DNA-directed nano-fabrication of high-performance carbon-nanotube field-effect transistors

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Abstract:

Biofabricated semiconductor arrays exhibit smaller channel pitches than existing lithographic feasibility. However, the metal ions within biolattices and the submicrometer dimensions of typical biotemplates result in both poor transport performance and small array uniformity. Using DNA-templated parallel carbon nanotube (CNT) arrays as model systems, we developed a rinsing-after-fixing approach to improve the key transport performance metrics by more than a factor of 10 folds over previous biotemplated field-effect transistors. We also used spatially confined placement of assembled CNT arrays within polymethyl methacrylate cavities to demonstrate centimeter-scale alignment. At the interface of high-performance electronics and biomolecular self-assembly, current approaches may enable scalable biotemplated electronics sensitive to local biological environments.

One Sentence Summary: High-performance transistors are constructed from biotemplates.

In projected high-performance energy-efficient field-effect transistors (FETs) (*1*, *2*), evenly-spaced small-pitch (spacing between two adjacent channels within individual FET) semiconductor channels are often required. Smaller channel pitch leads to higher integration density and on-state performance, but with the risk of enhanced destructive short-range screening and electrostatic interactions in low-dimensional semiconductors, such as carbon nanotubes (CNTs) (*3*); whereas evenly-spaced alignment minimizes the channel disorder that impacts the switching between on/off states (*4*). Therefore, although high-density CNT thin films exhibit on-state performance comparable with Si FETs (*5*, *6*), degraded gate modulation and increased subthreshold swing (*3*, *5*) are observed because of the disorder in the arrays.

Biomolecules such as DNAs (*7*, *8*) can be used to organize CNTs into prescribed arrays (*9*–*11*). Based on the spatially hindered integration of nanotube electronics (SHINE), biofabrication further scales the evenly-spaced channel pitch beyond lithographic feasibility (*12*). However, none of the biotemplated CNT FETs (*12*–*14*) have exhibited performance comparable with those constructed from lithography (*15*) or thin-film approaches (*3*, *5*, *6*, *16*–*18*). Meanwhile, during the surface placement of biotemplated materials, broad orientation distributions (*19*) prevent their large-scale alignment.

Here, we show that small regions of nanometer-precise biomolecular assemblies can be integrated into the large arrays of solid-state high-performance electronics. We used the parallel semiconducting CNT arrays assembled through SHINE as model systems (*12*). At the FET channel interface, we observed lower on-state performance induced by high-concentration DNA/metal ions. Using a rinsing-after-fixing approach, we eliminated the contamination without degrading CNT alignment. Based on the uniform inter-CNT pitch and clean channel interface, we constructed solid-state multichannel PMOS (p-channel metal-oxide-semiconductor) CNT FETs displaying high on-state performance and fast on/off switching simultaneously. Using lithography-defined polymethyl methacrylate (PMMA) cavities to spatially confine the placement of the CNT-decorated DNA templates, we demonstrated aligned arrays with prescribed geometries over a 0.35-cm^2 area substrate. Building high-performance ultra-scaled devices at the biology-electronics interface may enable diverse applications in the post-Si era, such as multiplexed biomolecular sensors (*20*) and 3D FETs, with nanometer-to-centimeter array scalability.

We assembled DNA-templated CNT arrays using DNA-based SHINE (*12*). We applied a rinsing-after-fixing approach (Fig. 1A) to remove DNA templates. Starting from the surface-deposited DNA-templated CNT arrays, both ends of the DNA-templated CNT arrays were first fixed onto Si wafer with deposited metal bars (first step in Fig. 1A). DNA templates and high-concentration metal salts (1 to 2 M) within DNA helices were gently removed through sequential rinsing with water and low-concentration H_2O_2 (second step in Fig. 1A and fig. S5). The inter-CNT pitch and the alignment quality of the assembled CNTs were not degraded during the rinsing (Fig. 1B, figs. S3 and S4) (*21*).

To explore the impact of single-stranded DNAs (ssDNAs) at channel interface, we first fabricated the source and drain electrodes onto the rinsed CNT arrays (Fig. 1C, left). Next, ssDNAs were introduced exclusively into the predefined channel area (first step in Fig. 1C, channel length ~200 nm). Finally, gate dielectric of HfO_2 and gate electrode of Pd were sequentially fabricated (second and third steps in Fig. 1C and fig. S6).

Out of 19 FETs we constructed, 63% (12 out of 19) showed typical gate modulation (on-state current density divided by off-state current density, I_{on}/I_{off}, exceeded 10^3, fig. S7). The other 7

devices exhibited I_{on}/I_{off} <5, which was caused by the presence of metallic CNTs within the array. At a drain-to-source bias (V_{ds}) of -0.5 V, one typical multichannel DNA-containing CNT FET (Fig. 1D) exhibited threshold voltage (V_{th}) around -2 V, I_{on} of 50 μA/μm (normalized to inter-CNT pitch) at gate-to-source bias (V_{gs}) of -3 V, subthreshold swing of 146 mV/decade, peak transconductance (g_m) of 23 μS/μm, and on-state conductance (G_{on}) of 0.10 mS/μm. Statistics over all the 12 operational FETs exhibited V_{th} distribution of -2 ± 0.10 V, I_{on} of 4 to 50 μA/μm, and subthreshold swing of 164 ± 44 mV/decade (fig. S7A). The transport performance was stable during repeated measurements (fig. S7C).

We annealed the above DNA-containing FETs at 400 °C for 30 min under vacuum to thermally decompose ssDNAs (*22*), and then recharacterized the transport performance. Compared to the unannealed samples, thermal annealing (Fig. 1D, figs. S7 and S16) slightly shifted the average V_{th} (around 0.35 V, V_{th} of -1.65 ± 0.17 V after annealing), and increased the average subthreshold swing by ~70 mV/decade (subthreshold swing of 230 ± 112 mV/decade after annealing). Other on-state performance, including g_m and G_{on}, as well as FET morphology, did not substantially change after annealing.

To build high-performance CNT FETs from biotemplates, we deposited a composite gate dielectric (Y_2O_3 and HfO_2) into the rinsed channel area, instead of introducing ssDNAs (Fig. 2, A and B, figs. S10 and S11) (*21*). Of all the FETs constructed, 54% (6 out of 11) showed gate modulation (fig. S12). The other 5 out of 11 FETs contained at least one metallic CNT within the channel (fig. S15). Using identical fabrication process, we also constructed another 9 operational single-channel DNA-free CNT FETs for comparing transport performance (fig. S8). The single-channel CNT FET (channel length ~200 nm) with the highest on-state performance exhibited on-state current of 10 μA/CNT (V_{ds} of -0.5 V) at the thermionic limit of subthreshold swing (that is, 60 mV/decade, Fig. 2C and fig. S9).

At V_{ds} of -0.5 V, the multichannel DNA-free CNT FET (channel length ~200 nm, inter-CNT pitch of 24 nm) with highest on-state performance (Fig. 2D and fig. S13) exhibited V_{th} of -0.26 V, I_{on} of 154 μA/μm (at V_{gs} of -1.5 V), and subthreshold swing of 100 mV/decade. The g_m and G_{on} values were 0.37 mS/μm and 0.31 mS/μm, respectively. The noise in the g_m-V_{gs} curves may originate from thermal noise, disorder and scattering within the composite gate construct. On-state current further increased to ~250 μA/μm, alongside with g_m of 0.45 mS/μm and subthreshold swing of 110 mV/decade, at V_{ds} of -0.8 V.

When the channel length scaled to 100 nm, we achieved I_{on} of 300 μA/μm (at V_{ds} of -0.5 V and V_{gs} of -1.5 V), and subthreshold swing of 160 mV/decade (fig. S14). Both the G_{on} and the g_m values were thus promoted to 0.6 mS/μm. The DNA-free CNT FETs exhibited comparable I_{on} to thin-film FETs from aligned chemical vapor deposition (CVD)-grown CNT arrays (*28, 29*), even at 60% smaller CNT density (~40 CNTs/μm vs. more than 100 CNTs/μm in (*28, 29*)). The effective removal of the contaminations, such as DNA and metal ions, and shorter channel length contributed to the high I_{on}. Notably, a previous study fixed CNTs directly with the source and drain electrodes (*13*). Because contamination could not be fully removed from the electrode contact areas, the on-state performance (g_m and G_{on}) decreased by a factor of 10.

At similar channel length and V_{ds} (-0.5 V), we benchmarked the transport performance (g_m and subthreshold swing) against conventional thin-film FETs using CVD-grown or polymer-wrapped CNTs (*3, 5, 16–18, 23–27*) (Fig. 2E, figs. S17 and S18). Both high on-state performance (g_m around 0.37 mS/μm) and fast on/off switching (subthreshold swing around 100 mV/decade) could

be simultaneously achieved within the same solid-state FET; whereas thin-film CNT FETs with similar subthreshold swing (~100 mV/decade) exhibited more than 50% smaller g_m.

Furthermore, the subthreshold swing difference between the multichannel (average value of 103 mV/decade) and the single-channel CNT FETs (average value of 86 mV/decade in fig. S9) was reduced to 17 mV/decade. Theoretical simulations suggest that, under identical gate constructs, uneven diameter of CNTs (*6*) and the alignment disorder (including crossing CNTs) (*5*) raise the subthreshold swing (*4*). We observed a wide diameter distribution of the DNA-wrapped CNTs in AFM images (fig. S2) and TEM images (fig. S1). Hence, the small subthreshold swing difference above indicated the effective gate modulation and evenly-spaced CNT alignment using SHINE (*12*), i.e. the absence of crossing/bundling CNTs within the channel area.

Statistics over all the operational multichannel DNA-free FETs exhibited V_{th} of -0.32 ± 0.27 V, I_{on} of 25 to 154 μA/μm (at V_{ds} of -0.5 V and V_{gs} of -1.5 V), and subthreshold swing of 103 ± 30 mV/decade. Different amounts of narrow CNTs (i.e. diameter < 1 nm) within FETs led to the wide distribution of I_{on}. Because the Schottky barrier and the band gap increase with narrower CNT diameter, lower CNT conductance is often observed than those with diameter above 1.4 nm (*30, 31*).

When comparing the transport performance differences between DNA-containing and DNA-free FETs (fig. S16), we observed largely negatively shifted V_{th} (-2V versus -0.32 V), higher drain-to-source current density (I_{ds}) at positive V_{gs} (mostly 10 to 200 nA/μm versus 0.1 to 10 nA/μm), and more than one order of magnitude smaller g_m (4 to 50 μS/μm versus 70 to 370 μS/μm). Thus, high-concentration ssDNAs and metal ions within multichannel FETs deteriorated the transport performance. Thermal annealing did not fully eliminate the impact because of the presence of insoluble annealing products, such as metal phosphates (*22*).

When CNT-decorated DNA templates were deposited onto a flat Si wafer, random orientations of DNA templates were formed through unconfined surface rotation. We solved this issue by using 3D polymeric cavities to confine the surface orientation during large-area placement. We first assembled fixed-width CNT arrays (fig. S19) (*21*) with prescribed inter-CNT pitch of 16 nm (2 CNTs per array). Next, in a typical 500 μm by 500 μm write-field on the PMMA-coated Si substrate (more than 20 write-fields on 0.35 cm^2 substrate), we fabricated densely-aligned crenellated parapet-like PMMA cavities (cavity density ~2×10^7 cavities/cm^2, fig. S20). The minimum and the maximum designed widths along *z* direction were 180 and 250 nm, respectively.

After DNA deposition and PMMA liftoff (Fig. 3A), >85% of the initial cavities (~600 cavities were counted) were occupied by DNA templates (Fig. 3B, fig. S21). The measured angular distribution, defined as the difference between the longitudinal axis of the DNA templates and the *x* direction of the substrate, was 56% within ± 1° and 90% within ± 7° (Fig. 3C), per scanning electron microscopy (SEM)-based counting of all of the remaining DNA templates within the 600 cavities sites. This value included improvable impacts from the fabrication defects of PMMA cavities sites, the variation during DNA placement, and any disturbance from PMMA liftoff. Notably, the angular distribution was still improved compared to previous large-scale placement of DNA-templated materials (*19*). CNTs were not visible under SEM, because they were embedded within the DNA trenches and shielded from the SEM detector by DNA helices.

Both the lengths of the DNA templates and the aspect ratio of the PMMA cavities affected the angular distribution. Longer DNA templates (length >1 μm) exhibited narrower angular distribution (0° ± 3.4° in Fig. 3D) than those of shorter DNA templates (length <500 nm, 1° ± 11°

in Fig. 3D). In addition, PMMA cavities with higher length-to-width aspect ratio (that is, 10 in in Fig. 3B and fig. S20) provided better orientation controllability than those with lower aspect ratio (that is, 1 in fig. S22). Hence, to further improve the angular distribution, longer DNA templates, as well as higher length-to-width aspect ratio of PMMA cavities, were beneficial. Because PMMA cavities were wider than the DNA templates, we observed up to 3 DNA templates, as well as the offset of DNA templates along the *x* and *z* directions, within a few PMMA cavities. Notably, DNA templates did not fully cover the PMMA cavities, even for a saturated DNA solution.

Two-dimensional hydrophilic surface patterns, with shape and dimensions identical to the DNA structures, could direct the orientation of the deposited DNA structures (*32*). However, it is difficult to design patterns adaptive to DNA templates with variable lengths. In contrast, effective spatial confinement relies mainly on the lengths of the DNA templates and the aspect ratio of PMMA cavities, and is applicable to irregular template lengths. Therefore, the anisotropic biotemplated CNT arrays could be aligned along the longitudinal direction of the cavities (Supplementary Sect. S4.1 and fig. S23) (*21*).

To further promote the on-state performance, scaling the inter-CNT pitch into sub-10 nm may be beneficial. However, at 2 nm inter-CNT pitch, the enhanced electrostatic interactions may impact the on/off switching. Therefore, the correlation between the inter-CNT pitch and performance metrics of CNT FETs needs to be verified. Combined with large-area fabrications through conventional lithography and directed assembly of block copolymers, biomolecular assembly could provide a high-resolution paradigm for programmable electronics over large area. The hybrid electronic-biological devices may also integrate electrical stimuli and biological input/outputs, producing ultra-scaled sensors or bioactuators.

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Supplementary Materials:

Materials and Methods

Supplementary Text

Figures S1-S23

References (*33*-*37*)

Fig. 1. Multichannel CNT FETs with ssDNAs at channel interface. (A) Design schematic for the rinsing-after-fixing approach. (B) Zoomed-in AFM image along the *x* and *z* projection direction for CNT arrays after template removal. The scale bar is 25 nm. See also figs. S3 and S4 in (*21*). (C) Design schematic for introducing ssDNAs at channel interface and FET fabrication. (D) The I_{ds}-V_{gs} curves (drain-to-source current density (I_{ds}) versus V_{gs} plotted in logarithmic at V_{ds} of -0.5 V) for multichannel DNA-containing CNT FET before (black line) and after (red line) thermal annealing. See also fig. S7.

Fig. 2. Constructing top-gated high-performance CNT FETs. (A) Design schematic for the fabrication of top-gated DNA-free FETs. (B) Zoomed-in SEM image along the *x* and *z* projection direction for the constructed multichannel CNT FET. Pink circle indicates the assembled CNT arrays. The scale bar is 100 nm. See also fig. S11 in (*21*). (C and D) The I_{ds}-V_{gs} curves (solid line, left axis, plotted in logarithmic scale) and g_m-V_{gs} curves (dotted line, right axis, plotted in linear scale) for single-channel (C) and multichannel (D) CNT FETs. Blue, red, and black colors in C and D represent V_{ds} of -0.8 V, -0.5 V, and -0.1 V, respectively. See also in figs. S9 and S12. (E) Benchmarking of current multichannel CNT FET in D with other reports of high-performance CNT FETs. Device performance from previous publications (*3*, *5*, *16* to *18*, *23* to *27*) are obtained at V_{ds} of -0.5 V and channel lengths ranging from 100 nm to 500 nm. See also in figs. S17 and S18.

Fig. 3. Centimeter-scale oriented placement of fixed-width arrays. (A) Design schematic for the oriented placement of the fixed-width CNT-decorated DNA templates on Si substrate. From left to right, fabricating cavities on a spin-coated PMMA layer, depositing CNT-decorated DNA templates onto the PMMA cavities, and liftoff to remove PMMA layer. (B) From left to right, zoomed-out and zoomed-in optical and SEM images of the aligned structures on Si wafer after PMMA liftoff. The scale bars in the bottom left, bottom middle, and bottom right are 10 μm, 1 μm, and 0.5 μm, respectively. The red rectangular circles indicate the selected areas for zoomed-in. The yellow arrows in the right panel indicate the aligned arrays. See also fig. S21 in (*21*). (C) The statistics of counts (left, red axis) and the cumulative percentage (right, green axis) for the aligned structures in (B) at each specific orientation. (D) Plot of angular distribution of the aligned arrays versus the lengths of the DNA templates.