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14. ABSTRACT
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Fabrication of highly ordered anodic aluminium oxide templates on silicon substrates

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Abstract: The controlled fabrication of highly ordered anodic aluminium oxide (AAO) templates of unprecedented pore uniformity directly on Si, enabled by new advances on two fronts – direct and timed anodisation of a high-purity Al film of unprecedented thickness (50 μm) on Si, and anodising a thin but pre-textured Al film on Si, has been reported. To deposit high-quality and ultra-thick Al on a non-compliant substrate, a prerequisite for obtaining highly ordered pore arrays on Si by self-organisation while retaining a good adhesion, a specially designed process of e-beam evaporation followed by in situ annealing has been deployed. To obtain an AAO template with the same high degree of ordering and uniformity but from a thin Al film, which is not achievable by the self-organisation alone, pre-patterning of the thin Al surface by reactive ion etching using a free-standing AAO mask that was formed in a separate process was performed. The resultant AAO/Si template provides a good platform for integrated growth of nanotube, nanowire or nanodot arrays on Si. Template-assisted growth of carbon nanotubes (CNTs) directly on Si was demonstrated via a chemical vapour deposition method. By controllably removing the AAO barrier layer at the bottom of the pores and partially etching back the AAO top surface, new CNT/Si structures were obtained with potential applications in field emitters, sensors, oscillators and photodetectors.

1 Introduction

It has been known for some time that anodisation of aluminium can form highly ordered arrays of nanopores and the resultant anodised aluminium oxide (AAO) nanopore array could serve as a template [1, 2] for the growth of highly ordered arrays of nanowires [3–5] and carbon nanotubes (CNTs) [6–15]. Controllable variation of the anodisation conditions gives rise to the possibility of tailoring the size and density of the template pores over a wide range. In prior demonstrations, the highly ordered AAO templates were obtained by anodising free-standing Al sheets [3–11]. However, nanowire or nanotube arrays grown within the free-standing AAO/Al templates are limited in applications because they are not readily interfaced electronically with external circuits. Integrating such highly ordered arrays, for example, CNT arrays, onto Si substrates will broaden their applications and provide possibilities for incorporating CNT material properties or functionality with the Si electronics, as uniformly sized and equally spaced arrays of vertically aligned CNTs are desired in many potential applications, including field-emission, microelectronics, electrochemical probes, gene delivery, molecular interfaces and microfluidic devices. Although the formation of AAO in evaporated or sputtered Al film on a Si substrate has been shown possible [12–16], attempts to grow highly ordered CNT arrays in this structure have met with little success partly because of the need for and the difficulty in forming thick enough high-quality Al on silicon and the added challenge in anodising the thick Al without thermal-stress-caused detachment during anodisation. To date, as to our knowledge, there was no report on this front using Al films thicker than 20 μm. Recent successes in obtaining highly ordered AAO films on silicon were made possible by anodisation of nanoimprinted Al, either by mould pressing or lithographical methods [17–21], in which cases the Al film can be kept thin at the cost of relying on nanolithography to achieve the uniformity and ordering. However, those methods might be economically unfavourable or physically size-limited.

In this work, we show that highly ordered AAO templates can be grown on Si substrates with either thick or thin Al films using two complementary techniques: self-organised nanopore arrays formation by direct anodisation of aluminium on silicon and a second-order template-based anodisation process. The self-organised AAO template was produced by directly anodising a thick e-beam evaporated high-quality Al film on a Si substrate, with no wafer size limitation from basic principles. Here, we deployed a specially designed process of e-beam evaporation and optimised the deposition conditions for obtaining thick Al films with good adhesion to the Si substrate. The second-order templating utilises thin Al film on silicon, and the high degree of ordering is achieved by reactive ion etching (RIE) through a highly ordered nanopore array membrane formed by anodisation of a separate aluminium film and placed on the Al evaporated on silicon. Using the as-made templates for CNT growth,

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we extend the method developed for growing highly ordered and highly uniform arrays of isolated nanotubes in a free-standing aluminium plate [6] to one by which nanotube arrays of the same high quality can be formed on a silicon wafer.

2 Experimental

For depositing thick, high-quality Al film on silicon, we used a specially designed high-vacuum e-beam evaporator that is equipped with a custom-made substrate holder, featuring temperature control and axial rotation to allow in situ annealing with minimal oxidation and high film uniformity. The Si substrates used here are p-type Si (boron doping, ⟨1 0 0⟩ orientation, University Wafer, Inc.). The Al source is high-purity Al pellets (99.999%, Cerac, Inc.) loaded into four crucibles. For one continuous run, deposited Al film with thickness up to 50 μm can be obtained. In order to ensure good adhesion during the anodisation, a thin layer (~5 nm) of Ti between Al and Si was predeposited in the case of the thick Al films.

Anodisation of the evaporated Al film was carried out using a two-step method [22]. Before anodisation, the Al film was electrochemically polished in a mixture of $HClO_4$ and ethanol. It was followed by the first anodisation step at 40 V in a 0.3 M oxalic acid at 10 °C for several hours. After chemically removing the anodised Al in the first step in a mixture of H_3PO_4 and CrO_3, and obtaining a surface with an array of nanopores 'foot-print', a second anodisation was carried out under the same conditions resulting in the formation of an array of uniform nanopores seeded at the sites of the 'foot-print' from the first anodisation. At the end of the anodisation process, there is a barrier layer at the bottom of the pores which can be removed either by chemical wet etching using 0.5 M phosphoric acid or by dry-etching using chlorine-based gases.

For a second method of forming a highly ordered nanopore array in a thin Al film on silicon, the Al surface can be indented to form the equivalent of the 'foot-print' described above in a process of three steps. First, a free-standing AAO nanopore membrane with thickness of 300–500 nm was fabricated by anodising a high-purity aluminium sheet followed by chemically removing the Al substrate and the alumina barrier layer at the bottom of the membrane. Then the membrane was placed on the Si substrate covered in advance with a layer of Al film (typically 2 μm or less). In the third step, indenting of the Al surface was carried out by RIE using BCl_3 and Cl_2 gases at flow rates of 5 and 20 sccm, with pressure of 15 mTorr and power of 100 W, respectively. Anodisation of the RIE-indented Al film followed by barrier layer removal etching was performed as before.

The two methods described above are complementary in the sense that (1) the use of thick Al film permits direct formation of highly ordered pore array by self-organisation and there is no principal limitation to applying the method on any wafer size; (2) the AAO-mask-based method allows the use of thinner Al films for the formation of highly ordered nanopore arrays and it does not require a second anodisation by substituting the first anodisation step with a plasma etching process which is shorter in time and requires no Ti adhesion layer, a technological importance for electronic applications. This method is, however, limited by the size of AAO masks.

CNT growth within the arrayed nanopores was then carried out by the same chemical vapour deposition (CVD) method as first demonstrated in 1999 and refined over the years [6]. The CNT arrays grown by this method are normally flush with the surface of the alumina template and capped by a thin glassy carbon layer. Before the exposure of the CNTs out of the alumina template, dry-etching by either RIE or ion-milling was used to clean the surface and remove the amorphous carbon top layer. To keep the long exposed nanotubes from sticking together, we apply a wet-etching process, using a mixture of 6% H_3PO_4 and 1.8% CrO_3 with dispersant of polymethacrylic acid or Gum Arabic, which we developed for both exposing the nanotubes to a given length and keeping the extruded nanotubes from bundling into haystacks [23].

3 Results and discussion

To obtain high-quality thick Al films by evaporation on silicon, we experimented with various deposition conditions, including the substrate temperature (up to 627 °C), the evaporation rate and the deposition time which correlates directly with the thickness of the Al film. High-quality Al films with thickness of 50 μm on Si substrates were achieved by finely tuning the deposition conditions, as evidenced in the anodisation results presented below. Clearly, it benefited from (1) in situ annealing of the Al film, (2) rate-controlled cooling of the sample and (3) substrate rotation during deposition. The minimal oxidation of the Al films is guaranteed by the high vacuum level of the evaporator as well as the high purity of the Al source. Energy-dispersive X-ray, carried by the scanning electron microscope (SEM), analysis results showed that oxidation of the Al films was negligible. The experimental results show that both substrate temperature and its cooling rate after deposition have a major effect on the Al film qualities, such as morphology and adhesion, which were characterised by optical and scanning electronic microscopes and/or manifested in the anodisation results. High substrate temperatures will result in rough or island-like Al films, with the formation of grooves or cracks at temperatures above 500 °C, as shown in Fig. 1*a*. But low substrate temperatures and high cooling rates may result in poor adhesion of the Al film. This can be partly explained by the evolution of the mechanical stresses in the film-on-substrate system. In the case of low deposition temperatures, the intrinsic stress is the main contributing factor, which decreases as the deposition temperature increases. In contrast, in the case of the high deposition temperature, the thermoelastic stress predominates. Therefore although the signs of these stresses are the same, there is an intermediate deposition temperature for which the whole stress is minimised. From our experimental

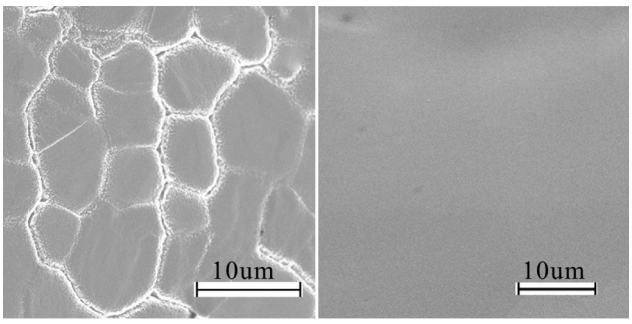

Fig. 1 *SEM images demonstrating the effect of substrate temperature on morphology of Al films (48 μm) deposited by e-beam evaporation*

a 627 °C
b 327 °C

results, such temperature lies in the range 200–400 °C. Fig. 1*b* illustrates the uniformity of an Al film deposited on Si substrates at substrate temperature of 327 °C. Our experimental results show that the use of a Ti adhesion layer prevented the pealing-off of the Al film from the Si surface during anodisation, especially in the case of thick Al films. This is likely because of the difference among the thermal expansion coefficients of Al, Ti and Si (with values of 25×10^{-6}, 8.5×10^{-6} and 3×10^{-6} °C^{-1}, respectively) and the fact that Ti is chemically less active than Al during anodisation.

After the two-step anodisation, the Al layer is completely consumed and turned into a nanopore array alumina film. Fig. 2*a* and *b* shows the straight vertical channels formed in the anodised alumina film on silicon and the interfaces between the film and the Si substrates. The original thickness of the deposited Al film is critical to the degree of ordering of the nanopores. A thin Al layer would not give the self-organisation process time to evolve enough to allow the nanopores to self-organise from their initial random distribution, as is the case shown in Fig. 2*c*. Highly ordered hexagonal pore arrays can be obtained from Al films of thicknesses greater than 40 μm, as shown in Fig. 2*e*. The periodic pore distribution or the ordering of the pore structures is more clearly assessed in the fast Fourier transform (FFT) images of both samples, for example, concentric rings for the thin Al film (Fig. 2*d*) and a well-defined 6-fold pattern for the AAO from a thick Al film (Fig. 2*f*) which is typical for hexagonal symmetry with long-range order. Although the mechanism of self-organisation is not fully understood, it was suggested that the mechanical stress at the metal/oxide interface with the expansion of the alumina during oxide formation was the cause of repulsive forces between neighbouring nanopores during the anodisation process, which led to self-organised formation of hexagonal nanopore arrays. To achieve good nanopore ordering, a prolonged anodisation time is required, hence a thick aluminium film is a prerequisite for the self-organised anodisation process. It has been proven that the thicker the Al film, the better is the pore ordering.

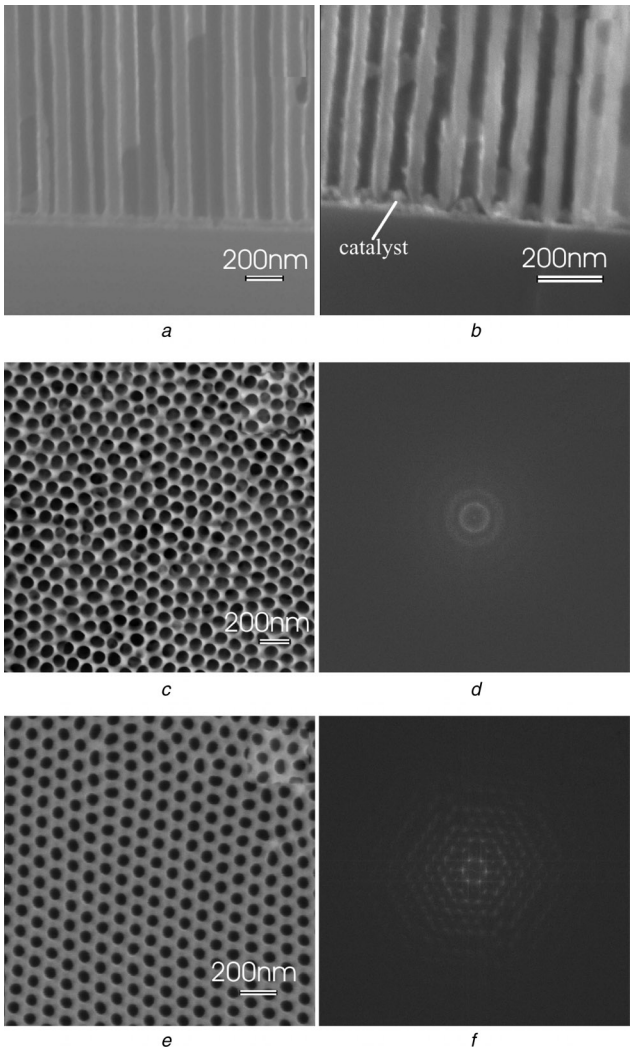

Fig. 2 *SEM images of AAO templates on Si*

a, *b* SEM images show cross-section view of channels in AAO and the interfaces between AAO and Si, where AAO barrier layer was removed by wet-etch and electrodeposition of Co catalyst was applied for *b*

c, *e* SEM top view images of the AAO pore arrays obtained from Al films with thickness of 10 and 40 μm, respectively, by anodising in 0.3 M oxalic acid at 40 V and wet-etching in 0.5 M phosphoric acid for 45 min

d, *f* FFT images reflecting the ordering of the pores

Fig. 3 shows the CNT arrays grown in these two types of AAO templates. It is clear that CNTs with wide diameter distribution and various distorted shapes were grown in the AAO template formed from a thin Al film (Fig. 3*a* and *c*), whereas uniform CNTs with a narrow size distribution and regular tubular shapes were obtained in the template formed by anodisation of a thick Al film (Fig. 3*b* and *d*). It should be mentioned here that, from TEM images (not shown here), CNTs grown from the AAO template are multiwalled and polycrystalline. Improved nanotube crystallinity was obtained via high-temperature annealing in vacuum or inert gas atmosphere at 1400–1700 °C, however, some defects remain, which is in agreement with the results from other groups [24, 25].

We note that normally a barrier layer would exist at the bottom of the anodised pores in the AAO film after the anodisation has consumed the entire aluminium. This barrier can, in principle, be removed via continued chemical wet-etching [26] and/or dry-etching (RIE or ion-mill, in case of thin AAO membranes) [27]. Hence, the CNTs

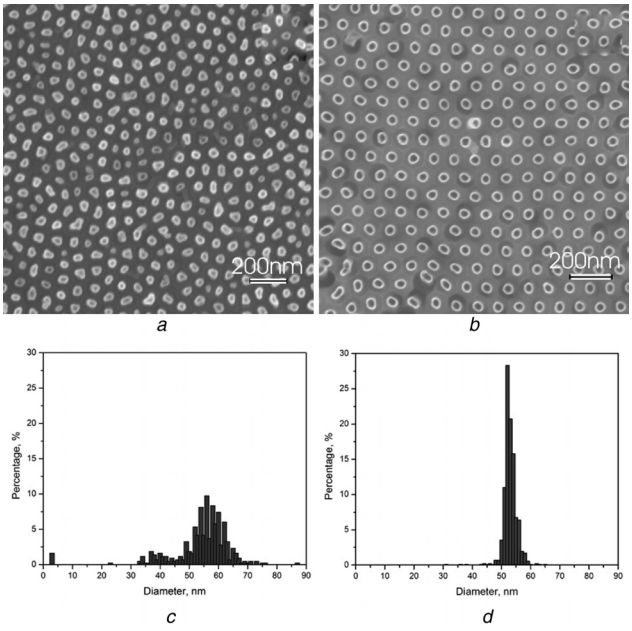

Fig. 3 *CNT arrays grown in two types of AAO templates*

a, *b* Top view SEM images of CNT arrays from thin (*a*, 10 μm) and thick (*b*, 40 μm) Al films, respectively, where AAO membrane was partially removed by wet-etch

c, *d* CNT diameter distribution graphs of the samples. The axes in both graphs are scaled in the same range for easy comparison

grown afterwards could, in principle, have direct physical contact with the Si substrate, and in effect, may form an electronic heterojunction structure. However, the anodisation process exhibited complex behaviour when the anodisation front approached the interface, with possible Si anodisation occurring after the Al film and the Ti buffer layer (if any) were totally anodised. The Si anodisation is to be avoided as it tends to yield irreproducible material property changes, and indeed could be prevented by addition of a buffer layer and carefully monitoring the current change associated with the crossing of the anodisation front from one material to another. Further development of the process protocols and precision control methods would be required if one desires high reproducibility of the electronically functional heterojunction at the metallurgical interface between the nanotube and the silicon. This is a topic that calls for more extensive future studies.

Although exploring ways for increasing the evaporated aluminium thickness are underway, we experimented with stacked multiple layers that were intermittently deposited and obtained interesting results. For consecutively deposited thick Al film from different crucibles of Al source (in our case, there were four crucibles in the chamber) with short time interruption (for switching crucibles) and constant substrate temperature, the anodisation behaviour exhibited no difference when compared with Al films from a single Al source. We also prepared Al films with long interruptions, that is, the evaporation process was paused and the sample was left in vacuum overnight with substrate temperature decreased to room temperature. The normal anodisation process for these films produced a new form of nanopore array alumina structure (stacked AAO layers). The time trace of the anodisation current (Fig. 4*a*) also manifested a multi-stage feature as imaged in the SEM (Fig. 4*b*). Apparently, a transition behaviour manifested itself in the anodisation current when electrochemical reactions reached the top of the first layer of Al (immediate above the Si substrate), indicating a restrained anodisation process. The underlying cause is likely the change of surface properties of the first layer during the pause period between the two deposition runs. This new phenomenon is intriguing as it seems to suggest a significant role played by the mechanical properties of the material which is what changed at the interface between the two consecutively deposited layers. It is notable here that (1) pore penetrating through the interface between the two layers seems discontinuous (Fig. 4*b*, inset), in accordance with the current drop as shown in Fig. 4*a* which indicates newly initiated pore development; and (2) the barrier layer of the top AAO film was totally dissolved forming a through-hole structure, which is due to the field-assisted chemical etching during the anodisation afterwards. The stacked layer structure provides further evidence of the importance of the film quality for the self-organisation. Fig. 4 is a spectacular demonstration how the stacked Al layers can switch the anodisation process.

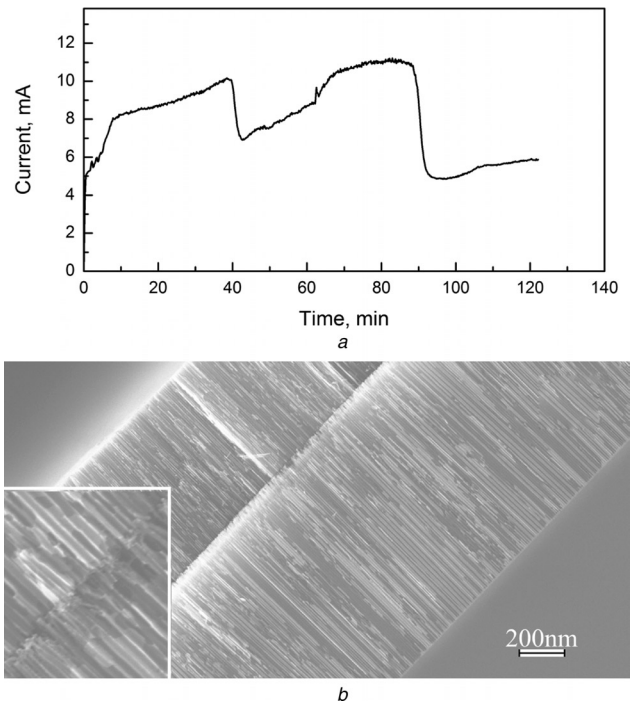

Fig. 4 *Demonstration of how the stacked Al layers can switch the anodisation process*

a Current against time curve from anodising intermittently deposited Al film in 0.3 M oxalic acid at 40 V and 10 °C, showing two stages which correspond to the anodisation of the two Al layers, respectively
b SEM image of cross-section view of the stacked AAO template, with Si substrate seen at the bottom-right corner; inset: higher magnification view along the interface between the two layers, showing the bottom of the pore channel of the top layer with its barrier layer totally etched away and newly initiated pores in the second layer

The second approach of forming highly ordered arrays of nanopores in alumina utilises a thin Al film deposited on the Si substrate, which is not feasible by the self-organised anodisation alone. The smaller Al thickness does offer the advantage of having good adhesion even in the absence of a Ti adhesion layer. Several methods for pre-patterning the Al surface have been reported, including moulding or indentation [17, 18], electron beam lithography [19], holographic lithography [20] and focused ion beam lithography [21]. In this case, we deployed another method, namely using a through-hole AAO membrane as an etching mask for patterning the Al surface. This method has not yet been reported in the literature, although AAO membranes have been used for fabrication of nanodot arrays and

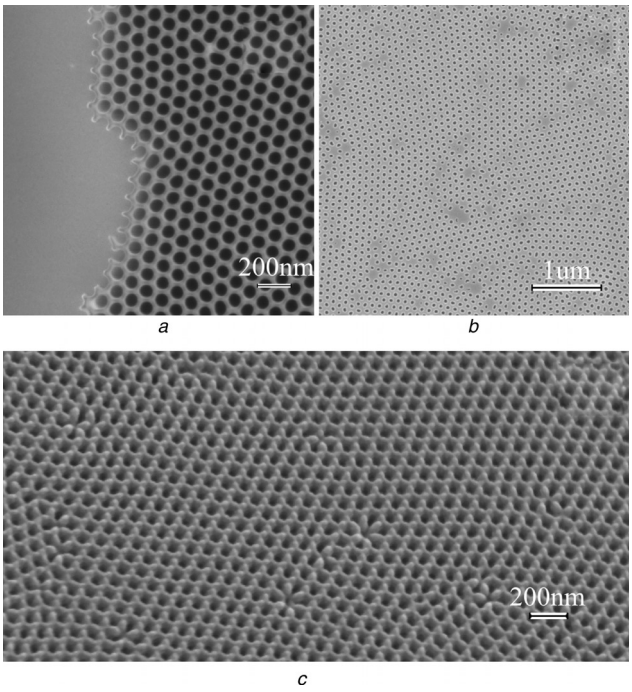

Fig. 5 *SEM images show surface morphology*

a AAO mask sitting on Al surface, viewed along one edge of the AAO film
b Al film patterned by RIE
c AAO template from 2 μm Al film anodised in 0.3 M oxalic acid at 40 V (15° tilted view)

pattern transfer [28]. Fig. 5*a*–*c* shows the surface morphology of the free-standing arrayed nanopore alumina membrane to be used as an RIE etch mask, the RIE-patterned Al film (2 μm) on silicon and the resultant anodised template for CNT growth, respectively. It is clear that the resultant alumina pore array on the Si substrate is a replication of the original AAO mask. This approach, when compared with those mentioned above, is relatively simple and economic.

Controllably exposing the CNTs from the AAO matrix follows the same process as described earlier [23]. Using the exposed CNT array as a platform for biosensing applications has been well demonstrated [29]. Studies of electrical transport, photocurrent response and mechanical resonance properties of these samples, both by our group and other groups, have shown that these CNT-Si arrays exhibited a pronounced electrical rectifying behaviour [30] and sharp electromechanical resonances and suggest that the fabrication methods reported here are promising and worth further development for applications in electronics, infrared photodetectors, bio-sensors and high-Q resonators.

4 Conclusion

Highly ordered nanopore array membranes of unprecedented uniformity on Si substrates can be fabricated directly and non-lithographically either by direct and time-controlled anodisation of a thick Al film deposited on a Si substrate or by an RIE pre-patterning followed again by time-controlled anodisation. These nanopore membranes can then be subsequently used as a template for CVD growth of CNT arrays on silicon. The substrate temperature during Al deposition and the temperature cooling rate after deposition played important roles for obtaining high-quality thick Al films and hence affected the subsequent growth of highly ordered CNT arrays on Si substrates. Compared with the approach based on thin Al films, the thick Al film approach offers a higher degree of ordering and greater uniformity in diameter and shape across a large area (a whole wafer). This work provides a controlled and scalable process for integrating highly ordered nanostructures, such as CNT arrays, onto Si, and thereby enables a wide range of applications in electronics, sensors, displays and resonators. Furthermore, the as-prepared AAO/Si substrate can also be used as a template for nanowire or nanodot (nanoparticle) fabrications for magnetic, electronic or optical device developments.

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