Mechanical characterization and cleaning of CVD single-layer h-BN resonators

Cartamil-Bueno, Santiago J.; Cavalieri, Matteo; Wang, Ruizhi; Houri, Samer; Hofmann, Stephan; van der Zant, Herre

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Hexagonal boron nitride (h-BN) is a 2D material whose single-layer allotrope has not been intensively studied despite being the substrate for graphene electronics. Its transparency and stronger interlayer adhesion with respect to graphene makes it difficult to work with, and few applications have been proposed. We have developed a transfer technique for this extra-adhesive material that does not require its visual localization, and fabricated mechanical resonators made out of chemical vapor-deposited single-layer hexagonal boron nitride. The suspended material was initially contaminated with polymer residues from the transfer, and the devices showed an unexpected tensioning when cooling them to 3 K. After cleaning processes in harsh environments with air at 450 °C and ozone, the temperature dependence changed with \( f_0 Q \) products reaching \( 2 \times 10^{10} \) Hz at room temperature. This work paves the way to the realization of highly sensitive mechanical systems based on hexagonal boron nitride, which could be used as an alternative material to SiN for optomechanics experiments at room temperature.

**RESULTS**

Fabrication of h-BN microdrums

Figure 1a shows the fabrication process of suspended h-BN devices. A continuous layer of CVD h-BN is grown on iron foils and characterized by transmission electron microscopy (TEM) to be single layer as previously reported.\(^{17}\) Due to the stronger adhesion of h-BN to surfaces, it has to be transported from the growth substrate to the final substrate by using sacrificial layers. Poly(methyl methacrylate) (PMMA) is spin coated on top of the h-BN, and the stack is delaminated with the bubbling technique:\(^{35}\) a voltage of 3 V is applied between the Fe/h-BN/PMMA stack (cathode) and a Pt wire (anode) of an electrochemical cell with 1 M NaOH (electrolyte). From scanning electron microscopy (SEM) images taken before the delamination, the grain size is estimated to be around 20 μm. The h-BN/PMMA stack is then transferred onto a piece of silicon covered with 300 nm of poly(vinyl alcohol).
1. Mechanical characterization of suspended CVD single-layer h-BN is performed by AFM nanoindentation. We use the Peak Force mode (ScanAssyst-Fluid probe) to deflect drums of 5 µm in diameter with a controlled force of 5 nN. Figure 3a shows the AFM image of the suspended drum from Fig. 1b with its corresponding deflection curve at its center. The tension and Young's modulus of the material are extracted by fitting the data for positive forces to the following equation valid for a circular structure under a central point load F:37

\[
F = \frac{4\pi^2E}{3(1-\nu^2)R^2} \pi N_0 \delta + \frac{te}{(1.05 - 0.15\nu - 0.16\nu^2)R^2} \delta^3, \tag{1}
\]

where \(N_0\), \(E\), and \(\nu = 0.18\) are the pre-tension, Young's modulus and Poisson's ratio of the suspended material (chosen as an intermediate value in the range reported in literature), respectively; \(t = 0.44\) nm is its thickness, \(R\) is the radius of the drum, and \(\delta\) is the deflection induced by the AFM tip. The Young's modulus \(E\) is extracted by fitting the large-deformation data to the cubic term, although for small applied forces the accuracy is limited. The thickness-dependent term of the linear spring constant is negligible in these atomically-thin devices as the transfer of the material usually introduces stress that dominates the response at small deformations. This membrane behavior (e.g., tension dominated suspended structures) is found experimentally in the h-BN drums as shown below. The extracted tension and Young's modulus values are shown in Table 1 for five different drums, and they are expected to be influenced by the polymer in a second order of importance. Note the large Young's modulus of an undamaged drum (device 5), which is comparable to that of pristine graphene,38 and the lower values obtained for the other perforated devices.

The mechanical properties are also measured indirectly from the drum dynamics with a laser interferometric technique in a
Fig. 2  Single-layer h-BN characterization. a Raman spectroscopy is used to determine the single-layer nature of the suspended material. As reported in ref. 9, we find a peak at 1370 cm$^{-1}$ that corresponds to a monolayer h-BN. Inset Vibrational mode of a h-BN ring for that Raman frequency. b AFM height image of a h-BN material edge before cleaning. The obtained thickness (5 nm) is larger than that expected for single-layer (0.44 nm). The material is covered with polymer contamination that can be removed by oven annealing at 450 °C in air and ozone as seen in c, where areas of 0.5 nm thick are found.

Fig. 3  Characterization of h-BN 5 µm in diameter drums. a AFM image of the h-BN drum showed in Fig. 1b. The CVD material shows wrinkles and sidewall adhesion as in CVD graphene (left panel). The force distance curve in the center of the drum is obtained by AFM indentation in Peak Force mode. From the retraction curve we extract the tension and Young’s modulus. b Mechanical frequency spectrum of the same drum at room temperature and ~10$^{-6}$ mbar when measured with the optical interferometric technique after cleaning. The second resonance peak appears around 1.5 times the fundamental frequency, indicating membrane behavior.37 Inset Mechanical fundamental frequency and phase analysis of the same drum before cleaning. By fitting the frequency peak to the driven harmonic oscillator equation, we extract the tension values.
cryo-station setup (Montana Instruments). A power-modulated diode laser (λ = 405 nm) photothermally actuates the drum, while a continuous-wave He-Ne laser (λ = 633 nm) allows the measurement of its resonance frequency \( f_0 \). Figure 3b displays the frequency spectrum of the same drum shown in Fig. 3a at a room temperature and pressure of \( \sim 10^{-6} \) mbar, showing the fundamental mode around 13 MHz and the second (splitted) mode around 1.5 times the fundamental one. The factor of 1.5 indicates that the single-layer h-BN acts as a membrane in which pretension dominates over the bending rigidity.\(^{35}\) The fundamental mode is given by:

\[
f_0 = \frac{2.4}{2\pi R} \sqrt{\frac{N_0}{\rho t}},
\]

where \( \rho \approx 2100 \text{ kg/m}^3 \) is the mass density of the suspended material. By fitting the fundamental peak to the response function of a harmonic oscillator, we extract the resonance frequency that is used to calculate the tension from Eq. 2. The pre-tension values are shown in Table 2.

Temperature dependence of the resonance frequency

Figure 4 shows the temperature dependence of the fundamental frequency when cooling the device from 300 to 3 K. Equation 2 has to be modified to take into account the temperature-induced tensioning of the membrane due to the in-plane expansion coefficients of the 2D material and the underlying substrate dominated by the thermal expansion of the Si:

\[
f_0(T) = \frac{2.4}{2\pi R} \sqrt{\frac{N_0}{\rho T}} + \frac{N_{\text{adhesion}}(T)}{\rho T},
\]

where \( \alpha_0 \) and \( \alpha_{2D} \) are the temperature-dependent expansion coefficient of silicon and the 2D material, respectively, in the basal plane; \( \Delta T = T - 300 K \) is the temperature difference from room temperature. \( N_{\text{sidewall}} \) is a new tensioning mechanism that originates from the adhesion of the suspended 2D material to the sidewalls of the circular cavity, which happens when the membrane tries to expand laterally inside the cavity. This adhesion could balance out \( N_{\text{sidewall}}(T) = 0 \) or increase the tension due to a biaxial in-plane strain \( \left(N_{\text{sidewall}}(T) = \frac{E_{\text{BN}} L^2}{R^2} \alpha_{\text{BN}}(T) \right) \), where \( L \) is the change of adhered length on the sidewall.\(^{40}\) When cooling down the device made out of single-layer h-BN with negative in-plane thermal expansion coefficient \( \alpha_{\text{h-BN}}(T) < \alpha_{\text{h-BN}}(0) \), we would expect a constant or monotonic increase of \( f^2 \), i.e., \( f^2(T) = f^2(300K) \alpha_x T \) with a zero or negative slope. The data, red circles in Fig. 4, show such a trend suggesting that \( N_{\text{adhesion}} = N_{\text{sidewall}} \). However, another possible explanation for this could be residual resist remaining on top of the h-BN after transfer.

### Impact of cleaning processes

To test this hypothesis, we clean the membranes by annealing the samples at 450 °C for 3.5 h in an air environment (78% \( N_2 \), 21% \( O_2 \)), and in a further cleaning step using ultraviolet (UV)-induced ozone environment for 30 min. The oven annealing is expected to cause thermal and chemical degradations of polymers (boiling and evaporation of PVA, oxidation and gasification thermolysis of PMMA), and the UV ozone cleaning should produce photo-induced and chemical decomposition of polymers (photolysis of remaining PVA/PMMA and ozonolysis of PDMS) and decompositions of other organic matter with the UV ozone cleaning. Despite these cleaning processes in harsh environments, the h-BN remains suspended and the material becomes invisible under the optical microscope as expected from its low absorption of visible light, thus indicating that the contaminating polymer layer has been largely removed. After the treatments, the fundamental mode frequency at room temperature increased for all drums, although slightly. We also repeated the measurement as a function of temperature as shown in Fig. 4 by the blue (after oven annealing) and black data points (after ozone cleaning). More details about the temperature dependence of the mechanical properties of the h-BN drums can be found in Supplementary Information together with the complete dataset. The conclusion from the data is that when cooling a slight increase in the resonance frequency remains, although the effect is smaller than before annealing.

### DISCUSSION

References 8 and 16 report the mechanical characterization by AFM indentation of similar devices made out of CVD multilayer h-BN (1–2 and 5–15 nm in thickness, respectively). In those works, the material is placed on top of cavities with different transfer techniques that also introduce polymer contamination and wrinkles as we have observed in our devices by SEM and AFM. The Youngs modulus reports (220–250 and 1160 GPa, respectively) are comparable to our measured values at room temperature, and in particular device 5 proves to have a stiffness close to the theoretical value of single-layer h-BN (885–995 GPa).

### Table 1. Tension and Young's modulus values determined from AFM nanoindentation measurements for five different drums of 5 \( \mu \)m in diameter

| Device | \( N_0 \) (10^{-3} N/m) | E (GPa) |
|--------|---------------------|--------|
| 1      | 21                  | 102    |
| 2      | 25                  | 199    |
| 3      | 21                  | 186    |
| 4      | 38                  | 81     |
| 5      | 48                  | 936    |

### Table 2. Fundamental frequency and tension of CVD single-layer h-BN drums at room temperature from the laser interferometric technique before and after cleaning in harsh environments

| Device | \( f_0 \) (MHz) | \( N_0 \) (10^{-3} N/m) | \( f_0 \) (MHz) | \( N_0 \) (10^{-3} N/m) | \( f_0 \) (MHz) | \( N_0 \) (10^{-3} N/m) |
|--------|----------------|------------------------|----------------|------------------------|----------------|------------------------|
| 1      | 8.5            | 2.7                    | 14.8           | 8.6                    | 9.1            | 3.3                    |
| 2      | 9.9            | 3.7                    | 13.5           | 7.2                    | 14.2           | 7.9                    |
| 3      | 10.0           | 3.8                    | 12.5           | 15.6                   | 19.9           | 15.6                   |
| 4      | 10.4           | 4.1                    | 9.9            | 3.9                    | 17.0           | 11.4                   |
| 5      | 9.1            | 3.1                    | 13.2           | 6.8                    | 20.3           | 16.3                   |
Fig. 4 Temperature dependence of the mechanical resonance frequency (fundamental mode) averaged for the h-BN drums before cleaning, after oven annealing, and after UV ozone cleaning. By cooling the drums from 300 K down to 3 K, the material is tensioned resulting in higher frequencies. Despite the expected negative in-plane thermal coefficient of h-BN, no compression is observed even after cleaning, hence suggesting that other tensioning mechanisms are present.

In another work on graphene, the temperature-dependence of the Youngs modulus was proven to be strongly influenced by the presence of wrinkles in the suspended membranes. More importantly, the previous works on h-BN present membranes that sag into the cavities due to the van der Waals forces between the material and the sidewalls. Considering that the 2D Youngs modulus scales with the thickness, it is expected that single-layer h-BN shows larger sagging than previously reported (20–50 and 15 nm, in the mentioned works). AFM scans of our devices show sagging of 50–60 nm, thus supporting the monolayer nature of the material.

Bulk h-BN has a negative in-plane thermal expansion coefficient for temperatures higher than 75 K. We assume that single-layer h-BN exhibits the same tendency as graphene with respect to graphite, i.e., that it has the same negative temperature dependence as in the bulk, but with a larger magnitude. The observed tensioning must therefore originate from (a) an unexpected positive in-plane thermal expansion coefficient for h-BN as a single layer caused by the presence of polymer contamination even after cleaning, (b) other tensioning mechanism such as the proposed sidewall adhesion or the presence of wrinkles, or (c) defect creation during UV ozone cleaning.

When comparing the tension values from the AFM and the laser interferometric studies as shown in Tables 1 and 2 (before cleaning), we observe a difference of one order of magnitude between the two. The indirect calculation of the tension from Eq. 2 includes the mass of the membrane as a parameter as opposed to the direct measurement, where the mass does not appear in Eq. 1. In fact, the thickness of the material measured by AFM before cleaning (5 nm, about 10 times that of single-layer h-BN) may explain the difference in values obtained from the AFM indentation and the laser interferometric techniques. The spread in values obtained with both techniques can also be justified with residues of inhomogeneous thickness and hence mass. In addition, the quality factors increased significantly with respect to the pre-cleaning values (see Supplementary Information), suggesting that the polymer residues play a significant role in damping the mechanical motion, probably due to their viscoelastic properties.

On the other hand, the increase of the fundamental frequencies at room temperature after cleaning (Table 2) indicates that either the oven annealing introduced larger pre-tensions (enhanced adhesion to the surface or the sidewall of the cavity) and/or a decrease in the mass (hence obtaining tensions more similar to those obtained from the AFM) according to Eq. 2. The mechanical quality factor of the resonators improved after each of the two cleaning steps (see Supplementary Information). This fact coupled to the disappearance of traces of polymer contaminants and the on-average increase in the resonance frequency of the resonators suggests that polymer contamination is the main damping mechanism encountered in these resonators. It is unclear if there remains some polymer residue, although the tensioning trend when cooling has been reported in graphene and several transition metal dichalcogenide membranes. However, this trend should be explained differently for 2D materials with a negative expansion coefficient like h-BN or graphene, and for that reason we propose an alternative tensioning effect caused by the sidewall adhesion, which should balance out or increase the tension when cooling certain 2D membranes, and explains our results and those on graphene drumheads. Other stronger cleaning routes for h-BN are available, although not all of them might be compatible with suspended single-layer structures.

This study has presented the fabrication, mechanical characterization, and cleaning of CVD single-layer h-BN drumhead resonators. We have developed a transfer technique that allows the transfer of this extra-adhesive material from the growth substrate to a patterned SiO2 surface with circular microcavities, resulting in suspended h-BN drums. The tension and Young’s modulus of the material were characterized by performing AFM indentation on several devices, and the tension values are compared to those measured indirectly from the resonance frequency by means of laser interferometry. The temperature dependence of the dynamics of the drum is realized by cooling the devices to 3 K, and corroborates the assumption of having polymer residues covering the h-BN that dominates the tensioning of the devices. After cleaning in harsh environments, we observe a substantial improvement in the quality factor of the drums, and a change in their temperature-induced tensioning, which demonstrates cleaning of the suspended material. However, the temperature behavior suggests that some residues may still be present or that the structural mechanics model needs to be revisited for 2D materials, thus requiring further research. The strong adhesion of h-BN layers compared to other 2D materials could be exploited to make highly sensitive mechanical systems. Its electrical and optical properties are different than those from graphene, hence being a desirable material for applications where low-optical absorption or electrical insulation is required. Its nearly-zero extinction coefficient above 500 nm and good reflectivity could be used to make large f0Q NEMS for optomechanics experiments. Moreover, it could be combined with other 2D materials to fabricate NEMS à la carte with new functionalities for transparent and flexible electronics.

METHODS
Raman spectroscopy
Raman spectroscopy on the h-BN material over Si/SiO2 was carried out with a Renishaw in Via Raman microscope. Measurements with a laser light of 514 nm were performed on supported areas. The backscattered light was collected with a ×100 objective (NA = 0.95) and recorded with a 1800 lines/mm grating, which gives a spectral resolution of ~1.75 cm⁻¹. The laser power was about 25 mW.

Atomic force microscopy
A Bruker Fastscan is used in tapping mode for imaging the topography and having a rough measurement of the material thickness (FastScan-A probe). For the mechanical characterization of suspended CVD single-layer h-BN, the AFM is calibrated and used in Peak Force mode for nanoindentation tests (ScanAsyst-Fluid probe, Peak Force setpoint of 5 nN).

Laser interferometry
The frequency response of the resonators is measured using a customized laser interferometer integrated into cryo-station (Montana Instruments). Two lasers are focused on the suspended drumheads with a microscope objective lens (Mitutoyo ×100, NA = 0.75). The suspended material is
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photothermally actuated with a modulated blue diode laser (Thorlabs LP405-SF10, \( \lambda = 405 \text{ nm} \)) at a power of 260 \( \mu \text{W} \), while a second continuous-wave linearly-polarized Helium–Neon laser (\( \lambda = 632.8 \text{ nm} \)) at 13 mW is used as optical probe for the detection. The reflected red light is captured by a high speed photo-detector (New-Focus 1601) whose signal is crossed with the driving signal of the diode laser connected to a network analyzer (Rohde & Schwarz, ZVB4). The measurements are carried out in vacuum (10\(^{-6}\) mbar) for room temperature measurements, and higher vacuum when cooling.

Cleaning procedures
After fabrication and first measurements, the membranes are annealed at 450°C for 3.5 h in an air environment (Lindberg/Blue tube furnace). After the immediate new round of measurements, we use a Novascan PSD-UVT decontamination system at room temperature to clean the samples using UV-induced ozone, which precedes another immediate round of measurements.

Data availability
The authors declare that the data supporting the findings of this study are available within the paper and its supplementary information files.

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