Microelectronic Engineering 142 (2015) 7–11

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

A flexible strain sensor made of graphene nanoplatelets/polydimethylsiloxane nanocomposite

M.K. Filippidou, E. Tegou, V. Tsouti, S. Chatzandroulis *

Institute of Nanoscience and Nanotechnology, NCSR "Demokritos", Agia Paraskevi, Attiki 15310, Greece

ARTICLE INFO

Article history:
Received 24 October 2014
Received in revised form 20 May 2015
Accepted 26 June 2015
Available online 2 July 2015

Keywords:
PDMS
GNP
Graphene
Strain sensor
Flexible sensors
Conductive nanocomposites

ABSTRACT

Carbon-based materials are commonly used as fillers in polymer matrices for the fabrication of flexible physical and chemical sensors. Among the carbon materials, graphene has emerged as a promising next generation material replacing the traditionally used carbon black. In this work, we present the fabrication process of flexible graphene nanoplatelets/polydimethlylsiloxane (GNP/PDMS) capacitive strain sensor. In order to optimize the GNP/PDMS suspension, the dispersion of GNP in different solvents for the formation of a homogeneous suspension is examined by FT-IR spectroscopy. The sensor is fabricated by a low cost, easy to implement and rapid method using layer by layer spin-coating. The sensor exhibits linear capacitance change to small strains (≤0.2%).

1. Introduction

Strain sensors find applications in biomedical [1], aviation and automotive industry [2]. This type of sensor is used to detect mechanical deformations due to an applied load or to translate human body movements into signals. In most cases, strain sensors are based in resistive measurements. However, at high temperatures, these sensors exhibit problems such as temperature dependence, delamination and non-linear gauge behavior [3]. On the other hand, the response of the capacitive strain sensors is not seriously affected by temperature changes [3].

Several kinds of capacitive strain sensors have been reported in the literature. An interdigitated capacitive strain sensor from silver lines on several substrates to be used at high temperatures and harsh environments is presented by Li et al. [3]. The sensor exhibited nominal capacitance 11.3 pF. Matsuzaki and Todoroki [2] presented a wireless flexible capacitive sensor with interdigitated electrodes for the strain measurements of an automobile tire. Zeiser et al. [4] presented a capacitor with interdigitated electrodes on two flexible polymers foils, polyimide and a liquid crystal polymer and studied the effect of the substrate material and the dielectric properties of the substrate during the sensor operation.

Various applications reported in literature suggest that a flexible and easy to implement material is required as the sensor substrate [2,4]. Polydimethylsiloxane (PDMS), a silicone based elastomer, is a good candidate for sensors applications due to its flexibility, biocompatibility, low cost, low toxicity, thermal stability, optical transparency, low permeability to water, low electrical conductivity and ease of micropatterning [5]. Furthermore, it is also used in microfluidics [6] and in microelectromechanical systems (MEMS) [1,7]. Nevertheless, in order to be used as the active component in a flexible microsystem, PDMS should be transformed to a conductive material first.

To this scope, up to date various methods have been proposed in the literature, that apply metal deposition on PDMS [3]. These methods usually present defects such as cracks. Carbon fillers added to polymer matrices have emerged as promising alternatives in order to generate conductive polymers. The fillers commonly used comprise carbon black, carbon nanotubes, and graphene [5,6,8–15]. More specifically, graphene, being a one-atom-thick planar sheet of sp2 bonded carbon atoms in a honeycomb crystal lattice, offers remarkable electronic, optical, mechanical, thermal, and electrochemical properties [16]. Thus, it has emerged as a promising next generation material replacing the traditionally used carbon black. Lee et al. [14] presented PDMS flexible strain sensors based on the combination of multi wall nanotubes and/or graphene nanoplatelets nanocomposites. Moreover, Wang et al. [5] used a graphene/PDMS nanocomposite for the fabrication of resistive strain sensors, while Bae et al. [12] used graphene for the fabrication of transparent flexible strain sensors.

A crucial issue in the preparation of the conductive nanocomposites is the dispersion of the carbon filler in the polymer matrices. A good dispersion results in better electrical properties of the nanocomposite [17,18], enhancing its electrical conductivity. For this reason, numerous reports exist in the literature addressing the critical parameters involved in the preparation of the nanocomposite using conductive carbon fillers

* Corresponding author.
E-mail address: stavros@imel.demokritos.gr (S. Chatzandroulis).

http://dx.doi.org/10.1016/j.mee.2015.06.007
0167-9317/

[18–21]. Among various methods reported [22], solvent blending leads to lower percolation thresholds, closely followed by in situ polymerization since these two methods usually enable better dispersions than the melt compounding method [21,23].

For the fabrication of strain sensors, several techniques have been developed, such as screen printing [24], inkjet printing [25] and laser micromachining [3], yet these techniques are usually labor intensive. In this work, we employ layer by layer spin-coating in order to fabricate flexible graphene nanoplatelets/polydimethlylsiloxane (GNP/PDMS) capacitive strain sensor. For the optimization of the GNP/PDMS suspension, the dispersion of GNP in different solvents for the formation of a homogenous suspension is examined by FT-IR spectroscopy. The operation of the sensor relies on the expansion (contraction) under tensile (compressive) strain of the two capacitor electrode plates and the corresponding thickness changes of the intermediate PDMS layer between the two plates, which both result in a change in sensor capacitance. In general the response of the sensor, for small strain values or a small poisson effect, is linear and is given by the following:

$$\Delta C = C_{stretch} - C_0 = \varepsilon_0\varepsilon_r \frac{(1-\nu\varepsilon_x)w(1+\varepsilon_x)L}{(1-\nu\varepsilon_x)d} - \varepsilon_0\varepsilon_r \frac{wL}{d} = \varepsilon_x C_0 \quad (1)$$

where

$C_{stretch}$ is the capacitance of the sensor when stretched,
$C_0 = \varepsilon_o\varepsilon_r \frac{wL}{d}$ is the initial capacitance, d is the electrode distance, L is the initial length w is the initial width of the capacitor electrodes, ε_r is the relative permittivity of the material, $\varepsilon_o = 8.854$ pF/m is the permittivity of vacuum, and
$\varepsilon_x = \frac{\Delta L}{L}$ is the strain applied parallel to x-axis, and ν is the Poisson's ratio.

2. Materials and methods

2.1. Materials

Polydimethylsiloxane (PDMS) SYLGARD 184 silicone elastomer was purchased from Dow Corning GmbH and was used for the fabrication of a) the sensor and b) the conductive material. The conductive filler, graphene nanoplatelets (GNP, 2–10 nm), was purchased from ACS Material. Cyclohexane was purchased from Merck KGaA (M: 84.16 g/mol) and was used as a solvent for the PDMS. Isopropanol was purchased from BASF and was used as the dispersing medium for the GNP fillers.

2.2. Characterization methods

In this section all the characterization methods used in the fabrication of the nanocomposite and the sensor are reported.

2.2.1. Morphology and thickness characterization

The nanocomposite morphology was characterized with JEOL JSM-7401f FESEM. The thickness of the polymer nanocomposite films was measured with Ambios XP-2 profilometer.

2.2.2. Electrical measurements

The I–V curves of the nanocomposite were measured with Prober Karl-Suss Micromanipulator 7000 LTE. In this way the conductivity curve of the nanocomposite was obtained.

2.2.3. FT-IR spectroscopy

For the collection of FT-IR spectra, a Bruker Tensor 27 FT-IR spectrophotometer, equipped with ZnSe windows, at 4 cm^{-1} resolution at 64 scans.

2.2.4. Capacitance measurements

Capacitance measurements were done to evaluate the sensor response to different loadings. The sensor capacitance response to strain was measured with a HP 4278A Capacitance Meter at 1 MHz.

2.3. Preparation of GNP/PDMS nanocomposite

In order to prepare the GNP nanocomposite suspension, the as-received GNP (ranging from 0.03 g to 0.2 g) and 5 mL of IPA/cyclohexane (30:70) are added into a glass beaker and sonicated for 30 min. A predetermined amount of PDMS base (0.91 g) is then added and the mixture is placed in an ultrasonic bath in order to produce filler concentration ranging from 3 to 20 wt.%. After 10–12 h ultrasonication the mixture is magnetically stirred for another 17 h. Then, PDMS curing agent (0.091 g) is added, in a ratio of 10:1 elastomer base to curing agent. Finally, the suspension is stirred until half of the volume is evaporated to achieve optimum viscosity of the nanocomposite.

2.4. GNP/PDMS sensor fabrication

PDMS and GNP are the main materials used in the device in a nine step fabrication process. The process flow for the fabrication of the device is shown in Fig. 1. The sensor is assembled on a silicon wafer. First, a Teflon layer is spin-coated (Fig. 1a) for easier detachment of the device from the silicon wafer substrate. Then a PDMS layer is spin-coated at 1000 rpm for 10 s and cured for 1 h at 100 °C at a thickness of 244 μm (Fig. 1b). Next, pieces of copper tape, to be used as contacts, are glued on the PDMS layer using PDMS adhesive, and baked for 45 min at 100 °C (Fig. 1c). Afterwards, pieces of scotch tape are used to cover parts of the PDMS layer leaving windows of uncovered areas into which the GNP/PDMS nanocomposite (750 μm thick) will be allowed to reside after spin-coating (Fig. 1d). In the next step (Fig. 1e), the scotch tapes are removed, effectively patterning the GNP/PDMS nanocomposite into two rectangles which will later constitute the two capacitor electrodes of the sensor. Immediately after patterning, the device is left to degas in a vacuum chamber for 20 min to remove any excessive solvent solution and finally baked for 30 min at 100 °C. After degassing, a top shielding layer of PDMS is spin coated at 1000 rpm for 10 s and cured for 1 h at 100 °C (Fig. 1f) and the sensor is detached from the Teflon coated Si substrate (Fig. 1g). Finally, the sensor is cut in two pieces (Fig. 1h): each piece will be used as one of the capacitor plates. The two plates are glued together using PDMS (Fig. 1i) and the device is baked for 1 h at 100 °C.

2.5. Experimental set-up

A custom made set-up is employed for the strain measurements of the flexible sensor (in-house). The sensor is mounted, using a double sided tape, on a cantilever beam whose one end is steadily fixed and the other is free to deflect under load as shown in Fig. 2.

A micrometer is used to deflect the cantilever perpendicularly to its axis. From the position of the sensor on the cantilever and the cantilever beam geometry the induced strain ε, is obtained from the following:

$$\varepsilon(x) = \frac{3hx}{2L^3} y \quad (2)$$

where h is the thickness of the cantilever beam, L is its length from the fixed end to the micrometer rounded tip, y is the perpendicular deflection and x is the distance of the sensor from the micrometer.