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An alternative and industrial method using low frequency ultrasound enabling to measure quickly tortuosity and viscous characteristic length

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In the past, several different methods have been proposed in order to determine tortuosity and characteristic lengths, using various physical approaches. For instance, the BET (from S. Brunauer, P. Emmett and E. Teller) method used the adsorption of argon molecules within the surface of the pores to measure thermal characteristic length (or the so-called specific surface). Ultrasonic methods, during the two last decades, were efficiently implemented to measure these parameters, using large frequency bandwidth, different gases including helium, and varying static pressures. The most standard and well-known method relies on phase spectrum measurements performed over short bursts, by plotting the inverse of the squared velocity as a function of the inverse of the square root of the angular frequency. Unfortunately, this method is quite sensitive on noise during the unwrapping procedure. We here propose a much simpler and efficient method, which is very robust with noisy signals, working at one single frequency, in most cases around 40 kHz. Measurements have been done onto a specific bench developed at CTTM, with some calibrated glass beads, enabling to recover the expected values of the physical parameters. This method is particularly well suited for industrial and "on line" applications.

1 Introduction

As it is well known, tortuosity and characteristic lengths (both viscous and thermal one) are basic physical parameters which are very useful in order to describe the propagation of acoustical waves at the same time in the frame of the so-called « equivalent-fluid » model, when the solid skeleton is motionless [1], and within the M.A. Biot theory, when some coupling exist between the waves propagated in the saturating fluid and inside the solid phase [2]. These parameters can be measured with different methods [3,4], but ultrasonic techniques are definitely very fast and quite efficient for that purpose, as it was described during the last 15 years ago [5,6]. For instance, propagation in different gases [6,7] or in air at different static pressures [8,9] was used in order to independently determine at the same time tortuosity and the two characteristic lengths. Most of the ultrasonic methods are based onto the very same physical principles. On one hand, dispersion curves enabling to obtain $c(\omega)$, i.e. the phase wave speed versus frequency, allows to determine tortuosity in the high frequency limit (see details in section 2 for some relevant equations). On the other hand, attenuation versus frequency $\alpha(\omega)$ provides some information onto the characteristic lengths, but in that case one needs to get two independent equations, which are given in turn by using two different gases [6,7] e.g. helium and air, or alternatively the same saturating fluid at two different (or more) static pressures [8,9]. At that time, several alternative techniques were implemented in Le Mans as well as in other laboratories (e.g. Katholieke Universiteit Leuven). In order to simplify these measurements for industrial applications, it is very convenient to consider that the ratiolinking the thermal to the viscous characteristic length has a given value, most often taken between 2 and 3. By the way, the viscous characteristic length is somewhat related to the average size of the small pores, while the thermal length is linked to the large ones. The influence of such ratio onto dispersion and attenuation curves is most often quite small and the above approximation is amply sufficient for industrial applications. Moreover, measurements performed in various gases or at different static pressures are time consuming, because in each case one needs to wait until thermal equilibrium (i.e. temperature recovery or stability) is reached after gas removing and / or during pressure step change. In the present work, such simplified two parameters determination procedure is used. Instead of using broad bandwidth transducers enabling to cover a significant portion of the dispersion curve after phase unwrapping, some narrow band ultrasonic sensors are utilised. These transducers are working around 40 kHz, producing tone-burst. Such frequency at the same time is sufficiently high in order to use the so-called high frequency asymptotic approximation (see next section for further details) and also is low enough to get a wavelength much larger than the pore size. This second point is very important to avoid diffusion process to occur. In other words, one needs to get a wavelength large enough to introduce a homogeneity volume covering a sufficient number of pores, some of them being small, close to the viscous characteristic length, the others being larger at the order of the thermal characteristic length. Beyond this homogeneity assumption which is fundamental when dealing with inhomogeneous porous materials, one also needs to verify that the viscous skin depth $\delta = (2\eta/\rho\omega)^{1/2}$, is smaller than the pore size, where ρ is the density of the saturating fluid and η its viscosity. For instance, at 40 kHz, such parameter lies around 10 µm, and accordingly porous structures with very tiny pore sizes (around and below 10 µm) cannot be measured with such ultrasonic technique unless frequency is increased in order to get a smaller skin depth, otherwise the full volume of the pore behaves like a viscous skin depth and consequently attenuation becomes drastic and ordinary propagation process no more takes place within the porous structure. Accordingly, some compromise definitely exists between frequency, average pore size and acoustical attenuation inside the free air. As stated above, the skin depth depends on the angular frequency ω by the inverse of its square root, and consequently some physical limit exists. When the average pore size is decreased, let say by 10, one needs to increase the frequency by 100 (in order to also decrease the viscous skin depth by 10), but in such case, the basic attenuation in the free air, and somehow within the porous material, is multiplied by 10 000 (being proportional to ω^2, an universal feature which is well known). The very best compromise in order to characterise porous media saturated by air at ambient static pressure (i.e. 1 bar or 10 500 Pa), in most ordinary cases, is to use a frequency in the range of 40 kHz. The following sections provide some details on the equations implemented in the software as well as on the numerical schemes and signal processing routines. The central frequency of the acoustic burst is a very basic and important feature in the numerical treatment. Some significant experimentation on glass beads are described for calibration purposes [10]. Comparisons are then performed between measured and predicted absorption coefficient of industrial porous materials, for which all intrinsic parameters are measured and used in the "equivalent fluid" model.

2 Analytical model

In the frequency domain of our application (≈ 40 kHz), from the high frequency asymptotic approximations of the dynamic compressibility and tortuosity [10], it is possible to express the refractive index $n = c_0/c(\omega)$ as follows:

$$\left(\frac{c_0}{c(\omega)}\right)^2 = \alpha_\infty\left[1+\delta\left(\frac{1}{\Lambda}+\frac{\gamma-1}{B\Lambda'}\right)\right] \qquad (1)$$

Where ω is the frequency, c_0 is the speed of sound in free air, $c(\omega)$ is the phase wave speed, $\delta=\sqrt{2\nu/\omega}$ is the viscous skin depth (defined with the kinematics viscosity ν), $\gamma = C_p/C_v$ is the ratio of the specific heats, $B=\sqrt{Pr}$ and Pr is the Prandtl number.

Λ and Λ' represent respectively the viscous and thermal characteristic lengths.

With the same frequency asymptotic approximations, the transmission coefficient takes the following form:

$$\ln[T(\omega)] = \ln\varepsilon - \frac{\omega}{c_0}\sqrt{\alpha_\infty}\left[\frac{\delta}{2}\left(\frac{1}{\Lambda}+\frac{\gamma-1}{B\Lambda'}\right)\right]L \qquad (2)$$

Where L is the thickness of the sample and ε is defined as follows:

$$\varepsilon = \left(-\frac{4\phi}{\sqrt{\alpha_\infty}}\right)\left(1+\frac{\phi}{\sqrt{\alpha_\infty}}\right)^{-2} \qquad (3)$$

For most porous materials ($\alpha_\infty \sim 1$ and $\phi \sim 1$), ε is close to one and the Eq.(2) can be expressed as a simplified relation of the attenuation per length unit a:

$$a = \frac{|\ln|T(\omega)||}{L} \approx \frac{\omega\sqrt{\alpha_\infty}}{c_0}\frac{\delta}{2}\left(\frac{1}{\Lambda}+\frac{\gamma-1}{B\Lambda'}\right) \qquad (4)$$

The new ultrasonic method described in this paper in based on time delay and attenuation measurements of a 40 kHz tone burst travelling across the sample. This time delay τ can be expressed as a function of the wave speed in free air, the phase speed of the wave in the sample and the thickness of the sample:

$$\tau(\omega) = L\left(\frac{1}{c(\omega)}-\frac{1}{c_0}\right) \qquad (5)$$

The equations system (1), (4) and (5) can be solved easily to give the following expressions of the tortuosity and the characteristic viscous length:

$$\alpha_\infty = \left[\left(\frac{|\ln(T)|c_0}{L\omega}\right) - \sqrt{\left(\frac{|\ln(T)|c_0}{L\omega}\right)^2 + \left(1+\frac{c_0\tau}{L}\right)^2}\right]^2 \qquad (6)$$

$$\Lambda = -\frac{\delta}{2}\left[\frac{NB+\gamma-1}{NB}\right]\left[1-\sqrt{1+\left(1+\frac{c_0\tau}{L}\right)^2\left(\frac{\omega L}{c_0|\ln(T)|}\right)^2}\right] \qquad (7)$$

As justified in the first section, these relations are obtained considering that the thermal and the viscous characteristic lengths are linked by a ratio (N in Eq. 6) between 2 and 3.

3 Experimental set-up and procedure

The measurements are performed by positionning the sample between two ultrasonic transducers, one emitter and one receiver (Fig.1). The emitter provides a burst of sine wave with a 40 kHz frequency.

The computer generates the signal and simultaneously records the received signal via a power amplifier.

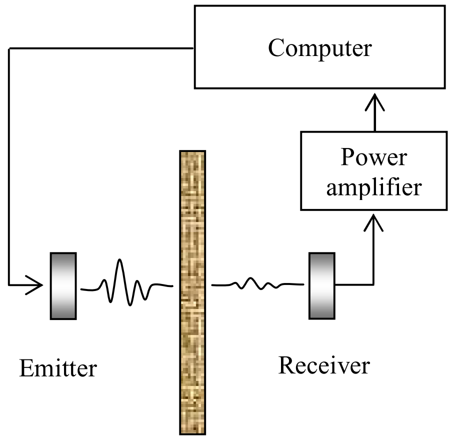

Fig. 1 Experimental set-up.

The measurement is performed in two steps: first, a reference signal is acquired without sample. Then, the same measurement is performed with the sample positionned between the two transducers. The comparison of the two registered signals allows to extract relative time delay and amplitude attenuation caused by the tested material (Fig.2). A correlation method is used to increase accuracy of the parameters estimation.

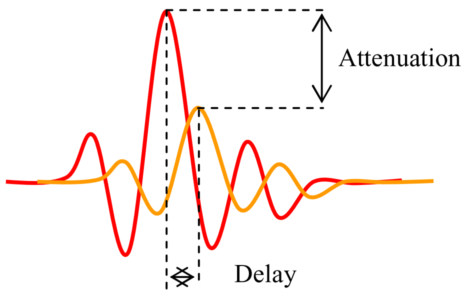

Fig. 2 Signal shapes with and without sample

The tortuosity and the characteristic lengths are deduced from the Eq.6 and 7. The measurements uncertainties are evaluated according to ISO "Guide to the expression of uncertainty in measurement" [11].
Just a few minutes are needed to performe the complete measurement.

4 Experimental validations

To validate the method, measurements were performed on calibrated size glass beads. The diameter is d = 1.46 mm, with a standard deviation equal to 0.02 mm.

The reference value of tortuosity, evaluated by conductivity measurements [10] is $\alpha_\infty = 1.37$.

The reference characteristic viscous lengths, evaluated by calculation [10], take the following value: $\Lambda = 90$ µm.

Series of experiments were conducted on the test bench with different thickness of glass beads (10 measurements for each thickness). The results of tortuosity and characteristic lengths measurements are presented in Table 1 with the standard deviation. A good correlation of the measured value of α_∞ and Λ with the reference values clearly appears.

Thickness	Mean tortuosity	Standard deviation	Mean viscous lenght	Standard deviation
13	1.37	0.034	87.3	7.7
17	1.35	0.014	76.1	2.9
21.5	1.4	0.017	95.7	4.9
All measurements	**1.37**	**0.03**	**86.4**	**9.7**

Table 1 Results of glass beads measurements

5 Application to porous materials

To illustrate the application of the proposed method, a complete characterisation was performed on two different porous materials: a plastic foam, and a felt.

The set of parameters used in the equivalent fluid model was measured on different benches at the CTTM (resistivity, tortuosity, characteristics lengths) and at the LAUM (porosity).

The software MAINE3A was used to predict the normal incidence absorption coefficient of the material.

Finally, a measurement of the absorption coefficient was performed using the Kundt technique. Based on these measurements, some fitting is computed to adjust the MAINE3A model.

The parameters values measured and fitted are presented in Table 2 and the predicted and measured absorption curves versus frequency are presented on Fig.3 and 4.

	Plastic foam		**Felt**	
	Measured values	Fitting values	Measured values	Fitting values
Thickness	13	13	18	18.7
Uncertainties	*0.2*		*1*	
Resistivity	3134	3600	26169	26000
Uncertainties	*200*		*3333*	
Porosity	0.99	0.99	0.98	0.98
Uncertainties	*0.05*		*0.05*	
Tortuosity	1.06	1.06	1	1
Uncertainties	*0.01*		*0.01*	
Viscous characteristic lengths	206.5	190	57.4	80
Uncertainties	*16.5*		*3.5*	

Table 2 Values of measured and fitted parameters

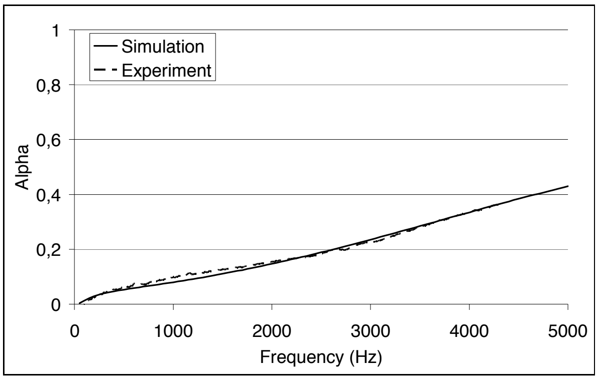

Fig. 3 Calculation/experiment comparison for plastic foam

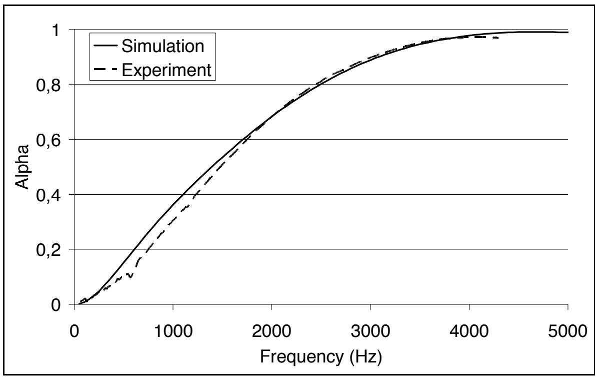

Fig. 4 Calculation/experiment comparison for felt

6 Conclusions

The proposed ultrasonic method for the estimation of tortuosity and characteristic lengths is easy to implement because it operates at single frequency and at standard ambient conditions of air pressure and temperature.

In opposition to the other ultrasonic methods [5 to 10], it allows to perform very fast measurements.

Based on this method, the CTTM has developed an automatic bench which complete the experimental devices for estimations of the set of parameters used in the equivalent fluid model. Other application of the proposed test bench is for example "on line" control.

The building of a prediction model (using MAINE3A for example) ideally requires the knowledge of the complete set of intrinsic parameters. These parameters are sometimes obtained through fitting absorption measurements. The drawback of this inverse method is that the model often yields to multiple or non realistic solutions. The most reliable method to estimate said parameters remains direct measurement such as the one proposed in this paper.

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