Composites Science and Technology 153 (2017) 95–102

Contents lists available at ScienceDirect

Composites Science and Technology

journal homepage: http://www.elsevier.com/locate/compscitech

Assessing local yield stress and fracture toughness of carbon nanotube poly(methyl methacrylate) composite by nanosectioning

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ARTICLE INFO

Article history:
Received 28 July 2017
Received in revised form
26 September 2017
Accepted 30 September 2017
Available online 4 October 2017

Keywords:
Fracture toughness
Shear yield stress
Nanosectioning
Carbon nanotubes
Polymer composites

ABSTRACT

A nanosectioning (cutting) method was used to test the local shear yield stress and fracture toughness (specific work of surface formation) of multiwall carbon nanotube (MWCNT) poly(methyl methacrylate) (PMMA) composites, and the effects of MWCNT content on the yield stress and toughness were investigated. The composites were prepared by a solution casting method, with MWCNT content varying from 0.05 to 1.0 wt%. Above 0.1 wt% MWCNT content, the yield stress reduced by the addition of MWCNTs. The fracture toughness of the composite was effectively enhanced by the addition of MWCNTs, ranging from 17 J/m^2 for the neat PMMA to 25 J/m^2 for the 1.0 wt% composite. The shear yield stresses obtained by nanosectioning were correlated to nanoindentation measurement, and possible contributions from the MWCNTs to the fracture toughness of the composite were analysed.

1. Introduction

Carbon nanotubes (CNTs), including singlewall CNTs and multiwall CNTs (MWCNTs), possess superior mechanical properties that can be potentially utilized to enhance the properties of polymers, metals, ceramics and many other materials [1,2]. Since the discovery of CNTs, considerable efforts have been made to incorporate CNTs into polymers homogeneously, which will significantly affect the deformation mechanisms and energy dissipation in polymers, for load carrying applications [3–5]. Studies have shown that the elastic modulus of CNT/polymer composite can often be increased compared to that of the neat polymer, while the tensile strength and fracture toughness may slightly increase and then decrease when the CNT content exceeds a critical value, especially for randomly oriented composites [6–8]. This non-monotonic effect is mostly due to CNT agglomeration [1].

It would be useful to microscopically investigate the underlying mechanisms that affect the basic mechanical properties of CNT/polymer composites as a function of CNT content. Most of the studies use linear elastic fracture mechanics (LEFM) tests to evaluate the strengths and fracture toughness of composites at the macroscopic level [6,9]. However, conventional LEFM tests are usually accompanied with the problem of crack blunting that would lead to an overestimation of fracture toughness [10,11], especially for toughened polymer materials. Another method, the pull-out test [12], besides being technologically challenging, is limited to quantify the interfacial properties of CNT based composites and only gives the local properties during nanotube pull-out. To further investigate the fracture processes in CNT/polymer composites, especially at micro- or sub-microscale, complementary experimental approaches need to be applied. More recently, material sectioning (cutting) has been used as an effective and robust approach in determining strengths and toughness of polymers without excessive crack-blunting [13]. Atkins [14], Williams et al. [11,15] have demonstrated that the shear yield stress (τ_y) and fracture toughness (G_c) of material can be determined by analysing the sectioning forces. Wang et al. [16,17] used the sectioning method to test the fracture toughness of nanoparticle/epoxy composites. To the authors' knowledge, small-scale sectioning which can characterise the local mechanical properties of materials has not yet been used to investigate the yielding and fracture properties of CNT nanocomposites and the dependence on CNT content.

In this work, MWCNT/PMMA composites with varying MWCNT

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https://doi.org/10.1016/j.compscitech.2017.09.034
0266-3538/

contents were prepared. The local shear yield stresses and fracture toughness (specific work of surface formation) of the composites were tested by nanosectioning in an ultramicrotome instrumented with force sensors, and the effects of MWCNT content on the mechanical properties of the composites were explored. Additionally, nanoindentation tests were performed to the composites to compare the yield stresses estimated by nanosectioning. Possible contributions from the MWCNTs to the fracture toughness of the composites are discussed.

2. Modelling

To determine the shear yield stresses and fracture toughness of the nanocomposites by sectioning, the following assumptions are made: (1) the low content of nanofillers (maximum is 1.0 wt% in this study) is assumed not to lead to any violation of the classical cutting principles developed for isotropic metal and polymer materials [18,19]; (2) the neat matrix and composites are assumed to be rigid-plastic; (3) Coulomb friction holds during the sectioning process. According to the force circle in Fig. 1, the friction force, f, and the normal force, N, on the chip-knife interface are expressed as,

$$f = F_c \sin\alpha + F_t \cos\alpha \tag{1}$$

$$N = F_c \cos\alpha - F_t \sin\alpha \tag{2}$$

where $\alpha = 40°$ is the rake angle of the knife, F_c is the force component along the sectioning direction and F_t is the force component normal to the sectioning direction, as shown in Fig. 1. During sectioning, the external work is provided only by F_c and is mainly dissipated by the plastic deformation on the shear plane (inclined at φ), friction on the chip-knife interface and crack propagation ahead of the knife. Based on these assumptions, Atkins [14] reformulated the energy relationship as,

$$F_c v = (\tau_y \gamma)(t_u w_u v) + [F_c \sec(\beta-\alpha)\sin\beta]\frac{\sin\varphi}{\cos(\varphi-\alpha)} v + R w_u v \tag{3}$$

where τ_y is the shear yield stress, γ is the plastic strain, R is the fracture energy (specific work of surface formation), β is the Coulomb friction angle, α is the knife rake angle, φ is the shear plane angle, t_u is the depth of cut, w_u is the width of cut and v is the sectioning speed.

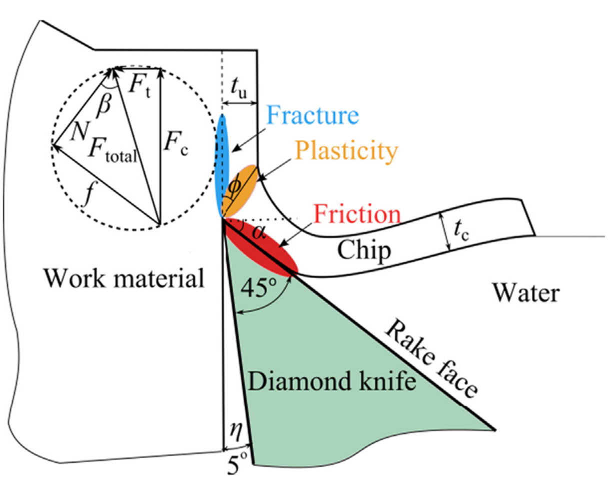

Fig. 1. Schematic of the material nanosectioning using an ultramicrotome.

The plastic strain γ and friction angle β are given as,

$$\gamma = \cos\alpha / \cos(\varphi-\alpha)\sin\varphi \tag{4}$$

$$\beta = \tan^{-1}(\mu) = \tan^{-1}(f/N) \tag{5}$$

where μ is the friction coefficient. Thus, Eq. (3) can be rearranged as,

$$\frac{F_c}{w_u} = \left(\frac{\tau_y \gamma}{Q}\right) t_u + \frac{R}{Q} \tag{6}$$

where $Q = [1 - \sin\beta\sin\varphi/\cos(\beta-\alpha)\cos(\varphi-\alpha)]$ is a friction parameter. From Eqs. (1), (2) and (5), an expression for F_t yields,

$$F_t = \left(\frac{\mu - \tan\alpha}{1 + \mu \tan\alpha}\right) F_c \tag{7}$$

Therefore, from Eq. (6) if F_c and t_u can be fitted linearly, the slope, S, and the intercept, I, of the linear plot are related to the shear yield stress and fracture toughness, respectively. During sectioning, the shear plane would adjust to a preferred orientation, seeking for the minimum consumption of external work. Atkins gave the implicit expression of φ in Ref. [14], and Williams et al. [15] later derived the closed-form solution as,

$$\cot\varphi = \tan(\beta-\alpha) + \sqrt{1 + \tan^2(\beta-\alpha) + Z[\tan(\beta-\alpha) + \tan\alpha]} \tag{8}$$

where $Z = R/\tau_y t_u$ is a dimensionless parameter. The values of τ_y and R can be obtained when φ is determined. The calculation procedure is described in Fig. 2.

The shear plane angle can be determined by experiment as well. Assuming that the material volume remains constant before and after sectioning, the thickness of the chip can be estimated by,

$$t_c = w_u l_u t_u / w_c l_c \tag{9}$$

where the subscripts 'u' and 'c' represent the uncut chip values and chip values, respectively. The validation of the estimation of chip

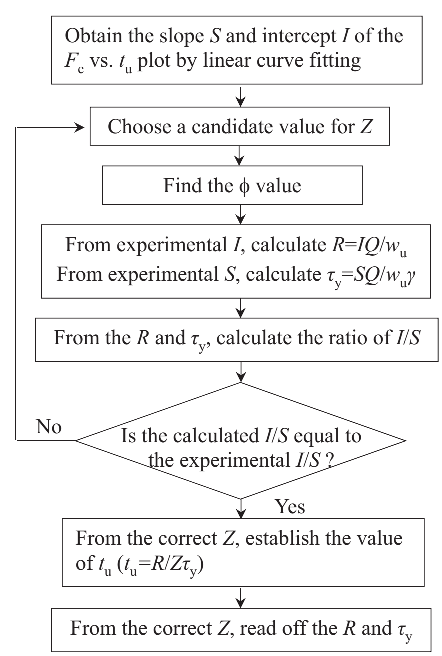

Fig. 2. The calculation procedure to determine the shear yield stress and fracture energy from sectioning tests.