

Confined Growth of Few-Nanometer MoS₂ Nanoribbons with Optical Anisotropy in Insulating Nanotubes

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ABSTRACT

One-dimensional (1D) nanoribbons of transition metal dichalcogenides (TMDs) are predicted to exhibit exotic quantum phenomena that make them attractive for nanoscale electronic and spintronic devices. However, exploring the potential of ultranarrow TMD nanoribbons remain a challenge due to the lack of a suitable platform. Here, we report the atomically precise growth of ultranarrow MoS₂ nanoribbons within boron nitride nanotubes (BNNTs). The insulating nature of BNNTs enables direct optical probing of the encapsulated species without perturbing their intrinsic properties. Atomic-resolution transmission electron microscopy reveals the preferential growth of bilayers along the zigzag direction. Raman spectra confirm that the encapsulated structures experience significant strain. Angle-resolved polarized Raman spectroscopy reveals strong optical anisotropy in the 1D geometry, markedly distinct from the isotropic 2D MoS₂ sheets. Our approach offers an ideal platform for exploring intrinsic optical properties and device applications in ultranarrow TMD nanoribbons.

With the growing interest in 2D materials, considerable efforts have been made to develop their structural modifications. Graphene nanoribbons (GNRs)—narrow and straight 1D stripes of graphene—have been widely studied in materials science. Depending on their edge configuration and width, GNRs can exhibit tunable bandgaps and ferromagnetic behaviors absent in pristine graphene.^{1,2} In particular, the semiconducting properties of GNRs make them attractive for future applications in electronics. A wealth of discoveries in intriguing properties of GNRs has spurred experimental and theoretical interest in inorganic analogs, especially transition metal dichalcogenides, whose generalized formula is MX_2 (M : transition metal of group 4–10 and X : chalcogen). Theoretically, quantum confinement and edge effects in MX_2NRs can result in metal–insulator transition,³ tunable bandgaps,⁴ enhanced thermoelectric performance,⁵ and valley/spin-polarized current.^{6,7} However, the practical exploration of MX_2NRs is hindered by challenges in precisely controlling their structures. Conventional lithographic techniques^{8,9} and vapor–liquid–solid growth^{10,11} have difficulties in fabricating atomically precise few-nm-wide species, where intrinsic 1D features are expected to be more pronounced. In addition, these ultranarrow structures are highly air-sensitive, further complicating the experimental studies.

A rational approach to overcoming these challenges is to use a template that promotes and stabilizes the growth of MX_2NRs . Carbon nanotubes (CNTs) have been increasingly utilized as templates for isolating 1D materials.^{12–22} Spatial confinement at the nanoscale facilitates the growth of atomically thin 1D structures. Furthermore, the encapsulated species are chemically and thermally stabilized, enabling direct visualization by transmission electron microscopy (TEM). To date, a variety of few-nm-wide MX_2NRs have been synthesized via CNT-templated reactions, and their structures are well characterized.^{23–25} However, there remains a challenge in characterizing their physical properties, as the metallic and semiconducting nature of CNTs often hinder electrical

and optical measurements. In this context, boron nitride nanotubes (BNNTs)—structural analogs to CNTs—have recently emerged as promising alternatives due to their wide bandgap ($E_g \sim 5.5$ eV), which is largely independent of diameter and wall number.^{26,27} In contrast to CNTs ($E_g = 0\sim 1$ eV), insulating BNNTs hardly interfere with the electronic structures of guest species, thereby allowing access to their intrinsic physical properties (Figure 1a). Despite these advantages, the template synthesis of MX_2NRs within BNNTs has remained elusive. This difficulty is likely due to the higher chemical inertness of BN walls compared to carbon, which hinders the nucleation and growth of guest species.²⁸ Establishing a reliable technique for the synthesis of MX_2NRs within BNNTs would enable detailed investigations of their atomic structures and intrinsic properties—an essential step toward exploring their potential in device applications.^{29,30}

Here we report the efficient growth of few-nm-wide MoS_2NRs within BNNTs (Figure 1b). We have previously reported the confined assembly of Mo_4S_4 clusters inside BNNTs through vacuum annealing.³¹ Upon further annealing, the encapsulated Mo_4S_4 clusters can transform into MoS_2NRs with atomic precision. Atomic-resolution scanning TEM (STEM) imaging revealed that bilayer MoS_2NRs preferentially grow along the $\langle 100 \rangle$ direction within the confined 1D channels. Raman shifts analysis indicates that the encapsulated MoS_2NRs are subjected to significant strain. Angle-resolved polarized (ARP) Raman spectroscopy indicates that the encapsulated MoS_2NRs exhibit highly anisotropic optical responses, in contrast to the isotropic behavior of 2D MoS_2 sheets.

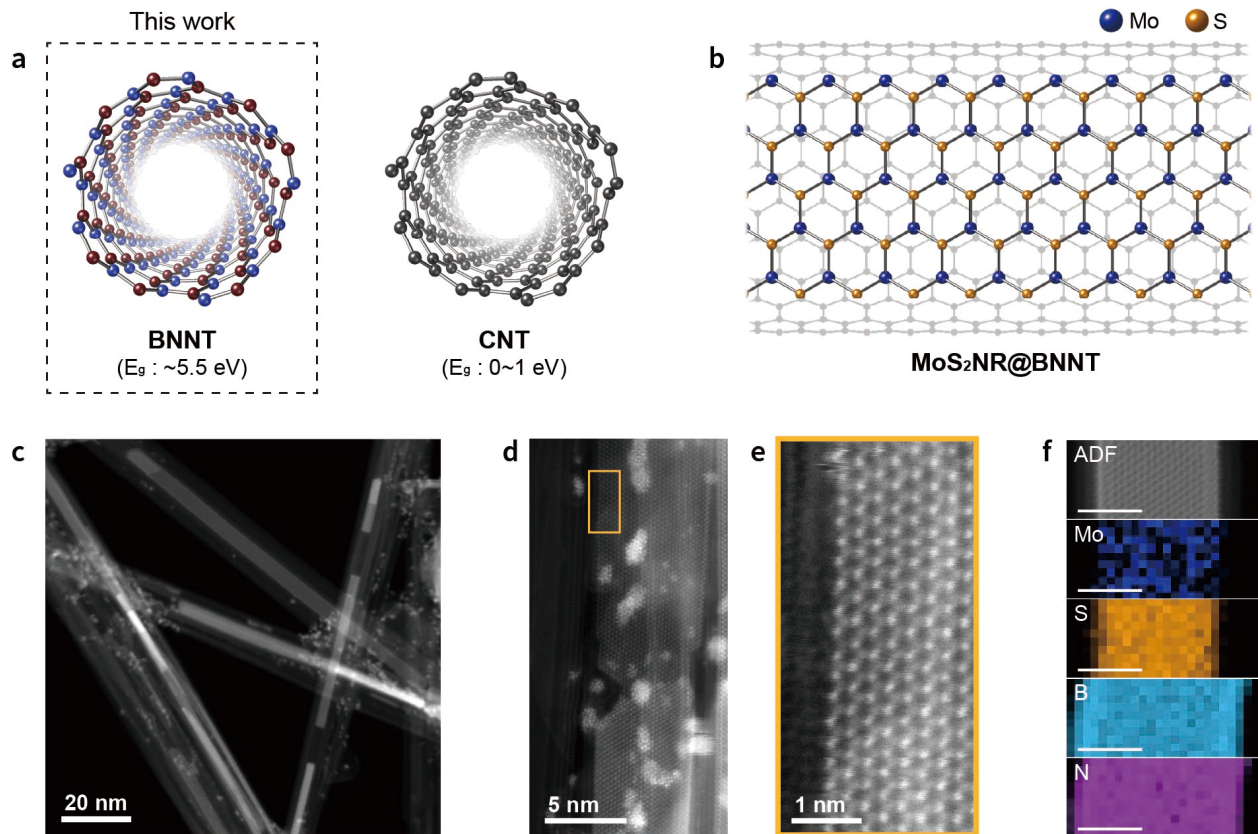


Figure 1. Template synthesis of MoS₂NRs inside BNNTs. (a) Scheme illustrations comparing BNNTs and CNTs as 1D nanoscale hosts for confined growth. (b) Atomic model of a monolayer MoS₂NR confined within a BNNT. (c) A HAADF image of the annealed BNNTs. (d, e) Magnified HAADF images of monolayer MoS₂NRs encapsulated inside BNNTs. (f) EELS elemental maps of an isolated MoS₂NR@BNNT, showing the spatial distribution of Mo, S, B, and N atoms. Scale bars: 5 nm.

In the present experiment, MoS₂NRs were fabricated on a two-step annealing process. To synthesize well-defined MoS₂NRs, Mo₄S₄ clusters were first encapsulated into BNNTs via a vapor-phase reaction. A stoichiometric mixture of MoCl₅ and S powders, along with open-ended BNNTs, were heated at 500 °C under vacuum. The obtained samples were observed using high-angle annular dark field (HAADF) STEM, confirming the encapsulation of Mo₄S₄ clusters bridged

by Cl cross-links (Figure S1). Upon subsequent higher-temperature annealing, the confined Mo₄S₄ clusters can be converted into MoS₂NRs. The formation of MoS₂ compounds was first confirmed using Raman spectroscopy (Figure S2): the Raman spectrum of the filled BNNTs show three characteristic peaks in the range of 400–500 cm⁻¹ due to the precursors, while the annealed BNNTs exhibit two new peaks at 381 cm⁻¹ and 404 cm⁻¹ absent before the annealing, which can be assigned to the E_{12g} and A_{1g} modes of 2H-MoS₂. Efficient growth of crystalline MoS₂NRs was achieved through annealing above 900 °C, whereas annealing at lower temperatures yielded defective NRs along with additional by-products (Figure S3).

A representative HAADF image of the annealed BNNTs is displayed in (Figure 1c, showing the formation of 1D structures along the axis of BNNTs with lengths on the several hundred nanometer scale. (Figure 1d shows a magnified HAADF image of an isolated 1D product, representing narrow and straight-edged stripes. Atomic-resolution STEM imaging of the products revealed that the configuration of the edges is distinctly different from the curved sidewalls of nanotubes (Figure 1e).^{32,33} The atomic-resolution HAADF image illustrates the hexagonal lattice with the spacing between lattice fringes is ~0.27 nm, corresponding to monolayer 2H-MoS₂. Elemental analyses of the products were performed by using electron energy-loss spectroscopy (EELS). The EELS maps of individual nanoribbons suggest that Mo and S atoms are confined within the inner space of BNNTs (Figure 1f), verifying the confined assembly of MoS₂NRs inside BNNTs.

Further HAADF imaging reveals that the encapsulated MoS₂NRs preferentially grow along a specific crystallographic direction. The crystal orientation of individual ribbons was investigated using fast Fourier transform (FFT) analysis. Figure 2a presents a representative HAADF image of

an encapsulated MoS₂NR and its corresponding FFT image. The FFT pattern displays a set of bright spots arranged in hexagonal symmetry, characteristic of the monolayer MoS₂ lattice. Notably, the FFT pattern in Figure 2a shows one of the hexagonal edges aligned with the ribbon axis ($\theta = 30^\circ$), indicating growth along the $\langle 100 \rangle$ direction. This alignment corresponds to zigzag edge termination, as illustrated in Figure 1e. Statistical analysis reveals that zigzag-edged configurations are predominant within BNNTs (Figure 2b), similar to the CNT-templated growth.^{23,34} This selectivity can be attributed to the relative stability of zigzag edges. According to early theoretical studies,³⁵ zigzag terminations are energetically more favorable than armchair and chiral ones. Importantly, zigzag MoS₂NRs are theoretically predicted to exhibit metallic behavior markedly distinct from semiconducting 2H- MoS₂ sheets.³

The confined growth within BNNTs enables the formation of ultranarrow MoS₂NRs. Figure 2c displays representative HAADF images of the encapsulated MoS₂NRs with successively smaller widths of 3.8 nm, 2.8 nm, and 1.5 nm. A statical analysis (Figure 2d) reveals an average width of approximately 3.8 nm, ranging from 1.4 nm to 8.9 nm. At these few-nm-wide widths, edge-induced 1D phenomena—such as spin polarization and bandgap modulation—are expected to become prominent.³

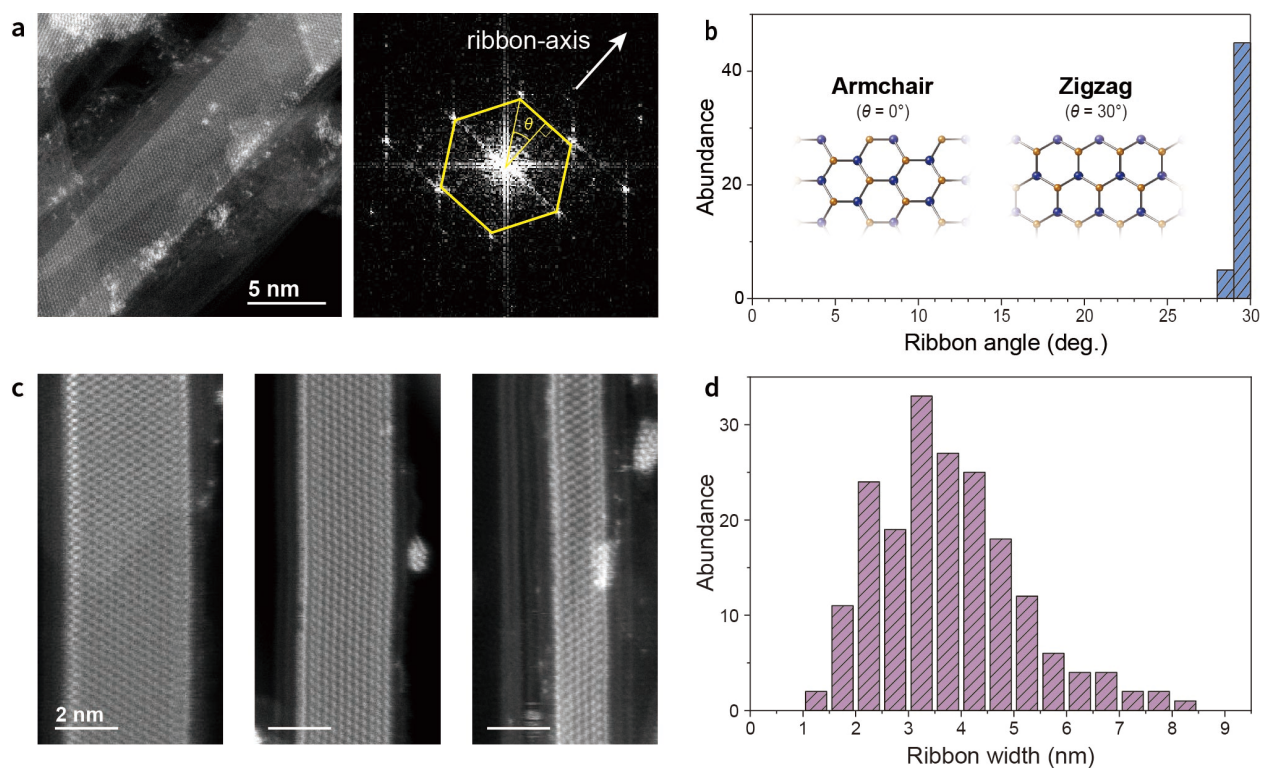


Figure 2. Structural characterization of MoS₂NRs confined within BNNTs. (a) Typical HAADF and corresponding FFT images of an isolated MoS₂NR, representing a zigzag-edged configuration with $\langle 100 \rangle$ alignment ($\theta = 30^\circ$). (b) Ribbon-angle distributions of 50 MoS₂NRs observed in the present experiment. (c) Magnified HAADF images of sub-4 nm-wide MoS₂NRs. (d) Width distribution of 190 MoS₂NRs confined within BNNTs.

A significant structural feature of the encapsulated MoS₂NRs is the preferential formation of bilayers. Depending on the inner diameter of host BNNTs, MoS₂NRs ranging from monolayer to multilayer structures—up to 6L—can be isolated (Figure S4a). Among them, bilayer configurations are significantly favored as shown in the statistical histogram (Figure S4b). The preferential formation of bilayer MoS₂NRs is likely governed by two factors: thermodynamic stabilization and spatial confinement. First, van der Waals interactions between MoS₂NRs and BNNTs contribute to the stabilization of the coaxial structure. Most of the observed BNNTs are slightly deformed

upon encapsulation (Figure S5), indicative of interfacial stabilization between the BNNT inner walls and the encapsulated MoS₂NRs. Similar behavior has been reported in CNT-templated growth.³⁶ Second, the geometry of the host cavities favors the formation of bilayers. The typical inner diameter of the host BNNTs is ~2 nm,^{32,33} while the interlayer spacing between BN and MoS₂ is ~0.5 nm, which matches well with van der Waals gap in vertically stacked *h*BN/MoS₂ heterostructures.³⁷ Within this spatial constraint, bilayer MoS₂NRs (~0.7 nm in thickness) are geometrically the most favorable configuration. As a result, the formation of trilayer or thicker MoS₂NRs is strongly suppressed under the present conditions.

Atomic-resolution imaging further reveals a variety of stacking patterns. Figure 3a and b illustrate two-view HAADF images of two representative stacking patterns: one with aligned hexagonal lattices and another with a lateral offset between layers. These configurations were verified through image simulations. Notably, the edge structures in some bilayers appear to deviate from ideal zigzag edges, likely due to edge folding or distortion. In this study, the term “nanoribbon” is used to also encompass flattened, closed-edged structures, owing to their similar flat cross-sectional geometries. Further atomic-level studies are warranted to elucidate the precise edge configurations.

Importantly, these bilayer nanoribbons are distinguishable from nanotubes. The intensity profiles in HAADF images show a uniform contrast across the cross-sectional direction, in contrast to the edge-enhanced profiles characteristic of tubular curvature (Figure S6). Moreover, twisting behavior was frequently observed during STEM observation. Both plan- and side-view configurations were simultaneously captured for an individual MoS₂NR (Figure S7), suggesting that the encapsulated species are twisting within BNNT cavities. Such twisting dynamics are incompatible with closed tubular structures and instead provide clear evidence for the formation

of mechanically flexible, flat ribbons. Similar dynamics have been reported for GNRs inside CNTs.³⁸

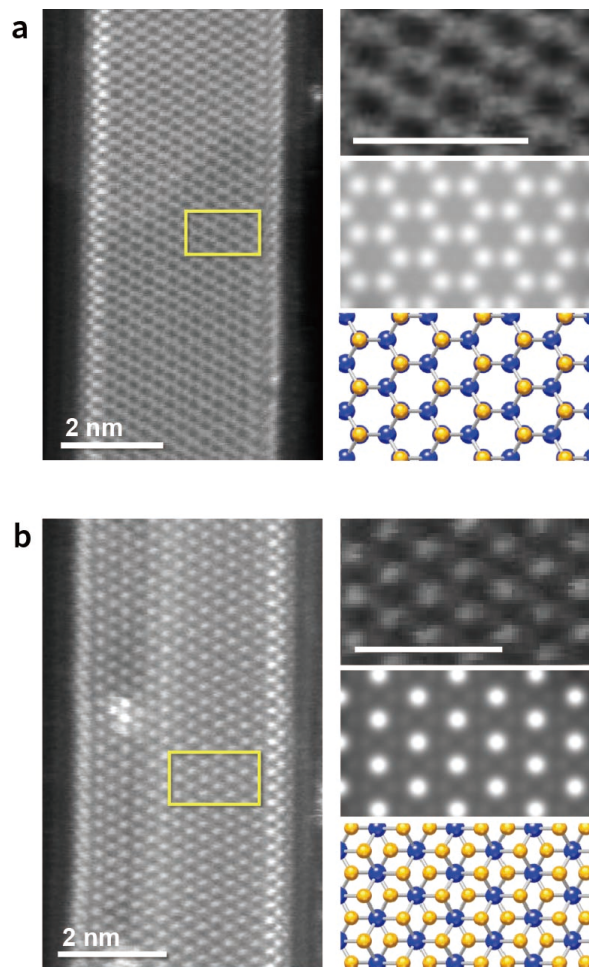


Figure 3. Atomic-resolution imaging of bilayer MoS₂NRs confined within BNNTs. (a, b) Top-view HAADF images of two representative bilayers stacking configurations. Each panel includes the corresponding atomic models and simulated images. The magnified images on the upper right correspond to the yellow boxed regions in the left-side images. Scale bars: 1 nm.

The encapsulated MoS₂NRs were further characterized using Raman spectroscopy. For the measurements, individual bundles were first suspended on TEM grids (Figure 4a), and their

encapsulation was confirmed via HAADF-STEM imaging (inset of Figure 4a). The clean surfaces of the bundles, with minimal impurities on the surfaces, facilitated reliable analysis of the vibrational properties of the encapsulated MoS₂NRs.

Detailed Raman studies on isolated bundles revealed the characteristics of the nanoribbons distinct from the 2D counterparts. Figure 4b displays Raman spectra of the confined MoS₂NRs compared to a reference MoS₂ bilayer grown by CVD. The CVD sample (blue) exhibits two characteristic peaks at 381 cm⁻¹ and 403 cm⁻¹, which can be attributed to the in-plane E_{1g} and the out-of-plane A_{1g} modes, respectively. In contrast, the encapsulated MoS₂NRs (red) exhibits two analogous peaks labeled E' and A', which are redshifted compared to the bilayer reference (Figure 4c). In particular, the in-plane E' peak shows a significant shift compared to the A' peak. These redshifts are likely due to the strain in the ribbons. According to early literature, the encapsulated MoS₂NRs are under strain up to 2%.³⁹⁻⁴² A likely origin is radial compressive stress exerted by the inner walls of BNNTs. Analogous to CNTs,^{43,44} the inner spaces of BNNTs can generate substantial local pressure, resulting in axial stretching of the confined ribbons. In addition, tensile strain may also arise from atomic rearrangements at the ribbon edges. Indeed, HAADF images of bilayers shows distinctive contrast at the edges (Figure 3b and c), suggesting local structural distortions in the encapsulated ribbons. Notably, given the extremely low abundance of irregular species (<1%), their contribution to the Raman response is negligible. Thus, the measured Raman signatures reflect the intrinsic vibrational properties of the encapsulated MoS₂NRs.

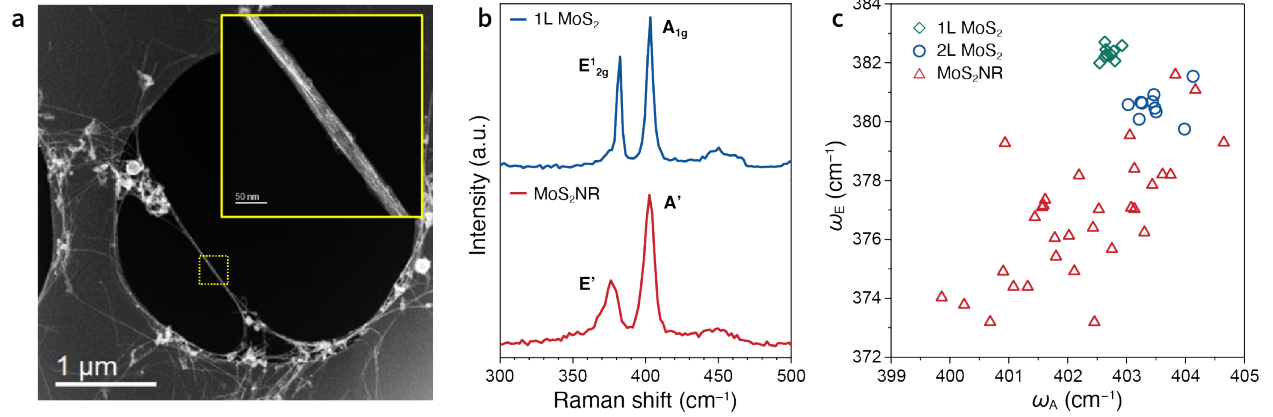


Figure 4. Raman characterization of suspended MoS₂@BNNTs. (a) HAADF image of a measured MoS₂@BNNT bundle. (b) Raman spectra of MoS₂NR@BNNTs (red) and CVD-grown MoS₂ bilayers (blue). (c) Correlation plot of two Raman modes for the suspended MoS₂@BNNT bundles. All the measurements for the MoS₂@BNNTs were performed when the polarization of the excitation laser was parallel to each bundle. In the correlation plot, ω_A and ω_E correspond to the A_{1g} and E_{12g} modes for layered MoS₂, and the A' and E' modes for MoS₂NRs, respectively.

To investigate the anisotropic vibrational properties of the encapsulated MoS₂NRs, we performed ARP Raman measurements on suspended MoS₂@BNNT bundles. The spectra reveal significant angular dependence, indicating strong optical anisotropy in the inherent 1D structures. Figure 5a and b show the ARP Raman spectra under co-polarized (VV) and cross-polarized (VH) configurations. Here, θ is defined as the angle between the ribbon axis and the laser polarization. In the present experiments, $\theta = 0^\circ$ corresponds to excitation polarized along the bundle direction. The polarization angle of the incident laser was changed by rotating the sample. In Figure 5a and b, the E' and A' modes vary periodically with the polarization angles, indicating anisotropic Raman scattering behavior.

Figure 5c–f presents the polar plots for the E' and A' modes under VV (purple) and VH (orange) configurations. The polar plots of each mode exhibit 2-lobed and 4-lobed patterns under the same configurations. The intensity maxima occur at $\theta = 0^\circ$ and 180° in the VV configuration, and at $\theta = 45^\circ, 135^\circ, 225^\circ$, and 315° in the VH configuration. These profiles can be properly fitted by functions $\propto \cos^4 \theta$ and $\cos^2 \theta \sin^2 \theta$, derived from the angle dependence of polarized Raman scattering in 1D systems.⁴⁵ The angular dependence observed in the present experiments closely resembles that of aligned CNTs.^{45–47} Such polarization-dependent behavior is markedly distinct from the isotropic Raman response of 2D MoS₂ sheets (Figure S8), and significantly exceeds the anisotropy observed in lithographically patterned samples,^{48,49} even those as narrow as 15 nm.⁵⁰ This enhanced anisotropy can be attributed to the narrow widths and highly aligned geometries of the encapsulated MoS₂NRs.

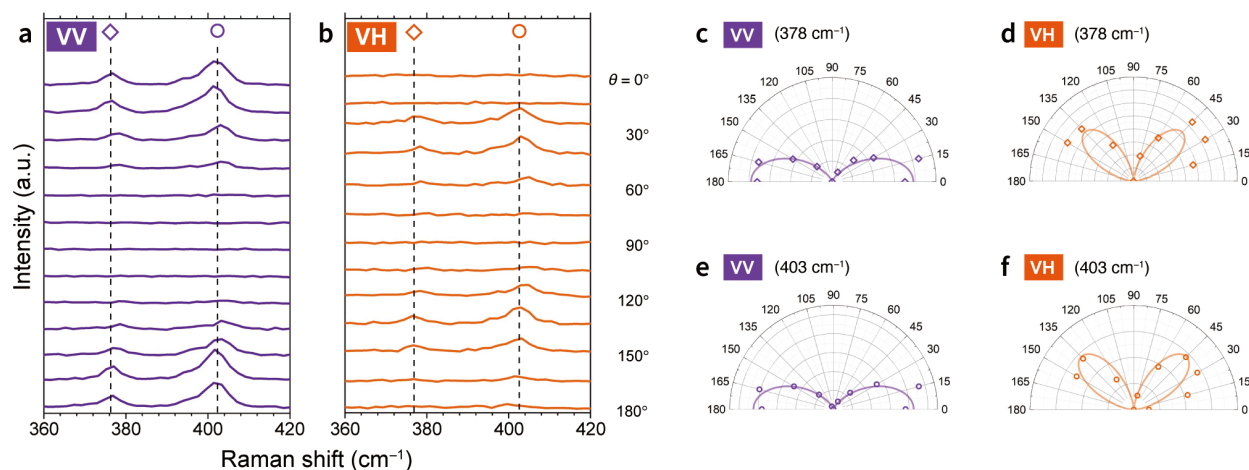


Figure 5. Anisotropic vibrational properties of MoS₂@BNNTs. (a, b) Polarized Raman spectra of a suspended MoS₂@BNNT bundle for (a) co-polarized (VV) and (b) cross-polarized (VH) configurations. (c–f) Polar plots of Raman intensity for 378 and 403 cm⁻¹ peaks (dashed lines with open diamond and circle in (a and b)) as a function of polarization angle θ under VV (purple) and

VH (orange) configurations. The purple and orange solid lines are the calculated curves for the Raman intensity under VV and VH configurations.

In summary, we demonstrate the atomically precise growth of ultranarrow MoS₂NRs inside BNNTs via thermal annealing of filled Mo₄S₄ clusters. The resulting MoS₂NRs are predominantly bilayers and selectively grown along the <100> direction. Raman spectroscopy revealed that the encapsulated MoS₂NRs are under strain up to 2%. ARP Raman scattering measurements revealed strongly anisotropic vibrational responses, in contrast to the isotropic behavior of 2D MoS₂ sheets. This anisotropy can be attributed to the directional confinement imposed by the insulating BNNT cavities. The present work represents the direct characterization of intrinsic optical properties in ultranarrow TMD NRs. Our template-based approach offers a general strategy for growing various 1D materials with enhanced anisotropy, paving the way for the discovery of novel quantum phenomena and their integration into nanoscale electronic devices.

Materials and Methods

Synthesis of MoS₂NRs inside BNNTs. Commercially available MoCl₅ (99.99%) and sulfur (99.998%) (purchased from Sigma Aldrich) were used as precursors. BNNTs were purchased from BNNT, LLC and pretreated as previously described.³¹ Briefly, the as-purchased BNNTs were heated in air at 800 °C for 90 min to open the tube ends, followed by annealing under vacuum at 1000 °C for 20 h to remove residual impurities. Typically, the open-ended BNNTs (~1 mg) were mixed with MoCl₅ (27.4 mg) and sulfur (6.4 mg) and sealed in a straight quartz ampule under vacuum (~10⁻⁷ Pa). The sealed ampoule was kept at 500 °C for 2 h and then cooled to room temperature.

The as-prepared BNNTs were sealed in a straight quartz ampule under vacuum ($\sim 10^{-7}$ Pa), and then heated to 600–1000 °C and kept for 12 h before being cooled to room temperature. The as-synthesized materials were dispersed in solution using a sonicator (Branson Sonifier SFX250) for 10 min and dropped onto TEM grids for STEM characterization and Raman probing, respectively.

STEM-EELS characterization. HAADF-STEM/EELS analyses were performed using a JEOL JEM-2100F-based electron microscope equipped with double JEOL Delta spherical aberration correctors and a Gatan Quantum EELS spectrometer operated at an electron accelerating voltage of 60 kV. STEM-EELS elemental maps of MoS₂NR were obtained based on L_{2,3} edge signal intensities of sulfur, M_{2,3} edge signal intensities of molybdenum, respectively. STEM image simulation was performed in Tempas simulation package, using imaging conditions similar to the experimental ones.

Optical probing. Raman spectra of the samples were taken at room temperature in a backscattering configuration using a micro-Raman setup (inVia, Renishaw) at 532 nm excitation. A 100× microscope objective (numerical aperture, NA = 0.85) was used to focus the laser beam and collect scattered light.

For the polarized measurements, the sample was set on a homemade manual rotation stage. A Glan-Taylor polarizer (GT10-A, THORLABS) was used to resolve the polarization of the scattered light. Raman spectra were collected every 15° with the same acquisition time.

Statistical analysis. All the data were collected from the HAADF images. The standard deviation of the data was presented in the form of “mean ± SD”. The statistical analyses were performed with OriginPro 2023.

Supporting Information. Additional structural characterization, such as STEM images and Raman spectra, as well as polarized Raman spectra of bilayer MoS₂ sheets for reference.

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Author Contributions

Y.N. conceived and designed the experiments. T.T., M.A., and Y.N. synthesized and characterized the materials. Y.S., R.S., and K.S. performed STEM-EELS analyses. T.T., Y.N., and Y.M. performed and analyzed Raman spectroscopy. Y.N. wrote the manuscript with feedback from the other authors. All the authors have approved the final version of the manuscript.

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Notes

The authors declare no competing financial interest.

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TOC graphic

