

Dimensional stability of materials based on Portland cement at the early stages

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ABSTRACT

In this work two fiber optic sensing techniques are used to study the dimensional stability in fresh state of different cementitious materials. A conventional Portland cement mortar and two commercial grouts were selected. The measurements were performed by using a Bragg grating embedded in the material and a non-contact Fizeau interferometer. The first technique was applied in a horizontal sample scheme, and the second one, by using a vertical configuration. In addition, a mechanical length comparator was used in the first case in order to compare the results. The evolution with time of the dimensional changes of the samples and the analysis of the observed behavior are included.

Keywords: Portland cement-based materials, dimensional stability, fiber optic sensing, Bragg grating, Fizeau interferometer.

1. INTRODUCTION

Portland cement based materials usually undergo shrinkage deformations along their life. Some shrinkage can occur at the early ages, and even in fresh state, when the water is mainly lost by evaporation and in some degree by the hydration of cement. All concretes, even shrinkage-compensated ones, may experience some degree of contraction which potentially leads to cracking. For several applications, the shrinkage of concrete is one of the most important considerations. As an example, the study of expansion or contraction processes in the fresh state is a requirement for grouts or mortars specially designed for sealing parts of structural elements. To optimize its performance, shrinkage must be correctly understood and addressed¹.

In recent years, much attention has been paid to cases of early cracking of pastes, mortars and concretes made with Portland cement. However, the methods that enable to evaluate dimensional changes in plastic state (fresh, before the setting of the material occurs) are very limited, since any device that is fixed on the fresh material causes alterations on it. An alternative to make such determinations is provided by optical methods like the use of the fiber sensors².

Optical fiber sensors are employed in several application areas like aerospace, structural, medical and energy (petroleum and gas projects). Characteristics such as the small size, low cost, and the capability of avoiding electromagnetic influence are some of their advantages. In particular, the use of fiber Bragg gratings (FBG) in sensing applications (especially for temperature and strain measurements) has received considerable attention³⁻⁵. Moreover, optical fiber sensors have been used to assist in describing the course of drying and evaporation of cement pastes^{6,7}. On the other way, Fizeau interferometers have also been employed in monitoring the curing process of polymers^{8,9}.

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In this work, the dimensional changes of cement mortars were measured from the first minutes up to several hours using the mentioned interferometer and / or FBGs. Later, a mechanical length comparator was also used to contrast the results obtained with optical methods. In order to characterize their expansion or contraction processes, different types of premixed cement mortars were studied. The evolution with time of the dimensional changes of the samples and the analysis of the benefits of each technique are included.

2. METHODOLOGY

2.1. Techniques employed

a) Embedded Fiber Bragg Grating (FBG) sensor

The fiber Bragg gratings used in this work, were recorded in our lab employing the fourth harmonic (266 nm) of the Nd:YAG laser emission, by using the phase mask technique. They have a typical reflectivity around 70-90%, spectral widths of ~ 0.15 nm @-3dB, good symmetry and reasonable levels of side lobe rejection. The spectral evolution of the gratings was monitored with an optical sensing interrogator device (Micron Optics, model sm125) (see Figure 1).

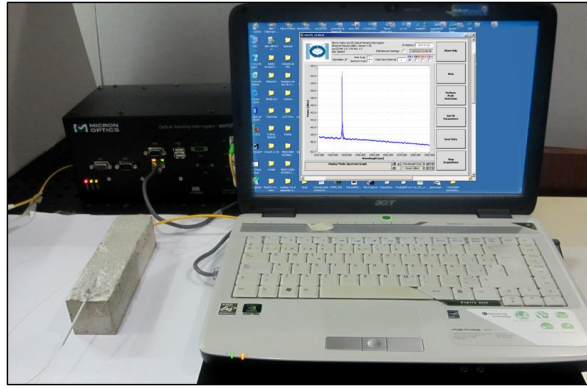


Figure 1. Setup for monitoring the spectral evolution of the FBG sensor.

The strain sensitivity S_ϵ and the temperature sensitivity S_T of the FBG sensor as a function of the changes in the refractive index of the optical fiber and the grating period with temperature and strain, can be defined as

$$S_\epsilon = \left\{ \frac{1}{n} \frac{\partial n}{\partial \epsilon} + \frac{1}{\Lambda} \frac{\partial \Lambda}{\partial \epsilon} \right\}; \quad S_T = \left\{ \frac{1}{n} \frac{\partial n}{\partial T} + \frac{1}{\Lambda} \frac{\partial \Lambda}{\partial T} \right\} \quad (1)$$

The normalized shift in Bragg's wavelength of the fiber grating subjected to both, strain (ϵ) and temperature (T) changes can be written as:

$$\frac{\Delta \lambda}{\lambda_0} = S_\epsilon \Delta \epsilon + S_T \Delta T \quad (2)$$

where $\Delta \lambda$ is the Bragg wavelength change and λ_0 is the initial Bragg wavelength. Besides, as can be seen from Ref. [10], $S_\epsilon = 1 - p$, being p the strain optical coefficient ($p = 0.22$ for silica fibers) and $S_T = \alpha_F + \xi$, with α_F the fiber optic thermal expansion coefficient ($\alpha_F = 0.55 \times 10^{-6} \text{ 1/}^\circ\text{C}$) and ξ the thermo-optic coefficient for a silicon fiber doped with germanium ($\xi = 7 - 8 \times 10^{-6} \text{ 1/}^\circ\text{C}$)^{11,12}. When the fiber sensor is bonded or embedded in a host structure of a different

material, the Bragg equation is modified to account for the thermally induced axial strain developed in the grating due to the mismatch between the thermal expansion coefficients of the optical fiber (α_F) and the host structure (α_H). In this case, a more generalized equation can be written as:

$$\frac{\Delta\lambda}{\lambda_0} = S_\varepsilon \{ \Delta\varepsilon + (\alpha_H - \alpha_F)\Delta T \} + S_T \Delta T \quad (3)$$

If the temperature change is negligible, the equation can be simplified as:

$$\Delta\lambda = \lambda_0(1 - p)\Delta\varepsilon \quad (4)$$

The change in the mechanical strain applied to the FBG sensor is related with the material deformation ΔL (expansion or contraction) through the expression:

$$\Delta\varepsilon = \frac{\Delta L}{L_0} \quad (5)$$

where L_0 is the initial length of the grating. From that value, it is possible to obtain the percent relative deformation of the material.

b) Fizeau Interferometer

Characteristics and properties of the Fizeau interferometer have been extensively described^{8,9}. Such interferometer operates by reflection and the output signal comes from the interference between rays reflected at the surface of the fiber optic connector and at the reflecting surface of the material which closes the cavity.

The phase φ of the signal modulation caused by the path difference of the interfering beams is given by:

$$\varphi = \frac{4\pi n_0 d}{\lambda} \quad (6)$$

where n_0 is the refractive index of the medium inside the cavity ($n_0 = 1$, considering air), λ is the working wavelength and d is the distance between the cavity surfaces. A maximum intensity is achieved when the phase of the interferometric signal is a multiple of 2π . It is clear that the separation between two consecutive wavelengths that produce maximum of interference occurs when

$$d = \frac{\lambda^2}{2(|\Delta\lambda|)} \quad (7)$$

with $|\Delta\lambda| = |\lambda_n - \lambda_{n-1}|$ and λ_{n-1}, λ_n are the wavelengths of two consecutive spectral maxima. Due to this, the accuracy of the technique allows to measure distance changes lower than one micron.

In practice, the interferometric cavity is formed between the end of the connector and the surface which is being measured. A basic diagram of the Fizeau set-up for a vertical arrangement is shown in Figure 2.

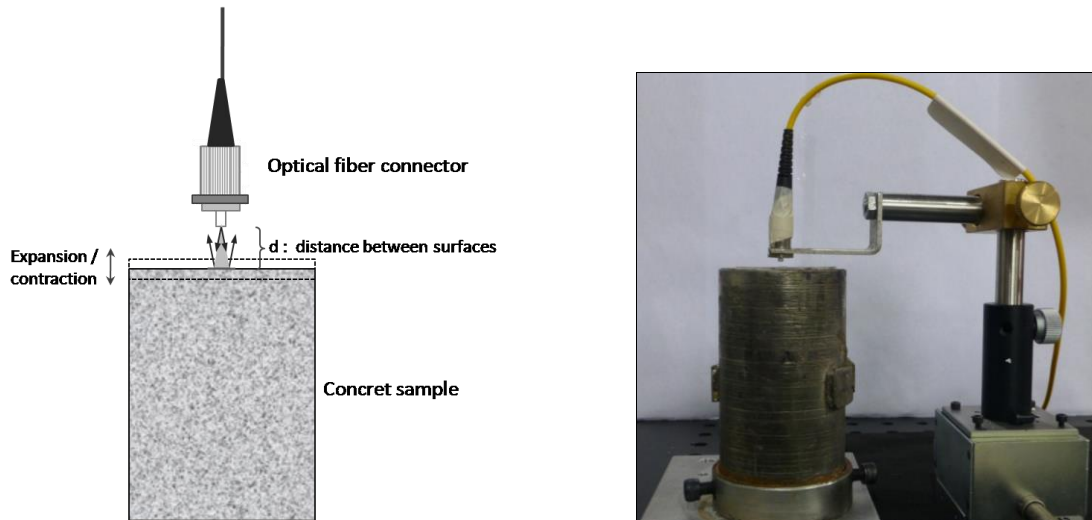


Figure 2. a) Scheme of the Fizeau interferometer set-up; b) Experimental arrangement.

Denoting with d_i and d_f the initial and final values of the distance between surfaces, respectively, and the absolute deformation of the material as $\Delta L = d_f - d_i$, we calculate the relative (%) deformation as

$$\frac{\Delta L}{L_0} [\%] = \frac{(d_f - d_i)}{d_i} \times 100 \quad (8)$$

where $L_0 = d_i$ is the initial length of the interferometric cavity.

c) Mechanical length comparator

It is a conventional device used to determine the change in the dimension of a mortar sample once the material has been stiffened, although with a very low strength level. It allows making comparative measurements respect to the initial value of the sample length. This digital comparator (SCHWYZ[®]) has a resolution of 0.001 mm. It was used to compare its measurement with the results obtained by means of the optical sensing techniques. Figure 3 shows a sample of the material being measured with this instrument.



Figure 3. Mechanical measurement setup

To find the deformation of the material using the mechanical digital comparator, the following expression is used:

$$\frac{\Delta L}{L_0} [\%] = \frac{\Delta L_{meas}}{L_0} \times 100 \quad (9)$$

being ΔL_{meas} the reading of the comparator, and L_0 the initial length of the sample.

2.2. Material analyzed

During the development of this work, mortar samples of different compositions illustrated in Table 1, were measured using the mentioned methods.

Table 1. Mixture proportions (kg/m³).

	S1	S2	S3
Water	385	263	422
Sand	--	1360	--
Grout-1	--	--	1880
Grout-2	1915	--	--
Portland cement	--	680	--

Samples S1 and S3 were made with two different types (Grout-1 and Grout-2) of premixed mortars and water. They are high strength grouts suitable for attachments, anchorages and fillings, whose formulations are based on the combination of cement, special additives and controlled grain sized aggregates. Sample S2 was a conventional Portland cement mortar, made with a fillerized Portland cement (CPF40), water and natural siliceous sand.

3. EXPERIMENTAL RESULTS AND DISCUSION

As was previously mentioned, the contraction or expansion processes that occurred during the first hours in cement mortars were measured by applying a fiber Bragg grating based sensor and a Fizeau interferometer. Both optical techniques allow for the monitoring of the dimensional evolution of these materials even in their fresh state, when mechanical methods cannot be employed.

By using the experimental arrangement shown in Figure 2 the interferometric measurement was performed. To do this, the sample was illuminated by employing a wide band superluminescent laser diode with a central emitting wavelength of 1550 nm, through a 2x1 3dB single mode fiber directional coupler. Light reflected by the sample interfered with rays reflected at the surface of the fiber optic connector and the resulting intensity was detected by an Optical Spectrum Analyzer (Yokogawa, model AQ6370B). The recorded signal was processed in order to obtain the time variation of the interferometric cavity length and therefore, the dimensional change of the sample. The absolute and relative deformations of the materials were calculated using the expressions mentioned in Section 2. Figure 4 shows the results obtained for samples S1 and S2. It is clear that, during the first hours, mortar S1 undergoes expansion while S2 shrinks.

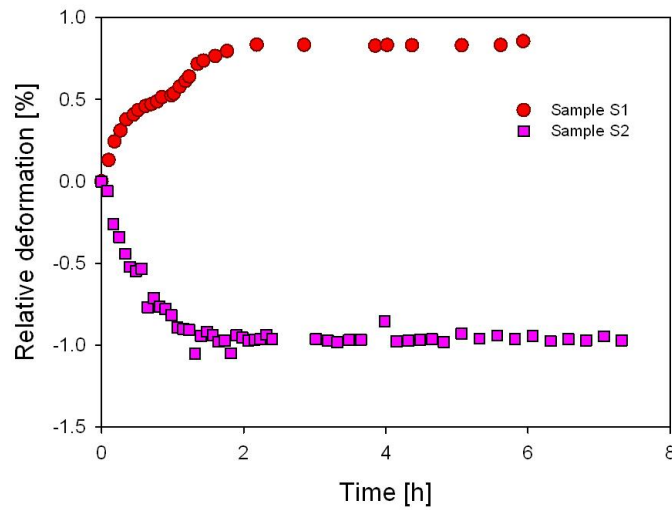


Figure 4. Relative deformation of two different mortars measured with a Fizeau fiber interferometer

The samples were also measured by using FBG-based sensors and a horizontal sample scheme (Figure 1). In that case, a fiber Bragg grating was embedded within the fresh material and a thermocouple was also placed into the sample in order to measure its temperature during the curing process. Once the mixture was prepared, it was placed into two exactly equal molds: one of them to perform the optical measurement and the other to be measured mechanically from the next day. The first sample was immediately started measuring with the FBG sensor. After approximately 24 h, when samples had acquired certain stiffening degree, both of them were demolded and measured with both techniques since that moment. By taking into account that the measured temperature changes were negligible, the absolute and relative deformations of the materials were calculated using the expressions above mentioned.

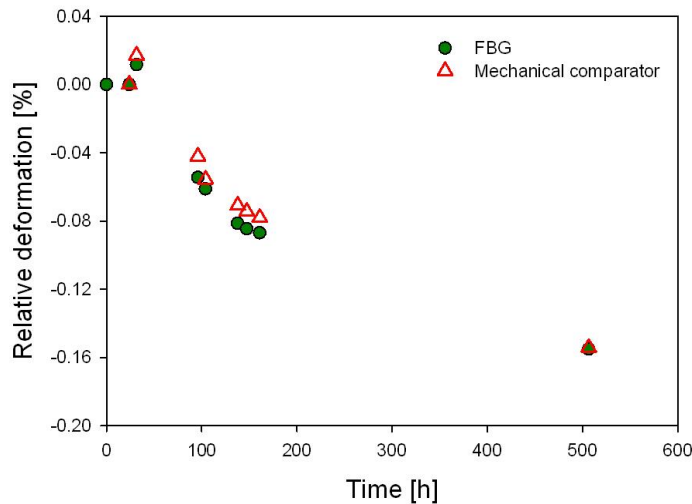


Figure 5. Relative deformation of sample S3 measured with a FBG sensor and a mechanical comparator.

In order to compare the results obtained with both techniques, Figure 5 shows the relative deformation experienced by sample S3 after 24 h. A good agreement can be seen between optical and mechanical measurements, allowing validation of the optical method based on FBG sensors for monitoring the evolution of the mortars deformation since fresh state. It can be observed that the material experienced shrinkage with an approximately exponential decreasing trend and a final contraction level of ~0.16% after 21 days.

4. CONCLUSIONS

This paper analyzes the evolution of the dimensional changes of cement based mortars since the fresh state. The investigated materials are heterogeneous mixtures (i.e. cement, fillers, sand and water); initially they are fluid or plastic and as time passes they stiffens and become a solid; the chemical reactions continue and this solid acquires strength. In this kind of materials it is very difficult to evaluate the dimensional changes during the first period of its life as the mechanical methods usually employed in hardened state cannot be applied in fresh state. They cannot evaluate the deformation process until some grade of solidification is achieved.

It was possible to perform the tracking of the complete process by using two optical measurement techniques: a FBG sensor embedded into the material and non-contact fiber Fizeau interferometer. Once the materials had acquired certain level of stiffness, it was verified a good correlation between mechanical and FBG measurements.

In the case of the Fizeau interferometer, the dimensional variations can be spectrally followed, which is a great advantage because it allows determining the precise moment when a change occurs in the material (expansion or contraction due to any physical or chemical transformation). The Bragg grating embedded in the material has the great advantage of not altering the chemical composition of the host and being immune to electromagnetic interference.

The results show the feasibility of the two optical techniques to follow the dimensional changes of cement based materials both in fresh and hardened state, even before the beginning of the hardening process. Besides, they have high sensitivity, resolution and repeatability.

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