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► **To cite this version:**

Rochdi El Abdi, Jean-Christophe Sangleboeuf. Effects of immersion duration in water on strength of instrumented composite materials. The Romanian Review Precision Mechanics, Optics and Mechatronics, 2015. hal-02385063

**HAL Id: hal-02385063**

**<https://univ-rennes.hal.science/hal-02385063>**

Submitted on 5 Dec 2019

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# Effects of immersion duration in water on strength of instrumented composite materials

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**Abstract** - The present paper reports on the study of the interfacial adhesion of an optical fiber embedded in a composite material aged in distilled water. Samples composed of optical fibers embedded in an epoxy vinylester resin or in resin/glass fibers systems were submitted to different aging duration in distilled water. Pull-out tests on optical fibers were carried out to measure the effect of water diffusion and glass concentration on fiber bonding. For resin/optical fiber samples, water diffusion leads to damage of the polymer/matrix interface from an aging duration of 15 days and the interfacial stress values present a low decrease up to an aging duration of 60 days. For the case of composite/optical fiber samples, the greater the glass fiber content, the less is the water damage at the polymer interface. On the other hand, a linear development of interfacial debonding stress with increasing glass fiber concentration is reported.

**Keywords:** Water diffusion, optical fiber, interfacial adhesion, smart composite materials, pull-out test.

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## 1. Introduction

The development of so-called smart structures, such as composite structures into services in which optical fibers such as sensors have been integrated, has led to characterization studies and analysis of the implementation of these fibers in composite materials that will be used in civil engineering work (bridges, footbridges, gateways, beams, etc.) [1]. The term “smart” comes from the dual functionality provided by the insertion of the optical fiber sensors into composite materials that provide information on the continuous evolution of damaged structures under mechanical stress [2].

But composite materials present some disadvantages, such as susceptibility to moisture absorption, and a non-negligible loss of tensile stiffness and strength can be obtained with immersion in water. For a long immersion period, the effect of water leads to significant damage to the interfacial shear strength [3].

The diffusion of water depends on immersion time and on many other parameters [4] (temperature, composition of resin and curing agent,...) and the

damage caused by the effect of water at the interface surface between an optical fiber embedded in the resin /reinforcement fiber composite is more complex to analyse and to quantify.

In this work, we developed composite specimens (epoxy vinylester resin + glass fibers) (with different fiber glass volumes) in which an optical fiber is embedded. These samples were immersed in distilled water (at laboratory temperature) for different durations (between 6 and 60 days).

## Methodology

### a/ Sample preparation

The optical fibers used are silica fibers with one layer of acrylate polymer coating. The diameter of the cladding is 80  $\mu\text{m}$  and the coating diameter is 101.8  $\mu\text{m}$ . These fibers are designed to be used at elevated temperatures and pressures in aggressive chemical environments.

The polymer resin used in this study is a mixture of epoxy vinylester resin provided by Derakane 470-36, and a catalytic system composed of Styrene, Percadox 16 and Trigonoc C, provided by DC

Pultrusion. The glass fibers used for the preparation of the composites come from “Roving Tex”. The powdered Perkadox 16 is firstly diluted in the styrene to form the “styrene/Perkadox 16” system. Then the Trigonoc C and the epoxy vinylester resin were added respectively. Before characterizing the interfacial adhesion between the optical fiber and the epoxy vinylester/glass fiber composite material, a simple system composed of an optical fiber embedded in epoxy vinylester resin was studied. The system is a unidirectional composite, where glass fibers have the same sample length direction and optical fiber direction. The objective was to obtain basic data on a simple system. A typical sample preparation consisted of pouring the resin into a mould and introducing the optical fiber into the middle of the sample. In the case of composites, the impregnated glass fibers were successively superimposed and the optical fiber gently introduced. Special care was taken to ensure that the optical fiber was straight. The set of moulds were then inserted into an oven at 80°C for 2 hours to polymerize the sample.

b/ Used tensile set-up

The single fiber pull-out test is a direct method for measuring the fiber matrix interfacial properties by the evaluation of the interfacial shear strength. Single pull-out tests were performed on optical fibers. The samples used for pull out tests were carefully prepared. If we applied a gripping force (imposed by the jaws of the LLOYD LR 50K tensile testing set-up) (Fig. 1) to the right hand side of the tested specimen and if the optical fiber was pulled, the fiber breakage occurred at the bit. Thus, a notch was introduced into the specimen and the jaws clamped the right hand part of the specimen. The crosshead speed was set at 1mm/min, which corresponds to a strain rate of about 0.04 min<sup>-1</sup>.

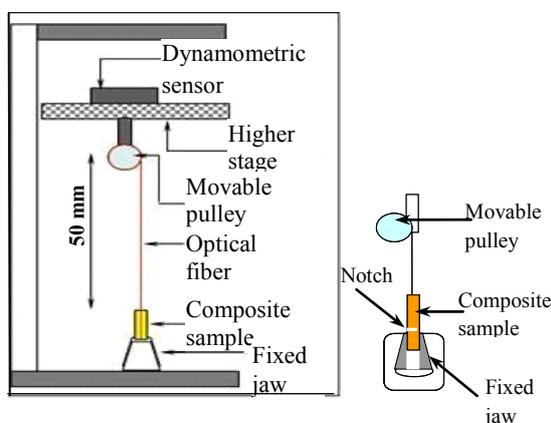


Figure 1: Description of the dynamic tensile test bench and detail of used composite sample

The debonding force  $F_d$  was taken as the maximum force preceding partial debonding. Different samples obtained with various glass fiber concentrations were tested.

c/ Water effect

The composite we studied will be a component of a civil engineering structure subject to bad weather. During manufacture, this structure receives protective layers to combat water diffusion. However, these protective layers are destroyed during its life and therefore the water diffuses through the composite material.

In recent years, many studies have focused on the analysis of moisture absorption in composite materials [5]. It has been shown that absorbed water can modify the elastic-plastic behaviour of the resin and lead to decohesion of the matrix/ fiber interface, and composite performance degradation may occur during use [6].

When composite material was aged in water, water diffusion had a noticeable effect on material properties and a study of the mechanical behaviour change of the interface between the matrix and optical fiber surface was undertaken.

Results and analysis

a/ Change of water content versus immersion time

The studied samples were either made of pure resin in which an optical fiber was embedded or made of resin and glass fibers with an embedded optical fiber. The composite samples contained different volume fractions of glass (0%, 40%, 50%, and 70%). The different samples were 30 mm in length, 10mm in width and 3 mm in thickness. For each case, four identical samples were used to obtain an average value.

The most used model to explain water recovery in polymers is classically based on the Fick's diffusion law [7] which gives water content changes versus the square root of time. Figure 2 gives the water content change for resin samples and for resin samples with an embedded optical fiber.

During the first 15 days, the water content increased in a linear manner with versus the square root of immersion time. After a transition period extending up to 50 days, the sample was saturated and water weight reached 1.25%.

Composite samples with various glass fiber concentrations were submitted to immersion in distilled water (Fig. 3).

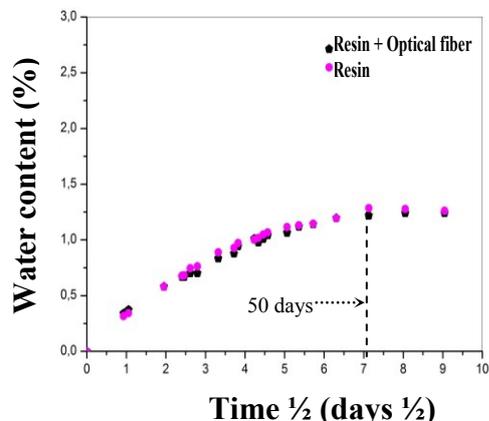


Figure 2: Water content versus immersion time for resin samples

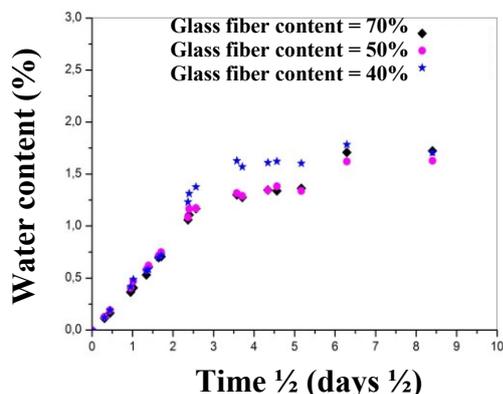


Figure 3: Water content for different glass fiber contents

Composite samples with 50% and 70% glass fiber content have similar behaviour. During the first 9 days, the water content had a linear behaviour, followed by a transition period up to 36 days, and then the saturation was obtained.

For the sample with 40% glass fiber concentration, the water content linearly increased up to 10 days then instantly stabilized.

For all the glass fiber contents, the same final water content of 1.7% (Fig. 3) was obtained. There was less water diffusion in resin samples (Fig. 2) than in composite samples (Fig. 3) where micro-voids and micro-cracks exist.

b/ Tensile tests analysis

Tensile tests on the optical fiber embedded in samples were undertaken:

a/ for resin samples immersed in distilled water for 10 days, 29 days and 60 days,

b/ for composite samples (with 40%, 50 %, and 70% glass fiber content) for 60 days.

\*Aging of resin samples

Figure 4 gives the force-displacement curves for different aging durations. All the force-displacement curves have the same appearance. The tensile deformation of the free part of the optical fiber gives the elastic behaviour. After the maximum value of the applied force, a harsh decrease is obtained and is indicative of decohesion of the optical fiber polymer. The second part of the curve indicates the optical fiber sliding in the resin. At first, this slippage is unstable (stick-slip), and then stabilizes at the end of the fiber pulling.

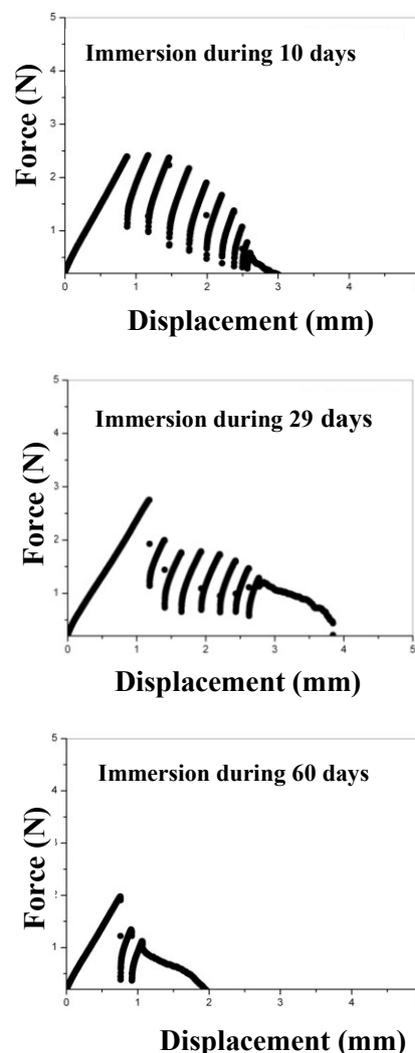


Figure 4: Force-displacement curves for different immersion durations in water for optical fiber embedded in resin samples and submitted to pull out test.

The mean interfacial stress decreased versus aging time. The water diffusion produced tensile radial stresses at the optical fiber/ resin interface and led to the interface decohesion. On the other hand, the water diffusion was near saturation after 15 days and the sample was completely saturated after 50 days. A decrease of resin/fiber adhesion was then obtained.

*\*Aging of composite samples*

Figure 5 gives the tensile tests results for composite samples with different glass fiber contents aged in distilled water for 60 days.

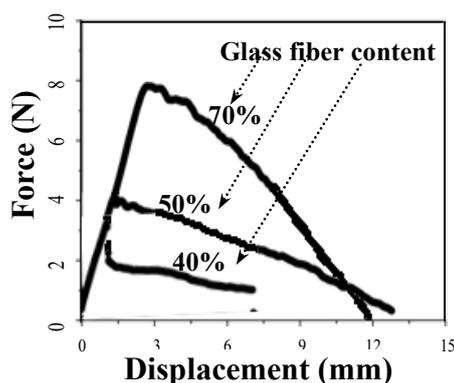


Figure 5: Force-displacement curves from pull-out test for composite samples aged in water for 60 days

All the force-displacement curves have the same appearance with a first zone corresponding to the elastic deformation of the free part of the optical fiber. After this phase, the interface decohesion was obtained. During the fiber slip in the composite, a decrease of applied force occurred until complete fiber extraction.

**Conclusion**

Using the tensile test procedure, the interfaces resin/optical fiber and composite/optical fibers were characterized after different aging durations in distilled water.

There was less water diffusion in resin samples than in composite samples where micro-voids and

micro-cracks exist. The damage of the interface between the polymer coating and the composite surface was due to the chemical-physical action of diffused water.

For resin/optical fiber samples, water diffusion led to polymer/matrix interface damage from an aging duration of 15 days and force values presented a low decrease up to an aging duration of 60 days. For the case of composite/optical fiber samples, the greater the glass fiber content, the lower the water damage at the polymer interface. We can also mention that glass fibers are stiffer than vinylester resin; therefore this effect can seriously affect the stiffness of the optical fiber surrounding the composite material.

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