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A new pressure sensor array for normal stress measurement in complex fluids

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Abstract

A new pressure sensor array, positioned on the bottom plate of a standard torsional rheometer, is presented. It is built from a unique piezo-capacitive polymeric foam and consists of 25 capacitive pressure sensors (of surface $4.5 \times 4.5 \text{ mm}^2$ each) built together in a 5×5 regular array. The sensor array is used to obtain a mapping of the normal stresses in complex fluids, which dramatically extends the capability of the rheometer. We demonstrate this with three examples. First, a pressure profile is reconstructed in a polymer solution, which enables the simultaneous measurement of the first and the second normal stress differences N_1 and N_2 , with a precision of 2 Pa. In a second part, we show that negative pressures can also be measured. Finally, we focus on the normal stress fluctuations that extend both spatially and temporally in a shear-thickening suspension of cornstarch particles. We evidence the presence of a unique heterogeneity rotating very regularly. In addition to their low cost and high versatility, the sensors show here their potential to finely characterize the normal stresses in viscosimetric flows. © 2021 The Society of Rheology. <https://doi.org/10.1122/8.0000249>

I. INTRODUCTION

Contrary to Newtonian fluids that are fully characterized by their viscosity η only, complex fluids exhibit a much more diverse behavior. Their microstructure, at an intermediate length scale between the molecule and the sample size (the radius of gyration of polymer coils in polymer solutions or the size of solid particles in suspensions), is altered by the flow. This often induces a nonlinear relation between the applied stress σ and the resulting flow velocity: its understanding is a fundamental challenge, central in many industrial processes. A striking example is the presence of normal stresses, appearing in the diagonal components of the stress tensor. In polymer melts and solutions, the normal stresses are responsible for a number of spectacular effects, such as the swelling of fluid in extrusion processes or its climbing on rotating rods [1], and have been the object of numerous studies [2–7]. Normal stresses are much less documented in other systems, even if they are essential to fully characterize the flow of complex materials. Yield-stress fluids, for example, develop moderate normal stresses in shear whose sign (positive or negative) and origin are still actively discussed [8–10]. In suspensions of solid particles, the sign of normal stresses is also the object of debate [11–16]. They are causing, in particular, particle migration [17] or edge fracture [18,19]. The normal stresses also increase dramatically in dense shear thickening suspensions: very high normal stresses, up to 10 000 Pa have been reported [20,21]. They are often associated with an inhomogeneous flow and generate localized forces strong enough to damage the rotors of mixing systems [21].

In viscosimetric flows, normal stresses are far less studied than shear stresses, mostly because their measurement is not straightforward. Indeed, on a torsional rheometer, the normal stress differences N_1 and N_2 are usually measured by combining two experiments with different geometries: the net thrust force gives access to N_1 in a cone/plate geometry and to $N_1 - N_2$ in parallel plate geometry [1,10,13,22,23]. N_2 is then calculated by finding the often small difference between two large experimental quantities, which amplifies the impact of any measurement error [7]. Other techniques have been developed to obtain a more reliable measurement of N_2 [3,13] or to avoid using cone plate geometry (unsuitable in some systems such as suspensions of large particles): the rotating rod rheometry [11,17,24,25], the measurement of the shape of a free surface [3], or the tilted through method [12,26]. Another method consists of measuring the pressure distribution as a function of radial position r in cone/plate or plate/plate geometry, by using a small number of pressure sensors integrated to the plate of a torsional rheometer. This technique, which gives both N_1 and N_2 in a single experiment, has been attempted in polymer solutions [2,4,7,27] and recently in a non-Brownian suspension [14]. Unfortunately, such setups are complex to build and measurements can be flawed. For example, Couturier *et al.* [12] used a rectangular channel that causes unwanted secondary flows near the corners of the cross section. The use of sensors must also meet a number of important requirements, for example, being small enough relatively to the rheometer plate, be extremely sensitive, and not cause any disturbance to the flow [28]. The pressure transducer membranes have to be positioned at exactly the same level as the disk surface: if not, a hole pressure has to be accounted for [29,30] and may lead to error measurements, as in the pioneering work of Adams and Lodge [2]. To this purpose, Dbouk and co-workers [14]

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coated the surface transducers with paraffin, so that no pressure hole effect is expected to take place. In addition, the number of sensors is often limited by the size of the plate and by the volume of the acquisition system.

Here, we demonstrate the potential of a new low-pressure sensor array in the measurement of normal stresses in non-Newtonian fluids, with a spatial resolution of $4.5 \times 4.5 \text{ mm}^2$. The number of sensors (25 in a $4 \times 4 \text{ cm}^2$ surface), their precision (up to 2 Pa for pressures below 1000 Pa), and the frequency of measurements (200 Hz) are unprecedented. In addition, the sensor array is highly versatile: the position, the size, and the number of sensors are easily varied by changing the bottom electrode only. They also do not drift with time and involve no pressure hole as the contact surface is flat and made of one piece. After presenting the sensor fabrication method and acquisition system, we demonstrate its potential to characterize three different flows in parallel plate geometry. First, in a polymer solution, we measure simultaneously the two normal stresses N_1 and N_2 , both in very concentrated and in very diluted systems. We then focus on a Newtonian fluid subjected to a secondary flow and measure this time negative pressures. Finally, we consider a shear-thickening fluid that exhibits flow heterogeneities: our sensors evidence the presence of a single aggregate rotating very regularly, an intriguing phenomenon that cannot be detected through the force sensor of the rheometer.

II. THE SENSOR ARRAY: FABRICATION, CALIBRATION, AND PROPERTIES

A. Sensor array

A distinctive feature of the pressure sensor array presented here is that the 25 sensors are not designed individually. As shown in Fig. 1(a), the sensor is made of two surfaces: a

solid *electrode network* (left) and a soft *measurement surface* (right) made of a polymeric material. As shown in Fig. 1(b), the electrode network is placed on the bottom plate of a torsional rheometer (Discovery HR-2, TA instruments) and covered by the measurement surface. A thin grid of double-sided tape is added between the two surfaces. The three-layer sandwich thus formed is the pressure sensor array itself, with a total thickness of $\sim 3 \text{ mm}$. It is presented in sectional view in Fig. 1(c).

The bottom layer is shown in dark blue and gray. It is a simple electrode network, custom-made by JLC PCB. The electrodes are 25 conductive square surfaces of $4.5 \times 4.5 \text{ mm}^2$, organized in a 5×5 regular matrix, and connected individually to the acquisition system. The total surface of the network is $4 \times 4 \text{ cm}^2$, which corresponds to the typical size of rheometer plate geometry.

On the top of the electrode network is attached a $25 \mu\text{m}$ thick layer of double-sided tape, as shown in Fig. 1(c). Using a laser cutter, 25 square holes (with size $5 \times 5 \text{ mm}^2$) are cut into the tape so that it does not cover the electrodes. The role of this intermediate layer is double: first, it ensures a good adhesion of the bottom layer to the measurement surface. In addition, its presence increases the sensitivity of the sensor by a factor 10 at very low pressures ($< 150 \text{ Pa}$). We interpret this as the consequence of a bending of the soft measurement surface within the grid (by typically $10\text{--}20 \mu\text{m}$), which can only happen when the measurement surface is slightly raised above the bottom electrodes.

The last layer is the measurement surface itself. Its core is a piezo-capacitive soft solid foam [shown in black in Fig. 1(c)] made from a polydimethylsiloxane polymer (PDMS) filled with 10% in weight of carbon black particle. This material was developed previously in our team, and its fabrication method is presented in detail in [31,32]. Here, the

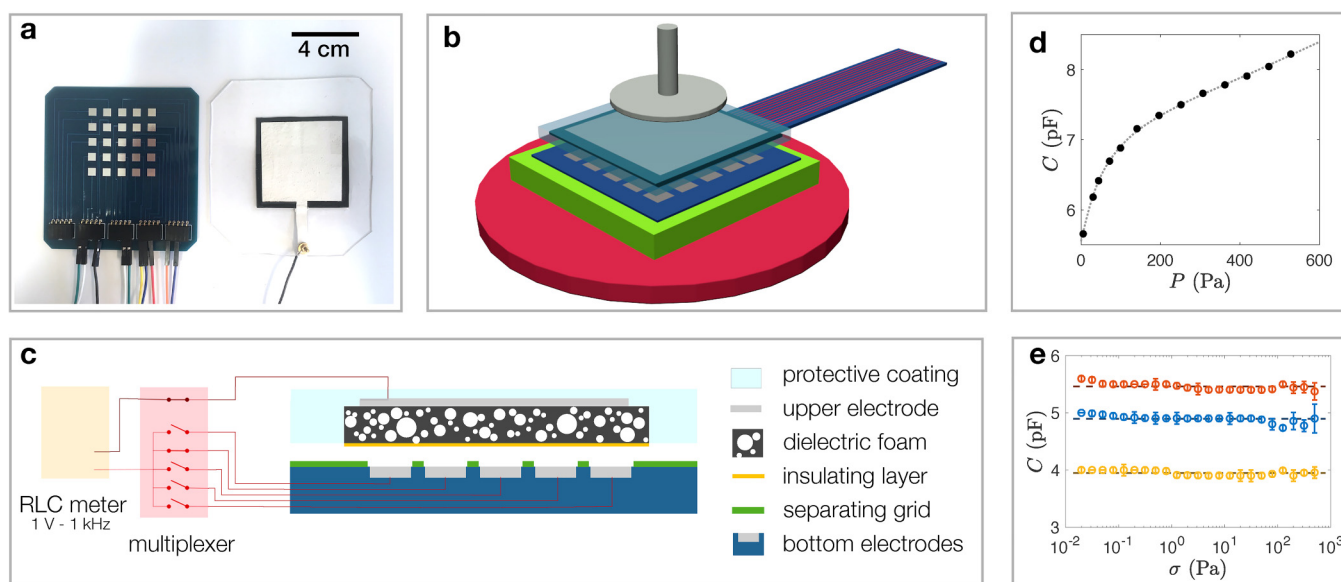


FIG. 1. (a) Picture of the electrode network (left) and the measurement surface (right) of a sensor array. (b) Experimental system: both layers are placed on the top of each other on the bottom plate of a parallel plate rheometer. The upper disk has a radius R of 2 cm, and the gap thickness h is 1 mm. (c) Side-cut of a capacitive sensor array. (d) Typical calibration curve of one sensor for applied pressures $P > 0$, showing the capacitance C as a function of hydrostatic pressure P . (e) Capacitance of three sensors when subjected to varying shear stress σ . One is positioned at the center of the geometry ($r=0$) and the other two are placed diagonally at a distance $r=12 \text{ mm}$ to the center.

solid foam is fully integrated into a multilayered sandwich to make a solid and reliable sensor. The foam itself is prepared as described in [32] through a water-in-oil emulsion process: a mixture of water and carbon-black particles is slowly added to PDMS under vigorous mixing. The emulsion then forms a paste, that is uniformly spread in a 1 mm thick sheet using an applicator (Zehntner ZUA 2000). The spreading is done on a Mylar surface covered by a 5 μm thick insulating layer of plain PDMS [shown in yellow in Fig. 2(c)], previously deposited with a spin-coater and cured for 1 h at 70 °C. After spreading, the paste is cured in a two-step process. First, it is placed in a bath of de-ionized water at 70 °C for 6 h to ensure cross-linking of the PDMS polymer. After curing, the solid is let to dry at 70 °C for 24 h so that all water contained in the pores evaporates. After curing and drying, the emulsion turns into a piezocapacitive soft solid foam. Its relative softness (with a Young modulus of 1.6 MPa [31]) is at the source of its remarkable piezocapacitive properties: when a pressure is applied on the material, the micropores (of size between 1 and 10 μm) deform, which induce a large variation of its permittivity and thus its capacitance. In the sensor array, the upper part of the foam is covered by a 25 μm thick paste of silver particles (Creative Materials), shown in gray in Fig. 1(c), which plays the role of a soft electrode. The upper electrode is connected to the acquisition system by planting a conductive screw attached to a wire through the silver film [as in Fig. 1(a)]. Finally, a protective coating of PDMS is cast around the three layers of the measurement surface. The PDMS, initially liquid, is let to rest for 30 min at ambient temperature to ensure that the measurement surface is flat. It is then cured at 70 °C for 1 h. We thus obtain a good alignment of the sensor with upper plate geometry. Small fluctuations of thickness (of the order of 20 μm) are observed when determining the zero position of the upper plate. These perturbations are small compared with the gap size in our experiments (1 mm) and the radius of the geometry (2 cm), and they are considered as standard in rheology.

The Young modulus of the whole measurement surface is close to the modulus of the piezocapacitive foam alone (≈ 1.6 MPa [31]). At small pressures (of the order of 100 Pa), the main source of deformation of the sensor originates in its local bending in the grid of a double-sided tape. The level difference between the disk and the coated sensor is thus less than 25 μm , while the gap between the disks is set to 1 mm. In such conditions, no pressure hole effect is expected. To check whether the softness of the sensor and the small thickness variations of the measurement surface might impact the flow, we compare in supplementary material, Fig. 1 [33] the flow curves of the two non-Newtonian fluids studied here (a polymer solution and a cornstarch suspension) when measured directly on the (solid) rheometer plate or on the soft sensor array. In both cases, the flow curves are almost identical, which indicates that the presence of sensor does not significantly disturb the flow.

B. Acquisition system

Due to its softness, the measurement surface does not redistribute the pressure, and the three-layers sandwich

sensor array behaves as 25 independent piezocapacitive elements. Their capacitance C (varying typically between 3 and 10 pF) is directly correlated to local pressure. Each sensor capacitance C is recorded as a function of time by an acquisition system, schematized in Fig. 1(c). It consists of two instruments: a multiplexer (Keysight 34980A Agilent technologies) and a precision LCR meter (Keysight E4980AL Agilent technologies), both controlled using an in-house Matlab code. The LCR meter imposes a sinusoidal signal at low frequency (1 kHz) and low voltage (1 V) and measures the capacitance of the connected circuit with a precision of 0.05%. Its internal impedance is automatically adjusted to the circuit: here, it is set to 5 k Ω . The multiplexer consists of 25 optical switches (one per sensor), with response time of 0.2 ms. During a measurement, the following steps are repeated: first, the switch corresponding to one bottom electrode is closed, and the LCR meter records the capacitance of the circuit between this electrode and the top electrode, averaged over 50 ms. The switch is then closed. This protocol is repeated for every sensor of the array—which takes approximately 1.5 s for all 25 sensors—before the next measurement cycle starts. In the following analysis of the data, an internal compensation calculation is done to account for the impedance of the multiplexer and wires. In most experiments, we use between 5 and 10 sensors, which automatically increases the measurement frequency, up to 2–3 cycles per second.

For higher frequency measurements, we use an electronic circuit custom made by Piwio, which miniaturizes the acquisition system. The circuit is similar in principle to the one described above, with three main differences. First, square waves of 1 V amplitude are used, with a frequency of 1 kHz. Second, there is one multiplexer for every four sensors, which increases the measurement frequency. Finally, the capacitance is measured with a low resolution of 0.1 pF, and it is not averaged. This system is much faster than the previous one (up to 200 Hz) but less accurate. In supplementary material, Fig. 2 [33], the typical response of the sensors when connected to the two acquisition systems is compared. When using the precision LCR meter, the noise (of the order of 1.5 Pa) is higher than the resolution of the acquisition system (0.000 01 pF, which corresponds to a pressure of 10^{-3} Pa). With this measurement method, pressures as low as 2 Pa can be measured, as further confirmed in Fig. 3(c). The Piwio integrated board, on the other hand, is limited by its resolution and cannot measure pressures below 10 Pa. These values of 2 and 10 Pa, which set experimentally the pressure detection threshold of our sensors, are close to the 4 Pa threshold of our rheometer (Discovery HR-2, TA instruments). However, since the surface of the sensors is 65 times smaller than the surface of rheometer geometry, the sensor array is sensitive to forces typically 50 times smaller than the rheometer. It should finally be noted that very low capacities (between 3 and 10 pF) are measured here, so that a non-negligible part of the noise in the measurements comes from stray fields in the lab environment. To limit their impact, we used in all experiments a nonconductive geometry (in PMMA), and we were careful to limit the length of the wiring.

C. Sensor calibration

The sensors are calibrated using hydrostatic pressure in compression and using a well-known flow in extension. In compression, a cylinder (with a diameter of 6 cm) is placed on the top of the measurement surface and made water-tight with grease. Water is then added, and the capacitance C_i of each sensor is measured as a function of water pressure $P = \rho gh$ (with h being the liquid height, ρ being water density, and g being gravity). A calibration curve is presented in Fig. 1(d): the sensor capacitance C , initially equal to $C_0 = 5.5$ pF, increases by 50% for a pressure variation ΔP of 600 Pa. The sensitivity $S = \frac{\Delta C/C_0}{\Delta P}$ is higher at low pressures, with $S = 1.8 \text{ kPa}^{-1}$ for $P < 100$ Pa. It remains excellent at higher pressures, with $S = 0.7 \text{ kPa}^{-1}$ for $200 \text{ Pa} < P < 1000$ Pa. While the sensors are particularly sensitive to the applied pressure, they do not react to shear stress. This is shown in Fig. 1(e), where the sensors' capacitance is recorded when shearing silicone oil with viscosity 100 000 cSt. At the stresses σ considered here, silicone oil is a Newtonian fluid, with constant viscosity and no measurable normal stress (see supplementary material, Fig. 3 [33]). The response of three sensors, placed either at the center of the geometry (blue dots) or at an equal distance $r = 12$ mm to the center (red and yellow dots), is shown. While the applied shear stress σ is varied over 5 orders of magnitude, the sensors' capacitance remains constant. Finally, the sensors are calibrated in extension using a Newtonian fluid sheared at high velocity. Indeed, in parallel plate geometry, a recirculation appears, which generates a negative pressure close to the static plate, varying quadratically with the angular velocity Ω of the rotating plate (see Sec. IV for more details). We use this theoretical pressure value as a reference to calibrate our sensors.

Two other properties of the sensor are also considered. First, we estimated the sensor response time by applying a constant pressure load (< 1000 Pa) on the sensors and removing it suddenly. The sensors' response time is limited by the dynamical response of the membrane, which deforms under the applied stress. It is of the order of 50 ms when increasing pressures are applied, but it can reach 100–200 ms when a high pressure (typically 1000 Pa) is suddenly lifted. Experimentally, we observed an increasing hysteresis when applying pressures $P > 3000$ Pa, which might correspond to a too large deformation of the measurement surface in the grid. For this reason, we will focus here on pressures between 0 and 1000 Pa, which correspond to the optimal operating mode of our sensors. In the experiments considered here, the measurement frequency varies between 1 and 20 Hz, so that the pressure is always averaged over a duration that is of the order or larger than the sensor response time. We finally checked that the sensors do not exhibit any significant drift in time. This is demonstrated in supplementary material, Fig. 4(a) [33]: the response of the sensors, submitted to a constant hydrostatic pressure of 300 Pa, is shown to remain constant. In addition, we compare in supplementary material, Fig. 4(b) [33] the sensors' response before and after a 2000 s experiment, after being subjected to pressures varying between 100 and 400 Pa. The difference in the

pressure measured at zero angular velocity before and after 2000 s is smaller than 4 Pa, which is of the order of the accuracy of the sensors. The potential drift is thus extremely small compared to what is reported in the literature [10,14], which can reach 15–30 Pa in 60 s [10].

D. Rheology procedure

We use a parallel plate geometry, with the measurement surface of the sensor as a static bottom plate. The rotating disk is made of PMMA (to limit the effect of stray magnetic fields on the sensors) and sanded, with a radius of $R = 2$ cm. This geometry was selected in all experiments so that the gap $h = 1$ mm is always much larger than the intrinsic variations in the thickness of the measurement surface or its variations due to compression with the normal stresses (which are both of the order of $20 \mu\text{m}$). This geometry, thus, limits the influence of the measurement surface on the flow: as presented in supplementary material, Fig. 1 [33], the flows in parallel plate geometry with and without the sensor are almost identical. The liquid of study is added directly on the measurement surface. After positioning the upper disk at the working gap ($h = 1$ mm), the liquid in excess is carefully cleaned, so that the meniscus at the edge of the plate is as vertical as possible. Since the PDMS covering the measurement surface is smooth and hydrophobic, the contact angle of the fluids used here (water-based solutions, glycerol) is of the order of 90° , as schematized in Fig. 2(c), and the meniscus is slightly concave. This does not affect the rheological measurements (as seen in supplementary material, Fig. 1 [33]), but it facilitates liquid ejection, which is observed at a lower angular velocity than on the rheometer plate (a rough and hydrophilic solid).

Once the liquid and the geometry are positioned, both the internal force sensor of the rheometer and the sensor array are set to 0. This consists in assuming that the system is prestressed by a constant pressure P_c (typically of the order of 50 Pa) due to the meniscus, that is subtracted in the following measurements. This assumption is valid as long as edge effects due to the distortion of the meniscus or instabilities such as edge fracture remain small enough to be neglected. The numerical simulations of situations where instabilities of the meniscus occur are complex. However, there seems to be both theoretical and experimental consensus on edge fracture [18,19,34]. The latter is expected to appear in fluids with a negative second normal stress N_2 , when $|N_2|$ exceeds a critical value $N_c \simeq 5\gamma/h$, with γ being the surface tension of the fluid and h being the gap size. In our experiments with a polymer solution of HPAM, N_2 never exceeds 16 Pa, which is much smaller than $N_c \simeq 350$ Pa. This value is also very large for the particle suspension system, where, in addition, we do not expect edge effects to directly impact our results, focused on the detection of flow heterogeneities. In other situations, such as instabilities induced by inertia (at thus at large shear rates $\dot{\gamma} > 200 \text{ s}^{-1}$), we checked that, in all experiments, the potential pressure variation induced by the increased meniscus deformation at the edge (of the order of 20 Pa) always remains small compared with the mean pressure in the flow.

During an experiment, a constant shear stress $\sigma = \sigma_{12}$ is imposed on the fluid by the upper disk (with radius $R = 2$ cm), where 1 and 2 are the directions of the velocity and the velocity gradient. The average normal stress is obtained with the built-in force sensor of the rheometer, which measures the mean vertical thrust on the geometry. The sensor array, on the other hand, measures the pressure $P(r, \theta) = -\sigma_{22}$ close to each sensor, with $\sigma_{22}(r, \theta)$ being the component of the stress deviator tensor (in the 22 = zz direction) in the position $(r, \theta, z = 0)$. In parallel plate geometry, we denote $\dot{\gamma}_R$ as the shear rate at the edge and $\dot{\gamma}$ as the local shear rate under the plate, with $\dot{\gamma} = \frac{r\Omega}{h}$. For shear rates $\dot{\gamma}_R < 200 \text{ s}^{-1}$, the velocity $\mathbf{v}$ of the fluid is (almost) purely orthonormal, with $\mathbf{v} \simeq v_\theta \mathbf{e}_\theta = r \frac{\Omega}{h} \mathbf{e}_\theta$ and $v_r, v_z \ll R\Omega$.

For a homogeneous liquid, the equation of motion along the r direction writes

$$\frac{1}{r} \frac{\partial}{\partial r} (r \sigma_{33}) - \frac{\sigma_{11}}{r} = -\rho \frac{v_\theta^2}{r} + \frac{\partial p}{\partial r}, \quad (1)$$

where 3 denotes the vorticity direction. Equation (1) can describe two types of flows that will be characterized separately in Secs. III and IV. First, we consider the flow of a non-newtonian fluid. For $\dot{\gamma} < 500 \text{ s}^{-1}$, the inertial effects [appearing in the term $\frac{\partial p}{\partial r}$ in Eq. (1)] are negligible compared with the effect of the normal stresses, and this term is neglected. The diagonal components in the stress tensor σ generate a radial variation of $-\sigma_{22}$, which is measured by the sensors. In Sec. IV, a Newtonian fluid is sheared at high angular velocities. In this configuration, the fluid does not develop normal stresses and the left-hand side of Eq. (1) is

negligible. The inertial term $\frac{\partial p}{\partial r}$ generates a slow recirculation of the liquid, and a negative pressure profile is measured.

III. POSITIVE NORMAL STRESS MEASUREMENTS

Long-chain polymer solutions are viscoelastic fluids, in which flow is characterized by three functions: their viscosity $\eta = \sigma/\dot{\gamma}$, the first normal stress difference $N_1 = \sigma_{11} - \sigma_{22}$, and the second normal stress difference $N_2 = \sigma_{22} - \sigma_{33}$. In polymer melts and concentrated polymer solutions, N_1 is typically positive and high, while N_2 is more than 10 times smaller and negative [1].

Using the pressure sensor array, both N_1 and N_2 are measured in a single experiment. We use a solution of a partially hydrolyzed polyacrylamid polymer (HPAM) with a high molar mass $M_w = 18 \times 10^6 \text{ g/mol}$. HPAM is a charged linear polymer consisting of acrylamid monomers, where 25–30% of the amine group are replaced by a carboxyl group. The concentration of HPAM in water is varied from 200 ppm (very diluted) to 5000 ppm (very concentrated). As shown in Fig. 2(a), the rheology of these solutions is characteristic of a viscoelastic fluid, with a strong shear thinning: the viscosity η decreases with $\dot{\gamma}_R$ following a power law $\eta \propto |\dot{\gamma}_R|^n$. n increases in magnitude with the concentration: in Fig. 2(a), the best fit (dotted lines) is obtained for $n = -0.84$ for 5000 ppm, $n = -0.70$ for 1000 ppm, and $n = -0.65$ for 200 ppm. At high shear rates, the liquid develops normal stresses, in which the average value is measured by the built-in force sensor of the rheometer, and is presented in Fig. 2(b). The thrust $F_z/\pi R^2$ averaged on the plate surface is plotted as a function of $\dot{\gamma}_R$ for three HPAM solutions. The concentrated solutions exhibit high normal stresses: for

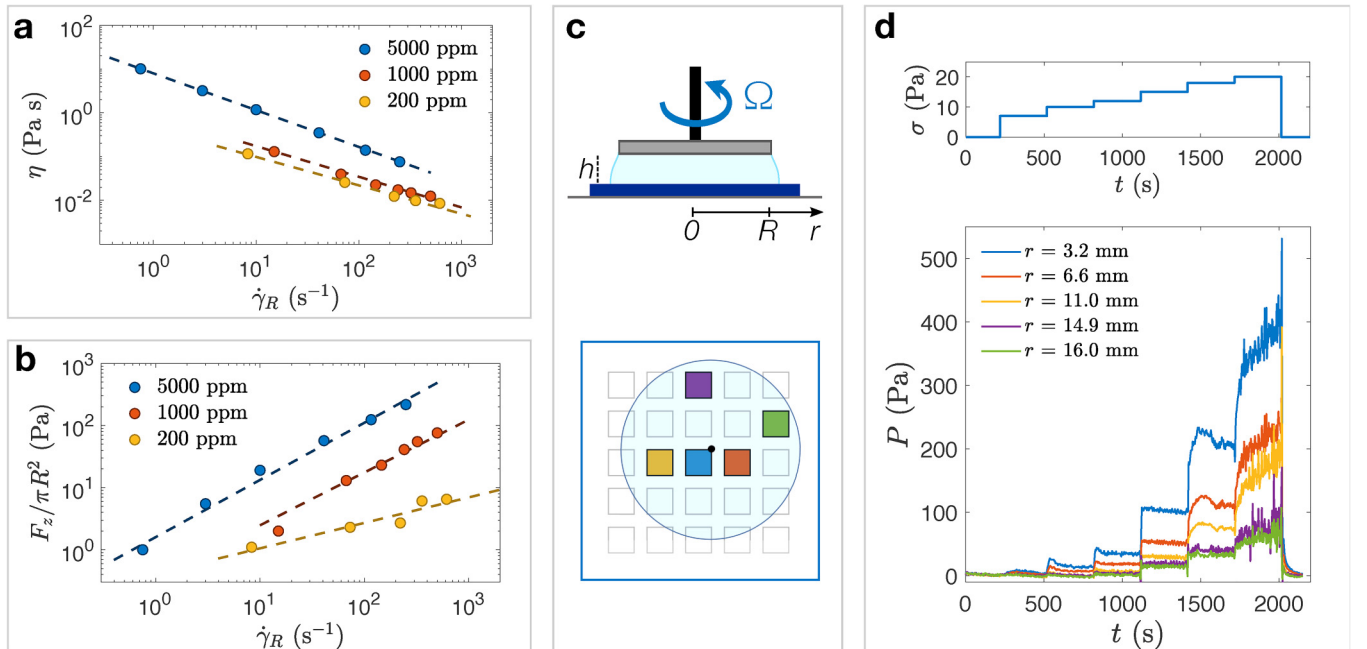


FIG. 2. Rheological measurements of HPAM solutions. (a) Viscosity η as a function of shear rate $\dot{\gamma}$ for three different concentrations of HPAM solutions: 5000 ppm, 1000 ppm, and 200 ppm. (b) Average vertical pressure $F_z/\pi R^2$ measured through the built-in sensor of the rheometer, as a function of $\dot{\gamma}_R = R\Omega/h$. (c) (Top) Side view of an experiment. The pressure profile $P(r)$ is measured as a function of radial distance r to the center of the geometry. (Bottom) Top view of the sensor array. The sensors used in the experiment are shown with darker (or colored) shades. (d) Response of the sensors during a typical experiment of stress-imposed steps $\sigma(t)$. The pressure $P = -\sigma_{zz}$ measured by five different sensors is plotted as a function of time t .

example, the mean normal stress of the 5000 ppm solution is more than ten times higher than the shear stress σ at high shear. In the region where they are present, the normal stresses increase proportionally to $\dot{\gamma}_R^m$. From Fig. 2(b), we find $m = 0.41$ for the 200 ppm solution, $m = 0.85$ for the 1000 ppm solution, and $m = 0.92$ for 5000 ppm. m is here always smaller than 2 and increases with the concentration, a phenomenon that has already been observed in similar solutions.

The sensors measure pressure $P = -\sigma_{22}$ as a function of radial distance r to the center of the geometry, as defined in Fig. 2(c) (top). For simplicity, we present here the pressure measurement of only five different sensors placed at varying distances r to the center of the geometry and distributed in all directions. Indeed, as presented in supplementary material, Fig. 5 [33], two sensors placed at an identical distance r to the center of the geometry measure the same pressure. In Fig. 2(c), the sensors are placed at a distance $r = 3.2$ mm, $r = 6.6$ mm, $r = 11.0$ mm, $r = 14.9$ mm, and $r = 16.0$ mm to the center of the geometry. The geometry is shown with a light circle: its center (indicated by a black dot) is placed at the level of the upper right corner of the central sensor.

A typical experiment is presented in Fig. 2(d), with a 5000 ppm HPAM solution. The shear stress σ (plotted on top) is increased by regular steps of 300 s, from $\sigma = 0$ Pa to $\sigma = 20$ Pa. The stress $P = -\sigma_{22}$ measured by each sensor is recorded as a function of time t and is presented in Fig. 2(d) with the same color code as in Fig. 2(c). As visible in Fig. 2(d), P increases with σ . At each stress-imposed step, the higher pressures are measured close to the center of the geometry (in blue): the pressure continuously decreases with r . While it remains almost constant for $\sigma \leq 15$ Pa (with a very slight decrease in time), the pressure signal becomes noisy for $\sigma > 15$ Pa. We interpret this as a consequence of an elastic turbulence phenomenon, which is likely to occur at low Reynolds numbers in our systems [35–37]. Such instabilities are driven by the stretching of the polymer chains in the azimuthal direction. Following Pakdel and Mc Kinley [38,39], we expect these instabilities to appear in parallel plate geometry when the criterion $(h/R)^{1/2} N_1/\sigma > M$ is met, with R being the streamline curvature and M being a constant, ranging between 1 and 6 depending on the rheological law of the fluid [40,41]. Anticipating that $|N_2| \ll N_1$, we calculate the Weissenberg number at the transition as $Wi = N_1/\sigma \simeq F_z/(\pi R^2 \sigma) \simeq 10$, which leads to $M \simeq 3$. This value is in good agreement with what is expected for elastic turbulence [37].

From Fig. 2(d), the pressure $P(r)$ measured at increasing distances r within the sheared polymer solution is extracted and is plotted for each shear stress σ . The results are presented in Figs. 3(a)–3(c), where $P(r, \sigma)$ is plotted for varying polymer concentrations [5000 ppm in Fig. 2(a), 1000 ppm in Fig. 2(b), and 200 ppm in Fig. 2(c)]. The error bars correspond to the error due to the sensor itself (which is done by comparing the pressures of two sensors placed at an equal distance to the center) and from the noise in the pressure measurements. For all polymer concentrations, the pressure P is positive and decreases with the distance r/R to the center of geometry. This is similar to previous observations of

Alcoutlabi *et al.* [7] in solutions of a different polymer. The pressure profiles $P(r)$ all seem to extrapolate to a positive pressure for $r = R$, which is consistent with a negative value for N_2 [1,7]. It should also be noted that the pressures detected here are significantly smaller than the ones measured in other polymer solutions [7]. In particular, minute pressures (smaller than 10 Pa) that develop in very diluted solutions [Fig. 2(c)] are detected, which opens the way to a better characterization of these much less studied systems.

The experiments are compared with the pressure profile $P(r)$ expected from the theory. Since the sensors are set to zero at the beginning of every experiment, the pressure difference due to the meniscus is already taken into account, so that the pressure measured by the sensors is equal to $-\sigma_{22}$. The effect of centrifugal forces is very small here compared with the normal stresses, so that the inertial term $\partial p/\partial r$ in Eq. (1) is neglected. Finally, using $N_1 = \sigma_{11} - \sigma_{22}$ and $N_2 = \sigma_{22} - \sigma_{33}$, the equation of motion within the fluid [Eq. (1)] is expressed as a function of σ_{22} and the two normal stresses only [14],

$$\frac{\partial \sigma_{22}}{\partial r} = \frac{\partial N_2}{\partial r} + \frac{(N_1 + N_2)}{r}. \quad (2)$$

We assume here that in the HPAM solutions, as in many polymer solutions, the two normal stresses are proportional to $\dot{\gamma}^m$ [3,4,7,25]. This comes to assume that N_1 and N_2 can be written as $N_1 = \alpha_1 \dot{\gamma}^m$ and $N_2 = \alpha_2 \dot{\gamma}^m$, with α_1 and α_2 being the normal stress coefficients. In other systems, this hypothesis might not hold: in this case, it is also possible to use our sensors slightly differently to measure N_1 and N_2 , as detailed in the supplementary material [33].

Using the expressions of N_1 and N_2 as a function of $\dot{\gamma}$ and $\dot{\gamma}_R = R\Omega/h$, Eq. (2) writes

$$\frac{\partial \sigma_{22}}{\partial r/R} = \dot{\gamma}_R^m (\alpha_1 + (m+1)\alpha_2) \left(\frac{r}{R}\right)^{m-1}. \quad (3)$$

This equation is integrated with respect to r . The boundary condition is found assuming that at the meniscus at the edge of the geometry (in $r = R$) the third normal stress is equal to the capillary pressure, which writes $\sigma_{33}(r = R) = -N_2(R) + \sigma_{22}(R) = P_c$, with P_c being the capillary pressure [14]. Since all sensors are set to zero at the beginning of the experiment (at zero angular velocity), this preload is already taken into account and $P_c = 0$. This gives

$$\begin{aligned} P(r) &= -\sigma_{22}(r) \\ &= -\dot{\gamma}_R^m \left[\frac{\alpha_1 + (m+1)\alpha_2}{m} \left(\frac{r}{R}\right)^m - \frac{(\alpha_1 + \alpha_2)}{m} \right]. \end{aligned} \quad (4)$$

The pressure profile $P(r)$ thus varies linearly with $(r/R)^m$. This formula is used to determine the two normal stress coefficients α_1 and α_2 . To do so, we combine all experiments for a given polymer solution and plot $P/\dot{\gamma}_R^m$ as a function of $(r/R)^m$. This is shown in Figs. 3(a)–3(c) for HPAM concentrations of 5000, 1000, and 200 ppm. The different location of the data on the r/R scale comes from the different

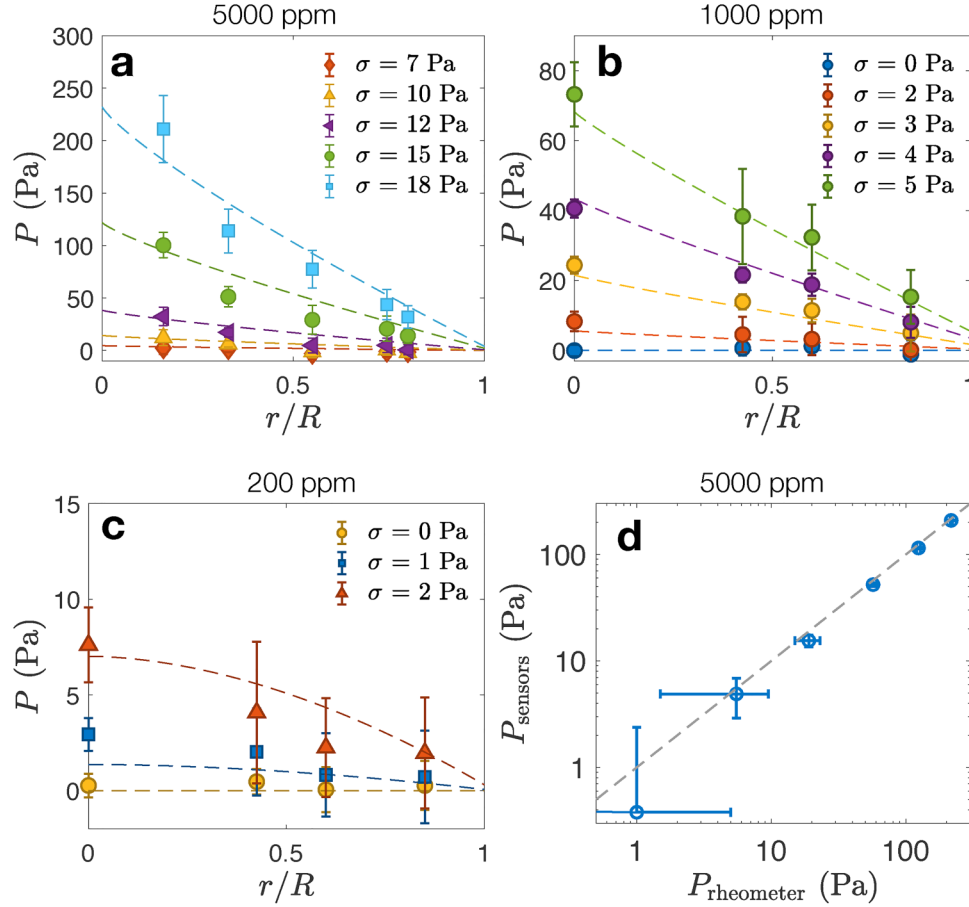


FIG. 3. Pressure profiles $P(r)$ within flowing HPAM solutions in plate/plate configurations, for varying shear stresses σ . The dotted lines are the best fit of the model, with $P(r) = -\dot{\gamma}_R^m[(\alpha_1 + (m+1)\alpha_2/m)(r/R)^m] - (\alpha_1 + \alpha_2/m)$. (a) Solution of 5000 ppm HPAM in water: $\alpha_1 = 4.7$, $\alpha_2 = -0.2$. (b) HPAM 1000 ppm: $\alpha_1 = 0.46$, $\alpha_2 = -0.02$. (c) HPAM 200 ppm: $\alpha_1 = 4 \times 10^{-4}$, $\alpha_2 = -3 \times 10^{-5}$. (d) Comparison of the integrated pressure from the sensor array P_{sensors} to the mean pressure given by the built-in force sensor of the rheometer $P_{\text{rheometer}}$. The dotted line has a slope equal to 1.

positions of the sensors. In Fig. 3(a), the rotation axis corresponds to one corner of the middle sensor [as schematized in Fig. 2(c)], while in the two other cases, the sensor is centered. m is deduced from the best fit of the mean vertical stress $F_z/\pi R^2 = N_1 - N_2$ as a function of $\dot{\gamma}$ [Fig. 2(b)]. The slope of the linear plot $P/\dot{\gamma}_R^m = f((r/R)^m)$ gives $-\frac{\alpha_1 + (m+1)\alpha_2}{m}$, and the intercept at the origin is $\frac{\alpha_1 + \alpha_2}{m}$, from which α_1 and α_2 are calculated. The error on α_1 is typically of the order of 25%, while for α_2 (which is one order of magnitude smaller than α_1) it is of the order of 100%. The corresponding pressure profiles (with a unique α_1 and α_2 per concentration) are plotted in Figs. 3(a)–3(c), with $\alpha_1 = 4.7$ and $\alpha_2 = -0.2$ for $c = 5000$ ppm, $\alpha_1 = 0.46$ and $\alpha_2 = -0.02$ for $c = 1000$ ppm, and $\alpha_1 = 4 \times 10^{-4}$ and $\alpha_2 = -3 \times 10^{-5}$ for $c = 200$ ppm. As expected for viscoelastic liquids, α_1 is positive and α_2 is negative, with $|\alpha_1| \gg |\alpha_2|$. Typically, α_1 decreases by a factor 10 between the 5000 and the 1000 ppm solutions, and decreases by a factor 1000 when comparing the 1000 ppm with the 200 ppm solutions. Remarkably, we can still measure the normal stress coefficients in the extremely dilute (200 ppm) polymer solution, for which the maximum mean normal stress is smaller than 10 Pa at high shear. To finally check the validity and precision of the sensors, the experimental pressure profiles are integrated to obtain the mean pressure P_{sensors} over the surface of the

geometry. In Fig. 3(d), P_{sensors} is compared to the pressure obtained through the built-in sensor of the rheometer $P_{\text{rheometer}}$. The error bars correspond to the pressure detection threshold (taken at 2 Pa for the sensor array and 4 Pa for the rheometer, see supplementary material, Fig. 2 [33]). All the data collapse on a line with slope 1 (indicated with a dotted line), which confirms that the pressure measured by the sensors is fully consistent with the pressure given by the rheometer instruments.

IV. NEGATIVE PRESSURES' MEASUREMENT

The sensors are also sensitive to negative pressures, which can be measured after a calibration in extension. To obtain controlled negative pressures, we use the same method than Adams [2], or, more recently, by Dbouk [14]. We take advantage of the fact that the flow in a parallel plate geometry is not purely viscosimetric. Even at small Reynolds numbers, there is always small recirculation because of inertia [14,42–44] [this appears in the term $\frac{\partial p}{\partial r}$ in Eq. (1)]. This secondary flow consists of an inward motion, close to the plate, and an outward motion, near the rotating disk that pulls the plate and the rotating disk together. The effect of recirculation on the rheology curves is presented in Fig. 4(a) for a Newtonian fluid (a mixture of 98% glycerol with water), with density $\rho =$

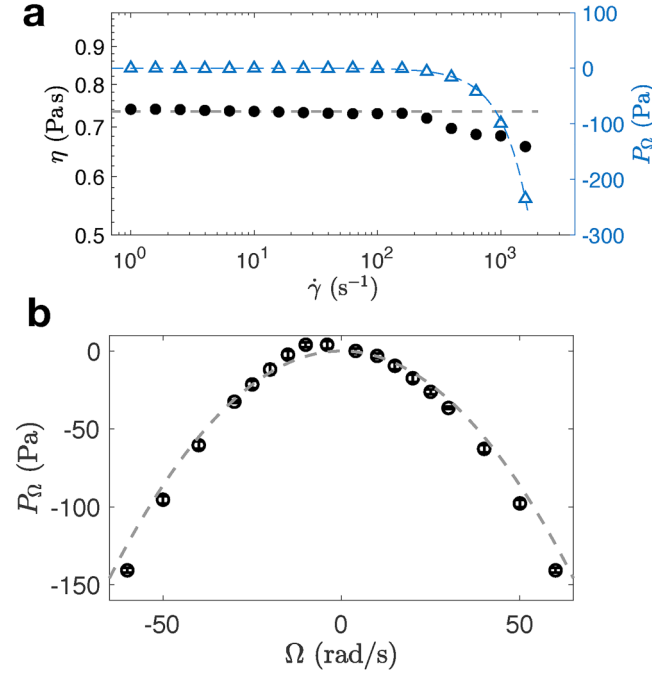


FIG. 4. (a) Viscosity η and mean normal stress (measured by the force sensor of the rheometer) of a 98% glycerol solution in water as a function of $\dot{\gamma}$. A value of $\dot{\gamma} = 200 \text{ s}^{-1}$ corresponds to an angular velocity $\Omega = 10 \text{ rad/s}$ in this geometry. (b) Mean normal pressure P_Ω measured through the force sensor of the rheometer, as a function of rotor angular velocity Ω . The dotted lines are the parabolic fits expected from the theory.

1250 kg/m^3 and viscosity $\eta = 0.74 \text{ Pa s}$ at 22°C . Usually, at low angular velocities, this secondary flow does not disturb the viscosity measurements. It simply induces a positive total normal stress on the plate, which corresponds to negative pressure P . However, depending on the value of the Laplace pressure in the meniscus, it may be at the origin of liquid ejection. This phenomenon is observed in our experiment at high shear rates $\dot{\gamma} > 200 \text{ s}^{-1}$: the ejection of a small amount of liquid comes with a decrease in the viscosity, which differs from the constant value expected for a purely Newtonian fluid (dotted line). Note that if the liquid were wetting, the ejection would occur at higher shear rate. In this situation, the recirculation might induce an increase in the viscous loss and the measurement of an effective viscosity increasing with the shear rate [44]. This ejection, however, does not modify the normal pressure on our small sensors, since they are at least 3 mm away from the meniscus.

What is of interest for us here is that recirculation generates negative pressure P in the fluid, in which absolute amplitude increases with Ω . This pressure is large enough to be detected by the sensor of the rheometer, as visible in blue in Fig. 4(a). The pressure profile generated by recirculation is found to vary quadratically with both the angular velocity Ω and the distance r to the center of geometry. Following [42–44], the exact expression of P is

$$P(r) = -\frac{3}{20}\rho\Omega^2(R^2 - r^2), \quad (5)$$

so that the mean theoretical pressure (averaged over the surface $S = \pi R^2$ of the geometry) writes $P_\Omega = -3/40\rho R^2\Omega^2$.

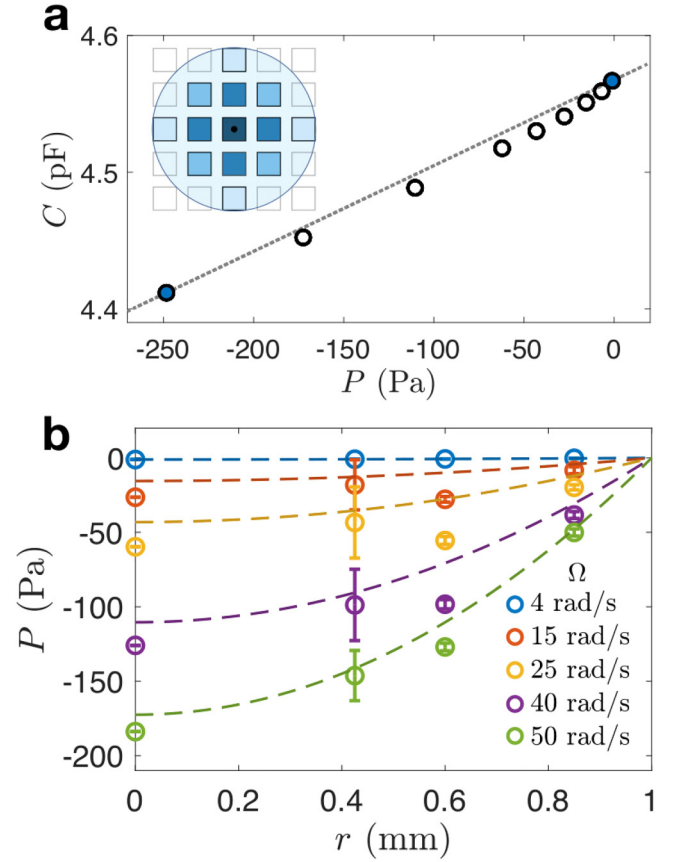


FIG. 5. (a) Calibration curve of a sensor, with (in dotted line) a linear fit. (b) Radial distribution of the pressure in a parallel plate torsional flow, for varying angular velocities Ω .

This prediction is shown in Fig. 4(a) with a blue dotted line and compared with the experiment (blue triangles). The model reproduces well the decrease in P_Ω for $\dot{\gamma} > 200 \text{ s}^{-1}$. This indicates that the effect of inertia on the meniscus and the potential fluid loss (visible in the viscosity measurement) remains of second order compared with the effect of recirculation on the pressure measurements. The parabolic shape of P_Ω is even more clearly visible when plotted in a linear scale, as presented in Fig. 4(b). In this experiment, the angular velocity Ω is varied between -60 and 60 rad/s : in this range of velocities, the experiment and model nicely overlap.

In the following, we use this negative pressure profile to calibrate the sensors in extension and to test them in a configuration where $P < 0$. As presented in the inset of Fig. 5(a), the sensor array is here centered with respect to geometry. The pressure P is measured at four different distances r from the center of geometry. The sensors are calibrated by measuring their capacitance at two extreme angular velocities: $\Omega = 0$ and $\Omega = 60 \text{ rad/s}$, and the corresponding pressure is deduced from Eq. (5). A linear interpolation between these two extreme points is sufficient here to calibrate the sensors. As presented in Fig. 5(a), the sensor capacitance C varies almost linearly with P in the region of negative pressures considered ($-300 \text{ Pa} < P < 0 \text{ Pa}$). The dotted line shows the linear interpolation between the extrema (blue points): it fits convincingly all intermediate measurements (black circles).

After calibration, we test the validity of our sensors to measure negative pressures. To do so, we compare the pressure profiles $P(r)$ measured experimentally to the theory for varying angular velocities Ω ($4 \text{ rad/s} < \Omega < 50 \text{ rad/s}$). In Fig. 5(b), the experimental results are shown in circles, with a different color for each Ω . The error bars show the difference in pressure measured by different sensors placed at an equal distance r from the center. The pressure profiles $P(r)$ are quadratic, as expected from Eq. (5). The theoretical profile is presented with a dotted line: it compares well with the experimental measurements. This demonstrates that the sensor arrays also detect negative pressures with a reasonable accuracy. In the following, the pressure measurement in an inertial Newtonian fluid flow will be used as a reference flow to calibrate the sensors for negative pressures.

V. EVIDENCING HETEROGENEITIES

Interestingly, the small size of the sensors when compared with the size of the geometry, combined with a high measurement frequency, makes them a very good tool to study heterogeneous flows. We consider here a suspension of non-Brownian solid particles (of cornstarch) in a neutrally buoyant Newtonian liquid. At high solid fractions ϕ , these suspensions exhibit a striking shear-thickening behavior: while they flow easily at low stirring, they become highly viscous at high stirring with an almost solidlike behavior. It is now admitted that this shear-thickening phenomenon

associated with an evolution from a lubricated interaction between the particles (at low shear) and a frictional contact (at high shear) due to an increase in normal stresses that push particles together [45–50]. Interestingly, close to the shear-thickening transition, large temporal fluctuations in viscosity, shear rate, or density are often observed in shear thickening fluids, including cornstarch [51–53]. Recent experiments and new measurement methods (magnetic resonance [54], ultrasound imaging [55], x-ray radiography [21], or boundary stress microscopy [56]) seem to indicate that these fluctuations are associated with a heterogeneous flow. This observation is further confirmed by recent simulations [57], but a lot remain to be understood, still, on the nature of the heterogeneities and their origin. Even if they seem to be consistently observed in some shear thickening systems, it is also not clear whether all shear thickening fluids become heterogeneous.

We use here a suspension of solid cornstarch particles, a popular shear-thickening system [20,55]. The solid particles are put in an isodensity salt of CsCl in water (55% w/w of CsCl). Here, we use a suspension of 41% in the weight of cornstarch, as done in Refs. [21,55] so that the fluid is in the discontinuous shear-thickening region. In Fig. 6(a), the viscosity of the 41% in weight cornstarch suspension is measured as a function of $\dot{\gamma}$ for stress steps of 60 s. At this weight fraction of 41%, cornstarch is shear-thinning at low shear rate ($\dot{\gamma} < 2 \text{ s}^{-1}$) and strongly shear thickening above a critical shear rate $\dot{\gamma}_c = 5 \text{ s}^{-1}$, with a viscosity η increasing by more than one order of magnitude. In Fig. 6(b), we report the

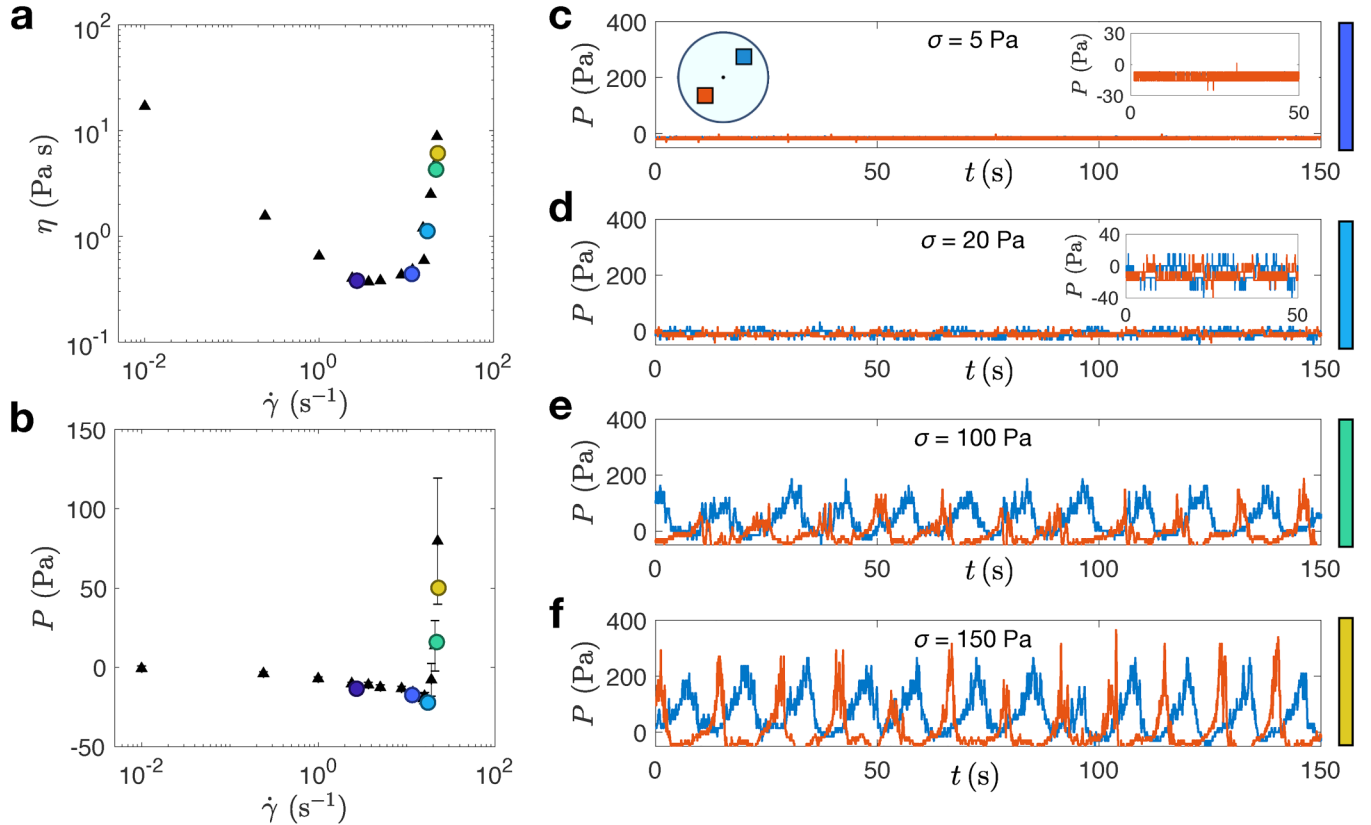


FIG. 6. Suspension of 41% of cornstarch particles in an isodensity water/CsCl solution. (a) Viscosity η as a function of shear stress $\dot{\gamma}$. (b) Normal stress P measured by the axis of the rheometer as a function of $\dot{\gamma}$. (c)–(f) Normal stress measurement of the sensors as a function of time, for varying imposed constant stresses σ . Two $4 \times 4 \text{ mm}^2$ sensors [shown in the inset of (c)] are placed in opposite directions on a diagonal. (c) $\sigma = 5 \text{ Pa}$. (d) $\sigma = 20 \text{ Pa}$. (e) $\sigma = 100 \text{ Pa}$. (f) $\sigma = 150 \text{ Pa}$.

mean normal stress generated by the flow as recorded by the rheometer. Here again, two regions can be distinguished: at low shear rates, normal stresses are small and negative and decrease slowly with $\dot{\gamma}$. For a shear rate $\dot{\gamma} > 15 \text{ s}^{-1}$, however, normal stresses become positive and increase rapidly with $\dot{\gamma}$. The error bars indicate the amplitude of pressure fluctuations, which increase dramatically in the shear-thickening region.

To the best of our knowledge, the sensor arrays presented in this work (accounting for normal stresses associated with fluctuations) have not been presented in the literature so far. We use here two sensors, placed at opposite directions below the rotating plate, at an identical distance $r = 12 \text{ mm}$ from the center [see the inset of Fig. 6(c)]. They record the pressure at a high frequency of 100 Hz. To study the dynamical behavior of the suspension, we impose steps of constant shear stress σ of 300 s, either in the shear-thinning region ($\sigma = 1$ and 5 Pa) or in the shear-thickening region ($\sigma = 20, 100, \text{ and } 150 \text{ Pa}$). These points are highlighted by circles in Figs. 6(a) and 6(b). We observe that the measurements corresponding to the 300 s steps coincide with the 60 s measurements, contrary to what was observed in other shear thickening suspensions [21]. We explain this by the combination of a limited particle migration in parallel-plate geometries [58,59] and the relatively short time scale of the experiment.

In Figs. 6(c)–6(f), we report the pressures measured by the two diagonal sensors [whose position is shown in the inset of Fig. 6(c)] as a function of time for each step. The typical response of the central sensor is also shown in supplementary material, Fig. 6 [33]. For $\sigma = 5 \text{ Pa}$, below the discontinuous shear-thickening region, the pressure is constant and identical for both sensors [Fig. 6(c)]. It is slightly negative ($P \simeq -10 \pm 3 \text{ Pa}$, see zoom in the inset), in good agreement with the global pressure measured by the rheometer. However, just after the shear thickening region is reached, oscillations become visible. The pressure variations are initially small, of the order of 30 Pa for $\sigma = 20 \text{ Pa}$ (see Fig. 6 and inset). They increase in amplitude with increasing σ : the pressure variation is of the order of 150 Pa for $\sigma = 100 \text{ Pa}$ and 350 Pa for $\sigma = 150 \text{ Pa}$. Very interestingly, the pressure fluctuations are extremely regular, with out-of-phase peaks of similar amplitude and a constant negative pressure in between. The period T between two peaks slightly decreases with increasing shear stress σ : $T = 15.1, 13.3, \text{ and } 11.3 \text{ s}$, respectively for $\sigma = 20, 100, \text{ and } 150 \text{ Pa}$. Interestingly, T is very close to twice the rotational period of the upper disk (respectively equal to 13.8, 11.3, and 10.7 s). All these results seem to indicate the presence of one large aggregate rotating with an angular velocity $\Omega_A \simeq \Omega/2$ within the parallel plate geometry. The presence of this aggregate is associated with local and very high normal stresses: for $\sigma = 150 \text{ Pa}$, the peak pressure is 7 times higher than the mean pressure obtained through the force sensor of the rheometer.

This observation is in good agreement with the recent work of Ovarlez *et al.*, which evidenced the presence of “density waves” in a similar cornstarch suspension [21]. In a Couette cell, the density waves move in the flow direction, more slowly than the rotor, similarly to what is detected here. Our results are also consistent with the observations of

Rathee *et al.* [56], who showed the presence of zones of high shear stress propagating with an angular velocity $\Omega_A = \Omega/2$ in a suspension of submicronic silica particles in glycerol. Here, for the first time, we evidence and measure the normal stresses associated with these heterogeneities. The stresses measured by the sensors are 5–10 times higher than the average pressure obtained through the rheometer. The asymmetric forces surely generate a large torque on the geometry and are thus most likely at the origin of the off-axis motion of the rotor in Couette cells. This phenomenon has been observed so far in cornstarch [21] and in latex particle suspensions [60]. It should be noted, finally, that even in a simple parallel plate geometry, the amplitude of the peak pressure remains undetected by the force sensor of the rheometer: the fluctuations are only visible when measured over an area significantly smaller than the plate size to avoid a compensation between the low and high pressure regions.

VI. CONCLUSION

In the study of Newtonian fluids or homogeneous flows, conventional rheometers, which give access to the average values of the shear stress and normal forces, are particularly relevant. However, heterogeneous flows often occur in complex fluids, as in cornstarch suspensions. The sensor array that we present here is a new tool that brings a new insight into these flows. Here, we access to $\sigma_{22} = -\sigma_{zz}$, an important parameter that is much less studied than the shear components of the stress tensor. Our results evidence the potential of the sensor array, not only to measure the normal stresses N_1 and N_2 (as done with the HPAM solution) but to evidence and follow heterogeneities (as in cornstarch suspensions). It is, to our knowledge, the only sensor capable of evidencing the normal stresses generated by rapidly moving objects, with very high signal to noise and with a frequency up to 200 Hz. In addition, and in contrast with other pressure sensors, our system is highly versatile. The size, number, and position of the sensors can be modified by replacing the bottom electrode array, which is independent of the measurement surface.

Further developments are also possible: The addition of a soft bottom electrode would make the whole sensor array bendable enough to be added to a Couette geometry. The measurement of normal stresses has never been reported in this very common geometry, despite a huge interest of the community. It would very nicely complement the experiments (of x-ray radiography or ultrasound imaging) that have been previously made in such a geometry [21,55] with heterogeneous flows.

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