Towards Graphene Based Flexible Force Sensor

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***Abstract*— Monolayer graphene transferred over flexible polyvinyl chloride (PVC) substrate combined with closely packed layer of nano-spheres (NSs) is fabricated for force sensing application. The force was applied from vertical direction through NSs which acts as lateral strain enhancers. The stack persuades lateral in-plane strain in the monolayer graphene for the applied vertical pressure through NSs. The electrical measurements demonstrate that the graphene layer is able to respond for soft touch range commonly perceived by human beings. The sensing stack was fabricated using simple approaches such as hot lamination graphene transfer process and drop casting of NSs. The device structure is flexible to conformably cover the non-planar surface for applications such as large area pressure sensing and robotic e-skin.**

Keywords—Graphene, pressure sensors, Nanosphere lithography, hot lamination process

I. Introduction

Touch sensors are gaining significant importance as they become major input and control modalities to operate modern electronic gadgets such as mobile phones, laptops, and robotics systems[1]. Successful sensing systems includes resistive and capacitive schemes fabricated using thin films of inorganic and organic materials [2, 3]. Additionally, highly sensitive large area sensing structures are desirable for technologies such as robotics, prosthetics, and bio-medicals instrumentation [4-6]. For example, artificial electronic skin (e-skin) for robots could be fabricated using large area tactile sensors with matching bio skin functionalities using synthetic (inorganic/organic) materials [7]. Such systems will be able to sense touch in the range of 0.02 to 10 N, which falls in the range of human touch perception. These requirements open opportunities for novel high sensitive devices and systems. 1D and 2D nanomaterials based sensors could play a major role to develop such architectures[8]. The advantage being their inherent discreteness, high surface to volume ratio and attractive physical and chemical properties. Importantly, unlike traditional thin films, inorganic nanostructures could be easily integrated with flexible materials. This finds interesting applications large area flexible electronics[9]. Graphene is an important 2D nano-structured material established to have synthesized over large area. Monolayer graphene based pressure sensing devices are attractive due to its high sensitivity, scalability over large area and flexibility for conformal coverage[10, 11]. Many sensing device architectures based on graphene have been explored in the past. Few techniques utilized multilayer graphene structure which took the advantage of the piezo resistive property[12]. Alternatively, graphene strain sensors reported in the literature involves tedious and expensive fabrication processes. Tedious line patterns have been reported

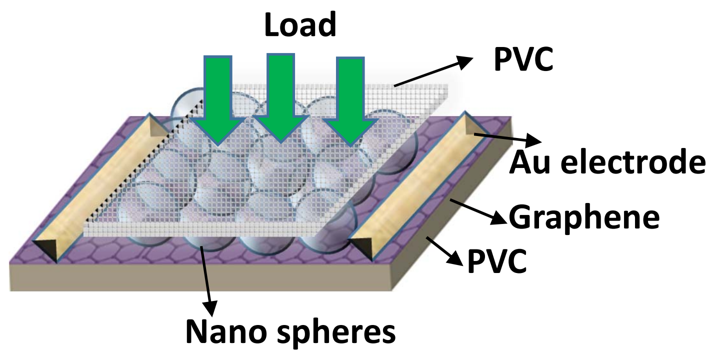

Fig.1. Schematic illustration of the electromechanical sensing structure

to act as pressure amplifying structure for graphene based force sensors[13]. These methods are not suitable for fabricating flexible large area sensing systems.

The current work aims to fabricate a force sensing structure with monolayer graphene as active material. The major difference with the existing methods lies in employment of cost effective fabrication methodologies and the ability to preserve the mechanical properties of monolayer graphene. The force sensing structure is based on interaction between silica NSs and graphene through application of external force. The sensing structure currently fabricated as two separable stacks. Microscopically, change in electrical conductivity of graphene with application of external load was measured. Tensile stress in graphene causes in-plane deformation due to stretching between C-C bonding. Theoretical estimates prove that graphene holds 200% strain in the elastic limit of the stress-strain curve. The change in resistance in this regime could very well comprise the pressure range a human skin senses.

II. Experimental Details

A. Sensor structure

The sensing device structure includes two layers facing each other as described in Fig.1. Graphene layer transferred over transparent PVC substrate[14] with an active area of 5 mm^2 with Au electrodes deposited at both ends. This active graphene layer attached over an immovable plate facing upwards to arrest its motion during external loading. The strain enhancer layer consists of monolayer of silica nano-spheres (NSs) deposited over silicon and PVC materials. This NSs layer (over Si and PVC) is inverted and placed for it to make contact with the active graphene region. The pressure is applied from rear side of NSs layer to enhance lateral strain on the graphene channel. The whole structure was made flexible and transparent with aid of chosen PVC substrate.

B. Fabrication methodology

CVD grown commercial graphene (Graphenea) on Cu substrate transferred over highly flexible PVC substrate using hot lamination (Fellowes gloss laminating pouch) approach. The Graphene/Cu stack placed over a white paper and these three layers sandwiched between two PVC sheets (125 µm thick) at 125ºC during lamination process. The back side PVC is detached from Cu substrate through white paper and this process results a stack of PVC/Graphene/Cu. Cu was dissolved by floating this stack over $FeCl_3$ etchant solution of 1M concentration for etching duration between 30 mins to 2 hrs. The etching process completed by washing the stack in flowing DI water for 5 minutes. Ti/Au electrodes of thickness 5/40 nm deposited using e-beam evaporation technique by making use of a plastic hard mask. The stack was diced into many samples of sensing structures using simple cutting using a scissor. The pressure enhancing stack consists of monolayer (ML) of silica NSs deposited over Si and PVC substrates. Nanosphere lithography (NSL) is a non-conventional technique which is used in this work to produce ML of silica NSs over PVC substrate. PVC substrate was needed to be converted into hydrophilic surface to increase wetting (contact angle) of suspension which leads to better anchoring of NSs. PVC surface was treated with mild oxygen plasma (Oxygen Barrel Asher - PlasmaFab 505) for 10-20 s. Monodispersed silica NSs in DI water was drop-casted over PVC surface which was kept in hot plate at 75°C for 5 minutes.

III. Results and Discussions

A. Structural characterization

Structural characterization of the sensing stack is characterized using Raman spectroscopy and scanning electron microscopy (SEM). The graphene transferred over PVC substrate by hot lamination technique is characterized by Raman spectroscopy. The lamination technique is advantageous as it did not require an adhesion interlayer for the successful transfer process. The lamination process carried out at temperature of 125ºC and solid contact pressure that ensures graphene layer adhered well with the PVC material. In addition, PVC becomes viscoelastic during lamination process as its glass transition temperature is 85ºC. A visual observation of the transparent PVC material shows contrast between graphene side of the PVC and bare back side.

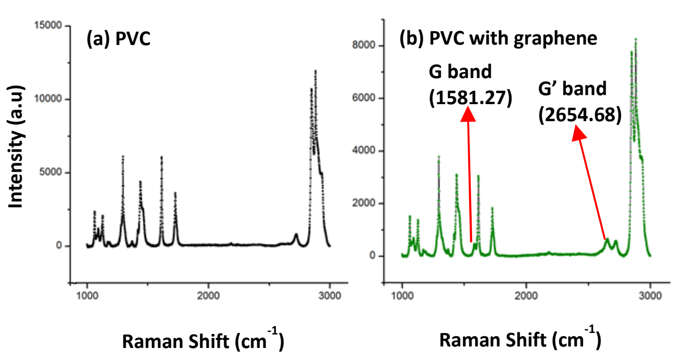

Fig.2. Comparison of Raman analysis of bare and graphene transferred over PVC. (a). Raman spectrum of bare PVC substrate. (b) Spectra of transferred graphene over PVC.

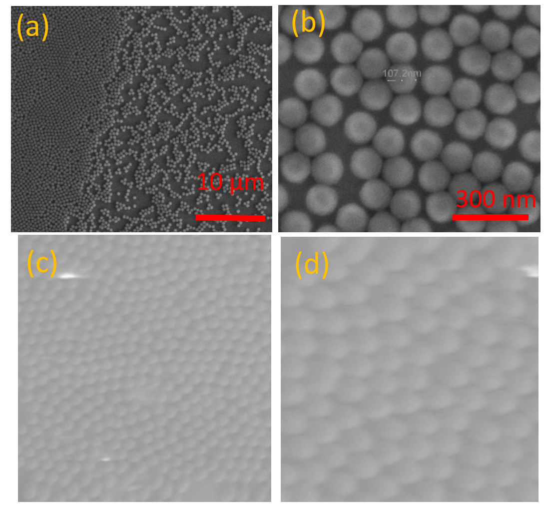

Fig.3. SEM images of the nano spheres assembled on Si and PVC substrates. (a-b). Nano spheres on Si substrate used as reference. (c-d). Monolayer of NSs assembled over PVC substrate by drop casting.

Raman tool Laser power is optimized to prevent the radiation damage. Fig.2 shows the Raman spectrum of the PVC with and without graphene layer for reference. The observation of Raman G and G´ (Fig.2b) bands confirms the presence of monolayer graphene layer. The diminished intensity of both the bands were attributed to high reflectivity of PVC to the incident photons. Scanning electron microscopy (FEI Nova NanoSem630) was used for characterization of self-assembled NSs over PVC substrate (Fig. 3). NSs of diameters ~500 (Fig.3a) and 100 nm (Fig.3b) on hydrophilic Si substrates have been shown for comparison. Study of NSs assembly on PVC is not reported previously by drop casted self-assembly method. SEM images evidently proves that the formation of large area monolayer of NSs over PVC substrate. The difference in contrast between Si/NSs and PVC/NSs arises due to the highly insulating nature of the PVC.SEM Imaging was carried out at 2 kV with thin Au layer over NSs. The NSs layer was inverted and placed over graphene to cover active sensing region.

B. Electrical characterization of the sensing structure:

Electrical characterization was carried using Agilent B1500A semiconductor device parameter analyser. The resistance of graphene transferred over PVC substrate was measured to be 4.4 kΩ which is higher compared to the graphene over Si/SiO_2 which is usually around 1-2 kΩ. This is attributed to enlarged voids or cracks during the lamination transfer process. This graphene was used for the fabrication of the proposed force sensing structure. The current-voltage characteristics of the sensing stack was studied under static loading conditions. Initial trials have been tested using graphene-NSs encapsulated structure. NSs were deposited over the monolayer graphene at 80ºC hotplate temperature. PDMS 20:1 composition was used to encapsulate the compact structure.

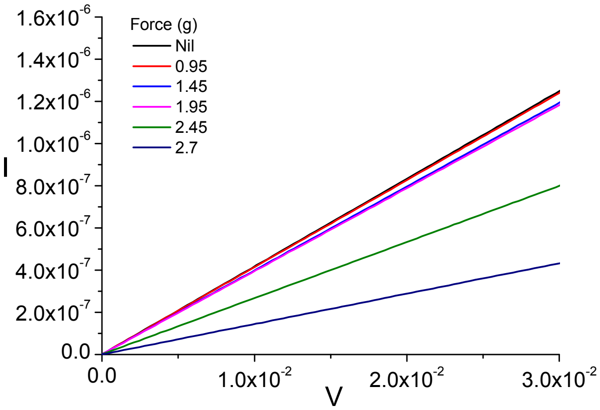

Fig.4. Electrical characteristics of the sensing structure under static loading conditions.

The choice of load was between 0.5 to 3 grams to evaluate the response to feather and soft touch. The electrical response of encapsulated stack was very poor as the applied force tend to absorbed by PDMS material. Further the measurements have been carried out in dual stack structure where the NSs on the PVC substrate made in contact with graphene during the measurement (Fig.4). The absorbance of the applied force is expected to be minimum as the load was applied over 85 µm thick PVC coated with NSs. Known weights in the range of 0.5 to 3 grams were added *in situ* during electrical probing. The current in graphene channel was observed to decrease an order within this applied load range. It is expected that the lateral strain in C-C bonds of graphene reduces resistance in channel. However, the chances for the void creation and crack enlargement also could cause change in resistance. The measurement is repeated with many different samples to verify the observation. Graphene layer was observed to be responsive to the applied external force. Currently, the stack is not robust to produce repetitive measurement under dynamic loading conditions. The work is a proof of concept to non-conventional NSs –graphene system for feather touch sensing device.

IV. CONLUSIONS

A graphene based simple force sensing structure is proposed for effective soft touch sensing applications. Monolayer of graphene is used as an active sensing layer in combination with silica NSs as lateral strain enhancers. Non-conventional processes such as hot lamination and soft lithographic techniques were employed in the fabrication process. This is viewed as a major advantage of this proposed design compared to the conventional graphene based sensing structures. Structural characterizations had ensured the presence of graphene and NSs monolayer. PDMS Encapsulated structure failed to produce electrical response due to force absorption. NSs acting as strain enhancing layer for the applied vertical force. The attachment of NSs over PVC was proven effective for many static measurements. However, permanent anchoring of NSs over PVC needs to be sought out for robust design. The whole structure is flexible and has potential for integrating over large area sensing system. The stack is observed to electrically respond for the soft touch within loading range of 0.5 to 3 grams. The lower sensitive limit of the structure needs to be identified to expand its potential. Effect of the NSs size with respect to the induced lateral stain needs to be elucidated.

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