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Test method

Towards the development of laser shock test for mechanical characterisation of fibre/matrix interface in eco-composites

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A B S T R A C T

This paper deals with the possibility of using the laser shock test for studying the adhesion between fibre and matrix in composite materials. Single hemp yarn in epoxy matrices - a fully synthetic one, Epolam 2020, and a partially bio-based one, Greenpoxy 56 - specimens have been tested. The water sorption effect on interfacial adhesion quality has been studied. Two different types of damage induced by laser shock have been observed: resin cracks appear only for high laser intensity levels, and specific cone-shaped interfacial damage appears for lower intensity values. The reproducibility of the threshold value evaluation has been demonstrated for the two resins. A numerical simulation by finite elements has also been performed to enhance the understanding of laser shock wave propagation in such samples. These preliminary results demonstrate the ability of the laser shock test to study and quantify the mechanical quality of yarn/matrix interface, which is needed to help design of such composites.

1. Introduction

Eco-composites are nowadays increasingly used for their numerous advantages [1]. However, one of the main problems when considering these materials is the weak fibre/matrix adhesion between the hydrophilic reinforcement and the hydrophobic matrix. This “mismatch” leads to a lack of compatibility between natural fibres and polymers. It causes poor adhesion between fibres and matrix, which reduces mechanical properties. Adherence can be seen as the sum of different contributions: interaction forces (like van der Walls), chemical bonds, residual stresses, etc. The crucial issue of adhesion between natural fibres and polymers has been examined in several review papers on green composites [2–5]. For example, Ku et al. demonstrates that the tensile properties of natural fibre reinforced polymers (both thermoplastics and thermosets) are mainly influenced by the interfacial bonding between the matrix and the fibres [2]. La Mantia et al. presented the role of different adhesion promoters, additives or chemical modifications that can enhance the interfacial adhesion [3]. To better integrate eco-composites on an industrial scale, it is necessary to improve the interfacial adhesion between natural fibres and polymers. Indeed, the best performance strength of a composite material is achieved when the ability to transfer stress across the fibre-matrix interface is high, i.e. when adhesion is the best. Therefore, the characterisation of fibre/matrix adhesion is an essential point for composite materials optimisation. Different types of tests are classically available, such as the fibre indentation test (push-out), the fibre extraction test (pull-out), the fragmentation test or the microbond test. In literature, there are plenty of papers dealing with these tests on synthetic fibres, for example on glass fibre with epoxy

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matrix [6–8], or on carbon fibres in acrylate or epoxy resin [9–11].

Concerning natural fibre composites, the influence of interface quality is highlighted in several publications [12–14]. Some papers on interfacial property measurements have been published, but many fewer than in case of synthetic fibres. For example, Doan et al. [15] and Sydenstricker et al. [16] present the pull-out test on sisal fibre in polyester matrix and jute fibre in epoxy matrix, respectively. Microdroplet debonding test was used by Park et al. [17] on hemp and jute fibres with polypropylene matrix and by Eichhorn et al. [18] on hemp fibre in epoxy matrix. Torres et al. [19] investigated the single fragmentation test on sisal-PE composite. Few papers deal with interfacial tests with a single yarn, for example on a hemp yarn with epoxy matrix [20] or on a hemp yarn with soy resin [21].

In this paper, the objective is to use another type of interfacial adhesion test, based on the laser shock wave method. This technique was first developed by Vossen and Gupta [22,23]. It can create a short but intense internal loading in the shocked sample. Thanks to the wave transmission/reflection through the irradiated material, localised tension in the loading direction can be generated. These stresses could open or not the interface. The laser shock adhesion test (LASAT) technique was developed for many applications, especially for metal assemblies or metal coatings, for which it is now well understood [24]. Recently, this technique has also been successfully used and optimised to test different bonding strengths of composite assemblies [25].

Following this principle, the present paper aims to investigate the possibility of using the laser shock adhesion test to study the adhesion between a single hemp yarn and an epoxy matrix. The characterisation of fibre-matrix interface has been performed on two different epoxy resins: a fully synthetic one and a partially bio-based one. Moreover, as the influence of moisture is a crucial problem in such composites, some samples have been tested after water sorption. After the shocks, all samples were recovered and damage micromechanisms have been analysed by microscopic observations.

The test campaign has been conducted using a laser source available in the Pprime Institute (France). Several samples have been tested in each configuration, with different laser shock intensities, in order to determine corresponding damage threshold of the fibre/matrix interface.

A first numerical modelling has also been developed to enhance the understanding of the complex phenomenon of laser-induced shock wave propagation in such single yarn samples.

2. Shock wave technique

The principle of shock generation by use of laser source is described in Fig. 1. The laser is focused on the specimen surface. An aluminium coating forces the laser/matter interaction to be produced on the sample surface, thus enabling shock generation inside the target. This is necessary because epoxy resin is about 70% transparent to the 1053 nm wavelength laser source. The aluminium coating previously deposited is ablated into high pressure plasma which expands rapidly. A shock wave is created by reaction inside the material (see Fig. 1a), and the propagation of shock waves through the specimen thickness can first be simply described by a schematic space/time diagram (see in Fig. 1b) [26,27]. Shock propagates through the specimen according to the material properties and geometry. When reaching the sample back face, this incident shock wave is reflected into a release wave propagating backwards due to impedance mismatch. This release wave then meets the incident release wave coming from the front-face and initiated at the end of the loading (back to the initial state). This crossing of two release waves leads to localised high tensile stresses which can damage the material if the local damage threshold is exceeded. This approach is quite simple, and relies on a one dimension description. In reality, phenomena are much more complicated and will be comprehended thanks to numerical simulation.

The Nd-Yag laser source available at Pprime Institute has the following characteristics: a wavelength of 1053 nm, a pulse-width about 25 ns and a maximal energy level of 20J. The energy level can be tuned by using optical densities in the beam path. Water confining was used to enhance the plasma expansion effect. It helps increasing the pressure level, and also increases the duration of the resulting pressure loading. The spatial distribution of the laser pressure profile is shown in Fig. 2. This representation is based on a visualisation in relief of a grey-scaled image of the laser beam. This image was recorded on a CMOS sensor thanks to a partially transparent mirror. The beam is circular with a total diameter of 4 mm (see in Fig. 2, for height zero). The vertical z-axis is proportional to the energy level of the laser beam. One can see that the laser energy is not perfectly constant over all the beam section. It is maximal in the centre of the beam, and decreases on the edges. This phenomenon is inherent to the laser technology of Nd-glass phosphate rods pumped by peripheral flash lamps: the spatial distribution is an almost top hat pattern with peripheral diffraction effects leading to a smooth decrease of energy at the boundaries of the rod. This particular shape will be taken into account for pressure profile in the finite element model.

3. Material and specimen preparation

The composite material studied is made of a single hemp yarn embedded in epoxy resin. The yarn is made of hemp fibres which have an average diameter of 13 ± 5 μm [28]. The yarn was produced with twist level of 324 tpm (yarn surface twist angle of 11°) and a linear density of 83tex. Besides the irregular cross-section, the hemp yarns have an apparent diameter of about 300 μm [20]. The fully synthetic epoxy resin, Epolam 2020, has a density of 1.10 g/cm^3 for cured resin [29]. The partially bio-based resin, Greenpoxy 56, contains 56% of bio-based carbon atoms, with density of 1.10 g/cm^3 (according to the manufacturer's data-sheet). Table 1 presents the values of the mechanical properties used in finite element model for the different materials. Composite plates were manufactured at Pprime Institute by casting. A specific mould has been developed, enabling hemp yarns to be well-positioned, with a distance