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ScienceDirect
Physics Procedia 3 (2010) 839–845
Physics Procedia
www.elsevier.com/locate/procedia

International Congress on Ultrasonics, Universidad de Santiago de Chile, January 2009

Characterization of mortar samples using ultrasonic scattering attenuation

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Abstract

In this paper, volume fraction and size of aggregates in highly dispersive materials have been estimated using ultrasonic techniques. Estimations were made by obtaining the best fit between experimental scattering attenuation and theoretical predictions obtained from an N-phase extension of the dynamic generalized self-consistent method (DGSCM) proposed by Yang [J. Appl. Mech. 70 (2003) 575]. Furthermore, a time-frequency procedure has been designed to estimate frequency-dependent scattering attenuation from experimental measures. The research was conducted with several cement mortar specimens made by taking into account different aggregate-to-cement ratio and water-to-cement ratio. Estimations of both size and volume fraction, were made and good agreements was found between experimental attenuation curves and theoretical predictions.

Keywords: Scattering attenuation, Ultrasonic characterization, cementitious materials

1. Introduction

The nondestructive testing (NDT) by ultrasounds of cementitious materials such as mortar and concrete is a fundamental research area that allows determining the structural quality and the degradation state of building materials. Moreover, ultrasonic NDT techniques relate relationships among physical and mechanical properties with the measurements of the ultrasonic pulse velocity (UPV), acoustic wave attenuation and backscattering noise characterization [1]. Nevertheless, cementitious materials exhibit a dispersive behavior due to their complex heterogeneity. Therefore, frequency dependence on velocity and attenuation has to be considered in the characterization of cementitious materials. Several research groups have successfully achieved the characterization of cementitious materials from frequency-dependent parameters. Punurai et al. [2] performed an estimation of both size and volume fraction of entrained and entrapped air voids using ultrasonic attenuation profiles in cement pastes. Research has been conducted on prediction of both velocity and attenuation of fresh mortar as well as on the

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doi:10.1016/j.phpro.2010.01.108

assessment of thermally damaged concrete [3, 4]. However, studies related to the characterization of mortar and concrete using frequency-dependent parameters are still in progress, especially related to the assessment of the content and size of aggregates.

The aim of this paper is to use scattering attenuation profiles to characterize the microstructure of cement mortar specimens, in order to estimate the average size and volume fraction of aggregates. To estimate these parameters, an optimization process is applied to determine the best fit between experimental measurements and theoretical predictions obtained from an N-phase extension of the dynamic generalized self-consistent model (DGSCM) proposed by Yang [5]. The paper is organized as follows: In section 2, mortar considered as a three-phase material is described and the definition of the effective complex wave number is given. Also, the N-phase extension of DGSCM is briefly outlined. Section 3 is divided in two parts: first the properties of the mortar specimens used in this study are highlighted; in the second part, experiment setup is presented as well as a proposed time-frequency (T-F) technique in order to compute scattering attenuation. The procedure of estimating the size and volume fraction of aggregates is explained in section 4. Results and discussions are summarized in this section. Finally, conclusions are presented in section 5.

2. Theory

2.1. Material model

In this study, mortar is considered a composite material of three phases: a solid phase (elastic matrix), another phase as elastic inclusions (fine aggregates) and a fluid phase which takes into account entrapped air voids (cavities) as shown in Fig. 1a. Mortar behaves as a dispersive material in which aggregates size is assumed to be much greater than the characteristic capillary pore size, but smaller or comparable to the ultrasonic wavelength. To describe the mean field propagating through the material and thus estimating the effective complex wave number $\langle k(\omega)\rangle$, the scheme of equivalent inclusion into the effective medium is used as shown in Fig 1b. The effective complex wave number is defined as:

$$\langle k(\omega)\rangle = \frac{\omega}{\langle V_L(\omega)\rangle} + i\langle \alpha_{sc}(\omega)\rangle \quad (1)$$

where $\langle V_L(\omega)\rangle$ and $\langle \alpha_{sc}(\omega)\rangle$ are the effective longitudinal phase velocity and the scattering attenuation, respectively. Angular brackets denote effective values. Furthermore, $\langle k(\omega)\rangle$ is related to the effective elastic constants such as Lamé modulus $\langle\lambda\rangle$ and shear modulus $\langle\mu\rangle$, as well as with the effective density $\langle\rho\rangle$.

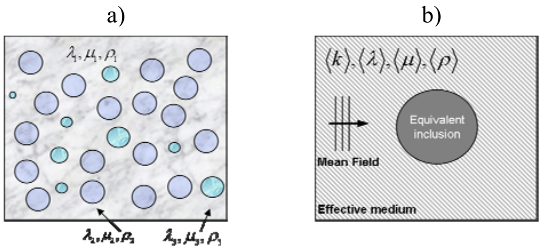

Fig.1 Cement mortar model: a) as a three-phase material, and b) as the equivalent inclusion embedded in the effective medium.

2.2. Multiple scattering model

To estimate $\langle k(\omega)\rangle$, an N-phase extension of DGSCM is proposed as described below. The DGSCM [5] assumes that every spherical inclusion of radius *a,* is surrounded by a matrix shell of outer radius *b* which in turn is embedded in an infinite medium with unknown effective properties. These radius are related to the volume fraction φ of inclusions by $\varphi=a^3/b^3$. However, DGSCM in its original form was formulated to examine bi-phase materials (see [5]), therefore in order to extend the study to three-phase materials, as defined cement mortar in section 2.1, the following expressions are used:

$$\langle k\rangle_{p+1}^2 = \langle k\rangle_p^2 + \sum_j \frac{3\varphi_j}{a_j^3} f_j(0) + \frac{9}{4\langle k\rangle_p^2} \sum_j \frac{\varphi_j^2}{a_j^6}\left(f_j^2(0) - f_j^2(\pi)\right) \tag{2}$$

where $f_j(0)$, $f_j(\pi)$ are the forward and backward scattering amplitudes of longitudinal waves in the far-field expressed as:

$$f_j(0) = \frac{1}{i\langle k\rangle_p} \sum_{n=0}^{\infty} (2n+1) A_{n,j}^*\left(\langle k\rangle_p\right) \tag{3}$$

$$f_j(\pi) = \frac{1}{i\langle k\rangle_p} \sum_{n=0}^{\infty} (-1)^n (2n+1) A_{n,j}^*\left(\langle k\rangle_p\right) \tag{4}$$

where i refers to the imaginary unit, the subscript j=1,2,3 indicates the corresponding properties of the phases: cement paste matrix, elastic inclusions and air voids, respectively. $A_{n,j}^*$ are the scattering coefficients that depend on the physical and geometrical properties of the inclusions. The solution of (2) is calculated iteratively with initial values of ⟨kp⟩ equal to those obtained by the self consistent model proposed by Sabina and Willis [6]. The iterative procedure is stopped when sufficient convergence is reached. The methodology to obtain the scattering amplitudes can be found in [5].

3. Experimental set-up

3.1. Materials

Eighteen cement mortar specimens (prismatic samples with dimensions of 40 x 40 x 160 mm^3) were made with different aggregate-to-cement ratios (a/c, by mass: 0.25/1, 0.5/1, 1/1) and water-cement ratios (w/c, by mass: 0.35, 0.40). Spherical glass microspheres were used instead of sand to make the mortar specimens for controlling volume fraction and size of aggregates. Specimens were divided in two groups (nine samples per group) according to their w/c ratios: 0.35 (group I) and 0.40 (group II). Moreover, a cement paste (reference specimen) for each group was made in order to measure matrix properties (subscript 1), obtaining for group I, V_{L1}= 4227 [m/s], V_{T1} = 2367 [m/s] and ρ_1=2316 [kg/m^3], while for group II, V_{L1}= 4087 [m/s], V_{T1} = 2289 [m/s] and ρ_1=2290 [kg/m^3]. The properties of glass microspheres (subscript 2) and entrapped air voids (subscript 3) employed were V_{L2}=5654 [m/s], V_{T2}=3387 [m/s] and ρ_2=2500 [kg/m^3], and V_{L3}=344 [m/s], V_{T3} = 0 [m/s], and ρ_3=1.24 [kg/m^3], respectively. Furthermore, the nominal diameter for both groups is shown in the second column of the Table 1 (see section 4), while the nominal volume fractions are shown in the third and eighth column for group I and II, respectively.

3.2. Ultrasonic attenuation measurements

Attenuation by scattering is measured by performing a longitudinal wave transmission (two 5MHz-broadband transducers Krautkramer H5K, 10mm diameter) experiment in immersion. To measure attenuation, the emitter transducer is excited with a chirp signal ranging from 500 to 3500 KHz generated with a function generator (TiePie, Handyscope HS3). Subsequently, the generated signal was injected into the specimen and the transmitted signal was received, pre-amplified (Panametrics, Preamp) by 54dB and averaged 128 times. Both input and output signals are recorded by an oscilloscope (Handyscope HS3). Ten measures per specimen were taken along the sample (center zone). Attenuation measures are calculated by means of the energy spectrum of the chirp signal, $E_s(\omega)$, travelling through mortar samples and the reference energy spectrum obtained from water measures $E_w(\omega)$, as follows:

$$\alpha(\omega) = -\frac{10}{d}\log_{10}\left(\frac{E_s(\omega)}{E_w(\omega)}\right) \tag{5}$$

where d is the path length of the specimen.

3.2.1. Estimation of scattering attenuation

$E_s(\omega)$ and $E_w(\omega)$ are estimated by means of calculating the frequency marginals that represent the energy densities, expressed as:

$$E_x = \int TSPWV_x(t,\omega)dt \tag{6}$$

where the subscripts x indicates the signal to be processed (s or w) and $TSPWV_x(t, \omega)$ denotes the *thresholded smoothed –pseudo Wigner-Ville distribution* defined as:

$$TSPWV_x(t,\omega) = \begin{cases} SPWV_x(t,\omega) & SPWV_x \geq \eta \\ 0 & SPWV \leq \eta \end{cases} \tag{7}$$

with

$$SPWV_x(t,\omega) = \int h(\tau)\int g(\upsilon - t)x(\upsilon + \tau/2)x^*(\upsilon - \tau/2)e^{-i\omega t}d\upsilon d\tau \tag{8}$$

where η is an energy detection threshold, h and g are the frequency and time smoothing windows, respectively. In order to detect the most representative frequency content, the threshold η was set to the half-maximum-energy of the signals. Fig. 2 summarizes the presented T-F procedure. As shown in Fig 2.g, a better estimation of attenuation is achieved by means of the proposed T-F technique (black line) instead of using Fourier transform (red line).

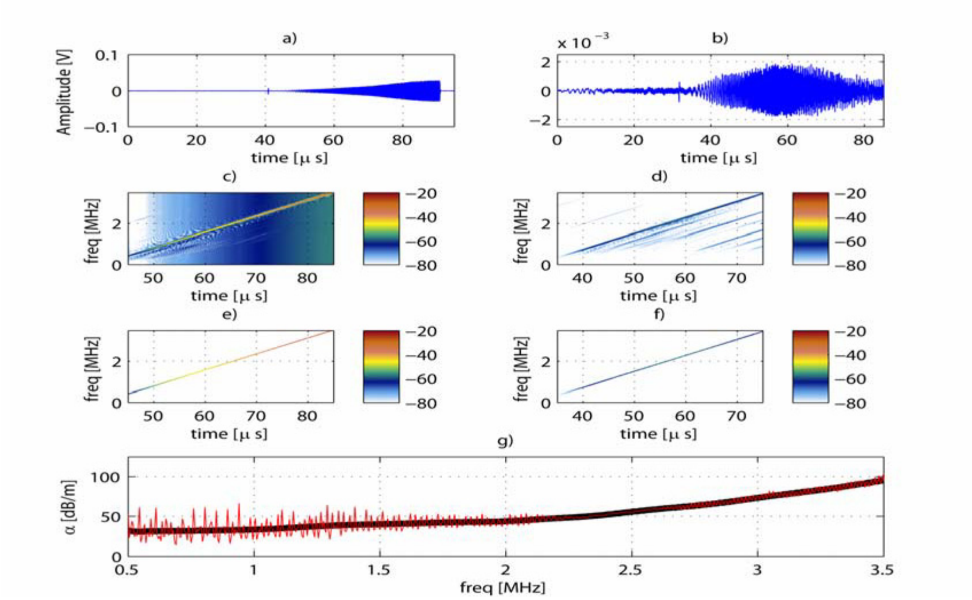

Fig.2 Example of the T-F procedure: a) chirp signal travelling in water x_w , b) chirp signal travelling into the specimen x_s, c) $SPWV_x(t, \omega)$ of x_w, d) $SPWV_x(t, \omega)$ of x_s, e) and f) $TSPWV_x(t, \omega)$ of both x_w and x_s for η sets to the half-maximum-energy, and g) Estimation of total attenuation by means of frequency marginals (black line) and Fourier transform (red line).

It has to be noted that attenuation measured by the above-mentioned procedure is the sum of absorption and scattering components. Therefore, attenuation by absorption (i.e. thermo-elastic energy loss) was measured from those reference specimens α_{ab}. In order to obtain experimental scattering attenuation by means of total attenuation α_{tot} and attenuation by absorption, the following expression was used [2]:

$$\alpha_{sc}^{ex} = \alpha_{tot} - (1 - \varphi_N)\alpha_{ab} \quad (9)$$

where φ_N is the nominal volume fraction of inclusions embedded into the matrix (see Table 1).

4. Estimates of size and volume fraction of aggregates and air voids in mortar

To estimate the average size and volume fraction of aggregates in mortar specimens, an optimization formulation was conducted. The objective function to be minimized is defined as follows:

$$F(\bar{x}) = \sum_{\omega}\left[\left\langle \alpha_{sc}(\omega,\bar{x})\right\rangle - \alpha_{sc}^{ex}(\omega)\right]^2 \quad (10)$$

where superscript *ex* denotes experimental values. The vector components of $\bar{x}$ include the average aggregate diameter d_s, the average volume fraction of inclusions φ_s, five different diameters of entrapped air voids d_v and their corresponding volume fractions φ_v. Lower and upper bounds were defined for d_s from 0.1 to 2.5 [mm], for φ_s from 5 to 50%, for φ_v from 0.1 to 3% per diameter of entrapped air voids and a size distribution of air voids from [0.25 0.5 1 1.25 1.5] to 3 [mm]. $F(\bar{x})$ was minimized using the *Levenberg-Marquardt* algorithm available in Matlab™ optimization toolbox.

4.1. Results and discussion

The results of the previously described procedure are outlined in Table 1, showing the average diameter and volume fraction estimations for microspheres and air voids of both groups I and II where the predictions of volume fraction of microspheres at low, medium and high concentrations (M025S1-M025S3, M050S1-M050S3 and M001S1-M001S3) are close to the nominal values.

Table 1. Estimates of average size and volume fraction of aggregates and air voids.

Samples	d_N [mm]	Group I w/c:0.35					Group II w/c:0.40				
		Nom.	Prediction				Nom.	Prediction			
		φ_N [%]	d_s [mm]	φ_s [%]	$\bar{d}_v$ [mm]	$\Sigma\varphi_v$ [%]	φ_N [%]	d_s [mm]	φ_s [%]	$\bar{d}_v$ [mm]	$\Sigma\varphi_v$ [%]
M025S1	018-0.30	13.4	0.47	13.74	1.73	1.65	12.6	0.52	13.28	2.40	1.45
M025S2	0.42-0.60	13.4	0.69	14.97	2.16	1.90	12.6	0.71	15.93	1.76	1.25
M025S3	2.00	13.4	1.97	15.06	2.03	1.73	12.6	1.98	13.98	2.08	2.19
M050S1	0.18-0.30	23.7	0.40	25.48	1.73	1.84	22.4	0.27	21.99	2.00	1.38
M050S2	0.42-0.60	23.7	0.61	23.84	1.67	2.02	22.4	0.51	23.00	1.83	1.58
M050S3	2.00	23.7	2.06	20.35	2.57	2.42	22.4	1.96	20.83	2.44	2.23
M100S1	0.18-0.30	38.1	0.45	37.94	2.11	1.74	36.6	0.26	35.25	1.99	1.97
M100S2	0.42-0.60	38.1	0.68	36.62	2.07	2.71	36.6	0.29	37.31	2.16	2.11
M100S3	2.00	38.1	1.99	34.07	2.17	1.68	36.6	1.95	32.79	2.47	1.64

The maximum prediction error for the specimens belonging to the group I was 14.14% (M050S3) whereas for the group II was 26.67% (M025S2). Nevertheless, the predictions errors in volume fraction for most of the specimens are less than 10%. In addition, despite of the aggregate-to-cement ratios being fixed a priori in mortar making, differences between nominal and real values occur [7]. For instance, a good fitting was obtained for specimen M025S2 of group II (worst case; see Table 1 and Fig. 3), but discrepancies between nominal values and volume fraction predictions were, however, found. Destructive measurements will be conducted in order to corroborate those results. On the other hand, the predictions of average sizes in the cases of radius with suffix S1 are much higher to those corresponding to the nominal values. In order to achieve better predictions, a broader frequency range should be used, however, it is worth noting that multiple solutions may occur due to combinations of size and volume fraction with the same attenuation profiles [2]. However, a good agreement between theoretical and experimental curves is obtained as illustrated in Fig. 3. As for volume content of entrapped air voids, the sum of volume fraction $\Sigma\varphi_v$ was estimated (see Table 1) regarding five-different size population of air voids, whose results lie in the expected physical range (<10%) as well as the average diameter of entrapped air voids, computed by a weighted average regarding the volume fractions, leading to values $\overline{d}_v$ < 3 [mm] which again are within the expect physical range [2, 3].

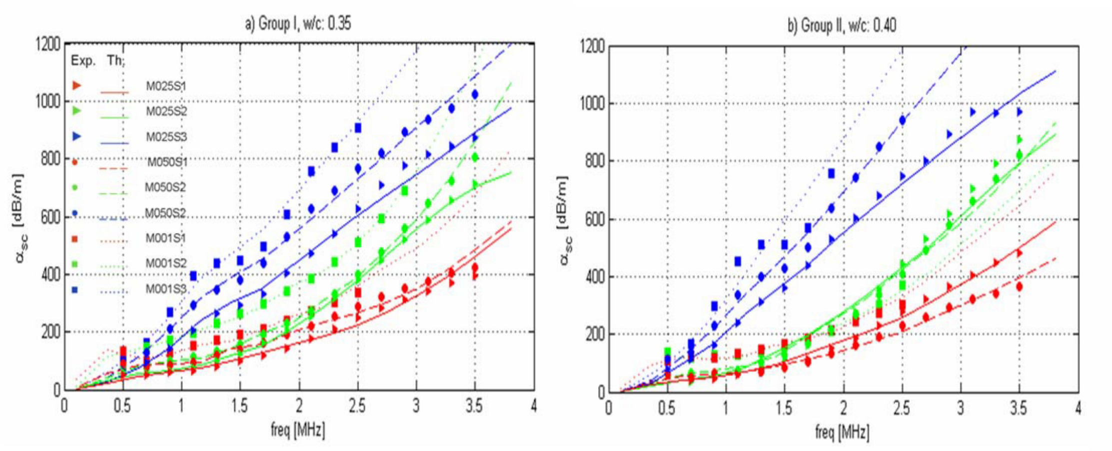

Fig.3 Scattering attenuation curves for a) group I and b) group II. *th* and exp denote theoretical and experimental values, respectively.

5. Conclusions

To assess the average size and volume fraction of the inclusions in cement mortar specimens, an inverse procedure of a multiple scattering model was carried out. Scattering attenuation profiles were calculated by means of an N-phase extension of the dynamic generalized self-consistent model, by taking into account a three-phase material: cement matrix, aggregates (microspheres) and entrapped air voids. It is worth noting that above-mentioned model can be used on any kind of composite material. In order to estimate the scattering attenuation curves, a time-frequency procedure was proposed. The predictions of both size and volume fraction are estimated, and the experimental attenuation curves are in a good agreement with the theoretical predictions. To further improve the accuracy of the estimation, velocity profiles will be added to the optimization process.

Acknowledgement

The financial support of the Spanish Science and Innovation Ministry (Project BIA 2006-15188-C03-01) and the Spanish Ministry of Public Works (C14/2006 and FOM 01/07) are greatly acknowledged. M. Molero was supported by the department of education of the Community of Madrid and the European Social Fund and The Mexican National Council for Science and Technology CONACYT: (186384).

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