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Influence of surface wettability on the accuracy of measurement of fluid shear modulus

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ABSTRACT

The shear moduli of liquids and their dependence on the degree of purity of the piezocrystal surface are measured by acoustic resonance method using a piezoelectric quartz resonator. As a criterion of surface condition the coefficient of static friction is accepted, which is measured by tribometer of Derjaguin–Lazarev design. The various methods of surface treatment which give a wide range of friction coefficients are used. At the maximum value of the coefficient of friction vibrating piezoquartz surface is completely wetted by the liquid and the effective value of the shear modulus of the liquid is the highest. © 2011 Elsevier B.V. All rights reserved.

1. Introduction

The most important structural characteristic of the liquid is a relaxation period τ – time of transition from nonequilibrium to equilibrium. In accordance to the theory of fluid by Frenkel et al. [1] the relaxation period is estimated on the basis of the rate of self-diffusion, by equating this period to time of the settled existence of individual molecules. Calculations give for low-viscosity liquids estimates of τ of the order 10^{-10} – 10^{-11} s. Estimates of the relaxation time in accordance to the nonequilibrium state of the Maxwell rheological model also gives the τ value of the order 10^{-10} s. Consequently, the dynamic shear elasticity of liquids could be detected in such periods of shear oscillations.

However, in [2,3] it was found by a resonance method using piezoelectric quartz that liquids possess shear elasticity at the frequencies of shear oscillations about 10^5 Hz regardless of viscosity and polarity. The formation of singular boundary layers of polar liquids on the lyophilic solid surfaces, with properties different from the properties of bulk liquid, was experimentally established in [4–7]. However, further investigations showed that the shear modulus is independent on the thickness within the range of thicknesses studied and the low-frequency shear elasticity is a property of a fluid in the bulk [8–11].

2. Theory

The essence of the acoustic resonance method used in this paper is as follows. The piezoquartz plate, vibrating at the main resonant frequency in its own plane, is loaded with a layer of test liquid, covered with a solid cover-plate. At the tangential displacement of piezoquartz plane a liquid layer will experience shear strain and damped shear waves should be established in the liquid layer. Piezoquartz used is of X-18.5° cut with a mass of 6.24g, the Poisson coefficient at the working surface is equal to zero. Resonance frequency is 74 kHz and plate dimensions are 34.5 mm \times 12 mm \times 5.5 mm.

The general formula expressing the complex resonance frequency shift Δf^* of the system piezoquartz – liquid layer – cover-plate has the following form [8]:

$$\Delta f^* = \frac{SG'k^*}{8\pi^2 M f_0} ctg\left(k^* H - \frac{\varphi^*}{2}\right),\tag{1}$$

where *S* is area of horizontal surface of piezoquartz, *G*^{*} complex shear modulus of the liquid, *k*^{*} the complex wave number, *M* mass of the piezoquartz, *f*₀ its resonance frequency, (the thickness of liquid layer, and φ^* complex phase shift upon reflection of the shear wave from the liquid – cover-plate border. Eq. (1) takes into account that the amplitude of the piezoquartz working surface oscillations varies in different places. The cover-plate weakly interacts with quartz and is almost at rest (φ^* = 0). Complex wave number corresponds to the formula $|k^*|^2 = \omega^2 \rho / |G^*|$. For the real and imaginary



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Fig. 1. Theoretical dependences of real $\Delta f'$ and imaginary $\Delta f'$ shifts of the resonance frequency versus the thickness of liquid interlayer.

parts one can obtain the following expressions:

$$\Delta f' = \frac{S\sqrt{G'\rho\cos(\theta/2)}}{4\pi M\sqrt{\cos\theta}} \cdot \frac{\sin 2\beta H - \tan(\theta/2) \operatorname{sh} 2\alpha H}{\operatorname{ch} 2\alpha H - \cos 2\beta H},\tag{2}$$

$$\Delta f' = \frac{S\sqrt{G'\rho}\cos(\theta/2)}{4\pi M\sqrt{\cos\theta}} \cdot \frac{\tan(\theta/2)\sin\beta H - \tan(\theta/2)\sin2\alpha H}{ch^2\alpha H - \cos2\beta H}, \quad (3)$$

where θ = arctan(G''/G') is the mechanical losses angle, α and β are real and imaginary parts of the complex wave number, respectively.

From expressions (2) and (3) it can be seen that the frequency shifts give damped oscillations, and with increasing the liquid layer thickness they tend to limit values $\Delta f'_{\infty}$ and $\Delta f''_{\infty}$. Theoretical dependences of $\Delta f'$ and $\Delta f'$ on the thickness of liquid layer for the case of $G' = 3 \times 10^5$ dyne/cm² and tan $\theta = 0.3$ are presented in Fig. 1.

From the analysis of expressions (2) and (3) three ways to measure the shear modulus of liquids follow, two of which are discussed in this paper. The first method is realized at small thickness of liquid layer, where it is much smaller than the shear wave, $H \ll \lambda$. In this case expressions (2) and (3) are extremely simplified and the real *G*' and imaginary *G*" parts of the complex shear modulus can be obtained from the following expressions:

$$G' = \frac{4\pi^2 M f_0 \ \Delta f' H}{S}, \qquad G'' = \frac{4\pi^2 M f_0 \ \Delta f'' H}{S}, \tag{4}$$

where *S* is the area of the cover-plate. For the tangent of mechanical losses we have:

$$\tan \theta = \frac{G''}{G'} = \frac{\Delta f''}{\Delta f'}.$$
(5)

The influence of asymmetry of the system, leading to a shift in the node of the resonator is considered in [12]. From these expressions, as well as from Fig. 1 we can see that the dependences of the frequency shifts Δf and $\Delta f'$ on the inverse value of the thickness of the liquid layer *H* should be linear. Imaginary shift of the resonance frequency is determined by the change of damping vibration system, i.e. it is equal to half of the change in the width of the resonance curve.

At large thicknesses when $H \rightarrow \infty$ shear wave is completely damped in the thick layer of liquid, the need for a solid cover-plate disappears and horizontal surface of piezoquartz can be loaded with a thick layer of the test liquid, i.e. $H \gg \lambda$. From the expres-



Fig. 2. Experimental dependences of real Δf (1) and imaginary $\Delta f'$ (2) frequency shifts on the inverse value of the liquid layer thickness for ethylene glycol.

sions (2) and (3) the following expressions for G' and $\tan \theta$ can be obtained:

$$G' = \frac{16\pi^2 M^2}{S^2 \rho} \cdot \left[(\Delta f_{\infty}'')^2 - (\Delta f_{\infty}')^2 \right],$$

$$\tan \theta = -\frac{2\Delta f_{\infty}' \cdot \Delta f_{\infty}''}{(\Delta f_{\infty}'')^2 - (\Delta f_{\infty}')^2}.$$
(6)

This expression shows that if liquid has bulk shear modulus then $\Delta f_{\infty}'' \gg \Delta f_{\infty}'$. If the liquid is Newtonian then $\Delta f_{\infty}'' = \Delta f_{\infty}'$. This method of measuring the shear elasticity of liquids is similar to the known impedance method of Mason, discussed in detail in [13]. From the expressions (6) it follows that to calculate *G* it is sufficient to measure the threshold values $\Delta f_{\infty}''$ and $\Delta f_{\infty}'$.

3. Results and discussion

In this paper we measure the shear modulus of various liquids and their dependence on the degree of wettability of working surface.

Fig. 2 shows the experimental dependence of the real Δf and imaginary $\Delta f'$ frequency shifts on the inverse thickness of the liquid layer for ethylene glycol, when the thickness is much smaller than the shear wave length, $H \ll \lambda$. As can be seen these dependencies are linear that, according to expression (4), indicates the presence of the complex shear elasticity in liquids studied at the oscillation frequency 74 kHz. Calculation by formulas (4) and (5) for the shear modulus and tangent of mechanical loss gives the values $G' = 0.91 \times 10^6$ dynes/cm², tan $\theta = 0.24$, respectively. Similar dependencies were obtained for all the fluids regardless of viscosity and polarity. The results of measurements for other liquids are given in Table 1. The presence of low-frequency shear elasticity of liquids suggests that in liquids, along with high-frequency relaxation process there is a low-frequency viscoelastic relaxation, determined by collective interactions of large groups of molecules (clusters).

Studies have shown that the thorough treatment of the working surface of piezoquartz to provide the best wettability is of great importance for the reproducibility of measurement results. At the maximum value of the friction coefficient, which means the surface purity, vibrating surface of piezoquartz is completely wetted by the liquid and the value of the measured shear modulus is the highest. Contrary, at the wittingly hydrophobized surface there is a hydrophobic slippage of liquid on the working surface of a vibrating piezoquartz due to poor wettability. In the case when quartz oscillates the energy transferred in contacted layer of liquid is minimal

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Table 1
Experimental results of measurement of fluid shear modulus.

Liquids	t, °C	ho, g/cm ³	$H \ll \lambda$		Hydrophilic surface		Hydrophobic surface	
			$G' imes 10^{-6}$, dyne/cm ²	$\tan \theta$	$\Delta f_{\infty}^{\prime\prime}$, Hz	$G' \times 10^{-6}$, dyne/cm ²	$\Delta f_{\infty}''$, Hz	$G' \times 10^{-4}$, dyne/cm ²
1. Water	22	0.99	0.31	0.3	13	0.25	1.5	0.35
Ethylene glycol	23	1.11	0.91	0.24	20	0.6	2	0.6
Triethylene glycol	22	1.12	1.28	0.27	23	0.8	3	1.38
4. Butyl alcohol	21	0.81	1.03	0.10	21	0.67	2.5	0.96
5. Octyl alcohol	23	0.82	1.34	0.16	25	0.9	4	2.45
6. Polymethyl siloxane – 100	22	0.97	0.6	0.5	16	0.4	1.5	0.35
7. Polymethyl siloxane – 900	23	0.97	1.35	0.8	25	0.96	4.5	3.1

and liquid does not fully exert its elastic properties. In this case, the shear modulus has a minimum value. Hydrophobic slippage is particularly clearly manifested in the measurement of limits of the frequency shifts $\Delta f_{\infty}'$ and $\Delta f_{\infty}''$, when the entire horizontal surface of piezoelectric quartz is loaded with a thick layer of the liquid. The second method most clearly shows the dependence of measured shear modulus on hydrophobicity of surface.

As a criterion of surface condition the coefficient of static friction is accepted, which is measured by tribometer of Derjaguin-Lazarev design [14]. The general form of the tribometer is shown in Fig. 3. Tribometer consists of the sample stage of the microscope M, where rails A with trolley C are installed on bolts. The trolley is able to move on these rails in a horizontal direction by means of inextensible thread D, which coils round shaft of the Warren's motor E, the rotating speed of 2 rpm. The investigated surface is placed on the trolley. The slider *K* is a disc supported by three needles α_1, α_2 , α_3 on the rubbing surface. At the top of the disc is placed a load, creating together with the weight of the slider the normal load. In its motion the trolley carries the slider, which rests against one of his legs at one end of an elastic rod T. The other end of the rod is fixed on a massive rack. As it moves, the rod T starts to bend until its reaction reaches the value of the force of static friction between the slider and the surface. At the beginning of sliding a deviation of an elastic rod decreases. Thus, the maximum deviation gives the value of static friction force, if the calibration of the rigidity of the rod is pre-made. Deviation of the rod T is measured by the measuring microscope *M*, equipped with an ocular scale.

In our case, the standard comparator IZA-2 was used as the measuring microscope and smoothly moving trolley. An optically polished quartz plate, fixed on the comparator stage, served as a surface under study. The slider in the form of a small platform sup-

Fig. 3. Ttribometer of Derjaguin-Lazarev design.

ported by three glass legs with spherical ends on a quartz plate which was connected to the calibrated elastic metal rod fixed on an independent stand by inextensible cord. Smooth movement of the stage was carried by micrometer screw of comparator. The accuracy of determining the coefficient of friction was high enough. For example, the strain of an elastic rod was determined with an accuracy $\pm 5 \,\mu$ m.

The measurement was carried out as follows. The working surface of piezoquartz and glass legs of platform were subjected to thorough cleaning. Next, the coefficient of static friction of the pair quartz–glass was determined and the shear elasticity of the test liquid purified by repeated distillation was measured by the method described above. For the purification the following methods were used: surfaces washing by double-distilled water and alcohol; exposure to the chromium mixture, followed by doubledistilled water washing; steaming; surface treatment in a glow discharge and in a hydrogen burner flame. These treatments gave a sufficient range of coefficients of static friction to investigate the dependence of the measured shear modulus on the condition of the working surface of piezoquartz.

Fig. 4 shows the results of experiments with ethylene glycol. The abscissa is the coefficient of static friction *K*, and the vertical axis – the corresponding values of shear modulus *G'*. It is seen that the measured shear modulus depends strongly on the surface condition. The maximum values of measured shear modulus for liquids correspond to the maximum values of the coefficient of static friction, which are reached by a thorough treatment of the working surface.

It should be noted that the best lyophilic surface is achieved by successive refinement by methods described above. The last step in



Fig. 4. The dependence of measured shear modulus on the value of static friction coefficient for ethylene glycol.

cleaning the surface is the hydrogen flame treatment during 2-3 s.

The limit value of an imaginary shift of frequency $\Delta f_{\infty}''$ for ethylene glycol at the maximum coefficient of static friction is 20 Hz. Calculation of *G'* by expression (6) gives a value of 0.6×10^6 dyne/cm². The measurement results for other liquids are given in Table 1. Measurement errors were close to 5%. The limiting value of the real frequency shift $\Delta f_{\infty}'$ for liquids with $\tan \theta < 1$ can be neglected, since the contribution made by them does not exceed 3%. The first method of shear modulus measuring ($H \ll \lambda$) is characterized by smaller area of contact between tested liquid and quartz, and is more preferable than the second method ($H \gg \lambda$) as more accurate in the measurement of shear modulus.

The effect of hydrophobic slippage of liquid on a solid surface, discussed in details in [15–17], manifests itself in the measurement of shear elasticity of polar liquids. Although the physical nature of the effect of hydrophobic slippage is still not clear, nowadays a model, in which slippage is associated with a reduction of viscosity in a thin boundary layer, is the most applicable. This reduction can be attributed to various factors, including increased nucleation of dissolved gas near a hydrophobic surface [16].

We measured the shear moduli of liquids on the knowingly hydrophobized surface of piezoquartz for comparison. For surface hydrophobization the cleaned and dried piezoquartz was placed in 20 - 30 min in 5% chlortrimethylsilan solution in benzene then washed clean with benzene. At knowingly hydrophobized surface of piezoquartz which corresponds to the minimum coefficient of static friction, the imaginary shift of the resonance frequency $\Delta f_{\infty}^{\prime\prime}$ for ethylene glycol was only 2 Hz. Calculation of *G*^{\prime} using formula (6) gives a value of 0.6×10^4 dyne/cm², i.e. much lower than that of carefully hydrophilized surface.

Thus, depending on the degree of hydrophobization of the working surface, the measured shear modulus of liquids the can vary widely.

A characteristic feature of the curve in Fig. 4 is a tendency to saturation of the shear elasticity with an increase in the coefficient of static friction, which depends on the degree of processing of the working surface. The highest values of the coefficient of static friction are obtained by the complex cleaning of surface, the last stage of which is the treatment by the hydrogen flame. Obtained results of measurements of the shear modulus of liquids are similar to the results obtained by other methods [18–20] on a hydrophilic surface.

4. Conclusions

Shear modulus of elasticity of liquids, measured by the resonance method, varies widely depending on the degree of purity of the piezoquartz surface. We obtain a monotone correlation between the measured shear modulus of elasticity of the liquid and the magnitude of static friction surface. With increasing friction coefficient the maximum value of the measured shear modulus is reached, corresponding to complete wetting of the liquid surface of a vibrating piezoquartz (i.e. absence of slip), and it is the true value of the measured shear modulus of the liquid studied. The best wettability of surfaces is achieved by an integrated piezoquartz cleaning using a hydrogen flame. Hydrophobicity of the working surface leads to decrease of the measured shear modulus of liquid.

Thus, our study shows that along with high-frequency relaxation process in liquids, there is a low-frequency viscoelastic relaxation process with a long relaxation time. Apparently, in fluids, in some cases, the main role is played by collective effects, which depend on the interaction of large groups of molecules (clusters). Relaxation of a large number of particles requires a concerted movement of many molecules, so it can be assumed that the existence of low-frequency shear elasticity of liquids is caused by their microheterogeneous structure. This microheterogeneous liquid structure, obviously, is the dynamic nature of the fluctuation.

Numerous works of researchers were devoted to studying the shear properties of liquids at high (MHz) frequencies, and the available data at low frequencies belongs to the study of highly viscous and supercooled liquids in the glass transition temperature. The applied methods do not allow to detect low-frequency shear elasticity of low-viscosity liquids. Our results converge with a series of recently published papers [21–26] which reveal solid-like behaviours of fluid above the melting point. Thus, the study of low-frequency shear elasticity of liquids is essential for understanding the nature of the liquid state of matter.

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