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Studying the ultrasonic characterization of industrial plasters

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Materials made from plaster contain gypsum and air scatterers. The latter strongly influence the mechanical strength and the volume density. Solutions for ultrasonic non-destructive characterization are designed to enable a better understanding of the influence of process parameters and to optimize this process. The model of homogenisation of Waterman-Truell is chosen to simulate the propagation of compression waves in the plaster medium. Validation tests of the model using ultrasound by immersion were set up for samples in vacuum sealed bag. The test results are close to those obtained from the model. The first results of characterization by ultrasonic air are also presented. The potential for non-destructive characterization of plaster using non-contact ultrasound is demonstrated.

1 Introduction

The plaster is an important part of the employed materials in civil engineering constructions. The manufacture process of industrial plasters needs to control the physical and mechanical characteristics of the final products. In order to insure mechanical resistance, thermal conductivity or phonic isolation, this process acts water/gypsum ratio and adds air bubbles during the manufacture. These bubbles increase the air volume ratio in the material and modify the volume mass of the product. Increasing the air volume ratio induces increasing modifications of thermal and phonic performances but also decreasing mechanical resistance.

To optimize the introduction of air bubbles in the plaster slurry it is important to develop measurement systems of air volume ratio in the final products. Forward it would be possible to measure these quantities directly during the processing and could ensure the enslavement of the air bubbles injection. Among non destructive testing methods, only the ultrasonic techniques can resolve the need of characterization for the variations of mechanical and physical properties taking into account the nature of plaster, its opacity and the prospect of future inspections during the process. Ultrasonic velocities are related to mechanical characteristics depending on air volume ratio in the material [1,2]. In these studies the air volume ratio depends on the w/g ratio (water on gypsum) but no air bubbles are introduced. Higher is the w/g ratio higher is the air volume ratio and lower are the mechanical characteristic in a common way.

The considered industrial foamed plasters are composed of gypsum matrix with a chosen w/g ratio and containing air bubbles which can be of different sizes and different volume ratios. Ultrasonic waves propagating in heterogeneous media composed of two phase material are scattered by inclusions. This scattering phenomenon can occur several times and one can observe multiple scattering of wave. Incoherent waves appear in the medium and the coherent part is affected by this transformation. The measurement of coherent wave parameters can inform about the origin of the scattering that is to say about the size and volume ratio of inclusion in matrix. The frequency behavior is very important in wave scattering phenomenon.

The analysis of the evolution of ultrasonic velocities and attenuation is done using analytical model of Waterman-Truell (WT) [3] and comparing results to experimental data obtained in laboratory condition. The link between ultrasonic parameters and air bubbles is thus established for the first time in gypsum materials.

After a brief description of the manufacture process, the model is described and the frequency behavior of velocity and attenuation is shown for different size and volume ratio of air bubbles. Then the two experimental set-ups are presented: the first one, used to validate model, was made by immersion technique and the second one allowed to obtain velocity measurement in aerial condition. Finally the results are exposed and discussed to conclude this study.

2 Manufacturing process of plasterboard

The plasterboard (figure1) usually composed by two paper sheets around the plaster. Sheet characteristic and plaster ones contribute to the performance and quality of the plasterboard. Field of use of the board depends on both different characteristics of the sheet and the plaster.

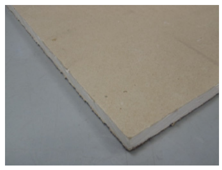

Figure 1: Plasterboard.

The final part of the manufacturing process associates paper sheets with plaster. The hydrated plaster is laid down on the first paper sheet and the second sheet is assembled on the plaster. The assembly is laminated on the production line to give the good thickness between the two sheets. Then the boards are dried with controlled atmosphere, temperature and time to obtain solid assembly with expected quality. Finally the boards are cut at standard dimensions to be marketed.

This study focalized on the manufacture of foamed plasterboard in which the addition of air bubbles is realized directly in the slurry (mixing of plaster and water). This foamed plaster is primarily composed of gypsum ($CaSO_4$+$2H_2O$) with some additives to control the hydration process. The obtained structure is done by small crystals of gypsum of few micrometers length which are tangled. This organization of the matter induced void between crystals depending on the w/g chosen for the formulation. These voids are called the micro-porosity of the plaster. In addition some air bubbles of about few hundred micrometers diameters and controlled volume ratio are injected in the liquid slurry. These air bubbles are called the macro-porosity. It results a solid material composed by gypsum, and air cavity of different sizes (figure2).

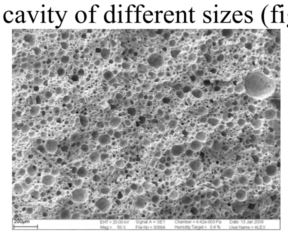

Figure 2: Structure of studied industrial plasters.

The total air volume ratio is an important parameter for mechanical, acoustical and thermal performances of the final product. This study is centered on the ultrasonic characterization of the macro-porosity introduced. At this scale, one can consider a homogeneous matrix with macro-porosity. As we can see on figure 2, the macro porosity is composed by air spherical inclusions the plaster matrix. The forms of these inclusions are very near to spherical ball and the sizes can be different. In case of process optimization, the diameters and the volume ratio of macro-porosity can be controlled.

3 Ultrasonic propagation model

The propagation medium is composed by a matrix containing punctual air spherical air inclusions. Each inclusion gives rise to scattering of ultrasonic waves. In the study we limit to the longitudinal wave propagating in the medium and we choose to use the Waterman-Truell's model [3]. This model is based on the principle of dynamic homogenization of the heterogeneous initial medium (figure 3) and it takes into account multiple scattering. This model has been validated for elastic [4] and liquid [5] matrix with spherical scatterers up to volume ratio of about 30%.

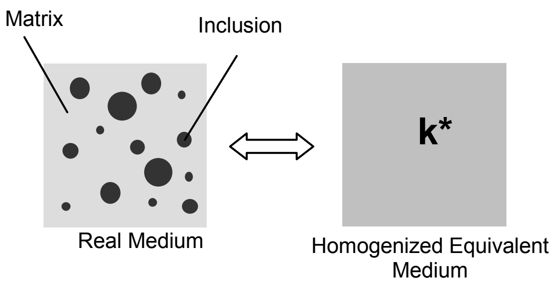

Figure 3 : Homogenization principle

The Waterman-Truell's model takes into account multiple scattering equations and applies averaging techniques to consider the random positions of the scatterers. In the initial form, all the scatterers are identical, can be spherical but with the same matter and the radius a. The volume ratio of air spheres in the matrix is included by the number of scatterers by unit of volume n_0. The common form for the model result is done by

$$\left(\frac{k^*}{k_1}\right)^2 = \left[1 + \frac{2.\pi.n_0.f(0,a)}{k_1^2}\right]^2 - \left[\frac{2.\pi.n_0.f(\pi,a)}{k_1^2}\right]^2 \qquad (1)$$

where k_1 is the wavenumber of plaster matrix and f(0,a) and f(π,a) are respectively the forward and backward scattering amplitude for one obstacle of radius a. Theses values can be easily obtained using equations provided for the case of longitudinal wave scattering by spherical scatterers by Brill [6].

The propagation characteristic which are the velocity (c*) and the attenuation (α*) of longitudinal wave are extracted of the wavenumber of the equivalent medium by

$$k^* = \frac{\omega}{c^*} + i.\alpha^* \qquad (2)$$

where ω=2.π.f is the wave pulsation and f the frequency.

Taking into account size distribution is possible by introduction of averaging on the size by

$$\langle f(\theta) \rangle = \int_a f(\theta, a).da \qquad (3)$$

The mean values for <f(0)> and <f(π)> are inserted in equation 1 instead of f(0,a) and f(π,a).

4 Experimental set-up

Experimental part of this work has several objectives. The first one was to demonstrate the feasibility of ultrasonic velocity and/or attenuation measurements on plasterboard (plaster and two sheets). This objective was verified and validated at the beginning of the study and it is not presented in this paper. The second objective was the improvement of the knowledge for the wave propagation in plaster. This is realized by the comparison of the result provided by theoretical model and ultrasonic measurements on specimen. Final objective was to demonstrate if ultrasonic in situ measurement by aerial technique is possible or not.

4.1 Specimens

The specimens, used to obtain ultrasonic measurement, are small plates of different plasters. The size is about 150x100x11 mm. For these specimens, we have only plasters matter without the two sheets.

For each presented specimen (see table 1) the same matrix was employed with a w/g ratio value of 0.85. For three specimen air bubbles was added for different mean value of diameter and different volume ratio. To produce air bubbles in plasters, a foam is mixed with liquid plaster paste. The quantities of added foam allow to control the volume ratio and the manufacture of the foam ensures diameter of air bubbles.

Table 1: Plaster specimens

		Macro-porosity	
	Volume mass (kg/m3)	Sphere diameter (μm)	Volume ratio (%)
Matrix	902	-	-
D1100V15	826	1100	15%
D850V18	818	850	18%
D200V24	673	200	24%

The values of sphere diameters and volume ratio are verified by tomography X for each specimen. Spheres diameter is the mean value of air bubbles diameters. The diameter distribution in the specimen was obtained and standard deviations are small in comparison with diameter values.

4.2 Immersion technique

In order to analyse and to compare the results provided by the model, we have to obtain experimental data for phase velocity and attenuation for the specimens. Contact measurements provide data about velocity but the amplitudes are perturbed by coupling conditions of the transducers. Due to heterogeneous nature of the medium the obtaining of two successive echoes can be difficult. Immersion techniques are adapted and performance but the porous and soluble nature of plaster prohibits direct immersion for specimens. It was chosen to seal all specimens in vacuum and waterproof bags.

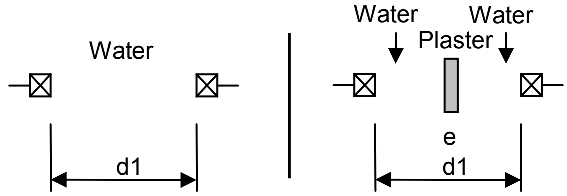

Figure 4 : Immersion measurement by comparison method

The ultrasonic measurements were done by comparison technique (figure 4) on two signals obtained by transmission: first in water and second in water and specimen.

Phase velocity in specimen c_p is obtained by

$$c_p = \frac{\omega.e}{\phi_p - \phi_w + \frac{\omega.e}{c_w} + \phi_{Div}} \tag{4}$$

where e is the sample thickness, ϕ_w and ϕ_p are the phases of the signals obtained respectively in water and in water and specimen. ϕ_{Div} is a term of divergence correction.

Attenuation α_p is obtained by

$$\alpha_p = -\frac{1}{e}.\ln\left[\frac{C_{Div}}{T_{ws}.T_{sw}}.\frac{A_p}{A_w}\right] \tag{5}$$

where A_w and A_p are the amplitudes of the signals obtained respectively in water and in water and specimen, Tws and Tws the transmission coefficients for the two crossed interfaces. C_{Div} is a factor of divergence correction.

The experimental chain is composed of an oscilloscope, an ultrasonic generator and two couples of transducers (Panametric: 2xV301 at 500 kHz central frequency and 2x V302 at 1 MHz central frequency). The signal is large frequency band and the analysis is done from Fourier analysis. All details of this experiment can be found in [4].

The effect of bag was evaluated by measurements on a plexiglass part. The effect on velocity seems to be negligible and the attenuation provided can be slightly over-evaluated (about few percent). This test needs to be improved to provide a factor which can correct the measured values of attenuation.

4.3 Aerial technique

Aerial ultrasonic measurements need high energy to explore internal material. This high energy can be obtained but the quality of signal decreases in comparison to the one obtained by immersion technique. The level of noise is important and the temporal lengths of echoes are long. It is observed superposition of different signals.

To obtain velocity measurement for plaster, a comparison method (figure 5) was chosen with the plexiglass part. This specimen was characterized, in a first time, with the immersion experimental set-up. In a second time, it is used like reference part for aerial measurements.

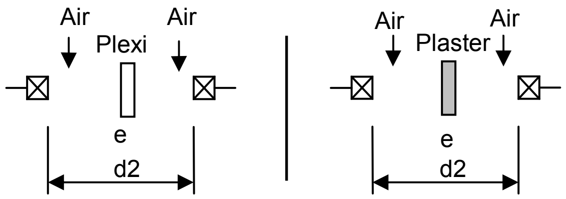

Figure 5 : Aerial measurement by comparison method

The ultrasonic measurements were done by obtaining the times of propagation for the two experimental configurations of figure 5.

The time of propagation for the first configuration of figure is

$$t_1 = \frac{(d_2 - e)}{c_{air}} + \frac{e}{c_{plexi}} \tag{6}$$

The time of propagation for the first configuration of figure is

$$t_2 = \frac{(d_2 - e)}{c_{air}} + \frac{e}{c_p} \tag{7}$$

where c_{air}, c_{plexi} and cp are respectively the ultrasonic velocities in air, in plexiglass and in the tested specimen.

By difference of the eq. 5 and eq. 6, on can obtain

$$c_p = \frac{c_{plexi}.e}{e - c_{plexi}.\Delta t} \tag{8}$$

where $\Delta t = t_1 - t_2$ is the time difference measured between the two configurations.

The experimental chain is composed of an oscilloscope, a computer card which generates signals for aerial ultrasonic sensor and a couples aerial sensors (Ultran NCG500 at 500 kHz central frequency). The signals are chosen sinusoidal to obtain the phase velocities. Velocity curve versus frequency is obtained scanning different frequencies in the pass-band of frequencies of the sensors.

5 Results and discussion

In a first time, the behavior of ultrasonic longitudinal wave in plaster without macro-porosity (Matrix) is studied. The phase velocity evolution versus frequency is presented on figure 6a) and the attenuation evolution versus frequency is on figure 6b).

Good agreement between the two couples of transducers is obtained for the phase velocity and also for the attenuation. Small dispersion is observed for the velocity behavior (about 200 m.s^{-1} on the total pass-band) with increasing velocity versus frequency. Attenuation is strongly frequency dependant and seems to have linear evolution versus frequency. This behavior is generally related to attenuation by absorption phenomenon. Theses two behaviors for velocity and attenuation are definitely in relation with micro-porosity which is include in the Matrix.

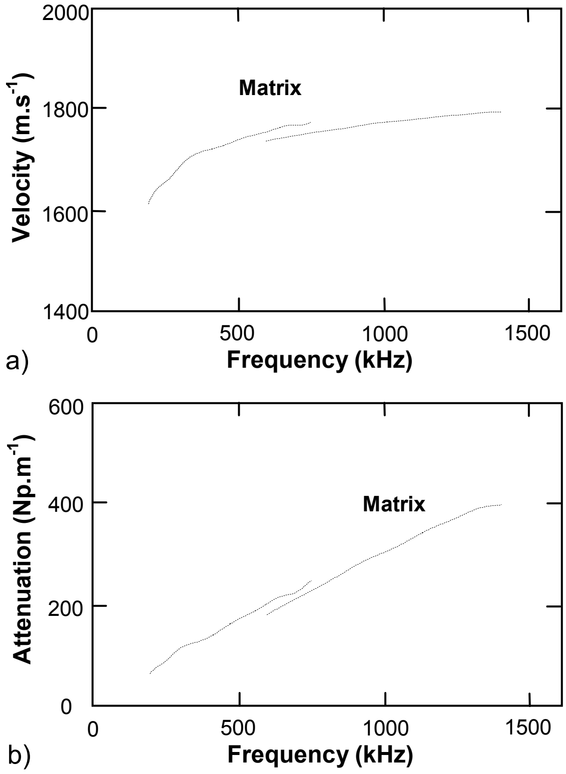

Figure 6: Experimental phase velocity and attenuation versus frequency in "Matrix" specimen

In a second time, the behavior of ultrasonic longitudinal wave in plaster with macro-porosity (D1100V15) is studied. The phase velocity evolution versus frequency is presented on figure 7a) and the attenuation evolution versus frequency is on figure 7b).

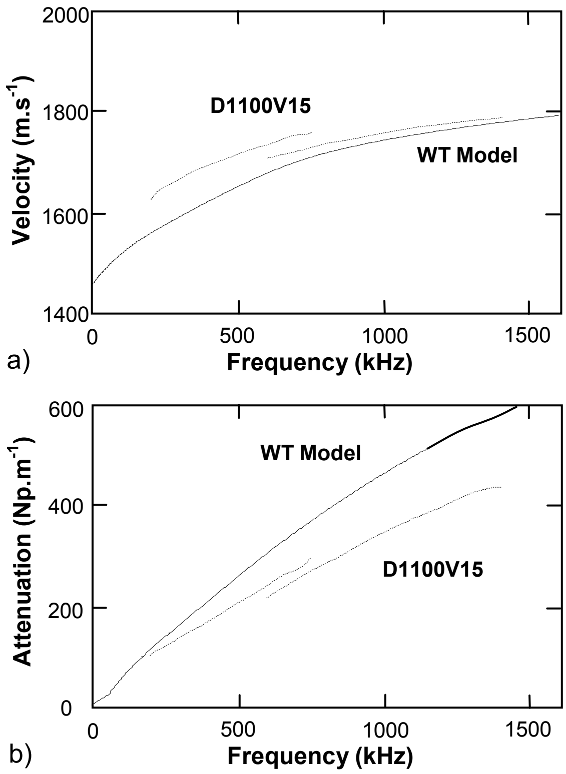

Figure 7: Phase velocity and attenuation versus frequency (– – – WT Model, — Exp: D1100V15)

On each curve, one can observe experimental data (dashed lines) and the Waterman Truell (WT) model result (continuous lines). The model curves are obtained integrating in the model (eq. 1) the matrix experimental data (figure 6) and 15% of air spherical inclusions with central diameter at 1100 µm. The introduction is done according to the diameter distribution obtained for each specimen from the tomography X.

Like previous observations, the phase velocity is slightly dispersive and the attenuation is strongly increasing with frequency. The values of velocity for D1100V15 are smaller than the ones of matrix specimen. The attenuation for D1100V15 is bigger than the one for matrix specimen. Introduction of air scatterers induces decreasing velocity and increasing attenuation, this is an expected result due to air characteristics and the scattering phenomenon.

The experimental attenuation seems to be linear but the model shows also linear dependence. The model takes into account the attenuation of the matrix but also the attenuation due to the scattering phenomenon. The velocity and the attenuation are in good agreement between experimental data and model ones. For high frequency typical divergence for attenuation can be observed. For higher volume ratio of air inclusions (Not presented here - D200V24 for example), the agreement becomes less good. The observed differences can be attribute to model limits but also to air bubbles repartition which can be inhomogeneous for some specimens. Some images obtained from tomography X suggest that air bubbles are not well distributed.

The model is able to describe qualitative evolutions of phase velocity and attenuation versus frequency, inclusions radius and volume ratio of inclusions. To conclude on quantitative agreement, this study needs to be completed with other in order to conclude on frequency limit or air volume ratio limit for the model.

In a third time, the phase velocity evolution versus frequency obtained by aerial technique for D850V18 specimen is studied and presented on figure 8. It is superposed with the one obtained by immersion technique.

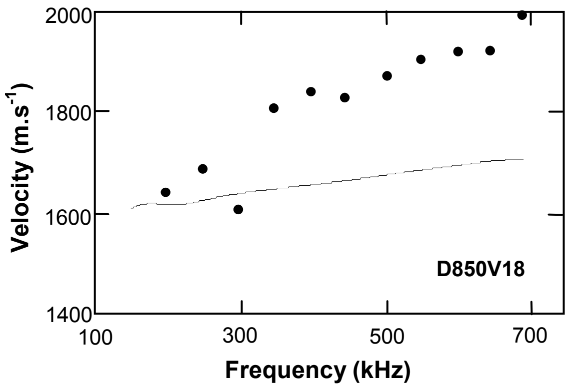

Figure 8: Measured phase velocity versus frequency in D850V18 specimen (— Immersion technique, • • • Aerial technique)

Due to the monochromatic method used for aerial technique, only few points are obtained in the pass-band. A good agreement is obtained for three points under 300 kHz. For other points the agreement is not good and measured difference is about 200 $m.s^{-1}$. This first measure with aerial sensors shows the feasibility of aerial measurement but also some difficulty due to the high energy involved in testing.

5 Conclusion

Ultrasonic characterization of gypsum-based materials is nowadays still relatively unexplored. In appearance one can conclude to the simplicity of structure for this material. Nevertheless the air bubbles introduction in the liquid plaster paste brings complexity in the structure geometry with the coexistence of porosity at two different scales.

The first experiments allow to concluding to a dispersive and attenuation media for ultrasonic waves in plaster without macro-porosity. Assuming the micro-porosity known, this study shows a solution possible solution for modeling macro-porosity. Good agreements are obtained between experimental data and modeled ones. The Waterman-Truell model provides qualitative evolutions for velocity and attenuation in heterogeneous plasters. Finally aerial techniques are implemented and the feasibility of phase velocity measurements is demonstrated. This latter result opens the way for new solutions of in situ ultrasonic measurements.

The general prospects of this work concern the quantitative validations of the model and the improvement for aerial measurements.

Acknowledgments

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