

Fracture mechanics of diamond-like carbon (DLC) films coated on flexible polymer substrates

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Abstract

Diamond-like carbon (DLC) films have been widely used for many industrial applications due to their outstanding physical properties such as high hardness, wear resistance and biological compatibility. The DLC films coated on polymer substrates have also been extensively used and investigated because recently, quite a few applications for the use of these polymer–DLC composites have been proposed and actively discussed. The applications range from DLC-coated Polyethylene Terephthalate film (DLC-PET), through DLC-coated Polycarbonate (DLC-PC) to other DLC-coated rubbers. In this work, thin DLC films coated on several polymer substrates possessing different chemical structures and Young's moduli were introduced. The DLC-polymer films were stretched to different strains and the extended surface was investigated by optical microscopy and scanning electron microscopy (SEM) to study the fracture mechanics of the DLC-coated polymer films. Horizontally and vertically aligned micro-cracks and micro-buckling were observed, constructing periodic lattice-like fracture patterns on the surface of the extended DLC-polymer films. It was found that the lattice patterns were significantly influenced by Young's moduli of polymer substrates and DLC films, and that the patterns were also dependent on the adhesion between the DLC films and the polymers.

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1. Introduction

Recently the demand for polymeric materials has been increasing and highly functionalized polymers are much desired for wider applications such as flexible materials with high gas barrier properties. In order to functionalize polymers, Diamond Like Carbon (DLC) coating is one of many useful approaches that are industrially broadly used for the surface modification. DLC is characterized by many unique properties, including high hardness, high wear resistance, low friction coefficient, chemical inertness and high gas barrier properties, which are used as commercial materials including DLC-coated PET bottles, improved tribological rubber surfaces and highly biocompatible polymers especially for medical purposes [1,2]. However there are obvious structural differences between

polymers and DLC, particularly on a microscopic scale. Polyethylene (PE), the most fundamental C–H based polymer, consists of plenty of two dimensional macromolecules partly connected to each other by crystallization (i.e. through relatively weak van der Waals force) and the distance between two molecules are typically ~ 2.5 , ~ 4.9 and ~ 7.4 Å, while DLC are intrinsically three dimensional carbon networks (i.e. connected through strong chemical bonding force) and the typical distance between two carbons is 2.5 Å, which is shorter (i.e. denser and heavier) than PE. Considering the use of the composite materials made of polymers and DLC, such structural differences between the two materials cannot be neglected and hence the resulting composite presents complex physical properties required to be investigated systematically. However, there have been few reports that have dealt with the structural and mechanical behavior of DLC films coated on polymer materials, particularly when the polymer–DLC composites are mechanically and cooperatively deformed.

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Fracture behavior of thin hard films coated on metals has been investigated by several groups. Shear strength and crack surface were observed by Agarawal et al. on copper coated by thin ceramic film [3]. They found transverse cracks when mechanically stretched. Ye et al. introduced three types of cracking processes on thin films due to residual strain in brittle substrates and weak interface between the films and substrates [4]. Chen et al. observed periodic cracking on thin TiN coated on stainless steel [5], while Ogwu et al. examined similar periodic cracking on thin DLC film coated on stainless steel when exposed to biological fluids [6]. Fracture surface and characterization of mechanical properties of DLC film were discussed by Choi et al., Cho et al. and Aoki et al. [7–9].

The deformation of the substrates becomes more significant as well as problematic, when softer polymeric materials are used as substrates instead of traditionally utilized hard metals. There have been a few papers that dealt with polymer–DLC composites. Ollivier et al. assessed the adhesion between DLC films and Polyethylene Terephthalate (PET) films through simple tensile testing and surface microscopy [10]. Aoki and Ohtake improved the wear resistance of DLC films by using segment-structured DLC films on aluminum substrates [9]. They developed this method after they studied the morphology of the DLC surface that presented a segmented surface (i.e. lattice-like fracture) automatically generated when DLC was coated on polymers. The composites of softer materials (i.e. polymers) and harder materials (i.e. DLC) should present complex physical behavior and when they are deformed by mechanical tension, the fracture properties of thin DLC films should be strongly dependent on the chemical and physical properties of both coated polymer substrates and DLC films. In this paper, we focused on the moduli of materials, of both polymers and DLC, and investigated how the resulting polymer–DLC composites act in terms of fracture behavior when they were mechanically deformed. The moduli of materials were changed by choosing different materials possessing different Young's moduli. Two different polymers with similar Young's moduli but with different adhesion to DLC films were selected to investigate the adhesion effects of polymer–DLC composites on fracture properties. Periodic fracture patterns of thin DLC films coated on soft polymers have been observed, showing lattice-like fracture surfaces of the DLC. It was also found that the periodicity and mechanism of fracture surfaces were strongly related to the moduli of polymers and DLC as well as the adhesion between both materials.

2. Experimental

2.1. Sample preparations

The following polymers were used in the experiments: Polyethylene (PE), Polyethylene Terephthalate (PET), Polystyrene-block–Polyisoprene-block–Polystyrene Copolymer (SIS), Polydimethylsiloxane (PDMS) and Polypropylene (PP). PDMS at “Silastic Medical” grade were obtained from Dow Corning Toray Corporation, already cut in shape for the experiments.

Other polymers were molded by compact size heat press equipment (AH-2003, As One Corporation) from polymer pellets purchased from Aldrich Chemical Corporation (PE: high-density polyethylene #181900, PP: isotactic #427896, SIS: 17 wt.% of polystyrene #432407). The molding temperature was set at temperature approximately 20 °C above the melting points or the glass transition temperatures if any, of the materials. The melting points of PE, PET and PP are 134 °C, 253 °C and 164 °C respectively and the glass transition temperature of SIS is ~ 90 °C, all measured using differential scanning calorimetry (DSC: DSC822e of Mettler Toledo Corporation). Young's moduli of the polymers were measured using tensile testing machine of AG-50NIS MS of Shimadzu Corporation: Young's moduli of PET, PP, PE, PDMS and SIS are 1500 MPa, 600 MPa, 460 MPa, 6 MPa and 1.4 MPa respectively. The detail of the mechanical testing results can also be referred to in our coming paper [11].

DLC films were deposited on polymer surfaces by the radio frequency (rf) plasma enhanced chemical vapor deposition (RF-CVD) method (PD-10, Samco Corp.) where rf power was 200 W, deposition time was 15 s and the process gases were changed according to the moduli of the targeted DLC films: acetylene (C₂H₂) was used at 13 Pa of air pressure to synthesize usual DLC and the compound gas of C₂H₂ (5.2 Pa) and C₂F₆ (7.8 Pa) was used to synthesize flexible fluorinated DLC (F-DLC: fluorinated DLC with atom ratio F/C = ~ 12%). Fig. 1 shows the result of the Raman spectroscopy scanned through DLC coated on HDPE. The G-peak and the D-peak were detected at 1371.3 cm⁻¹ and 1607.8 cm⁻¹ respectively. The ratio of the intensity of the D peak $I(D)$ and G peak $I(G)$ was $I(D)/I(G) = 1.28$, revealing that the coated film was DLC. The Young's modulus of DLC was ~ 100 GPa and the Young's modulus of F-DLC was ~ 13 GPa. The thickness of the polymer substrates was ~ 0.1 mm, and the thickness of DLC films were ~ 50 nm. The polymer–DLC composites, i.e. the DLC coated polymers, were cut into 30 × 5 mm in length and width. They were then stretched to different strains and the

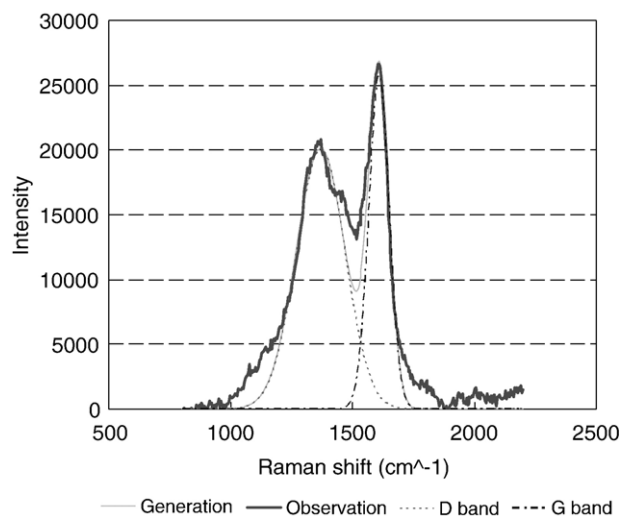


Fig. 1. Raman spectroscopy results of diamond like carbon (DLC) film coated on HDPE.

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