



Quasi in-situ observation of the elastic properties changes of the graphene–low-density polyethylene composites

Krzysztof Gajewski^{a,*}, Witold Szymański^b, Witold Kaczorowski^b, Piotr Niedzielski^b, Teodor Gotszalk^a

^a Wrocław University of Science and Technology, Faculty of Microsystem Electronics and Photonics, ul. Z. Janiszewskiego 11/17, Wrocław PL-50372, Poland

^b Łódź University of Technology, Institute of Materials Science and Engineering, ul. Stefanowskiego 1/15, Łódź PL-90924, Poland

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ABSTRACT

In this paper we present the results of electromechanical studies of graphene–low-density polyethylene (LDPE) composites. Two types of graphene were transferred onto LDPE using PMMA support: graphene grown by physical vapor deposition on liquid metallic matrix (so called high strength metallurgical graphene, HSMG) and commercial chemical vapor deposited (CVD) graphene. Raman spectroscopy was used for preliminary characterization of graphene, whereas Kelvin probe force microscopy (KPFM) combined with electrical measurement setup and symmetrical stretching stage was used for observations of the electrical properties changes of elongated graphene. Maximum elongation of graphene–LDPE composites were 10%. Resistance of the HSMG graphene was changing from 130 kΩ up to 900 kΩ for HSMG graphene and from 100 kΩ up to 300 kΩ for CVD graphene. Observed resistance changes were compared with contact potential difference recorded by KPFM. KPFM showed that resistance changes can be attributed rather to the structural discontinuities for the CVD graphene sample. In case of the HSMG graphene sample such behavior was not observed.

1. Introduction

Graphene, the first representative of 2D materials family is still extensively studied by the scientists [1–3]. It is related not only to its outstanding properties [4–7], but also potential use in modern devices like van der Waals heterostructures [8], or elastic and transparent electronics [9–11].

Materials like polymers, textiles, and so on are used as a support [12–16] for elastic electronics. Due to the high quality material needed for electronics, needs of high scalability, and mass production, physical as well as chemical vapor deposition (PVD, CVD) processes has to be used [17] for graphene growth. But, these processes are incompatible for direct growth on elastic substrates (especially on polymers due to their low temperature of decomposition [18]). For that reason transfer processes are needed to investigate graphene onto elastic base. Graphene transfer can be done with [19,20] or without [21,22] additional support. After every technology step performed onto active layer, a material characterization has to be done. In case of 2D materials, Raman spectroscopy and scanning probe microscopy (SPM) related techniques are very helpful, e.g. conductive atomic force microscopy [23–25], magnetic force microscopy [26,27], scanning thermal microscopy [28,29], and Kelvin probe force microscopy

(KPFM) [30,31]. Combining both techniques it is possible to obtain information about electromechanical properties of investigated materials with nanometer resolution.

Y. Wang et al. using just Raman spectroscopy concluded, that graphene properties were almost no affected by various type of substrates [32]. They observed, that only silicon carbide (SiC) modified the Raman spectra of graphene. It was due to strain caused by the covalent bonding between SiC substrate and epitaxial graphene. This interaction has to be much higher than van der Waals forces between graphene and substrates like SiO₂ or PDMS. R. Wang et al. stated contrary conclusion [33]. Using Raman spectroscopy and KPFM they observed graphene interactions with substrates like PMMA, SiO₂, and SiO₂ modified by silanes. They showed, that by the use of different substrates it is possible to modify doping level and work function of graphene. But, R. Wang et al. investigated smaller set of substrates than Y. Wang et al. Liu et al. observed graphene oxide–poly(3-hexylthiophene) (P3HT) nanofibers interactions combining KPFM and Raman spectroscopy [34,35]. P3HT had different nanostructure controlled by solvent quality and concentration. Liu et al. concluded, that stronger interactions are present between nanofibers than between nanofibers and graphene oxide. Nevertheless, they used Si as a substrate, so they were unable to observe electromechanical interactions in their nanostructures.

* Corresponding author.

E-mail address: krzysztof.gajewski@pwr.edu.pl (K. Gajewski).

Despite many works appeared about graphene composites and graphene deposited on polymers [36–38], to our knowledge there is lack of investigations showing electrical behavior of graphene on polymers subjected to mechanical elongation. Here, He et al. use stretching stage to tune the CPD of elongated graphene [39]. They obtained 0.161 eV change for 7% strain. This result was in the agreement with theoretical predictions [40,41]. Nevertheless, the obtained results was too much localized (small scan area) and they observed relatively large CPD difference between the minimum and the maximum of the observed area.

In this paper we present the results of electromechanical studies of graphene–low-density polyethylene (LDPE) composites. Two types of graphene were transferred onto LDPE using PMMA support: graphene grown by physical vapor deposition on liquid metallic matrix (so called high strength metallurgical graphene, HSMG) and commercial chemical vapor deposited (CVD) graphene. Raman spectroscopy was used for preliminary characterization of graphene, whereas Kelvin probe force microscopy (KPFM) combined with electrical measurement setup and symmetrical stretching stage was used for observations of the electrical properties changes of elongated graphene. Maximum elongation of graphene–LDPE composites were 10%. Resistance of the HSMG graphene was changing from 130 k Ω up to 900 k Ω for HSMG graphene and from 100 k Ω up to 300 k Ω for CVD graphene. Observed resistance changes were compared with CPD recorded by KPFM. KPFM showed that resistance changes can be attributed rather to the structural discontinuities for the CVD graphene sample. In case of the HSMG graphene sample such behavior was not observed.

2. Materials and methods

2.1. Samples preparation

Two distinct types of graphene were investigated. The first one was commercially available CVD graphene (Graphene Laboratories Inc.). The next one was HSMG produced using Kula et al. method [42]. In this method copper/nickel composite (substrate, 72% Cu, 28% Ni) was initially pre-heated above its melting temperature (1200–1250 °C) in argon protective atmosphere (constant pressure of 100 kPa). Substrate was kept at this temperature range for 1 min to decrease amount of defects which could influence the graphene growth. Next, the substrate was cooled to the temperature of 1050 °C at the cooling speed of 0.5 °C/min. At this time, the mixture of acetylene, hydrogen and ethylene at the partial pressure of 3 kPa was injected into the chamber. More detailed procedure of HSMG growth can be found elsewhere [43,44].

Graphene transfer was done using the modified method with PMMA as a support for graphene. Graphene on copper/nickel substrate was coated by PMMA dissolved in chlorobenzene (46 mg/ml) and dried at 50 °C. Next, Cu/Ni substrate was etched in iron chloride water solution (1 M FeCl₃) at 50 °C for 24 h. After this time, PMMA foil with graphene was extensively washed in deionized water. Then, PMMA/graphene foil was transferred onto LDPE substrate. To obtain a good adhesion of graphene to LDPE, sample was placed on a hot plate and heated up to 70 °C. During heating PMMA foil with graphene was gently pressed to LDPE substrate. Next, the PMMA foil was dissolved by submerging in acetone for 24 h. During PMMA dissolution acetone was exchanged five times.

Raman spectra of LDPE expose background and mode frequencies in proximity of the graphene D, G, 2D bands. This could makes the direct analysis of graphene on LDPE substrates troublesome. For that reason Raman spectroscopy was performed on benchmark graphene samples transferred onto glass substrates. They were prepared by the same way described above. Nevertheless, Raman spectra of graphene–LDPE composites before and after stretching can be found in the supporting materials.

Electrical contacts to the CVD and HSMG graphene transferred onto LDPE were done using a silver conductive paint (Silver Conductive

Adhesive 503, Electron Microscopy Sciences). After electrical contacts deposition, samples were cut to form approximately 5 mm \times 25 mm stripes to fit to the sample holder on the symmetrical stretching stage. Active graphene area was approximately 6.5 mm \times 5.5 mm.

2.2. Raman spectroscopy

inVia confocal micro-Raman spectrometer (Renishaw) equipped with a 532 nm laser was used for Raman spectroscopy. In order to avoid sample heating and destruction of the samples, power of the laser was maintained below 1 mW. Laser spot diameter was approximately 500 nm. Spectra were collected through the \times 100 objective. Obtained spectra were deconvoluted in a Peakfit 4.11 software. Graphene properties like number of graphene layers, doping and strain present in the structure were extracted based on the Raman peaks shape, their mode frequencies as well as relative positions [45,46].

2.3. Atomic force microscopy - sample stretching and Kelvin probe force microscopy

Commercial Veeco/Bruker Nanoman V with a Nanoscope V controller was used for observation of the sample stretching process. KPFM measurements were done using PPP-EFM probes (Nanosensors, spring constant 2.8 N/m, resonant frequency 75 kHz) using a two-pass procedure. Each scan line was scanned twice - at the first scan topography of the sample was collected. During the second scan CPD between sample and scanning tip was measured. The measurement setup of the microscope is presented in Fig. 1.

AFM microscope was equipped with a home-made symmetrical stretching stage [47]. Sample was mounted on the stretching arms and the AFM scanning head was set in the middle of them. Even if the imaged area was suspended between the stretching arms, imaged samples weren't vibrating in any way which could influence overall measurements. Each sample was stretched 5 times and relaxed up to its initial length. Relative elongation of the sample was calculated by the measurement of the distance between selected details on each KPFM image after each elongation/relaxation process. Between each elongation/relaxation steps and KPFM imaging sample resistance was measured using a Keithley 2000 multimeter.

Influence of the resistance change of the silver contacts during stretching was also estimated. To do so, LDPE stripe was fully covered by the silver paint. Initial resistance of such stripe was approximately 2 Ω . This resistance increased up to tens ohms after \sim 20–30% elongation. Taking into account, that electrical contacts of graphene/LDPE

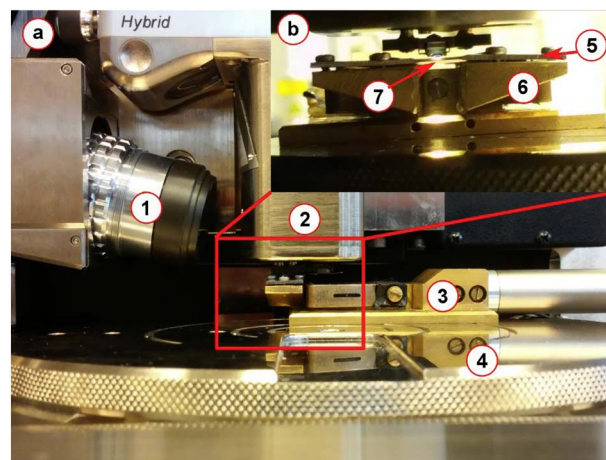


Fig. 1. AFM setup for the sample stretching experiments. a) Side view, b) front view. Numbers on the image show - 1) AFM optical camera, 2) AFM head, 3) sample stretching stage, 4) movable sample stage of the AFM microscope, 5) sample holder, 6) stretching arms, and 7) investigated sample.

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