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# Surface modification of aramid fibres by graphene oxide nano-sheets for multiscale polymer composites



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#### ABSTRACT

Aramid fibre–graphene oxide (GO) multiscale reinforcement fibres were prepared by a novel technique where a plasma co-polymerized functional primer coating was initially applied on fibres followed by chemically attaching GO unilamellar sheets to the functionalized surface of the fibres. The functionalized fibres were immersed in purified GO aqueous suspensions at 60 °C under two pH conditions (slightly acidic GO suspension at pH 6 and alkaline ammonia water GO suspension at pH 9). The multiscale hybrid fibres were characterized by XPS, FTIR, Raman spectroscopy, SEM, AFM and tensile measurements. The fibres treated in alkaline ammonia aqueous suspension showed better strength and adhesion characteristics. The mean tensile strength value for the modified fibres showed an improvement over 7.9% whereas the interfacial shear strength in modified fibre–epoxy model composites, as determined by micro-bond technique, improved by 44.68%.

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#### 1. Introduction

Aramid fibre is a well-known class of reinforcement fibres for polymer composites. A combination of low density, high specific strength and high toughness makes these fibres extremely suitable for applications which demand light weight but superior mechanical properties of composite. However, high crystallinity of fibres makes their surface chemically inert and microscopically smooth. As a result their adhesive bonding with most of the matrices is poor and some sort of surface treatment is usually conducted to enhance the reinforcing effect. The common approaches for surface modification of reinforcement fibres include: (a) plasma surface treatment of fibres [1,2], (b) chemical activation of the fibre surface [3,4] and (c) coating with coupling agents or finish formulations to form chemical bonding with resins on the surface of the fibre [5]. A recent approach is the grafting of carbon nanotubes (CNTs) on fibre surface to produce multiscale reinforcement, which increases the strength of Kevlar fibre by 12% [6]. This approach has the advantage of simultaneously improving mechanical performance and surface activity. The nanostructured interface created by the grafted carbon nanotubes enhanced the interlaminar strength in modified fibre– bismaleimide composite by 30%. However, cylindrical geometry and entanglement of tubes forming the bundles make it difficult to align nanotubes along fibre surface. The higher cost of CNTs and the use of highly toxic chemicals are another point of concern.

Graphene, an allotrope of carbon, is a two dimensional monolayer of  $sp^2$  bonded carbon atoms in a honeycomb lattice. It has unique physical. optical and mechanical properties, and its potential has been widely explored in recent years in various disciplines and as nano-reinforcement in polymer composites [7–10]. It has been shown that at a low nanofiller content, graphene platelets performed significantly better than carbon nanotubes in terms of enhancing a variety of mechanical properties in nano-composites (tensile strength, Young's modulus, fracture toughness and fracture energy) [11]. This better efficiency has been attributed to the enhanced specific surface area of graphene platelets, improved mechanical interlocking at the nano-filler-matrix interface, and the two dimensional geometry of graphene platelets. Graphite oxide (GO) is an oxygen-rich carbonaceous layered material which is produced by the controlled oxidation of graphite. Each layer of GO is essentially an oxidized graphene sheet commonly referred to as graphene oxide. A number of studies [12–14] suggest that GO consists of intact graphitic regions interspersed with sp<sup>3</sup>-hybridized carbons containing hydroxyl and epoxide functional groups on the top and

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bottom surfaces of each sheet and sp<sup>2</sup>-hybridized carbons containing carboxyl and carbonyl groups mostly at the sheet edges. Hence, GO is hydrophilic and readily disperses in water to form stable colloidal suspensions. Graphene oxide is nonconductive and mostly insoluble in organic solvents, but it can be reduced to conductive graphene and functionalized to increase it's solubility in organic solvents.

Martirosyan and Zyskin<sup>[15]</sup> simulated the elastic properties of reinforced Kevlar-graphite unidirectional fibre composites in the form of Kevlar yarn with a thin outer layer of graphene. The study suggests that introducing outer enveloping layer of graphene sheet linked to polymer chains by strong chemical bonds may significantly strengthen Kevlar fibre with respect to transversal deformation. Inspired by the simulation of Martirosyan and Zyskin and the approach of Chen et al. [6] for multiscale reinforcement, the potential of graphene oxide as nano-scale phase is explored in present research. Graphene oxide nano-sheets have been chemically attached to the functionalized surface of aramid fibres to produce multiscale reinforcement and create nanostructured interface in model composites. It is expected that this treatment may enhance the strength of the fibre from the strength contribution of graphene oxide nano-sheets and functionalities on GO surface coupled with the nano-architecture interface may result in enhanced adhesion. Plasma co-polymerization of Allylamine-Octadiene (PCP AA-OCD) was used to create a primer layer on fibre surface containing surface exposed amine functionalities. It has been previously reported that plasma co-polymerization of Allylamine-Octadiene produced nanometre thick, conformal and highly adherent functional coatings on the fibre surface that mostly retained amine functionalities and improved adhesion of carbon fibres to the epoxy matrix [16]. The functionalities created on the fibre surface can be utilized for further reactions at the surface and in present research these have been used

to attach chemically the graphene oxide sheets through reduction reactions. Graphene oxide produced by controlled oxidation of graphite contains epoxide and other oxygen containing functional groups on its surface are reactive towards these functionalities on the fibre surface [17]. The schematic diagram of the reaction mechanism is illustrated in Fig. 1. Plasma deposits are not characterized by repeating monomer units as conventional polymers. The 'R' in Fig. 1 represents highly cross-linked structure of monomers and their dissociation products (resulting from the large variety of precursor chemical bond dissociation processes).

#### 2. Experimental

#### 2.1. Materials

The aramid fibres used in this work were finish-free Kevlar® 49 yarn, type 968, by E. I. DuPont de Nemours (dtex 4800, den 4320). The fibres were cleaned with 1,2 dichloromethane in a Soxhlet apparatus for 24 h, washed with distilled water and dried overnight in a vacuum oven at 80 °C. Fibres were randomly extracted from the tow for further processing and characterization. Allylamine and Octadiene monomers were of reagent grades, purchased from Sigma and degassed in three *freeze-pump-thaw* cycles before plasma co-polymerization step. Graphite powder (<20 µm), sodium nitrate (purity  $\geq$  99.0%), sulfuric acid (purity of 95–98%), potassium permanganate (purity  $\geq$  99.0%), hydrogen peroxide solution (30% w/w in H<sub>2</sub>O) and hydrochloric acid (37%) were also purchased from Sigma and used without any further purification. Huntsman cold curing epoxy system based on Araldite® LY 5052 and hardener Aradur® 5052 was used for making model composites for microbond test.







Fig. 1. Scheme adopted for chemical attachment of graphene oxide nano-sheets to aramid fibre surface (for description of 'R', reader is referred to Section 1).

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