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In situ measurement of elastic properties of cement by an ultrasonic resonant sensor

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Abstract

The ultrasonic sensor is composed of a resonator excited by a piezoelectric element. It is in the shape of a solid metallic cylinder with a fine cylindrical tip. The tip is dived into the cement to be tested, where it generates propagating and standing waves. The frequency of resonance of the system gives us information on the elastic properties of the paste. The sensor is mostly sensitive to the shear modulus G' of the medium. With the described sensor, the range $10^6 < G' < 10^7$ Pa is tested. Other ranges can be selected by changing the size of the cylinder or the tip.

Keywords: Ultrasonic measurement; Cement; Rheology; Mechanical properties; Hydration

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1. Introduction

During the hydration of cements and concrete, strong variations of rheological properties are observed [1–3]. The setting and hardening result from a complex chemical process involving numerous chemical components. Elastic properties of cements vary drastically and continuously as the paste passes from a quasi-liquid state to a solid. There are several empirical methods to check the loss of workability and the hardening of the medium [4]. One method tests the breaking point of the sample by measuring the slump of a standardised volume (e.g., the "Abrams cone"). A second method examines the penetration of a solid in the paste (e.g., the so-called "Vicat's needle"). These tests sometimes are not carried out directly on the product, but on samples poured simultaneously and supposed to be in the same conditions as in the bulk.

Rheometers are used to follow the modifications of mechanical properties. During the early stages of hardening, rheometers follow the viscosity of a fluid sample between two cones or plates [5–10]. A rheometer using an oscillating probe has also been proposed [11] to minimise the shear of the samples.

For mature cements, solid samples are tested, with different sample geometries. Few apparatus permit mechanical measurements from the beginning up to the complete solidification of a same sample. The results of different methods can be linked, in order to follow the full range of setting–hardening, from the early age to the complete strength [12].

Acoustic waves are used successfully for the determination of mechanical properties of cement pastes and mortars, from the mix to the cure [3,13,14]. With compression waves, the methods have little efficiency in the early stages of the hardening because of the relative insensitivity of the longitudinal velocity to the formation of the gel structure.

Methods using acoustic transverse wave are also proposed [15,16] as method including these two kinds of waves [17–20]. The "vibroscope," for instance, measures the velocity of both types of waves at low frequency (100–5000 Hz).

We proposed in Ref. [21] a novel acoustic method and a compact-sized and easy-to-handle probe to monitor the hardening. But the indicated values were not related to the intrinsic properties of the material. This paper will show that our apparatus follows the complex shear modulus and can be calibrated.

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As a viscoelastic medium, the cement paste can be described by the Lammé coefficients λ and μ, which for sinusoidal excitation takes a complex form, $\mu = G' + iG''$.

In a freshly mixed cement, the modulus λ is always strong (λ>5 GPa) due to the low compressibility of the medium, combining water and minerals, and evolves slowly. On the contrary, the shear modulus μ begins with very low values, as the fresh paste is mostly viscous and increases by several orders of magnitude as the paste cures. This effect can be observed by sound velocity measurements:

$$V_L = \sqrt{\frac{\lambda + 2\mu}{\rho}} \quad \text{and} \quad V_T = \sqrt{\frac{\mu}{\rho}}$$

where the transverse velocity V_T is much lower than the longitudinal velocity V_L.

There are many studies [22–27] that relate the elastic properties of the cement to its compressive strength, and all have shown a definite relation between the elastic modulus E and the strength, affected by the nature and composition of the aggregates. We do not enter into this discussion but indicate only that the workability (i.e., the ability of the cement to flow) must be in the same manner strongly related to the shear modulus of the paste.

As we will show below, our sensor is sensitive to the shear modulus of the cements or concrete; we expected that it would serve to test the workability of the paste.

In opposition to other systems that measure the velocity on isolated samples, we present a sensor, which is placed directly in the medium to be tested, without any preparation. This in situ nondestructive test gives obvious information on the real state of the medium, not biased by any differential humidity or temperature conditions.

2. Experimental set-up

The sensor presented in Fig. 1 is composed of a metallic cylinder (tip A) dipped in the medium to be tested. It can be put in resonance by a second larger cylinder (B) and a piezoelectric element. The device works very simply: just after the mix, the cement is semiliquid and the tip A vibrates easily in the medium. When the cement hardens, the sensor is blocked and only the emerged part of the sensor can vibrate. The resonance frequency of the system then passes

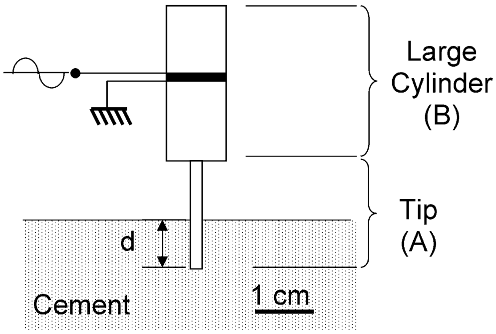

Fig. 1. Acoustic sensor. The tip (A) is bound to the body (B) of the sensor. The tip is immersed in the cement in order to measure rheological properties.

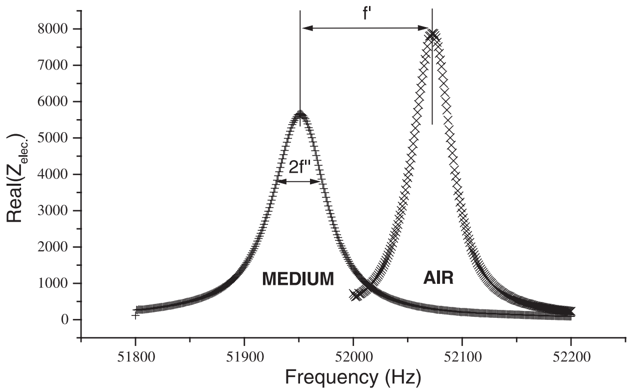

Fig. 2. The two parameters measured by applying a frequency ramp to piezoelectric element: the frequency shift of the resonance f' and the half width of the resonance f''.

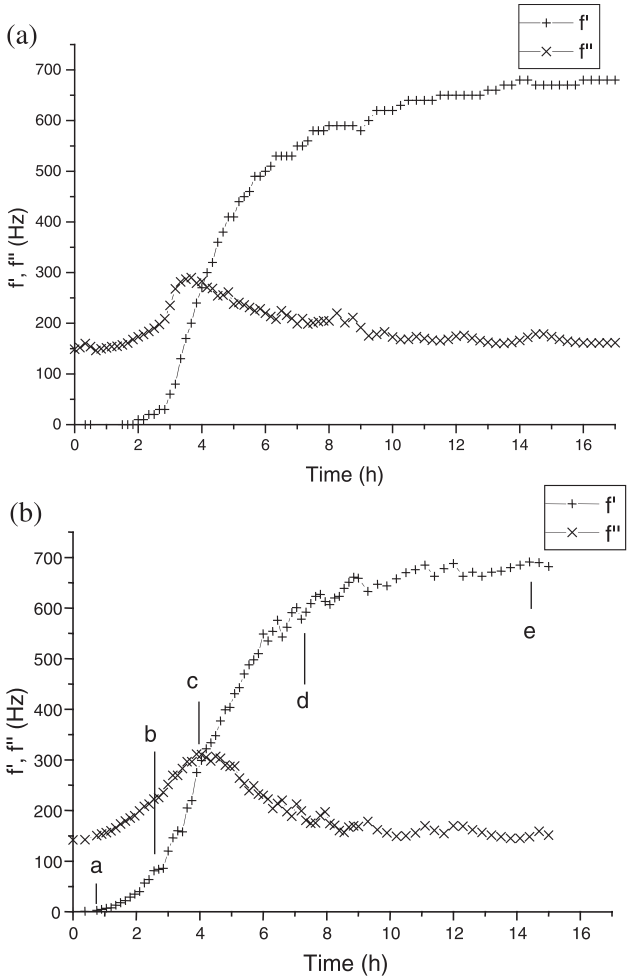

Fig. 3. (a) Experimental curves f' and f'' versus time of hydration. f' is the shift of resonance frequency. f'' is the half width of the resonance. (b) Theoretical curves f' and f'' versus time of hydration.

from one value to another, as the cement cures. The sizes of the two cylinders (A and B) are selected to adapt the impedance of the cement to the piezoelectric element and to select the range of hardness to which the system will be sensitive.

The main difference between our sensor and the previously cited rheometers is that the vibration amplitude is very small (< 100 nm), resulting in a negligible shear rate of the sample during experiments.

A pure cement paste (Lafarge ‘Ciment Gris,’ water/cement ratio = 0.30) has been prepared and poured out in a plastic vessel (≅200 cm^3) to form the sample. The sensor is then dipped at a deepness of 10 mm. By applying a frequency ramp to piezoelectric element, the frequency shift of the resonance f' and the half width of the resonance f'' are measured and recorded, each for 5 min, during the cure of the paste (Fig. 2). The results are presented in Fig. 3.

3. Calibration

To test and calibrate the above sensor, we operated in parallel with the sensor presented in Fig. 1, an acoustic rheometer to measure the elastic properties of the medium at the same frequency. The acoustic rheometer measures the longitudinal and transverse propagation velocity in a small cylinder of cement, in the same frequency range as the sensor.

In Table 1, the elastic properties of the studied cement are reported over the first 10 h of the solidification. We calculated the corresponding wavelength and penetration of the waves. As mentioned above, when the paste is fresh (< 1 h), the wavelength is much shorter than the cylinder’s radius and the wave expands cylindrically around the cylinder. Later, the wavelength is greater than the tip, and the cement is then oscillating in a standing wave mode. Such complex vibrations cannot be represented by simple analytical expressions and a numerical analysis is needed. A finite element calculation (axisymmetric, nine-point element) gives, at the frequency of the experiment (around 45 kHz), the deformation and the strain everywhere in the vessel. The mechanical impedance of the immersed part of Cylinder A is then calculated:

$$Z_{\text{mech}} = \frac{\text{Strain in the section of the tip A at the surface of the sample}}{\text{Velocity of deformation in this section}}.$$

Table 1
Lammé coefficient λ, Lammé coefficient $\mu = G' + iG''$, wavelength λ_s, and penetration depth δ_s for the transverse waves, versus the time since the beginning of hydration

Time (h)	λ (GPa)	G'(MPa)	G'' (MPa)	λ_s (mm)	δ_s (mm)
1	5.6	0.32	0.04	0.3	0.8
2	5.7	3.2	0.3	0.9	3.2
3	5.9	14	1	1.9	8
4	6.1	37	2.4	3.1	16
5	6.3	72	4.1	4.3	24
6	6.6	140	7	6	40
8	7.4	380	16	10	75
10	8.4	700	26	13.5	115
15	11.1	1600	50	20	210

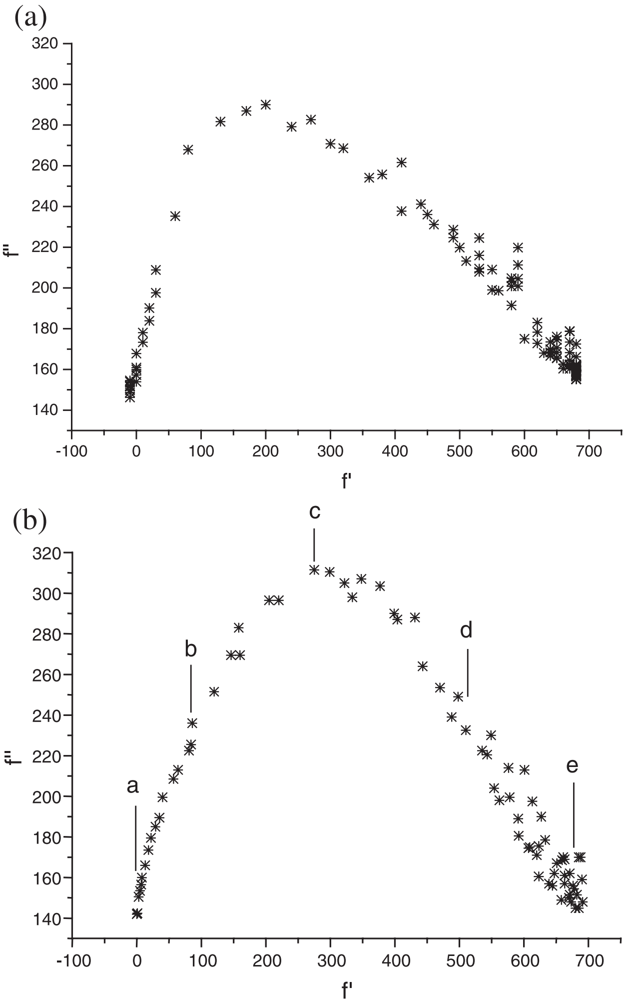

Fig. 4. (a) f'' versus f'—experimental curve. (b) f'' versus f'—theoretical curve. The theoretical curve is continuous but seems erratic in some parts for the reasons described in Fig. 2b.

This impedance (a complex number) describes precisely the interaction between the cement paste and the sensor. The imaginary part of the impedance is related to the standing waves in the medium, and the real part to the energy dissipated in the medium. The external part of the sensor (emerged part of A and the whole of B) is a resonant system by itself, whose frequency is determined by its size and by the above impedance Z_{mech}. By means of a simple 1D scheme [28], the electric impedance Z_{elec} across the piezoelectric element can be deduced at each frequency f. From the calculated Z_{elec} (f) curve, the complex resonance frequency $f = f' + if''$ is calculated and is presented in Fig. 3 [the complex resonance frequency is the pole of the Z_{elec} (f) function].

4. Discussion

Taking into account the diverse approximations made in the calculations, notably the free boundary assumption for the cement in the vessel, the model appears in be in accordance with the experiment. The frequency shift and the width of the resonance are correctly reproduced.

We now discuss what information can be deduced from the measurements (i.e., which elastic constants are measured effectively):

1. Firstly, we noted that the elastic constants (λ, G', and G'') vary monotonically during the cure, whereas the f'' increases and decreases. The output (f' and f'') is then nonlinear versus the input constants. The nonlinearity is explained simply by the way the initial information (the complex modulus G) is transferred in the measured values (the complex impedance Z_{elec}), via the mechanical impedance, by strongly nonlinear relations.

2. There is no discontinuity in the experimental data. We do not observe a viscosity decrease in the initial stages of the experiments. This confirms that the probe does not cause structural breakdown in the paste.

3. The letters (a), (b), (c), (d), and (e) are corresponding points in the input and output graphs of Fig. 4. In the first period (a–b), the shear modulus is small and the paste behaves in a fluid manner. The presence of the free surface of the cement gives a low pressure in the whole sample and the forces applied on the sensor are mostly determined by the shear constant G. At the end of the experiment (d–e), the cement is hard and the force depends on both λ an G constants.

4. Large variations of λ or G'' (doubling the actual values near the point c) have little effect on f' or f''. Then the calculated frequencies f' and f'' are mostly related to the real shear modulus G' of the medium. In fact, the resonance frequency appears equally sensitive to G' and G'' variations, but the effect from G'' is negligible as G'' is nearly 10 times lower than G'.

5. The small irregularities shown on the frequency curves are due both to interferences in the vessel and to numeric errors related to the variable mesh grid used in the finite element calculation.

5. Conclusion

We described an ultrasonic sensor placed directly in the cement to be tested. The sensor creates a near-field oscillation in the paste. As the mechanical properties of the cement pass from a fluid to a solid, the system goes continuously from a free vibration mode (Point a) to a blocked mode (Point e). The intermediary point (Point c) is reached for a shear modulus G' near 30 MPa. The main interest of the device is that it tests the real state of the cement, in bulk form.

In spite of its nonlinearity, our sensor tests the G' shear modulus in a large interval of G' values, and is relatively insensitive to the other constants (λ and G''). For instance, G' can be measured in the range 0–40 MPa by the induced frequency shift with a nearly linear relation between 0 and 300 Hz. For higher G' values, the system saturates and less precise properties can be deduced.

In reason of the nonlinearity, our sensor cannot be considered really as a measure instrument, but more effectively as a hardness indicator (efficient in the 10–50 MPa interval). By changing the size of the cylinders, other values can be selected, allowing the sensor to be adapted to any point of the cement hardening curve.

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