

FABRICATION AND EVALUATION OF CARBON NANOTUBE-PARYLENE FUNCTIONAL COMPOSITE-FILMS

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Abstract: We present the design, fabrication and mechanical testing results from Parylene-Carbon Nanotube (CNT)-Parylene composite-films. Utilizing SWNT materials as the active layer, we have fabricated flexible devices consisting of 10 μ m thick parylene membranes wrapped around active SWNTs. The tensile test results show that parylene-CNT-parylene sandwich has a linear-elastic response up to a strain value of $\epsilon \cong 2\%$ and the composite fails at $\epsilon_{\text{Fail}} \cong 7.5\%$ strain. The load-unload testing of the sample shows that a very small change in resistance ($\sim 1\%$) was observed when applying a strain ranging from 0% to 2% and the results were repeatable up to 100 times. The resistance of the CNT-parylene film increased 9 fold at failure. Potential applications of this work include interconnect materials for flexible electronics devices.

Keywords: Flexible Parylene-C Substrates, Carbon Nanotubes, Flexible Electronics

1. INTRODUCTION

There is a growing interest in making sensors, optoelectronic and electronic devices on flexible polymer films. One of the main limitations in the field of organic electronics is that the mobility of the transistors is relatively low. Carbon nanotubes (CNTs), first discovered by Iijima in 1991 [1], are unique materials due to their excellent electrical, mechanical and thermal properties, and have good chemical stability. With their exceptionally high mobility exceeding 75,000cm²/Vs, CNTs can potentially be utilized in electrical device applications [2]. Furthermore, the hollow structure and closed topology allow extreme strains under tension (40%) without showing signs of brittle behavior, plastic deformation, or bond rupture [3-4]. Thus, CNT based network architectures on flexible media present a promising technology for Flextronics.

Applications of Flextronics place challenging demands on optical properties, dimensional stability, and solvent/moisture resistance of the substrates. Polymers, such as PEN [5] and PET [6] have currently been utilized as candidates for flex-film substrates because of their widely available manufacturing techniques for more traditional

applications. PDMS with a relatively low elastic modulus (~ 1 MPa) allows large strains and has been used in various applications [7]. However, it is difficult to manufacture very thin PDMS substrates and such substrates lack the robustness to be of value as flexible substrates and are also not compatible with IC fabrication processes.

Parylene, an inert, conformal and mechanically strong thin film, is a potentially suitable yet rather a rarely explored candidate as a substrate for Flextronics applications [8-9]. Deposited at room temperature, it is a lightweight, transparent, and stress-free material which is compatible with IC



Fig. 1 Optical photograph of the CNT-parylene sandwich-films

fabrication processes. These properties suggest that Parylene can be a strong player as a substrate material for flexible electronics.

In this paper, we present our recent results of CNT-parylene composite-films for flexible electronic devices (Fig.1). The fabrication results from SWNTs based lateral interconnect structures embedded in between two Parylene-C layers and a study of their electrical characteristics under tensile loading are reported.

2. FABRICATION

One of the potential application areas of the parylene/CNT/parylene sandwich is creating lateral interconnects. Accordingly, we have fabricated SWNT-based structures embedded in between two Parylene layers. The fabrication process (Fig.2) starts with the deposition of a thin parylene layer (10 μ m) on 3" silicon wafers. Then, we deposit Au electrodes (1500 \AA) utilizing a transparency shadow mask. Next, a solution of SWNTs was drop casted and air dried. A 10 μ m parylene layer was next deposited to encapsulate the SWNTs interconnects. We then opened contact areas on the second parylene layer with an inductively coupled plasma (ICP) etching tool using shadow mask patterning.

2.1 Sample Preparation

After fabrication, the CNT-parylene sandwich was peeled off from the silicon wafer. The overall shape and dimensions of a typical sample tested for strain and electrical conductivity is shown in Fig.3, with test area of 2 \times 10mm. The gold leads are placed away from the test area on the lower

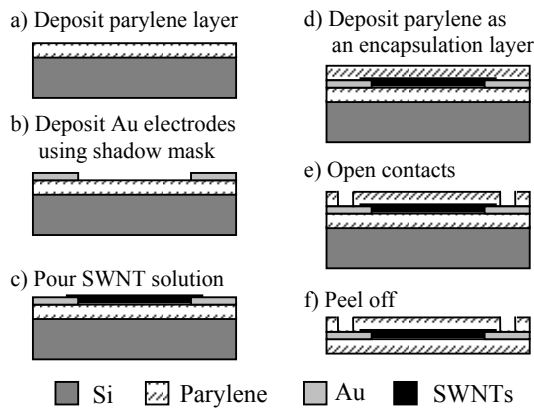


Fig.2 Fabrication process

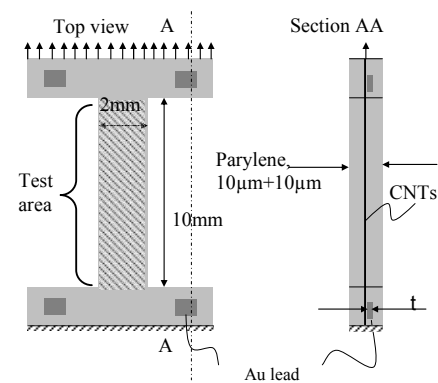


Fig.3 The tested dimension of sample

and upper horizontal corners of the specimen. Since the entire sample surface is covered with SWNTs, it is crucial to reduce the strain near the electrodes. These sections are further reinforced by gluing 250 μ m thick cardboards on both sides (inset of Fig.4). The goal of this standard exercise is to reduce the strain experienced in the top and bottom horizontal sections and for the strain measurement. A finite element model of this “structure” has been built. The strain distribution in the specimen (Fig.4) shows that the horizontal sections experience approximately 1/1000th of the strain experienced in the test area.

3. RESULTS AND DISCUSSION

3.1 Mechanical Testing

The test specimen is attached to a Universal Testing Machine made by CETR (Campbell, CA). Special attention is paid to ensure the grips hold the lower and upper horizontal sections without slip. A strain rate of 0.0033mm/sec is applied to

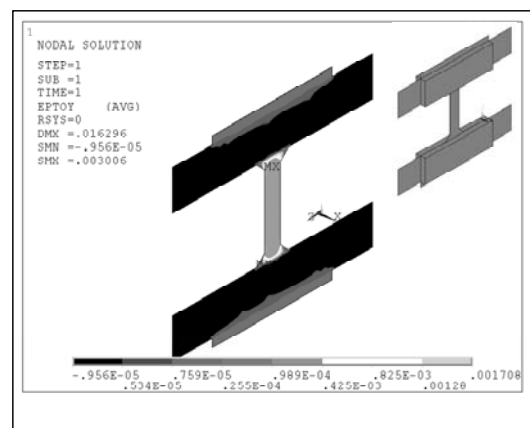


Fig.4 Strain distribution in the test specimen by FEA

the specimen. During the test, resistance is measured through a Fluke ModelNo 189 (Everett, WA) multimeter. The normal-stress (σ) is calculated based on the measured force and the cross sectional area ($40 \times 10^{-9} \text{m}^2$). The normal strain (ϵ) is calculated based on the gauge length of 10mm and the imposed elongation. The history of stress, strain, and electrical resistance is stored during the test.

3.2 Mechanical Properties

A tensile test showed a linear-elastic response until a normal-strain value of $\epsilon \cong 4\%$; the material failed at $\epsilon_{\text{Fail}} \cong 8\%$ strain. The elastic modulus and the ultimate tensile stress were measured to be $E = 1.8 \text{GPa}$ and $\sigma_{\text{ult}} = 55 \text{MPa}$. These values were similar to those obtained from a $20 \mu\text{m}$ thick parylene without the nano-tubes.

Fig.5a shows the stress-strain curve, and Fig.5b shows the relative change in resistance $\Delta R/R_0$, during this test as a function of strain. Initial resistance of the CNT-parylene thin-film was $R_0 = 66 \text{Ohms}$. The resistance of the film increased 9 fold until failure. The sample was still conductive when the film failed. The relative change of resistance change follows a cubic-polynomial trend ($R^2 = 0.9995$) up to 7% strain.

The details of 0 – 3% strain-range are plotted on the inset of Fig.5b. This figure shows that the rate of change of resistance, $dR/d\epsilon$, is fairly uniform until 2% strain, where $\Delta R/R_0 = 1.5\%$. The rate of resistance change increases thereafter, and at 3% strain the $\Delta R/R_0$ becomes 4%.

3.3 Load-unload tests

Flex-films were subjected to cyclical tensile strain using two different instruments at room temperature. First a micrometer based fixture (strain resolution 0.25%) which allowed only the control of strain was used for a small number of cycles. Next, the CETR tester was used to subject the specimens to a large number of tensile load-unload cycles.

Using the micrometer based tester for load-unload tests, a specimen was strained to 1.3% and relaxed to 0% for four consecutive times. Then it was strained to 1.5%, 1.8% and 2% strain, and each strain level was repeated four times. The

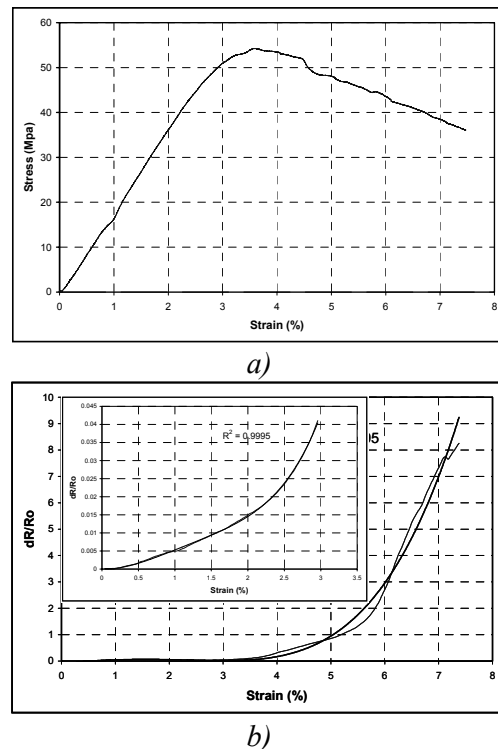


Fig.5 a) Stress-strain test of $20 \mu\text{m}$ CNT-parylene film b) Change in resistance

results for 2% strain case are given in Fig.6. Generally, a very small change in resistance ($\sim 1\%$) was observed when applying the strain range from 0% to 2% and the results were repeatable, with negligible hysteresis, as shown in Fig.6.

Using the CETR tester a specimen was tested for load-unload in the 0 – 1% strain ranges for 100 repetitions. Fig.7 shows the normal stress σ and the change in resistance $\Delta R/R_0$ in the specimen as a function of normal ϵ strain, for the 1st and the 99th cycles. While the σ - ϵ curve shows a very small amount of hysteresis for the 1st-cycle of loading,

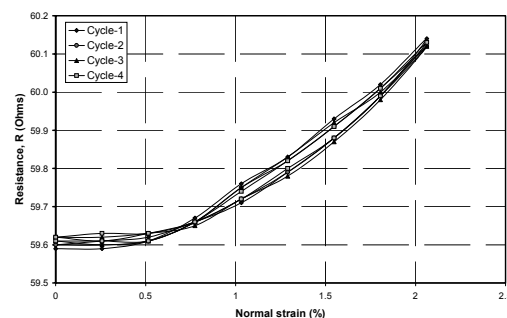


Fig.6 R - ϵ plots for 4-cycles up to 2 % strain

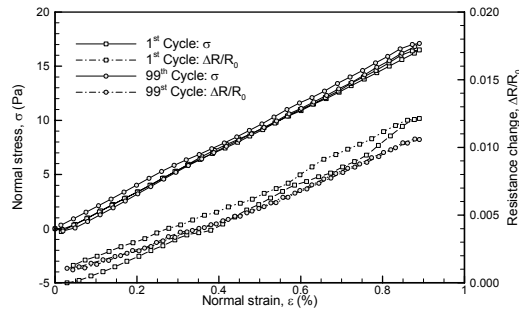


Fig.7 Normal stress and strain and the change in resistance for the 1st and 99th cycles

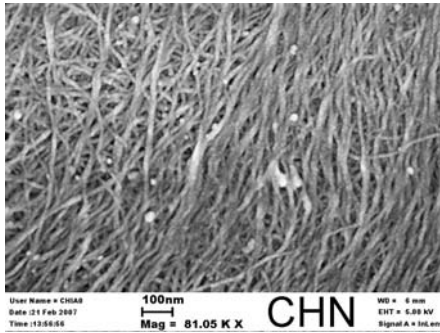


Fig.8 SEM micrograph of the bundles of SWNT on the parylene surface as deposited

the 99th cycle behaves nearly perfectly elastic. This same observation applies to the $(\Delta R/R_0)$ -vs- ϵ behavior during the test. The film was still functional at the end of 100 cycles, where we stopped testing.

The distribution of the CNTs on the parylene was captured by an SEM image as shown in Fig.8. The drop casting process results in CNT bundles to dry on the surface in different orientations, and in an overlapping manner. It is likely that this overlapping in different orientations enabled continuous electrical conductivity in this thin flex film, even for large strain values.

4. CONCLUSIONS

We fabricated a CNT-parylene composite thin-film and characterize mechanical properties. The macro scale samples (H, W, and L of $20\mu\text{m} \times 2\text{mm} \times 10\text{mm}$) were subjected to tensile testing. Change of electrical resistance of the samples was monitored as a function of normal strain. Load-unload tests in the elastic range of the Parylene showed small resistance change ($\sim 1\%$) and a small

amount of hysteresis in the R- ϵ data. Test to failure showed that the sample was conductive up to the failure strain of 8%. Furthermore, the resistance increased 9 fold during the failure test and the R- ϵ has a nice fit by a cubic polynomial. Single-walled carbon nanotubes (SWNTs) embedded in Parylene-C layers remains conductive for reasonably high strain levels. This opens up the possibility of their use as device and interconnects applications for Flextronics.

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