

Fiber-optic sensor design for chemical process and environmental monitoring

R.S. Mahendran, L. Wang, V.R. Machavaram, S.D. Pandita, R. Chen, S.N. Kukureka, G.F. Fernando *

Sensors and Composites Group, School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

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ABSTRACT

“Curing” is a term that is used to describe the cross-linking reactions in a thermosetting resin system. Advanced fiber-reinforced composites are being used increasingly in a number of industrial sectors including aerospace, marine, sport, automotive and civil engineering. There is a general realization that the processing conditions that are used to manufacture the composite can have a major influence on its hot-wet mechanical properties. This paper is concerned with the design and demonstration of a number of sensor designs for *in situ* monitoring of the cross-linking reactions of a commercially available thermosetting resin system. Simple fixtures were constructed to enable a pair of cleaved optical fibers with a defined gap between the end-faces to be held in position. The resin system was introduced into this gap and the cure kinetics were followed by transmission infrared spectroscopy. A semi-empirical model was used to describe the cure process using the data obtained at different cure temperatures. The same sensor system was used to detect the ingress of moisture into the cured resin system.

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1. Introduction

Fiber-reinforced composites (FRC) consist of three primary components: (i) the fiber, (ii) the matrix and (iii) the interface between the fiber and the matrix. The properties of FRC are, in general, dictated by the nature of the reinforcing fibers and the interface. However, the matrix can influence the so-called hot-wet and compressive properties of FRC. A number of previous studies have shown that absorbed moisture and elevated temperatures can cause significant and detrimental changes in the mechanical properties of FRC. Absorption of moisture can bring about changes such as (i) lowering of the glass-transition temperature (T_g), (ii) reduction in the resin modulus over a wide temperature range, (iii) swelling stresses induced by absorbed moisture and (iv) degradation of the resin and leaching, especially at high temperatures and prolonged exposure. The response of resins to moisture can be influenced by several factors including (i) exposed area, thickness of the sample, (ii) temperature, relative humidity and (iii) the cross-link density, morphology, free-volume, functional groups present and the resin-hardener system used.

A wide range of fiber-optic sensor systems have been used for monitoring the cross-linking process and these can be classified into qualitative and quantitative techniques [1]. The qualitative techniques include intensity-based sensor designs [2–4]. Quantitative analysis of the cross-linking kinetics is obtained using

sensor designs that enable UV-visible [5], infrared [6] and evanescent wave [7] spectra to be obtained. The development of a multi-functional sensor for monitoring various parameters of relevance to thermosetting resins and composites has also been reported recently [8].

A number of papers have also reported on the use of optical fiber-based sensor designs for sensing humidity [9–11]. However, only a limited number of publications have reported the use of optical fiber sensors to study the diffusion of water in thermosetting resins [12–14]. In general, optical fiber-based sensor designs for detecting moisture ingress tend to be based on polymer-coatings on a de-clad portion of the wave guide; the presence of chemicals are detected using evanescent wave spectroscopy [15–17].

The versatility of the extrinsic fiber Fabry–Perot (FP) interferometric (EFPI) sensors was demonstrated in a previous publication [18]. This current paper exploits the basic EFPI sensor design and describes three simple sensor configurations to monitor the cross-linking reactions in a commercial thermosetting resin. One of the sensor designs was then used for monitoring the ingress of moisture in the cross-linked resin.

2. Experimental details

2.1. Fabrication of sensor packaging fixtures

The basic sensor configuration investigated here involved securing cleaved optical fibers with a defined end-face separation.

* Corresponding author. Tel.: +44 121 414 8244; fax: +44 121 414 5232.
E-mail address: g.fernando@bham.ac.uk (G.F. Fernando).

The gap between the cleaved optical fibers was used as a cell for conducting transmission infrared spectroscopy of the resin system. Three types of fixtures were fabricated and evaluated for securing the cleaved optical fibers in position.

Schematic illustrations of the fabrication sequence for the first sensor design (Type-I) are shown in Fig. 1(a)–(c). Here, a precision-bore capillary tube with inner and outer diameters of 128 and 300 μm , respectively, was secured in a silicone mould as illustrated in Fig. 1(a). An epoxy/amine thermosetting resin system, LY3505/XB3403, (Huntsman Advanced Materials, UK) was poured into the mould. The mould was then transferred to an air-circulating oven that was set at 60 °C and the resin was processed for 8 hours. After this period, the oven was switched off and the mould was allowed to cool to room temperature. The cross-linked resin, with the embedded capillary, was removed from the mould, see Fig. 1(b).

The dimensions of the cross-linked resin sample were 40 mm (length), 5 mm (width) and 0.5 mm (thickness), see Fig. 1(b). A sacrificial optical fiber with an outer diameter of 125 μm was inserted into the embedded capillary. The function of the optical fiber was to support the capillary and prevent debris from contaminating the bore during subsequent cutting and grooving operations. A diamond-tipped cutting wheel (0.5 mm thick and 22 mm diameter) was used to obtain rectangular sections of the cross-linked resin with the embedded capillary and sacrificial optical fiber. The edges of the short rectangular sections were smoothed using 1200 grit SiC grinding papers. The approximate dimensions of these rectangular sections were 15 mm (length),

4.2 mm (width) and 0.5 mm (thickness). A groove was cut across the rectangular section using the diamond-tipped cutting wheel. The sacrificial optical fiber section was removed and the bore of the capillary was cleaned using a 125- μm -diameter tungsten wire. A cleaved multi-mode step-index silica optical fiber was inserted into the embedded capillary as illustrated in Fig. 1(c). The cleaved optical fibers were secured in position using a UV-curable resin (UV 304-T). The gap between the cleaved end-faces of the optical fiber was set at 100 μm . A photograph of the sensor assembly is shown in Fig. 1(d).

A schematic illustration of the second design (Type-II) is shown in Fig. 2(a). Here, a stripped optical fiber was inserted into two precision-bore capillaries of length 10 mm. This assembly was bonded on to a surface of another ‘substrate’ capillary using the UV-curable epoxy with a defined gap between the first two capillary end-faces. A schematic illustration of this design is presented in Fig. 2(a) along with a photograph in Fig. 2(b).

The third sensor design (Type-III) was achieved by partially abrading a quartz capillary along its length. Prior to the abrading operation, a sacrificial optical fiber was inserted into the precision bore to support the capillary during the abrading operation. The capillary with the sacrificial optical fiber was mounted on a metal block with a V-groove of 0.375 mm depth and inclusive angle of 45° using low-melt mounting wax (Contamac Ltd, UK). The capillary with the sacrificial optical fiber were abraded and polished using 2400 grit SiC abrasive paper and 3 μm diamond paste, respectively. The partially abraded capillary was cleaned with isopropanol in an ultrasonic bath for 1 minute to remove any

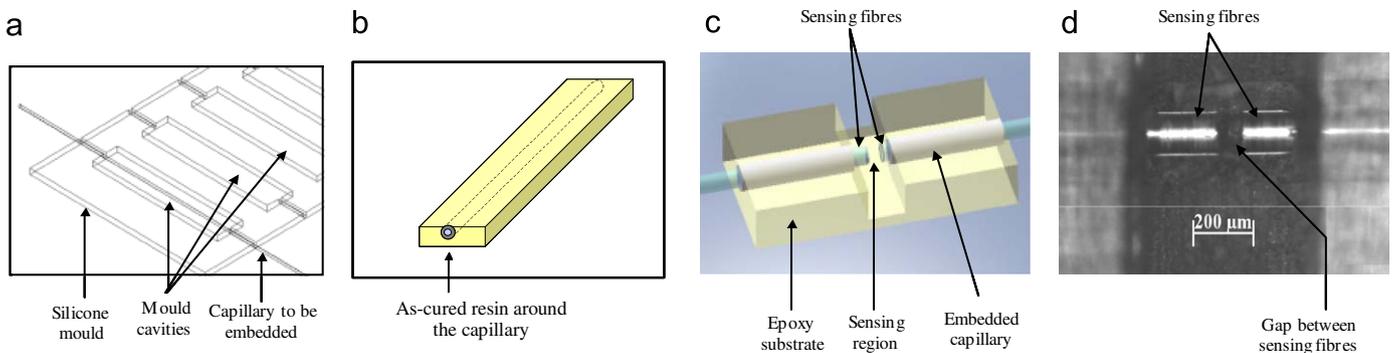


Fig. 1. Schematic illustrations of the fabrication sequence for sensor Type-I: (a) the silicone mould that was used to cast the resin around the capillary; (b) cross-linked resin with the embedded capillary; (c) a rectangular cell for conducting infrared spectroscopy and (d) micrograph of the sensor assembly.

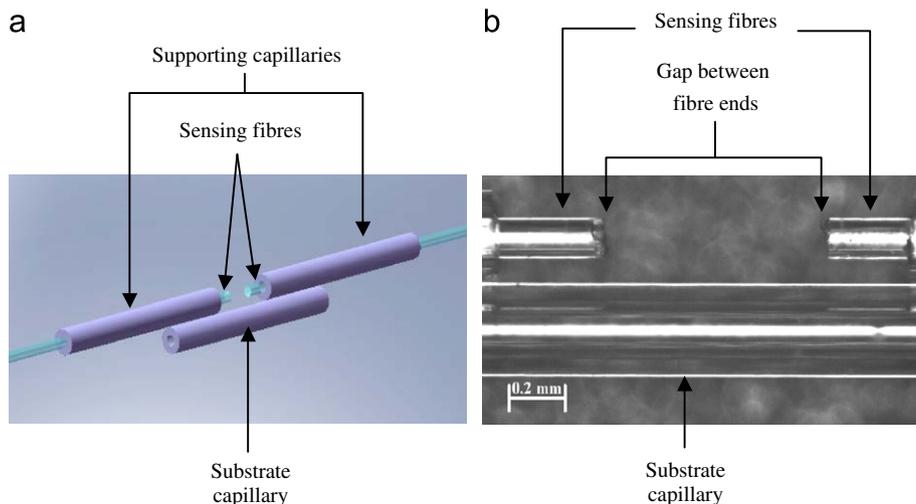


Fig. 2. (a) Schematic illustration of sensor Type-II and (b) micrograph of the sensor assembly.

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