Characterization of Dimensional Changes of Cement Pastes and Mortars in Fresh State Applying an Interferometric Technique

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Abstract—**The effect produced by the incorporation of additives in Portland cement based materials over dimensional changes occurring during the setting process was evaluated employing a fiber optic Fizeau interferometric sensor. The sensor system employed a broadband light source (SLED) centered at 1550 nm, whose spectral emission was modulated by the interferometer formed between the material surface and the end of the optical fiber used to illuminate the sample. An optical spectrum analyzer was used to monitor the variation of the modulated spectrum, while the mentioned process took place. The expansion or contraction experienced by materials with different compositions was observed and quantified. Results obtained point out the accuracy and the potential of the technique.**

Keywords— Optical Fiber Sensor, Cementitious Materials, Dimensional Change In Fresh State, Interferometric Technique

I. INTRODUCTION

Concrete is a composite material where fine and coarse aggregates are included in a porous matrix made of cement and water that combine into a cement paste binder. The aggregates act as inert filler materials restraining the dimensional variations while the cementitious matrix covers the aggregates, fills the voids and is the main responsible for the strength and durability of the material.

During the last years, great attention has been paid to the development of new mixtures based on cementicious materials and other additives, in order to create a new generation of materials with enhanced properties. The main concept in the design is to minimize the number of defects, avoiding the early cracking of pastes (cement + water), mortars (paste + fine aggregates) and concretes (mortar + coarse aggregates), and to achieve a greater percentage of the ultimate load capacity of its components. The contraction or expansion occurring within the material (which may be significant during the first 24 hours) can lead to the development of these internal cracks, so the detection and quantification of those processes is of great interest [1].

When the water and cement become in contact the hydration of the cement starts, then a series of physical changes appear as an expression of the ongoing chemical process.

Thus, the cement paste changes from being a "viscous liquid" to be a "tough solid". First, a reduction of free water occurs in the system due to the early hydration reactions, the adsorption of water on the surface of the poorly crystalline hydration products and the evaporation, and, as a result, the paste loses plasticity and stiffens. The reactions continue and, after a few hours, they produce what is called setting and then hardening. The setting represents the solidification of the paste and it takes place progressively. At this moment the strength of the paste is very low, the reactions continue and the paste gains strength over the time (months).

The hydration of cement compounds is an exothermic process, and the heat of developed hydration can produce some expansions affecting the dimensional stability of the system. Several factors, such as the composition and fineness of the cement or the mixture proportions modify it. However, Portland cement based materials usually show a shrinkage response both in fresh and hardened state; several reasons such as settlement, chemical reactions (the hydration products occupy less volume than the non hydrated Portland cement and water), water evaporation and the movement of water in pores are some of the main reasons. Shrinkage that occurs in the material during the early hours is generally ignored because its magnitude is assumed to be much smaller than the long-term shrinkage, but this assumption can be erroneous. Concrete shrinkage is a complex phenomenon that is affected by many factors including drying and internal self-desiccation deformations, curing procedure and environmental conditions.

Then, the dimensional stability of these materials is a major concern in order to prevent the development of cracks. In structures where it is necessary to avoid the shrinkage, some chemical admixtures can be included. The study of the mentioned processes in the fresh state is a requirement for some commercial grouts especially designed for sealing parts and structural elements [2].

As mentioned above, the volume of concrete begins to change shortly after the mixture has been made. This is due to some competition between chemical shrinkage and thermal expansion of the material, which is generally present from early stages.

Very few methods allow evaluating dimensional changes from the plastic state (before the setting of the material occurs). Piezoelectric transducers embedded in the material are one of the devices that have been used to perform that task [3].

On the other hand, optical measurement techniques offer an excellent alternative [4]. Optical fiber sensors have been employed in aerospace, structural control, medical and chemical applications among others, for more than thirty years [5,6]. The main reasons are their predominant advantages such as small size, low cost and capability of avoiding electromagnetic influence. In the same way, the concrete setting process has been monitored by measuring the optical fiber loss changes [7,8]. Most applications use fiber Bragg gratings (FBG) sensors, especially for sensing temperature and strain. In addition, FBG sensors have been utilized to assist in describing the process of drying and evaporation of cement pastes [9-14].

Different fiber optic interferometric sensor systems, such as the Mach-Zender interferometer, the extrinsic Fabry-Perot Interferometer (EFPI) or the SOFO system ("Surveillance d'Ouvrage par Fiber Optique" or Monitoring of Structures by Fiber Optic Sensors), have been employed for civil engineering applications and for monitoring changes induced by different processes in materials [15,16]. On the other hand, we have employed the Fizeau sensor for monitoring the photopolymerization process of dental resins [17,18].

In a previous work [19], we analyzed the suitability of two fiber optic sensing techniques (Bragg grating sensor and Fizeau interferometer) to study the dimensional stability of cementitious materials. These results were compared with those obtained with a commercial mechanical device after the samples solidified. Measurements performed with the mentioned techniques showed a good agreement between them.

In the present work, the Fizeau interferometric scheme (basically a non-contact technique) was applied to characterize different cement-based pastes and mortars by measuring their dimensional evolution from the very early stages and until the materials had reached their hardening. This technique was able to detect dimensional changes immediately after the mixture preparation was finished, which was the actual moment of zero deformation. The effect produced by the incorporation of some additives was evaluated, as well as the impact generated by changing the environment temperature over one of the samples.

II. METHODOLOGY

In this section, a brief description of the methodology employed to calculate the deformation of the material is explained as well as an estimation of the corresponding error.

The Fabry-Perot interferometer is one of the most used fiber optic sensors due to its sub-micrometric accuracy in applications such as the measurement of temperature, pressure, displacement or vibration. When the finesse of the Fabry-Perot cavity made with fiber optic components is low, or the length of the cavity formed by the surfaces of the reflective elements is much greater than the core diameter of the fiber used, the device may be called extrinsic Fizeau interferometer. Such interferometer operates by reflection and the output signal is generated from the interference between rays reflected on the surface of the fiber optic connector and on the reflecting surface which closes the cavity (that is, the surface which is being measured). The theoretical description of the operating principle of the device was described in previous works [20,21].

In Fig. 1 we show a basic scheme of the Fizeau interferometer. By considering "d" as the separation of the reflective elements and $n=1$ the refractive index of the medium inside the cavity (air), the frequency separation $\Delta \nu$ (constant along the spectrum) between two consecutive intensity maxima corresponding to two consecutive maxima of interference, can be expressed as:

$$\Delta \nu = \frac{c}{2d} \qquad (1)$$

Considering that $|\Delta \nu| = (c/\lambda^2)\Delta\lambda$, we can express the spectral separation of the peaks as:

$$\Delta\lambda = \frac{\lambda^2}{2\,d} \qquad (2)$$

and

$$d = \frac{\lambda^2}{2\,(\Delta\lambda)} \qquad (3)$$

Due to this, the accuracy of this technique or the minimum value of d that can be measured may reach values in the order of one micron, depending on the resolution of the detection system (Optical Spectrum Analyzer - OSA) and the measurement range. Denoting with d_i and d_f the initial and final values of the distance between surfaces, respectively, we can calculate the absolute deformation of the material (Δd) as:

$$\Delta d = d_f - d_i \qquad (4)$$

and the percent relative deformation is given by:

$$\frac{\Delta d}{L_0}[\%] = \left(\frac{d_f - d_i}{L_0}\right) \times 100 \qquad (5)$$

where L_0 is the initial length of the sample measured.

Error estimation: we can estimate the error in determining the value of d as:

$$\delta d = \frac{\lambda}{\Delta\lambda}\,\delta\lambda + \frac{\lambda^2}{2(\Delta\lambda)^2}\,\delta\Delta\lambda \qquad (6)$$

Considering L_0 = 10 cm, λ = 1550 nm, $\delta\lambda$ = 0.02 nm (OSA resolution), a spectral window of 20 nm and $\delta\Delta\lambda = 2\delta\lambda$, the accuracy of the technique results better than 1×10^{-3} [%].
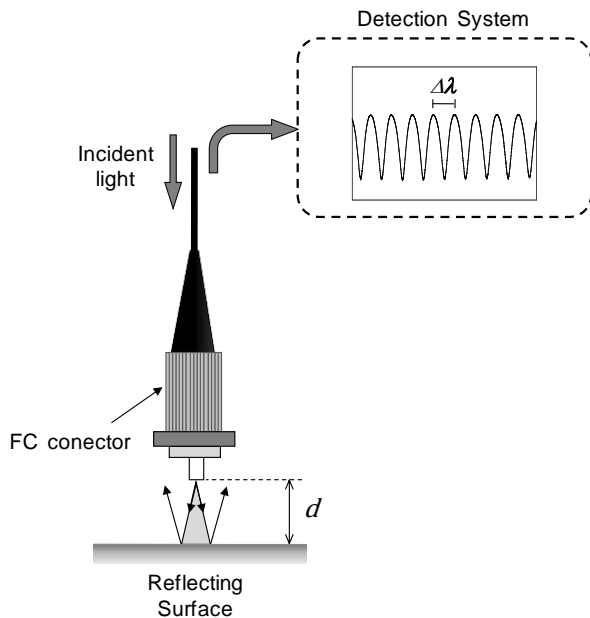
Fig. 1. Basic scheme of the Fizeau interferometer.

III. EXPERIMENTAL IMPLEMENTATION

A. Experimental Set-up

In order to perform the measurement, the set-up shown in Fig. 2 was implemented. The illumination system employed a broadband superluminescent laser diode (SLED) whose emission was centered at 1550 nm. The resulting intensity pattern, coming from the interferometric cavity, was detected and registered by an OSA (Yokogawa, model AQ6370B). The evolution of the spectral structure registered by the mentioned instrument determined whether the cement-based material had expanded or contracted.

Once each mixture was prepared, it was immediately placed in a cylindrical mold of rigid walls, 10 cm high and 5 cm in diameter, and the deformation of the material was observed in the vertical direction. In order to improve the intensity reflected by the sample surface and therefore increase the fringes' contrast, a small aluminum foil was placed on the material and was aligned with the fiber connector. Subsequently, in order to compare the behavior of one of the materials at different temperatures, the sensor scheme was placed inside an adiabatic box with a thermal source.

B. Materials

During the development of this work, two Series of cement-based mixtures of different compositions were measured using the Fizeau interferometer. The first Series compared the behavior of mortars that, according to their composition, could have expansion or contraction during the first hours after the mix was prepared. The effect of the temperature on mixture deformation was also studied.

Series 2 studied high-strength mortars, suitable for structure repair, which incorporated different types and amount of mineral additions. The inclusion of very fine particles, like those of mineral additions, can modify the dimensional stability of the mixtures. The mineral additions, that can react or not in order to produce more hydrated
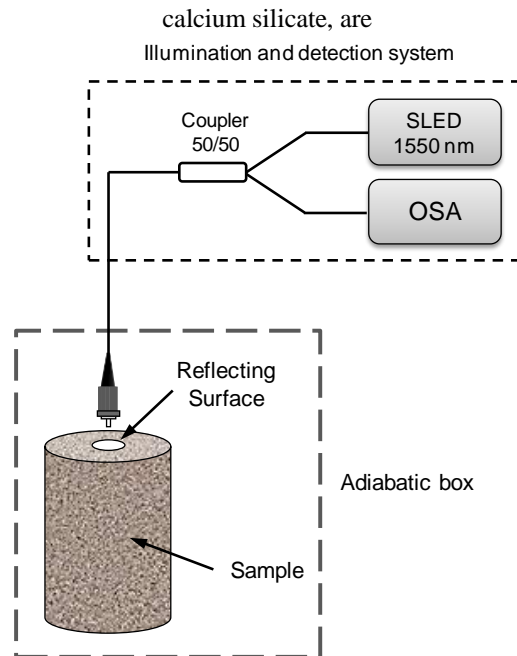
calcium silicate, are

Fig. 2 Experimental implementation of the Fizeau Interferometer including the monitoring system, the sensor itself and the sample.

usually employed in mortars that require high strength, good cohesion and high fluidity. In addition, as this type of mortars has a high volume of paste, a conventional mortar prepared with the same cement was included; in this way the effect of the volume and water/cement ratio of the paste can be shown. The main characteristics of each mixture are given in Table I.

TABLE I. MATERIALS AND MIX PROPORTIONS.

Mortar Materials (kg/m^3)	Series 1			Series 2				
	A	B	C	D	E	F	G	H
Water	250	367	291	299	299	302	299	240
Sand	1290	--	580	800	660	665	660	1480
Grout	--	1635	1245	--	--	--	--	--
Portland Cement	645	--	--	1065	1065	1080	960	480
Cement TYPE	CPF40	--	--	CPN50	CPN50	CPN50	CPN50	CPN50
Silica Fume	--	--	--	--	107	--	107	--
Calcareous filler	--	--	--	--	--	108	107	--
HRWRA	--	--	--	8.6	10.6	11.0	10.6	--
Water/Cement	0.39	--	--	0.28	0.28	0.28	0.31	0.50
Sand/Cement	2.00	--	--	0.75	0.62	0.62	0.69	3.08
Water/Binder	0.39	0.22	0.23	0.28	0.25	0.25	0.25	0.50
Sand/Binder	2.00	--	0.47	0.75	0.56	0.56	0.56	3.08

Series 1 includes 3 mixtures named A, B and C. Mixture A was a conventional Portland cement mortar, made with a fillerized Portland cement (CPF40®), sand and water. The mentioned commercial product is based on a combination of ordinary Portland cement and calcareous filler, with high mechanical strength, suitable for mortars and concretes. Mixtures B and C were made with a premixed commercial mortar (Grouter®) and water. Mortar C was prepared using the same grout as mortar B with the incorporation of natural siliceous sand. Mortar B was tested at 22 and 38 ºC. The commercial product is a high strength grout suitable for anchorages and fillings, whose formulation is based on the combination of cement, special admixtures and controlled

grain sized aggregates. One of the main characteristics of this product is that it must show some expansion during the fresh state. As a greater volume of inert material is present in mortar C, a reduction in the mixture expansion is expected.

Series 2 included five mortars (D to H) made with an ordinary Portland cement (CPN50®), sand, water, high range water reducer admixture (HRWRA) and, in some cases, mineral additions. Two mineral additions with very different characteristics were selected, silica fume and calcareous filler. The silica fume is a very fine and active powder that contributes to the strength of the mortar; the calcareous filler is an inert addition, of similar fineness to the cement, which contributes to cement dispersion. High-strength mortars have very low water/cement ratio. Mixture D did not incorporate any addition; mortars E and F had the same water/cement ratio as mortar D, but they incorporated silica fume (E) or calcareous filler (F); in mixture G a combination of both mineral additions was used. Finally, mixture H was a typical conventional-strength mortar, with water/cement ratio equal to 0.50 and without any mineral addition.

IV. RESULTS AND DISCUSSION

As it was previously mentioned, dimensional changes of the investigated materials in their early stages cannot be evaluated employing traditional mechanical techniques. Generally, to make use of them it is necessary that the material has acquired certain degree of rigidity. However, it is probable that in the first hours after preparation, the mixture experiences deformations that should be considered. Non-contact optical methods, like the Fizeau interferometer, are an interesting option in these cases, because they are able to evaluate the entire hardening process.

Fig. 3 shows two typical interferograms obtained with the OSA while the material was hardening. This illustrates the phase shift caused by the path difference of the interfering beams. The initial trace (dashed line) has $\Delta\lambda_i \approx 1.14$ nm and was acquired immediately after the mixture was prepared and put into the mold. The other trace (solid line) has $\Delta\lambda_f \approx 1.82$ nm and was registered 130 min after the beginning of the measurement process. In this case, the material underwent a process of expansion.

For each mixture analyzed, the interference signals were recorded by the OSA for several hours. Then, they were processed by applying the equations above mentioned and it was possible to determine the deformation of the materials under test. The traces shown in Figs. 4 and 5 correspond to the mixtures of Series 1 and 2 respectively. All of them were measured at 22 ºC, and mixture B was also measured at 38 ºC. As it can be seen, the main dimensional change occurred in the first two hours for the materials used in this work.

Fig. 4 shows that Mixture A experienced a contraction process, while samples B and C showed some expansion until a constant level was achieved. As expected in mortar C, due to the presence of a greater volume of inert material, the expansion was lower. In this sense, the used technique is suitable for measuring these processes.

As it is well known, the environmental conditions can affect the chemical reactions of the cement; the differences in the rate of hydration can modify the expansion rate and level. To take into account the effect of ambient temperature, the expansion during the setting process of mixture B was also evaluated at 38 ºC (Fig. 4, empty triangle trace). As expected, the expansion rate and the relative deformation were quite higher at the highest temperature.

The changes in the behavior of mortars when some mineral additions were included can be observed in Fig. 5.

In this case all mixtures present shrinkage during the fresh state, varying the magnitude between near 0.5 and 0.9 % according to the cementitious material (cement + mineral addition) and proportions used. Comparing the mortars without additions, it can be seen that the conventional mortar H had lower contraction than mortar D, due to the higher content of sand (inert material).

It can be seen that during the first six hours, when the material had not hardened yet, the silica fume, as it is a very reactive and fine material, increased the retention of water and reduced the contraction of the mortar (compare E and D) while the calcareous filler increased the contraction (compare F and D). When a higher amount of both additions was used in a combined way (mortar G) the volume changes were closer to the reference mixture (D), but they were smaller.
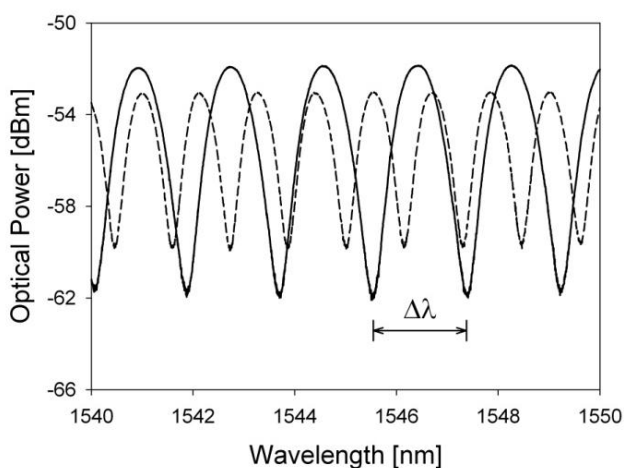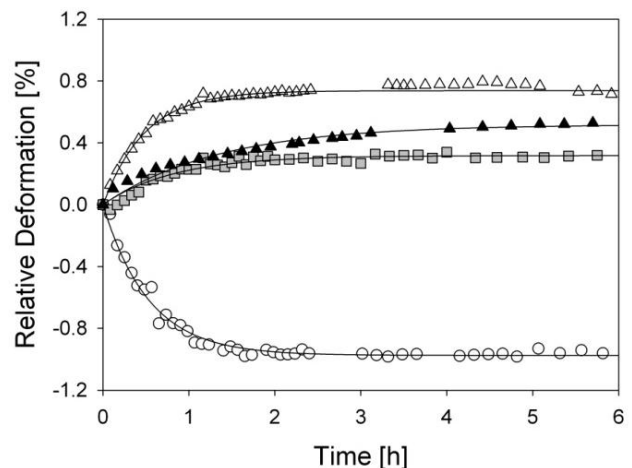

Fig. 3 Typical interferometric signals obtained with sample B immediately after placing the mixture in the mold (dashed line) and two hours later (solid line).

Fig. 4. Dimensional evolution of cement-based materials shown in Table 1. A: empty circle, B: black triangle, C: gray square (all at 22ºC), and B: empty triangle (at 38ºC).
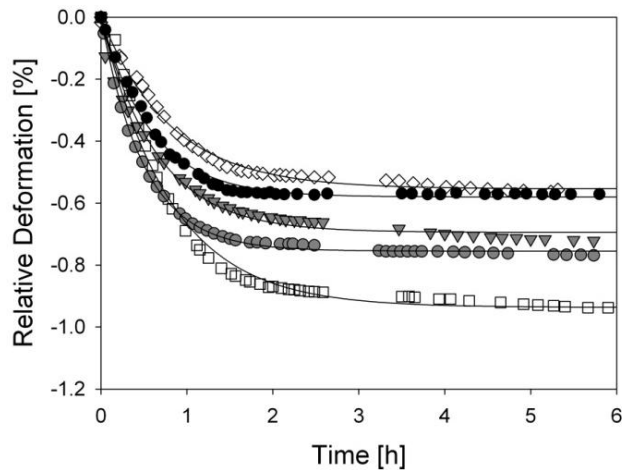
Fig. 5. Dimensional evolution of cement-based materials shown in Table 1. D: gray circle, E: empty diamond, F: empty square, G: gray triangle and H: black circle (all measured at 22ºC).

The temporal evolution of the vertical deformation of each material was fitted with a simple exponential curve of the type:

$$y(t) = A(1 - \exp(-kt)) \qquad (7)$$

where A is the relative deformation [%] and k is the evolution rate [time^{-1}]. Table II shows the corresponding coefficients for the different materials.

TABLE II.   EVOLUTION PARAMETERS OBTAINED FROM THE CURVE FITTINGS.

Sample (at 22 ºC)	A [%]	k [hs^{-1}]
A	-0.97	1.88
B	0.52	0.74
B (at 38 ºC)	0.74	2.1
C	0.32	1.21
D	-0.75	2.00
E	-0.55	1.24
F	-0.94	1.24
G	-0.69	1.55
H	-0.58	1.71

The relative deformation parameter in Table II (the values are positive for expansion processes) represents the limit average value obtained after eight hours, while k gives an indication about the velocity of the process. As it can be seen, for the materials used in this work, the main dimensional change appeared in the first two hours. One aspect to highlight is the low dispersion of the measurement results.

V. CONCLUSION

The dimensional evolution of cement pastes and mortars during the setting process was evaluated from the fresh state employing the Fizeau interferometer technique. It was possible to monitor the complete solidification process of these materials, even at the early stages, allowing determining the true initial position of the absolute zero deformation point. Mixtures with different compositions were analyzed and the results were in good agreement with the relative changes expected for the formulations employed. Tests performed on mixtures of identical composition under similar conditions showed a good repeatability, variations in the measured deformation were usually lower than 0.05 %.

By applying this optical technique, the expansion or contraction experienced by the sample is spectrally codified and it can be determined the precise moment when a change occurs in the material due to any physical or chemical transformation. Besides, not being involved in the physicochemical process that takes place, this technique has high sensitivity, resolution and repeatability. The methodology employed also allows the use of small quantities of materials without loss of resolution.

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