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ULTRASOUND – A USEFUL TOOL TO INVESTIGATE COMPLEX MATERIALS

Mariusz KACZMAREK

Institute of Environmental Mechanics and Applied Computer Science
Bydgoszcz University, 85-064 Bydgoszcz, Chodkiewicza 30, Poland

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Abstract

In the paper ultrasonic techniques more frequently used for studies of complex materials are reviewed. The scope of the paper is limited to methods that serve for evaluation of material properties, neglecting other methods as for example those, which are applied in image analysis. Discussion of selected applications of the discussed techniques for identification of properties of some complex materials and remarks to model approaches used within the procedures are included.

Keywords: ultrasonic waves, inhomogeneous, multiphase materials, non-destructive techniques, modeling.

1. Introduction

Most natural materials such as soils, rocks or biological tissues as well as a new generation of engineered materials, belonging to the class of smart or functional media, are complex with respect to their microscopic constitution, internal structure and majority of macroscopic properties. Among characteristic features of such materials are their multiphase or multicomponent nature, complex internal structure, scale dependent properties, inhomogeneity, anisotropy, existence of various dissipation mechanisms significant during mechanical deformations and sensitivity of physical properties of the materials to non-mechanical external loads such as changes of chemistry of the surrounding environment, magnetic or electric fields, etc. (*Fig. 1*).

Due to the above mentioned features, in order to evaluate mechanical or structural properties of complex materials, special techniques are required, which must refer to a particular scale and apply appropriate models representing internal and external interactions.

The purpose of this paper is to present frequently used ultrasonic techniques and to show the applicability of the methods to study complex materials. Selected results for model and real complex materials are described to illustrate different

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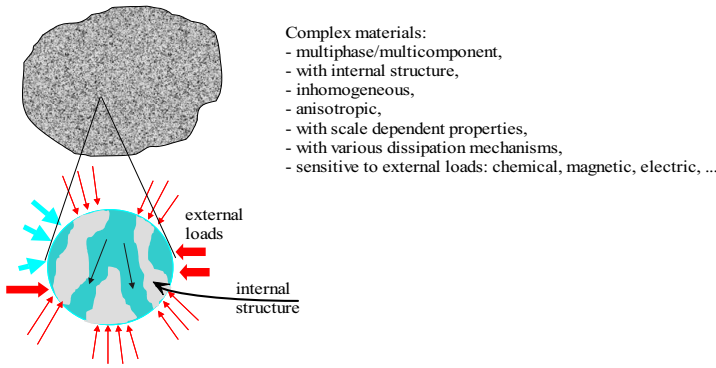

Fig. 1. Features of complex materials

capabilities of ultrasonic methods. A short discussion of basic modeling approaches used to evaluate mechanical and structural properties of the materials is included in the Appendix.

2. Ultrasonic Techniques

Different ultrasonic techniques using low energy mechanical waves have been developed to study properties of materials including their mechanical and structural characteristics. Among the most widely used techniques are (see e.g. [5], [28]:

- pulse/tone burst methods with in time domain analysis of signals,
- broadband ultrasonic spectroscopy,
- continuous wave methods,
- resonance spectroscopy,
- critical angle reflectometry,
- acoustical microscopy.

All the techniques, with the exception of resonance spectroscopy, apply running or standing pure bulk waves (longitudinal or shear waves), surface waves (e.g. Raleigh or Love waves) or geometrically dispersed modes. The method of resonance spectroscopy requires the solution of a boundary value problem to determine complex modes of vibrations which are the result of interaction of the applied disturbance with boundaries of studied samples.

2.1. Pulse/Tone Burst Methods with Time Domain Analysis of Signals

Short – broadband pulses or wave packets containing a number of cycles referred to as tone bursts (or bursts), most frequently used as ultrasonic disturbances are

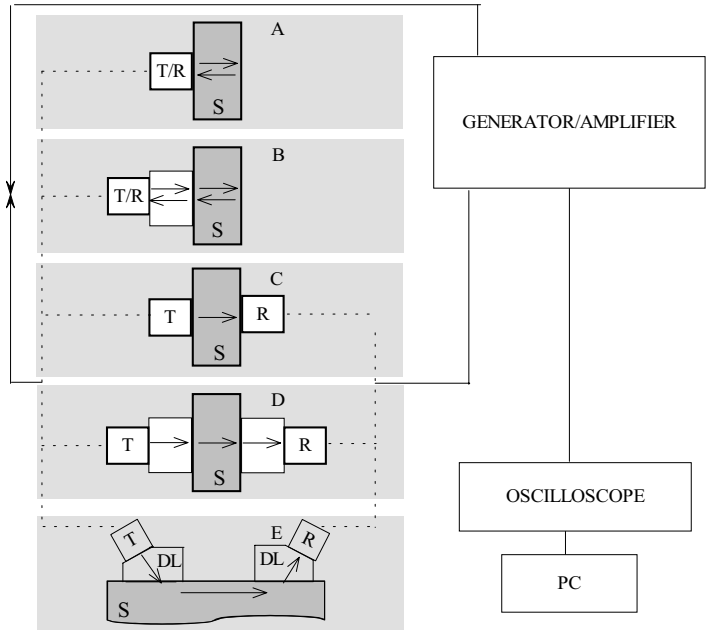

Fig. 2. Experimental setup for pulse/tone burst tones: A) reflection (echo) method, B) reflection method with delay line, C) transmission method, D) transmission method with double delay line, E) transmission method for studies of surface waves

applied to investigate materials, see e.g. [10], cite21. The pulse or burst mechanical disturbance is usually generated by appropriately excited piezoelectric transducer – transmitter (T) (exciting voltage is of a short spike type or packet of sinusoidal voltage), it enters the studied material (S) (in some cases indirectly through a delay line (DL)) and after transmission through or reflection from the internal boundary of the sample it is transformed by the receiving transducer (R) (in the case of echo mode the transmitting transducer works also as the receiver) into voltage, which is recorded and/or measured, see *Fig. 2*. The analysis of time of flight or comparison of amplitudes of the measured signals with a reference signal is then used to determine the wave velocity and attenuation. The evaluation of time of flight in the pulse method is usually done by detecting, e.g. [24]:

- first arrival transit time, t_a,
- 10% threshold transit time, t_t, or
- first zero crossing transit time, t_z, see *Fig. 3*.

Similar criteria for determination of time of flight with additional zero crossing times are used in the case of tone burst signals, see *Fig.3b*. An alternative way for determination of time of flight is the method of correlation, see e.g. [8]. From the

time of flight the velocity is evaluated as the ratio of distance that the wave passes through the studied material, L, and the determined time of flight, t_i, i.e. $v = L/t_i$, where i refers to one of the above indicated methods of evaluation of time of flight (a, t, or z).

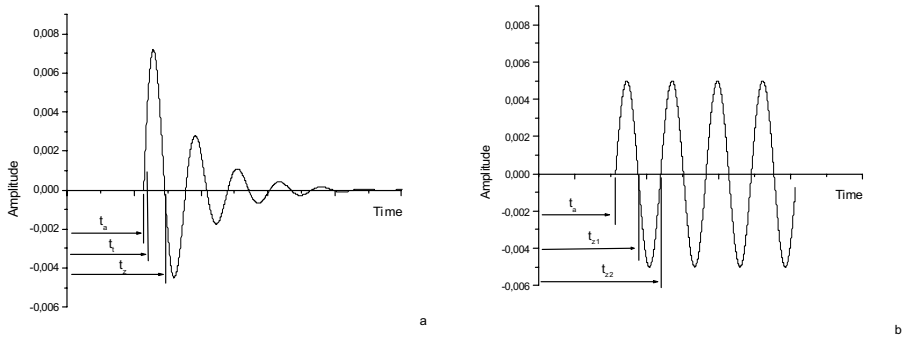

Fig. 3. Criteria for determination of time of flight in case of pulse or tone burst method: t_a – first arrival transit time, t_t – 10 % threshold transit time, and t_{zi} – ith zero crossing transit time

When the frequency dependence of wave velocity (dispersion) is insignificant the time of flight method gives good approximations of both phase and group velocities; otherwise the time of flight gives a better approximation of the group velocity, ([35]). Measurement of attenuation coefficient directly from recorded in time domain signals can be performed using the definition of attenuation α as the logarithm of the ratio of amplitudes of measured and reference signals, A_m, A_r, respectively, divided by the length of pathway of the wave through the tested sample, L, see (*Fig. 4*)

$$\alpha = \frac{1}{L} \ln \frac{A_m}{A_r}.$$

The reference signal may correspond to the wave transmitted through a low attenuating material, as compared with the studied one, or to the wave transmitted through a shorter sample of the investigated material. In the latter case the distance L is equal to the difference in pathway the waves pass through the two samples.

While using the pulse method the determined value of attenuation gives approximation corresponding to the center frequency of the spectrum of the pulse, the burst technique allows for measurement of attenuation for the frequency of the wave packet of burst.

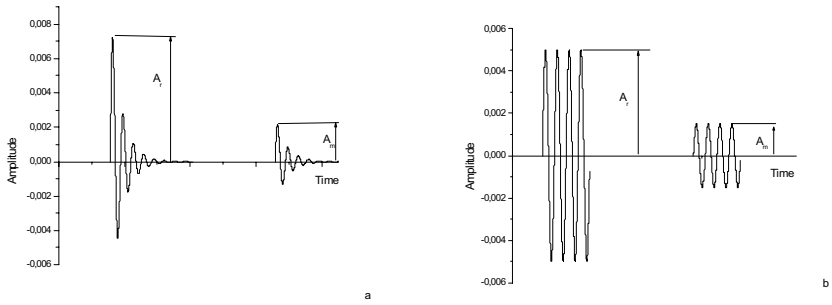

Fig. 4. Amplitudes required for determination of attenuation from pulse methods

2.2. Broad Band Ultrasonic Spectroscopy

The technique is based on application of broad band pulses in reflection or transmission mode, see. *Fig. 2*, and on the analysis of the signals in the frequency domain, e.g. [10], [44].

For determination of wave propagation parameters for a range of frequency reference and measured signals are needed. In reflection mode the reference signal is usually one of the echoes recorded from the back surface of the sample. In transmission mode the reference signal is obtained as transmitted through a reference material or as transmitted through the studied material of different thickness than the measured one (the technique is also called the substitution method). A significant advantage of the latter option is the fact that it allows to avoid correction for loss of energy due to the reflected wave. The spectral analysis of pulses is usually performed numerically with help of the Fast Fourier Transform algorithm giving amplitude and phase spectra of the reference and measured signals, see *Fig.5*.

In the case of measurements performed by transmission method for two samples of the same material and different thickness, L_1 and L_2, the propagation parameters, phase velocity v and attenuation coefficient α as functions of frequency f can be calculated as follows, ([44]:

$$v(f) = \frac{2\pi f(L_2 - L_1)}{\varphi_2(f) - \varphi_1(f) - 2\pi n}$$

and

$$\alpha(f) = \frac{1}{L_2 - L_1} \ln \frac{A_1(f)}{A_2(f)},$$

where $A_1(f)$ and $A_2(f)$ are amplitudes, φ_1(f) and φ_2(f) are phases of the spectral components of the reference and measured pulses, n is the total number of wavelength for a given frequency, which is contained in the distance $L_2 - L_1$.

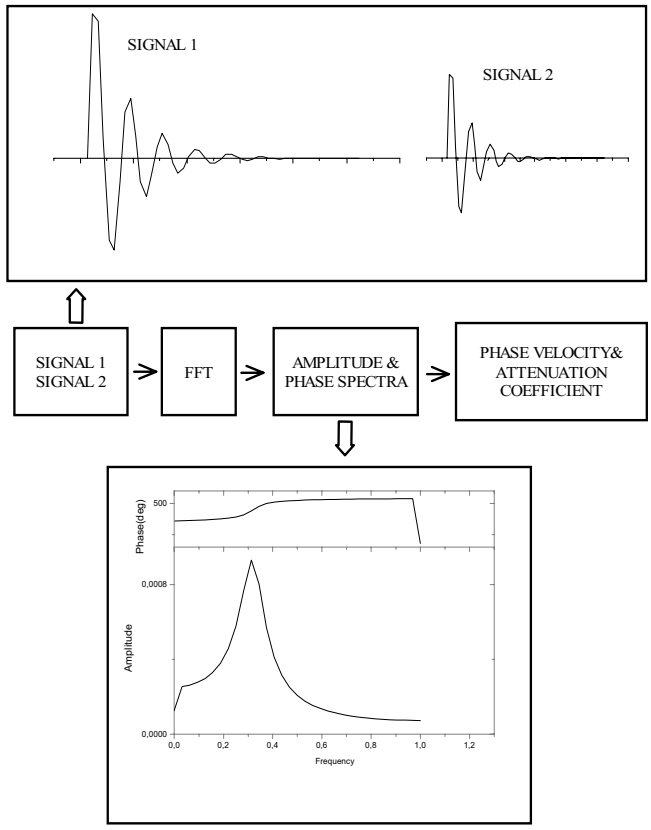

Fig. 5. Schematic view of signal analysis done in broad band ultrasonic spectroscopy along with the example of phase and amplitude spectra for one of the signals

2.3. Continuous Wave Methods

The methods are mostly used for precise measurement of phase velocity of ultrasonic waves. A continuous harmonic excitation of transmitter is applied and amplitude or phase of the signal obtained by the receiver are recorded, while frequency of the transmitted signal or thickness of the sample change (the latter method may be used only for study of fluids), ([38], [39]. A schematic view of experimental setup for the two applications of continuous wave methods with tuning of frequency is shown in *Fig. 6*. The amplitude method is based on detection of the phenomenon of standing wave in a sample of tested material. Assuming insignificant changes in phase velocity at a given frequency range of excited waves, the detection of frequencies f_i corresponding to successive maxima of amplitude of standing waves

enables one to determine the propagation velocity in studied material as:

$$v = \frac{2l(f_k - f_n)}{k - n},$$

where k and n are selected peaks of amplitude and l is the thickness of sample.

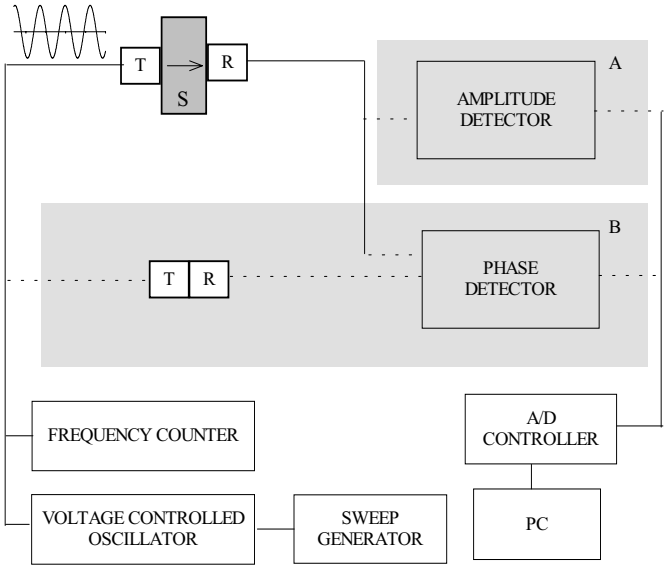

Fig. 6. Setup for continuous wave methods with tuned frequency: A. The branch with amplitude detection. B. The branch with phase detection

The phase method is based on the comparison of phase changes of continuous wave transmitted through a specimen and a reference channel with transducers being in contact through a coupling medium. The phase velocity can then be determined from the equation

$$v = 2\pi l \frac{\Delta f}{\Delta \varphi},$$

where Δf is the change of frequency of the applied excitation and $\Delta\varphi$ is the corresponding difference of phases as measured from comparison of signals transmitted through the sample and the reference channel.

2.4. Resonant Spectroscopy

Resonant spectroscopy is focused on studies of vibrations of free standing sample made of tested material ([20], [42]). Having experimentally determined resonance

frequencies of vibrating modes f_n^m and analytical or numerical solutions appropriate for a given geometry of tested sample along with the predicted resonance frequencies f_n^t the material parameters (e.g. wave velocities, elastic modules) are found by minimizing the error function E which can be defined as e.g. $E = \sqrt{\sum_{n=1}^{N} \left(f_n^m - f_n^t \right)^2}$. It is observed that the resonant methods have become currently important practical tools due to significant development of computer methods of simulation of mechanical vibrations of solid bodies.

2.5. Critical Angle Reflectometry

The technique employs measurements of amplitude and/or phase of reflection coefficients as the functions of angle of incidence of the wave that first travels in fluid and then hits the flat surface of tested material (*Fig. 7*) e.g. [34], [1] [33]. The excited wave is usually of the pulse type. The knowledge of extreme values of the components of reflection coefficient (its amplitude and phase) together with the generalized Snell's law are used to determine propagation velocities of waves. In some cases, depending mostly on the properties of studied materials, the method can be useful to determine velocities of longitudinal and shear waves, but it is mostly applied to measure the velocity of surface waves of Rayleigh or Love type. An appropriate model of wave propagation in the tested material must always be used.

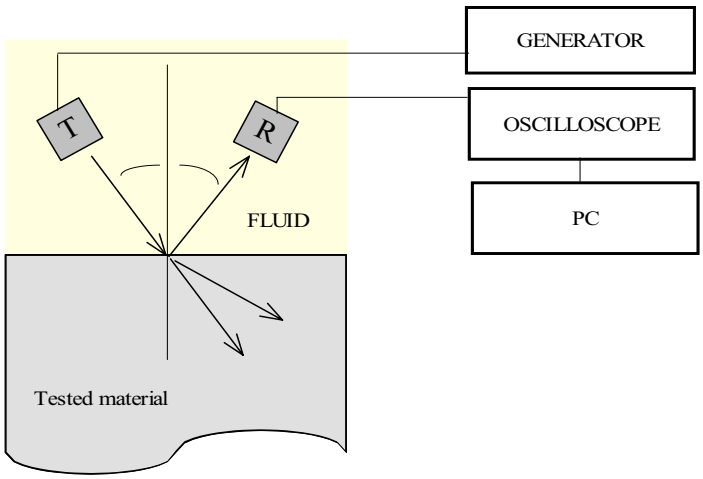

Fig. 7. Experimental setup for critical angle reflectometry

2.6. Acoustic (Ultrasonic) Microscopy

Acoustic or ultrasonic spectroscopy uses waves of the frequency range from 50 to 2000 MHz to visualize the microstructure of materials (acoustic imaging) or to study their local mechanical properties such as elastic modules, wave velocities and attenuation coefficients ([12]). The methods of acoustic imaging called scanning acoustic microscopy serve as techniques for surface inspection in particular to detect structural characteristics of phases: surface fractions, characteristic size of inclusions, distributions of inclusions, parameters of fabric, flaws, etc. For measurement of local mechanical properties in a region being comparable with the wavelength of the applied waves in acoustic microscope the reflected or transmitted waves in one of the configurations shown in *Fig. 8* can be used together with analysis of interference pattern of the running and reflected waves, see e.g.[13] .

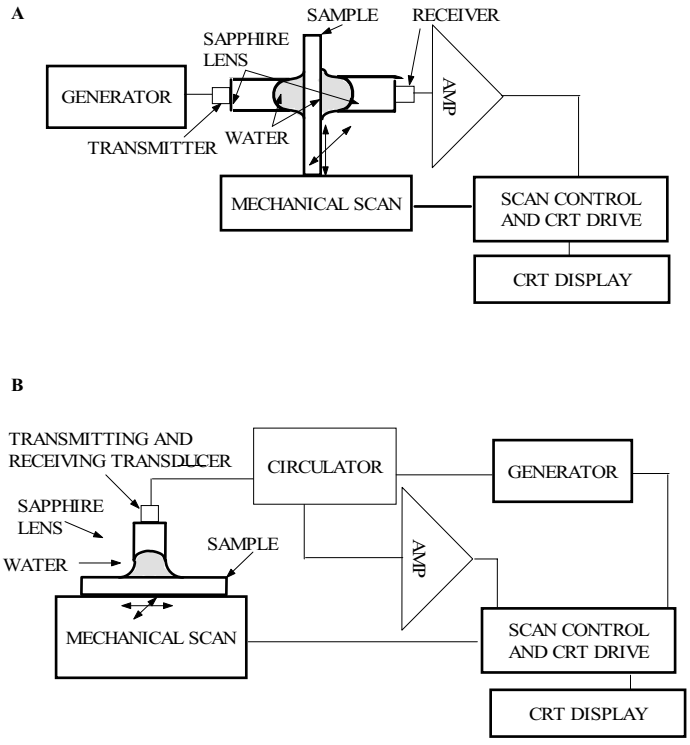

Fig. 8. Schematic diagrams of scanning acoustic microscopes in transmission (A) and pulse (B) modes

3. Selected Applications of Ultrasound

Examples of application of ultrasonic methods to study properties of complex materials will be discussed paying attention mostly to the qualitative results. Details of experimental systems, applied measurement techniques and models used are not discussed in details.

3.1. Studies of Dispersion and Frequency Dependence of Attenuation of Ultrasonic Waves in Saturated Porous Materials

Due to continuity of fluid and solid phases in saturated porous materials with open porosity the materials may transmit additional wave modes as compared with usual – impermeable solid materials, see e.g. [4]; [15] . In isotropic case three bulk waves: two longitudinal waves (called the fast and slow ones) and one shear wave can propagate in fully saturated materials. The schematic view of an experimental configuration that enables the observation of the waves (immersion method with rotated sample) on the example of the three pulses recorded for water saturated sintered porous glass with average diameters of glass beads of 80 μm are shown in *Fig. 9*, see e.g. [30] and [23].

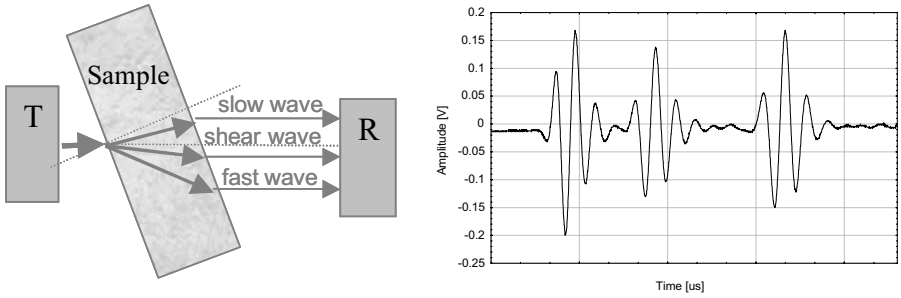

Fig. 9. Immersion method and pulses of fast, shear, and slow waves (the order corresponds to wave velocities) recorded for water saturated porous glass

The dependence of phase velocity and attenuation coefficient on frequency for each wave mode is determined using pulses transmitted through two samples of different thickness and application of spectral analysis (broad band ultrasonic technique). The results for water saturated glass with an average diameter of glass beads equal to 80 μm are given in *Fig. 10*.

The slow wave exhibits the highest attenuation and the strongest dependence on frequency. Such behavior is in agreement with macroscopic Biot's theory of wave propagation in saturated porous materials, which assumes that viscous dissipation due to interaction of phases is the dominant attenuation mechanism and

neglects scattering of waves on inhomogeneous elements of microstructure. When the wavelength, which is equal to the ratio of wave velocity and frequency, becomes comparable to the size of micro-inhomogeneities (e.g. pores or grains) the role of scattering becomes significant both for velocity and attenuation and Biot's model is no longer appropriate to describe wave parameters. The examples of propagation parameters obtained for the two longitudinal waves in water saturated sintered glass beads of average diameter equal to 275 μm are given in *Fig. 11*.

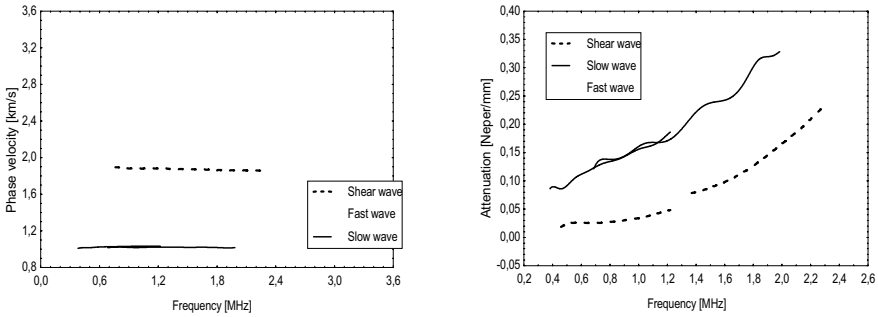

Fig. 10. Frequency dependence of phase velocities and attenuation coefficients for the three bulk waves in water saturated sintered glass beads of average diameter 80 μm

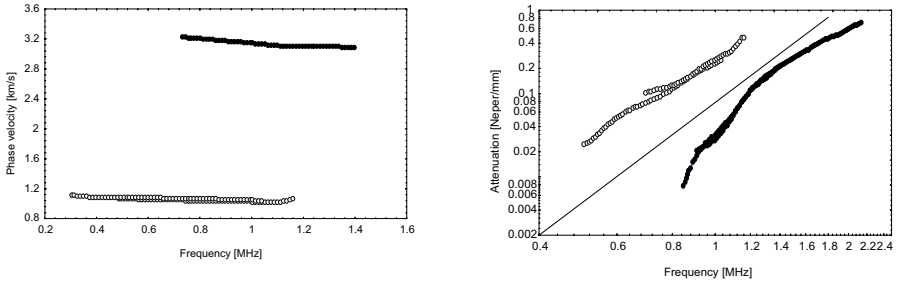

Fig. 11. Frequency dependence of phase velocities and attenuation coefficients for the longitudinal waves in water saturated sintered glass beads of average diameter 275 μm

The observed decrease of phase velocity with frequency (negative dispersion) and power law type dependence of attenuation on frequency are characteristic for Rayleigh scattering range of wave propagation. An extension of the Biot's model is necessary to describe the behavior of scattered waves, see [17].

3.2. Determination of Micro- and Macro-Parameters of Pore Structure

Internal structure of inhomogeneous materials is described by structural parameters referred to as microscopic parameters such as characteristic size of inhomogeneities or distance between inhomogeneities and macroscopic parameters the examples of which are volume or mass fractions of phases. In porous materials the important microscopic parameters are characteristic size of pores and single elements of skeleton (grains, fibers, etc.). The fundamental macroparameters are porosity, permeability and tortuosity, which are in the Biot's model. Since the structural properties play important role in the physical behavior of porous materials and influence mechanical and transport characteristics of the materials (e.g. permeability is the key parameter for advective flow, while tortuosity determines diffusive and ionic transport in pore fluid) the identification of the parameters is extremely important from the point of view of modeling and application of porous materials. While the standard methods of determination of structural characteristics are mostly based on different microscopy techniques the ultrasonic methods can also be useful, see e.g. [22] and [2][16] [37]. Taking into account the fact that scatterings of waves appears when the wavelength becomes comparable with the size of inhomogeneity and different type of scattering can be distinguished depending on the ratio of the two quantities, the analysis of attenuation and dispersion can lead to evaluation of average size of pores or grains. In *Fig. 12* the propagation characteristics for the fast wave are presented for sintered glass with average diameter of beads of 550 μm showing a transition from the Rayleigh scattering (negative dispersion and attenuation proportional approximately to frequency up to power four) to stochastic scattering (positive dispersion and attenuation proportional to square of frequency). The correlation between wavelength corresponding to the bounds of the particular scattering region and size of inhomogeneities can serve as the basis for determination of characteristic size of micro-inhomogeneities.

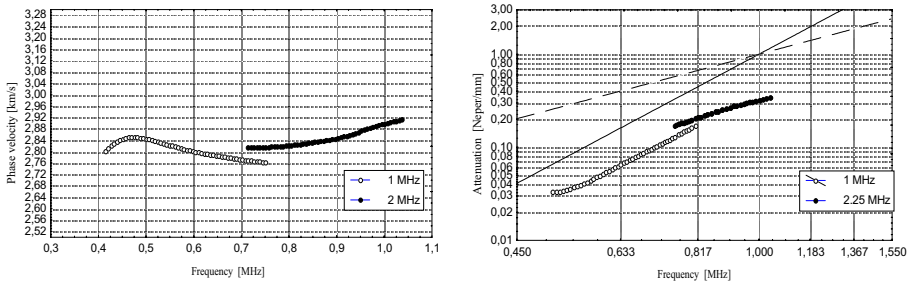

Fig. 12. Dispersion and frequency dependence of attenuation for sintered glass beads of diameter of 550 μm

Determination of macro-structural parameters of porous materials through ultrasonic techniques requires the application of a model including the searched

parameters. An example of such procedure which applies ultrasonic data from non-scattering region and can be used to find permeability and tortuosity from wave parameters determined by broad band spectroscopy is shown in *Fig.13*.

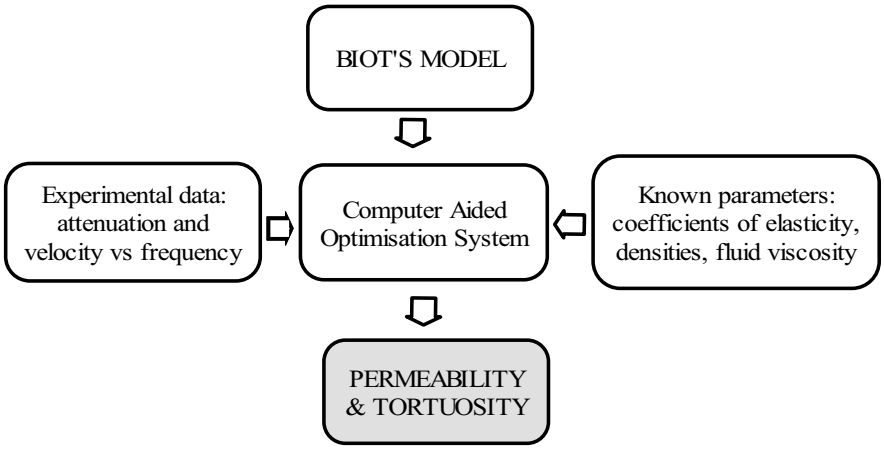

Fig. 13. Diagram of the optimization procedure used for determination of structural parameters

The experimental results supplemented by known material parameters (in this case coefficients of elasticity, porosity, densities and fluid viscosity) with Biot's model allow to formulate the numerical optimization problem with an objective function which depends on the differences between theoretical and measured values of wave propagation parameters (attenuation and velocity) determined in a set of frequency points. The optimization problem should also incorporate the constraints, which in considered case require that permeability must be positive and tortuosity is not less than 1.

3.3. Evaluation of Quality of Tissues (Bones)

Noninvasive low energy ultrasound is not only widely used in medical imaging but also in determination of the quality of tissues, for example bone tissues and then in diagnosis of bone disorder related to ageing or diseases (e.g. osteoporosis). One of the standard methods in this field called quantitative ultrasound is based on determination of wave velocities through bones (mostly in heels) and on value of attenuation or increase of attenuation in a particular frequency range (called broad band attenuation), see e.g. [9]; [26]; [7] . The propagation parameters are correlated with density of bones determined by X-ray methods and other techniques. Despite the above mentioned acknowledged application of ultrasonic technique intensive

studies of bones are led mostly, *in vitro*, to extend the range of predictions of quality of bones from ultrasound. Particular attention is devoted to the possibility of determination of changes in structure of trabecular bones related to osteoporosis. One promising direction of such efforts is associated with studies at higher frequencies of ultrasound, ([26] and [19]). In *Fig. 14* the examples of ultrasonic signals transmitted through a sample of marrow saturated bovine trabecular bone studied by the transducers with resonance frequencies equal to 0.5, 1 and 2.25 MHz are shown.

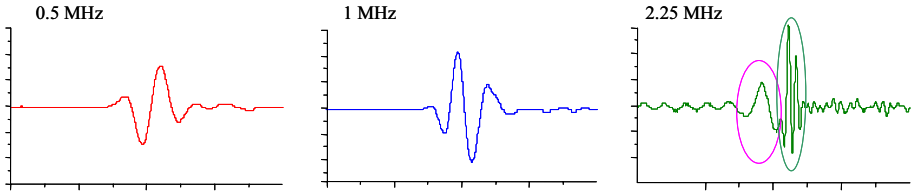

Fig. 14. Pulse waves of different frequencies (0.5, 1, 2.25 MHz) recorded as transmitted through marrow saturated trabecular bone

The signals received from the transducers of 0.5 and 1 MHz contain single wave modes, i.e. the fast wave. In turn the signal from 2.25 MHz transducers is composed of two pulses: a lower amplitude pulse of fast wave and a higher amplitude pulse of slow wave mode. It can also be noticed that the period of fast wave is significantly longer than the period of slow wave. The obtained results prove that studies of bones at higher frequency can supply additional data as compared with standard studies at frequencies below 1 MHz. Since the slow wave is essentially influenced by the structure of porous materials the properties of the wave could be particularly useful as predictors of structural changes in bones.

3.4. Study of Magnetically Sensitive Materials

An example of application of ultrasound to study the coupled effect of external fields on mechanical properties of materials are the results obtained for magnetically sensitive materials such as ferrofluids and ferrogels, see e.g. [29]; [40]; [18]; [36]. Ferrofluids are colloidal suspensions of magnetic particles in a carrying fluid such as water, kerosene or oils. The typical diameter of magnetic particles varies from 5 to 10 nm and the surface of the particles is covered with a surfactant in order to prevent their coagulation. When a magnetic fluid is exposed to constant magnetic field certain amount of particles gather into clusters. In turn clusters interact with each other constituting chains. The evolved microstructure forms a soft skeleton that can carry some mechanical load and influences the effective stiffness of the material. This process is clearly confirmed in studies with application of wave

propagation, where attenuation and phase velocity change due to the variation of strength of magnetic field, see *Fig. 15*.

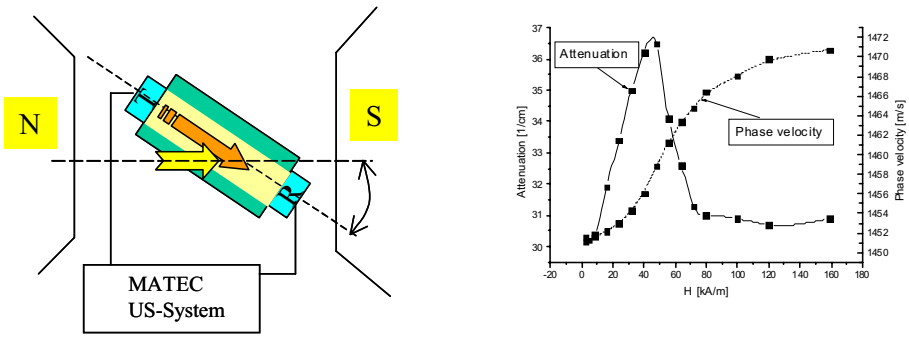

Fig. 15. Experimental configuration and dependence of attenuation and phase velocity of ultrasonic waves in ferrofluid on strength of magnetic field; $\theta = 0$, frequency was 1.18 MHz.

With the increase of magnetic field the wave velocity increases showing the effect of saturation. For weaker magnetic fields the attenuation increases proportionally to the strength of magnetic field. The behavior can be related to the growing number of clusters interacting through viscous forces with the surrounding liquid. Then because the relative motion of clusters in liquid is limited by mutual magnetic interactions among clusters, the attenuation decreases. Such scenario can also be predicted by the two-phase model of magnetic fluid with the postulated evolution of stiffness of skeleton, see [18]. The predictions of the model for wave parameters showing qualitative agreement with experimental results are shown in *Fig.16*.

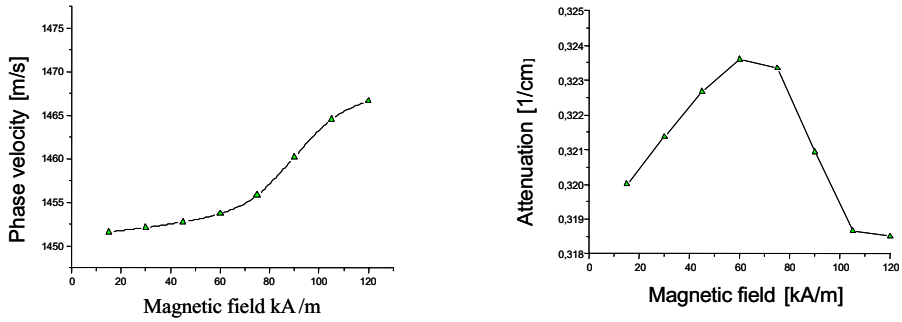

Fig. 16. Predictions of two phase model of magnetic fluid

4. Conclusions

The presented discussion of ultrasonic methods is not complete since it neither includes specific techniques of image analysis nor other methods which could be distinguished from the point of view of methods of signal generation, its reception or applied signal analysis. The usefulness of ultrasonic methods to study materials, their mechanical and structural parameters and the influence of external loads on some material properties were illustrated presenting a few examples. The capabilities of the methods, however, are much broader including advanced applications as determination of size and size distributions of particles or clusters in suspension, diagnosis of progress of chemically driven degradation of materials such as concrete and polymers and evaluation of influence of external conditions (e.g. temperature or electric field) on mechanical properties of materials.

In all cases depending on the type of materials and involved fields the appropriate models must be applied, incorporating evolution laws for internal interactions and form of influence of external fields on the material properties.

Appendix

Remarks to Modeling Approaches Used in Description of Waves in Complex Materials

The necessary components of identification procedures of complex material properties are models incorporating the studied effects, for example constitution, internal structure, interactions between phases and/or influence of external fields on the materials. If the considered materials are made of a single phase or from number of phases having macroscopically the same kinematics (there is no relative movement among them), as in the case of solid composites, the models can be formulated starting with the balance of mass and linear momentum for the single material continuum ([11]):

$$\frac{\partial}{\partial t}\rho + \operatorname{div}(\rho \mathbf{v}) = 0,$$

$$\frac{\partial}{\partial t}(\rho \mathbf{v}) + \operatorname{div}(\rho \mathbf{v}\mathbf{v}) - \operatorname{div}\mathbf{T} - \rho \mathbf{b} = 0,$$

where ρ, $\mathbf{v}$, $\mathbf{T}$ and $\rho\mathbf{b}$ denote mass density, velocity vector, stress tensor and body force, respectively. Specific properties of single phase materials are in most cases expressed by a form of proposed constitutive function for stress tensor. In particular, the dissipation and scattering effects which result from internal friction or energy loss due to reflections on micro-inhomogeneities of complex materials are determined through different types of viscoelastic models, being generalizations of Hooke's model of elastic materials. In the case of harmonic excitations or equations transformed into frequency domain the viscoelastic models are expressed by

equation

$$\mathbf{T} = \mathbf{A}\boldsymbol{\varepsilon},$$

where the stress tensor $\mathbf{T}$ is related to the strain tensor $\boldsymbol{\varepsilon}$ and $\mathbf{A}$ is a constant or a frequency dependent tensor of material coefficients represented by complex numbers. While considering wave propagation the real and imaginary components of $\mathbf{A}$ are related to transmission and loss of energy of the waves, respectively, see e.g. [43] and [21]; . The extensions into frequency dependent form of the moduli are applied to describe dispersive properties of waves due to complex internal friction or scattering mechanisms ([3]).

In order to develop a model for multiphase materials when independent kinematics plays a role the concept of superimposed continua must be used with separated balances of mass and linear momentum for each phase, as in the following forms ([6]):

$$\frac{\partial}{\partial t}\rho_i + \operatorname{div}(\rho_i \mathbf{v}_i) = \dot{m}_i,$$

$$\frac{\partial}{\partial t}(\rho_i \mathbf{v}_i) + \operatorname{div}(\rho_i \mathbf{v}_i \mathbf{v}_i) - \operatorname{div}\mathbf{T}_i - \rho_i \mathbf{b}_i = \mathbf{R}_i.$$

The equations contain mass densities ρ_i, velocity vectors $\mathbf{v}_i$, stress tensors $\mathbf{T}_i$, body forces $\rho_0 \mathbf{b}_i$, intensities of mass m_i and momentum $\mathbf{R}_i$ exchange between phases. When a transport process takes place through the described material one also needs to consider balances of mass for components of phases. For consideration of influence of electric or magnetic fields on the material behavior, equations governing the additional fields and coupled effects in the mechanical model should be added. In some cases the rotational degrees of freedom and balance of linear momentum must also be considered. In dynamical processes the mass exchange between phases is usually not important ($m_i = 0$). In general, in order to close the system of equations and express the interactions between phases as well as the external fields and materials, and to describe the evolution of properties of materials a set of equations for m_i, $\mathbf{T}_i$ and $\mathbf{R}_i$ is required specifically constituted for given materials and existing loads. If, as an example, one considers the widely applied model of fluid saturated porous materials the basic set of constitutive functions in isotropic and elastic cases has the form ([4]):

$$\begin{aligned}
\mathbf{T}_s &= 2N\boldsymbol{\varepsilon}_s + (Atr\boldsymbol{\varepsilon}_s + Qtr\boldsymbol{\varepsilon}_f)\mathbf{I},\\
\mathbf{T}_f &= (Qtr\boldsymbol{\varepsilon}_s + Rtr\boldsymbol{\varepsilon}_f)\mathbf{I},\\
R_s &= -R_f = \mathbf{c}(\mathbf{v}_f - \mathbf{v}_s) + \mathbf{d}(\dot{\mathbf{v}}_f - \dot{\mathbf{v}}_s),
\end{aligned}$$

where indexes s and f refer to solid and fluid phase, N, A, Q, and R stand for elastic parameters of the system, and coefficients c and d are responsible for viscous and dynamic interaction between phases and are dependent on parameters describing the internal structure of the materials such as porosity, tortuosity, and permeability. It is now evident that the range of applicability of the above model is much broader than modeling saturated porous materials and can be used for example for liquid suspensions (e.g. [27]), structured liquids (e.g. [18]) and gels (e.g. [41]; [14]).

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