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Self-aligned mechanical attachment of carbon nanotubes to silicon dioxide structures by selective silicon dioxide chemical-vapor deposition

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A self-aligned thin-film deposition technique was developed to mechanically attach carbon nanotubes to surfaces for the fabrication of structurally robust nanotube-based nanomechanical devices. Single-walled carbon nanotubes were grown by thermal chemical-vapor deposition (CVD) across 150-nm-wide SiO2 trenches. The nanotubes were mechanically attached to the trench tops by selective silicon tetraacetate-based SiO2 CVD. No film was deposited on the nanotubes where they were suspended across the trenches. © 2003 American Institute of Physics. [DOI: 10.1063/1.1636267]

Single-walled carbon nanotubes (SWNTs) have been shown to possess extraordinary nanomechanical properties, such as a Young’s modulus of ~1 TPa and a high aspect ratio. These properties are currently limited by a mechanically weak Van der Waals attraction between the carbon nanotube (CNT) and the substrate. One method, developed to rigidly attach CNTs to AFM probe tips, uses an acrylic adhesive obtained from briefly sticking the probe tip to carbon tape before manually attaching the tube. Another method involves welding a CNT onto a silicon AFM probe tip using a scanning electron microscope (SEM) beam. While each method provides rigid attachment and significantly improves AFM resolution, they are both time consuming and yield CNT probes with inconsistent lengths and diameters.

We are pursuing self-aligned methods for nanomechanical fabrication. Self-alignment is currently a key technology in silicon device manufacturing, and could benefit nanomechanical fabrication processes because

1. patterned layers can be produced without additional lithography steps and
2. self-alignment can provide more accurate alignment than lithography.

Additionally, self-aligned methods are useful on three-dimensional structures for which traditional lithography is difficult. For example, self-aligned processes have been useful for defining nanoscale features on AFM probe tips.

We have developed a rigid attachment method to overcome the difficulties of individual CNT attachment, using the self-aligning thin-film deposition technique described here. A CNT was grown over a lithographically produced trench [Figs. 1(a) and 1(b)] and SiO2 was then deposited over the trench [Fig. 1(c)] to rigidly attach the CNT. The film was deposited selectively on the SiO2 surface, and did not cover the CNT where it was suspended over the trench. This resulted in a self-aligned attachment of the CNT where it contacted the SiO2 substrate. If this method had been nonselective, it would have also coated the CNT in the suspended region, increasing its diameter and altering its nanomechanical properties.

The trenches were produced by e-beam lithography in poly(methyl methacrylate) that was spun onto a SiO2 surface. Dry etching was used to transfer the pattern into the SiO2, resulting in trenches 150 nm wide and 40 nm deep.

The trench wafer was dipped into a 150 μg/ml ferric nitrate nonahydrate in isopropyl alcohol catalyst solution. CNTs were then grown on the trench sample by chemical-vapor deposition (CVD) at 700 °C. The CVD was done at atmospheric pressure with flow rates of 150 sccm argon, 100 sccm hydrogen and 5.5 sccm ethylene for 6 min.

The SiO2 was thermally deposited from a silicon tetraacetate precursor in the reaction

\[ \text{Si(O(O)CCH}_3\text{)}_4 \rightarrow \text{SiO}_2(s) + 2(\text{CH}_3\text{CO})_2\text{O}(g) \]

which occurs at 170 °C. The (CH₃CO)₂O, or acetic anhydride (bp
The Si(O(O)CCH₃)₄ precursor was heated to 100 °C, just below its melting point, and the top of the grown film. Height measurements are performed on planar Si substrates, and the Si(O(O)CCH₃)₄ precursor was heated to between 97 and 104 °C for all cases. Oxide thicknesses before and after deposition were measured using ellipsometry. After deposition, the wafer showed a significant increase in the strength of the SiO₂ peak, relative to the Si–Si peak. This shows that the deposited layer was SiO₂.

To confirm that SiO₂ films were grown with our CVD apparatus, the SiO₂ was first deposited on planar Si substrates and analyzed. The deposition rate was determined by ellipsometry to be 0.2 nm/min (Table I). Composition and chemical bonding of the deposited film were studied by XPS. Figure 3 shows Si 2p XPS narrow scans before and after CVD of SiO₂. The lower binding energy component of these spectra comes from the bulk Si and the higher energy component comes from the Si in SiO₂. The chemical shift of ~4 eV from the bulk Si 2p XPS peak is consistent with Si bonded to four oxygen atoms, for example, SiO₂. Prior to SiO₂ CVD, the ratio of Si in SiO₂ to Si in bulk silicon was 0.31. After CVD, this ratio increased significantly to 16. The increase of the SiO₂ peak in relation to the Si peak verified that the deposited layer was SiO₂. The XPS data was also used to calculate the SiO₂ film thickness to confirm the ellipsometry data.

Characterization of deposited films was followed by deposition on suspended CNT samples illustrated in Fig. 1. One of the several CNTs that spanned multiple trenches was found on the trench–nanotube sample by AFM, and landmarks were recorded so that the tube could be located again after processing. The CNT was measured by AFM to be 1.9 nm in diameter (a single-walled CNT), and rested on top of the trenches (Fig. 4). SiO₂ was deposited by CVD on top of
the trenches and CNTs for 55 min at 172 °C. The sample was then imaged with AFM [Fig. 5(a)]. AFM height measurements showed a difference of 8.2 nm between the top of the NT and the top of the trenches [Figs. 5(b) and 5(c)]. This height difference shows that a film covered the NT where it was in contact with the substrate, but that very little or no film was deposited on the suspended region of the tube, confirming that we achieved selective SiO2 deposition. The SEM image in Fig. 5(d) shows both the covered and suspended segments of the NT under the deposited film.

The selectivity of the deposition is consistent with the expected surface chemistry. While the CNT, graphene surface should be chemically inert to Si(O(O)CCH3)4, surface silanols (═SiOH) of the patterned trench tops should readily react with Si(O(O)CCH3)4 to form acetic acid (HOC(O)CH3) and SiOSi(O(O)CCH3)3. This is the start of the chemisorbed SiO2 layer, which continues to grow according to the previously mentioned reaction Si(O(O)CCH3)4(g)→SiO2(s)+2(CH3CO)2O(g), at 170 °C.

Silicon tetraacetate-based SiO2 CVD provides a self-aligned method to rigidly attach CNTs to SiO2 structures. Other oxide structures could also be compatible with this process, and should be explored. The self-aligned nature of the process will allow rigid NT attachment on three-dimensional SiO2 structures such as AFM probe tips. This process is compatible with mass fabrication of CNT AFM probes and could aid in making them available to a wider range of researchers. This process may be particularly valuable for producing CNT AFM tips for fluid imaging, where rigid attachment to the tip is critical.

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10 The relative areas of the Si 2p peaks from bulk silicon and SiO2 were used to calculate the SiO2 film thickness using standard XPS theory [C. S. Fadley, R. J. Baird, W. Siekhaus, T. Novakov, and S. A. L. Berström, J. Electron Spectrosc. 4, 93 (1974)]. Literature values for the mean free paths of Si 2p photoelectrons in silicon (1.6 nm) and silicon dioxide (2.6 nm) were used in these calculations [M. F. Hochella and A. H. Carim, Surf. Sci. 197, L260 (1988); M. Suzuki, H. Ando, Y. Higashi, H. Takemata, H. Shimada, N. Matsubayashi, M. Imamura, S. Kurosawa, S. Tanuma, and C. J. Powell, Surf. Interface Anal. 29, 330 (2000)]. Ellipsometry is known to yield thickness measurements up to 40% higher than XPS [M. F. Hochella and A. H. Carim, Surf. Sci. 197, L260 (1988)]. Here, our ellipsometry measurements were ~25% higher than our XPS measured thicknesses, within the expected agreement of the methods.