

Homochiral carbon nanotube van der Waals crystals

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5 For applications of single-walled carbon nanotubes (SWNTs) in integrated circuits, it is crucial to have high tube-density arrays of SWNTs that are well-aligned and purely semiconducting. We report on the direct growth of close-packed SWNT arrays on hexagonal boron nitride (hBN) substrates, demonstrating high alignment and uniform chirality within each array. Molecular dynamics simulations suggest a unique self-assembly growth mechanism resulted from the intertube van der Waals attraction and the ultralow sliding friction of SWNTs on the atomically flat hBN substrate. Field-effect transistors constructed from the grown SWNT array exhibit high performance at room temperature, with mobilities of up to 2000 square centimeters per volt per second, on/off ratios of $\sim 10^7$, and a maximum current density of ~ 6 milliamperes per micrometer.

The ultrahigh carrier mobility (1), excellent thermal conductivity (2), and nanoscale size of single-walled carbon nanotubes (SWNTs) make them promising candidates for next-generation electronic materials (3, 4). Field-effect transistors (FETs) based on semiconducting SWNTs have potential advantages over traditional silicon-based FETs such as faster operating speed, better energy efficiency, and higher integration density (5-8). Fabrication of high-performance SWNT-based microprocessors requires high-density, well-aligned arrays of pure semiconducting SWNTs to ensure device uniformity and to maximize current capacity and on/off ratio.

Controlled growth through chemical vapor deposition (CVD) (9-18) and the assembly of SWNTs with post-growth techniques (7, 19-26) have been used to produce SWNT arrays. However, several challenges remain in processing high-quality SWNT arrays, including the need of improving chirality selectivity during the CVD growth, effectively removal of surfactants and polymers after assembling SWNTs, and reducing bundling and disorder in alignment for both approaches (27). These imperfections in the raw materials have strongly limited the performance of SWNT-based integrated circuits.

Here we report on an approach for the direct growth of two-dimensional (2D) close-packed SWNT arrays on hexagonal boron nitride (hBN) substrate. Each SWNT array consists of SWNTs that have uniform chirality and are arranged precisely parallel to each other with a constant spacing of 0.33 nm, forming a crystalline structure. The van der Waals (vdW) interactions between SWNTs within this structure help drive the alignment and produce the uniform intertube spacing. Molecular dynamics (MD) simulations revealed that the growth proceeds through self-assembly facilitated by the atomically flat and very low friction hBN surface. Field-effect transistors (FETs) fabricated from the as-grown SWNT arrays exhibited superior electronic characteristics compared to those fabricated from previously reported SWNT arrays or films produced through other approaches (7-9, 11, 13, 14, 22-25, 28-36). We observed carrier mobilities of up to $\sim 2,000 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$, on/off ratios larger than 10^7 and limiting current transport capability greater than $6 \text{ mA}/\mu\text{m}$.

Growth and characterizations

The 2D SWNT array samples were grown through a catalytic CVD approach. Experimentally, catalytic iron nanoparticles were first deposited onto hBN flakes residing on a SiO_2/Si substrate. The system was then heated in a CVD tube furnace under a mixture of argon and hydrogen at atmospheric pressure. After reaching a growth temperature of about 850°C , methane was introduced as the carbon source to initiate the growth of SWNT arrays for a period lasting 60 mins. After growth was complete, the system was cooled down to room temperature under a protective gas of argon and hydrogen (See Methods and fig. S1).

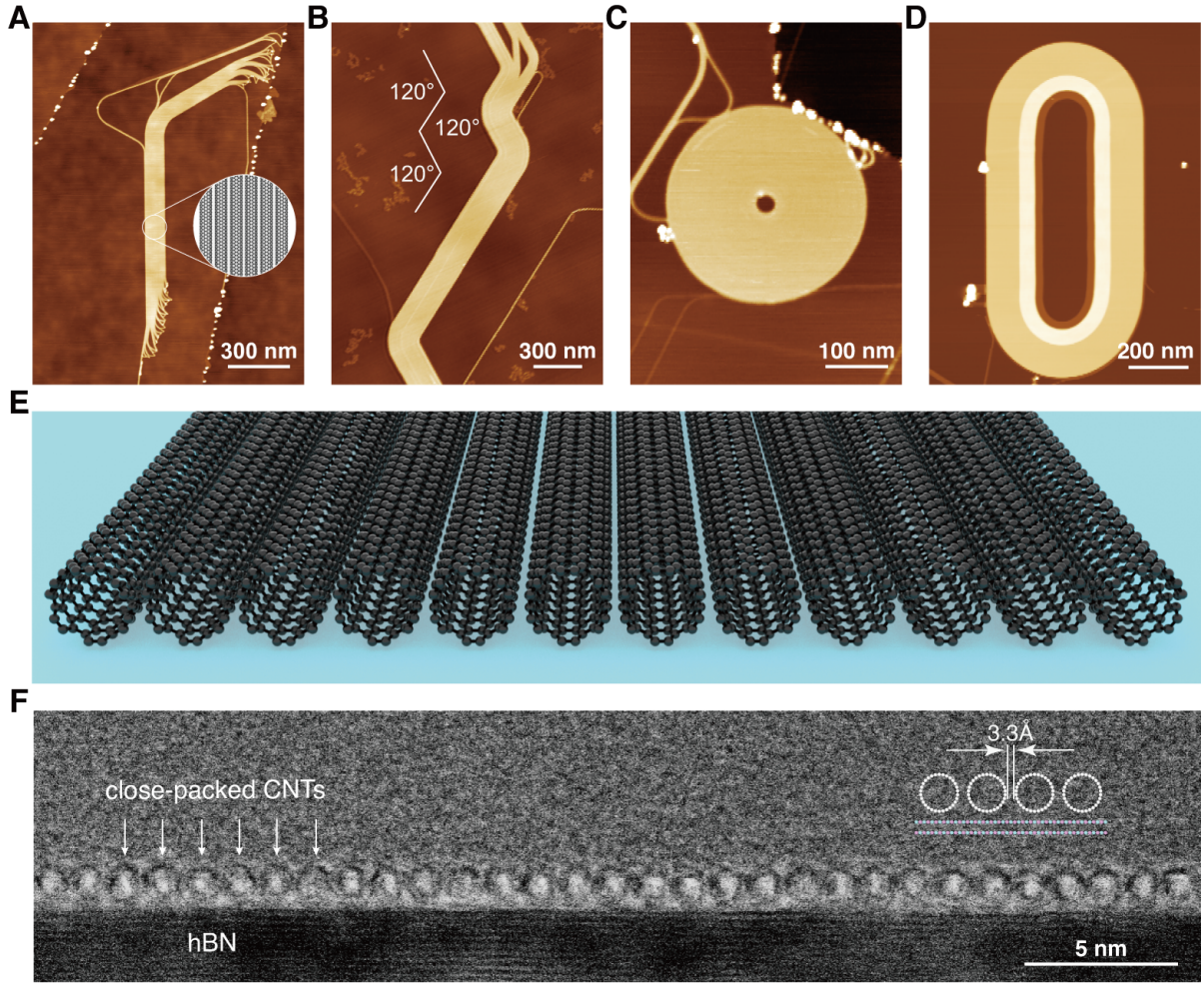


Fig. 1. Closely-packed carbon nanotube (SWNT) 2D array structures. (A and B) Atomic force microscopic (AFM) images of SWNT arrays. (C and D) AFM images of closed-loop shaped SWNT arrays, including disk shape (C) and running-track shape (D). (E) A schematic of the close-packed SWNT array. (F) Scanning transmission electron microscopy (STEM) cross-sectional image of a SWNT array on hBN substrate, where close-packed SWNTs with uniform spacing can be clearly seen. The intertube distance of ~ 3.3 Å is a result of inter-tube vdW interaction.

Such growth procedures typically produce random and isolated single-walled SWNTs. However, the introduction of atomically flat hBN substrates led to well-aligned SWNT arrays. We characterized the structure of as-grown 2D SWNT arrays using a combination of atomic force microscopy (AFM) and scanning transmission electron microscopy (STEM). AFM images in Fig. 1, A to D, depict a few representative SWNT arrays that were either the linear arrays (Fig. 1, A and B, also see fig. S2) or closed-loop arrays (Fig. 1, C and D, also see fig. S3).

The majority of linear arrays were several micrometers in length and a few hundred nanometers in width (see fig. S4), and they sometimes exhibited a bend at a fixed angle of 120° (Fig. 1B, figs. S2 and S5). The closed-loop arrays exhibited various shapes, including disk shapes (Fig. 1C), running-track shapes (Fig. 1D), triangle shapes (Fig. 3I), and some irregular circles (figs. S3 and S5).

Most SWNT arrays had a consistent thickness of 1~2 nanometers (roughly equivalent to the SWNT diameter, fig. S4), indicating stacking of a monolayer of SWNTs into the 2D arrays. The schematic microstructure of the 2D SWNT array is illustrated in Fig. 1E. The STEM image of a cross-sectional view of a SWNT array displayed in Fig. 1F revealed regularly arranged nanosized circles, each representing an individual SWNT. The intertube spacing was constantly measured to be 0.33 nm, equivalent to the interlayer spacing found in multilayer graphene, indicating that the SWNTs within the array were the most closely packed.

We further characterized the structure and chirality of the SWNT arrays. The SWNTs within each array exhibited uniform chirality. An AFM topography image in Fig. 2A displays a representative SWNT linear array, and a zoomed-in AFM image in Fig. 2B was obtained using super sharp AFM probes (see Methods, figs. S6 and S7). Parallel SWNTs within the array were well-aligned (with a constant intertube distance of 0.33 nm), and a line profile (black line) in Fig. 2C shows the uniform period of 1.53 nm across the SWNTs, indicating the same diameter and potentially the same chirality.

Polarized Raman spectroscopy was performed on this SWNT array, and a series of Raman spectra for various incident angles were depicted in Fig. 2D. Characteristic G- and 2D-peaks confirmed the presence of sp^2 carbon in this structure. The G- and 2D-peak intensities were maximum when the light was polarized along the array, and almost disappeared (decreased to ~2% of the maximum intensity) when the light was polarized perpendicular to the array. The polarization angle dependence of Raman G- and 2D-peak intensities in the inset of Fig. 2D displayed a two-fold rotational symmetry, agreeing well with the parallelly aligned SWNT structure. The Raman intensity in the perpendicular polarization direction was nearly zero, consistent with the high degree of alignment of the SWNTs inside the array (7, 35).

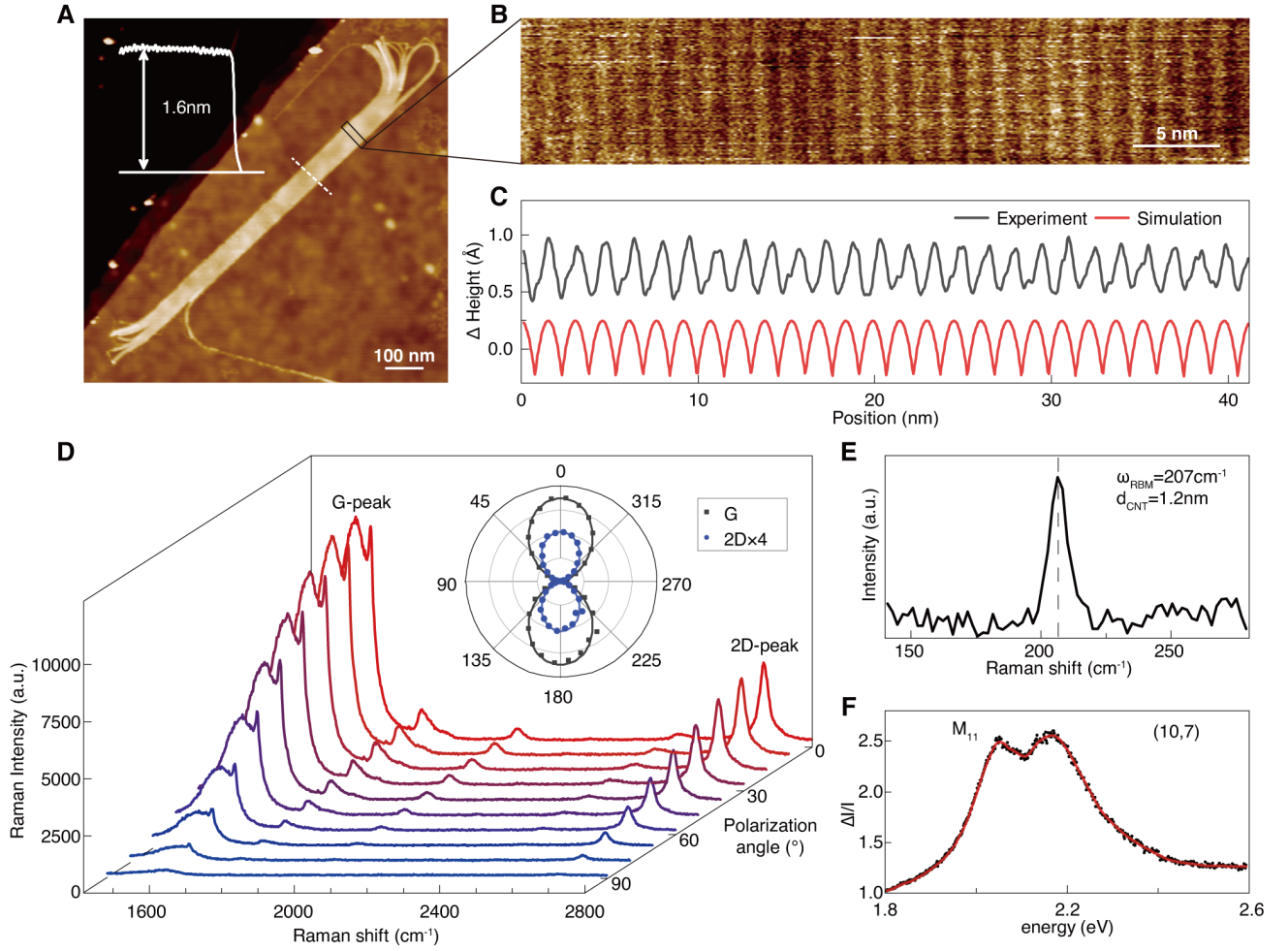


Fig. 2. Characterization of a homochiral SWNT vdW crystal. (A) AFM image of a typical SWNT array. Inset shows its height profile (about 1.6 nm) extracted from the white dashed line. (B) A zoom-in high-resolution AFM topography image of the SWNT array shown in (A), where closely-paced individual SWNTs can be readily identified. These SWNTs are well-aligned and parallel to each other, exhibiting a crystalline structure. (C) Experimental (black line) and simulated (red line) height profile of the SWNT array in (B), showing oscillations with a uniform period of ~ 1.53 nm. (D) Polarized Raman spectra of the corresponding sample at different angles between the SWNTs and the polarization direction of incident light. Inset displays the angle-dependent intensity of the Raman G- (black markers) and 2D- (blue markers) peaks in polar coordinates. When the polarization direction was perpendicular to the SWNTs, the Raman intensity was nearly zero, reflecting a near-perfect alignment of the SWNTs. (E) Raman radial breathing mode (RBM) of the same sample, a single RBM peak at 207 cm^{-1} , suggesting that all of the SWNTs share the same diameter. (F) Rayleigh spectrum of the same sample, indicating the chirality of the SWNTs is uniformly (10, 7).

Additionally, the radial breathing mode (RBM) of the same SWNT array was also observed. The SWNT array exhibited a single RBM mode at $\omega = 207\text{ cm}^{-1}$ (Fig. 2E), suggesting that all of the SWNTs in this SWNT array had a diameter of $d \approx 1.20\text{ nm}$, calculated through (37) $\omega = 254/d$.

To further determine the SWNT chirality, Rayleigh scattering spectroscopy was performed on the same

SWNT array (details in Methods and fig. S10). Rayleigh scattering spectrum in Fig. 2F revealed two resonant peaks at 2.05 eV and 2.17 eV, based on which a single chirality of (10, 7) can be derived (38). This demonstrates the homochiral nature of the SWNT arrays. The diameter of the (10, 7) SWNT was calculated as $d = \frac{a}{\pi} \sqrt{n^2 + m^2 + nm} \approx 1.2 \text{ nm}$, where $a = 0.246 \text{ nm}$ is the lattice constant of graphene, consistent with the Raman RBM-mode determined SWNT diameter.

Combining the SWNT diameter of 1.20 nm and the array period of 1.53 nm, we obtained an intertube wall-to-wall distance (39) of 3.3 Å. This value is near that of the interlayer spacing in crystalline multilayer graphene, and is determined by the vdW interactions, confirming the close packing nature of the SWNTs. Based on all of the above characterizations and analysis, we concluded that the SWNTs within an array exhibited uniform chirality, were parallel to each other, and were closely packed with an inter-tube vdW distance of 0.33 nm, leading to the designation of the as-grown SWNT arrays as SWNT vdW crystals.

Growth and formation mechanism

We investigated the growth and formation mechanism of the SWNT vdW crystals. We first considered a series of U-shaped turns at the terminals of the linear arrays (Fig. 3A), and each U-turn connecting neighboring SWNT sections within the array. This structure indicated that the SWNT array was created by repeatedly folding an individual long SWNT multiple times, rather than assembling different SWNTs together. Based on this folding structure, we propose the growth process illustrated in Fig. 3B. First, SWNTs grow from the catalysts through a base-growth model (40), in which the newly grown SWNT segment pushes the existing SWNT segment to slide forward on the hBN surface (Fig. 3B i). When encountering hBN steps or other obstacles, the moving SWNT bends and clings to the hBN step-edge (Fig. 3B ii). After clinging along the step-edge to a certain length, the sliding friction becomes large enough to force the SWNT to move in the opposite direction. The oppositely directed SWNT adheres to the previously grown nanotube through vdW attraction (Fig. 3B iii and iv). The SWNT repeats this process and bends back and forth several times, eventually forming a densely packed SWNT array grown from a single SWNT (Fig. 3B v). Coarse-grained (CG) MD simulations have reproduced the above growth process, a video of which can be found in movie S1.

Energetically, a folded SWNT array structure could be more favorable than a straight individual SWNT because of the reduction in intertube vdW potential energy (see Fig. 3C and fig. S12). However, folded SWNT array structures were not reported in previous studies of SWNT growth on other substrates (9, 11, 13, 41). Presumably, the atomically flat hBN substrates plays an important role by providing a very low friction surface for the SWNT moving freely to reach the energetically favorable folded structure. We speculate that the formation of the 2D SWNT array was facilitated by a combination of the vdW attraction between neighboring SWNT sections and the sliding of the SWNT on the hBN surface.

To validate this possibility, we conducted fully atomistic MD simulations (see Methods for details) to calculate the sliding friction and the vdW attraction between SWNT sections. Specifically, we calculated the sliding potential energy surface for a (20, 0) SWNT sliding on a flat hBN substrate (Fig. 3D), from which a maximal friction of about 2.02 pN/atom was extracted (see Methods for calculation details). Such low friction allowed the smooth sliding motion of SWNTs on hBN substrates. The vdW

potential between two parallel SWNT segments approaching each other was simulated and recorded in Fig. 3C, and the total potential energy exhibited multiple local minima as the separation distance varied. When the separation distance was $< 5 \text{ \AA}$, the sum of the sliding potential energy and vdW potential monotonically decreased, enabling SWNT sections to overcome friction and adhere to each other (inset of Fig. 3C). High-temperature MD simulations (see Methods and movie S2 for details) revealed that, at experimental temperatures ($\sim 1000 \text{ K}$), the pronounced atomic vibrations allowed SWNTs to overcome the sliding potential barrier, and facilitated the adhesion of SWNTs even with an initial separation distance of a few nanometers.

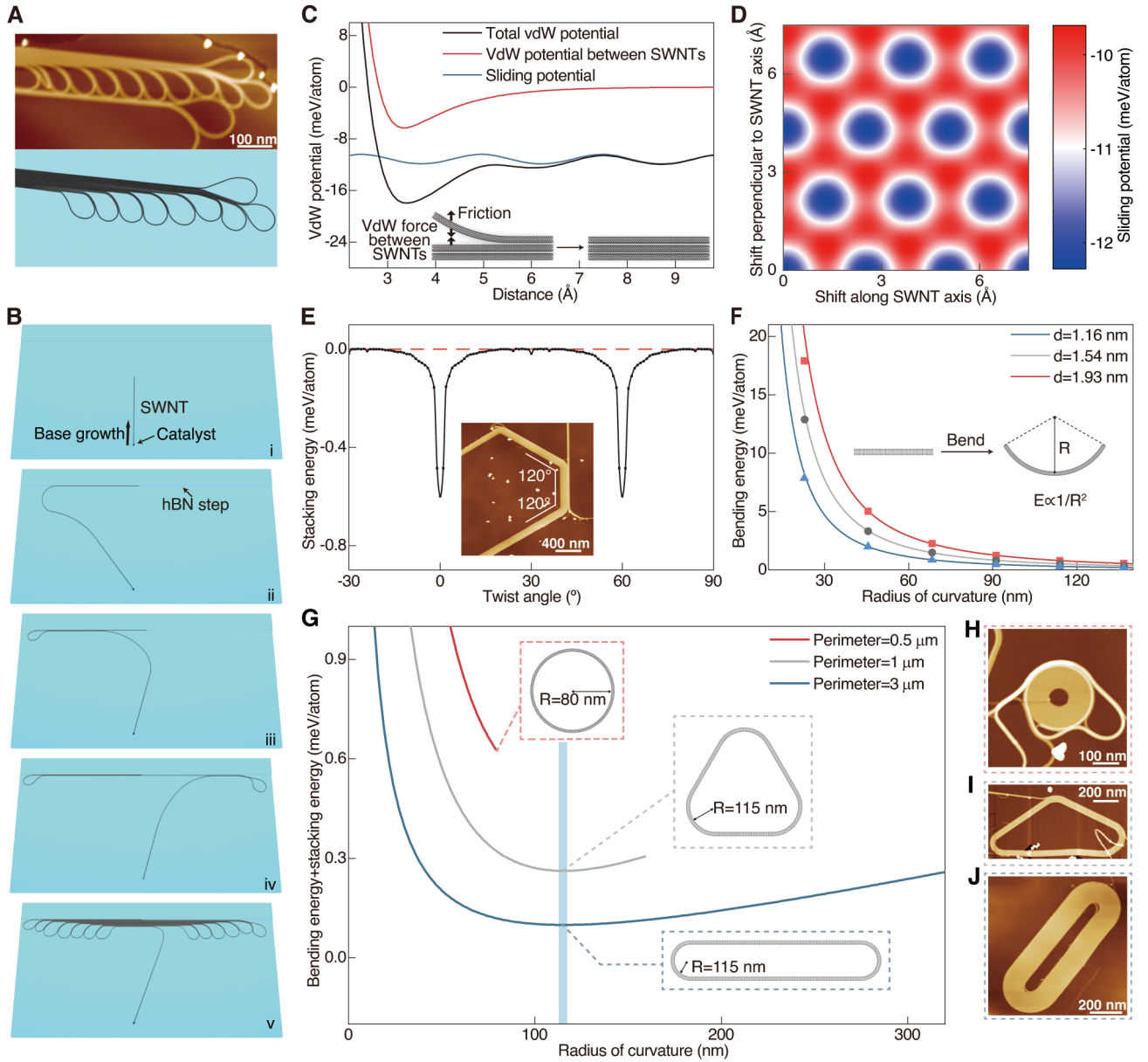


Fig. 3. Growth mechanism of SWNT vdW crystals. (A) AFM topography image (above) and illustration (below) of terminal structure of a representative SWNT array. (B) Schematic growth process of an open-linear SWNT array. (C) The vdW potential between SWNT segments (red line), sliding potential energy (blue line) and the sum of the sliding potential energy and vdW potential

between SWNT segments (black line). Inset, the schematic of neighboring SWNTs approaching together arising from the vdW attraction enabled by the low-friction hBN substrate. **(D)** Sliding potential energy of a SWNT on hBN substrate. The sliding potential energy line in (C) was extracted from the horizontal axis. **(E)** The stacking energy between a (20,0) SWNT and hBN substrate as a function of twist angle. Here the stacking energy of the misaligned configuration was set as zero. The inset AFM topography image showed selectivity in the stacking direction. **(F)** Dependence of SWNT bending energy on the bending curvature radius R . Bending energies of SWNTs with different diameters were all proportional to $1/R^2$. **(G)** Dependence of total energy of a closed-loop SWNT array with fixed perimeters on the curvature radius of the arc parts. The red line was shifted downward by 0.6 meV/atom, and the blue line is shifted upward by 0.4 meV/atom. The shaded area represents the optimal curvature radius. The insets showed schematics of structures with minimum energies. **(H to J)** AFM topography images of three close-loop structures, corresponding to the structures described in (G).

We analyzed the formation mechanism of the distinctive shapes of the SWNT arrays, which were closely related to their stacking and bending energies. The SWNT array shapes include polylines, triangles, rings, etc., as shown in Figs. 1 (A to D) and 3 (H to J). The majority of these SWNT arrays show directional selectivity with bending angles of 60° , 120° , or 180° , indicating that SWNTs preferred to align to specific directions of the hBN lattice with threefold rotational symmetry. These bending angles could be attributed to the symmetry in the stacking energy between a SWNT and the underlying hBN substrate, which was calculated by MD simulations (Fig. 3E). The stacking energy exhibited a pronounced 60° rotational symmetry toward the twist angle. Notably, the vdW stacking energy sharply decreased when the SWNT aligned with the hBN, signifying a substantial increase in the system stability at specific twist angles. This analysis could account for the formation of open polyline structures with specific bending angles.

Closed-loop SWNT arrays could be divided into linear and arc segments. The overall energy of the closed-loop array could be reduced by forming longer arc segments (increasing the curvature radius) to lower the bending energy, or by allowing longer linear segments to stack more effectively along the crystal orientation of the hBN substrate, thereby minimizing the vdW stacking energy. The final structure of the closed-loop SWNT array was governed by the competition between bending and stacking energies, with the actual curvature radius of the arc part determined by minimizing the total energy of the system (as shown in the simulation in Fig. 3G, and AFM images in Fig. 3, H to J).

Quantitatively, the bending energy of a SWNT was obtained through the classical beam theory as $DL_{\text{arc}}/2R^2$, which agreed well with our MD simulation results (Fig. 3F). Here, D represents the bending rigidity of the SWNT, R is the radius of curvature and L_{arc} is the length of bended arc segments. For a closed-loop SWNT with a fixed perimeter C , the stacking energy is $-\Delta\gamma R(C - L_{\text{arc}})$, where $\Delta\gamma$ is the stacking energy difference per unit length between misaligned and aligned SWNT/hBN configurations. By minimizing the total potential energy, the optimal radius of curvature of the arc segment was calculated as $\sqrt{D/2\Delta\gamma}$ (see Methods for detailed derivation). At sufficiently

large perimeter, the optimal radius was ~ 115 nm for a SWNT with a diameter of 1.54 nm (light blue shaded area in Fig. 3G), which aligned well with the experimental observations (Fig. 3, I to J). For the closed-loop SWNT arrays with tinier sizes, the maximum curvature radius may be even less than the

optimal radius, which would lead to a circular structure being the most energetically favorable (Fig. 3H). As a result, SWNT arrays with smaller perimeters tended to form a perfectly circular shape. A possible growth process of the closed-loop SWNT arrays can be found in movie S3.

5 High-performance SWNT-array FETs

We fabricated standard field-effect transistors (FETs) from our homochiral and highly aligned SWNT arrays (Fig. 4A). We deposited Pd/Au (or Cr/Au) electrodes on top of the two ends of a SWNT array to serve as the source and drain, and the silicon substrate with 285 nm oxide acted as the back gate to tune the Fermi level of the SWNT array system (see details in Methods). The transfer characteristics of a semiconducting SWNT array device are displayed in Fig. 4B, from which we derived on/off ratio near 10^7 . The carrier mobility extracted from the transfer curve reached $1910 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, as estimated through $\mu = \frac{dG}{dV_g} \times \frac{L}{WC_g}$, where G is the measured conductance of the SWNT array FET, L is the channel length, W is the SWNT array width, and C_g is the effective back gate capacitance per unit area.

The output curves were displayed in Fig. 4C, exhibiting a high on-state current approaching $1.2 \text{ mA}/\mu\text{m}$ under a bias $V_{sd} = 1.0 \text{ V}$. To further explore the current carrying capacity of the SWNT-array FET, we applied a large source/drain bias and measured the current. As shown in Fig. 4D, a SWNT-array could sustain a current of at least 6.5 mA per micrometer in width. This current density corresponded to a current of $\sim 10 \text{ }\mu\text{A}$ per SWNT, which was comparable to previously reported maximum values of current in individual SWNT devices (42, 43).

To evaluate the performance of our SWNT array FETs, we benchmarked our results to those previously reported in Fig. 4, E to G. Our SWNT array FETs exhibited high values of on/off ratio, carrier mobility, and SWNT density simultaneously (Fig. 4, E and F, red stars) compared to other SWNT-array candidates (7, 9, 11, 13, 14, 22-25, 28-36). Especially, the electronic features of our SWNT array even fell within the target zones (marked by the blue regions in Fig. 4, E and F, originally proposed in ref(27)). Notably, our SWNT-array devices showed a high on-state current of $1.2 \text{ mA}/\mu\text{m}$ at $V_{sd} = 1.0 \text{ V}$, despite having a relatively long channel length of 400 nm (Fig. 4G). This performance surpasses the target of $\sim 1 \text{ mA}/\mu\text{m}$ set for silicon FETs by the international roadmap for devices and systems (IRDS) (44). Additionally, the on-state current was higher than that of solution-assembled SWNT-array devices with comparable channel lengths (45), although it was lower than that of devices with shorter channel length ($\sim 50 \text{ nm}$) (Fig. 4G) (8, 33, 45-50). The exceptional electrical performances can be attributed to the extremely small inter-tube distance, precise alignment, and very high semiconducting purity. It is important to note that while uniform chirality was maintained within each SWNT array, there were still variations between different arrays. Achieving wafer-scale chiral uniformity remains a significant challenge.

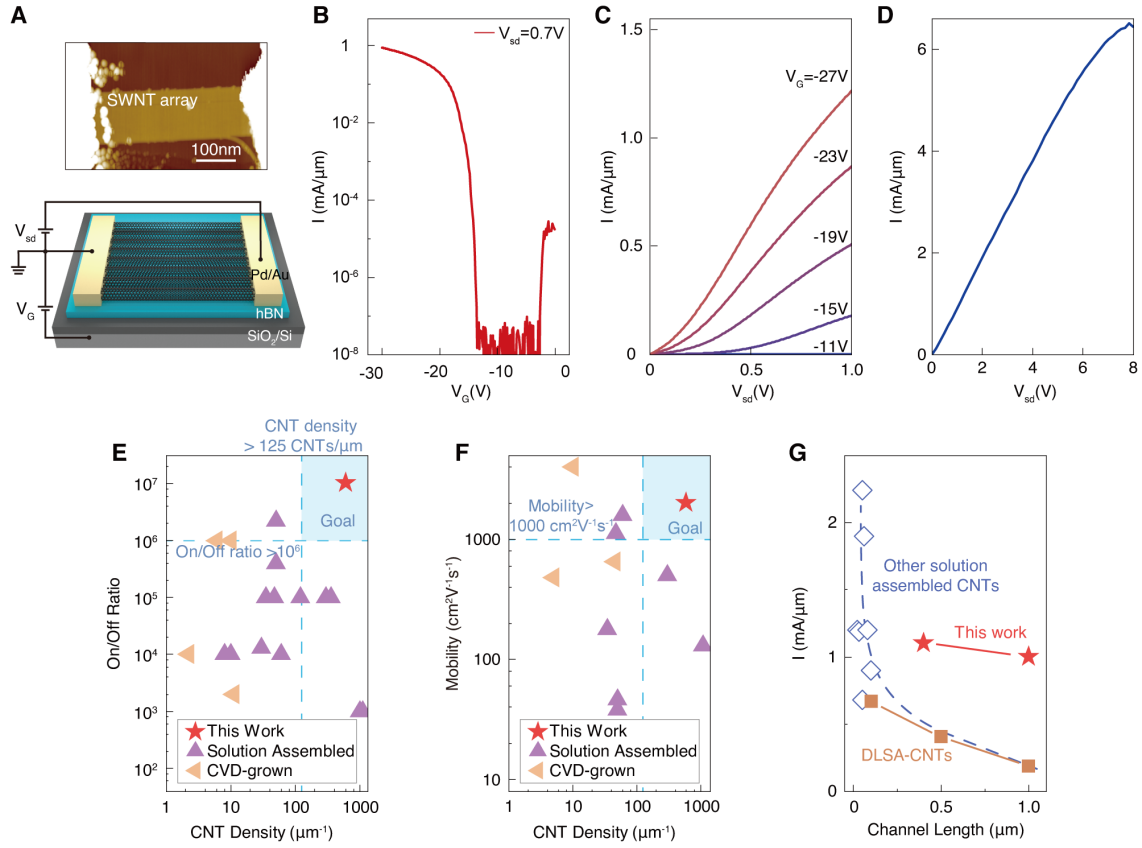


Fig. 4. Superb electrical performance of homochiral SWNT-array field-effect transistors (FETs).

(A) AFM topography and structural schematic of a SWNT-array FET. (B and C) Transfer (B) and output (C) characteristics of a 1-μm-channel-length semiconducting SWNT array device. (D) Saturation current of a 400-nm-channel-length SWNT array device under large source/drain bias. (E to G) Benchmarking of our SWNT array FET results and FETs from other literatures (7-9, 11, 13, 14, 22-25, 28-36, 45-50). Compared to the results found in existing reports, our homochiral SWNT FETs (marked by red stars) exhibited superb on/off ratio, carrier mobility, and current carrying capacity simultaneously (E to G). These brilliant merits stemmed from the excellent SWNT array structures with ultra-high density, high alignment, and uniform chirality. The target goals for the array density, on/off ratio, and mobility of ideal SWNT-array FETs (blue dashed lines in E and F) were originally proposed by ref(27), and blue regions in (E and F) marked the target zones with features better than the goals.

Discussion

We have reported the discovery of closely packed SWNT arrays with high chirality and alignment, and outstanding electrical performance. These results mark a step toward the practical use of SWNTs in nano-electronic devices and circuits. The growth mechanism presented here provides a new approach for producing intricate nanostructures, especially for developing new vdW materials.

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