



Nanoindentation and thermal study of polyvinylalcohol/graphene oxide nanocomposite film through organic/inorganic assembly



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ABSTRACT

In this study, hydrogen-bonding interactions between polyvinylalcohol (PVA) and graphene oxide (GO) were utilized as the driving force to fabricate organic/inorganic PVA nanocomposite films with homogeneous dispersion of GO. The nanomechanical and nanotribological performances of pure PVA and PVA/GO films were investigated by using nanoindentation technique. The results demonstrated that the incorporation of 0.5 wt% GO in PVA gives the highest improvement in nanomechanical and nanotribological properties. Compared to pure PVA, the elastic modulus and hardness of 0.5 wt% GO/PVA were notably increased by 122.8% and 64.5%, respectively. Furthermore, the film showed self-lubrication effect and enhanced anti-scratch performance. TGA study reveals an enhanced maximum decomposing temperature of 68.4 °C with the addition of 0.5 wt% GO in PVA. The stability of hydrogen bonding between PVA and GO accompanied with the formed organic/inorganic assembled lamellar micro-structure of PVA/GO films is the main reason for the distinct improvements in nanomechanical, nanotribological and thermal properties of PVA/GO nanocomposite films.

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1. Introduction

Polyvinylalcohol (PVA) is an important commercial water-soluble polymer with excellent film forming, emulsifying and adhesive properties. Due to its favorable mechanical, thermal resistance (melting point > 200 °C), biodegradable properties and flexibility, PVA has been widely applied in manufacturing fibers, coatings, food packages and medical devices [1–3]. Besides, its nontoxic, odorless and biocompatible features make PVA films promising candidates in biotechnological applications, such as tissue engineering, drug delivery, articular cartilage, and biosensors [2,4]. Therefore, it has attracted tremendous interests from the research community in recent years. Studies indicated that PVA hydrogel is an excellent articular cartilage repair biomaterial due to its desirable biocompatibility and bio-tribological properties [5–7].

However, pure PVA shows some drawbacks, such as low elastic modulus, poor abrasion resistance and severe creep problem, which lead to early failure of pure PVA and restrict its use in industry [2,8–10]. In order to overcome the aforementioned drawbacks,

researchers have developed PVA nanocomposites with improved properties by using various inorganic nanofillers, such as clay [11], carbon nanotube [12], graphene [13,14], nano-TiO₂ [15]. Some of them showed obvious improvement on the mechanical and electric properties of PVA [11–16].

Up to now, however, studies have shown that the reinforcing effect of most nano-fillers is still far from the expected level [8,11,14,17]. The unsatisfactory performance was usually attributed to the poor dispersion and severe agglomeration of the nano-fillers in polymer matrix. Hence, it is desirable to develop PVA composites reinforced with fillers at nanoscale dispersion, and therefore the physiochemical properties of the nanocomposites can be more efficiently improved.

Graphene and graphene oxide (GO) are two-dimensional nanomaterials prepared from natural graphite. Although graphene is envisioned as promising nanofiller for the next generation of nanomaterials, a number of practical challenges must be addressed to achieve truly homogeneous dispersion of graphene in their host polymer matrix [13,14]. As for GO, it is hydrophilic and can be easily exfoliated into monolayer nanosheets stably dispersed in water, primarily due to the plenty of hydrophilic oxygenated groups [13,18–20]. These functional groups play a significant role to improve the dispersion of GO in solvents or polymer matrix through covalent or non-covalent bonding [17,18].

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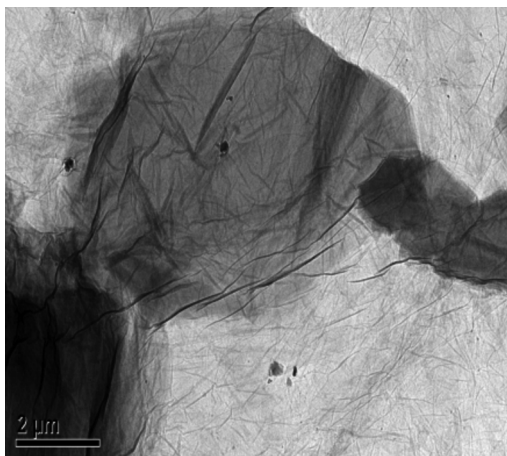


Fig. 1. TEM image of GO.

It is well known that the formation and development of stronger interfacial interaction is critical for the strength and rigidity of nanocomposites. GO has abundant functional groups which are desirable for the fabrication of high strength nanocomposites. PVA molecules can be easily coated onto exfoliated GO sheets to yield building bricks for synthesizing various functional materials through hydrogen bonding [17]. Therefore, GO has been found to be a promising filler for the improvement of mechanical properties of PVA due to its unique properties. For instance, Chen [14] reported a PVA nanocomposites with 76% increase in tensile strength and 62% improvement of modulus after dispersing 0.7 wt % GO using water solution processing method.

In our daily lives, polymer surface damage occurs frequently because of scratches, which leads to irreparable cracks [16]. Sliding friction and plowing have important roles during scratching a surface. However, to the best of our knowledge, there is rare report on the mechanical and tribological performances of PVA/GO nanocomposites investigated at nano- and micro-scales. In the present work, we utilized hydrogen-bonding interactions between PVA and GO as the driving force to fabricate assembled organic/inorganic nanocomposite thick films with uniform dispersion of GO. This simple solution-mixing method is environmentally friendly as it only uses water as the solvent. The nanomechanical and nanotribological performances of GO reinforced PVA nanocomposite films were systematically investigated by employing the nanoindentation technique. Both nanoindentation and nanoscratch tests were performed. It is expected that this study can provide insights on nano- and micro-scale reinforcement of GO in PVA, and promote the application of nanoindentation to evaluate mechanical and tribological performances of polymer films at localized region.

2. Experiment

2.1. Materials

Natural graphite powder (SP-1) was purchased from Bay Carbon Inc., USA. PVA (99% hydrolyzed, Mw ~ 89,000–98,000) were purchased from Sigma–Aldrich. DI water and other reagents were of analytical grade and used without further purification.

2.2. Synthesis of GO and PVA/GO nanocomposite films

GO was synthesized from graphite powder by the modified Hummers method [21]. TEM morphology and Raman spectroscopy of the synthesized GO are shown in Figs. 1 and 2. The procedures for preparing PVA/GO nanocomposite films are described as follows.

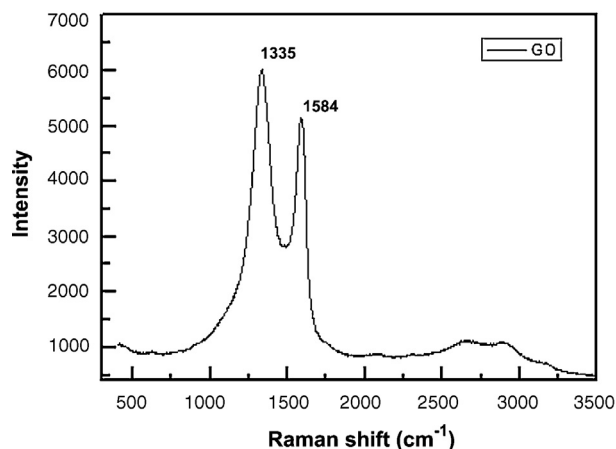


Fig. 2. Raman spectrum of GO.

The synthesized GO was diluted in 50 mL DI water and dispersed with ultrasound (300 W) for 45 min to make a homogeneously dispersed GO/H₂O (1 mg/mL) solution. PVA powders were dissolved in DI water at 90 °C and the solution was subsequently cooled to room temperature. The GO aqueous suspension was gradually added to the PVA solution and ultrasonicated at room temperature for 30 min. Then, the solution was accomplished by keeping it under magnetic stirring at 90 °C for 24 h to obtain homogeneous PVA/GO solution. Finally, the above solution was cast onto glass plate and kept at 30 °C for film formation until its weight equilibrated. The films (thickness: 0.3–0.35 mm) were cut into certain species for measurements. The weight fraction of GO in the nanocomposite films described above were controlled to be 0.25, 0.5, 0.75, 1.0 and 1.5 wt%, and denoted the sample name from PVA1 to PVA5, respectively.

2.3. Nanoindentation and nanoscratch tests

Nanoindentation was used to investigate the nanomechanical properties, including hardness and elastic modulus, of the PVA/GO nanocomposite films with different contents of GO. The nanoindentation and scratch tests were conducted at room temperature (25 °C, humidity 50–55%) on a nanotriboindenter (Hysitron Inc., USA) using a conical diamond indenter. For all the nanoindentation tests, the holding time at the F_{max} was 10 s to minimize the time-dependent plastic effect. For each indentation testing condition, at least 12 tests were repeated. In the nanoscratch tests, the conical shape diamond indenter pressed vertically into the films and moved horizontally on the film surface under designed load with 10 μm scratch length.

2.4. Characterization

Transmission electron microscope (TEM) was performed using a Philips CM-200 TEM with a LaB6 filament at an accelerating voltage of 120 kV. The GO/H₂O solution was dropped onto carbon-coated copper grids (mesh size 300) and allowed to dry under ambient conditions. Raman measurements were performed with a Horiba Jobin Yvon LabRAM HR high resolution Raman microscope, in a backscattering configuration. The microscope was focused onto the desired area of the sample and then the laser was turned on and focused for the measurement. The laser spot was about 1 mm in diameter. Scanning electron microscopy (SEM) was performed on a high magnification SEM (Ultra ZEISS) with low acceleration voltage 1–2 kV and samples were scanned without gold coating.

Atomic force microscope (AFM, Digital Instruments) images of scratches and scratch profiles of PVA samples were performed

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