

INVESTIGATION OF LIQUID FLUOROPOLYMERS AS POSSIBLE MATERIALS FOR LOW-TEMPERATURE LIQUID WALL CHAMBERS FOR ULTRACOLD-NEUTRON STORAGE

*Yu. N. Pokotilovski**

*Joint Institute for Nuclear Research
141980, Dubna, Moscow Region, Russia*

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Several hydrogen-free liquid low-temperature fluoropolymers are investigated from the point of view of their possible use as the material for walls of ultracold-neutron traps with low losses. Viscosity was measured in the temperature range 150–300 K and neutron scattering cross-sections were measured in the temperature range 10–300 K and in the neutron wavelength range 1–20 Å. Some conclusions are made for their possible ultracold-neutron bottle properties. Quasi-elastic neutron reflection from the surface of a viscous liquid is considered in the frame work of the Maxwell dynamic model.

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1. INTRODUCTION

The goal of present-day investigations of free neutron decay is to reach a precision better than 0.1 % for parameters of the V–A theory of the neutron beta decay through measurement of the asymmetry of neutron decay and the neutron lifetime. The neutron lifetime measurements are necessary for determining the fundamental coupling constants of the weak interactions. Additionally, it is an important parameter in astrophysical calculations concerning the creation of nuclei in the early stage after the Big Bang, including the helium abundance in the Universe, the number of species of light stable neutrinos, and solar neutrino flux calculations.

An almost order-of-magnitude improvement of the accuracy of measurement of the neutron lifetime has occurred in recent years [1]. This progress was reached entirely due to the application of ultracold-neutron (UCN) storage in closed volumes. A review of the neutron lifetime measurements up to 1990 was published by Schreckenbach and Mampe [2] (see also the review by Pendlebury [3], on wider fundamental applications of UCN). For a recent review of investigations of free-neutron decay see [4, 5]. The most precise

measurements of the neutron lifetime were carried out with very low surface temperature beryllium and solid oxygen traps [6], or with traps covered with Fomblin oil [7–10].

The experiments with very cold (near 10 K) beryllium UCN traps have led [11, 12] to the observation of surprisingly large UCN losses in the traps, exceeding theoretical predictions by two orders of magnitude. At room temperature, the wall losses exceed the theoretical ones by an order of magnitude [12]. The reason of this anomaly is not understood yet and is a serious impediment to further significant progress in precision of neutron lifetime measurements by this method.

The losses of UCN stored in traps with walls covered with solid oxygen [6, 12] or cooled graphite [13] also exceed those calculated according to cold-neutron transmission cross-sections by two to three orders of magnitude.

Application of the hydrogen-free perfluoropolyether (PFPE) oil (Fomblin) was first proposed by Bates [14] and was tested for neutron lifetime measurement in [15]. Fomblin has the chemical formula $\text{CF}_3(\text{C}_3\text{F}_6\text{O})_n(\text{OCF}_2)_m\text{OCF}_3$, with $m/n = 20\text{--}40$, and the molecular weight near 3000 [16].

The UCN losses caused by Fomblin wall collisions in a large UCN trap (volume up to 721) were around 20 % (at 10 °C) and 10 % (at 4 °C) of the beta decay in [15]

*E-mail: pokot@nf.jinr.ru

and subsequent experiments [7, 8] and were the most important source of systematic errors when corrections for these losses were made. There were plans [17] to continue the neutron lifetime experiments with an upgraded installation in the same way as in [7, 8].

Careful measurements of UCN losses in Fomblin traps with the aim to obtain the UCN energy dependence of the loss coefficient were performed in [18]. In the expression for the UCN loss probability averaged over the isotropic angular distribution,

$$\bar{\mu}(E) = 2\eta \left[\frac{V}{E} \arcsin \left(\sqrt{\frac{E}{V}} \right) - \sqrt{\frac{V-E}{E}} \right], \quad (1)$$

where E is the energy of the neutron and V is the boundary potential of the trap, the experimental UCN reduced loss coefficient η was found to be $\eta = 2.35(0.10) \cdot 10^{-5}$ for the Fomblin oil and $\eta = 1.85(0.10) \cdot 10^{-5}$ for the Fomblin grease at 21 °C. It is interesting to note that these values of the measured UCN loss coefficient η in Fomblin are significantly lower than the limiting anomalous loss coefficient $\eta_{anom} = 3.3 \cdot 10^{-5}$ in beryllium traps at 10 K [12]. At a lower temperature of Fomblin, 4 °C, the loss coefficient was as low as $\eta = 1.3(1) \cdot 10^{-5}$, the best one ever obtained experimentally and 2.5 times lower than the anomalous loss coefficient in experiments [12].

We use the standard formalism for the complex potential U describing the UCN interaction with walls,

$$\eta = \frac{\text{Im } U}{\text{Re } U}, \quad U = \frac{4\pi\hbar^2}{2m} \sum_i N_i b_i, \quad \text{Im } b = \frac{\sigma}{2\lambda}, \quad (2)$$

where m is the neutron mass, N_i is the number of nuclei in a unit volume of the wall material, b_i is the coherent scattering length on a bound nucleus of the wall, and σ is the cross-section of inelastic processes for neutrons with wavelength λ ; this formalism allows calculating the part of wall loss coefficient attributed to neutron capture in Fomblin. This part is as low as $3.6 \cdot 10^{-7}$ (the experimental value $\text{Re } U = 106.5 \text{ neV}$ [19] was used in this calculation). This implies that the main component of UCN losses is the inelastic scattering. For Fomblin, a strong dependence of UCN wall losses on temperature was observed: the losses decrease by about 3% as the temperature is lowered by 1 °C [7]. This requires a very good wall temperature uniformity over the trap surface for precision measurement of the neutron lifetime. But the use of Fomblin at lowered temperatures did not prove to be promising in view of crumbling of the Fomblin surface in solid and near-to-solid states [7, 9], resulting in a significant increase of the UCN losses. The authors of [7] found that below

0 °C, the Fomblin oil becomes too viscous for respraying over the wall surface.

But for a liquid Fomblin surface, contrary to solid surfaces, the difference between the measured UCN loss coefficients and the ones calculated from transmission measurements is not large.

Except for the indication [7] that transmission measurements with $\lambda = 60 \text{ \AA}$ neutrons at 20 °C Fomblin agree with the cross-section of UCN losses within the factor 1.5, there is no (known to the author) quantitative experimental information on neutron inelastic scattering in PFPE; the possibility for calculating it suffers from uncertainties about dynamics of thermal motion in this liquid polymer. It is interesting to compare the total inelastic UCN cross-section in PFPE extracted from [7, 18] in accordance with Eq. (2) and extrapolated to the thermal point in accordance with the inverse-velocity law $\sigma_{extr}^{PFPE} = 8.65 \text{ b}$ ($\approx 0.51 \text{ b}$ per atom of PFPE) with the experimental data for a «similar» compound, Teflon $(\text{CF}_2)_n$ [20]. For the lowest neutron energy 0.376 meV used in these measurements, at which the elastic coherent scattering effects are believed to be negligible (below all the Bragg peaks in the cross-section measured in [20]), the cross-section per atom extrapolated to the thermal point is 0.63 b, which satisfactorily agrees with 0.51 b.

In [21], it was proposed to try low-temperature liquid wall chambers for storing UCN. This proposal was prompted by publication [22] that reported about new recently synthesized fully fluorinated compounds whose physical properties are promising from the point of view of using them in liquid-wall UCN traps. These new compounds — perfluorinated polyaldehydes — have a large liquid range and good low-temperature properties. Their formula is $\text{C}_4\text{F}_9(\text{OCF}_2)_n\text{C}_4\text{F}_9$. Depending on the number n (between 6 and 10), the boiling temperature of this polymer varies between 125 and 200 °C; the melting point is between –145 and –152 °C.

There is the hope that at lower temperatures — in the vicinity of the melting point (e.g., –100 °C), inelastic upscattering, which is believed to be the most important component of UCN losses at collisions with chamber walls, decreases by several times. Corrections for the UCN loss at inferring the neutron lifetime from the UCN storage data are decreased respectively.

It is not yet clear to what extent the recently observed [23] small UCN cooling and heating during storage in Fomblin traps is an important component of UCN losses from the liquid-wall traps.

The lack of any information about dynamical properties of this new compound (as well as for Fomblin)

only allows making a very approximate estimate of gain in UCN losses (UCN upscattering cross-section) with lowering the temperature from 10 to -120°C . It is assumed in this estimate that dynamical properties of PFPE and this new polymer are similar, and the frequency distribution is described by the Debye model. From the known specific heat of Fomblin, $0.24 \text{ kal}/(\text{g}\cdot\text{grad})$ [24], the Debye temperature of Fomblin can be calculated in standard way as 750 K ; it in turn allows calculating the UCN upscattering cross-section in the incoherent approximation. The result of this calculation is that the upscattering cross-section decreases by five times from room temperature to -120°C , and changes by 0.7% per 1°C at room temperature. The latter figure is four times lower than was reported [7], and our estimate therefore seems to be a lower bound of the possible decrease of UCN upscattering with lowering the temperature.

To our regret, we were unable to obtain this substance [22] in our disposal. But it turned out that low-temperature fluoropolymers with similar properties are produced in Russia¹⁾.

The goal of this work was to investigate several possible low-temperature fluoropolymers that are candidates for a low-temperature liquid-wall UCN bottle. Measured were: viscosity as a function of temperature because viscosity determines the temperature range at which the liquid wall can be practically used and cold neutron cross-sections as a function of temperature.

2. VISCOSITY AND NEUTRON CROSS-SECTION MEASUREMENTS

Viscosity was measured with the simplest possible method of a «sinking ball» [25]. The results are presented in Fig. 1. As can be seen POM-310 and POM have the most promising properties. They are the mixtures of complex fluoropolyoximethilenes with the general formula $\text{CF}_3\text{O}(\text{CF}_2\text{O})_n(\text{CF}_2\text{CF}_2\text{O})_m(\text{OCF}_2\text{CF}_2\text{O})_l\text{CF}_3$ with $n : m : l = 65.8 : 3.1 : 0.2$ and molecular weight 4883 for POM-310, and $n : m : l = 30.3 : 1.5 : 0.2$ and molecular weight 2354 for POM.

It can be seen from Fig. 1 that the viscosity of the most promising liquids, POM and POM-310, at -90°C is close to the viscosity of Fomblin at a temperature near 0°C , at which it can still be used in the exper-

¹⁾ The investigated substances were produced by Perm branch of the Russian Scientific Center «Applied Chemistry» and by the State Scientific Institute for Organic Chemistry and Technology, Moscow.

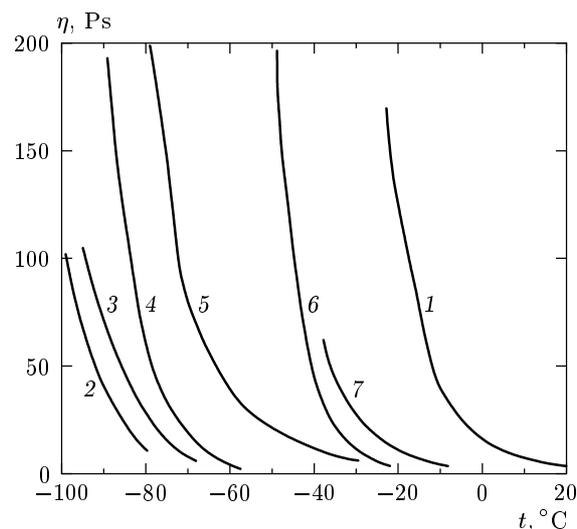


Fig. 1. Viscosity of different liquid fluoropolymers as a function of temperature: 1 — Fomblin; 2 — POM-310; 3 — POM, 4–7 — several other liquid fluoropolymers with a different chemical content

iments on neutron lifetime measurement [8, 15]. This means that from the experimental standpoint, they are appropriate for a similar use as in experiments [7].

Neutron cross-section measurements were performed at the channel 6B of the reactor IBR-2. The main goal was to measure the total cross-sections at the lowest possible energies in a wide temperature range that would allow inferring the upscattering part of the cross-section from these measurements. This can be used to estimate possible UCN losses in storage experiments.

In view of an overwhelming flux of delayed thermal neutrons in the direct beam time-of-flight spectrum below the neutron energy about 1 meV , diffraction from the stack of mica (artificial fluorophlogopite [26, 27]) with the lattice parameter 9.97 \AA for the (001) plane was used for neutron monochromatization. A significant suppression of the delayed thermal neutron component in the diffracted neutron beam was reached in this way. The spectrum of neutrons diffracted from the stack of mica (thickness about 1.8 mm) is shown in Fig. 2.

The total cross-sections were measured at several Bragg angles of neutron diffraction from mica, from 45 to 83.5° . In Fig. 3, we show the total neutron cross-section for three room-temperature fluoropolymers as a function of the neutron wavelength. Strong coherent effects can be seen in a wide wavelength range up to the largest wave length reached in this measurements, 20 \AA .

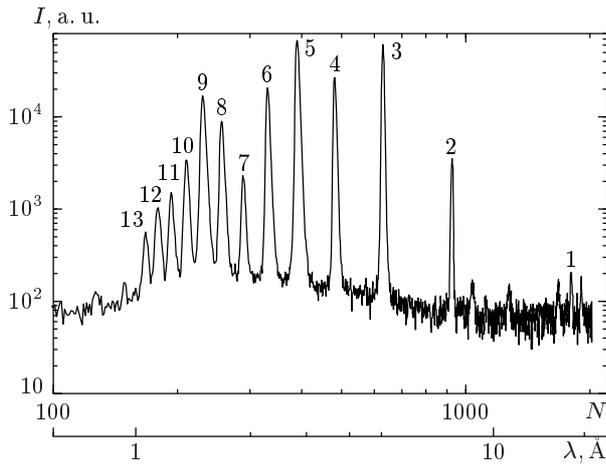


Fig. 2. Time-of-flight spectrum of neutrons diffracted from the stack of artificial fluorogopite at the Bragg angle 45° . Numbers indicate the order of diffraction

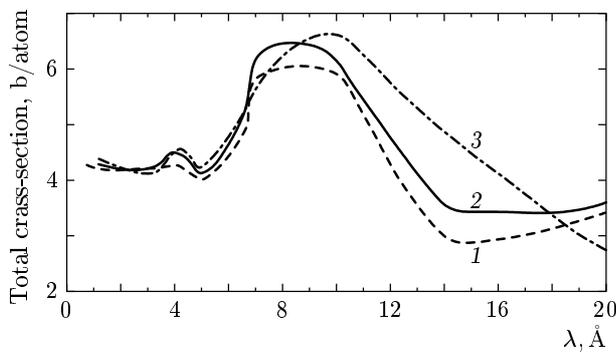


Fig. 3. The total neutron cross-section for liquid fluoropolymers at temperature 290 K as a function of the neutron wavelength: 1 — Fomblin; 2 — POM-310; 3 — POM

This behavior is similar to the behavior of the cross-section for an other well-known solid fluoropolymer, Teflon. Our measurements of the total cross-section for Teflon are in a good agreement with the previous one [20].

To obtain the value of the upscattering probability for UCN in traps with walls covered with these liquids, the measurements of the total neutron cross-section were performed in wide temperature range from 290 down to 10 K and in the wavelength range from 1 to 20 Å. In Figs. 4 and 5, we show temperature dependence of the total cross-section of long-wavelength neutrons for Fomblin and POM-310. The cross-section for POM (not shown) is close to the cross-section for POM-310.

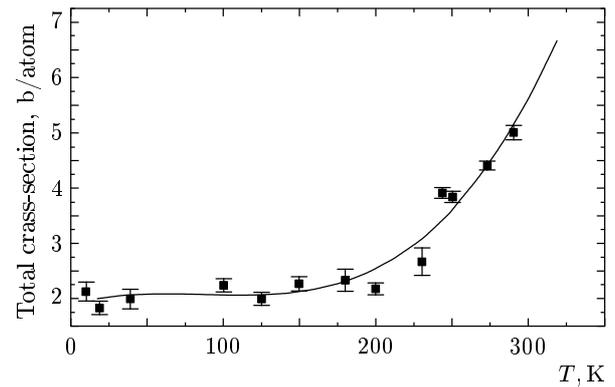


Fig. 4. The total neutron cross-section for Fomblin versus temperature for the neutron wavelength 14.1 Å

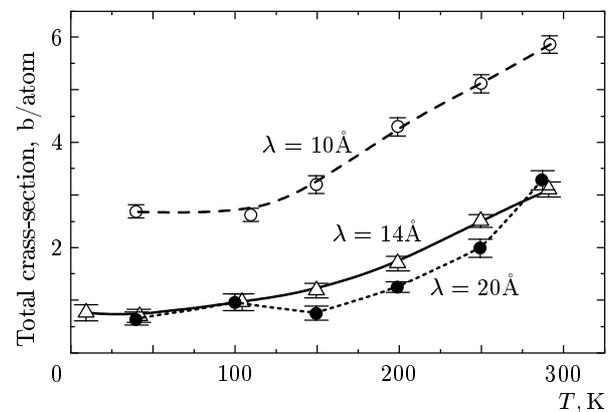


Fig. 5. The total neutron cross-section for POM-310 versus temperature for several neutron wavelengths

In the temperature range of interest (200–300 K) and wavelength larger than 10 Å, upscattering is a strongly dominating component of inelastic scattering (for neutron energies $E_n \ll kT$). At low temperatures (10–100 K), the cross-section is independent of the temperature and is the one of entirely elastic scattering. The upscattering cross-section can therefore be obtained after subtracting this elastic component from the total cross-section. In Fig. 6, we show the result of this operation for the neutron wavelength 20 Å. It is seen that in these measurements, we did not reach the neutron energy range where the upscattering cross-section behaves according to the inverse velocity law. This is because of a strong coherent inelastic contribution (at $T \geq 100$ K) in a wide range of wavelengths around 10 Å. Measurements of the total cross-section for these substances at room temperature for neutrons in the wavelength range 200–800 Å demon-

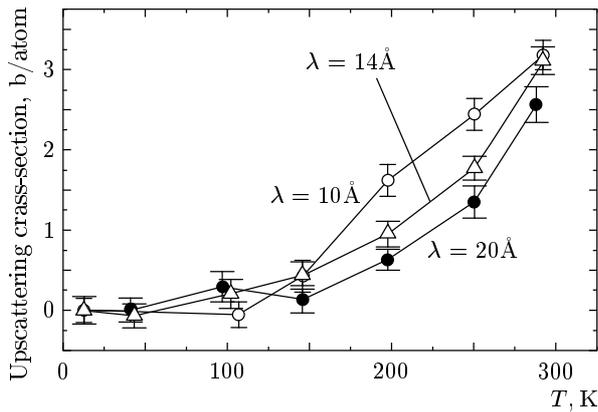


Fig. 6. The upscattering neutron cross-section for POM-310 versus temperature obtained as a result of subtracting the elastic scattering contribution from the total cross-section

strated a good agreement with the inverse velocity law dependence of the cross-section and yielded coincident results with the present measurements at 20 Å for the value of the upscattering cross-section extrapolated to the thermal point at room temperature [28].

At large wavelengths, the upscattering cross-section as a function of temperature, can be taken as a starting point for calculation of the possible upscattering contribution to the UCN loss in liquid-wall traps.

From the upscattering cross-section 3 b/atom for Fomblin at $\lambda = 20 \text{ \AA}$ and room temperature, it follows that $\text{Im } b = \sigma/2\lambda = 0.75 \cdot 10^{-17} \text{ cm}$, which leads to the loss coefficient $\eta \approx 1.25 \cdot 10^{-5}$ (the mean value of the real part of the scattering length for Fomblin was calculated to be $\text{Re } b \approx 5.9 \text{ F}$). The experimental value $\eta = 2.35 \cdot 10^{-5}$ [18] inferred from UCN storage experiments is almost twice the value inferred from the transmission data of the present measurements. Some additional loss processes possibly occur when UCN reflect from the liquid surface: due to some hydrogen contamination of the surface increasing the upscattering probability, or a significant influence of quasi-elastic scattering (small heating [23]) due to the surface excitation of a viscous liquid [29].

Upscattering cross-sections for other liquid fluoropolymers are close to the Fomblin one. Most important is the temperature behavior of the cross-section for low-temperature liquids. It follows from Fig. 6 that at temperature around 190 K, where viscous properties are appropriate for experiments with a UCN bottle, the upscattering cross-section at the wavelength 20 Å is 0.5–0.6 b. For the expected loss coefficient of UCN due to upscattering at this temperature, this gives

$\eta \approx 2 \cdot 10^{-6}$. This figure is an order of magnitude lower than the experimental one for Fomblin [8, 18]. We can therefore hope to decrease the UCN loss correction in the neutron lifetime experiment approaching the value $3 \cdot 10^{-4}$ for the precision of neutron lifetime measurement.

3. INFLUENCE OF QUASI-ELASTIC UPSCATTERING

It was shown in [29] that another possible source of UCN losses in liquid-wall traps — small neutron heating during collisions with the walls [23] — can be explained as a result of neutron interaction with thermal surface excitations of the liquid surface. Quantitative experimental data on UCN quasi-elastic scattering on liquid surfaces are scarce: the differential probability of scattering as a function of the incident UCN energy and of the energy change is not measured yet, the integral probability is known very approximately because absolute calibration of the detecting apparatus is difficult and the range of integration over the incident and final neutron energy is not determined precisely. It is possible, however, to determine parameters of the dynamic model of a viscous liquid with some precision using even this scant information.

First, it is easy to show that in typical experimental situations of our interest, the capillary wave contribution to a small UCN energy change is inessential. For practical needs, the viscosity range of liquid polymers used for UCN storage is between one and dozens poise, and the surface tension of our liquids is about 20 din/cm^2 . As is well known [30, 31], capillary waves are not damped when the dispersion curve $\omega = \sqrt{\sigma/\rho} q^{3/2}$ for capillary waves lies above the line $\omega = (2\eta/\rho)q^2$. Here, ω is the frequency of surface oscillations, σ is the surface tension, η is the viscosity, q is the surface wave vector, and ρ is the density of the liquid. The critical wave vector $q_c = \sigma\rho/4\eta^2$ (about 0.4 cm^{-1} at $\eta = 5 \text{ Ps}$) corresponds to the critical capillary frequency $\omega_c = \sigma^2\rho/8\eta^3$. It is as low as 1 s^{-1} and outside measurement capacity in any neutron experiments. The capillary waves in the energy range of our interest, $0.1\text{--}100 \text{ neV}$ ($10^5 \text{ s}^{-1} \leq \omega \leq 10^8 \text{ s}^{-1}$), are strongly overdamped.

But at small time scales ($t \leq 10^{-5} \text{ s}$), viscous liquids demonstrate elastic properties: they deform under the influence of an external force, and a shear stress then relaxes with a characteristic time τ . Viscoelastic prop-

erties can be described introducing [32] the complex viscosity via the Maxwell formula

$$\eta = \eta_0 / (1 - i\omega\tau), \quad (3)$$

which is simply the Fourier transform of the exponential time dependence of elastic deformations in a viscoelastic liquid.

As shown in [33, 34], taking elastic properties into account leads to significant variability of the spectrum of surface fluctuations: the spectrum of capillary waves is suppressed and narrowed, and energy dissipation of surface vibrations is increased at the expense of arising at higher frequencies of the wave motion of a phonon nature. These elastic effects totally prevail in the frequency range of our interest.

To find the effect of surface fluctuations on the UCN quasi-elastic interaction with a liquid surface, we use the results in [35] for the dynamic structure factor of a viscous liquid,

$$S(\mathbf{q}, \omega) = (2\pi)^3 \langle \xi_{\mathbf{q}, \omega_{\mathbf{q}}}^2 \rangle, \quad (4)$$

where $\langle \xi_{\mathbf{q}, \omega_{\mathbf{q}}}^2 \rangle$ is the mean squared surface fluctuation with the wave vector \mathbf{q} and frequency $\omega_{\mathbf{q}}$. The structure factor is given by

$$S(q, \omega) = \frac{8k_B T \operatorname{Re}[\eta(\omega)] q^3}{|D(q, \omega)|^2} \times \left\{ 1 + \frac{1}{2 \operatorname{Re}[\alpha(q, \omega)]} - 2 \operatorname{Re} \left(1 + \frac{1}{1 + \alpha(q, \omega)} \right) \right\}, \quad (5)$$

where the surface-mode dispersion relation [31, 33, 36–38] is $D(q, \omega) = 0$, with

$$D(q, \omega) = [i\omega + 2\nu(\omega)q^2]^2 - 4\nu(\omega)^2 q^4 \alpha(q, \omega) + \sigma q^3 / \rho. \quad (6)$$

Here, $\nu(\omega) = \eta(\omega) / \rho$ is the kinematic viscosity; the complex frequency-dependent viscosity $\eta(\omega)$ in Eq. (3) crosses from the viscous behavior at low frequencies to the elastic behavior at high frequencies with $\eta_0 = G\tau$ [32, 35] and

$$\alpha(q, \omega) = \sqrt{1 + \frac{i\omega}{\nu(\omega)q^2}}. \quad (7)$$

In expressions (5)–(7), k_B is the Boltzmann constant, T is the temperature, σ is the surface tension, ρ is the density, τ is the liquid-polymer stress relaxation time, and G is the frequency-independent shear modulus of the polymer network.

In this simple Maxwell model, the surface dynamics of the liquid and the interaction of neutrons with

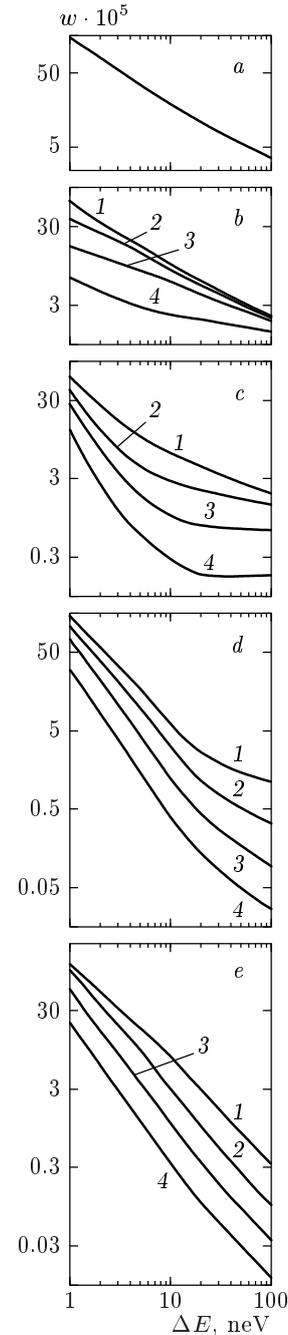


Fig. 7. Results of calculations of the probability (per 1 neV) of quasi-elastic upscattering of a neutron with the energy 50 neV, incident at the angle $\pi/4$ on the surface of room-temperature liquid with the surface tension 20 din/cm². The model of liquid with a complex viscosity was assumed (Eq. (3)) with $\tau = 10^{-3}$ (a), 10^{-5} (b), 10^{-6} (c), 10^{-7} (d), and 10^{-12} s (e); $\eta_0 = 0.3$ (1), 1.0 (2), 3.0 (3), and 10.0 Ps (4)

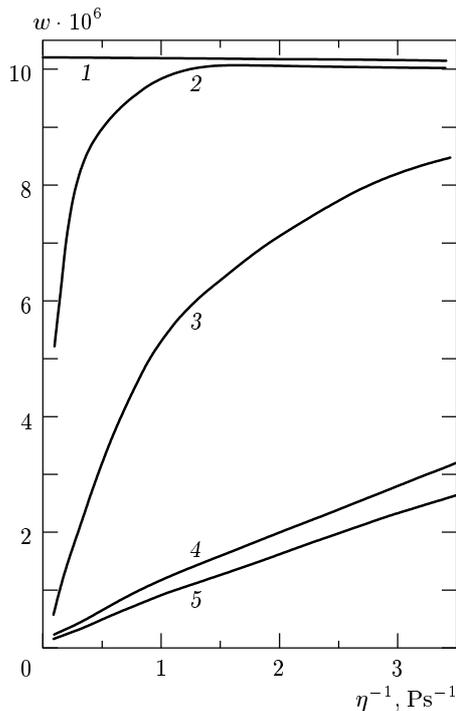


Fig. 8. The probability (per 1 neV) of quasi-elastic upscattering of a neutron with the energy 50 neV incident on the liquid surface at the angle $\pi/4$ with the energy change $\Delta E = 50$ neV as a function of the inverse viscosity η_0^{-1} , each curve for constant $\tau = 10^{-3}$ (1), 10^{-5} (2), 10^{-6} (3), 10^{-7} (4), and 10^{-8} s (5)

the liquid surface are determined for any (\mathbf{q}, ω) by four parameters, T , ρ , G , and τ .

The probability of quasi-elastic reflection of neutrons from liquid surface fluctuations is given by [29]

$$w(\mathbf{k}_0 \rightarrow E) = \frac{16k_{0\perp}}{(2\pi)^3} \frac{1}{2\hbar} \int S\left(q, \frac{\Delta E}{\hbar}\right) k_{\perp q} dq d\phi. \quad (8)$$

In Fig. 7, we show typical results of calculations according to Eq. (8) of the probability of quasielastic upscattering of a neutron with the energy 50 neV, incident at the angle $\pi/4$ on the surface of the room temperature liquid with the surface tension 20 din/cm².

Apart from the possibility (very restricted in view of the lack of experimental information mentioned above) of a quantitative comparison of the calculated probabilities with the experimental data [23], it is now possible to infer some parameters of the dynamic model of liquid. It follows from the recently measured [39] viscosity dependence of quasielastic upscattering from the Fomblin surface demonstrating a close proportionality of the upscattering probability to the inverse viscosity, at least in the viscosity range between 0.5 and

15 Ps. A detailed analysis of the calculated (Eq. (8)) UCN upscattering probability as a function of the stress relaxation time τ and shear modulus G shows that this proportionality is observed only in the τ range $(1-3) \cdot 10^{-7}$ s and the G range 10^6-10^8 din/cm². This behavior is demonstrated in Fig. 8, showing the probability of quasi-elastic upscattering of a neutron with the energy 50 neV incident on the liquid surface at the angle $\pi/4$ with the energy change $\Delta E = 50$ neV as a function of the inverse viscosity η_0^{-1} ; each curve corresponds to a constant τ . A similar picture is observed when the upscattering probability is presented as constant- G curves.

Computational analysis shows that the data for the probability of UCN quasi-elastic scattering on a liquid surface as a function of the incident neutron energy and the energy transfer allow determining the Maxwell model dynamic parameters of a viscous liquid.

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