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Nanomechanical resonators fabricated by atomic layer deposition on suspended 2D materials

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Abstract

Atomic layer deposition (ALD), a layer-by-layer controlled method to synthesize ultrathin materials, provides various merits over other techniques such as precise thickness control, large area scalability and excellent conformality. Here we demonstrate the possibility of using ALD growth on top of suspended 2D materials to fabricate nanomechanical resonators. We fabricate ALD nanomechanical resonators consisting of a graphene/MoS₂ heterostructure. Using atomic force microscope indentation and optothermal drive, we measure their mechanical properties including Young's modulus, resonance frequency and quality factor, showing a lower energy dissipation compared to their exfoliated counterparts. We also demonstrate the fabrication of nanomechanical resonators by exfoliating an ALD grown NbS₂ layer. This study exemplifies the potential of ALD techniques to produce high-quality suspended nanomechanical membranes, providing a promising route towards high-volume fabrication of future multilayer nanodevices and nanoelectromechanical systems.

The properties of 2D materials, in particular their ultralow weight and ultrahigh mechanical flexibility, provides them with an excellent sensitivity to external forces [1–3]. Hence, resonators from 2D materials have become a popular choice for the next generation of nanoelectromechanical systems [4, 5]. Recently, there is surge towards stacking different 2D materials into heterostructures often exhibiting better sensing properties. Such heterostructures are used for tunable resonators and oscillators [6], and can potentially lead to better sensors in microphone and pressure sensing applications [5].

To achieve high-performance nanomechanical resonators, clean interfaces between different 2D materials are important [7]. Therefore, bottom-up synthesis methods were developed, of which chemical vapor deposition (CVD) is the most attractive due to its large-scale and high-quality growth. The main shortcoming of CVD, however, is the difficulty

to accurately control the thickness and morphology of grown 2D materials. Atomic layer deposition (ALD), a vapor phase thin film deposition technique based on self-limiting surface reactions, inherently yields atomic-scale thickness control, excellent uniformity, and conformality [8]. ALD processes exists for a large variety of materials ranging from pure elements to metal oxides and chalcogenides [9]. In terms of 2D materials, ALD was applied to fabricate 2D-based field effect transistors, p–n diode devices, solar cells and photodetectors, displaying high electrical and optical uniformities [10]. Since experimental research of ALD materials for nanomechanical resonators is unexplored territory, it is of interest to study the potential of such devices and evaluate their mechanical performance.

In this work, we show two types of nanomechanical resonators fabricated using ALD: one consists of a heterostructure made from exfoliated graphene

(bottom layer) and ALD MoS₂ (top layer) and the other is ALD NbS₂. We use atomic force microscope (AFM) indentation to determine their Young's moduli and use an optomechanical method to study their resonance frequency and corresponding quality factor in vacuum conditions. The extracted parameters from our measurements agree well with literature values for 2D exfoliated or CVD resonators. Furthermore, by fitting a relation between the quality factors before and after ALD, we verify a low-level dissipation induced by ALD MoS₂. Our work indicates the potential of ALD fabrication techniques for realizing multilayer nanomechanical membranes and resonators with enhanced functionality and thickness control.

Results and discussion

The ALD layers are deposited (see figures 1(a) and (b)) by plasma-enhanced ALD (PE-ALD) technique using an Oxford Instruments Plasma Technology FlexAL ALD reactor. The base pressure of the system is 10⁻⁶ Torr. The metal-organic precursors bis(tert-butylimido)-bis(dimethylamido) molybdenum (STREM Chemical, Inc. 98%) and bis(tris-niobium) are used for MoO_x and NbO_x growth, respectively [9, 11]. The Mo and Nb precursors are kept in stainless steel bubblers at 50 °C and 65 °C, respectively and are bubbled using Ar as the carrier gas. In both the processes, O₂ plasma is used as the coreactant. The MoO_x and NbO_x films are deposited at 100 °C and 150 °C, respectively. More details on the PE-ALD recipes can be found in supplementary S1.

Both the MoS₂ and NbS₂ films are synthesized by a two step approach. As the first step, metal oxide (MoO_x or NbO_x) film is deposited by PE-ALD technique. Next, the metal oxide film is sulfurized at 900 °C in H₂S environment (10% H₂S and 90% Ar) to form metal sulfide film (MoS₂ or NbS₂). As shown in figures 1(a) and (c), the MoS₂ film is synthesized by PE-ALD on top of suspended graphene drums, resulting in 10 resonators with a radius $r = 4 \mu\text{m}$. Note that device D3 and D9 broke (buckled, figure 1(c)) during fabrication and will not be considered further. On the other hand, NbS₂ film is synthesized by growing NbO_x on glassy carbon followed by sulfurization at 900 °C. Then, we fabricate the NbS₂ resonators by transferring the NbS₂ films from glassy carbon substrate over circular cavities in a SiO₂/Si substrate to form suspended drums using the Scotch tape method [5] (see figure 1(b)). We tested the transfer of ALD nanoflakes with different thicknesses grown on different substrates and found that only the transfer of thick NbS₂ films from glassy carbon to the substrate with cavities was possible (see table SI). The cavities have a depth of 285 nm and were fabricated by reactive ion etching (see supplementary S1). The fabricated NbS₂ resonators, shown

in figure 1(d), have a radius of $r = 4 \mu\text{m}$ (devices D1 and D2) or $r = 3 \mu\text{m}$ (devices D4 and D5). We use AFM (tapping mode) to scan the surface of our fabricated samples, to determine the thickness of the 2D materials. By calculating the height difference between the membranes and substrate (see the statistics in figures 1(e) and (f)), we extract the mean thickness of the graphene $t_g = 13.3 \text{ nm}$ (40 layers), MoS₂ $t_m = 7.8 \text{ nm}$ (12 layers) and NbS₂ $t_n = 56.1 \text{ nm}$ (92 layers), respectively. The total thickness of the heterostructure is thus $t_h = t_m + t_g = 21.1 \text{ nm}$.

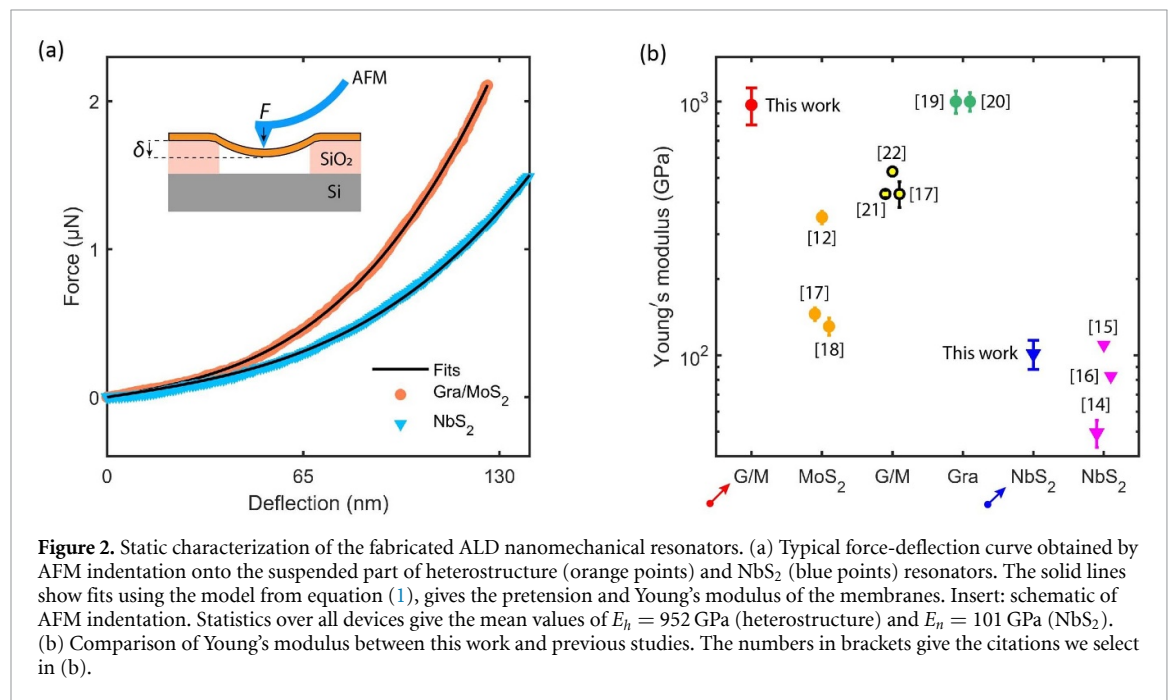
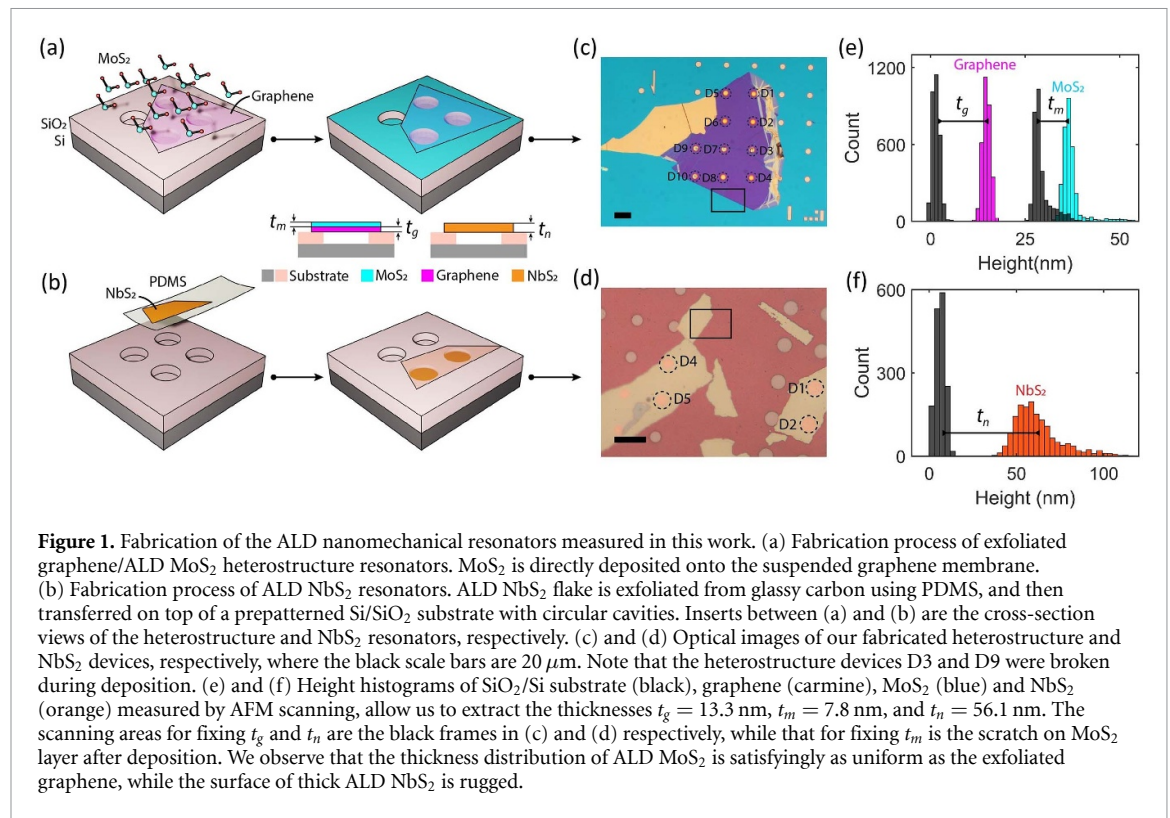
Raman spectra of both ALD heterostructures and NbS₂ devices, obtained with a 515 nm green laser at room temperature (see supplementary S2), clearly show the expected Raman modes and thereby verify the quality of fabricated devices after the high-temperature ALD synthesis processes. We also measure and theoretically analyze the Raman intensity ratio between the Si peak of suspended MoS₂/graphene membrane and that of substrate (see figure S4), from which we conclude that ALD MoS₂ layer is only deposited on top (but not on bottom) of the exfoliated graphene membrane in the fabricated heterostructure devices.

After fabrication, we determine the Young's modulus of the ALD devices by indenting with an AFM (contact mode) cantilever at the center of the suspended area (see figure 2(a), insert). Following literature [12], the applied vertical force F versus membrane deflection δ for a circular membrane (composed of single material), as depicted in figure 2(a), is given by

$$F = \left(\frac{16\pi D}{r^2} \right) \delta + n_0 \pi \delta + Etq^3 \left(\frac{\delta^3}{r^2} \right), \quad (1)$$

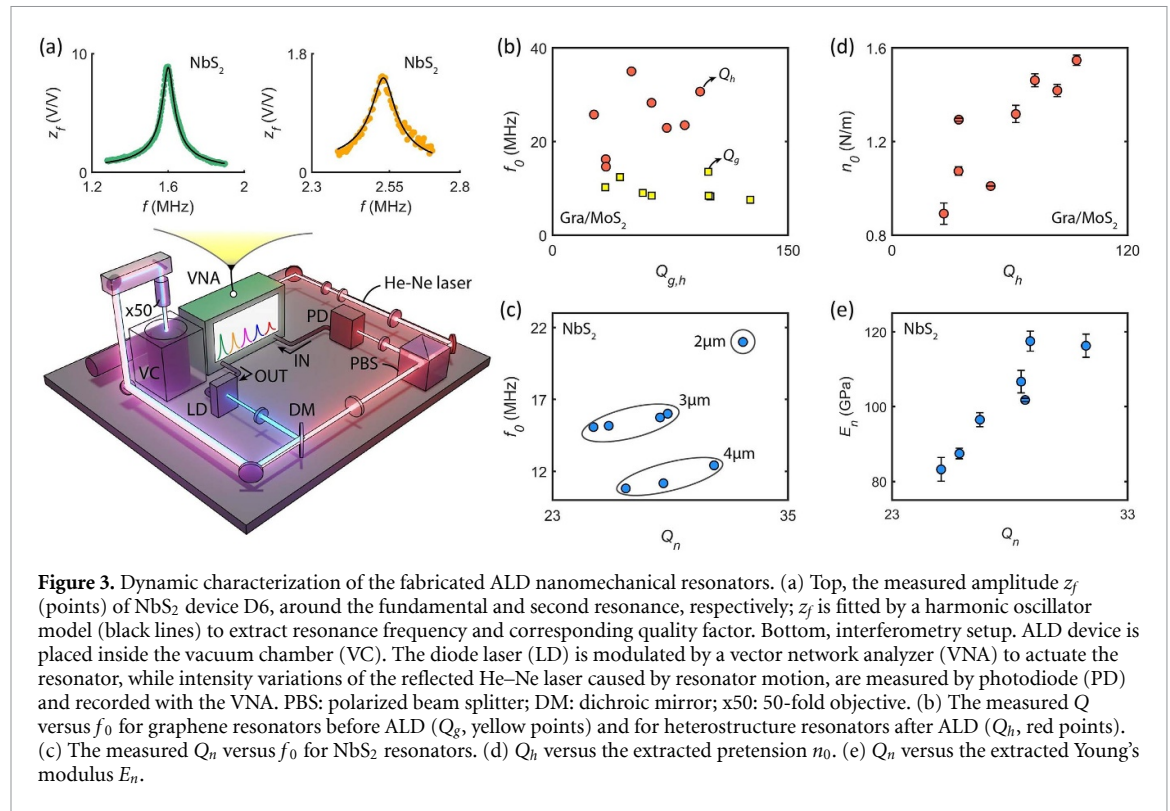
where $D = \gamma Et^3 / (12(1 - \nu^2))$ is bending rigidity, E is Young's modulus, ν is the Poisson ratio, n_0 is pre-tension, $q = 1 / (1.05 - 0.15\nu - 0.16\nu^2)$, and γ is a factor that quantifies the effect of interlayer shear interactions on D in multilayer 2D materials [13]. The first two terms in equation (1) scale linearly with δ ($F \sim \delta$) and are set by D and n_0 ; while the third cubic term ($F \sim \delta^3$) is due to the geometric nonlinearity of the membrane, which lead to an increase in the in-plane stress that depends on its Young's modulus E . Note that equation (1) is suitable for NbS₂, while for heterostructures, it contains contributions from graphene and MoS₂ layers (see equation (S5)). We use the bulk Poisson ratios $\nu_g = 0.165$, $\nu_m = 0.25$ and $\nu_n = 0.28$ of graphene, MoS₂ and NbS₂, respectively, in further analysis. In addition, considering the measured layer numbers of graphene, MoS₂ and NbS₂ membranes, we use the factors $\gamma_g = 0.1$ and $\gamma_m = 0.4$ from literature [13] and assume $\gamma_n = \gamma_m$.

We extract E_h and E_n by the fitting the measured F versus δ with equations (S5) and (1), respectively, which nicely describe the experimental data for the NbS₂ device D1 (blue points) and the heterostructure



device D2 (orange points) as shown in figure 2(a). The extracted statistics of effective Young's moduli for heterostructure devices (E_h) and Young's moduli (E_n) for NbS₂ devices give the mean values of $E_h = 952 \pm 161$ GPa and $E_n = 101 \pm 13$ GPa, respectively. In figure 2(b), we compare E_h and E_n with values reported in the literature: E_n shows a good agreement with the reported values of 75 ± 35 GPa [14–16]; E_h is between the reported values for MoS₂ 250 ± 120 GPa and graphene membranes 1025 ± 125 GPa

[12, 17–20], but higher than the reported values for similar fully exfoliated heterostructures 461 ± 43 GPa [17, 21, 22]. The larger E_h might be caused by the stronger interlayer adhesion or the larger intrinsic Young's modulus of ALD MoS₂. The standard deviations in extracted Young's moduli, ± 13 GPa and ± 161 GPa for NbS₂ and heterostructure resonators, respectively, are comparable to the ones reported in literature for exfoliated materials. This illustrates the high homogeneity of ALD materials.



In addition to the Young's modulus, we also extract the pretension n_0 for each device. Supplementary tables SII and SIII show a complete overview of the obtained parameters from the fitting to equation (1). The extracted n_0 ranges from 0.45 to 1.55 N m⁻¹ for all heterostructure and NbS₂ resonators, which are similar to values reported in the literature for resonators made by exfoliation and CVD [4, 21, 23].

Let us now focus on the dynamics of the ALD resonators. We measured the dynamic response of the membranes with a laser interferometer [23] (see figure 3(a), bottom). A power modulated blue diode laser ($\lambda = 405$ nm) photothermally actuates the resonator, while the reflection of a continuous-wave red He–Ne laser ($\lambda = 632$ nm) is sensitive to the time-dependent position of membrane. A vector network analyzer (VNA) provides a signal at drive frequency f (OUT port) that modulates the blue laser intensity while the intensity of the red laser recorded by a fast photodiode is connected to the IN port. The VNA thus measures a signal z_f that is proportional to the ratio of the membrane amplitude and actuation force. By sweeping the drive frequency f , we locate the resonance peak in the range from 100 kHz to 100 MHz. Laser intensities are set to 0.3 mW (blue) and 1.1 mW (red), respectively. These intensities are low enough for the resonator to vibrate in the linear regime. All measurements were performed at room temperature in vacuum at a pressure of 10⁻⁵ mbar.

Figure 3(a) (top inserts) shows the measured signal z_f of NbS₂ device D6, at around the fundamental and second resonance frequency, respectively.

By fitting z_f to the response function of a harmonic oscillator, we extract $f_0 = 16.0$ MHz with $Q = 28.9$ and $f_1 = 25.3$ MHz with $Q = 34.4$. For vibrations of clamped drums, we can compute the resonance frequencies f_i using [3]

$$f_i = \left(\frac{\mu_i}{2\pi} \right) \sqrt{\frac{D}{\sigma r^4} \left[\mu_i^2 + \frac{n_0 r^2}{D} \right]}, i = 0, 1, \dots, \quad (2)$$

where $\sigma = \eta \rho t$ is the areal mass density, η is a correction factor of mass considering the contaminations on resonators, and μ_i is a mode-specific factor. We have $\mu_1 = 2.4048$ for the fundamental mode and $\mu_2 = 3.8317$ for the second mode. For an ideal membrane, in which $n_0 r^2$ is much larger than the flexural rigidity D and thus $n_0 r^2 / D \rightarrow \infty$, we have $f_1 / f_0 = \mu_1 / \mu_0 = 1.59$; while for an ideal plate where D is much larger than $n_0 r^2$, we have $n_0 r^2 / D \rightarrow 0$ and thus $f_1 / f_0 = (\mu_1 / \mu_0)^2 = 2.54$. The measured f_1 / f_0 are 1.595 ± 0.167 and 1.959 ± 0.642 for heterostructure and NbS₂ devices, respectively (see supplementary tables SII and SIII), suggesting that the modes of heterostructure resonators are near the membrane limit, while the modes of NbS₂ resonators are in between the membrane and plate limit. We plot the extracted Q versus f_0 for all heterostructure resonators (Q_h) and NbS₂ resonators (Q_n) in figures 3(b) and (c), respectively, including the quality factor Q_g of the exfoliated graphene membranes before ALD. As expected from equation (2), f_0 decreases with increasing r for the NbS₂ resonators, while f_0 for heterostructure resonators varies widely from 14.6 to 30.7 MHz. This is attributed to the inhomogeneities like wrinkles

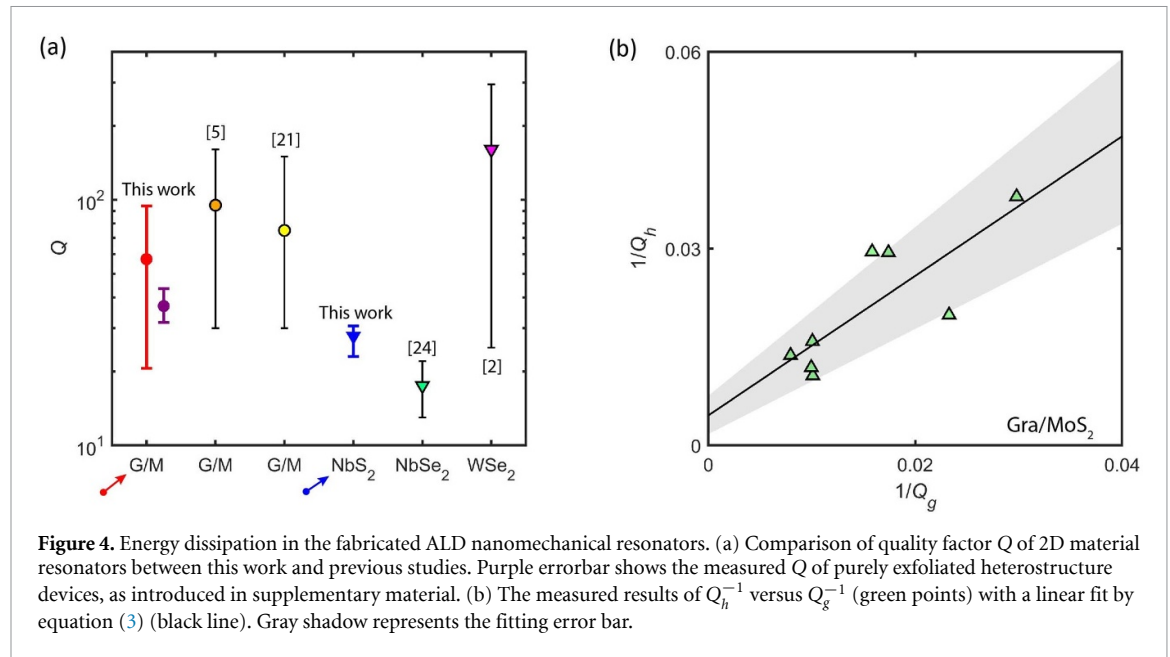


Figure 4. Energy dissipation in the fabricated ALD nanomechanical resonators. (a) Comparison of quality factor Q of 2D material resonators between this work and previous studies. Purple errorbar shows the measured Q of purely exfoliated heterostructure devices, as introduced in supplementary material. (b) The measured results of Q_h^{-1} versus Q_g^{-1} (green points) with a linear fit by equation (3) (black line). Gray shadow represents the fitting error bar.

and crumples in the heterostructures (see images in figure S5) and large differences in pretension. All measured resonance frequencies are comparable to those in literature reported for similar devices [3, 12].

The extracted values of Q_n and Q_h are also comparable to values of previously studied resonators made by exfoliation and CVD [2, 6, 21, 24], as illustrated in figure 4(a). To gain insight into the damping, we plot Q_h versus n_0 for heterostructure resonators and Q_n versus E_n for NbS₂ resonators, respectively, as plotted in figures 3(d) and (e). For both cases, we observe a linear relation, indicating that pretension plays a more important role on damping than bending rigidity for heterostructure resonators, while it is on the other way around for NbS₂ resonators. This is exactly as expected based on the ratio f_1/f_0 . On the other hand, we do not see clear relations of Q_h versus E_h and Q_n versus n_0 as plotted in figures S6(a) and (b), respectively.

Concerning the effective masses, we determine the correction factors η_h and η_n by substituting the measured f_0 , and the extracted n_0 and E into equation (2) (see values in supplementary tables I and II). We obtain $\eta_h = 1.34 \pm 0.92$ and $\eta_n = 2.87 \pm 0.83$, respectively. The high η_n of NbS₂ devices is attributed to the contaminations from the PDMS stamping. The values η_h for the heterostructure are surprisingly close or even below 1. This suggests the absence of any residues and possibly even the thinning of the graphene membrane during the ALD process, while the ALD MoS₂ layer is mainly deposited on top of the suspended graphene membrane instead of bottom (see supplementary S2).

We also observe a general decrease of quality factor in heterostructure resonators after ALD ($Q_h < Q_g$), as shown in figure 3(b). Considering the

dissipation mechanism for two parallel membranes, the overall Q_h can be modeled as

$$1/Q_h = \alpha/Q_g + 1/Q_m, \quad (3)$$

where α can be different than 1 on account of structural changes in the graphene because of the ALD process, and Q_m^{-1} is a fit parameter that represents the damping in the heterostructure originating from the ALD MoS₂. We fit the measured Q_h^{-1} versus Q_g^{-1} with equation (3) (see figure 4(b)) and extract $\alpha = 1.1 \pm 0.1$ and $1/Q_m = 4.7 \pm 3.1 \times 10^{-3}$. The fact that the obtained α (within errors) is close to 1, provides evidence that there little to none increase of the dissipation in the graphene during the ALD process. A control experiment has been done with purely exfoliated graphene/MoS₂ heterostructures (see figure S7), giving us $\alpha = 1.1 \pm 0.2$ and $1/Q_m = 17.6 \times 10^{-3}$. The lower $1/Q_m$ of ALD heterostructure compared to exfoliated layers can be attributed to a better conformality of the ALD layer and the absence of contamination by transfer polymers.

Compared with PE-CVD method that grows 2D materials under a temperature more than 400 °C, the reaction temperature window for ALD here is much lower (100 °C for MoO_x deposition), which significantly improve the survival rate of suspended graphene membranes. Therefore, PE-ALD is a safer and more efficient method for fabricating high-performance 2D heterostructure resonators. In addition, although ALD is known to be capable of wafer-scale synthesis, the dimensions of our fabricated devices are still quite small due to the use of exfoliation in the fabricating process. A strategy could be to grow transfer less suspended CVD 2D material membranes like graphene [25], and subsequently grow ALD material

heterostructures from them. In addition, ALD could benefit from a method to precisely control the flatness, so as to avoid the cragged surfaces of nanoscale devices as illustrated in figures S5(a) and (b).

2. Conclusions

In conclusion, we presented the fabrication and mechanical characterization of nanomechanical resonators consisting of ALD 2D materials. We developed two PEALD based approaches to suspend ALD flakes on a patterned Si/SiO₂ substrate: one is dry transfer using PDMS (exfoliate ALD Nb₂ flakes from glassy carbon); the other is ALD deposition of MoS₂ on mechanically exfoliated suspended graphene drums. AFM indentation allows us to determine their Young's moduli as 101.4 ± 13.3 GPa and 951.7 ± 161.0 GPa. Using an optomechanical method, we extracted their resonance frequencies and the corresponding quality factors. All of the above parameters are well comparable to the reported values of exfoliated and CVD resonators. We found experimental indications that the dissipation of ALD MoS₂ membranes in heterostructures is roughly 3.7 times lower than that of purely exfoliated MoS₂ membranes, which is promising for high-performance 2D heterostructure resonators. Our results show possibilities toward exploiting ALD technique for nanomechanical resonators in explorations on atomically thin tunable resonators and 2D sensors. Further work could focus on the thickness control of ALD resonators, which can bring significant improvements in device performance and lead to new functionalities.

Methods

Sample fabrication

A Si wafer with 285 nm dry SiO₂ is spin coated with positive e-beam resist and exposed by electron-beam lithography. Afterwards, the SiO₂ layer without protection is completely etched using CHF₃ and Ar plasma in a reactive ion etcher. More details on the fabrication of substrate can be found in supplementary S1. The edges of cavities are examined to be well-defined by scanning electron microscopy and AFM (see figures S2(a) and (b)). After resist removal, few-layer graphene and ALD NbS₂ nanoflakes are exfoliated by Scotch tape, and then separately transferred onto the substrate at room temperature through a deterministic dry stamping technique.

Optothermal drive

We use an interferometer setup [26, 27] to measure the resonance frequency and Q factor of the fabricated resonators. An intensity-modulated blue laser ($\lambda = 405$ nm) irradiates the membrane resulting in a periodic heat flux to actuate it, while a intensity-fixed red laser ($\lambda = 633$ nm) is utilized to detect the motion. The heat flux results in a motion of the drum

due to the thermal expansion force. All measurements are performed at room temperature inside a vacuum chamber at 10^{-6} mbar. A vector network analyzer (VNA) modulates the intensity of a blue laser at frequency ω to optothermally actuate a resonator while it analyzes the resulting intensity modulation of the red laser caused by the mechanical response of the same resonator. The red and blue laser powers used are 1.20 and 0.13 mW respectively, where the resonators vibrate in the linear regime and the temperature increase due to self-heating was negligible.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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Conflict of interest

The authors declare no competing financial or non-financial interests.

Author contributions

H L, S L, P G S and G J V conceived the experiments. H L performed the optomechanical measurements. H L and S L fabricated and inspected the samples. S B B and A A B synthesized and characterized ALD materials. H L, G J V and P G S analyzed and modeled the experimental data. H S J v d Z, A A B and P G S supervised the project. The paper was jointly written by all authors with a main contribution from H L. All authors discussed the results and commented on the paper.

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