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Herein, through a novel and effective catalyst design (Trojan-Mo catalysts), we successfully synthesize ultra-high density (as high as 160 tubes/\mu m) SWNTs with large area on sapphire surface.
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ABSTRACT
For realizing the full potential of single-walled carbon nanotubes (SWNTs) in realistic electronic devices, a scalable approach to obtain SWNT arrays with high density and large area is essential and still a great challenge. Herein, we reported an improved synthesis method for large area growth of ultra-high density SWNT arrays on sapphire surface by combining Trojan catalysts (release from substrate, assure ultra-high density) with Mo nanoparticles (loaded on surface, stabilize the released Trojan catalysts) as cooperating catalysts. Dense and perfectly aligned SWNTs covered the full growth substrate and the local density could be as high as 160 tubes/μm. Field-effect transistors (FETs) built on such arrays demonstrated the output current density up to -488 μA/μm at $V_{ds} = V_{gs} = -2\,V$, corresponding to the on-conductance per width of 244 μS/μm. These results confirm a vast potential application of Trojan-Mo catalysts in the structure-control growth of SWNTs.

1 Introduction
Horizontally aligned single-walled carbon nanotube (SWNT) arrays on the flat substrates are suitable candidates for the fabrication of future integrated circuits due to their superior electrical properties [1-3]. However, for sufficient current output and large-scale device integration, one of the key challenges is placing SWNTs at very tight (5-10 nm) pitch with well alignment to allow for density scaling and source/drain contact scaling, corresponding to the density of SWNT arrays must be 100-200
tubes/µm [4, 5]. Two kinds of techniques are developed for this goal. One is post-treatment approach, such as multiple transfer method [6, 7] and solution-based SWNT placement methods [8-12]. While these approaches significantly increase the SWNT density, the nanotube density even reach 500 tubes per micro [8] and there is no limit to the area of assembly, the tubes in post-treatment processes are always defective and in poor orientation which severely degrade the performance of SWNT devices. Another approach is the direct chemical vapor deposition (CVD) growth and the high quality SWNTs with perfect alignment on substrates can be obtained [13-15].

Over the past few years, CVD methods have made rapid progress in growth of dense SWNT arrays with perfect alignment on quartz or sapphire substrates [16-18]. In CVD system, the density and area of SWNT arrays largely depend on the activity and aggregation of catalysts [19, 20]. How to keep catalysts uniformly distributed over large area and restrain catalysts from aggregating and poisoning during SWNTs growth are always being pursued [21-23]. Many groups have provided a number of solutions, such as using monometallic catalysts (i.e., Fe, Co, and Cu), through sequentially patterning catalysts [23], periodic growth [24], and multiple-cycle growth [25] etc. Large area, high quality, and aligned SWNTs were successfully obtained on the quartz substrates. However, the density merely reached 10-60 tubes/µm, which was far away from the requirement for superior device performance. In these reported methods, the catalysts were exposed to the growth atmosphere all at once in the initial process of SWNTs growth, which caused the lateral diffusion and thus sintering together. Therefore, most catalysts were deactivated before catalyzing the growth of SWNTs. These deficiencies were the main reasons for the low density. Until very recently, our group developed a new method to prepare the catalysts, called Trojan catalysts. This method was effective for growing SWNT arrays on sapphire surface with high density, as high as 130 tubes/µm [26]. Briefly, the Fe catalysts are dissolved into the substrate and stored, and then they can be released gradually during SWNT growth. Note that all Trojan catalysts mentioned in this paper refer in particular to Fe nanoparticles dissolved into sapphire substrate, unless otherwise specified. The gradually released mode could reduce the interaction of Trojan catalysts and the ultra-high density SWNT arrays were obtained eventually. Even the dissolve-and-release process was able to prevent the aggregation of part Trojan catalysts and improved the efficiency of catalysts up to some extent, some released Trojan catalysts still tended to migrate and merge into large ones which restricted the area of high density SWNT arrays. The general results were in the range of several hundred-micron-widths, which severely limited their further application in large-scale device integration. Recently, we found the surface structure of substrate had a great influence on the growth of SWNTs. The terrace-step-kink sites were formed on the surface of substrate after an annealing process. These terrace-step-kink sites were along with unsaturated bonds or dangling bonds, providing more adsorption sites for the released Trojan catalyst. Thus, the released Trojan catalyst could be pinned on the sites, and then the high density SWNT arrays could be realized. Thus, inhibiting catalysts aggregation on flat substrates during CVD process is the key issue for obtaining large area SWNT arrays with ultra-high density.

It is well known that Fe–Mo combinative catalysts can promote the SWNT growth because Mo catalysts can stabilize active Fe nanoparticles in CVD system [27-29]. Herein, we developed a rational method that combining Trojan catalysts (release form substrate, assure ultra-high density) with Mo nanoparticles (loaded on surface, stabilize the released Trojan catalyst) as cooperating catalysts (we named the synergetic catalysts as Trojan-Mo catalysts) for growth of ultra-high density SWNT array with large area. Figure 1 schematically illustrates the procedure of the method. Firstly, the process of preparing Trojan catalysts was the same as reported previously [26]. Fe
catalysts underwent a necessary annealing process (at 1100 °C in air for 8 h) to dissolve into the a-plane sapphire substrate. Then the precursors of Mo catalysts were dispersed onto the substrate. Afterwards, sapphire substrates containing Trojan-Mo catalysts were placed into a CVD system for the growth of SWNT arrays (the details described in methods). When Fe were released gradually from the substrate and started to form Fe nanoparticles, part Mo nanoparticles had nucleated for the growth of SWNTs. Meanwhile, the migration and agglomeration of the released Fe nanoparticles were effectively suppressed by Mo catalysts as well as the nanotubes grown from them. The introduction of Mo catalysts on the surface can pin the released Trojan Catalyst and create more opportunities to nucleate SWNTs, which are beneficial to expand the growth area of SWNT arrays while retaining the ultra-high density.

2 Results and discussion

Utilizing Trojan-Mo catalysts, the full sapphire wafer (15 mm×30 mm, Figure 2a) were covered with ultra-high density SWNT arrays from scanning electron microscopy (SEM) observations (Figure 2b, 2c). The observed SWNTs were confirmed by Raman spectroscopy [30]. Figure 2d presented the typical Raman spectra with 514.5 nm excitation. Obvious radial breathing mode (RBM) peaks at low-frequency region and barely noticeable defect-induced D band at ~1350 cm⁻¹ indicated high quality SWNTs were synthesized on sapphire substrate. SEM and atomic force microscopy (AFM) are two most common used facilities for characterizing horizontal SWNT arrays [31-33]. Due to the electron beam-induced charge accumulation within the insulating sapphire substrate, the sapphire surface around SWNTs was imaged instead of SWNTs themselves. Thus the SWNTs appeared thicker in SEM images [34]. The width of an individual nanotube image was about 10 nm for 1 KeV electrons after sputtering gold observed in our SEM (Hitachi S4800 field emission). For ultra-high density SWNT arrays (ideal value > 100 tubes/μm) on sapphire, SWNT images overlap each other and form a continuous film under high magnification as shown in Figure 2c. Therefore, the density of the SWNT arrays was so high that we couldn’t accurately measure by SEM. But, SEM is still a fast and convenient method for the determination of the array area and uniformity in the scale of microns. Similar SEM images were found over the whole substrate (Figure S1 in the Electronic
Figure 2  Photo image (a) and SEM image (b, c) of the SWNT arrays using Trojan-Mo catalysts, with super high density and uniform dispersion on the wafer. d) Raman spectra of the as-grown SWNT with 514.5 nm excitation. (e) Typical AFM image of the as-grown SWNT arrays. (f) The corresponding diameter distribution of the SWNTs. The red solid line is Gaussian fitting peak. (g-i) HRTEM images of ultra-high density SWNT arrays transferred onto the ultrathin carbon membrane supported on a copper grid at low and high magnification, respectively. HRTEM image (i) at higher magnification confirm that the density of SWNT arrays is as high as 160 SWNTs/µm (each of 16 SWNTs in100nm length area has been marked in figure i).

Supplementary Material (ESM), confirming uniform density dispersion with large area coverage. Meanwhile, the distance between two SWNTs in the samples being smaller than the AFM tip (radius of curvature ~10 nm) led to large convolution effect and loss in the lateral resolution [35]. The SWNT arrays in different magnification AFM images (Figure S2 and Figure 2e) both approached a full monolayer. So, the AFM also suffered from the decline of lateral resolution power and tended to underestimate the array density. Relative to density, AFM height was reliably measurable. Figure 2f displayed a statistical analysis of the SWNT diameters from AFM measurements. The diameter distribution of most SWNTs was within 0.7-1.8 nm with an average diameter of 1.42 nm.

High-resolution transmission electron microscopy (HRTEM), as a higher resolution tool, is able to directly observe SWNTs at the atomic scale [36, 37]. Thus, it is competent to measure the density of nanotubes more accurately. For SWNT arrays on the substrate surface, the preparation of a HRTEM specimen is extremely difficult. The isolated SWNTs are very easy to form tube bundles or break during
the transfer process [25, 38]. Thus far, HRTEM images of horizontal SWNT arrays are rarely reported, let alone the ultra-high density SWNT arrays which are more challenging. In our former reported work, the ultra-high density SWNT arrays from Trojan catalysts were small area and usually grown at the edge of substrate which further frustrated the realization of transfer and other applications. Now, large area ultra-high density SWNT arrays from Trojan-Mo catalysts covered the full substrate were obtained. Through trial and error, we successfully transferred the ultra-high density SWNT arrays grown on sapphire surface to the ultrathin carbon membrane supported on a copper grid for HRTEM characterization (more details of the SWNT transfer process discussed in methods). Figure 2g-i were typical TEM images at different magnification. SWNTs transferred onto the ultrathin carbon membrane were well isolated and parallel arranged, maintaining the original morphology on sapphire. To distinguish the SWNTs from each other, we need a high magnification in HRTEM, which would decrease the observing area. From TEM observations at high magnification, the local tube density can be even as high as 160 tubes/µm (Figure 2i). The slight increase of density compared to our previous results (as high as 130 SWNTs/µm) at least proved that the larger area SWNT arrays synthesized by Trojan-Mo catalysts still kept the ultra-high density. Of course, it is still difficult to ensure larger area with well-distributed density as high as 160 tubes/µm and sub-micron uniformity at this point. Nevertheless, the result has been the highest reported

Figure 3 (a-c) AFM images of sapphire substrates loaded different catalysts (a: only Fe catalysts (without annealing), b: only Mo catalysts (without annealing), c: Trojan-Mo catalysts) after H₂ treatment at 830 °C for 5 min. (d) AFM images of sapphire substrates loaded Trojan-Mo catalysts after H₂ treatment at 830 °C for 30 min. AFM images (a-d) are the same Z-scale (-4nm~4nm). (e) The corresponding counts and average diameters of catalyst particles statistics in the areas (1µm×1µm). The error bars of counts and average diameters were standard deviations which calculated from 5 time statistic results. (f) XPS of Mo3d peaks at surface and in bulk (3 nm depth) of the sapphire substrate loaded Mo precursors after 830 °C in air for 30 min. Obviously, Mo catalysts didn’t dissolve into the sapphire substrate at the SWNT growth temperature.
density of grown SWNT arrays to date [26, 39] Since an obvious increase of the SWNT array area and preserving the ultra-high density have been achieved utilizing the improved synthesis method, it is

![Figure 4](image_url)

**Figure 4** (a) Schematic illustration of Trojan-Mo catalyzed the constant growth of SWNTs in the CVD process. Mo nanoparticles had nucleated for the growth of SWNTs in advance of Fe gradually released and formed Fe nanoparticles. (b-d) SEM images of SWNTs grown from different catalysts (b: only Trojan catalysts, c: only Mo catalysts (without annealing), d: Trojan-Mo catalysts) after SWNT growth 1 min. SEM images (e,f) and HRTEM image (g) of ultra-high density SWNT arrays over large areas using Trojan-Mo catalysts after SWNT growth 30 min.

necessary to further investigate the detailed mechanism. Many reported works about SWNT growth have shown that the catalyst-substrate interaction strongly influences the efficiency of catalysts, and the interaction is closely related to surface energies [35, 40]. Metal catalysts generally have higher surface energies than oxide substrates leading metal catalysts to dewet on oxide substrates [40]. As a result, metal catalysts are easy to migrate and accumulate on the surface of oxide substrates. Fe, with higher surface energy than Mo, is expected to dewet easier on sapphire surface [26].

This is supported by previous studies that metal species with more electropositive are more likely to wet the oxide layer (e.g., Mo instead of Fe) [41]. So, Fe and Mo catalysts show very different degrees of dewetting behaviors on sapphire, and Mo nanoparticles adhere better to a-plane sapphire than Fe nanoparticles which will restrain the aggregation of active catalysts during the growth of SWNTs [28, 29].

In order to confirm this speculation, Fe(OH)$_3$/ethanol solution and (NH$_4$)$_6$Mo$_7$O$_{24}$·4H$_2$O /water solution were dispersed on the surface of sapphire substrates...
by spin coating. Then the precursors were reduced by H₂ at 830 °C for 5 min. Figure 3a and Figure 3b show the AFM analysis of the surface of sapphire, large agglomerated particles were obviously observed for the Fe catalyst (Figure 3a), The average diameter of Fe nanoparticles was up to 7.36 nm (Figure 3e). The phenomenon could be a good proof that the migration and aggregation of the Fe catalysts on sapphire were severe. While, the sizes of most Mo catalysts were less than 2 nm (Figure 3e), which were suitable for the SWNT growth. This observation indicated a strong anchorage of the Mo nanoparticles onto the sapphire surface. Figure 3c and Figure 3d were AFM images of sapphire substrates loaded Trojan-Mo catalysts after H₂ treatment at 830 °C for 5 min and 30 min respectively. We can see that with the reduction time prolonging, the size distribution of catalyst particles did not change obviously, most catalyst sizes were still less 3 nm after H₂ reduction for 30 min (Figure 3e). This was attributed to Mo nanoparticles stabilized the released Fe Trojan catalysts [42, 43]. Thus, SWNT arrays with a higher SWNT density and larger growth area were obtained. In addition, the sapphire substrate loaded Mo precursors after 830 °C in air for 30 min were characterized by X-ray photoelectron spectroscopy (XPS). According to XPS of Mo 3d peaks (Figure 3f) at surface and in bulk (3 nm depth), it was found that Mo catalysts only stayed on the surface, didn’t dissolve into the sapphire substrate at the SWNT growth temperature.

On the basis of the above-mentioned analysis, we proposed that part of Mo nanoparticles prior nucleated for the growth of SWNTs at the early growth stage. With extension of growth time, Mo catalysts as well as the nanotubes grown from them suppressed the migration/ agglomeration of the released Trojan catalysts. The synergistic effect of Trojan-Mo catalysts resulted in expanding the growth area of ultra-high density SWNT arrays. The process was schematically illustrated in Figure 4a. To verify our hypothesis, some control experiments were performed. Firstly, we compared the growth results at the same CVD condition using different catalysts. Only using Trojan catalysts for SWNT growth 1 min, no SWNTs were found (Figure 4b). There were also no catalysts found on the surface (Figure S3a in the ESM). This result was in good agreement with our previous finding [26]. Trojan catalysts gradually released from the substrate needed certain time, but 1 min was not enough. However, the Mo catalysts, loaded on sapphire surface, didn’t undergo dissolving and releasing processes. They were able to nucleate and catalyze the growth of SWNTs in 1 min, which were analyzed from Figure 4c. Large area SWNT arrays with density of -1 tube/μm were obtained only using Mo catalysts. Similar results were observed from Trojan-Mo catalysts after 1 min growth (Figure 4d). The density of SWNTs increased with the growth time extension. Finally, horizontally aligned and large area uniform SWNT arrays (Figure 4e-f) with ultra-high density (Figure 4g) were obtained after growth of 30 min. In addition, only using Mo catalysts (without 1100 °C annealing) for SWNT growth 30min, the final density was about 10 tubes/μm (Figure S3b in the ESM). These results demonstrated Mo catalysts, on the one hand stabilized the released Trojan catalysts to expand the growth area, on the other hand made it possible to further increase the density of SWNT arrays by combining the advantages of Mo catalysts and Trojan catalysts.

The SWNT arrays synthesized by Trojan-Mo catalysts have much larger area while keeping the attractive high density, which are more suitable for fabrication of large-current electronic devices [25, 44]. Figure 5a was schematic illustration of SWNT FET with a self-aligned U-gate structure [45]. The as-grown SWNTs on sapphire were directly fabricated into top gate transistors. The gate stack was composed of YOx/Ti and source/drain electrodes were made by Ti (0.5 nm)/Pd (70 nm). Figure 5b showed the SEM image of top-gated FETs made from these SWNT arrays with gate length $L_g = 0.5 \, \mu m$, channel length $L_{ch} = 2 \, \mu m$ and channel width $W = 15 \, \mu m$. Compared to TEM for measuring the density of nanotubes, the SWNT transistors span a much wider width, and thus their on current density serves as a better indication of the large area tube density. Figure 5c and Figure 5d presented the representative transfer
and output characterization of the fabricated FETs.

![Figure 5](image)

(a) Schematic illustration of ultra-high density aligned SWNT field-effect transistor (FET) with a self-aligned U-gate structure. The horizontal dimension of the each ungated area is about 100 nm. (b) SEM image showing the core region of the FET devices fabricated on the as-grown SWNT arrays with S, D, and G electrodes. $W = 15 \mu m$, $L_{ch} = 2 \mu m$, $L_g = 0.5 \mu m$. (c) A typical transfer characteristic curve of SWNTs device with $V_{ds} = -1 V$. (d) Output characteristics of the high current density devices. $V_{tg}$ varies from 0 V to -2.0 V in steps of 0.5 V.

The largest on-state current could reach -488 $\mu A/\mu m$ at $V_{ds} = V_{gs} = -2 V$, even matched our previous best results of -467 $\mu A/\mu m$ at $V_{ds} = V_{gs} = -2 V$ with the similar channel length. The corresponding on-conductance per width was 244 $\mu S/\mu m$, which could be a fair parameter for comparison with the published data of drive-current. [8, 9, 25, 26] The remarkably high current density and on-conductance were strong evidences that the SWNT arrays grown from Trojan-Mo catalysts kept ultra-high density over larger area. These dense and perfectly aligned SWNTs with high current-carrying capability and large active area make them promising candidates for interconnects in electronic applications.

3 Conclusions

In summary, through a novel and effective catalyst design (Trojan-Mo catalysts), we successfully synthesize ultra-high density (as high as 160 tubes/\mu m) SWNTs with large area on sapphire surface. Detailed characterizations verify that Mo catalysts prior nucleate and suppress the agglomeration of Fe nanoparticles resulting in expanding the growth area of SWNT arrays while retaining ultra-high density. In addition, transistors based on the as-grown SWNTs exhibit high current density and on-conductance, showing the great potential for SWNT applications in future nanoelectronics. Trojan-Mo catalysts offer more
choices for the structure-control growth of SWNT arrays. Further studies and optimization of the CVD conditions are ongoing in our group for electrical properties and chirality selection with careful use of Trojan-Mo catalysts. Finally, this work provides better understanding of the mechanism of SWNTs growth on surface and might represent a step forward in large area synthesis of ultra-high density SWNT arrays on the substrate.

Experimental method

Synthesis of ultra-high density aligned SWNT arrays: Trojan catalysts were prepared according to reference [26]. Briefly, 0.05 mmol/L Fe(OH)3/ethanol solution dispersed onto clean α-plane sapphire substrates (15 mm×30 mm, bought from Hefei Kejing Materials Technology Co.) underwent a annealing process (1100 ℃ in air for 8 h) to dissolve into the substrate. Then 0.05 mmol/L (NH4)6Mo7O24·4H2O/water solution was loaded onto the substrate mentioned above by the spin-coating method and no annealing at 1100 ℃ again. Next, sapphires substrates containing Trojan-Mo catalysts were placed into a 1 inch tube and heated in air to 830 ℃. After the system was purged with 300 standard cubic centimeters per minute (sccm) argon, 100 sccm hydrogen and 50 sccm argon through an ethanol bubbler were introduced for the growth of ultra-high density SWNT arrays. After 30 min growth, the hydrogen was turned off and the furnace was cooled down to room temperature in argon atmosphere.

Transfer of ultra-high density aligned SWNT arrays onto ultrathin carbon membrane: PMMA solution (MW = 950K, 4wt% ) was spin-coated onto the sapphire substrate and then baked at 170 ℃ for 10 min to form a thin film that encapsulated SWNT arrays. This PMMA/SWNTs film was separated from the sapphire substrate in KOH aqueous solution (1 mol/L, 70 ℃). Before PMMA/SWNTs film was attached to the ultrathin carbon membrane supported on a copper grid, some ethanol solution was dropped onto the ultrathin carbon membrane to make it wet. Next, they dried off at 50 ℃ for 1h. This allows the tightly contact of the ultrathin carbon membrane with PMMA/SWNTs film. Finally, PMMA film was removed by decomposing at 320 ℃ in argon atmosphere for 2.5 h.

Characterization: The structures of the as-grown SWNTs and catalysts were studied with scanning electron microscopy (SEM, Hitachi S4800 field emission, Japan), High-resolution transmission electron microscopy (HRTEM, Tecnai F30, FEI), atomic force microscopy (AFM, Veeco NanoScope IIIA, Veeco Co.), a Raman spectrometer (Horiba HR800 Raman system), and XPS (ESCALab250, Thermo Scientific Corporation).

Fabrication and measurements of FET devices: Top-gated FETs based on the as-grown SWNT arrays were fabricated through a self-aligned U-gate process [45]. Electron beam lithography (EBL) was used to define gate and contact electrodes. The length of gate was 0.5 μm, the source and drain electrodes were deposited by Electron-Beam Evaporation (EBE) of Ti/Pd (0.5/70 nm), and then 10 nm Pd were deposited as self-aligned contact electrode. The dc characteristics of our devices were measured in air with a semiconductor analyzer (Keithley 4200 SCS).

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References


Electronic Supplementary Material

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Figure S1 SEM images of SWNT arrays grown on the full sapphire wafer (15 mm×30 mm) using Trojan-Mo catalysts, uniform density dispersion cover the whole wafer.
Figure S2 Larger area AFM image of SWNT arrays synthesis from Trojan-Mo catalysts.

Figure S3 (a) AFM images of the sapphire substrate loaded Trojan catalysts after H₂ treatment for 1 min. There were no catalysts on the surface. (b) SEM images of SWNT arrays with density ~10 tube/μm after growth 30 min using Mo catalysts (without annealing).

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