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Thickness-, alignment- and defect-tunable growth of carbon nanotube arrays using designed mechanical loads

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ABSTRACT

We demonstrate the thickness-, morphology-, and defect-tunable growth and simultaneous integration of aligned carbon nanotube (CNT) arrays using a novel microscale platform. This platform consists of a micromechanical spring of desired stiffness, which applies a precise vertical load to a vertically aligned CNT array during its growth by chemical vapor deposition (CVD). The micromechanical spring is strained by the extrusive growth force output from the aligned CNT array during its growth and, at the same time, exerts a mechanical restoring force against the buckling resistance of the CNTs. This application of a designed vertical load on the CNTs allows modulation of the thickness and degree of alignment of the CNT array, as well as the structural quality of the individual CNTs. Consequently, the electrical resistance between two opposing CNT arrays can be tuned by adjusting the vertical load. In addition, their sensing responsiveness toward chemical species can also be enhanced by applying larger vertical load on the CNTs. In contrast to conventional growth methods for producing aligned CNT arrays, our approach offers an efficient way for the growth engineering and on-chip integration of aligned CNT arrays in a single step of the CVD.

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1. Introduction

Research on the growth kinetics [1,2], assembly [3], and methods of integrating [4] densely packed and aligned arrays of carbon nanotubes (CNTs) has led to the development of CNT-based devices with high mobility and flexibility for electronics [5,6], fast electron transfer reaction and high energy density storage [7,8], and improved contact reliability in micromechanical switches [9,10]. These distinctive properties arise from the unique and remarkable mechanical [11], electrical [12], thermal [13], and geometric [14] characteristics of aligned arrays of CNTs. To exploit and extend the properties of CNTs for even more diverse applications, the controlled growth and integration of aligned CNT arrays is essential, since the properties and functionalities of CNT-based devices depend strongly on the thicknesses and morphologies of the integrated CNT arrays [15–18]. Moreover, the introduction of defects in the atomic structures of the CNTs may alter their physical properties and provide chemical functionalization sites, leading to new potential applications [19–21].

However, sufficient and effective modulation of the thickness, degree of alignment, and structural quality of aligned CNT arrays still remains a challenge. For example, it is possible to generate disordered carbon in CNTs using growth-temperature modulation [22], ion irradiation [23], the application of high currents [24], and oxidation at high temperatures in air and by an acid treatment [25] during or after the CNT growth. However, these methods involve multiple growth

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steps or require repetitive post-growth treatments in order to introduce various defects and disordered states. To modulate the thickness and alignment of CNT arrays, the effects of various parameters of the chemical vapor deposition (CVD) have been studied, such as the growth time, precursor species, pressure, and temperature, as well as the thicknesses and compositions of the catalysts used and the conditions for their pretreatments [2,26–31]. In situ monitoring of the growth rate during CVD using laser diffraction, reflectivity and interference analyses [1,32,33], time-resolved photography, [34,35] and laser displacement sensors [36] has enabled better control over the thickness of the CNT arrays than has timed growth. Even though these approaches can elucidate the growth mechanisms of CNT arrays and their termination behaviors, obtaining CNT arrays with various controlled thicknesses and alignments using CVD involves multiple steps in the growth processes, repeated observations, and additional apparatuses such as laser sources and photodetectors.

An alternative approach has been to place a heavy mold on the catalyst-coated substrate prior to growth, which transfers the shape of the mold to the grown CNT array, allowing its thickness to be controlled [37]. In addition, it was also found that an increase in the weight placed on the catalyst led to a decrease in the degree of alignment of the CNT arrays [37]. However, this approach is only suitable for growing CNT arrays perpendicular to the planar substrate. Furthermore, in order to fabricate CNT-based devices, post-transfer processes are inevitably required for the site-specific integration of the CNT arrays into the desired micro- and nanostructures. This increases the process complexity as well as the possibility of contamination of the CNTs. On the other hand, the direct growth of CNTs on gold-coated microelectrodes leads to arrays of different lengths, depending upon the thickness of the gold layer [38]. However, in such arrays, the gold layer forms uncontrollable electrical paths, preventing the fabrication of practical CNT-based devices.

In this work, we describe the development of a platform containing a micromechanical spring that enables the thickness-, alignment- and defect-tunable growth and integration of aligned CNT arrays onto specific microstructures. The extrusive force generated during the growth of the CNT array is delivered to the spring, and, accordingly, a vertical load is applied perpendicularly to the aligned CNT array during the CVD. This is owing to the restoring force produced in the strained micromechanical spring against the buckling resistance of the CNT array. The vertical load can be easily adjusted by changing the stiffness of the spring. We were able to experimentally demonstrate that the application of a larger vertical load during the growth process resulted in CNT arrays with smaller final thicknesses and lower degrees of alignment. In addition, the CNTs in these arrays had a deteriorated structural quality. Furthermore, all the process steps could be potentially scalable for the manufacture of CNT-based devices such as sensors. In this work, the micromechanical platform was fabricated on a 4-inch silicon-on-insulator (SOI) wafer, and the CNTs were grown on a diced chip of a SOI wafer inside a 1-inch tube furnace. Thus, the method can be scaled up by merely increasing the sizes of the wafer and tube furnace. The electrical characteristics of the integrated CNT arrays were investigated, and their applications as chemiresistive and electrothermal pressure sensors were demonstrated. The dependence of the responsiveness of the chemical sensor on the amount of vertical load on the CNTs was also analyzed.

2. Experimental

The mechanism for the tunable growth and integration of aligned CNT arrays is depicted in detail in Fig. 1. The platform is composed of a silicon-processed anchor and a micromechanical spring that are initially separated by a set gap (Fig. 1a). In the multiple platforms tested, the length of the spring was varied to obtain different stiffnesses, while its width and height were kept constant. After the deposition of the iron catalyst on the microstructures, aligned CNT arrays were grown on the sidewalls of both the anchor and the micromechanical spring by CVD; the growing CNT arrays subsequently filled the gap completely. In this state, the thickness of each CNT array was about half the initial gap, regardless of the stiffness of the spring used (Fig. 1b). Even after the gap was filled completely, the CNT arrays kept growing; this resulted in the micromechanical spring being strained owing to the growth force exerted by the CNT arrays. This extrusive growth force generated by the growing CNT arrays has been reported previously as well. For example, it was observed that growing CNTs could lift a mass placed on top of a catalyst layer [37], a deposited metal film [39], and stacked and multilayered CNT arrays [40,41]. It has been found that the high-density growth of CNTs and the van der Waals interactions between neighboring CNTs inhibit the overgrowth of CNTs toward and penetration into an opposing array of CNTs [9,42]. Thus, the force generated by the growth of the CNTs was entirely delivered to the micromechanical spring.

A growth force of approximately 10^{-1} nN has been reported for a multiwalled CNT [37], but this may vary depending on the diameter, number density, and length of CNTs, which are in turn related to the catalyst preparation, pretreatment, and CVD conditions used [29–31,43,44]. However, in our study, this force was large enough to deform the compliant micromechanical spring during the growth process. Thus, the CNT arrays are under a vertical load once they make contact each other. With all the other CVD parameters and the growth area being constant, the magnitude of this load is dependent on the stiffness and deformation of the spring. As the growth process continues, the spring deforms and the vertical load increases further, until the growth force from the CNT array balances the restoring force from the spring. At this point, the spring stops deforming and the vertical load remains constant. Growth after this point does not increase the final thickness of the CNT array and only changes the morphology of the individual CNTs, making them rather buckled and less aligned in shape. Hence, the final thickness of the CNT array decreases as the length of the mechanical spring decreases (Fig. 1c).

The micromechanical platform was fabricated by bulk micromachining on a SOI wafer with a 20-μm-thick arsenic-doped single-crystalline silicon device layer ($\rho < 0.005\ \Omega$ cm), as described in Fig. S1 of Supporting Information. A

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