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THE INFLUENCE OF A PRESSURE MEDIUM ON THE ACCURACY OF MEASUREMENTS IN ULTRASONIC INVESTIGATIONS OF SOLIDS

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The effects related to leaking of an ultrasonic wave from a solid sample through its end faces to a pressure medium were analysed in this paper. The influence of these effects on the results of measurements of the attenuation and the velocity of ultrasonic waves was experimentally investigated for several liquid and gaseous pressure media. The measurements were performed at a wave frequency of 10 MHz and in a pressure range up to 1 GPa.

1. Introduction

The pulse method of measuring the velocity and attenuation of ultrasound in a solid sample placed together with an ultrasonic transducer in a pressure medium has been applied by many investigators. The results of such measurements are influenced by pressure fluctuations of the acoustic properties of the medium, a layer coupling the transducer with the sample and the transducer itself. The geometry of the measuring system also changes during the experiment. The thicknesses of the sample, the transducer and the bound layer are reduced. The analysis of the effect of hydrostatic pressure on an ultrasonic transducer has already been presented in the literature [1]. Owing to this analysis, the influence of pressure phenomena related to the latter on the accuracy of measurements can be assessed. An increase of hydrostatic pressure increases the resonance frequency of an ultrasonic transducer. This results from the reduction in the thickness of the transducer and from the increase of the velocity of ultrasound propagating in it. The relative increment of the resonance frequency per unit of the hydrostatic pressure increment for a quartz X-cut transducer was equal 1.46×10^{-5} MPa^{-1}. VARIUKHIN et al. [2] has investigated experimentally several pressure media in order to obtain the maximal value of the reflection coefficient of an ultrasonic wave from the sample-pressure medium interface. Their differential measurements performed on samples of various lengths revealed that a change in the attenuation of ultrasound in the material of the sample due to the increase in hydrostatic pressure may be tens of times weaker than the change due to leaking of ultrasound into the pressure medium. Also the changes in the phase of the wave reflected from the sample-ultrasonic transducer interface was taken into account in accurate measurements of the velocity of ultrasonic waves [1]. However, the above observations applied only to the case of an ultrasonic transducer having one free surface, i.e. radiating only in one direction, to the sample. The value of the correction of the measured travel time of the wave due to the above mentioned effect can approach one half of its period. This means that the lower the wave frequency is, the greater can be the error made in the measurement of the time of travel of an ultrasonic signal through the sample. Since the acoustic impedance of the pressure medium changes together with hydrostatic pressure, the conditions of leaking of the wave through the end faces of the sample into the pressure medium also have to change. The aim of this paper is to analyse the influence of the pressure medium and the layer of coupling material on the results of measurements of the attenuation coefficient and the velocity of ultrasonic waves.

2. Theoretical

Let us consider a measurement system consisting of an ultrasonic transducer of thickness b and acoustic impedance Z_T, attached to a solid sample having flat and parallel end faces by means of a layer of a bonding material of thickness a, and acoustic impedance Z_B. The material of the sample is isotropic of acoustic impedance Z_s. A free surface of the sample and a free surface of the ultrasonic transducer adjoin the pressure medium of acoustic impedance Z_M. The case is illustrated in Fig. 1.

The method which is most frequently applied for measuring the travel time of the ultrasonic wave through the sample has been proposed by Papadakis [3]. It is based on the choice of a release frequency of an oscilloscope presenting successive echoes reflected from the back side of the sample, so that the time between a pair of echoes chosen for measurements is equal to the period of pulses releasing the time base of the oscilloscope. In such a case a superposition of ultrasonic pulses occurs on the oscilloscope screen. For two following echoes the travel time T, registered in the experiment, can be expressed as a sum of three components:

$$T = \delta + n/f - \varphi/2\pi f \tag{1}$$

where δ is the actual travel time of the wave, n — an integer expressing the possibility of an error in the time measurement by the multiple of the wave period, φ — the sum

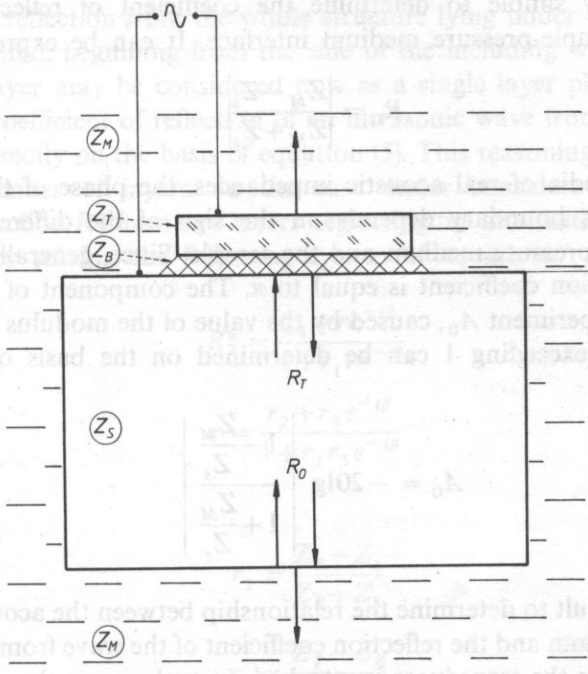

FIG. 1. Geometry of acoustical system. Z_s – acoustic impedance of sample, Z_B – acoustical impedance of bond, Z_T – acoustic impedance of transducer, Z_M – acoustic impedance of pressure medium, a – bond thickness, b – transducer thickness

of reduced (without π) phases of the coefficients of reflection on both sides of the sample, and f is the frequency of the wave. With this method the travel time of ultrasound through the sample can be measured with the accuracy better than 0.1 ns.

The attenuation of ultrasound A, measured on the path equal to the double length of the sample can be expressed in decibels as follows:

$$A = -20\lg(R_0 R_T e^{-2\gamma l}) \tag{2}$$

where l is the length of the sample, γ — the coefficient of attenuation of the wave in the material of the sample, and R_0, R_T — the moduli of the reflection coefficient of the wave from both end faces of the sample, on the side of the pressure medium and the side of the transducer, respectively.

The above mentioned relationships do not take into account the diffraction effects which occur when the wave propagates through the sample [3÷5].

Both coefficients of reflection are responsible for the leakage of ultrasound from the sample to the pressure medium, hence increasing the registered attenuation. As the pressure can influence the value of these coefficients, it is not possible to assess and investigate in direct observation the effect produced by hydrostatic pressure only on the sample subjected to it. Because of this, it is important to know the effect of parameters R_0 and R_T on the quantities observed in experiments.

It is relatively simple to determine the coefficient of reflection R_0 of the wave from the sample-pressure medium interface. It can be expressed as follows

$$R_0 = \left|\frac{Z_M - Z_s}{Z_M + Z_s}\right| \tag{3}$$

In cases of media of real acoustic impedances, the phase of the coefficient of reflection from this boundary depends on the sign of the difference of acoustic impedances of the pressure medium and the sample. Since, generally, $Z_M < Z_s$, the phase of the reflection coefficient is equal to π. The component of the attenuation registered in the experiment A_0, caused by the value of the modulus of the reflection coefficient R_0 not exceeding 1 can be determined on the basis of relation:

$$A_0 = -20\lg\left|\frac{1 - \frac{Z_M}{Z_s}}{1 + \frac{Z_M}{Z_s}}\right| \tag{4}$$

It is more difficult to determine the relationship between the acoustic impedance of the pressure medium and the reflection coefficient of the wave from the second face of the sample, where the transducer is attached. In such a case the wave is reflected from the parallel layers places between two elastic halfspaces. This problem may be solved with the method well-known from the literature, developed mainly for the needs of optics [6]. It requires, however, an inconvenient calculation multiplication of matrices, solution of a system of equations. Ready formulae can be found too, (e.g. [7]). Applying the known dependence of the reflection coefficient of an ultrasonic wave from a single layer of impedance Z_2 and of thickness x, placed between two elastic half-spaces of acoustic impedances Z_1 and Z_3 [8] we obtain

$$R^* = \frac{r_{12} + r_{23}e^{-i2xk}}{1 - r_{21}r_{23}e^{-i2xk}} \tag{5}$$

where

$$r_{ij} = \frac{Z_j - Z_i}{Z_j + Z_i} \tag{6}$$

and k is the wave number in the layer.

Generally, the effects due to the attenuation in the layer and the halfspaces surrounding it can be taken into consideration by applying the complex forms of the wave impedances of media and the wave number. Considering a system of two parallel layers between elastic halfspaces, as it is shown in Fig. 1, it is possible to express the reflection coefficient of the wave from these layers by an expression similar to the one obtained for a single layer. The reflection coefficient from the first layer-second layer interface (denoted by symbol r_{23} in eq. (5)) must be replaced with the coefficient of reflection from the whole structure lying under the first layer. The layers are numbered, beginning from the side of the including wave. The structure under the first layer may be considered now as a single layer placed between two halfspaces. The coefficient of reflection of an ultrasonic wave from such a layer can be determined directly on the basis of equation (5). This reasoning can be applied to an arbitrary number of layers. In the case under consideration, the pressure coefficient of reflection R_T^* from two layers neglecting the attenuation of the wave can be determined by solving the following system of equations:

$$R_T^* = \frac{r_1 + re^{-i\vartheta}}{1 + r_1 re^{-i\vartheta}} \tag{7}$$

$$r = \frac{r_2 + r_3 e^{-i\beta}}{1 + r_2 r_3 e^{-i\beta}} \tag{8}$$

where

$$r_1 = \frac{Z_B - Z_s}{Z_B + Z_s} \tag{9}$$

$$r_2 = \frac{Z_T - Z_B}{Z_T + Z_B} \tag{10}$$

$$r_3 = \frac{Z_M - Z_T}{Z_M + Z_T} \tag{11}$$

$$\vartheta = 2ak_B \tag{12}$$

$$\beta = 2bk_T \tag{13}$$

while k_B and k_T denote wave numbers in the coupling layer and in the transducer, respectively. The solution of the above equations results in the following expressions for the modulus R_T and the phase Φ of the reflection coefficient that was searched for:

$$R_T = \left[1 - \frac{1 - r_1^2 r_2^2 r_3^2 - r_1^2(1 - r_3^2) - r_2^2(1 - r_1^2) - r_3^2(1 - r_2^2)}{1 + r_2^2 r_3^2 + r_1^2 r_2^2 + r_1^2 r_3^2 + 2r_2[r_1(1 + r_3^2)\cos(\vartheta) + r_3(1 + r_1^2)\cos(\beta)] + 2r_1 r_3[\cos(\vartheta + \beta) + r_2^2\cos(\vartheta - \beta)]}\right]^{1/2} \tag{14}$$

$$\mathrm{tg}(\Phi) = \left[(r_1^2 - 1)\frac{r_2(1 + r_3^2)\sin(\vartheta) + r_3[\sin(\vartheta + \beta) + r_2^2\sin(\vartheta - \beta)]}{r_1(1 + r_2^2)(1 + r_3^2) + 4r_1 r_2 r_3\cos(\beta) + r_2(1 + r_1^2)(1 + r_3^2)\cos(\vartheta) + r_3(1 + r_1^2)[\cos(\vartheta + \beta) + r_2^2\cos(\vartheta - \beta)]}\right]. \tag{15}$$

When the transducer works at its resonance frequency which is the most frequent case, expressions (14) and (15) are simplified because in this case:

$$\cos\beta = 1 \tag{16}$$

$$\sin(\beta) = 0 \tag{17}$$

This leads to the relation:

$$R_T = \left[1 - \frac{1 - r_1^2 r_2^2 r_3^2 - r_1^2(1-r_3^2) - r_2^2(1-r_1^2) - r_3^2(1-r_2^2)}{1 + r_1^2 r_2^2 + r_1^2 r_3^2 + r_2^2 r_3^2 + 2r_2 r_3(1+r_1^2) + 2r_1(r_2+r_3)(1+r_2 r_3)\cos(\vartheta)}\right]^{1/2} \tag{18}$$

$$\operatorname{tg}(\Phi) = \left[\frac{(1+r_2 r_3)(r_2+r_3)(r_1^2-1)\sin(\vartheta)}{r_1(r_2^2+1)(r_3^2+1) + 4r_1 r_2 r_3 + (r_2+r_3)(1+r_1^2)(1+r_2 r_3)\cos(\vartheta)}\right]. \tag{19}$$

If the real part of the reflection coefficient R_T^* is negative, then $\varphi = \operatorname{arctg}(\Phi)$.

The modulus of the reflection coefficient influences the registered value of the attenuation of ultrasound in the investigated material. In the case of measurements applying two successive echoes, the attenuation measured on a path equal to the double length of the sample is increased by term A_T, which can be expressed by

$$A_T = -20\lg(R_T). \tag{20}$$

This parameter versus the ratio of the impedance of the pressure medium to the impedance of investigated sample is presented in Fig. 2. The dependence is determined for several values of the parameter ϑ. This parameter characterizes elastic properties of the coupling layer, as well as its thickness, because it defines the ratio of the thickness of the layer to the length of the wave propagating in it. After suitable transformations it can be proved that for $\vartheta = 0$ the coefficient A_T in terms of the impedance of the pressure medium is identical to the one obtained from formula (4), which describes the relationship between the attenuation component A_0 in the relative value of the pressure medium acoustic impedance. In this case the moduli of coefficients of reflection remain equal to each other in spite of the fact that one side of the sample adjoins the transducer, while the other side directly adjoins the pressure medium. This effect occurs, because the transducer (considered here as a parallel layer) is transparent for the wave at its resonance frequency, provided we neglect attenuation of the ultrasonic wave in the material of the transducer. For greater values of the parameter ϑ the A_T dependence on Z_M/Z_s practically coincides with this dependence for $\vartheta = 0$ and small values of Z_M/Z_s. When the ratio Z_M/Z_s is increased, the curves become divergent and the greater is the value of the parameter ϑ, the earlier this occurs.

The character of this dependence is somehow different for even greater values of the parameter ϑ. In this case a rapid increase of the attenuation accompanying an increase of impedance of the pressure medium for small Z_M/Z_s is observed, and then, after the maximum is exceeded, a decrease to zero for greater values of Z_M/Z_s occurs. A high ϑ can correspond to a thicker coupling layer. The analysis of expression (18)

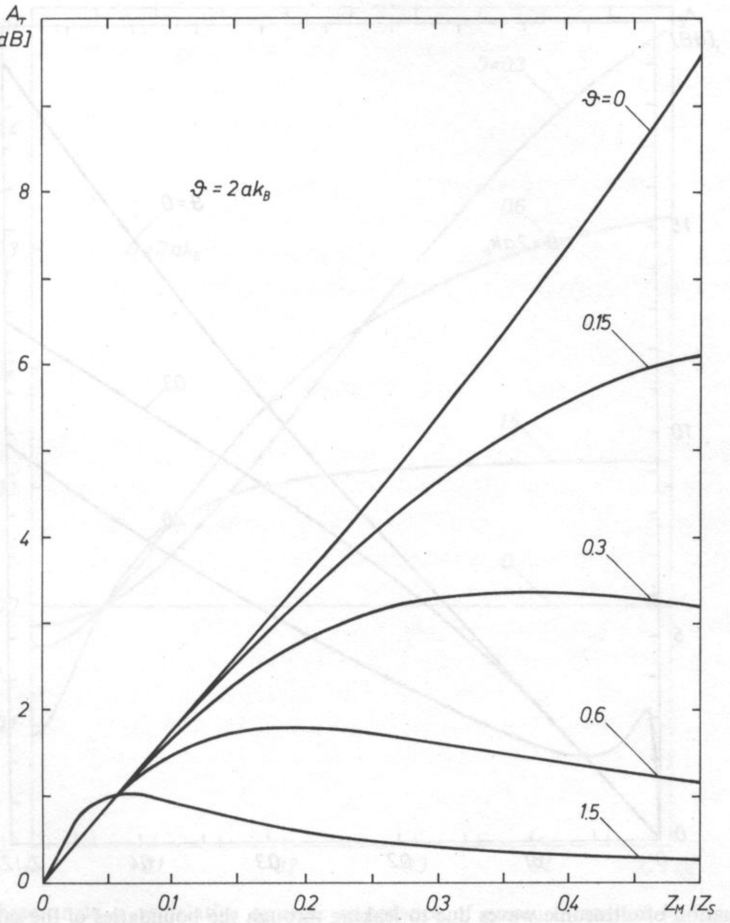

FIG. 2. Attenuation of ultrasonic waves due to leaking through the sample-transducer boundary vs relative acoustic impedance of pressure medium $A_T = 20\lg(R_T)$

results in a conclusion that when the impedance of the pressure medium is equal to the acoustic impedance of the coupling layer $Z_M = Z_B$, then the value of the attenuation component A_T is independent of the value of the ϑ parameter and, hence, of the thickness of the coupling layer. It is equal to $A_T = -20\lg|r_1|$. Figure 3 illustrates the total effect of the reflection coefficient at both sides of the sample. It presents the attenuation component A_E, due to leaking of ultrasound into the pressure medium, which is a sum of parameters A_0 and A_T.

$$A_E = A_0 + A_T \tag{21}$$

in terms of the ratio of the impedance of the pressure medium to the impedance of the sample. It can be seen in this figure that the parameter ϑ, which characterizes the coupling layer, has a considerable influence on this dependence. As for A_T in Fig. 2,

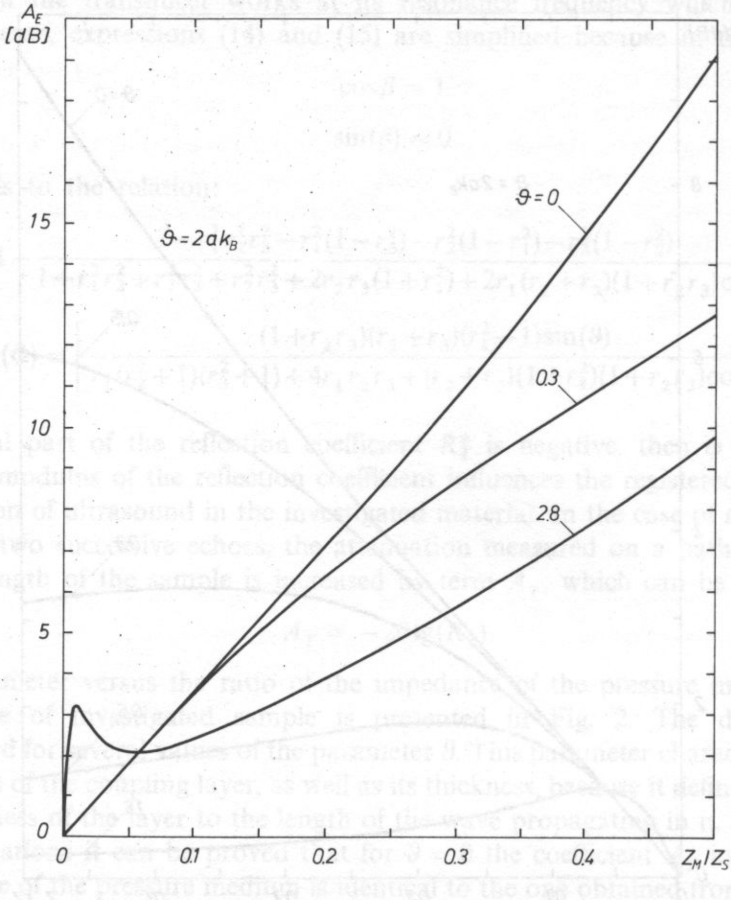

FIG. 3. Attenuation of ultrasonic waves due to leaking through the boundaries of the sample vs relative acoustic impedance of pressure medium $A_E = 20\lg(R_0 R_T)$

the attenuation component A_E is monotonic for small values of the parameter ϑ. A_E increases when the impedance of the pressure medium is increased, whereas for higher values of the parameter ϑ a very rapid increase of the attenuation of ultrasound due to its leaking to pressure medium is observed. In this case the curve reaches a maximum, then its values decrease and futher on then increase monotonically for higher values of the argument Z_M/Z_s when the curve asymptotically approaches the value of component A_0.

The influence of an increase of the acoustic impedance of the pressure medium on the phase of the reflected wave is shown in Fig. 4. It can be seen that if the transducer is placed directly on the sample, the phase of the reflected wave does not depend on the impedance of the pressure medium. In all other cases the phase increases when the parameter Z_M/Z_s increases. For $Z_M = 0$ the parameter φ assumes

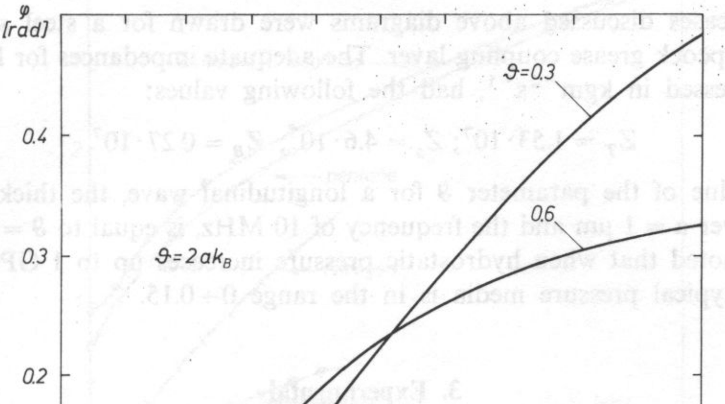

FIG. 4. Phase angle φ vs relative acoustic impedance of pressure medium ($\Phi = \varphi + \pi$)

the following value

$$\varphi = \operatorname{arctg}\frac{(1-r_1^2)\sin(\vartheta)}{2r_1-(1+r_1^2)\cos(\vartheta)}. \tag{22}$$

Hence, if $\cos(\vartheta) = 2r_1/(1+r_1^2)$, then $|\varphi| = \pi/2$. For small Z_M/Z_s values the higher is the value of the coefficient ϑ, the greater becomes the rate of these changes. When $Z_M = Z_B$, the phase of the reflected wave does not depend on the thickness of the coupling layer, which was the case for A_T and A_E, while for the sample exceeding Z_B/Z_s for very small values of ϑ, the phase changes are very small. For greater values they rapidly increase, achieve maximum and then decrease. When is equal to about $\pi/2$, the phase for increasing values of the impedance of the pressure medium practically remains unchanged. Because of the periodical character of described functions, definitions concerning the parameter ϑ refer to the first period only.

In all cases discussed above diagrams were drawn for a steel sample with a nonaq stopcock grease coupling layer. The adequate impedances for longitudinal waves, expressed in $kgm^{-2}s^{-1}$, had the following values:

$$Z_T = 1.53 \cdot 10^7; \; Z_s = 4.6 \cdot 10^7; \; Z_B = 0.27 \cdot 10^7$$

The value of the parameter ϑ for a longitudinal wave, the thickness of the coupling layer a = 1 μm and the frequency of 10 MHz, is equal to $\vartheta = 0.06$ rad. It should be noted that when hydrostatic pressure increases up to 1 GPa, the ratio Z_M/Z_s for typical pressure media is in the range $0 \div 0.15$.

3. Experimental

In order to verify the previously derived relationships in practice, the attenuation, as well as the travel time of ultrasonic pulses in a steel sample on a path equal to its double thickness, were measured. The measurements were made at the temperature of 300 K in a pressure vessel presented in Fig. 5. Acoustic waves were generated by circular ultrasonic transducer of 7 mm in diameter. They were made of X– and Y–cut quartz and fixed to the sample with nonaq stopcock grease. The thickness of the coupling layer did not exceed 1 μm. Transducers were activated with electric pulses of duration of approximately 1 μs and filling frequency of 10 MHz. The sample was cylindrical, 14 mm in diameter and 20 mm high. It was made of bearing

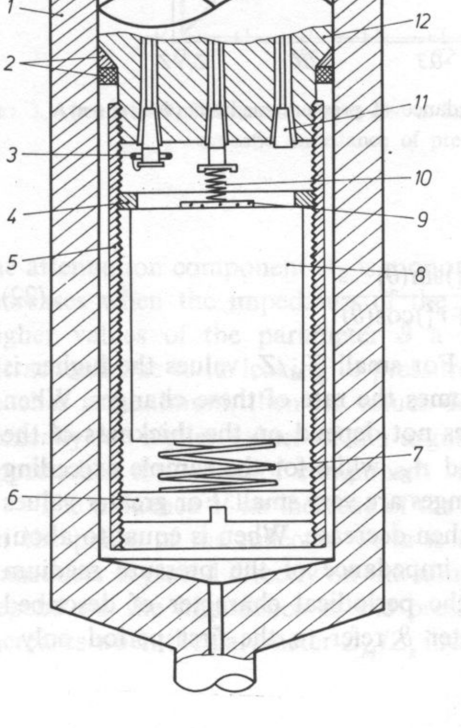

FIG. 5. Equipment used for ultrasonic investigation of solids under high hydrostatic pressure [9], *1* – pressure vessel, *2* – sealing, *3* – manganin gauge, *4* – screw ring, *5* – sample holder, *6* – screw, *7* – spring, *8* – sample, *9* – transducer, *10* – R. F. lead spring, *11* – electrical fit through, *12* – plug

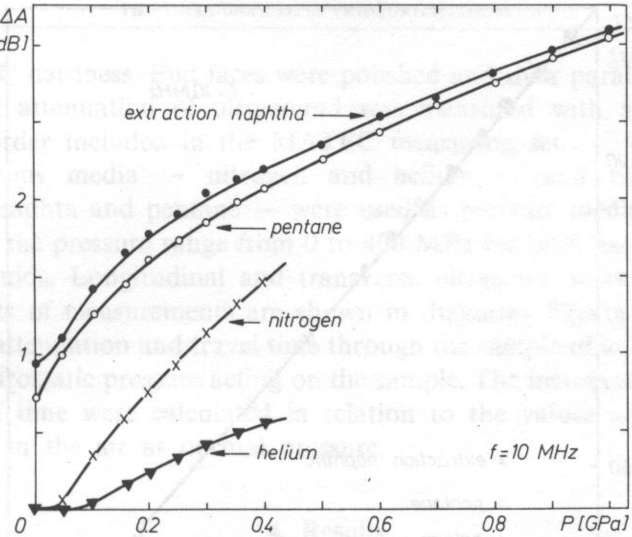

FIG. 6. Increment of attenuation of longitudinal ultrasonic wave vs hydrostatic pressure for various pressure media. ($\Delta A = A(P) - A_0$) A_0 is the attenuation measured in air at ambient pressure

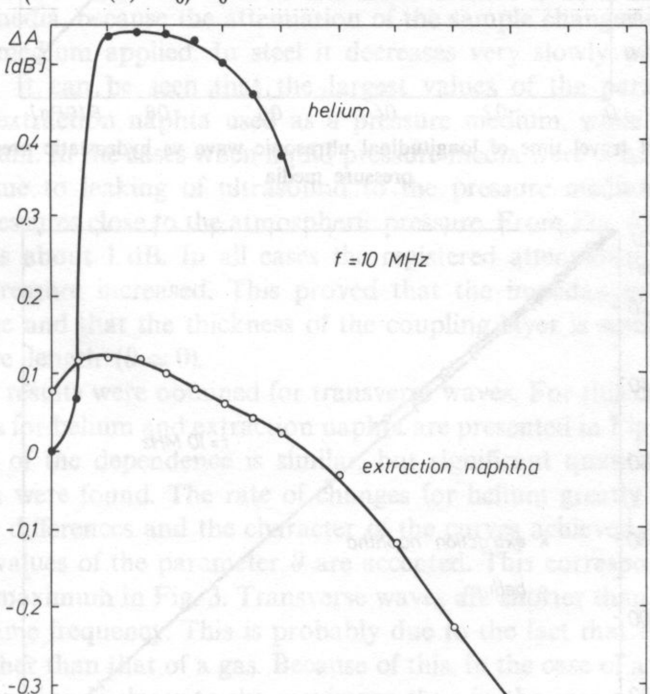

FIG. 7. Increment of attenuation of shear ultrasonic wave vs hydrostatic pressure for extraction naphta and gaseous helium

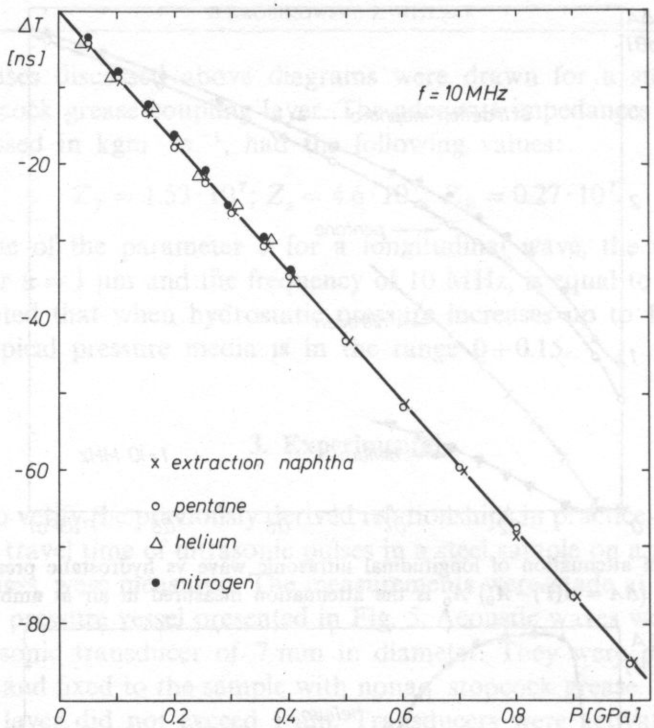

FIG. 8. Increment of travel time of longitudinal ultrasonic wave vs hydrostatic pressure for various pressure media

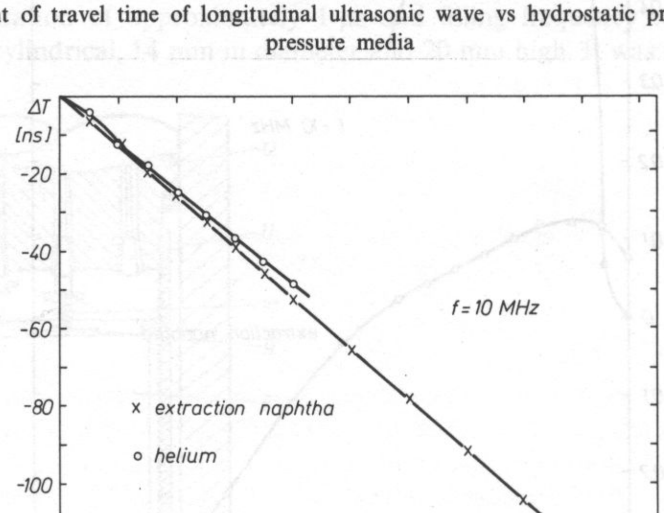

FIG. 9. Increment of travel time of shear ultrasonic wave vs hydrostatic pressure for extraction naphta and gaseous helium

steel of 62 HRC hardness. End faces were polished and their parallelism was better than 30″. The attenuation of ultrasound was measured with an automatic attenuation recorder included in the MATEC measuring set.

Two gaseous media – nitrogen and helium – and two liquid media – extraction naphta and pentane – were used as pressure media. Measurements were made for the pressure range from 0 to 400 MPa for both gases and from 0 to 1 GPa for liquids. Longitudinal and transverse ultrasonic wave were applied.

The results of measurements are shown in diagrams Figs. 6–9. They present increments of attenuation and travel time through the sample of an ultrasonic signal in terms of hydrostatic pressure acting on the sample. The increments of attenuation and the travel time were calculated in relation to the values achieved from the measurements in the air at normal pressure.

4. Results

All differences observed in Fig. 6 result from the difference of the attenuation of the pressure media, because the attenuation of the sample changes independently of the pressure medium applied. In steel it decreases very slowly when the pressure increases [2]. It can be seen that the largest values of the parameter ΔA were achieved for extraction naphta used as a pressure medium, while the lowest were found for helium. In the cases when liquid pressure media were used, an increment of attenuation due to leaking of ultrasound to the pressure medium, was observed already for pressures close to the atmospheric pressure. From Fig. 6 it results that the increment was about 1 dB. In all cases the registered attenuation increased when hydrostatic pressure increased. This proved that the impedances of the pressure media increase and that the thickness of the coupling layer is small in comparison with the wave length ($\vartheta \approx 0$).

Different results were obtained for transverse waves. For this case the results of measurements for helium and extraction naphta are presented in Fig. 7. In both cases the character of the dependence is similar, but significant quantitative differences between them were found. The rate of changes for helium greatly exceeds that for liquids. These differences and the character of the curves achieved can be explained when higher values of the parameter ϑ are accepted. This corresponds to the curve with a single maximum in Fig. 3. Transverse waves are shorter than the longitudinal ones of the same frequency. This is probably due to the fact that the impedance of a liquid is higher than that of a gas. Because of this, in the case of a liquid, the point under consideration is closer to the maximum than in the case of a gas. Therefore, the attenuation increments are smaller for a liquid than for a gas in a similar range of pressures.

It should be noticed that changes of elastic properties of the coupling layer, occurring when pressure in increased, may be of greater importance in case of transverse waves, than of the longitudinal ones, since the liquid viscosity increases with pressure and so does its capacity of transferring shear stresses.

The results of measurements of a pulse travel time through the sample are presented in Figs. 8 and 9. The travel time of ultrasonic pulses through the sample decreases when hydrostatic pressure increases. According to the expression (1), this is due to the changes of the velocity of ultrasonic waves, the length of the sample and the conditions of reflection of ultrasound at both faces of the sample in terms of pressure. As it can be seen in Fig. 8, all results for pentane, extraction naphta and both gases in the case of longitudinal waves are very similar. The maximal difference between them is equal to only 2 ns. From among the factors considered only the component resulting from the change of the phase of the ultrasonic waves, when they are reflected from the sample-ultrasonic transducer interface, can be the source of these differences, for the phases of reflected waves depend on the acoustic impedance of the pressure medium at a given pressure. As it was mentioned previously, the maximal correction to the time measurements due to this mechanism can be equal to a half period. This is 0.05 μs for the frequency of 10 MHz. In Fig. 4 it can be seen that the changes in the pressure medium impedance weakly influence the phase of the waves reflected from both faces of the sample for small values of the parameter ϑ. Hence, the same concerns the results of time measurements. The comparison of the results for transverse waves, shown in Fig. 9, indicates a qualitative compatibility with the results of theoretical calculations presented in Fig. 4. Due to the fact that the impedance of a liquid exceeds the impedance of a gas at a fixed value of pressure, the phase change of the wave reflected from the sample-ultrasonic transducer interface is greater for the liquid than for the gas at the same pressure. It also increases when the value of the parameter ϑ increases. This results from Eq. (1) showing that changes of the travel time are quicker for a pressure medium of higher acoustic impedance. The changes are quicker in the case of a liquid than that of a gas, as can be seen in Fig. 9.

5. Conclusions

– In investigations of longitudinal wave attenuation helium was found to be the least disturbing medium. In this case the registered increments of attenuation per unit of hydrostatic pressure increment were the smallest. The highest values of attenuation were registered for extraction naphta as a pressure medium.

– The influence of the pressure medium on the results of measurements of ultrasound velocity in megahertz frequency range seems to be very small and may be neglected in many cases (long samples, thin coupling layer, high frequency).

– The measurements made with the application of transverse waves (attenuation, in particular) seem to be more sensitive to impedance changes of the pressure medium. It is mainly the result of the higher value of the parameter ϑ in the case than in the case of longitudinal waves of the same frequency.

– The phase of the wave reflected from the sample-transducer interface is independent of the impedance of the pressure medium when the latter is equal to the acoustic impedance of the coupling material. Then the attenuation component due to leaking of the wave to the pressure medium is independent of the thickness of the coupling layer in this case. Therefore, the same liquid should be used as a pressure medium and the coupling material in accurate ultrasonic measurements.

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