



Frictional behaviour of polycrystalline graphene grown on liquid metallic matrix

Lukasz Kolodziejczyk*, Piotr Kula, Witold Szymanski, Radomir Atraszkiewicz, Konrad Dybowski, Robert Pietrasik

Institute of Materials Science and Engineering, Lodz University of Technology, Stefanowskiego 1/15, 90-924 Łódź, Poland

ARTICLE INFO

Article history:

Received 17 July 2014

Received in revised form

10 October 2014

Accepted 2 December 2014

Available online 10 December 2014

Keywords:

Polycrystalline graphene

Friction

Lateral force microscopy

ABSTRACT

In this work friction characteristics of polycrystalline graphene grown on the metallic matrix from the liquid phase via new metallurgical method were investigated using atomic force microscopy under the range of applied normal loads. Polycrystalline graphene films were transferred onto silicon oxide using a modified polymer supported transfer process. Lateral force microscopy mode with both silicon and diamond-like carbon coated probes was used to measure the friction properties. Ogletree method was employed in order to calibrate the lateral force and estimate the average friction coefficient of polycrystalline graphene. As a comparison the friction of SiO₂ surface, commercially available CVD grown graphene monolayers transferred onto SiO₂ as well as highly oriented pyrolytic graphite were also measured under the same experimental conditions.

© 2014 Elsevier Ltd. All rights reserved.

1. Introduction

During last several years the number of methods for graphene production has significantly increased. Some of these include mechanical [1–3] or chemical [4] exfoliation, CVD [5,6] or epitaxial growth [7], arc discharge, the reduction of graphene oxide [8,9], and many others. Li et al. [10] found that low-pressure CVD synthesis of graphene on Cu foil provides a good way of fabricating uniform single-layer graphene films. They showed that the continuous films are formed by connecting randomly oriented, irregular-shaped, and micrometre-sized graphene flakes. Employing a liquid matrix has been proposed as effective means for controlling the nucleation process in CVD graphene production [11]. It completely removes the grain boundaries in polycrystalline solid, allowing a uniform distribution of graphene nucleation sites, and enabling self-assembly of graphene flakes into compact and ordered structures.

Based on this phenomena a new method of manufacturing of polycrystalline graphene from liquid matrix via metallurgical process has been proposed [12,13].

Frictional behaviour of graphene has gained recent attention [14], with several studies demonstrating the frictional anisotropy

of graphene grains primarily due to the development of wrinkles. Graphene has been found to lower friction when compared to bare substrates. This general result was found in case of exfoliated, epitaxially-grown, or CVD grown graphene supported on several substrates (e.g. copper, nickel, silicon dioxide). However, compared with the works on general properties of graphene [15], the tribological investigation of graphene is still quite limited. Additionally to wrinkles formation the friction of graphene is greatly affected by graphene-to-substrate adhesion and number of graphene layers [15,16]. Theoretical simulations predicted that the low friction of graphene is highly dependent on the sliding interfaces (whether the tip is sliding on the graphene surface or two graphene surfaces are sliding against each other) and on the number of graphene layers. The modelling of the AFM tip-graphene interactions [17] demonstrated that the friction force decreases as the number of layers increases. The great majority of experimental studies of graphene tribology at the nano-scale have used lateral force microscopy (LFM) [18–22]. The LFM measurements demonstrated the friction dependence on the number of graphene layers [19,21,23] in agreement with the predicted simulation results. Li et al. [23] performed LFM studies in ambient conditions using silicon probes. Both free-standing graphene and graphene supported on SiO₂ showed a decrease in friction with number of layers due to the puckering effect. However, observed thickness dependent friction is not present for graphene strongly bonded to the substrate (e.g. mica) [23].

The main idea of the presented work is the evaluation of frictional characteristics of the metallurgical graphene obtained

* Corresponding author. Tel.: +48 42 631 30 48.

E-mail addresses: lukasz.kolodziejczyk@p.lodz.pl (L. Kolodziejczyk), piotr.kula@p.lodz.pl (P. Kula), witold.szymanski@p.lodz.pl (W. Szymanski), radomir.atraszkiewicz@p.lodz.pl (R. Atraszkiewicz), konrad.dybowski@p.lodz.pl (K. Dybowski), robert.pietrasik@p.lodz.pl (R. Pietrasik).

by new technology of graphene growth on liquid metallic matrix. The evaluation has been done with reference to the graphene produced by commonly known CVD method. Additionally, complete characterisation of metallurgical graphene has been conducted, including qualitative and quantitative analysis with the use of optical, scanning and atomic force microscopy and Raman spectroscopy.

2. Materials and methods

2.1. Samples preparation

In the presented work following materials were used: highly oriented pyrolytic graphite (HOPG) grade ZYH (Bruker Corporation, USA), silicon dioxide on (1 0 0) p-doped silicon with thermal oxide thickness of 300 nm (Semiconductor Wafer Inc., Taiwan), single layer CVD graphene (latter in the text abbreviated as CVD-G/SiO₂) already transferred onto 285 nm thick SiO₂ (Graphene Laboratories Inc., USA) and multilayer polycrystalline graphene grown on liquid metallic matrix (referred as metallurgical graphene and abbreviated as M-G/SiO₂) [24]. CVD graphene films were transferred onto SiO₂ using a PMMA assisted transfer method [6,25]. Polycrystalline graphene production consisted of following steps. First, the multilayer metallic substrate, the copper/nickel composite (72% Cu, 28% Ni) [24,26], was heated to the temperature of 1200–1250 °C in argon protective atmosphere at the constant pressure of 100 kPa. Then, the substrate was kept within this range of temperature for 1 min. Afterwards, the plate was cooled to the temperature of 1050 °C at the cooling speed of 0.5 °C/min, also in argon atmosphere at the constant pressure of 100 kPa. During the first two aforementioned thermal stages, the mixture of acetylene, hydrogen and ethylene in the proportions of 2:1:2 [27], at the partial pressure of 3 kPa, has been simultaneously introduced into the process chamber [12,28].

For the polycrystalline graphene grown on liquid matrix the modified method of graphene transfer from metallic substrates on thin PMMA film has been employed. This method and its variations are most often utilized in procedures for graphene transfer on any substrate (Si/SiO₂, glass, PE, etc.) [6,29–31]. Graphene on nickel/copper substrate was coated with PMMA in chlorobenzene (46 mg/ml). The thickness of the etched Ni/Cu substrate was 0.3 mm. Surface prepared in this way was dried at 50 °C for 1 h. PMMA/Ni/Cu stack was placed in a FeCl₃ solution (1 M) for 24 h. Occasionally, prepared PMMA foil was covered with the residues of FeCl₃ on the free side (without graphene). In that case PMMA foil (only free side) was rinsed in concentrated HF solution and 5% H₂O₂ and then in deionized water. Prepared PMMA foil with graphene, prepared in this way, was ready to follow the procedures for the transfer of graphene to any substrate. Selected piece of dH₂O wetted foil was placed on the SiO₂ target substrate. The entire part was placed on a hot plate and heated to a temperature of about 70 °C. After stretching the foil on the substrate the setup substrate-foil-graphene was immediately transferred into the atmosphere of boiling acetone vapour for about 10 min. Then, the sample was removed and cooled down. Such a procedure ensured good adhesion of graphene to the substrate. Next, the PMMA foil was dissolved by submerging it in acetone for 24 h (four times exchanging the solvent). Polycrystalline graphene samples prepared using above described transfer method were then ready for investigation.

2.2. Materials characterization

2.2.1. Optical and scanning microscopy

JEOL JSM-6610LV scanning electron microscope integrated with MiniCL-GATAN Cathodoluminescence Imaging and Oxford

Instruments systems was used for analysis of the possible AFM tip wear. Hitachi scanning electron microscope S-3000M, working in SE mode at accelerating voltage of 5 kV, was used for qualitative analysis of metallurgical graphene layers morphology. The quality of graphene layers after the transfer process onto SiO₂/Si substrate was studied using an optical microscope Nikon Eclipse MA 200. Acquisition of the optical images was obtained using Nis Elements BR software.

2.2.2. Raman spectroscopy

For every graphene sample on SiO₂ substrate the Raman spectroscopy using Ntegra Spectra Solar spectrometer (NT-MDT, Russia) was conducted. The Raman scattering in the range of 1000–3000 cm⁻¹ using visible –2.33 eV (532 nm) excitation of Ar⁺ laser was studied. The laser power held at 3.2 mW did not cause any changes on the surface (local heating and damage of the samples). The spectra were registered using NovaPX software (NT-MDT). Data processing was performed with use of PeakFit software. The spectra were deconvoluted using Gauss–Lorentz curve. Obtained peaks intensities and half-widths were used for the calculation of characteristic peak ratios of graphene.

2.2.3. Atomic and lateral force microscopy

Surface morphology, topography and lateral force measurements were measured using Multimode atomic force microscope equipped with Nanoscope V controller (Bruker Corporation, USA). All investigations were performed under ambient conditions. Prior to topography measurements and LFM experiments the areas of 500 × 500 nm were scanned (up to several times in one scanning direction along slow scan axis) in contact mode at the very small loads (2–3 nN) to clean the surface from mainly polymer deposits remaining after graphene transfer process and move them outside measurements' area.

Topography measurements were made in contact mode at applied load of ca. 2 nN and the size of the images were 500 × 500 nm. The images were shaded in to enhance contrast. Commercial silicon cantilevers type HQ:CSC15 (MicroMasch, Estonia) with nominal tip radius ~8 nm and nominal cantilever spring constant of 0.3–0.8 N/m were used. Image acquisition was performed with use of Nanoscope 7.3 software and further image processing was done using Nanoscope Analysis 1.5 (Bruker Corporation, USA) and MountainsMap Premium 5.0 (Digital Surf, France) software. From the topography images commonly used roughness parameters – the average roughness (R_a) and root-mean square roughness (RMS) – of the samples were defined (average values taken from 512 surface profiles). The error was calculated as the standard deviation among all surface profiles.

For friction measurements the smaller areas of 100 × 100 nm within previously cleaned zone were used. The average friction measurements were done at increasing loads up to ~20 nN (10 normal loads ramping over the single image) at 1 Hz scan rate. For friction tests two different types of AFM tips: commercial silicon cantilevers type HQ:CSC15 (same as for topography images) and diamond-like carbon (DLC, hydrogen-free ta-C) coated probe cantilevers type HQ:XSC11 (MicroMasch, Estonia) with resulting tip radius ≤ 20 nm and nominal cantilever spring constant of 0.2 N/m were used. Precise value of cantilever spring constant was specified using thermal tune method before each measurement. Both, deflection sensitivity of the cantilever and adhesion of the tip to the scanned surface were determined from force–distance measurements. The friction tests were carried out in air at temperature 21–23 °C and relative humidity 39–42%. At least seven results from measurements under same load were taken to calculate friction coefficient for single LFM test, and at least six LFM tests using same type AFM tip were performed on each

Download English Version:

<https://daneshyari.com/en/article/614231>

Download Persian Version:

<https://daneshyari.com/article/614231>

[Daneshyari.com](https://daneshyari.com)