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METHOD OF STUDYING VISCOELASTIC PROPERTIES OF POROUS POLYMER FILMS

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A method is proposed for studying the viscoelasticity of polymer films with the aid of a quartz resonator. Results are reported pertaining to thin copper films.

In order to produce reliable quartz-type air humidity transducers, devices of great practical importance, one must know the viscoelastic properties of thin porous coatings. In another study [1] was demonstrated the feasibility of using a compound vibrator for solid specimens of sections smaller than the end section of the piezoelectric bar. With the use of such specimens it becomes possible to study the viscoelastic properties of polymers in which energy losses are high and the acoustic velocity is low. Unlike a conventional compound vibrator, the one in the method to be described here the piezoelectric cell has a specimen (film) deposited on its entire surface.

The diagram in Fig. 1 depicts an X-cut piezoelectric cell. The excitation electrodes are deposited on the $l_0 l_m$ face. The entire surface of this cell is coated uniformly with a polymer film of thickness Δ' by immersion in a preheated 3-5% solution of the polymer in an appropriate solvent and subsequent spinning in a centrifuge. This process yields then uniform films with excellent adhesion to the surface of the piezoelectric cell. When the latter vibrates and the adhesion is strong, the film will perform the same vibrations as the cell surface, i.e., there will be no sliding of the film. Assuming that the piezoelectric cell vibrates longitudinally along the y axis and that its strains ε are equal to strains in the film, one can write the equation of motion for the cell with film in the form

$$(m_1 + m_2)\ddot{\varepsilon} + (N_1 + N_2)\dot{\varepsilon} + (G_1 + G_2)\varepsilon = F. \tag{1}$$

According to the electrochemical analogy, the electric-circuit equation equivalent to Eq. (1) can be written as

$$(L_1 + L_2)\ddot{q} + (R_1 + R_2)\dot{q} + \left(\frac{1}{C_1} + \frac{1}{C_2}\right) q = U. \tag{2}$$

The relation between parameters in Eq. (1) and those in Eq. (2) can be defined in terms of the coefficient β [2]

$$\beta = 2L_1/m_1 = 2(L_1 + L_2)/(m_1 + m_2). \tag{3}$$

The equivalent electrical parameters L_1 and L_2, R_1 and R_2, C_1 and C_2 are determined through measurement [3]. The reproducibility of results is high. The resonance frequencies f_1 of the piezoelectric cell and f_2 of the specimen with the cell are determined from Eq. (1), namely

$$f_1 = [G_1/2\pi^2 m_1]^{1/2}, \tag{4}$$

$$f_2 = [(G_1 + G_2)/2\pi^2 (m_1 + m_2)^{1/2}. \tag{5}$$

Expressions (4) and (5) are used for calculating the stiffness parameters G_1 and G_2. The

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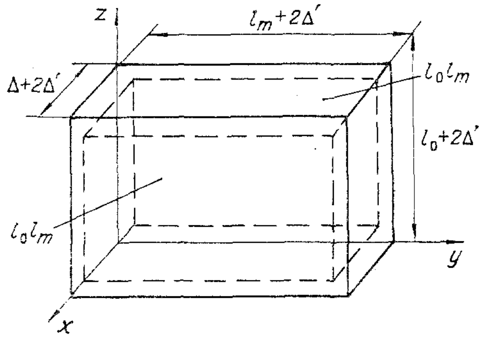

Fig. 1. External view of X-cut piezoelectric cell coated with polymer film.

masses of the cell, needed for this calculation, are found from the dimensions or by measurement.

Since it is difficult to determine the mass of the film through measurement, on account of its being too small, one finds it from the change in equivalent inductance of the piezoelectric cell in accordance with relation (3).

When the density ρ_1 of the film material is known, then one can determine first the volume and then the thickness of that film. From its stiffness and its mass, in turn, one finds its resonance frequency

$$f_3 = [G_2/2\pi^2 m_2]^{1/2}, \tag{6}$$

which can differ from the resonance frequency of the piezoelectric cell.

It is interesting to note that results based on relation (6) are close to those based on the relation [1] usually applied to a compound vibrator.

The acoustic velocity in the film is

$$v = 2f_3(l_m + 2\Delta'). \tag{7}$$

For determining the viscosity of the film material we have used the relation [4] which a comparison of the damping increment for a plane wave and the damping decrement for the vibrating film with cell yields:

$$\eta = \frac{R_2(l_m + 2\Delta')}{\pi^2(l_0+\Delta+2\Delta')\Delta'\beta}. \tag{8}$$

For verification of this method, measurements were made on thin copper films electrolytically deposited on the electrodes of a piezoelectric cell. Sliding of the film was thus prevented, with the earlier stipulated condition of equal strains in film and cell satisfied. As the piezoelectric cell was used at DT-cut quartz plate with dimensions $l_m = 1.559 \cdot 10^{-2}$ m, $l_0 = 0.608 \cdot 10^{-2}$ m, and $\Delta = 0.062 \cdot 10^{-2}$ m. Its resonance frequency was 300 kHz. The fundamental vibration mode was in circumferential shear. Since the frequency of such vibrations is determined by the cell width, dimension l_m in relations (7) and (8) must be replaced with dimension l_0. Measurements were made on two such cells with copper films of somewhat different thicknesses. The experimentally determined shear modulus was $C_{44} = (5.88 \pm 0.4) \cdot 10^{10}$ Pa. For comparison were taken results [5] pertaining to crystalline copper and averaged by a special method. According to W. P. Mason, $C_{44} = 5.46 \cdot 10^{10}$ Pa. The feasibility of measuring the viscosity of thin metal films was tested by comparing the experimentally determined hysteresis constant of copper with published data [5]. The hysteresis constant is well known to be

$$H = C_{44}/Q \tag{9}$$

and related to the viscosity through the equality

$$H = \omega\eta. \tag{10}$$

According to experimental data, the Q-factor of a copper film at a frequency of 300 kHz was $Q = 1852 \pm 95$, corresponding to a hysteresis constant of $H = 3.17 \cdot 10^7$ Pa. According to Mason's data, the hysteresis constant of copper vibrating torsionally at this frequency of 300 kHz was of order $3.2 \cdot 10^7$ Pa. The results are thus entirely satisfactory, considering that the vibrations in or experiment were not torsional.

Following the verification of this method on metal films, a study was made of polymer sorption coatings. The results have revealed the mechanism by which energy of elastic vibrations is dissipated in dry and in moist Capron films. The frequency characteristic of the Q-factor of Capron film in a dry medium and in a moist medium was measured with the aid of an XYS + 1°30'-cut piezoelectric cell having a resonance frequency of 100 kHz and excitable in the fundamental mode as well as in the third and fifth harmonics. In dry air the Q-factor of Capron film hardly changes with frequency, which suggests a hysteresis mechanism of energy loss. In a moist medium the Q-factor of Capron film is inversely proportional to the frequency, which indicates a viscous mechanism of energy loss. An interesting peculiarity of porous polymer films is their much lower modulus of elasticity in dry air and its increase with increasing humidity. In an analysis of polymer sorbents in a moist medium it is important to take into account the thickness of the acoustic layer, within which most of the energy of elastic vibrations of the piezoelectric cell is lost.

In conclusion, we will now estimate the attainable accuracy of this method. It depends on the error with which the equivalent parameters of quartz resonators are measured, on the accuracy with which the mass of the piezoelectric cell is determined, and on the thermal conditions of the experiment. The largest error is usually the one with which the equivalent parameters of quartz resonators are measured. The viscosity of media was determined with the aid of quartz resonators in one study [6]. With the temperature stabilized, the error of viscosity determination reached the order of 12.5%. Such a relatively large error could be explained by the fact that the damping decrement was determined from the width of the resonance curve of a quartz resonator placed in the medium under investigation. The resonance curve of a high-Q quartz resonator is tens of hertz or, in the more favorable case, hundreds of hertz wide, equivalent to 10^{-5}-10^{-6} of the resonance frequency. In this case electronic-counter frequency meters, which measure the resonance frequency very accurately (error within 10^{-6}%), cannot measure the width of the resonance curve with sufficient accuracy. For this reason, the error of measurements of the equivalent parameters based on the width of the resonance curve is of the order of 5-10%.

A special device ensuring a higher accuracy has been developed [7] for measuring the equivalent parameters of quartz resonators. The equivalent impedance of a quartz resonator is in this device determined with the use of a standard reference resistor and a cathode-ray voltmeter of 2-3% accuracy. The author notes, however, that the internal friction in a specimen is calculated with the aid of tables listing piezoelectric constants and the values of these constants can differ by as much as 20% from experimentally obtained ones. This introduces an additional error into the measurements.

In this experiment the equivalent parameters of quartz resonators were measured with special instruments [3] ensuring a reproducibility of results within ±1% through use of a self-balancing bridge circuit and of an alternating voltage with small amplitude across the quartz resonator so as to minimize fluctuations of mechanical stresses in the test specimen. The mass of a piezoelectric cell was determined from its dimensions, the latter measured with a micrometer accurately within ±1.7%. The temperature and the relative humidity of air during tests were maintained constant within ±0.2°C and ±2%, respectively, in a model "Feutron" climatic chamber (made in the German Democratic Republic). Thus the largest contribution to the measurement error came from determination of the mass of a piezoelectric cell on the basis of its dimensions. The overall error of measurement of the viscoelastic properties of polymer films can be estimated at 4-8%, but it can be reduced substantially by use of better methods for determining the mass of a piezoelectric cell.

NOTATION

X, cut of a piezoelectric cell; l_m, length of a piezoelectric cell; l_0, width of a piezoelectric cell; Δ, thickness of a piezoelectric cell; Δ', thickness of a polymer film; y, orientation axis of a piezoelectric cell; ε, strain; m_1, equivalent mass of a piezoelectric cell; m_2, equivalent mass of the coating specimen; N_1, equivalent mechanical resistance of a piezoelectric cell; N_2, equivalent mechanical resistance of a coating specimen; G_1, equivalent stiffness of a piezoelectric cell; G_2, equivalent stiffness of a coating specimen; L_2, equivalent inductance of a coating specimen; R_1, equivalent electrical resistance of a piezoelectric cell; R_2, equivalent electrical resistance of a coating specimen; C_1, equivalent capacitance of a piezoelectric cell; C_2, equivalent capacitance of a coating specimen; U, voltage; q, electrical charge; β, electromechanical coupling coefficient; f_1, resonance frequency of a piezoelectric cell; f_2, resonance frequency of a coating specimen with piezoelectric cell; f_3, resonance frequency of a coating specimen alone; ρ_1, density of the coating material; v, acoustic velocity in the coating material; η, viscosity of the coating material; H, hysteresis constant; Q, Q-factor of a coating (film) specimen; ω, radian frequency; XYS + 1°30', cut of a piezoelectric cell in this study; C_{44}, shear modulus; and L_1, equivalent inductance of a piezoelectric cell.

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THERMAL CONDUCTIVITY OF INERT GASES OVER A WIDE TEMPERATURE RANGE

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We present the results of correcting and correlating experimental data on the thermal conductivity of inert gases in the temperature range 90-6000°K.

The thermal conductivity of monatomic gases at atmospheric pressure has been rather well investigated over a wide range of temperatures. The experimental material accumulated up to 1976 was systematized and correlated [1, 2] in the form of smoothed empirical relations or tables of reference data for temperatures up to ∿2000°K. In [3, 4] the high-temperature experimental data on the thermal conductivity of neon, argon, krypton, and xenon were correlated and represented by a power-law dependence on the temperature in the range from 500-800 to 5000°K. It was noted that the results of shock-tube measurements in [5-8] are systematically lower than values obtained by steady-state methods in the overlapping temperature range as a result of the improper use in [5-8] of a power-law dependence of the thermal conductivity on the temperature

$$\lambda = \lambda_0 \left(\frac{T}{T_0} \right)^b \quad (1)$$

with a constant value of the exponent in the temperature range 300-5000°K. On this basis the results in [5-8] were increased in the correlation by a certain amount for each gas [3, 4].

The presently available experimental data on the thermal conductivity of inert gases in the temperature range 90-6000°K plotted in Fig. 1 show definite regularities in the difference between the results obtained by steady and unsteady methods.

At temperatures above 1000°K there is a small (in principle within the 3-4% limits of experimental error), but systematic divergence of the data, with the values of the conduc-

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