



The reinforcement effect of exfoliated graphene oxide nanoplatelets on the mechanical and viscoelastic properties of natural rubber



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ABSTRACT

The mechanical properties of natural rubber reinforced with exfoliated graphene oxide (GO) nanoplatelets have been studied to evaluate the reinforcement efficiency of the filler. Tensile tests on rectangular specimens have been carried out at different strain rates for deformations up to 600%. Tensile tests showed a significant increase in the Young's modulus of the composites even at low filler loading (+50% @ 1.00 wt%) that corroborates the strong interfacial interaction between GO and natural rubber. The viscoelastic properties are investigated through relaxation tests and loading–unloading cycles at different strain rates. It is shown that the Young's modulus increases with the strain rate and the dissipation increases (+250% @ 0.75 wt%) for increased GO content because of the increased friction caused by the presence of high aspect-ratio GO platelets. These findings indicate significant improvements in the mechanical properties at much lower filler concentration than those usually achieved with carbon black or carbon nanotubes.

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1. Introduction

In industrial applications rubber is used extensively; and reinforcement with fillers, such as carbon black, further improves the mechanical, viscoelastic and thermal properties [1,2]. This improvement in the properties of the material is dependent on a number of factors that include particle size, shape, dispersion, aspect ratio and surface functionalization. It is for this reason that significant interest is currently engaged in the exploitation of nano-dimensional fillers (such as nanoclays [3], carbon nanotubes [4,5] and graphene [6–8]) which have large specific surface areas and excellent physical properties that give them the potential to provide significant improvements compared to existing solutions.

Since the discovery of single layer graphene [9], nanocomposites based on a rubber–graphene matrix have been increasingly developed. Graphene is nano-dimensional with a large aspect ratio that gives it an exceptionally large surface area. It is also highly conductive, thermally stable, has very high in-plane stiffness and can be customised by functionalization of the surface. All of this makes it an excellent nanofiller material in composites and has often been used in polymer systems in the glassy state [10,11]; pro-

viding significant improvements. The improvements that are seen in the properties are often dependent on the physiochemical properties of the graphene and so functionalization of the graphene sheets is very important [12]. Graphene oxide (GO) is often a precursor material to graphene, and is functionalised with oxygenated groups that can provide reaction sites for further functionalization and even potentially enhance the interactions between the filler and bulk matrix, therefore enhancing the properties [6,13]. This has in some part been transferred to elastomers but the mechanisms are different and require a slightly different understanding [14].

Natural rubber (NR) is extracted in the form of a latex that consists of a colloidal suspension of rubber latex particles, non-rubber components and water [15]. Non-vulcanized NR displays characteristics analogous to the cross-linking of vulcanized NR in that the complexity of the long polymer chains forms entanglements and enhancements in residual (van der Waals) forces [14]. This is in addition to functional groups at the ends of chains that are thought to interact with non-rubber components and form a 'natural' pseudo network [16,17]. Additional cross-linking of the NR is typically performed by a vulcanization procedure; giving enhanced mechanical properties and increased durability, however the vulcanization process also adds additional complexity. It is thought that, to get a better understanding of the filler–matrix interactions, it is worthwhile to look first at a non-vulcanised nanocomposite.

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Non-vulcanized NR has demonstrated an upturn in stress at high strain (>200%) that has not been observed in non-vulcanized synthetic rubber and it has been shown that this is due to Strain-Induced Crystallization (SIC) [17]. Unlike the glass transition, crystallization includes structural change, an increase in density and the release of latent heat [14].

The addition of platelet-like fillers to NR has provided significant mechanical advantages (see for instance [18] for nanoclay fillers or [6] for functionalised graphene sheets). Their lamellar structure is thought to accelerate the stiffening of the nanocomposite through the formation of a filler network larger than the one seen with spherical particles [18]. This effect could be further increased by the presence of immobilised rubber trapped within the filler network [3].

In the present work, the effects of GO nanoplatelets on NR are quantified in terms of Young's modulus, fracture toughness and dissipation energy. Both elastic and viscoelastic properties of the nanocomposite are affected by the presence of GO which provides excellent stress transfer within the matrix and cause the hysteresis losses during cyclic tests to be increased. The response of the NR/GO compound at large strain is also discussed and new insights are provided which show a possible reorientation of the nanoplatelets in the direction of the applied force.

2. Description of materials and experimental techniques

2.1. Materials

Natural rubber (NR, 60% HA, Chana Latex Co., Ltd., Thailand) Latex is obtained from a private source and used as received, without further modification. Graphene oxide (GO) was prepared from

graphite according to the Hummers–Offeman method described in our previous report [19].

NR–GO Nanocomposites were prepared by “latex mixing” of NR latex and aqueous dispersions of GO platelets. A Petri dish has 3 g of NR latex added, followed by 3 ml of aqueous GO platelets (whereby the concentration of the GO dispersion is controlled to give the required wt% of GO). Mixing is achieved with a vibrating plate. The solution is then left to dry in the Petri dish for 48 hours. Nanocomposites of GO concentrations $\chi = 0, 0.25, 0.5, 0.75$ and 1.0 wt% have been produced and tested.

2.2. Structural characterisation

TEM and AFM analysis of GO precursor was performed by air drying freshly prepared aqueous dispersions of GO on to carbon coated copper grids and freshly cleaved mica respectively. As shown in Fig. 1d, TEM images revealed the presence of ultra-thin flat sheets ranging from 500 nm to 1 μm in size. Tapping mode AFM studies showed the presence of delaminated sheets that were 1–1.4 nm in thickness (Fig. 1a), which was consistent with the formation of completely exfoliated atomically thick GO.

Fig. 1c shows Fourier-transform spectra of graphite, graphene oxide, natural rubber and natural rubber–graphene oxide nanocomposites. The FTIR spectrum of graphite did not exhibit many prominent features apart from features at 3446 cm^{-1} , $2925/2852\text{ cm}^{-1}$ and 1627 cm^{-1} corresponding to O–H, C–H and C=C stretching vibrations from adsorbed water, alkyl groups at the edges and graphitic sp^2 hybridized network. On the other hand, GO samples showed additional peaks corresponding to hydroxyl, carbonyl/carboxylic acid and epoxide groups at 3378 cm^{-1} , 1718 cm^{-1} and 1225 cm^{-1} respectively [20]. The vibration spec-

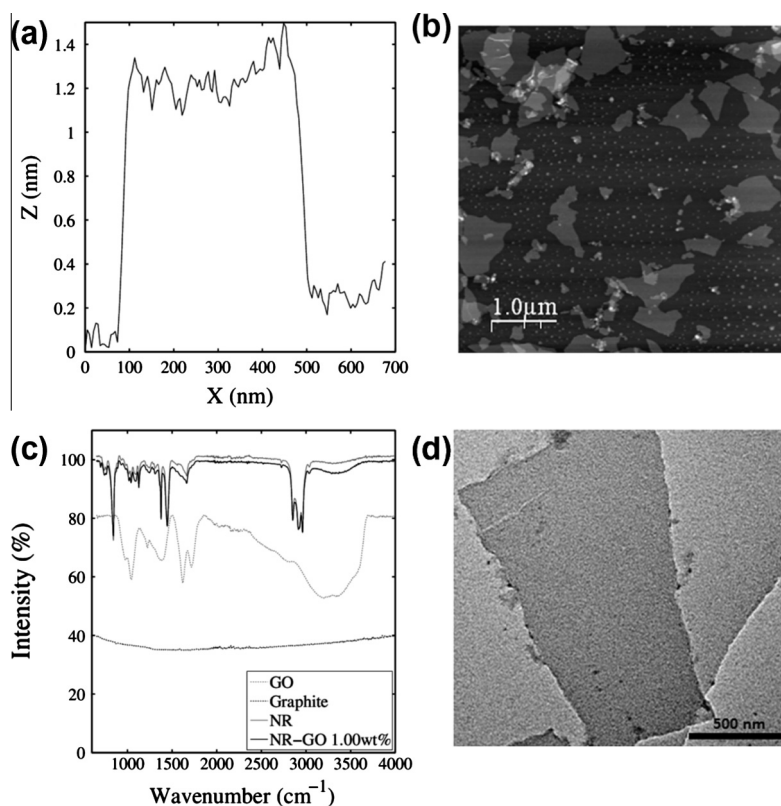


Fig. 1. (a) AFM images of GO platelets reveal the delaminated sheets to be 1–1.4 nm in thickness, consistent with completely exfoliated single layer GO; (b) AFM image showing the typical dispersion of GO platelet size; (c) Fourier-transform spectra of GO, graphite, natural rubber and NR–GO nanocomposites containing 1.00 wt% GO; and (d) TEM image of GO platelet reveals ultra-thin flat sheet between 500 nm and 1 μm in size.

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