfies the application requirements.

The efficacy of tunable nanomechanical resonators in enhancing mass sensors' sensing performance could be beneficial to numerous applications. For instance, realizing selective detection of trace pollutants or hazardous substances with varying masses in the field of environmental monitoring,³¹ enabling early detection of disease biomarkers in medical diagnostics³² and ensuring precise measurement of drug compounds.³³ Although electrostatic tuning and temperature variation-induced tuning are capable of improving or controlling $\mathcal{R}$ and δM to realize the aforementioned benefits, they present certain challenges in practical applications. In the electrostatic tuning method, it is crucial to keep the applied voltage V_g within a safe range to ensure the maximum stress of the resonator should not exceed the critical value that results in a catastrophic failure of the nanoresonator device. For monolayer MoS₂, the mechanical fracture usually occurs at the average mechanical fracture strength value around 23 GPa.³⁴ Also, when the displacement of the MoS₂ covers the electrode gap would indicate the end of the tuning range (and in a scenario when the MoS₂ membrane makes contact with the gate electrode is likely to damage the device).

5.4 Conclusion

In this chapter, we explored the application of ultrathin Si NEMS resonators for gas sensing, focusing on their mechanisms, performance characteristics, and potential enhancements through tuning strategies. Our studies have demonstrated that these resonators, due to their low mass and high surface-to-volume ratio, exhibit significant responsivity to gas adsorption, making them highly effective for detecting gas such as CO₂.

The experimental results showed that the ultrathin Si NEMS resonators could achieve a responsivity of approximately 0.019 Hz/ppm for CO₂, even without any surface coating or sensitive layer. This performance is competitive with many MEMS resonator-based gas sensors that utilize sensitive coatings. This underscores the inherent advantages of using ultrathin Si NEMS resonators in gas sensing applications, offering high responsivity, simplicity in design, and potentially lower costs.

Moreover, we investigated the theoretical models and practical implementation of enhancing mass sensing performance through gate voltage tuning. The ability to adjust the resonance frequency and nonlinearity dynamically offers a promising approach to improve sensor responsivity and detection resolution during the operational phase. By incorporating sensitive coatings or surface treatments, the responsivity of these resonators could be further enhanced, potentially realizing ultra-sensitive performance in nano-scale devices. Future work will focus on integrating these enhancement techniques and exploring additional surface modifications to further boost sensor performance and expand their applicability in various industrial and environmental monitoring applications

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Chapter 6 Conclusion

6.1 Summary of accomplishments

This work aims to overcome the fabrication challenges and demonstrate the ultrawide electrostatic tuning of both resonance frequency and nonlinear behaviour in monolithically fabricated ultrathin Silicon (Si) NEMS resonator devices. By deeply exploring their dynamic performance, we examine their ability to operate as sensors and investigate methods to enhance sensing performance through the ultrawide tunability of resonance frequency and nonlinearity. This study is expected to bring remarkable operational flexibility and new capabilities to this extraordinary tool influencing many technological areas. The main accomplishments and conclusions of this thesis are listed as follows:

(1) Fabrication and Characterization of Ultrathin Silicon NEMS Resonators

By carefully designing the dimensions and employing a scalable microfabrication process involving advanced electron-beam-lithography and optimized deep reactive-ion etching, ultrathin Si NEMS resonators (width ranges from 20 nm to 300 nm, length ranges from 100 μm to 300 μm , and height is 7.5 μm) are monolithically fabricated. The largest aspect ratio can reach around 15000. The characterization of fabricated NEMS resonators realized by a customized detection circuit and a digital lock-in amplifier highlighted their exceptional mechanical properties, including high resonance frequencies and stress-dominated dynamic behaviour. By gradually decreasing our device's width from 300 nm to 20 nm, we demonstrate a gradual transition from beamlike to string-like dynamic behaviour indicated by increased stress-dominated resonance frequency with thinning of width.

(2) Ultrawide Electrostatic Tunability of Resonance Frequency

We experimentally demonstrate and theoretically investigate the ultrawide resonance frequency tunability in the developed ultrathin Si NEMS resonators. By applying DC gate voltages between the actuation electrode and nanoresonator structure, ultrawide electrostatic tuning ($\sim 70\%$) of (initial) resonance-frequency ($\sim 7\% \text{ V}^{-1}$) at room temperature is observed in a fabricated ultrathin Si NEMS (width $\sim 40 \text{ nm}$, length ~ 200

μm) resonator device. Such wide tunability is comparable to atomically thin resonators, such as graphene and MoS_2 -based resonators. An approximate electromechanical model is established to explain the tuning mechanism and explore the tuning behaviours under various conditions. Furthermore, the temperature-induced resonance frequency tuning and temperature dependence of electrostatic tunability are investigated by varying the device's substrate temperature.

(3) Nonlinear Behaviour Investigation and Tuning

According to the experimental resonance response results of fabricated Si NEMS resonators under the nonlinear regime, the transition from linear to nonlinear regimes is identified as increasing driving voltage. By manipulating the DC gate voltage, the nonlinear coefficients can be effectively tuned from softening type to hardening type. Even the cancellation of nonlinearity can be approximately reached when flipping the sign of nonlinearity. The linear dynamic range of the device can be broadened around this cancellation point, reaching around 86 dB based on the estimation of one device with a width of 100 nm and a length of 100 nm. The study also demonstrated the activation of internal resonance without requiring special structural designs, showing the versatility of Si NEMS resonators in nonlinear regime applications, including advanced signal processing and energy harvesting.

(4) Sensing Application and Performance Enhancement

The high surface-to-volume ratio and low mass of the Si NEMS resonators significantly enhanced their responsivity to gas molecules, demonstrated by the experimental detection of CO_2 with high responsivity. The study also explored methods to improve sensing performance through electrostatic tuning, allowing real-time adjustments to the resonator's responsivity and detection limit. This work highlights the potential for ultrathin Si NEMS resonators to be beneficial to various sensing applications, offering high responsivity, operational flexibility, and potential for integration into various industrial and environmental monitoring systems.

6.2 Future work

Achieving ultrawide tunability in microfabricated Si NEMS resonator devices holds significant potential for the rapid commercialization of ultrathin, tunable NEMS resonators across various applications. These applications include high-precision sensing, signal processing, and energy harvesting. The integration of such highly tunable devices into existing technologies can lead to significant advancements in performance and functionality, particularly in fields requiring adaptive and sensitive detection mechanisms.

Future research will focus on key areas to further enhance the tunability and responsivity of these NEMS resonators and explore their integration into complex systems for real-world applications. In terms of device design and fabrication, we believe wider tuning of resonance frequency can be realized by optimizing the geometry and combining other tuning methods like applying an external force to actively adjust the axial stress. The coupling of frequency and nonlinearity tuning have been discussed at the end of chapter 4. An effective decoupling method will be beneficial for precisely controlling the ideal resonant properties.

Concerning the gas sensing application, there is no surface treatment or sensitive layer coating applied on the Si NEMS resonator in this study. To further improve the responsivity and specificity of gas sensing, future research will explore the integration of surface coatings and functionalizations. These modifications target specific gas molecules, enhancing selectivity and responsivity. Besides, the development of multiparametric sensing capabilities will be investigated. Integrating multiple sensing mechanisms (e.g., temperature, pressure, humidity) into a single device can create more versatile and robust sensors for various applications.

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List of Publications

Journal Paper

- [1] Wei Yu, Yuma Ohara, Claude Meffan, Jun Hirotani, Amit Banerjee*, Toshiyuki Tsuchiya. Achieving Ultrawide Tunability in Monolithically Fabricated Si Nanoresonator Devices. *Nano Letters*, 23 (2023) 11517-11525.
- [2] Wei Yu*, Amit Banerjee, Jun Hirotani, Toshiyuki Tsuchiya. Enhancing responsivity and detection limit in tunable NEMS resonator mass sensors. *Japanese Journal of Applied Physics*. 63 (2024) 03SP74.
- [3] Wei Yu, Amit Banerjee*, Jun Hirotani, Toshiyuki Tsuchiya. Temperature Dependence of Electrostatic Frequency Tunability of Ultrathin Si Nanoresonators. *Sensors and Materials*. 36 (2024) 3395-3405.

Conference

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