Composites Science and Technology 71 (2011) 1692–1698

Contents lists available at SciVerse ScienceDirect

Composites Science and Technology

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

A surface-property relationship of atmospheric plasma treated jute composites

Abdullah A. Kafi *, Kevin Magniez, Bronwyn L. Fox

Centre for Material and Fibre Innovation, Institute for Technology and Research Innovation, Deakin University, Geelong, VIC 3217, Australia

ARTICLE INFO

Article history:
Received 27 November 2010
Received in revised form 7 June 2011
Accepted 17 July 2011
Available online 31 July 2011

Keywords:
D. Atomic force microscopy (AFM)
A. Textile composites
B. Interfacial strength
B. Fibre/matrix bond

ABSTRACT

Jute fabric was treated for various periods of time under atmospheric plasma glow discharge (APGD) using helium (He), helium/nitrogen (He/N), and helium/acetylene (He/Ac) gases. It was found that, for all gases studied, 10 s of treatment was enough to significantly improve the wetting behaviour of the fabric. Different levels of improvement of up to 55%, 62%, and 40% in flexural strength, flexural modulus, and interlaminar shear stress respectively were observed in composites produced from plasma treated fabrics. The storage modulus and glass transition temperature were also improved by up to 200% and 16 °C, respectively. Efforts were made in order to correlate the changes in surface roughness, tip-surface adhesion, and surface chemistry of the fibres (measured by XPS and FTIR) with the performance of the composites. In light of some of the trends, it has been postulated that low-molecular weight oxidised species have formed on the fibre surface during plasma and that the chemical nature of these species must have changed considerably depending on the type of gas mixture used, inducing various synergistic or antagonistic effects.

1. Introduction

The focus on the use of natural fibres such as jute, sisal, flax, and hemp in order to produce eco-friendly composites has increased considerably over the past few years. Among these, jute (which is a lignocellulosic bast fibre) has been identified as one of the most promising candidates for the composite industry because of its high specific modulus (19 GPa) and low cost [1]. However, the poor interfacial adhesion properties displayed by jute often result in composite materials having poor mechanical properties.

Many physical and chemical treatments including photo oxidation by UV irradiation [2] ionising radiation [3], corona [4], cold plasma [5], and atmospheric pressure plasma [6] have already been employed to overcome the incompatibility of various substrates. Surface modification of natural cellulosic fibres such as jute is primarily conducted in order to remove surface components such as wax, pectin or hemicelluloses from the amorphous region which increases the concentration of oxygen related functional groups on the fibre surface. Atmospheric plasma is of interest to this study because it is cost effective, continuous in operation and provides a relatively uniform and stable treatment throughout the surface compared to low pressure plasma systems [7]. Depending on the material of interest to be modified, atmospheric plasma flow can cause ablation, cross-linking or surface activation. Ablation consists of the removal of organic residues as well as surface layers at a molecular level. Cross-linking occurs as a result of the interaction between two or more radicals leading to the formation of covalent links whilst surface activation increases the surface energy as a result of generation of polar groups on the fibre surface. Generally, inert gases such as argon or helium are used to achieve an ablation or etching effect (in order to remove possible contamination from the surface), whereas grafting gases such as nitrogen, oxygen, and hydrogen can be used to impart specific functionality. Gases including oxygen [8], helium [9], helium and nitrogen [10], air [11] plasma have been used to modify the surface of natural fibres. However, little has been studied the effect of atmospheric plasma on surface characteristics of jute fibres as well as surface property relationships in jute/polyester composites.

Scanning force microscopy is especially useful to measure the intermolecular forces between the tip and the sample surface in addition to surface imaging for the chemically inhomogeneous and rough surface of natural fibres. When a tip is brought in contact and pulled from the surface, the interaction between the two surfaces is recorded as a force curve [12]. Analysis of the force curve provides measurement options like estimation of surface energy [13] and mean adhesion [12]. AFM techniques have been previously used to characterise the chemically modified flax fibre which could be a strong tool in direct visualisation of the effect of chemical modification. For instance, Balnois et al. [14] found that the adhesion properties of flax fibre at the tip were found to increase after sodium hydroxide treatment but decreased after formic acid-treatment as a result of the changes in surface hydrophilicity. Although AFM has been used to measure the tip-surface adhesion of protein fibre [12], it is not very common for studying

* Corresponding author. Tel.: +61 3 52272038.
E-mail address: abdullah.kafi@deakin.edu.au (A.A. Kafi).

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doi:10.1016/j.compscitech.2011.07.011

the surfaces of bast fibre such as jute [15]. Furthermore, only a very few literature reports exist on the correlation between surface roughness, tip-surface adhesion and the role of adhesion in the performance of composites.

Natural fibre based thermoset composites are commonly manufactured using methods which include wet lay-up [16], resin transfer molding (RTM), compression molding [17], and vacuum assisted resin infusion [18]. In this work we looked at an alternative manufacturing technique namely the Quickstep™ process, a patented fluid filled floating mould technology [19]. The process operates by rapidly applying heat to the uncured laminated surface by close contact through a silicon bladder filled with a heat transfer fluid of high heat capacity and thermal conductivity. The heat transfer fluid acts as a thermal sink which maintains constant cure temperature and removes any excess heat generated in an exothermic reaction. Efficient temperature control is maintained by circulating the fluid through the pressure chamber. This provides rapid and precise heating/cooling of the laminate and allows excellent consolidation to be achieved at low applied pressures.

This study is designed to investigate the effect of helium (He), helium/nitrogen (He/N), and helium/acetylene (He/Ac) gases mixtures under atmospheric plasma glow discharge (APGD) on the wicking, topography, adhesion properties and surface chemistry of jute reinforcement. The changes in surface properties of the jute fibres were correlated to the mechanical and thermo-mechanical properties of the corresponding composites.

2. Materials and experimental section

2.1. Materials

Unbleached woven jute fabrics were obtained from Bangladesh Jute Mills Corporation, Dhaka. The unsaturated polyester resin used was ESCON 62-333 procured from fibreglass International in Australia was premixed manually with a cobalt napthenate promoter solution (1.5 wt%) and 36 wt% styrene crosslinking agent. The curing agent was methyl ethyl ketone peroxide (MEKP).

2.2. Experimental section

2.2.1. Plasma treatment

Throughout this study we have used a semi-industrial atmospheric pressure glow discharge plasma process (Sigma International APC 2000) in order to treat the surface of the jute fabric. This device contains two aluminium electrodes mounted above a ceramic-coated aluminium roller electrode. Because of the nature of the electrodes and issues related to instability of the plasma, helium is recommended as the main stream plasma gas (using a fixed flow rate of 14 L/min). Other gases often generate an undesired localised corona discharge rather than a uniform plasma treatment. Although helium is not overly economical (approximately USD $ 1 per litre), the surface area of the roller allows to treat up to 0.25 m^2 of fabric per revolution.

Three different gas mixtures were used to treat the jute fabric: (1) helium (He) (Flow rate: 14 L/min); (2) helium (He) and acetylene (Ac) (Flow rates: 14 L/min and 0.7 L/min, respectively); and (3) helium (He) and nitrogen (N) (Flow rates: 14 L/min and 0.7 L/min, respectively). For clarity of discussion, gas treatments (1), (2) and (3) will be referred to as He, He/Ac, and He/N respectively throughout the text. The fabric was attached to the treatment roller and rotated past the plasma source. Treatment was done for 5, 25, 50 and 100 revolutions of the treatment roller. The fabric was exposed to the plasma source for 0.424 s per revolution. The power of the treatment plasma was 970 W and the frequency of the power supply was 90 kHz.

2.2.2. Lay-up process

Square samples of woven jute fabrics (250 mm in length) were used to produce the composites. Polyester resin premixed with 1 wt% MEKP (approximately 300 g in total) was applied evenly onto each layer of woven jute fabric with an approximate resin to jute weight ratio of 3:1. For the overall stacking sequence, a total of 12 layers of fabric were placed with a [0/90] orientation (in both warp and weft directions) on a stainless steel mould coated with a poly (vinyl alcohol) (PVA) release agent. A breather material used as the final layer and the stack was vacuum bagged at −85 kPa before curing.

2.2.3. Composite fabrication: quickstep process

Composite panels cured using Quickstep QS5 Technology was positioned in a clamshell style mould with flexible silicon bladders containing a heat transfer fluid (HTF). The pressure created by the HTF within the bladder system during the curing process was 10 kPa at maximum. Two thermocouples were inserted one on each side of the laminate before vacuum bagging to record resin temperature and to control the cure cycle. The laminates were heated from room temperature to 95 °C at a rate of 3 °C/min, followed by a dwell time of 30 min. The DSC data in our previous publication [20] showed that the resin was fully cured using this cure cycle (data not shown here). The resultant composites were stored in the fumehood overnight to remove unfixed styrene vapour.

2.2.4. Wicking measurement

Six discs of equal size were cut from the jute fabric using a hole puncher to measure the wicking of the jute fabric. A 500 ml glass beaker half filled with distilled water was conditioned in the laboratory environment for 8 h prior to the test. Each sample disc was dropped into the water and the time required for water to penetrate completely into the fabric disc was recorded for each sample.

2.2.5. Scanning probe microscopy

2.2.5.1. Surface topography. Topographic height surface images for both untreated and plasma treated fibres were collected with a Digital Instruments Dimension 3000 SPM in contact mode using silicon nitride probes with pyramidal tips on cantilevers with a low spring constant (0.12 N/m). At least three single fibres were pulled out from the fabric and fixed on a glass slide with double sided tape. The test was repeated on a minimum of five different spots (5×5 μm) on each fibre to obtain a representative fibre surface topography.

The surface roughness was quantified by two parameters viz. average roughness (R_a) and root-mean square roughness (R_{rms}), which represent the standard deviation of the z values within a given area and the arithmetic average of the deviations from the centre plane, respectively. The surface roughness, R_{rms} and R_a were calculated using NanoScope software (V5.31) [21].

2.2.5.2. Adhesion. Force–volume mode (contact) with Digital Instruments Dimension 3000 SPM using the same tip was used to collect 16×16 arrays of force–distance curves in an area of 5×5 μm of the surface of a single fibre. Data were collected from 5 to 6 areas over 2 single fibres at ambient condition. The data were then converted to adhesion maps using an in-house developed volume data analysis software. Up to 256 adhesion data points were collected from various locations on the surface of the studied fibres using in-house developed software. The resulting data were statistically analysed by the histogram technique following our previously reported method [22]. Adhesion values are reported in nm of deflection. Approximate values of adhesion are reported in terms of nm deflection cantilever can be obtained by multiplying with the nominal spring constant (0.12 N/m).