

Physical properties of novel free-standing polymer–nanotube thin films

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Abstract

Novel polymer–nanotube composites have been fabricated by the introduction of polymeric material into the free volume contained in carbon nanotube (CNT) mats grown by chemical vapour deposition. The resulting composite material can then be peeled from the substrate to give a free-standing film, the properties of which are controlled by the nanotubes. The nanotube mat acts as a percolative network resulting in increases in surface and bulk conductivities by factors of 10^7 and 10^8 , respectively. The presence of CNTs also causes the Young's Modulus of the polymer film to double. It is shown that the presence of CNTs has no effect on the morphology of the polymer itself.

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1. Introduction

Due to their highly desirable physical and chemical properties, carbon nanotubes have been extensively investigated since their discovery in 1991 [1]. Probably the most readily attainable application for these materials is as a filler in polymer or epoxy-based composite systems. Polymer–CNT composites can be used for a large variety of applications including solar cells [2], flat panel displays [3,4], electromechanical actuators [5], electrostatic dissipation [6], electromagnetic interface shielding [6], flexible electronics [7], and nanosensors within a polymer to monitor deformations [8] and polymer transitions [9]. Many of the advantages of CNTs over metal fillers are associated with their greater flexibility, lower density, better durability and of course their one-dimensional nature. However, problems connected with the commercialisation of CNTs include difficulties scaling up the production process, and the challenges of handling and selectively positioning such

a low density, nano-sized material. One of the most likely methods for commercial growth is chemical vapour deposition (CVD) [10–12]. CVD allows for the in situ growth of CNTs onto a range of substrates. This technique is also scalable and can be used to produce a large number of CNT arrays at once.

The incorporation of CNTs into a polymer matrix allows us to combine the desirable properties of the nanotubes with the processing advantages of the polymer. Many composite production techniques have been demonstrated including extrusion [13], intercalation into Buckypaper [14], electrospinning [15] and solution processing [16]. However, in all cases achieving controlled dispersion of the nanotubes in the matrix is a non-trivial problem. We address this issue by introducing a novel composite formation method whereby the polymer matrix is incorporated around a pre-formed nanotube network. Using this technique we can selectively position the CNTs within the composite, as previously demonstrated by the authors [17]. This technique results in a uniform nanotube distribution as controlled by the pre-deposited catalyst and growth process. In this letter we report initial studies on the properties

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of a CNT–polymer composite system produced by this technique.

2. Experimental

Nanotubes to be used in the composite were grown by chemical vapour deposition. This process involves the thermal decomposition of a carbon source which can then restructure and grow as CNTs in the presence of a catalyst material. We focus on acetylene as the carbon source. The catalyst we use is poly(styrene–vinylferrocene). The vinylferrocene part of this copolymer contains an iron core which is active as the catalyst for CNT growth. The copolymer was dissolved in toluene to form a solution which could then be drop cast onto silicon oxide (SiO_2) substrates. The substrates are left for 24 h for the solvent to evaporate. Previous thermo-gravimetric analysis of the catalyst copolymer revealed that 5.1% of the copolymer remains at 700 °C, by 900 °C this has reduced to 1.1% (TGA graphs not shown). We therefore choose 700 °C as a suitable temperature for the CVD process.

3. Results and discussion

Fig. 1 shows a scanning electron microscopy (SEM) image of as-grown CNTs on a SiO_2 substrate. Nanotubes cover the surface of the SiO_2 . The average tube diameter in the SEM below is in the region of 90 nm, which includes a 2×15 nm gold layer deposited for imaging purposes. (This gold layer is absent from CNT arrays incorporated into composites.) In this work we choose to grow nanotubes as an entangled mat rather than as aligned arrays. This will result in a composite film with an isotropic nanotube distribution. However, it would be just as simple to

grow an aligned nanotube forest, giving an anisotropic nanotube distribution in the resulting composite.

Composite formation was achieved by spin coating a curable polymer onto the CNT mat. The polymer used was Sylgard 184 silicone elastomer (Dow Corning). It should be pointed out that although we have chosen to use an elastomer in this work, any soluble polymer can be used for composite formation using this technique. The transparent polymer comes as a base and a curing agent; the two are mixed together in a 10:1 ratio. The medium viscosity solution is then allowed to sit for a period of 1 h so that air bubbles, introduced by mixing, can dissipate. The polymer solution is then spin coated, at 500 rpm for 60 s, onto the as-grown CVD nanotubes. The solution intercalates into the free volume between the CNTs and can be left to cure under atmospheric conditions for a period of seven days. Normal cure time is 24 h at 25 °C but to guarantee a fully cured system we allow longer. Once fully cured, the composite film can be peeled away from the substrate to produce a free-standing thin film approximately 250 μm thick. During CVD nanotubes grow with one end attached to the substrate. On polymer deposition, these tubes become securely embedded in the elastomer and detach from the substrate as the cured composite is peeled away. As seen from the inset of Fig. 1, the resulting composite is opaque due to the dense CNT network embedded throughout the transparent polymer matrix. Derivative thermogravimetry measurements (not shown) on the composite films show the presence of a very small nanotube oxidation peak between 550 and 650 °C. Integration of this peak allows us to estimate the nanotube content of the composites to be ~ 0.4 wt%.

In this case the nanotubes are randomly oriented which should allow for the formation of a three-dimensional

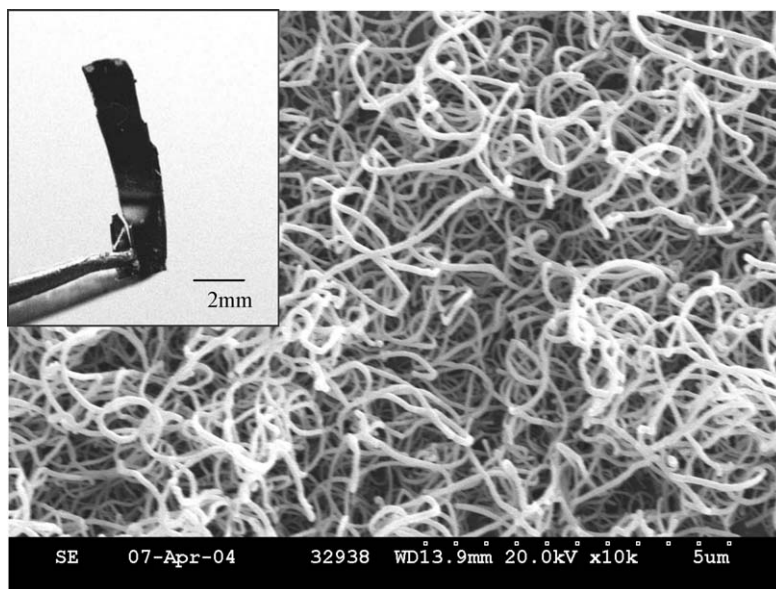


Fig. 1. SEM image of entangled CNTs grown on SiO_2 . The tubes are then incorporated into a polymer matrix by spin coating a curable silicone elastomer onto the as-grown CNTs. The inset shows a photograph of the free-standing cured composite thin film held by a tweezers.

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