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INVESTIGATION OF LIQUID FLUOROPOLYMERS AS POSSIBLE MATERIALS FOR LOW TEMPERATURE LIQUID WALL CHAMBERS FOR STORAGE ULTRA COLD NEUTRONS

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1 Introduction

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

The last years brought almost an order of magnitude improving the accuracy of measurement of neutron lifetime[1]. This progress was reached entirely due to application of ultracold neutron (UCN) storage in closed volumes. The review of the neutron lifetime measurements up to 1990 was published by Schreckenbach and Mampe[2] (see also review of Pendlebury[5], on wider fundamental applications of UCN). More recent review of investigations of free neutron decay see in[3, 4]. The most precise measurements of neutron lifetime were carried out with very low surface temperature beryllium and solid oxygen traps[6], or with traps covered with Fomblin oil[7, 8, 9, 10].

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

Losses of UCN when they are stored in traps with walls covered with solid oxygen[6, 12] or cooled graphite[13] also exceed

two-three orders of magnitude the expected ones calculated according to cold neutrons transmission cross sections.

Application of the hydrogen-free perfluoropolyether (PFPE) oil (Fomblin) was first proposed by Bates[14] and tested for neutron lifetime measurement in[15].

Fomblin has chemical formula $CF_3(C_3F_6O)_n(OCF_2)_m OCF_3$, with m/n=20-40, molecular weight $\sim$3000[16].

UCN losses caused by the Fomblin wall collisions in large UCN traps (volume up to 72l) were around 20% (at 10° C) and 10% (at 4° C) of the neutron beta decay in[15] 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 upgraded installation in the same way as in[7, 8].

Careful measurements of UCN losses in Fomblin traps with the aim to obtain UCN energy dependence of the loss coefficient were performed in[18]. It was found that experimental UCN reduced loss coefficient η for UCN with energy E in the trap with boundary potential V, in the expression for the averaged over isotropic angular distribution UCN loss probability:

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

is $\eta = 2.35(0.1) \cdot 10^{-5}$ for the Fomblin oil and $\eta = 1.85(0.1) \cdot 10^{-5}$ for the fomblin grease at 21° C. It is interesting to note that this figures for 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 lower temperature of Fomblin: 4° C, the loss coefficient was as low as η=1.3(1)$\cdot 10^{-5}$, the best one ever obtained experimentally and 2.5 times lower than anomalous loss coefficient in experiments[12].

Using the usual formalism for the complex potential U, de-

scribing UCN interaction with walls

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

(2)

where m is the neutron mass, N_i is the number of nuclei in a unit volume of a wall material, b_i is the coherent scattering length on a bound nucleus of the wall, and σ is cross-section of inelastic processes for neutrons with wavelength λ, it is possible to calculate that part of wall loss coefficient, which is attributed to neutron capture in Fomblin. This part is as low as $3.6 \cdot 10^{-7}$ (the experimental value ReU=106.5 neV[19] was used in this calculation). It means that the main component of UCN losses is inelastic scattering. For fomblin it was observed strong dependence of UCN wall losses on temperature – decrease with lowering temperature of about 3% for a 1° C temperature change[7]. For precision measurement of neutron lifetime, it demands very good wall temperature uniformity over trap surface. 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 in near to solid state[7, 9] which led to significant increasing of UCN losses. The authors[7] found, that below 0° C the Fomblin oil became too viscous for respraying over the wall surface.

But contrary to solid surfaces in the case of liquid Fomblin surface the difference between measured UCN loss coefficients and calculated ones from transmission measurements was not large.

Except for indication[7], that transmission measurements with $\lambda = 60\mathring{A}$ neutrons at 20° C Fomblin had given within the factor 1.5 agreement with UCN losses cross-section, 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 extracted

3

from[7, 18] according to Eq.2 total inelastic UCN cross-section in PFPE, extrapolated to thermal point in accordance with inverse-velocity-law σ_{extr}^{PFPE}=8.65 b ($\approx$0.51 b per atom of PFPE) with the experimental data for "similar" compound – teflon $(CF_2)_n$[20]. For the lowest neutron energy used in these measurements 0.376 meV, at which elastic coherent scattering effect are believed to be negligible (below all the measured in[20] Bragg peaks in the cross-section), the extrapolated to thermal point cross-section per atom is $\simeq$ 0.63 b, which is in satisfactory agreement 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] which contained the report about new recently synthesized fully fluorinated compounds, which have promising physical properties from the point of view of using them for liquid wall UCN traps. These new compounds – perfluorinated polyformaldehydes – have large liquid range and good low temperature properties. Their formula is: $C_4F_9(OCF_2)_nC_4F_9$. Depending on n (between 6 and 10) – the number of OCF_2 units, the boiling temperature of this polymer is varied between 125 and 200° C; the melting point is between -145 and -152° C.

There is hope that at lower temperature - in vicinity of melting point (e.g. -100° C), inelastic upscattering, which is believed is the most important component of UCN losses at collisions with walls of chambers, will decrease several times. Corrections for UCN loss at inferring neutron lifetime from UCN storage data are decreased respectively.

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

In view of lack of any information about dynamical properties of this new compound (as for Fomblin) it was possible to make only very approximate estimation of gain in UCN losses (UCN

upscattering cross-section) with lowering temperature from 10 to -120° C. It was assumed in this estimation that dynamical properties of PFPE and this new polymer are similar, and frequency distribution is described by Debye model. From known specific heat of Fomblin 0.24 kal/(g·grad)[24], it is possible to calculate in standard way the Debye temperature of Fomblin $\simeq 750°$ K, and from the latter, in the incoherent approximation, to calculate the UCN upscattering cross-section. The result of this calculation yields: the upscattering cross-section decreases five times from room temperature to -120° C, and changes as 0.7% per 1° C at room temperature. This latter figure is four times lower than was reported[7], and therefore our estimation seems to be a low estimation of the possible decrease of UCN upscattering with lowering temperature.

To our regret we were unable to obtain in our disposal this[22] substance. But it turned out that the low temperature fluoropolymers with similar properties are produced in Russia[1].

The goal of this work was investigation of several possible low temperature fluoropolymers – candidates for low temperature liquid wall UCN bottle. Measured were: viscosity as a function of temperature, for just viscosity determines the temperature range at which the liquid wall may be practically used, and cold neutron cross sections as a function of temperature.

2   Viscosity and neutron cross sections measurements

Viscosity was measured with the simplest possible method of "sinking ball"[25]. The results are presented in Fig. 1. As is seen

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[1]The investigated substances were produced by the Perm branch of the Russian Scientific Center "Applied Chemistry" and by the State Scientific Institute of Organic Chemistry and Technology, Moscow

the most promising properties have POM-310 and POM. They are the mixtures of complex fluoropolyoximethilenes with general formula $CF_3O(CF_2O)_n(CF_2CF_2O)_m(OCF_2CF_2O)_l CF_3$ 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 is seen from this figure that the most promising liquids: POM and POM-310 have viscosity at $-90°$ C close to viscosity of Fomblin at a temperature near $0°$ C, at which it was still possible to use in the experiments on neutron lifetime measurement[15, 8]. It means that from experimental point of view they are appropriate for using in similar way as it was 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 as low as possible energies in wide temperature range so that it could be possible to infer from these measurements the upscattering part of cross section. The latter may be used for estimation possible UCN losses in storage experiments.

In view of overwhelming flux of delayed thermal neutrons in the direct beam time of flight spectrum below neutron energy $\sim$1meV, diffraction from the stack of mica (artificial fluorophlogopite [26, 27]) with lattice parameter 9.97 Åfor (0,0,1) plane was used for neutron monochromatization. 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 $\sim$1.8mm) is shown in Fig. 2.

The total cross sections were measured at several Bragg angles of neutron diffraction from mica: from $45°$ to $83.5°$. Fig.3 shows the total neutron cross section of three room temperature fluoropolymers as a function of neutron wavelength. There are seen strong coherent effects in wide wave length range up to the largest reached in this measurements - 20Å. This behaviour is

similar to cross section for other well known solid fluoropolymer - teflon. Our measurements of total cross section for teflon are in good agreement with the previous one[20].

In order to obtain the value of upscattering probability for UCN in traps with walls covered with these liquids, the measurements were performed of total neutron cross section in wide temperature range: from 290° down to 10° K and in the wavelength length from 1 Å up to 20 Å. Figs. 4 and 5 show temperature dependence of 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.

In the temperature range of interest (200°-300° K) and wave length larger 10 Å strongly dominating component of inelastic scattering is upscattering (neutron energy $E_n \ll kT$). The cross section at low temperatures (10°-100° K) does not depend of temperature and is entirely elastic scattering. Therefore after subtracting this elastic component from the total cross section the upscattering cross section may be obtained. Fig. 6 shows the result of this operation for neutron wavelength 20 Å. It is seen that we did not reach still in this measurements the neutron energy range where the upscattering cross section behaves according to inverse velocity law. The reason for that lies in the strong coherent inelastic contribution (at $T \geq 100$ K) in a wide range of wavelengths around 10 Å. The later measurements of total cross section of these substances at room temperature for neutrons in the wave length range 200-800 Å demonstrated good agreement with inverse velocity law dependence of cross section and yielded coincident results with the present measurements at 20 Å for the value of extrapolated to the thermal point upscattering cross section at room temperature[28].

The upscattering cross section at large wavelengths as a function of temperature may be taken as a starting point for calculation of possible upscattering contribution to UCN loss in the liquid wall traps.

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

Upscattering cross section for other liquid fluoropolymers are close to the Fomblin figures. The most important is temperature behaviour of cross section for low temperature liquids. It follows from Fig. 6 that at the temperature around 190° K where viscous properties are appropriate for experiments with UCN bottle, upscattering cross section at the wave length 20 Å is 0.5-0.6 b. It gives for the expected loss coefficient of UCN due to upscattering at this temperature $\eta \approx$2·10^{-6}.

This figure is an order of magnitude lower than experimental one for Fomblin[18, 8]. Therefore we can hope to decrease respectively the UCN loss correction in the neutron lifetime experiment approaching to the value 3·10^{-4} for the precision of neutron lifetime measurement.

3   Influence of quasielastic upscattering

It was shown[29] that another possible source of UCN losses in liquid wall traps - small neutron heating during collisions with

the walls[23] - may be explained as a result of neutron interaction with thermal surface excitations of liquid surface. Quantitative experimental data on UCN quasielastic scattering from liquid surface are scarce: differential probability of scattering as a function of incident UCN energy as well as of energy change is not measured yet, integral probability is known very approximately in view of difficulty of absolute calibration of detecting apparatus and not precisely determined range of integration over incident and final neutron energy. It is possible however using even this scant information to determine with some precision parameters of dynamic model of viscous liquid.

First of all it is easy to show that in typical experimental situations of our interest capillary wave contribution to small UCN energy change is inessential. From practical needs viscosity range of liquid polymers used for UCN storage is between 1 and dozens Ps, 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 for capillary waves $\omega = \sqrt{\sigma/\rho} \cdot q^{3/2}$ lies above the line $w = (2\eta/\rho)q^2$. Here ω is frequency of surface oscillations, σ is surface tension, η is viscosity, q is the surface wave vector and ρ is density of liquid. Critical wave wave vector $q_c = \sigma\rho/4\eta^2$ (~ 0.4 cm^{-1} at η=5 Ps) corresponds to critical capillary frequency $\omega_c = \sigma^2\rho/8\eta^3$. It is as low as 1 s^{-1} and out of any capability to be measured in any neutron experiments. The capillary waves in the energy range of our interest: 0.1-100 neV ($10^5 s^{-1} \leq \omega \leq 10^8 s^{-1}$) are strongly overdamped.

However at small time scales (t$\leq 10^{-5}$ s) viscous liquids demonstrate elastic properties: they deform under the influence of external force, and shear stress then relax with characteristic time τ. Viscoelastic properties may be described introducing[32] the complex viscosity by means of Maxwell formula

$$\eta = \eta_0/(1 - i\omega\tau), \tag{3}$$

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

As was shown in[33, 34] the account of elastic properties 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 more high frequencies of the wave motion of phonon nature. These elastic effects totally prevail in the frequency range of our interest.

To find the effect of surface fluctuations on UCN quasielastic interaction with liquid surface we use the results of derivation[35] of dynamic structure factor for viscous liquid:

$$S(\vec{q}, \omega) = (2\pi)^3 < \xi^2_{\vec{q},\omega_{\vec{q}}} >, \tag{4}$$

where $< \xi^2_{\vec{q},\omega_{\vec{q}}} >$ is the mean squared surface fluctuation with wave vector $\vec{q}$ and frequency $\omega_{\vec{q}}$. The structure factor has the form:

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

where the surface mode dispersion relation[31, 33, 36, 37, 38] is $D(q, \omega) = 0$, with

$$D(q, \omega) = (i\omega + 2\nu(\omega)q^2)^2 - 4\nu(\omega)^2 q^4 \alpha(q, \omega) + \sigma q^3/\rho. \tag{6}$$

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

$$\alpha(q, \omega) = \left(1 + \frac{i\omega}{\nu(\omega)q^2}\right)^{1/2}. \tag{7}$$

In these expressions k_B is the Boltzmann constant, T is the temperature, σ is surface tension, ρ is density, τ is 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 liquid for any $(\vec{q}, \omega)$, as well as the interaction of neutrons with liquid surface is determined by four parameters T, ρ, G and τ.

The probability[29] of quasielastic reflection of neutrons from liquid surface fluctuations:

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

In Fig.7 are shown typical results of calculations according to Eq. 8 of probability of quasielastic upscattering of neutron with energy 50 neV, incident at the angle $\pi/4$ at the surface of room temperature liquid with surface tension 20 din/cm^2.

Apart from possibility (very restricted in view of above mentioned lack of experimental information) of quantitative comparison of calculated probabilities with experimental data[23] it is possible now to infer some parameters of dynamic model of liquid. It comes from recently measured[39] viscosity dependence of quasielastic upscattering from Fomblin surface which demonstrates close proportionality of upscattering probability to inverse viscosity at least in viscosity range between 0.5 and 15 Ps. Detailed analysis of calculated (Eq. 8) UCN upscattering probability as a function of stress relaxation time τ and shear modulus G shows that such proportionality is observed only in the τ range $(1\text{-}3)\cdot10^{-7}$s, and G range $(10^6\text{-}10^8)$din/cm^2.

Fig.8 demonstrates this behaviour. It shows the probability of quasielastic upscattering of neutron with energy 50 neV incident at the liquid surface at the angle $\pi/4$ with energy change $\Delta E=50$ neV as a function of inverse viscosity η_0^{-1}, each curve for constant τ. Similar picture is observed when upscattering probability is presented as curves for constant G.
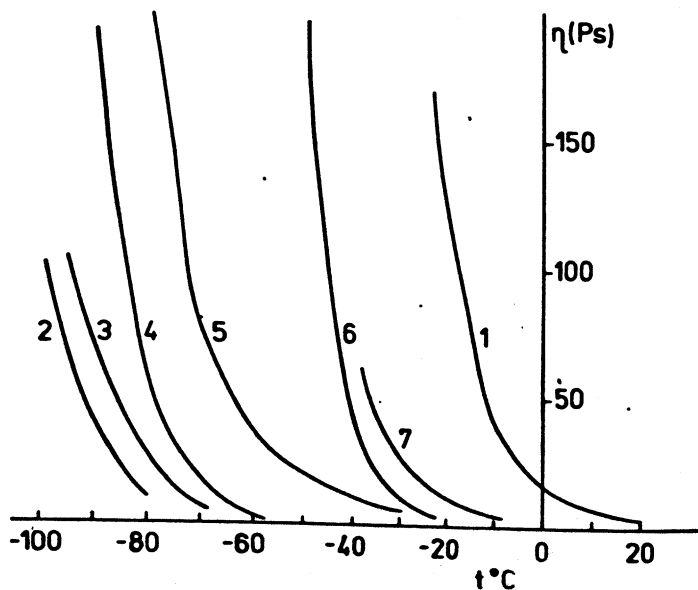
11

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 different chemical content.
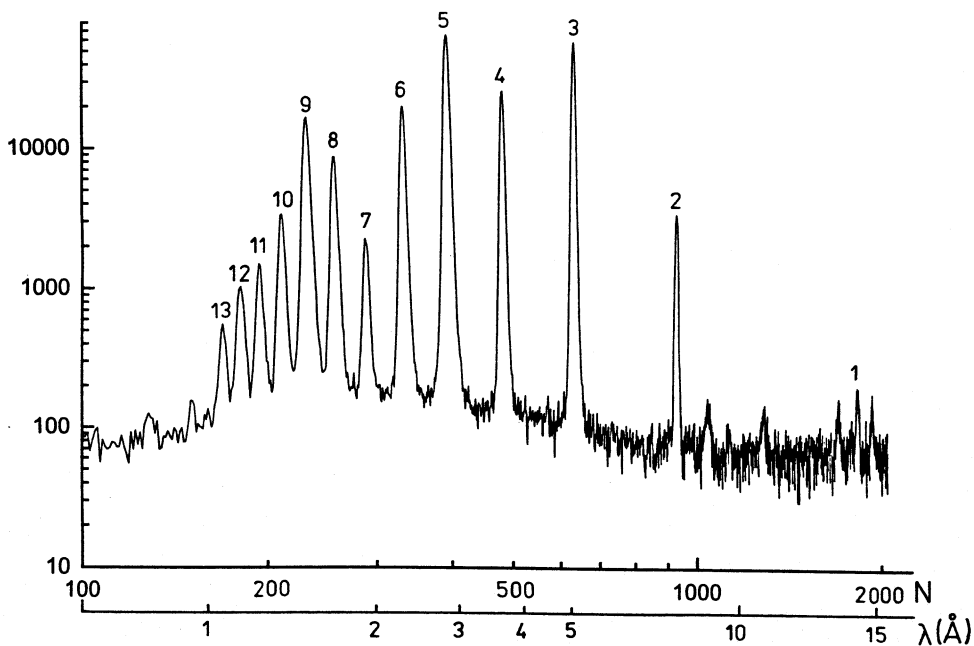
Fig.2.Time of flight spectrum of neutrons diffracted from the stack of artificial fluoroflogopite at the Bragg angle 45°; numbers indicate the order of diffraction.
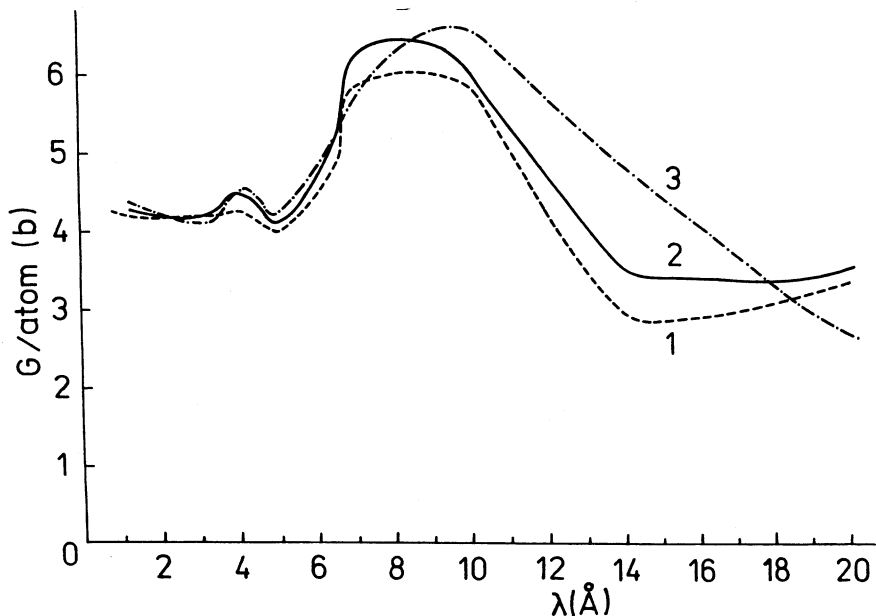
Fig.3.Total neutron cross section of liquid fluoropolymers at the temperature 290° K as a function of neutron wavelength: 1 - Fomblin, 2 - POM-310, 3 - POM.
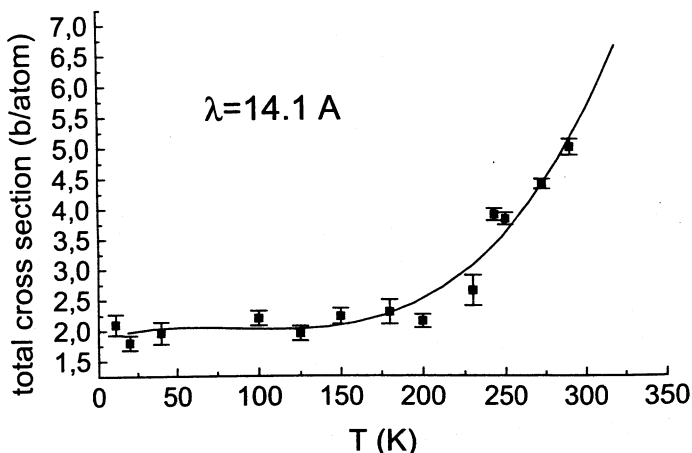
Fig.4.Total neutron cross section of Fomblin vs temperature for neutron wavelength 14.1 Å.
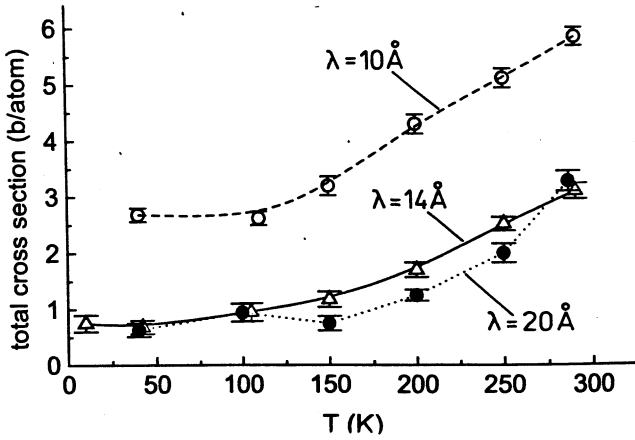
Fig.5.Total neutron cross section of POM-310 vs temperature for several neutron wavelengths.
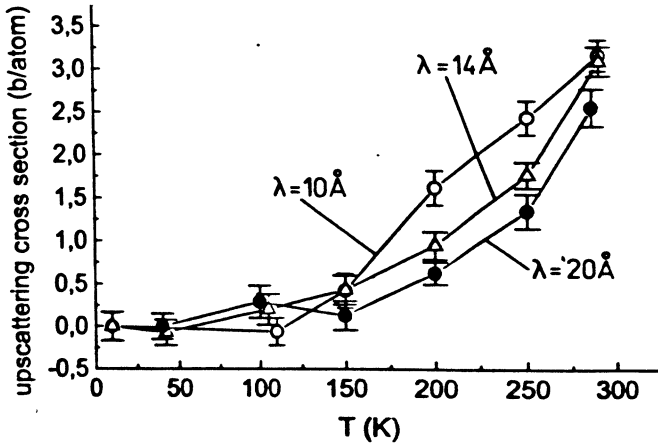
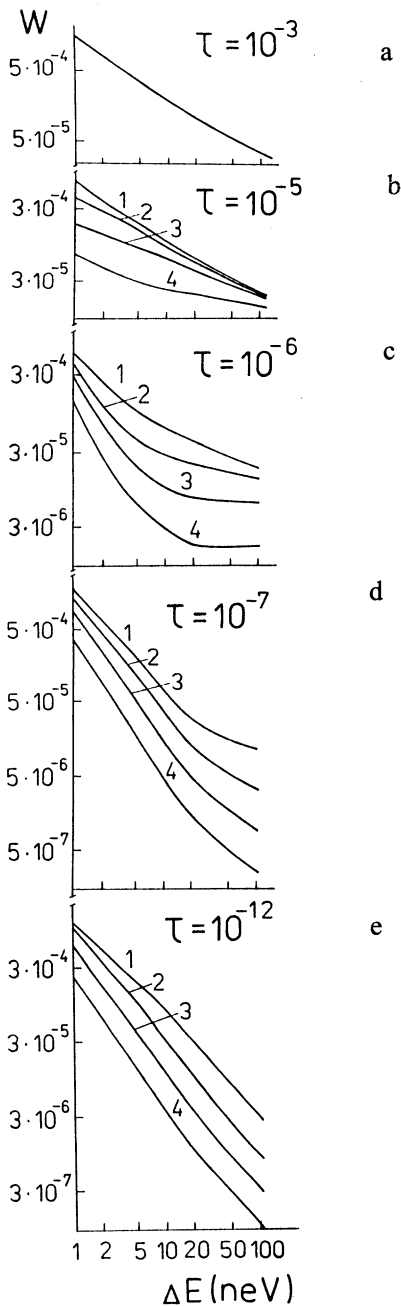Fig.6.Upscattering neutron cross section for POM-310 vs temperature obtained as a result of subtracting the elastic scattering contribution from total cross section.

15

Fig.7. Results of calculations of probability of quasielastic up-scattering of a neutron with energy 50 neV, incident at the angle $\pi/4$ at the surface of room temperature liquid with surface tension 20 din/cm^2. Model of liquid with complex viscosity was assumed (Eq. 3):

a - $\tau = 10^{-3}$ s,
b - $\tau = 10^{-5}$ s,
c - $\tau = 10^{-6}$ s,
d - $\tau = 10^{-7}$ s,
e - $\tau = 10^{-12}$ s,
1 - $\eta_0 = 0.3$ Ps,
2 - $\eta_0 = 1.0$ Ps,
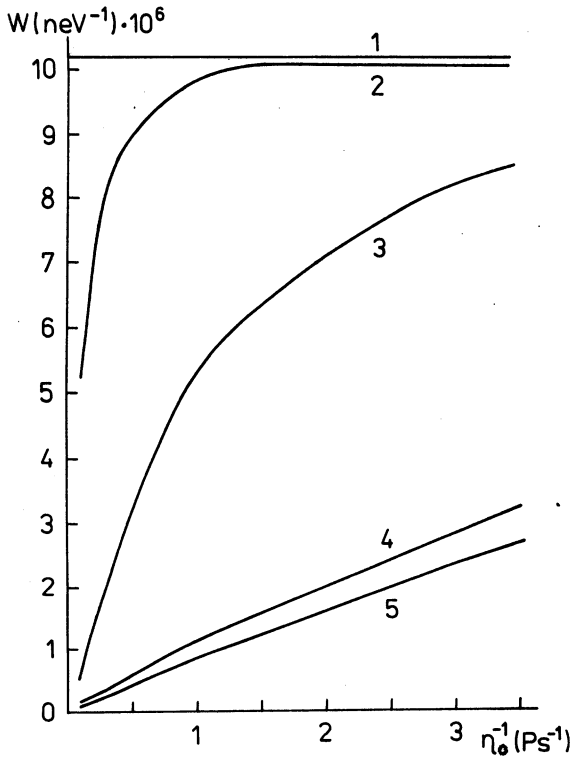3 - $\eta_0 = 3.0$ Ps,
4 - $\eta_0 = 10.0$ Ps.

16

Fig.8.Probability of quasielastic upscattering of neutron with energy 50 neV incident at the liquid surface at the angle $\pi/4$ with energy change $\Delta E=50$ neV as a function of inverse viscosity η_0^{-1}, each curve for constant τ: 1 - $\tau=10^{-3}$s, 2 - $\tau=10^{-5}$s, 3 - $\tau=10^{-6}$s, 4 - $\tau=10^{-7}$s, 5 - $\tau=10^{-8}$s.

Computational analysis shows that having data for the probability of UCN quasielastic scattering from liquid surface as a function of incident neutron energy and of energy transfer permits to determine the Maxwell model dynamic parameters of viscous liquid.

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Покотиловский Ю. Н.   E3-2002-59
Исследование жидких фторполимеров
как возможных материалов для низкотемпературных камер
с жидкими стенками для хранения ультрахолодных нейтронов

Исследовано несколько безводородных фторполимеров с низкой температурой плавления с точки зрения их возможного использования в качестве материалов стенок ловушек ультрахолодных нейтронов с низкими потерями. Измерены вязкость в диапазоне температур 150–300 K и нейтронные сечения в диапазоне температур 10–300 K для длин волн нейтронов 1–20 Å. Сделано заключение об их возможном применении для ловушек ультрахолодных нейтронов. Рассмотрено квазиупругое отражение нейтронов от поверхности вязкой жидкости в рамках модели Максвелла.

Работа выполнена в Лаборатории нейтронной физики им. И. М. Франка ОИЯИ.

Pokotilovski Yu. N.   E3-2002-59
Investigation of Liquid Fluoropolymers as Possible Materials
for Low Temperature Liquid Wall Chambers
for Storage Ultra Cold Neutrons

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

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR.

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