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

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Supplementary material for this article is available [online](#)

## 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/MoS<sub>2</sub> 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 NbS<sub>2</sub> 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 MoS<sub>2</sub> (top layer) and the other is ALD NbS<sub>2</sub>. 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 MoS<sub>2</sub>. 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<sup>-6</sup> Torr. The metal-organic precursors bis(tert-butylimido)-bis(dimethylamido)molybdenum (STREM Chemical, Inc. 98%) and bis-tris-niobium are used for MoO<sub>x</sub> and NbO<sub>x</sub> 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, O<sub>2</sub> plasma is used as the coreactant. The MoO<sub>x</sub> and NbO<sub>x</sub> 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 MoS<sub>2</sub> and NbS<sub>2</sub> films are synthesized by a two step approach. As the first step, metal oxide (MoO<sub>x</sub> or NbO<sub>x</sub>) film is deposited by PE-ALD technique. Next, the metal oxide film is sulfurized at 900 °C in H<sub>2</sub>S environment (10% H<sub>2</sub>S and 90% Ar) to form metal sulfide film (MoS<sub>2</sub> or NbS<sub>2</sub>). As shown in figures 1(a) and (c), the MoS<sub>2</sub> 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, NbS<sub>2</sub> film is synthesized by growing NbO<sub>x</sub> on glassy carbon followed by sulfurization at 900 °C. Then, we fabricate the NbS<sub>2</sub> resonators by transferring the NbS<sub>2</sub> films from glassy carbon substrate over circular cavities in a SiO<sub>2</sub>/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 NbS<sub>2</sub> films from glassy carbon to the substrate with cavities was possible (see table S1). The cavities have a depth of 285 nm and were fabricated by reactive ion etching (see supplementary S1). The fabricated NbS<sub>2</sub> 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), MoS<sub>2</sub>  $t_m = 7.8 \text{ nm}$  (12 layers) and NbS<sub>2</sub>  $t_n = 56.1 \text{ nm}$  (92 layers), respectively. The total thickness of the heterostructure is thus  $t_h = t_m + t_g = 21.1 \text{ nm}$ .

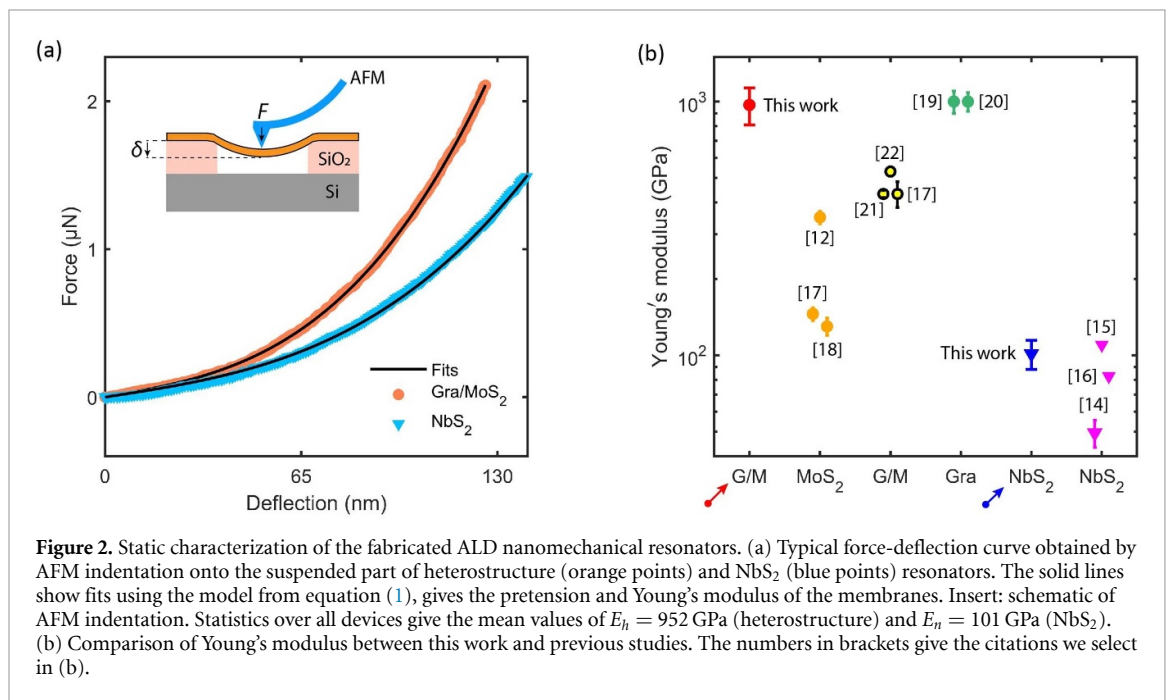
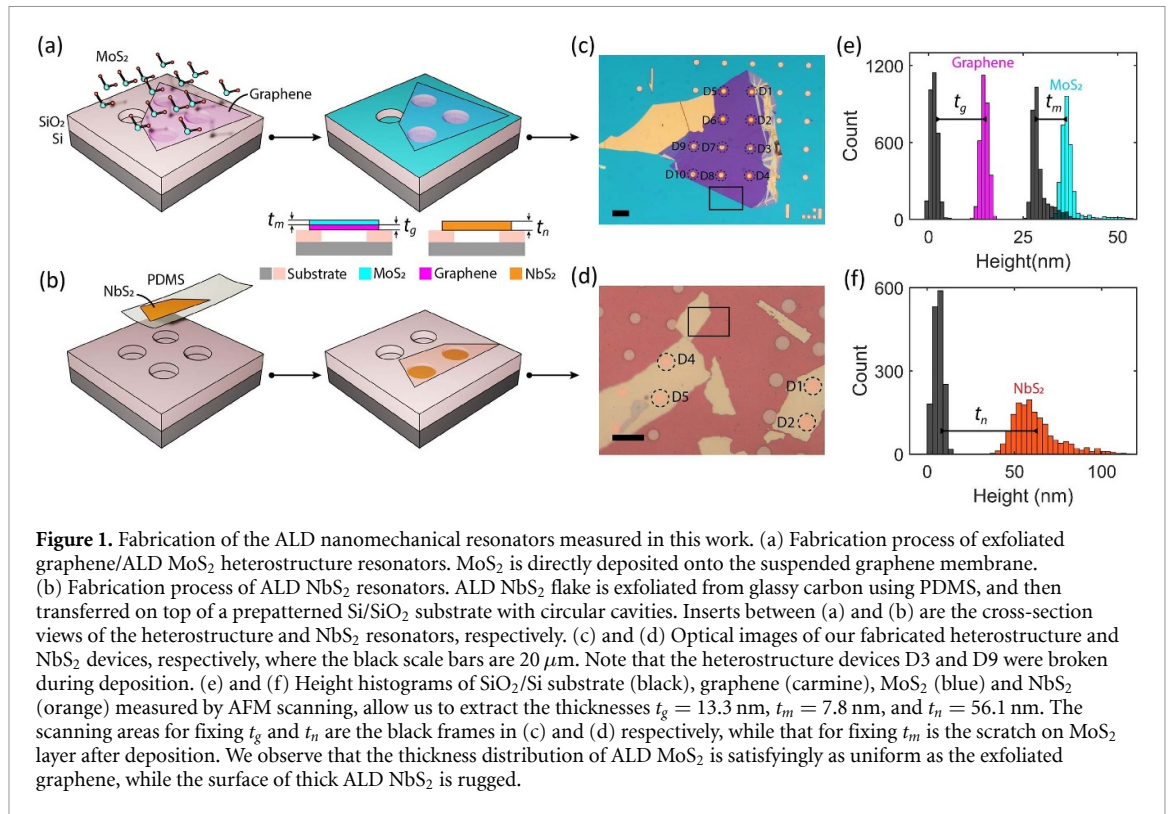
Raman spectra of both ALD heterostructures and NbS<sub>2</sub> devices, obtained with a 515 nm green laser at room temperature (see supplementary S2), clearly show the expected Raman modes and thereby verify the quality of fabricated devices after the high-temperature ALD synthesis processes. We also measure and theoretically analyze the Raman intensity ratio between the Si peak of suspended MoS<sub>2</sub>/graphene membrane and that of substrate (see figure S4), from which we conclude that ALD MoS<sub>2</sub> 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  $\delta$  for a circular membrane (composed of single material), as depicted in figure 2(a), is given by

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

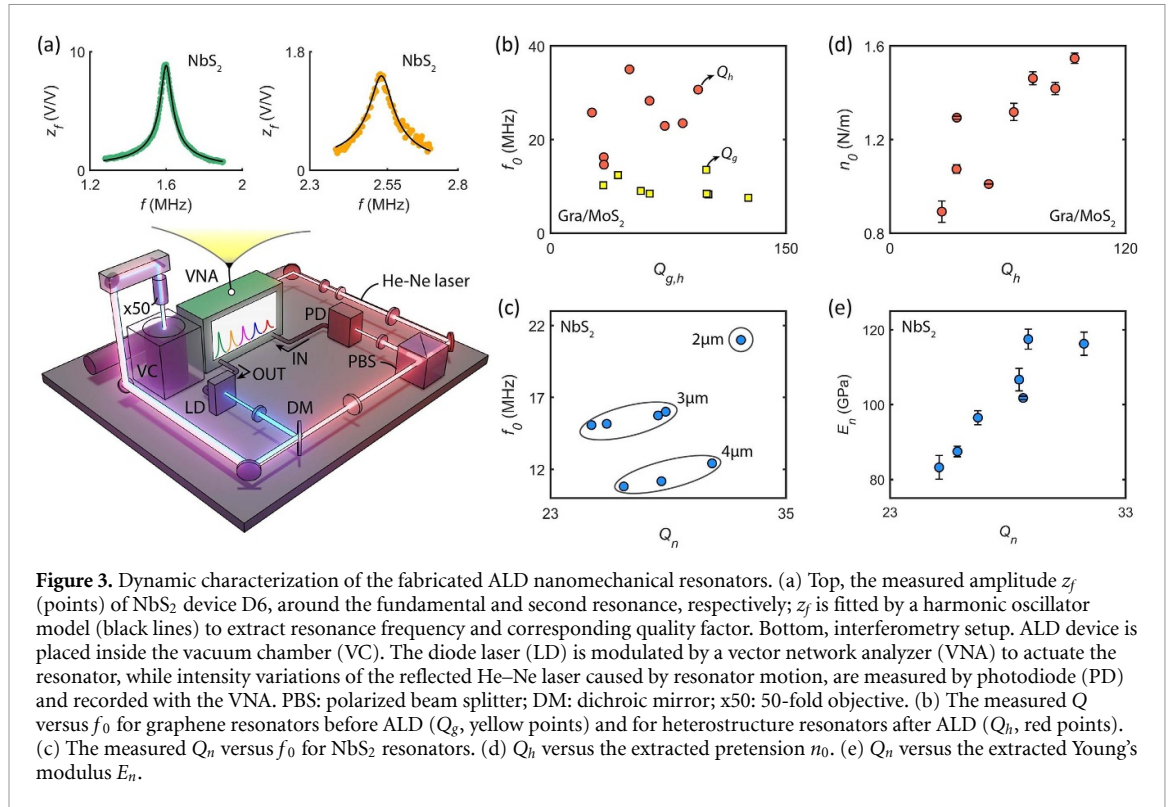
where  $D = \gamma Et^3 / (12(1 - \nu^2))$  is bending rigidity,  $E$  is Young's modulus,  $\nu$  is the Poisson ratio,  $n_0$  is pre-tension,  $q = 1 / (1.05 - 0.15\nu - 0.16\nu^2)$ , and  $\gamma$  is a factor that quantifies the effect of interlayer shear interactions on  $D$  in multilayer 2D materials [13]. The first two terms in equation (1) scale linearly with  $\delta$  ( $F \sim \delta$ ) 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 NbS<sub>2</sub>, while for heterostructures, it contains contributions from graphene and MoS<sub>2</sub> 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, MoS<sub>2</sub> and NbS<sub>2</sub>, respectively, in further analysis. In addition, considering the measured layer numbers of graphene, MoS<sub>2</sub> and NbS<sub>2</sub> 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  $\delta$  with equations (S5) and (1), respectively, which nicely describe the experimental data for the NbS<sub>2</sub> 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 NbS<sub>2</sub> 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  $75 \pm 35$  GPa [14–16];  $E_h$  is between the reported values for MoS<sub>2</sub>  $250 \pm 120$  GPa and graphene membranes  $1025 \pm 125$  GPa

[12, 17–20], but higher than the reported values for similar fully exfoliated heterostructures  $461 \pm 43$  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 MoS<sub>2</sub>. The standard deviations in extracted Young's moduli,  $\pm 13$  GPa and  $\pm 161$  GPa for NbS<sub>2</sub> and heterostructure resonators, respectively, are comparable to the ones reported in literature for exfoliated materials. This illustrates the high homogeneity of ALD materials.



**Figure 3.** Dynamic characterization of the fabricated ALD nanomechanical resonators. (a) Top, the measured amplitude  $z_f$  (points) of NbS<sub>2</sub> device D6, around the fundamental and second resonance, respectively;  $z_f$  is fitted by a harmonic oscillator model (black lines) to extract resonance frequency and corresponding quality factor. Bottom, interferometry setup. ALD device is placed inside the vacuum chamber (VC). The diode laser (LD) is modulated by a vector network analyzer (VNA) to actuate the resonator, while intensity variations of the reflected He–Ne laser caused by resonator motion, are measured by photodiode (PD) and recorded with the VNA. PBS: polarized beam splitter; DM: dichroic mirror; x50: 50-fold objective. (b) The measured  $Q$  versus  $f_0$  for graphene resonators before ALD ( $Q_g$ , yellow points) and for heterostructure resonators after ALD ( $Q_h$ , red points). (c) The measured  $Q_n$  versus  $f_0$  for NbS<sub>2</sub> resonators. (d)  $Q_h$  versus the extracted pretension  $n_0$ . (e)  $Q_n$  versus the extracted Young's modulus  $E_n$ .

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<sup>-1</sup> for all heterostructure and NbS<sub>2</sub> resonators, which are similar to values reported in the literature for resonators made by exfoliation and CVD [4, 21, 23].

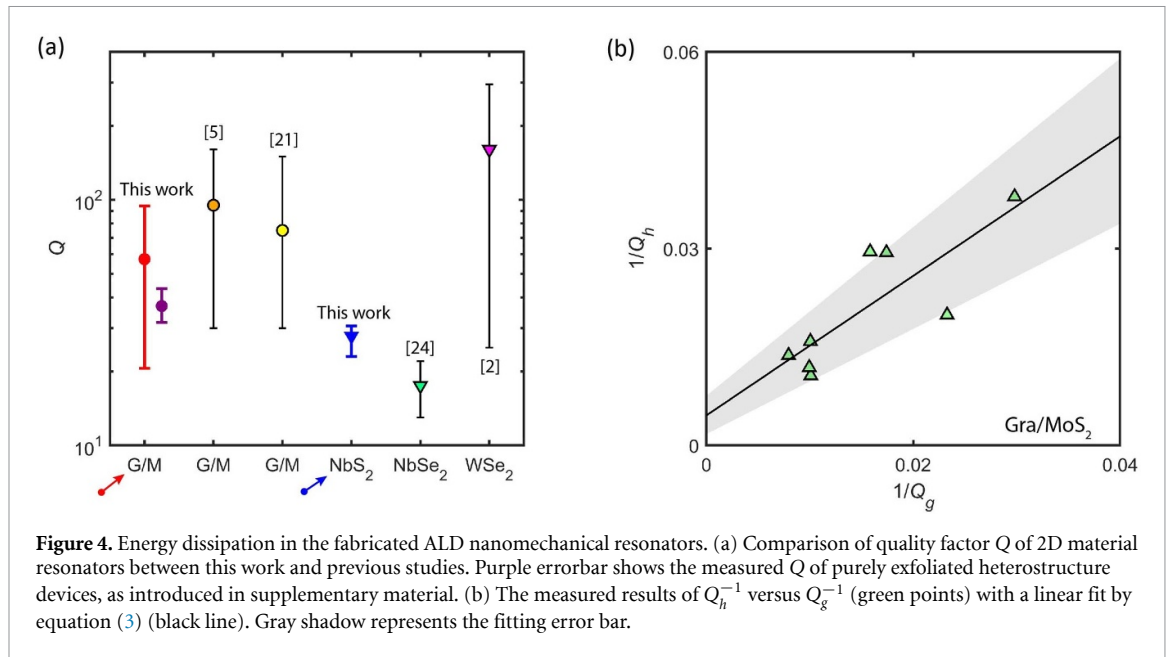
Let us now focus on the dynamics of the ALD resonators. We measured the dynamic response of the membranes with a laser interferometer [23] (see figure 3(a), bottom). A power modulated blue diode laser ( $\lambda = 405$  nm) photothermally actuates the resonator, while the reflection of a continuous-wave 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 NbS<sub>2</sub> 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,  $\eta$  is a correction factor of mass considering the contaminations on resonators, and  $\mu_i$  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  $1.595 \pm 0.167$  and  $1.959 \pm 0.642$  for heterostructure and NbS<sub>2</sub> devices, respectively (see supplementary tables SII and SIII), suggesting that the modes of heterostructure resonators are near the membrane limit, while the modes of NbS<sub>2</sub> resonators are in between the membrane and plate limit. We plot the extracted  $Q$  versus  $f_0$  for all heterostructure resonators ( $Q_h$ ) and NbS<sub>2</sub> 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 NbS<sub>2</sub> 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 NbS<sub>2</sub> 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 NbS<sub>2</sub> 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  $\eta_h$  and  $\eta_n$  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  $\eta_n$  of NbS<sub>2</sub> devices is attributed to the contaminations from the PDMS stamping. The values  $\eta_h$  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 MoS<sub>2</sub> 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  $\alpha$  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 MoS<sub>2</sub>. 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  $\alpha$  (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/MoS<sub>2</sub> 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<sub>x</sub> 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/SiO<sub>2</sub> substrate: one is dry transfer using PDMS (exfoliate ALD Nb<sub>2</sub>S<sub>2</sub> flakes from glassy carbon); the other is ALD deposition of MoS<sub>2</sub> on mechanically exfoliated suspended graphene drums. AFM indentation allows us to determine their Young's moduli as  $101.4 \pm 13.3$  GPa and  $951.7 \pm 161.0$  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 MoS<sub>2</sub> membranes in heterostructures is roughly 3.7 times lower than that of purely exfoliated MoS<sub>2</sub> 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 SiO<sub>2</sub> is spin coated with positive e-beam resist and exposed by electron-beam lithography. Afterwards, the SiO<sub>2</sub> layer without protection is completely etched using CHF<sub>3</sub> 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 NbS<sub>2</sub> 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  $\omega$  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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