Highlighting a Solid-Like Behavior in RTILs: Tri-octylmethylammonium Bis(trifluoromethanesulfonyl)imide TOMA-TFSI

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Identification of Shear Elasticity at Low Frequency in Liquid n-Heptadecane, Liquid Water and RT-Ionic Liquids [emim][Tf2N]

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ABSTRACT

By improving the boundary conditions between the sample and the substrate whereby the stress and the measurement are transmitted, novel dynamic experiments enable the detection of subtle properties as the identification of a no-zero low frequency shear elasticity in various liquids. This study is for the first time extended to molecular liquids as simple liquids (heptadecane), room temperature ionic liquids ([emim][Tf2N]) and water, confirming the generic character of the elastic properties identified at the sub-millimeter away from any phase transition.

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1. Introduction

Little work has been done on quantitative methods to probe long time scales in liquids. Indeed, the low frequency domain is typically unexplored since liquids are not supposed to exhibit shear elasticity in contrast to solids or plastic fluids that require a finite stress to flow. However, several recent results have revived the assumption of long-range elastic correlations and therefore of long time scales, hitherto neglected in the liquid state even away from any phase transition. The measurement of macroscopic finite shear elasticity at the sub-millimeter scale has been essentially enabled by minoring the disturbance of the liquid during the mechanical solicitation and by improving the boundary conditions of the liquid to the substrate [1–5]. This method was successfully applied to glass formers (polymer melts, polypropylene glycol, o-terphenyl, glycerol) revealing in the low frequency range (typically 10^{-2}–10^2 rad/s), a finite shear modulus and a finite viscous modulus. The shear modulus being at least one order of magnitude higher than the viscous modulus, the elasticity dominates the mechanical response.

In the present paper, we extend this analysis to a series of three new molecular liquids, including water. We use the improved boundary method [2] and examine for the first time the shape of the shear stress signal generated by the liquid during the sinusoidal shear strain solicitation. The evolution of the signal is observed from low to high strain amplitudes, thus evolving from a solid-like behavior up to the regime where a purely viscous behavior is recovered.

The three chosen fluids represent 3 large classes of liquids (Table 1): a Van der Waals liquid: the n-heptadecane ($C_{17}H_{35}$); a salt, more particularly a room temperature ionic liquid (RTIL): the 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([emim][Tf2N]); and an H-bond liquid: distilled water probed at room temperature. We reveal in all cases, a weak but non-ambiguous solid-like low frequency response at weak shear strain solicitation. The present work thus extends, details and generalizes the identification of solid-like correlations previously reported above the melting point, of glycerol [4], of o-terphenyl [5], of polypropylene glycol [3] and more generally in polymer melts [1,3].
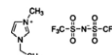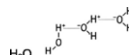
2. Experimental

We aim at presenting dynamic properties of liquids close to their equilibrium state. For that, we analyze the response of the liquid submitted at low frequency, to a mechanical impulse (low shear strain stresses) as non-disturbing as possible to keep intact the material properties (linear conditions) [6]. A strong interaction between the liquid and the substrate is required since the efficiency of the transmission of the strain from surface to the sample, and thus the validity of the measurement itself is completely dependent on the interaction between the surface and the material. The boundary conditions are optimized by using alumina surfaces to provide total wetting properties [7]. The total wetting favours a direct contact of the liquid to the surface, expulsing the air layer formed by trapped microbubbles around asperities and at the interface between the liquid and the substrate (Fig. 1). The trapped air favors an interfacial disruption during the mechanical solicitation. It is therefore important that the liquid forms strong bonds to the surface to optimize the stress transmission.

The sample is placed in the gap between two coaxial disk-like alumina surfaces. The zero gap is set by contact without sample; the

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Table 1
Comparative table of the characteristics of the three liquids.

Class	Van der Waals liquid	Ionic liquid	H-bond liquid
Samples	Heptadecane	1-Ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide: [emim][Tf2N]	Water
Chemical formula	$C_{17}H_{36}$		H_2O
Supplier	Aldrich	Aldrich	Distilled water
Molecular weight	$M = 240$ g.mol^{-1}	$M = 391.3$ g.mol^{-1}	$M = 18$ g.mol^{-1}
Bulk Transition temperatures (atm. pressure)	Crystal $> T_m = 21$ °C $>$ liquid	$T_g = -87$ °C, $T_m = -18$ °C	Crystal (ice point $> T_m = 0$ °C $>$ liquid
Working temp.	24 °C (liquid)	24 °C (liquid)	21.5 °C (liquid)

error is positive and less than 0.005 mm. A shear strain of amplitude γ_0, is transmitted to the sample by imposing a rotating sinusoidal motion of variable frequency (ω) by contact with one disk (strain imposed mode). The second disk is immobile and coupled to a sensor. It measures the stress transmitted by the sample via the torque (σ) transmitted by contact to the disk. Oscillatory motion and torque measurement are provided by a replace by "rheometer" (Ares II). Simultaneously, a 7-digit voltmeter (Keitley; rate: 300 data/s) measures the voltage of the motor imposing the oscillation (input wave associated to the strain amplitude), while another 7-digit voltmeter measures the voltage associated to the sensor (output wave associated to the torque). This setup permits the simultaneous measurement of the shear strain and to the shear stress signals, and of the dynamic profile using the conventional relationship: $\sigma(\omega) = G_0.\gamma_0.sin(\omega.t + \Delta\phi)$ with $\sigma(\omega)$ is the shear stress, G_0, the shear modulus, γ_0 the strain amplitude defined as the ratio of the displacement to the sample gap and $\Delta\phi$ the phase shift between the input and the output waves. This equation can be also expressed in terms of shear elastic (G') and viscous (G'') moduli: $\sigma(\omega) = \gamma_0.(G'(\omega).sin(\omega.t) + G''(\omega).cos(\omega.t))$, with G' the component in phase with the strain, and G'' the out of phase component. It should be stressed that the formalism in terms of G' and G'' supposes that the resulting stress wave keeps the shape of the imposed strain wave (sinusoidal-like). We will in particular show that the study of the wave shape is very instructive since the output stress wave keeps actually a sin shape in two extreme situations only, corresponding to the very weak and to the high strain fields respectively.

3. Results

3.1. Application to the alkane: the n-heptadecane

Fig. 2a displays over one period, the input sin strain wave and the output shear stress wave (displayed with the same amplitude as the strain wave) of the n-heptadecane at 24 °C solicited at low strain
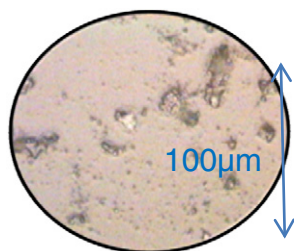

Fig. 1. Microscopic photograph of the wetting surface (Al_2O_3). The surface is flat at a molecular scale with small dispersed irregularities of ± 0.4 μm height.

amplitude ($\gamma_0 = 2\%$) and using total wetting boundary conditions. At this temperature, the n-heptadecane is liquid [8]. The first order transition from crystal to liquid state can be optically determined at $T_f = 21$ °C. With seventeen atoms of carbon, the heptadecane (above T_f) belongs to the category of ordinary alkyl liquids. This alkyl liquid presents the advantage of having a low vapor tension, and therefore a low volatility which is useful when the measurement requires long times. Fig. 2a shows that both input and output signals are almost superposed indicating a quasi-instantaneous sample response ($\Delta(\Phi) = 15°$ – the viscous character dominates when $\Delta(\Phi) > 45°$), i.e. a solid-like response. This result indicates that when the liquid is firmly anchored on the surfaces, its response to a weak mechanical solicitation is solid-like. A similar in-phase signal is observed over the wide range of frequency tested in Fig. 2b. Since the output shear stress is sinusoidal, a description in terms of elastic G' and viscous G'' moduli (linear regime) is justified. Fig. 2b gathers the evolution of G' and G'' versus frequency. It indicates that the shear modulus G' dominates the viscous component. Both are weakly dependent on the frequency in agreement with a solid-like response and with the quasi in-phase shear stress output wave depicted in Fig. 2a. This result is coherent with those previously obtained on glass formers (glycerol, o-terphenyl, polymer melts). Different interpretations have been proposed, in particular that the solid-like behavior would be related to clustering effects away from the glass transition [9,10]. The n-heptadecane excludes an effect of glass transition since the material is crystalline by lowering the temperature. An interpretation in terms of long range intermolecular interactions has to be privileged in agreement with the early assumptions of Derjaguin [11].

The solid-like signal is delicate and can be easily shear-melt by increasing the strain amplitude as previously observed by different authors [1,3–5,9,12]. The evolution of the stress wave by increasing the strain amplitude is for the first time presented; Fig. 2c displays the stress wave at weak, moderate and strong strain amplitudes for the n-heptadecane.

At low shear strain, both input (shear strain) and output (shear stress) waves are quasi-superimposable (Fig. 2c at left). At intermediate strain rates ($\gamma = 10\%$, middle figure of Fig. 2c), the output wave is no more sinusoidal but is distorted with apparent harmonics. The occurrence of the distorted signal coincides with the entrance in the non-linear regime where the values of G' and G'' are strongly dependent on the strain rate. Since the signal is no more sinusoidal, a modeling in simple terms of G' and G'' has no physical meaning. An accurate observation of the wave distortion shows that harmonics can be interpreted as the result from a truncation/recombination of the primitive wave. A part of the backward side of the wave is cut and the lacking part seems to be stuck in advance to the phase, suggesting a "slip-stick" mechanism, i.e. an alternation of a sliding state and a stick state [13]. This process is an alternating succession of a solid friction of stored energy and of a fast release of stored energy. This effect is typically related to elastic effects stored in the material, i.e. in the liquid.
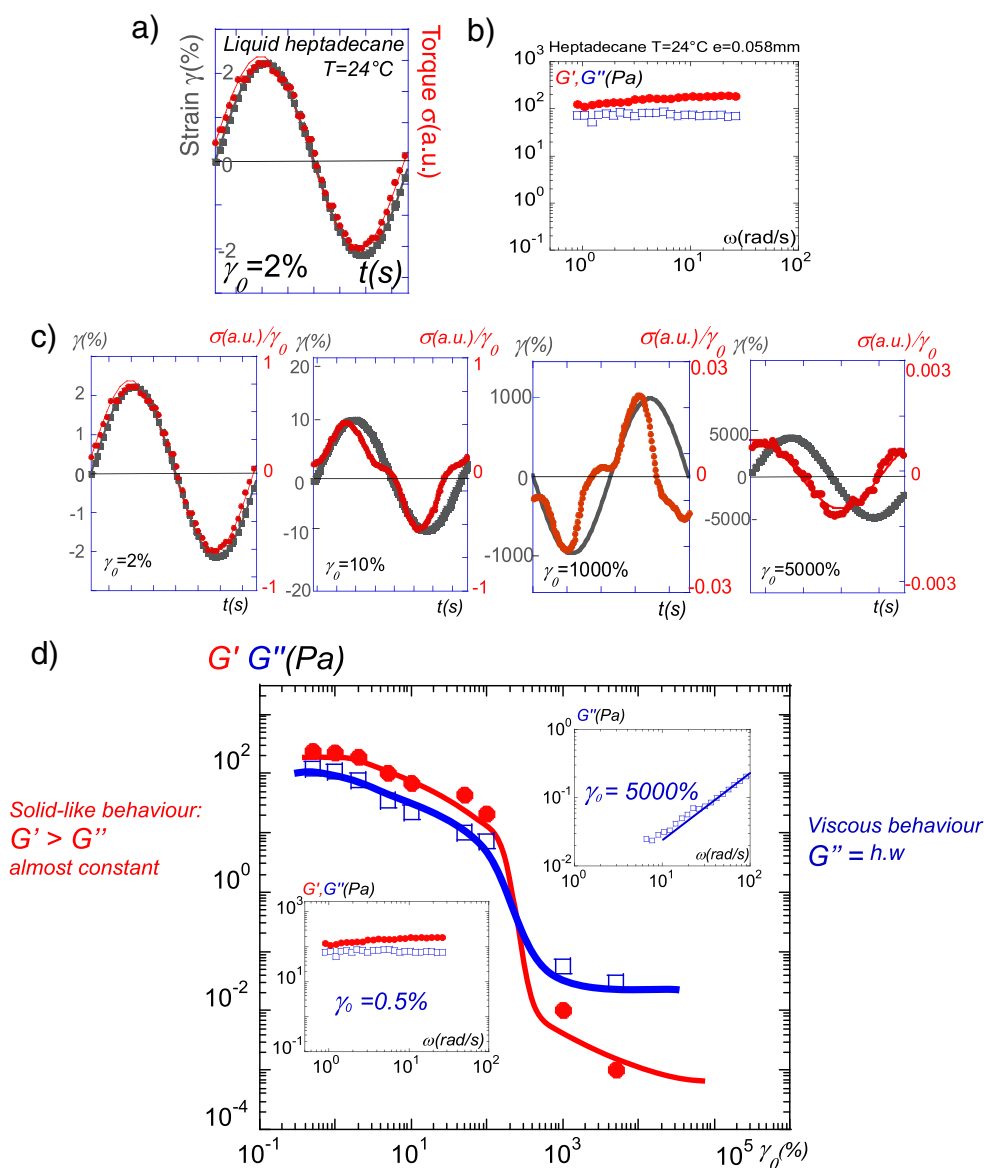
Fig. 2. a: Low strain solid-like response obtained in liquid n-heptadecane at 24 °C at 0.058 mm thickness and 2% shear strain. The input sinusoidal wave (■) corresponds to the strain amplitude and the output shear stress σ(a.u) (●) wave corresponds to the sample response measured by the torque transmitted to the sensor. The continuous lines correspond to sinusoidal fits. The phase shift $\Delta\phi$ of the output signal is in advance of 15°.2b: Treatment of the low strain signal in terms of elastic (G': ●) and viscous (G'': □) moduli of heptadecane at 3 °C above the crystallization ($Tf = 21$ °C) temperature and 0.058 mm gap thickness. Measurement carried versus frequency.2c: From left to right: input sin wave (■: strain amplitude γ (%)) and output shear stress wave (●: shear stress: σ (a.u.)) corresponding to the liquid response of the n-heptadecane at 24 °C measured at different imposed strain amplitudes ($\gamma_0 = 1$, 10, 1000 and 5000%; $e = 0.058$ mm). The stresses are normalized by their amplitudes. Similar behaviors are observed with other fluids (polypropylene glycol, water, ionic liquids …). The continuous red line in the first and the last figures corresponds to a modeling by a sinusoidal fit.2d: Transition from solid-like to viscous behavior induced by increasing the strain amplitude (γ_0).The data points display the shear (G' (●)) and the viscous (G'' (□)) moduli at 1 rad/s frequency. At low strain amplitude, a solid-like behavior is observed. The moduli in the intermediate non-linear regime are not presented since the signal is not a sinusoidal wave. At very large strain amplitudes, an apparent viscous behavior is displayed.

At large shear strain ($\gamma = 5000$%), the front wave completely collapses, only the part in advance of the phase remains. In this regime, the wave recovers a sinusoidal shape with a phase advance of about $\pi/2$ (Fig. 2c at right). This output sin wave can be expressed in terms of G' and G''. The associated spectrum displays a viscous behavior as indicated in the right inset measured at $\gamma = 5000$%. The curve obeys to the ω-scaling with a slope of $\eta = 2.2 \cdot 10^{-3}$ Pa.s, of the order of the tabulated dynamic viscosity value ($\eta = 3.4 \cdot 10^{-3}$ Pa.s) [14]. It is interesting to note that the evolution from the low strain amplitude sinusoidal wave to the large strain amplitude sinusoidal-like wave does not correspond to a simple shift of the phase but to a strong modification of the signal which is no more identifiable as a sinusoidal wave (Fig. 2c).

Fig. 2d summarizes the transition from solid-like to viscous behavior viewed from the evolution of the viscoelastic moduli of the n-heptadecane. This representation has a physical meaning as long as the stress wave reproduces the shape of the input strain wave. Similar evolutions are observed on the two other liquids as a function of the strain amplitude, but are not represented for sake of clarity.

3.2. The ionic liquid: [emim][Tf2N]

The second liquid belongs to the group of ionic liquids, dominated by Coulomb interactions. [emim][Tf2N] is a typical room temperature ionic liquid (RTIL), chosen for its relatively low hygroscopy. Ionic liquids have recently gained a huge interest because of new molecules
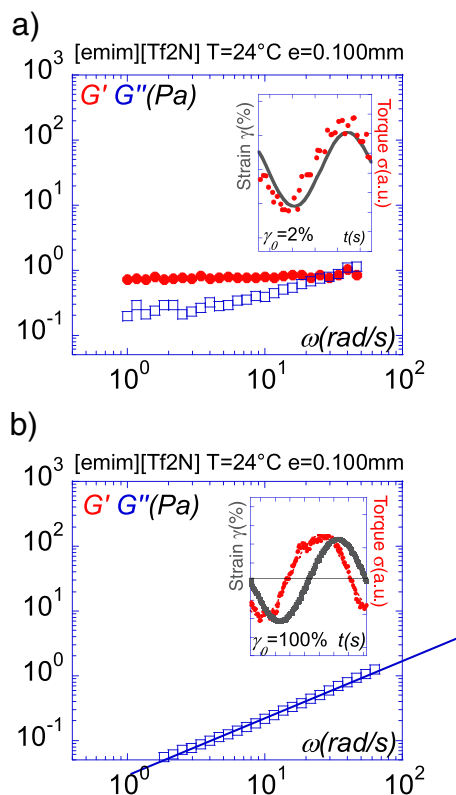
a)

b)

Fig. 3. a: Low strain frequency dependence of the moduli of [emim][Tf2N] at 0.100 mm gap thickness and $\gamma = 5\%$. At low frequency, the shear modulus G' (●) is independent of the frequency and higher than G'' (□). The inset displays the input strain wave (■) and output shear stress (●) wave at ($\omega = 6$ rad/s).3b: Large strain frequency dependence of the moduli of [emim][Tf2N] at 0.100 mm gap thickness and $\gamma = 100\%$. The viscous modulus G'' (□) obeys to the ω-scale law (dotted line). The shear modulus G' is several decades below the viscous modulus at high frequency. The inset displays the input strain wave (■) and output shear stress (●) wave at ($\omega = 1.7$ rad/s).

providing liquid properties at room temperature [15–17]. Their physicochemical properties are also very attractive since they are generally characterized by nonvolatility, nonflammability, thermal stability, and excellent solvating properties. The understanding of these novel ionic liquids and of their physical properties is currently a very active investigation field that arises numerous questions. Some even wonder why these ionic liquids are liquid [18], or if they are really liquids ranging from "normal" viscous liquids to glassy or liquid crystalline materials [19]. Deviations from a Maxwell behavior are observed at low frequencies indicating a topological network. Similarly light scattering investigations [20] indicate long range structures reminding the clusters observed by E.W. Fischer [21]. Elastic [17] and inelastic neutron scatterings [22] seem to indicate a weak frontier between liquid and solid states in terms of structural correlations and of observation of slow dynamic mode.

The inset in Fig. 3a displays the input sin wave and the output shear stress wave of the ionic liquid: 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide: [emim][Tf2N] measured at room temperature at 0.100 mm gap thickness and 2% strain amplitude.

The output stress wave at weak mechanical solicitation exhibits a relatively noisy sinusoidal signal in phase with the input strain wave. The dispersion of the data points and the relatively low moduli (Fig. 3a) might be explained by a less efficient wetting due to the high viscosity of the RTIL. However, the shear stress wave keeps the sinusoidal shape of the strain wave with a phase shift lower than the accuracy. The sample response is unambiguously solid-like. The modeling of the output shear stress by a sinusoid wave, enables the extraction of the elastic G' and the viscous G'' moduli (Fig. 3a). At low frequency, the shear modulus G' dominates the viscous component.
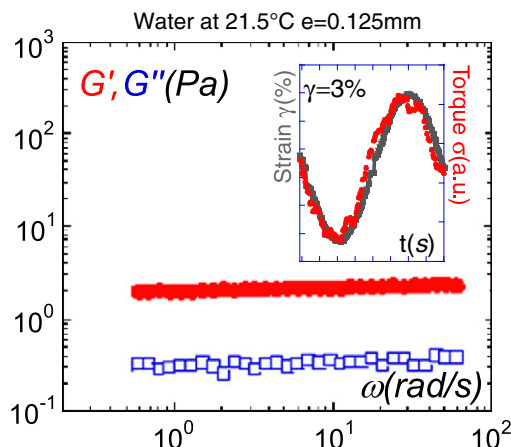

Fig. 4. Low strain frequency dependence of the viscoelastic moduli of liquid water measured at room temperature (21.5 °C), at 0.125 mm gap thickness and 2.5% strain amplitude. The shear modulus G' (●) is independent of the frequency and higher than G'' (■). The inset displays the input strain wave (■) and output shear stress (●) obtained at low strain ($\omega = 1$ rad/s).

Both are weakly dependent on the frequency obeying to a low frequency (linear) solid-like response.

At high strain amplitude (Fig. 3b), the low frequency shear modulus collapses similarly as it was observed for the n-heptadecane (scheme of Fig. 2d) and in agreement with several previous observations [1–4,9], giving rise to a second linear domain characterized by a dominant viscous modulus fitting with the conventional ω-scale behavior, the slope of which indicating the dynamic viscosity ($G'' = \eta.\omega$). The value estimated from the linear fit of Fig. 3b ($\eta = 0.020$ Pa.s at 24 °C) is in agreement with the tabulated value [15].

The identification of a low frequency solid-like response at more than 40 °C above its melting point is an important information in the current debate on RTILs. The novelty of these materials and the interest that they create are an opportunity to redefine the relevant parameters not only in ionic liquids by more generally of the "liquid" state.

3.3. Application to distilled water

The last example concerns the most ubiquitous and most studied liquid on Earth: the water. It is out of purpose to summarize here several decades of studies about the physico-chemistry properties of water. It has been established that the properties in confined geometry (capillaries, at interface or in thin layers) are specific and differ from the "bulk" properties [23]. These measurements reveal scale-dependent interactions at molecular scale or at a multiple of that as reported for example in Surface Forces Apparatus experiments [24–26]. These studies are restricted to several molecular thick layers, i.e. typical layers of less than the micron. At this length scale, several authors found an increased viscosity up to 1000–10,000 Å away from the surface. These results were interpreted by surface-induced effects and some by intrinsic properties [24,27]. Concerning mechanical approaches (Surface Force Apparatus, flow measurements), the measurements were mainly obtained without wetting considerations of the interactions at the boundary. By improving the interactions between the liquid and the surface, the present results on liquid water report on subtle elastic properties at several hundred microns length scale (Fig. 4). These new data invite to reconsider the previous interpretations in terms of surface-induced effects versus intrinsic properties.

The inset in Fig. 4 shows quasi-superposed input and output waves. This solid-like response is obtained because the water is gently probed (low strain amplitude) between attractive surfaces; i.e. the liquid is firmly anchored on the surfaces. This sinusoidal

output shear stress enables a description in terms of elastic G' and viscous G'' moduli (linear regime) (Fig. 4). The shear modulus G' dominates the viscous component. Both are weakly dependent on the frequency obeying to a (linear) solid-like response, in agreement with the in-phase signal depicted in the inset. A detailed description of water is beyond the present scope and will be detailed in another letter.

4. Conclusions

Finite shear elasticity has been measured by applying a soft mechanical shear stress to several molecular liquids (n-heptadecane, room temperature ionic liquid [emim][Tf2N] and distilled water) usually considered as purely viscous in the low frequency range away from any phase transition. The shear elasticity is non-ambiguously identified by a quasi-in phase response to a low amplitude sinusoidal mechanical shear strain. The conservation of the input strain sinusoidal shape to the output shear stress wave enables an analysis in terms of (in-phase) elastic and (out-of-phase) viscous moduli. The study of the low frequency range (10^{-1}–10^2 rad/s) indicates in agreement with the quasi-in-phase response, that the shear modulus G' dominates the viscous component, both G' and G'' being weakly dependent on the frequency. The predominance of the shear modulus indicates that these liquids exhibit primary a solid-like response. The low frequency shear modulus is of the order of a hundred Pascal for the n-heptadecane and about 2 Pa for the water. These values are extremely weak compared to "normal" solids characterized by elastic moduli of the order of several GPa or compared to the solid response of liquids at MHz or GHz solicitation frequency [28]. Nevertheless, the low frequency elasticity is of prior importance since it tells that liquids resist to a shear flow before flowing.

Because the low frequency elasticity is measured at several tens to hundred microns length scale, surface-induced effects are excluded. Because simple molecules such as the water, the n-heptadecane and the room temperature ionic liquid exhibit the low frequency elasticity, it is a generic property. The measurement of elasticity in RTIL [emim][Tf2N] should constructively contribute to current debates on the true nature of ionic liquids [19] in which by definition oscillates between "normal" viscous liquids and glassy or liquid crystalline materials.

Only few experiments report on this low frequency elasticity. Actually, the measurement of an elastic response at macroscopic scale remains a hard experimental challenge that requires a high care of the boundary conditions between the surface and the liquid [2,3]. This delicate signal is obtained at low strain amplitude, at the sub-millimeter scale and using total wetting surfaces. These conditions improve the transmission of a very weak mechanical stress to the liquid, keeping the liquid as much as possible close to its equilibrium state. The first observation of a finite low frequency shear elasticity in different liquids including water is reported by Derjaguin using the atomic vibration of a piezomembrane to impose the strain [11]. This property, observed at several microns scale at 73 kHz, is clearly interpreted as an intrinsic condensed matter property. Similarly, a low-frequency gel-like behavior is reported in untangled polymer melts up to 50 μm gap thicknesses and interpreted by dynamic heterogeneities (clusters) associated to the glass transition [9]. The present results on the n-heptadecane exclude effects of glass transition clustering [9] of confinement-induced solidification or ill-defined supercooled states. X-ray photon correlation spectroscopy shows that the analysis of capillary waves of supercooled PPG requires an elastic term in addition to the viscous contribution [29]. Recent dielectric developments might indicate a slow relaxation mode so far unidentified [30]. These different results show that time scales much longer than the α-relaxation exit in liquids.

The low frequency elasticity indicates that the intermolecular interactions are actually long ranged. This claim excludes a macroscopic

description of liquid properties based on individual molecular dynamics or close-neighbor clusters. The liquid state has to be considered first of all as a condensed matter [31–33], with long range elastic correlations contributing significantly to the energy of the condensed state. In this frame, the model of Ref. [33] predicts a finite value of the static shear modulus for liquids. This shear modulus is temperature and pressure dependent, and also size dependent, decreasing with increasing sample size, a behavior observed in our experiments with sub-millimeter thick samples [1–3]. Quantitative analysis of some of the present data and data at higher frequencies found in the literature is currently underway in the frame of this model.

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