Fiber Optics Technique for Quality Control and Monitoring of FRP Installations

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Synopsis: Advantages such as light weight, high mechanical properties and resistance to aggressive and chemical agents, non invasive installation, low tooling and machinery costs, possibility to repair the structure without interrupting its use have determined great expectation on Fiber Reinforced Polymer (FRP) composites for the strengthening of civil structures. However, their diffusion is often limited by the lack of procedures and methods to assess the quality of installations. The main focus of this paper is to present a non destructive methodology based on a fiber optic refractometer for the quality control of FRP wet lay-up systems. After summarizing the basic parameters necessary for monitoring the cure reaction and introducing the principles of the proposed technique, some preliminary experimental tests are discussed. The analysis of laboratory outcomes allows identifying the aspects that need further research before the proposed technique can be implemented as a field tool for quality assessment of FRP installations.

Keywords: curing; fiber optic sensors; quality control; refractometer; wet lay-up systems

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INTRODUCTION

Over the last years it has been recognized that FRP materials could represent a very effective solution for the upgrade of existing structures and their installation has been done in the majority of applications by manual wet lay-up. This installation procedure is characterized by the mixture of a two-component resin, typically epoxy, that is then used to impregnate a dry FRP sheet. Once impregnated with resin, the FRP system goes through a chemical process over time that provides the cured laminate with desirable properties. The success of such chemical process is dependent upon many factors such as: use of proper mixed amounts of each resin component; storage environmental conditions of resin components; surface temperature, moisture and exposure; atmospheric conditions during application; curing of the resin (Nanni et al. 2001). This means that the structural performance of the externally bonded reinforcement are strongly dependent on the proper and full curing of the resins used to impregnate the fibers. Such aspect assumes even more importance in the case of some emerging strengthening systems where the resin is obtained by automatic mixture of the two components. The advantage of such techniques is that the resin can then be sprayed on the support with great saving of time and manpower; however, there is a high risk related to a potential improper automatic mixture that is hard to detect.

The importance of developing protocols for quality control of FRP installations is also recognized by European (*fib* bulletin 14 2001) and North American (ACI 440.2R.02 2002) design guidelines for FRP systems externally bonded to reinforced concrete structures. Even though both documents underline the need for quality control checks and suggest some standard tests, there is a lack of procedures and methods that could be used in practice to assess the quality of an FRP installation. The development of such a methodology could be key towards the definition of inspection protocols for FRP systems and it could make it possible to quantitatively assess the quality of its installation. By increasing the trust of practitioners and reducing the uncertainties and the risks related with the adoption of innovative materials, this achievement could represent a crucial step towards a wide diffusion of FRP in construction with high potential for new jobs and business opportunities in the construction industry.

The possibility of checking the quality of an FRP installation depends on the availability of a non-destructive technique (NDT) that allows monitoring some properties of FRP through the use of a certain type of sensor. During the last years, different systems have been applied to civil infrastructures. Electric sensors (Chen and Liu, 2003), Acoustic Emissions (Chang and Liu, 2003), Ultrasonic tests (Mariìn et al., 2003), piezoelectric sensors (Gowripalan, 2001), termography (Starnes et al., 2003) and microwaves (Feng et al., 2002) have been used to measure the strains in the FRP composite or to detect defects at its interface with the support. Lately, the possibility of using fiber-optic sensors to measure displacements, strains and temperature has been also demonstrated (Inaudi, 2003; Katsuki, 2003; Imai et al., 2003).

A large project is under development at the Center of Excellence on Structural Composites for Innovative Construction (SCIC) based at the University of Naples. Its overall objective is the implementation of a field protocol for the quality control of wet lay-up installations by means of an innovative NDT technique based on the use of fiber-optic sensors embedded through the reinforcing fibers of the FRP laminates. The sensors are connected to a refractometer that, using the Fresnel reflection laws, makes it possible to measure the variation of the refraction index associated to the reaction of reticulation and then monitor the advancement of the cure reaction of the resin. The present paper discusses the findings of a first set of research activities that have been focused on two main lines, namely: the kinetic characterization of a typical thermoset resin used to impregnate the fibers in civil applications, and the assessment of the potential of the fiber optic refractometer to provide reliable information about the advancement of the resin cure under typical conditions of temperature and humidity.

CURE REACTION MONITORING

Refractive index as a process state parameter

The Lorenz-Lorentz law (eq.1), applied to polymeric material (clarifies the relationship between the refractive index n, the density ρ and the polarizability β (Ku and Liepins, 1987):

$$\frac{n^2-1}{n^2+2}=\frac{N}{3M\varepsilon}\rho\beta \tag{1}$$

where N is the Avogadro number, M is the molecular weight of polymer repeat unit and ε is the free space permittivity. In other terms, the refractive index reflects the variation of the polymer density that is an useful process parameter in the case of thermoset manufacturing. In fact, the cure of low molecular weight prepolymer involves the transformation of a fluid resin into a rubber then into a solid glass; this is the result of the exothermic chemical reactions of the reactive groups present in the system, which develop a progressively denser polymeric network.

The growth and branching of the polymeric chains are due to intramolecular reactions that initially occur in the liquid state until a critical degree of branching is reached and an infinite network and an insoluble material is formed (gelation or gel point). After the gelation, successive crosslinking reactions increase the crosslink density and stiffness of the polymer is steadily increased, leading, at the end of the process, to the glassy structure of the fully cured thermoset (Nicolais and Kenny, 1991). Since the curing process strongly influences the density of the material, direct relation between the extent of cure and refractive index is expected. This finding leads to the possibility to develop a compact, robust, cost effective and high resolution fiber optic sensing system for non destructive evaluation of the polymeric matrix characteristic features during the manufacturing process.

Fiber optic refractometer

The use of fiber optic sensor offers a very powerful tool to perform remote, on-line, in-situ monitoring of composite manufacturing processes (Afromowitz et al., 1995; Cusano et al., 2000a); Lew, et al., 1984; Culshaw et al., 1997; Crosby et al., 1997). As well known, in fact, the fiber optic is free from electromagnetic interference, and is characterized by high chemical and high temperature resistance. Moreover, due to the capability of the fiber optical sensors to be multiplexed in a large number of independent channels, due to the fact that fibers are readily embedded into the composite and due to their small size that make them minimally intrusive in the host structures, this approach provides useful tools for implementing integrated sensing networks within the material itself.

The proposed sensor is based on the principle Fresnel reflection, here the transducer is simply the fiber-optic/host material interface. This leads to a more simple, less intrusive, and lower cost sensing system. The response of the sensor is a function of the mismatch in refractive index between the fiber optic and the host interface.

Figure 1 shows the optical part of the proposed sensor and Figure 2 is a picture of the optical refractometer and of the acquisition system during the consolidation of an FRP on a concrete specimen. A laser beam lights a mono mode optical fiber, which is embedded into the under test resin. At the interface between the fiber and the resin, due to the mismatch of the two refractive indexes (of the fiber core and the resin at the laser wavelength) part of the light beam is transmitted and part is reflected. The reflected signal is collected on a photodetector by means of a fibre Y-couple.

According to Fresnel's equations, the field amplitude reflection coefficients at the fiber-end/resin interface for the perpendicular and parallel polarisation, respectively r_n and r_p, can be expressed as (Cusano et al., 2000b):

$$r_n = \frac{n_f \cos\theta_1 - n_m \cos\theta_2}{n_f \cos\theta_1 + n_m \cos\theta_2}$$
$$r_p = \frac{n_m \cos\theta_1 - n_f \cos\theta_2}{n_m \cos\theta_1 + n_f \cos\theta_2} \qquad (2)$$

where ϑ_1 is the angle of incident light and ϑ_2 is the angle of the transmitted light, n_f and n_m are respectively the effective refractive index of the fiber and the sample refractive index. If step index optical fibre is used, a number of propagating modes are guided. If a mono mode fiber is used, it can be assumed that the fundamental guided mode travels along a paraxial path in the fiber, in this hypothesis, ($\vartheta_1 \cong \vartheta_2 \cong 0°$) and $|r_p|=|r_n|=r$. As a consequence, the intensity reflection coefficient R can be expressed by (Ku and Liepins, 1987):

$$R = \left|r_p\right|^2 = \left|r_n\right|^2 = \left|\frac{n_f - n_m}{n_f + n_m}\right|^2 \qquad (3)$$

Thus, by monitoring the light intensity reflected from the fiber end/host interface detailed information about the sample refractive index are available. In fact, the signal at the photodetector V in hypothesis of monochromatic light source can be expressed as:

$$V = h \;\; \beta \;\; \alpha \;\; P_{INC} R \qquad (4)$$

where h is the gain of the photodetector, α the coupling coefficient introduced by the 1x2 coupler, β accounts for signal losses along the optical chain and P_{INC} represents the optical power impinging the sample/fiber end interface.

EXPERIMENTAL RESULTS

The studied resin system was a mixture of an epoxy resin MAPEWRAP31 and its hardener (amine) supplied by Mapei (2003). The amine to epoxy volumetric ratio was 4:1 as suggested by the supplier. The complete consolidation of this resin at environmental temperature occurs in 7 days. Therefore, at first, the polymerization of the MAPEWRAP31 resin has been analysed at 70 °C, that is a sufficient level of temperature to attain a complete conversion. Its kinetic behaviour has been investigated both by measuring the refractive index with the developed fiber optic refractomer and performing a Differential Scanning Calorimetry (DSC) experimental test. The DSC is a conventional technique, suggested also by the European and American guidelines for the cure monitoring of FRP. It allows to investigate the material phase transitions and the chemical kinetic by measuring the absorbed or released heat flow during a dynamic or an isothermal test. Since the resin polymerization is an exothermal chemical reaction, the measurement of the heat released by the analysed sample gives indications on the chemical conversion.

Figure 3 compares readings of the degree of cure (DoC) over time obtained from both DSC test and refractive index measurements; at any time t, DoC is computed according to the following relationship:

$$DoC = \frac{f(t) - f(0)}{f(\infty) - f(0)} \quad (5)$$

where f(t) is the heat emission in the DSC test and the refractive index in refractometer test, measured between 0 (beginning of test) and ∞ (time when the asymptotic value is reached). A good agreement can be observed between the two sets of data confirming the capability and reliability of the fiber optic sensor apparatus. For more details about the data analysis both by DSC and fiber optic refractometer see Cusano et al., 2000.

This preliminary test was useful to know the required time for the resin to attain a specified conversion level at 70 °C. Then, various experimental tests have been performed at selected degrees of cure and at different temperature values between 10-35 °C that is the effective operative range. In particular, the resin refractive index has been measured in the temperature range 10-35 °C at conversion levels of 0, 0.3, 0.7, 1. To attain an incomplete cure reaction (i.e., DoC of 0.3 and 0.7), the analysed sample has been first subjected to an isothermal heating at 70 °C for 5 and 15 min, respectively; according to the DSC curve shown in Figure 3, these time values correspond to DoC of 0.3 and 0.7, respectively. As an example, Figure 4 reports the refractive index as function of time during the whole experimental test for the conversion level of 0.7. As expected, since the refractive index is related to the density (see eq. 1), one should observe that, at the beginning, the refractive index decreases as the temperature increases due to the resin density reduction.

Then, as the polymerization reaction takes place, the optical properties changes result dominated by the formation of cross-links between the reactive groups of the polymer resin that develop a progressively denser network. This behaviour can be observed also in the diagram of Figure 5, where the refractive index of the partially cured resin is shown as function of temperature during the cooling and heating in the temperature range 10-35 °C. Figures 6 and 7 summarize the results of whole experimental tests reporting the resin refractive index as function of temperature and of the cure degree. The availability of these diagrams could be useful during the in-situ application of this resin system allowing to know if the resin has completely reacted by measuring the environmental temperature and the refractive index.

CONCLUSIONS AND FUTURE WORK

The preliminary assessment of the potential of using the proposed system to check the degree of curing of the resin has demonstrated that it could be possible to embed fiber-optic sensors into the laminates in order to monitor the advancement of the curing reaction of the resin over time in conjunction with temperature measurements. The experimental data herein discussed represent a first set that will need to be enriched in order to calibrate a direct relationship between the degree of cure and the refractive index. Once the behaviour of the resin has been studied and a reliable tool is available to follow the evolution of the resin reaction, the next effort will be devoted to defining reference values that, for different combinations of environmental temperature and humidity, can be used to judge whether the quality of an FRP installation is structurally acceptable and eventually plan the appropriate actions necessary to overcome curing problems in order to make the installation satisfying the minimum quality requirements.

The availability of such instructions collected into a manual could be of crucial importance for contractors, practitioners and inspectors because it will contain responses to present needs, namely: how the curing reaction of the resin can be controlled after the execution of a manual wet lay-up process and which actions should be taken in order to solve issues related to an imperfect curing of the resin. However, the information obtained at material level are not enough to achieve this goal. It is necessary to move the attention from the material level to the structural level and assess which is the minimum amount of chemical imperfection that could result in a structural imperfection; this is done with the objective of avoiding negative evaluations of installations that are not perfect from a chemical standpoint but whose chemical imperfections do not affect the structural effectiveness.

In order to define these tolerance thresholds for imperfect resin curing in terms of structural performance, it is planned to perform pull-off tests on FRP laminates bonded on concrete or masonry supports. The outcomes of these tests will highlight the influence of different levels of imperfect curing on the bond of the FRP laminates and provide a database to determine at which extent a deviation of the key parameters (environmental conditions, support conditions, mixing proportions) from the standard conditions indicated by the manufacturers could be still structurally accepted. It is also planned to perform tests on structural elements in order to check the feasibility of the overall quality control protocol. FRP laminates will be installed on concrete and masonry specimens and fiber-optic sensors will be embedded through the dry fibers. Many samples will be prepared by changing environmental and concrete surface conditions. Readings of the sensor will be used to test if the technique is able to highlight the curing problems theoretically expected for each given installation condition created in the laboratory. The results will be important to check the sensitivity of the sensors to the rolling of fibers typically performed during the impregnation by manual wet lay-up.

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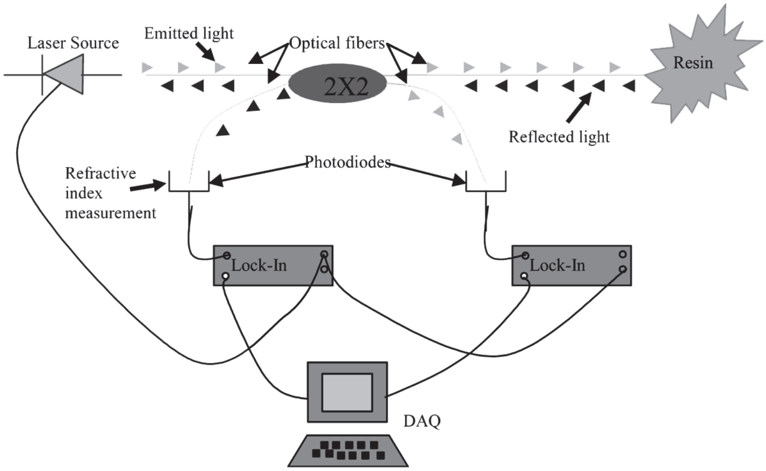

Figure 1 — Schematic of the fiber optic refractometer.

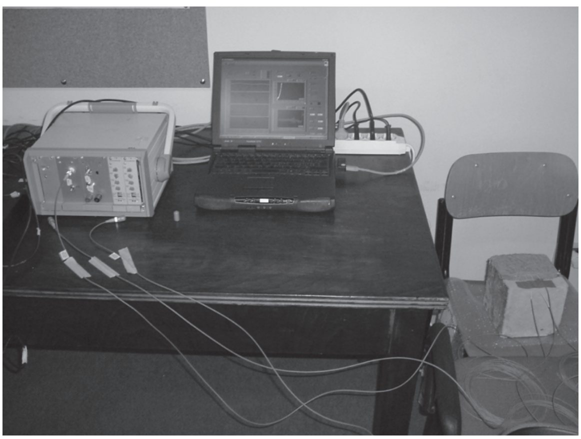

Figure 2 — Fiber optic refractometer and its in-situ application.

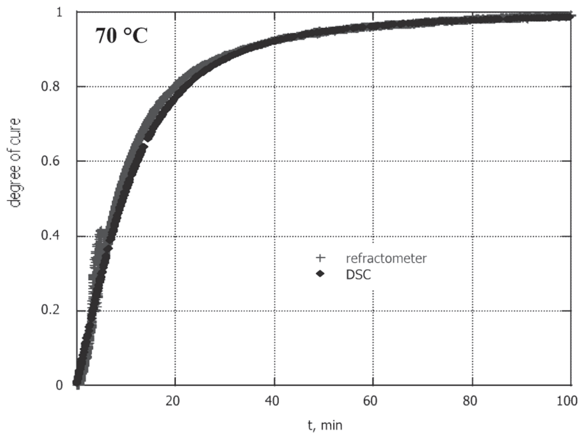

Figure 3 — Conversion evaluated by DSC test and refractive index measurements.

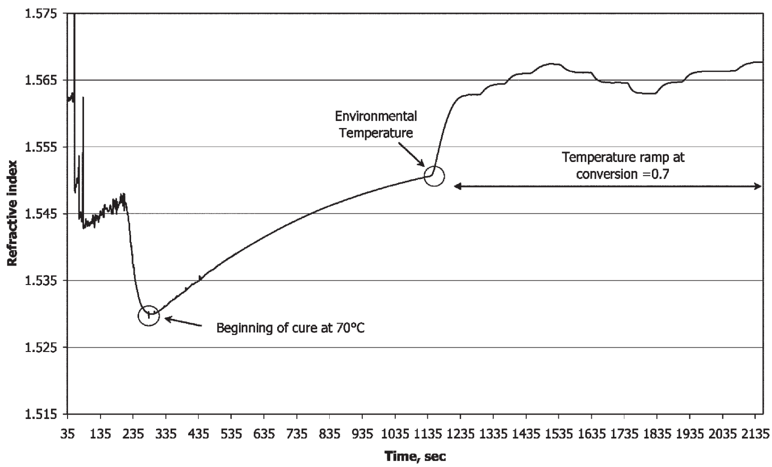

Figure 4 — Refractive index as function of time during the incomplete polymerization at 70°C and the temperature ramps.

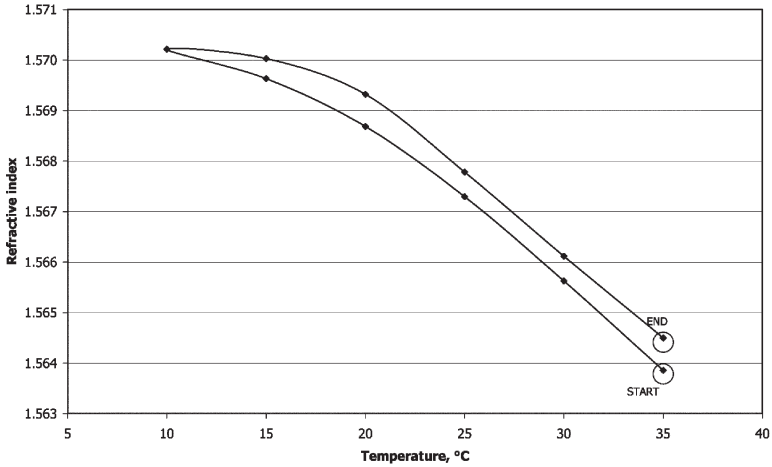

Figure 5 — Refractive index of the partial cured resin (conversion=0.7) as function of temperature during the temperature ramps in the range 10-35 °C.

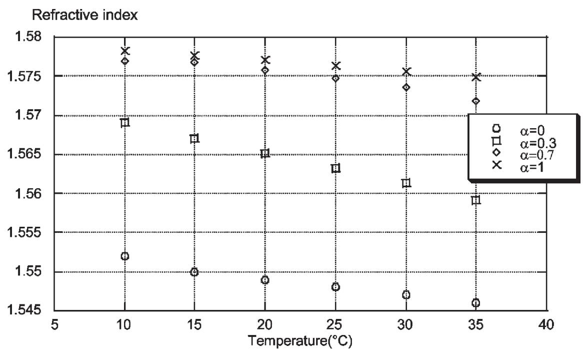

Figure 6 — Refractive index as function of temperature for different level of resin conversion.

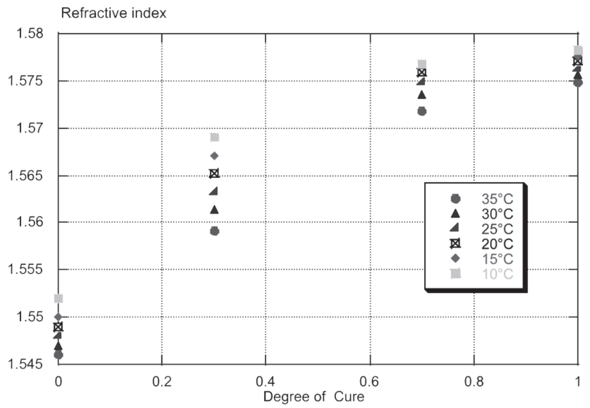

Figure 7 — Refractive index as function of cure for different temperature levels.