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Transfer of III-nitride epitaxial layers onto pre-patterned silicon substrates for the simple fabrication of free-standing MEMS

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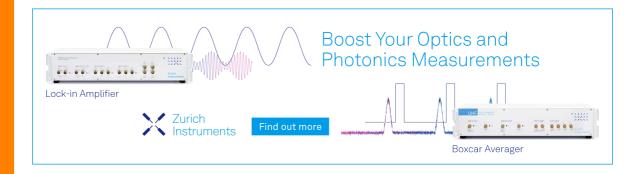


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ABSTRACT

In recent years, the remarkable properties and potential applications of III-nitride (III-N) semiconductors have sparked a significant interest in the field of microelectromechanical systems (MEMS). Traditionally, III-N MEMS are fabricated through a process involving the epitaxial growth of III-N epilayers on a silicon substrate followed by etching the handle wafer to generate free-standing structures. In this study, we explore the potential of a relatively simple approach based on the two-dimensional (2D) material-based liftoff and transfer to fabricate III-N mechanical resonators. The methodology involves van der Waals epitaxy of III-N layers on 2D hexagonal-boron nitride (h-BN), which leverages the weak van der Waals adhesion between h-BN layers to lift off and transfer these layers from their original growth substrate to an alternative host substrate. The employed method is demonstrated by fabricating 600 nm thick GaN/AlGaN and 2.5 μ m thick h-BN microresonators onto pre-patterned cavities etched in silicon substrates. These devices are characterized using laser Doppler vibrometry, enabling the observation of well-defined modes of vibration and resonant frequencies. Furthermore, finite element method simulations are performed to gain insights into the experimental observations and the mechanical properties of the transferred layers. This approach could be extended to transfer high-quality III-N MEMS devices onto various host substrates, including flexible substrates, and could be used to assess the mechanical properties of emerging III-N semiconductor materials.

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Gallium nitride (GaN) has garnered a significant attention in the field of microelectromechanical systems (MEMS) due to its intrinsic crystalline quality stemming from its wurtzite crystal structure, ¹ notable wide bandgap, ² piezoelectric and piezoresistive characteristics, ^{1,3,4} high temperature stability, ^{5,6} chemical robustness, ⁷ and seamless integration capabilities with GaN electronics. ^{8,9} Moreover, the AlGaN/GaN interface hosts a two-dimensional electron gas (2DEG) ^{10,11} that can function as a strain transducer with enhanced sensitivity due to its exceptional sheet carrier concentration (>10¹³ cm⁻²) and electron mobility (>2000 cm²/V s), without intentional doping. ^{1,12,13} These outstanding attributes of

AlGaN/GaN heterostructures position them as highly promising candidates for MEMS operations demanding high-frequency capabilities, superior performance, and sensitivity. ^{13–15}

GaN MEMS innovative components and systems include GaN Lamb wave resonators, ¹⁶ GaN-on-SOI resonators catering to infrared detection, ¹⁷ and GaN-on-silicon filters designed for specific filtration purposes. ¹⁸ The conventional process for fabricating GaN MEMS involves growing GaN epitaxial layers on silicon substrates, followed by substrate etching to create free-standing structures. ^{1,13-15} Silicon substrates are advantageous for easy micromachining but pose challenges for achieving high-quality crystalline III-N layers. ^{1,19} GaN-on-silicon

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substrates commonly exhibit high threading dislocation densities, mainly screw-type dislocations.²⁰

The heterogeneous integration of GaN MEMS to substrates that are not well-suited for epitaxy (e.g., flexible substrates) typically requires thin film transfer method, which involves isolating grown epilayers from their initial growth substrate for subsequent transfer onto the desired host substrate. Methods like laser liftoff,²¹, implantation-induced interface splitting, ^{23,24} and wet etching ^{25,26} serve this purpose. However, these approaches have limitations including slow processing, laser-induced interface damage, material constraints, limited thickness options, and potential ionic contamination.²⁶ Alternatively, the 2D material-based layer transfer (2DLT) technique is also pioneered^{29,30} that uses hexagonal-boron nitride (h-BN) as a sacrificial layer for the liftoff and transfer of III-N layers grown on h-BN/ sapphire templates to foreign substrates. Owing to the weak van der Waals interactions within the h-BN layers, the III-N epilayers can be mechanically lifted off from their native sapphire substrate, providing a route for convenient transfer onto another substrate. The adoption of this approach offers the potential for recycling the growth sapphire substrate, leading to reduced footprint and cost effectiveness. Notably, this technique has been used to fabricate flexible HEMTs gas sensors, LEDs,^{32–34} and solar cells.³⁵

In this context, this paper extends the 2DLT technique to the fabrication of free-standing III-N MEMS. This approach eliminates the requirement for etching a substrate or a sacrificial layer to release the movable parts of the microsystem; instead, the III-N layers are transferred to pre-patterned substrates. In order to validate the viability of the proposed technique, this study focuses on the process of lifting-off and transferring epitaxial layers of GaN (300 nm)/AlGaN (300 nm), grown onto a 3 nm h-BN sacrificial layer/sapphire template, onto prepatterned cavities etched in silicon substrates. Furthermore, this study underscores that h-BN can serve as more than just a transfer sacrificial layer; it also possesses the potential to function as the active layer in

MEMS. This is accomplished by transferring 2.5 μ m thick h-BN epilayers grown on sapphire onto pre-patterned silicon substrates. The AlGaN/GaN bi-layer and h-BN thin films are suspended above $180 \times 60~\mu\text{m}^2$ and $450 \times 150~\mu\text{m}^2$ rectangular cavities, respectively. Dynamic characterizations were performed on both structures. Finite element method (FEM) simulations using COMSOL were conducted to obtain the eigenfrequencies and the shapes of the first resonant modes and to gain insights into the experimental observations. In summary, this study demonstrates the transfer and the integration of III-N epitaxial layers onto pre-patterned silicon substrates. The method can be extended to different substrates, rigid or flexible, thereby enhancing the versatility of device integration and advancing the field of III-N MEMS fabrication

The schematic representation of the pick-and-place process employed for the fabrication of self-standing III-N MEMS resonators is depicted in Fig. 1. The process starts with the liftoff of the III-N epilayers grown on the h-BN/sapphire template, by employing a $15 \times 15 \,\mathrm{mm}^2$ double-side water tape affixed to a stamp. The stamp is gently lowered to stick to the epilayer on the sapphire substrate [Fig. 1(a)]. During the pickup step, pressure is first applied on the stamp during 30 s to ensure the uniform adhesion of the adhesive tape to the epilayer, followed by the swift raise of the stamp at a speed of 1 mm/s [Fig. 1(b)]. Then the III-N layer is transferred onto a host substrate using capillary assembly where the drying of a water film at the interface of the III-N film and the substrate is used to create a large surface area to promote adhesion via van der Waals forces.³³ To this aim, after lifting the III-N layers with the stamp, a thin film of water is applied using a wetted microscope lens wipes [Fig. 1(c)]. The stamp is then positioned atop of a silicon substrate patterned with microcavities [Fig. 1(d)]. The placement process first entails lowering the stamp until the III-N thin film contacts the silicon surface. Then, a pressing force of 50 mN (equivalent to stamp weight of \sim 5 g) is exerted for 1 min to facilitate the wetted III-N film's adhesion to the silicon substrate

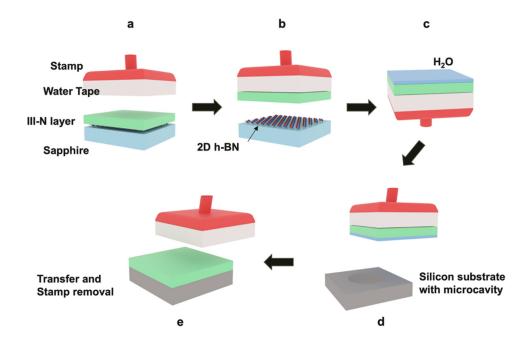


FIG. 1. Schematics of the proposed method for the fabrication of free-standing III-N MEMS on a host substrate by a liftoff and transfer process (where the III-N layer is GaN/AlGaN or BN). (a) and (b) Water tape is used to pick up the epilayer grown onto the h-BN/sapphire template. (c) and (d) The epilayer is transferred using pressure and capillary forces onto a silicon substrate with etched cavities and (e) the stamp is dissolved to obtain the self-standing III-N structure held to the silicon substrate by van der Waals forces.

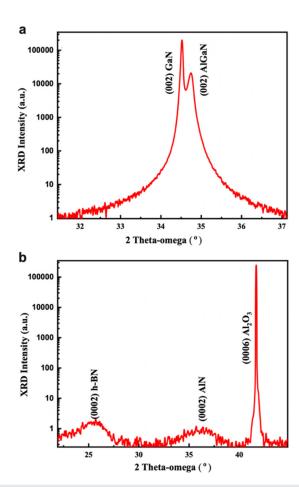


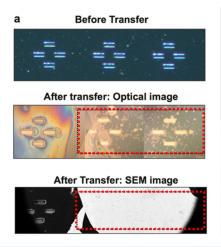
FIG. 2. High-resolution x-ray diffraction (HR-XRD) $2\theta-\omega$ scans of (a) the 300 nm GaN/300 nm AlGaN heterostructure on a 3 nm h-BN/sapphire wafer and (b) the 2.5 μ m thick h-BN epilayer on a sapphire wafer.

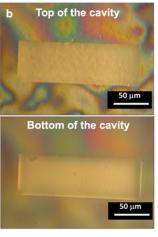
through capillary forces. Following placement, the stamp is removed, by dissolving the water tape upon immersion of the silicon sample along with the stamp in a water-filled beaker for 30 min [Fig. 1(e)]. This final step removes the stamp, leaving the III-N epilayer, held by van der Waals forces, on the silicon sample. The presence of the cavities in the silicon substrate results in the formation of self-standing structures.

Two sets of samples are used in this study: (1) a GaN (300 nm)/ Al_{0.14}Ga_{0.86}N (300 nm) heterostructure grown onto a 3 nm h-BN/ sapphire template and (2) a 2.5 μ m-thick h-BN epilayer grown on a sapphire substrate. These samples are grown on epi-ready sapphire substrates in a closed coupled showerhead (CCS 3×2 in.) reactor by a metal-organic chemical vapor deposition (MOCVD) technique. The growth conditions are described in great detail in previous works.^{31,36} Figure 2 shows the high-resolution x-ray diffraction (HR-XRD) 2θ – ω scans of the GaN/AlGaN heterostructure [Fig. 2(a)], and the $2.5 \mu m$ h-BN film grown on sapphire [Fig. 2(b)]. As evident in the figure, diffraction peaks for the GaN/AlGaN heterostructure are observed at 34.5° and 34.7°, corresponding to the (002) reflections of GaN and AlGaN layers, respectively. The HR-XRD 2θ - ω scan for the h-BN layer exhibits a discernible peak at 25.8°, which corresponds to the (0002) planes of the sp² BN. It is worth noting the presence of an AlN peak at 36.3° in the scan, which is attributed to the nitridation of the sapphire substrate during growth (utilizing NH3 flow) that preceded the h-BN growth.

The host silicon substrate with pre-patterned cavities is obtained by etching cavities of various geometries and sizes by deep reactive ion etching (DRIE). Standard 100 mm, P-type silicon wafers are used for this purpose, and the patterns are created using AZ 125NXT photoresist and an Alcatel AMS 4200 for dry etching of silicon. Among others, cavities of $180 \times 60~\mu\text{m}^2$ and $450 \times 150~\mu\text{m}^2$ rectangular shapes and $22~\mu\text{m}$ deep are obtained. After the etching process, the photoresist is removed using AZ100 remover followed by an oxygen plasma treatment (Tepla). The wafers are then cleaved into chips, of size $\sim 4 \times 1.5~\text{cm}^2$, onto which transfer of III-N layers is performed.

Figure 3 provides a summary of the transfer results of the GaN (300 nm)/AlGaN (300 nm) layers onto silicon substrates with microcavities. Optical and scanning electron microscopy (SEM) observations





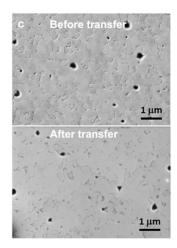


FIG. 3. (a) Silicon substrate with microcavities before and after transferring a GaN/AlGaN layer observed by optical microscopy and SEM (the location of the transferred film is indicated by the red dashed rectangle), (b) inside of a cavity covered by a GaN/AlGaN film observed by optical microscopy, and (c) surface morphology of the GaN surface before and after transfer observed by SEM.

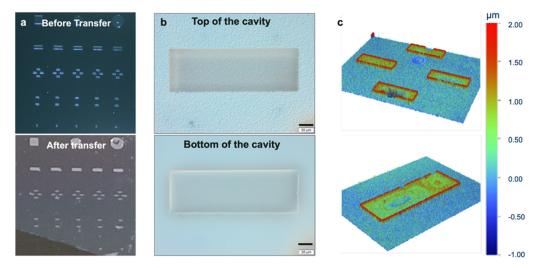


FIG. 4. (a) Optical images of the cavities before and after transfer of 2.5 μ m thick h-BN. (b) Observation of the cavity interior with optical microscopy by focusing on the top and bottom of the cavity (scale bar 20 μ m). (c) Optical profilometer images of the h-BN plates covering the cavities etched in the silicon wafer.

were employed to examine the transferred layer, revealing anticipated transparent and opaque surface under optical and SE microscopy, respectively [Fig. 3(a)]. Notably, the transparency of the layer under the optical microscope facilitates the visualization of the cavity's interior and bottom through the thin film overlay [Fig. 3(b)]. Importantly, Fig. 3(b) showcases a cavity free from any contaminants. This ensures that the intended function of the cavity, namely to provide free space for structural deformation of the transferred film, remains unchanged after the film transfer process. Additionally, SEM observations [Fig. 3(c)] of the transferred epilayers on the cavities show the absence of GaN morphology degradation following the transfer procedure.

The transfer process of the $2.5\,\mu m$ thick h-BN layer, which is identical to the process used to transfer GaN/AlGaN layers, was carried out on silicon microcavities, and results are shown in Fig. 4. The structures were further observed with an optical profilometer (Vecco NT9080), and the resulting images [Fig. 4(c)] indicate that the h-BN layer covering the cavity is flat and adhere uniformly to the silicon surface with clean edge clamping (the step observed at the edge of the cavities is an interface artifact of the profilometer).

Next, the mechanical characterization of the fabricated freestanding GaN/AlGaN and h-BN plates was conducted by performing dynamic studies of the corresponding mechanical resonators. To this

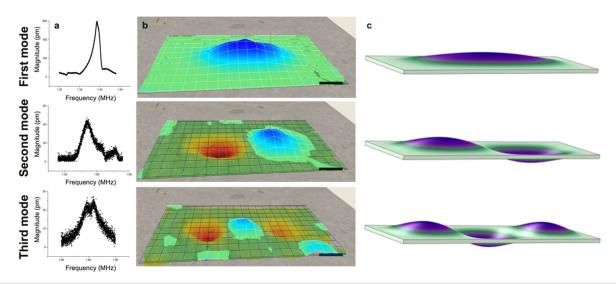


FIG. 5. (a) Resonance spectra of the first three modes of a rectangular AlGaN/GaN plate on silicon (where the amplitude of vibration is measured at the largest displacement location, which depends on the mode shape); (b) corresponding measured 3D displacement maps showing the maximum deflection amplitude of the first three modes (scale bars: 20

µm); and (c) COMSOL simulation of the three modes of vibration.

aim, the silicon samples were mounted on a PZT piezoelectric actuator used as an external mechanical excitation source. The displacement of the self-standing layers was mapped using a laser Doppler vibrometer (MSA-500 Micro System Analyzer from Polytec) upon actuation. The amplitude of vibration of the resonator was recorded while sweeping the excitation frequency.

Figure 5 shows the dynamic response of a 300 nm GaN/ 300 nm AlGaN heterostructure transferred onto a $22 \,\mu\mathrm{m}$ deep, $180 \times 60 \,\mu\mathrm{m}^2$ rectangular silicon cavity. The resonant frequency peaks of the first three consecutive flexural modes of resonance are displayed in Fig. 5(a). Peaks were clearly identified from the corresponding mode shapes [Fig. 5(b)], resulting in measured resonance frequencies of 1.39, 1.58, and 1.85 MHz. Analogously, the first resonance modes of $450 \times 150 \,\mu\mathrm{m}^2$ rectangular $2.5 \,\mu\mathrm{m}$ thick h-BN plates were characterized. Figure 6(a) shows the shapes of the first and the third observed modes with the corresponding displacement profiles at resonance along the length of the plate through its center [Fig. 6(b)].

The natural frequencies of a rectangular plate is given by³⁷

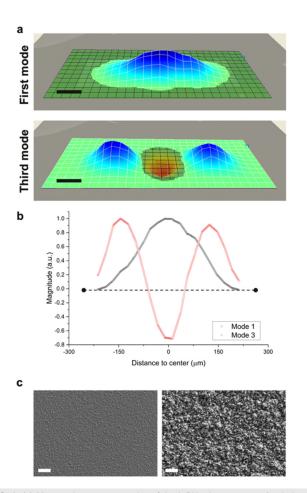


FIG. 6. (a) Measured resonance modes of the h-BN micro-resonator (scale bars: $50~\mu\text{m}$); (b) normalized deflection profile of the film at the line of maximum deflection; and (c) SEM image of the $2.5~\mu\text{m}$ thick h-BN epilayer grown on a sapphire wafer (scale bar: left = $10~\mu\text{m}$, right = $1~\mu\text{m}$).

$$f_{ij} = rac{\lambda_{ij}^2}{2\pi a^2} \left[rac{Eh^3}{12\gamma(1-
u^2)}
ight]^{rac{1}{2}},$$

where a and h are the plate length and thickness, respectively, i and j are the number of half-waves in mode shape along the length and width, E is the modulus of elasticity of the plate, ν is the Poisson's ratio, and γ is the mass per unit area. λ is a dimensionless frequency parameter that depends on the clamping conditions (boundary conditions applied at the edges of the plate), the plate length to width ratio, and the mode number. The mode sequence and the frequency ratio between a given mode and the fundamental mode are an indication of the boundary conditions (free, clamped, supported): in our case, we experimentally observed that the first three modes are the (11), (21), (31) modes with measured frequency and frequency ratio listed in Table I.

FEM simulations were performed using COMSOL to determine the eigenfrequencies and mode shapes of a bi-layer AlGaN/GaN thin film and a h-BN film suspended on a $180\times60\,\mu\text{m}^2$ and a $450\times150\,\mu\text{m}^2$ rectangular cavity, respectively. Values for the Poisson's ratio and the density for all plate materials were those found in the literature (with $\nu_{\text{AlGaN}}=0.2,^{38}_{\text{A}}$ $\nu_{\text{GaN}}=0.18,^{39}_{\text{A}}$ $\nu_{\text{BN}}=0.21,^{40}_{\text{AlGaN}}$ and the Young's modulus was used as an adjusting parameter. Simulations were conducted with fully clamped boundary conditions at the edge of the cavity according to the measured mode sequence and the frequency ratios. 37

Figure 5(c) shows the first three simulated modes of the AlGaN/GaN resonator that are in the same sequential order of the observed experimental modes. Table I summarizes the theoretical and experimental resonance frequencies of the first three modes along with the calculated frequency ratios of the n-mode over the fundamental mode (i.e., $\lambda_{ij}^2/\lambda_{11}^2$). It is important to note that using the typical Young's modulus values found in the literature (i.e., $E_{\rm GaN} = 261~\rm GPa$ and $E_{\rm AlGaN} = 285~\rm GPa)^{41}$ results in theoretical values of the resonance frequencies and frequency ratios close to the experimental ones.

The sequence of the modes obtained by simulation is also the same as the experimental ones for the h-BN plate. However, the Young's modulus value that leads to theoretical results (in terms of resonance frequency and frequency ratios) in agreement with experimental values is $E_{\rm BN} = 18\,{\rm GPa}$, which is far from the $\approx 0.8\,{\rm TPa}$ value consistently reported in the literature for the single-layer h-BN. 40 This later value, which is fairly constant for up to 8 h-BN sheets, has been estimated to be much lower for thicker layers. For instance, the Young's modulus of 292 nm thick pristine h-BN flakes exfoliated from their layered bulk was estimated at 248 GPa. 43 Also the modulus of \approx 100–120 nm thin films of BN synthetized by pulsed laser deposition was measured in the range of 120-180 GPa using nanoindentation. 44 However, these values are still far from the ones derived from our parametrical fit. Hence, we have used the FEM to take into account other parameters that could account for this large discrepancy in modulus, namely the presence of internal stress (since the h-BN layer is lifted from the substrate, it should be stress-free; however, additional stress could be induced during the transfer process) or modified clamping conditions that would influence the size of the plate. Simulations were conducted with $E_{\rm BN} = 120\,{\rm GPa}$, the lowest value found in the literature, 44 and either the internal stress or the plate size was adjusted to fit the resonant frequency of the first mode to the experimental value. Table I presents the corresponding results. A fit is

TABLE I. Experimental and simulated values of measured resonant frequencies.

	$\frac{\text{Al}_{0.14}\text{Ga}_{0.86}\text{N/GaN plate}}{300 \text{ nm/300 nm}}$ $180 \times 60 \ \mu\text{m}^2$				BN plate $2500\mathrm{nm}$ $450\times150\mu\mathrm{m}^2$								
Thickness													
Dimensions													
Resonance modes	Experir	nent	$E_{GaN} = 26$	FEM $E_{GaN} = 261 \text{ GPa}$ $E_{AlGaN} = 285 \text{ GPa}$		Experiment		$\begin{array}{c} FEM \\ E_{hBN} = 18 \text{ GPa} \end{array}$		$FEM \\ E_{hBN} = 120 \text{ GPa} \\ x \times y = 725 \times 241 \mu\text{m}^2$		$\begin{aligned} & \text{FEM} \\ & \text{E}_{\text{hBN}} = 120 \text{ GPa} \\ & \sigma_{11} = \sigma_{22} = -95.5 \text{ MPa} \end{aligned}$	
	Frequency (kHz)	Ratio	Frequency (kHz)	Ratio	Frequency (kHz)	Ratio	Frequency (kHz)	V Ratio	Frequency (kHz)	Ratio	Frequency (kHz)	Ratio	
Mode 1	1390		1248		355		354		355		355		
Mode 2	1580	1.14	1391	1.11			394	1.11	395	1.11	382	1.07	
Mode 3	1850	1.33	1654	1.32	476	1.34	469	1.32	469	1.32	531	1.49	
Mode 4					604	1.7	581	1.64	580	1.63	814	2.30	
Mode 5					782/812	2.20/2.28	731	2.06	730	2.05	1209	3.40	

obtained for a bi-axial compressive stress of \approx -95 MPa, which seems large when compared to the stress of, for instance, silicon dioxide thermally grown onto silicon that is generally of the order of a few hundred MPa. 45 In addition, and more importantly, the resonance frequencies of higher modes do not match. On the other hand, a good correspondence between simulated and experimental frequencies is obtained for $E_{\rm BN} = 120 \, \rm GPa$ and a plate size of $725 \times 241 \, \mu \rm m^2$, which is 1.6^2 times larger than the actual plate size. However, the profiles of the plate for the first and the third modes at maximum amplitude deflection [Fig. 6(b)] tend to indicate that the length of the plate corresponds to the size of the cavity. Hence, these hypotheses are not fully satisfactory, and we stipulate that the Young's modulus of our grown h-BN layer must be lower than what was previously reported. The presence of AlN (as illustrated from Fig. 2) in between the sapphire substrate and the BN layer should not influence the mechanical properties of the plate since it is very unlikely that the AlN would delaminate during the lift because it forms 3D epitaxial bonds with sapphire (which is much stronger than the inter h-BN layer van der Waals interactions). In fact, a low Young's modulus could be explained from the growth process leading to distinct structural properties of our BN. Indeed, SEM observations of the h-BN surface [Fig. 6(c)] indicate a granular surface morphology, which is owed to the high growth rate (470 nm/h), causing transition from layered to turbo static growth mode of h-BN layers.³ As a result, following the two-dimensional h-BN structure formed at the film/sapphire interface, the top h-BN layer is organized in short domains that is irregularly oriented, losing the long range order, which could explain our observations.

The passive resonators fabricated by the pick-and-place method introduced in this work have been shown to vibrate with well-defined modes of vibration and resonant frequencies in agreement with theoretical studies conducted using the FEM, at least in the case of AlGaN/GaN epilayer-based plate resonators. This simple method can be used in the determination of the mechanical properties of III-N layers, such as the h-BN thick film used in this study. Moreover, the fabrication of active MEMS integrating transduction schemes can be envisioned by

carefully controlling and optimizing each fabrication step after growth to avoid spontaneous delamination of the device that could arise from mechanical strains, as it has been demonstrated with the fabrication of HEMT devices on 2D h-BN. Most importantly, the adopted approach of pick-and-place of epilayers and their transfer to prepatterned cavities can also be extended to the fabrication of flexible GaN MEMS. Indeed, although we have tested the viability of this method on a silicon substrate, we foresee that it can be extended to flexible substrates, such as polyethylene terephthalate (PET) or Kapton. The realization of flexible III-N MEMS, which are difficult to manufacture through conventional micromachining processes, would open the door to a wide range of applications, from wearable devices for health monitoring to the internet of things, even if further technical developments are needed to reach the level of yield and device performance offered by conventional fabrication methods.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Rajat Gujrati: Investigation (equal); Visualization (equal); Writing – original draft (equal). Ali Kassem: Investigation (equal); Writing – original draft (equal). Cédric Ayela: Investigation (equal). Fabrice

Mathieu: Investigation (equal). Liviu Nicu: Investigation (equal); Writing – review & editing (equal). Suresh Sundaram: Investigation (equal). Isabelle Dufour: Writing – review & editing (equal). William Hunt: Writing – review & editing (equal). Abdallah Ougazzaden: Funding acquisition (supporting); Resources (equal). Jean-Paul Salvestrini: Conceptualization (equal); Funding acquisition (equal); Resources (equal). Thierry Leichlé: Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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