



# Intrinsic mechanical properties of graphene oxide films: Strain characterization and the gripping effects



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## ABSTRACT

Graphene oxide films are excellent structural and functional materials for their outstanding performance. Recent measurements reported widely scattered mechanical properties, which have been attributed to the difference in the chemistry and complex microstructures that rely on the fabrication process. In this work, we present an experimental study showing that the procedure of mechanical characterization is also critical for measuring intrinsic mechanical properties of graphene oxide films. We find that the specimen geometry and loading conditions could lead to significant variation in the measured stress, strain and toughness. To quantify these effects, we propose a rigid-plasticity shear-lag model to capture the effect of interfacial slippage at gripping ends, which can not only eliminate artifacts in measurements, but also be used to determine interfacial mechanical properties of gripping. Effects of grip pressure, length and loading rate are also discussed, following which suggestions for the experimental setup are provided. These understandings lay the ground for probing intrinsic mechanical properties of graphene oxide films in a reliably way.

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## 1. Introduction

With emerging low-dimensional materials and technological development of synthesis and processing techniques, nano-structured assemblies in forms of films and fibers have been found wide applications as structural and functional components in relevant applications [1–6]. Building blocks and microstructures are the key information encoded into the macroscopic assemblies, which could be tuned to control the material performance [7–12]. Considering the outstanding mechanical properties of graphene, such as its tensile stiffness (1 TPa), strength (120 GPa) and resilience (20% strain to failure) [13–17], one would expect the films and fibers assembled in the layer-by-layer fashion to exhibit superior mechanical performance. Graphene oxide (GO), a hydrophilic derivative of pristine graphene, which assembles in the solvated phase, holds great promise in this scenario. GO films and fibers fabricated from their dispersions via vacuum-assisted filtration [1],

evaporation induced self-assembly [18], electrospray coating [19], or wet spinning techniques [20] demonstrate impressive mechanical performance, including tensile stiffness of 6.8 GPa and 5.4 GPa, strength of 453 and 102 MPa and strain to failure up to 5.6% and 10.1%, respectively [21,22]. However, it should be remarked here that the performance reported for these materials in the literature is highly scattered, which has been attributed to the variation in the chemistry of GO sheets [23,24], and the microstructures that depend on the film or fiber forming process [20]. Moreover, one should be noted that due to the limitation in fabrication processes, the geometry of GO films is quite special, and may have significant impacts on the measurement for their high mechanical resistance.

The American Standard Test Method (ASTM) D882 is issued for mechanical tests of thin sheets under tension, which report the elongation, Young's modulus, tensile strength and strain to failure [25]. The ASTM D882 is designed for thin sheets with a length of ~100 mm and a thickness below ~1 mm to minimize the effects of interfacial sliding between grips. Accordingly, this can be done for GO films in practice by specifying the specimen geometry and validated further by quantifying the slippage at gripping ends and stress distribution in stretched sample. However, GO films prepared using current technology can hardly conform to the requirements declared in ASTM D882. Specifically, GO films fabricated via

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vacuum filtration are limited in both the lateral size and thickness. In the typical laboratory setup, the lateral size is defined by the container that is usually on the order of 10 mm, while the thickness  $h$  is about 10  $\mu\text{m}$  as defined by the filtration or evaporation process. Consequently, slippage or shear in grips may lead to significant interfacial displacement that is incorrectly counted in the tensile strain of sample, which is measured based on the grip-to-grip distance  $L$ , as called grip separation. This issue, however, has been overlooked in previous studies. Our literature survey shows that the geometry of GO specimen is widely dispersed (Fig. 1a). For example, the grip separation  $L$  and width  $W$  ranges mostly from 4 to 20 mm and 1–6 mm, with the thickness  $h$  from 1 to 40  $\mu\text{m}$  [1,18,21,26–49]. The effects of the specimen geometry and loading conditions then have to be well elucidated for a reliable design of measurements and industrial applications of GO films, as the specification in current test standards needs to be adjusted according to the material properties. In this work, we carried out a set of uniaxial tensile tests to address these issues, following by model analysis that quantitatively clarifies the underlying mechanisms. Practical rules are then suggested for measurements of intrinsic mechanical properties for GO films. Interfacial mechanical properties of gripping interface are also discussed based on the model analysis and results from mechanical tests.

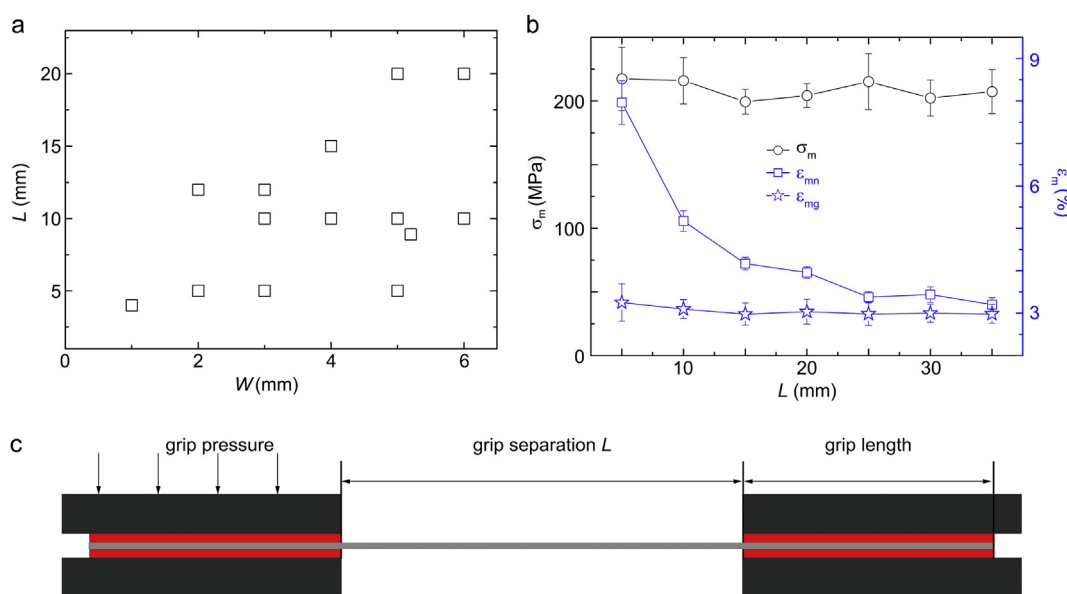
## 2. Materials and methods

### 2.1. GO film fabrication

GO sheets with an average lateral dimension of 23  $\mu\text{m}$  were synthesized by a modified Hummers method from natural graphite at a relatively low oxidation temperature of 0  $^{\circ}\text{C}$  [50]. Natural graphite powder (325 mesh, 3.0 g) was dispersed in 70 mL concentrated sulfuric acid under constant stirring with a speed of 350 rpm at 0  $^{\circ}\text{C}$ . After stirring for 2 h, 9 g potassium permanganate was added slowly over a period of 2 h. The reaction mixture was kept stirring for addition 8 h under the temperature at 0  $^{\circ}\text{C}$ . Then, 150 mL water was slowly added to the reaction mixture over 6 h by a peristaltic pump. All the reaction process was strictly controlled at

0  $^{\circ}\text{C}$ . The final reaction mixture was poured into 100 mL ice-water mixture to terminate the reaction, followed by the addition of hydrogen peroxide (30% aqueous solution) under stirring until no gas escape from the dispersion. The mixture was then filtered and washed with diluted (3.7%) hydrochloric acid aqueous solution to remove the metal ions, followed by washing using ultrapure water. The resultant slurry was then dispersed in distilled water under mild magnetic stirring to form a GO suspension. The GO suspension was purified by dialysis for two weeks. After that, it was centrifuged repeatedly at 3000 rpm to remove non-exfoliated aggregations. Finally, the GO suspension was concentrated by centrifugation at 10,000 rpm for 1 h. The concentration is confirmed to be 7.40  $\text{mg mL}^{-1}$  by weighting the drying solid from 1 mL concentrated GO dispersion.

We follow the facile dispersion-evaporation method by cast drying 7.4  $\text{mg mL}^{-1}$  GO dispersions on a polystyrene petri dish under ambient condition, which could be easily peeled for further characterization. The evaporation process typically takes 3 days at 25  $^{\circ}\text{C}$ . Fig. S1a in Supplementary Information (SI) shows the GO film fabricated from the dispersion-evaporation process. Fig. S1b in SI shows the scanning electron microscope (SEM) images taken by Sirion 200 field emission SEM that operates at a voltage of 10 kV, where the layered microstructure is clearly visualized. The size distribution of GO nanosheet histogram is measured from the SEM images (Fig. S2a, SI). It implies that the GO nanosheets have an average lateral size of  $\sim 23$   $\mu\text{m}$ . AFM image indicates that the average thickness of GO nanosheets is  $\sim 1$  nm, which is comparable to that of a GO monolayer (Fig. S2b, SI). The X-ray photoelectron spectroscopy (XPS) spectra of GO shows the presence of four type carbon atoms, C–C/C=C (284.8 eV), C–O (286.8 eV), C=O (287.8 eV) and O–C=O (289.0 eV), implying that several types of oxygenated groups are attached on to the GO sheet (Fig. S2c, SI). The Raman spectrum of GO flakes on the Si/SiO<sub>2</sub> substrate displays a D-band at 1348  $\text{cm}^{-1}$  and a G-band at 1590  $\text{cm}^{-1}$  (Fig. S2d, SI). The intensity ratio between the D- and G-band ( $I_D/I_G$ ) is 1.97, indicating the structural integrity of GO nanosheets from the modified Hummers procedure carried out at a relatively low oxidation temperature of 0  $^{\circ}\text{C}$ .



**Fig. 1.** (a) A literature survey on the geometry (length  $L$ , width  $W$ ) of GO film specimen in previous studies [1,18,21,26–49]. (b) Strain to failure measured by the gauge and gripping methods, and the tensile strength determined for test specimens with different grip separation  $L$ . (c) The experimental setup of uniaxial tensile tests. (A colour version of this figure can be viewed online.)

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