A novel rheo-optical device for studying complex fluids in a double shear plate geometry

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A new rheo-optical shearing device was designed to investigate the structural evolution of complex material under shear flow. Seeking to keep the area under study constantly within the field of vision, it was conceived to produce shear flow by relying on the uniaxial translation of two parallel plates. The device features three modes of translation motion: step strain (0.02–320), constant shear rate (0.01–400 s⁻¹), and oscillation (0.01–20 Hz) flow. Because the temperature is controlled by using a Peltier module coupled with a water cooling system, temperatures can range from 10 to 80 °C. The sample is loaded onto a user-friendly plate on which standard glasses can be attached with a depression vacuum pump. The principle innovation of the proposed rheo-optical shearing device lies in the fact that this suction system renders the microscopy glasses one with the plates, thereby ensuring their perfect planarity and parallelism. The gap width between the two plates can range from 0 to 5 mm. The device was designed to fit on any inverted confocal laser scanning microscope. In terms of controlled deformation, the conception and technical solutions achieve a high level of accuracy. Moreover, user-friendly software has been developed to control both shear flow parameters and temperature. The validation of specifications as well as the three modes of motion was carried out, first of all without a sample, and then by tracking fluorescent particles in a model system, in our case a micro-gel. Real values agreed well with those we targeted. In addition, an experiment with bread dough deformation under shear flow was initiated to gain some insight into the potential use of our device. These results show that the RheoOptiCAD® promises to be a useful tool to better understand, from both a fundamental and an industrial point of view, the rheological behavior of the microstructure of complex fluids under controlled thermo-mechanical parameters in the case of food and non-food systems. © 2013 American Institute of Physics. [http://dx.doi.org/10.1063/1.4774395]

I. INTRODUCTION

Complex fluids, such as foams, emulsions, gels, and doughs, are readily found in food and non-food products consumers use on a daily basis. These fluids, which are representative of food systems, are often characterized by the presence of mesoscopic-sized objects (0.1–100 µm). The meso-structure, for example, droplets in emulsions or three-dimensional networks in gels or doughs, gives the fluid specific visco-elastic and non-newtonian properties. Under shear, and depending on the experimental thermodynamic conditions (including temperature, pressure, pH, and aging time), the structure of the fluid may undergo rearrangement, leading to anisotropic structure destabilization. To bring under scrutiny the behavior of such complex systems, optical rheometry—otherwise referred to as rheo-optics—has proven to be a relevant and rewarding technique. Based on optical methods, it permits at once flow (rheometry) and structural observation. The aim is to correlate the changes in the structure of complex materials to the flow field, as well as to better understand the relationship between the morphology and the rheological properties of materials, thanks to the fact that the deformation of materials at the micromechan scale has an optical signature.1 The development of multiple observation techniques under shear, up until 1998, has been reviewed by Fuller2 in the case of 2D-rheo-optics and by Wagner1 in the case of 3D-rheo-optics. Since the end of the 1990s, innovation in the field of rheo-optic techniques has been focused on the development of new rheological and observational tools. van der Linden3 has recently summarized the devices

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developed throughout the last decade in the food as well as in the non-food sciences. The rheo-optical devices which have been developed are either: (i) an observation device adapted to an existing or commercial shearing system or (ii) a shearing device adapted to an existing or commercial observation system. To take advantage of the well-proven accuracy of the commercialized rheometer, some teams have developed observation techniques adapted to it. However, the power of magnification of these devices is low and the size of the area maintained within sight is highly limited. Moreover, compared to the development of an optical device, it is faster, simpler, and more economical, in addition to being more scientifically precise, to develop a mechanical shearing system to fit an existing or commercial observation system. Indeed, much work has been focused on the development of shearing tools adapted to an observation device.

To carry out deformation under controlled conditions, different means, such as a controlled strain rheometer, parallel plates, 4-roll mills, parallel plates, and others have been used. In terms of the means of producing shear in rheo-optic devices, either rotation or translation is used. Rotation has the advantage, over translation, of producing an infinite shear and strain. But from a purely technological point of view, the axis of the motor driving the rotation motion often falls into line with the axis of observation of the objective of the microscope, thereby compromising the field of vision. Motion driven by translation does not present this problem because the axis of motion is orthogonal to the axis of observation when shearing between two parallel plates.

The techniques of observation available in rheo-optic devices are numerous depending on the material and the observation scales. Often, CCD video cameras are used, capped with a magnification lens or directly mounted on a microscope. In the case of a turbid system, after the fluorescent labeling of the material, a confocal laser scanning microscope (CLSM) is used. Observations can also be carried out using laser scattering methods.

Most of the devices which have been developed are laboratory prototypes, and only a few have made their way to the market. The Linkam® shear cell is the first commercialized device for observation under rotational shear flow. It can heat the sample but its sole mobile bottom plate does not make it possible to keep the object or the region under study within the observation window, except in the case of a deformation small in amplitude. Moreover, it does not allow any measurements in terms of force. Among the non-commercial devices, the shear cell recently developed by Wu et al. based on motion by translation, retained our attention as it is the approach which best suits our scientific aims: it was designed to be adapted to an inverted CLSM and its mechanical features are highly accurate. However, several drawbacks needed to be overcome. First, the preparations before the observation of the samples, including their step of gluing the glasses to the plates, is very time consuming (around 3 h). Second, a pair of cassettes must be purchased for each sample. Third, it turns out that, in practice, it is difficult to ensure reproducible sampling. In addition, temperature cannot be controlled. Bessel-ing et al. have recently developed the “confocal rheoscope,” a combination of a rheometer and a confocal observation. The size and the weight (>10 kg) of these cells, as well as their cost, limit their use. For our needs in the food sector, we set out to build a shearing device, one that would be light and compact as well as adaptable to any inverted CLSM or other type of optical microscope. We sought to achieve a high accuracy in terms of the rheological parameters while including the possibility of regulating temperature. Our overall aim was to develop a shear cell that could be used for a wide range of textures and consistency of complex soft materials.

In this paper, we present the RheOptiCAD®, a novel rheo-optical device designed to study complex systems under thermo-regulated conditions using controlled double translation shear plates. The conception and specifications are presented as well as motor configuration and its validation. We also present and discuss our initial experiments with the device, investigating at first a simple model system, a microgel containing fluorescent particles, and then a more complex one, bread dough.

II. CONCEPTION AND SPECIFICATIONS OF THE RHEOPTICAD®

The RheOptiCAD® was designed so that three functions can be carried out, simultaneously or not:

- applying strain, constant shear rate or oscillation-controlled shear flow;
- observing a wide range of complex materials ranging in texture from a liquid to a soft-solid; and
- controlling the temperature of samples.

In our case, in which we bring food systems under observation, we sought the capacity to deform and visualize a wide range of textures and structures including emulsions and foams, gels, doughs, and solids. To define our specifications, we took into account the physico-chemical characteristics of these products as well as the available knowledge in the fields of rheology and the microscopy of complex food systems. The three main features of the RheOptiCAD®, including the various parameters that must be brought under control as well as the technological solutions that allow for this, are presented below.

A. An overall view of the RheOptiCAD®

The first prototype of the RheOptiCAD® was manufactured by CAD Instruments (Les Essarts-le-Roi, France). It is shaped like a cube with a height of 20 cm and it weighs about 5 kg. A computer-controlled electronics device relies on Ethernet communication to run the motors and RS-232C to control temperature. Figure 1 shows the developed shear cell mounted on an inverted CLSM. It has been designed to be compact and easily transportable by simply grabbing the two handles integrated right into the device’s skeleton.

B. Controlling strain and shear rate

1. Motion and motor specifications

In the case of the RheOptiCAD®, the shearing of the sample is carried out by uniaxial translation, generated by the motion of two parallel plates. Each plate is driven by
its own motor. The targeted strains and shear rates are set within the range of 0.01–10 and 0.1 s\(^{-1}\)–100 s\(^{-1}\), respectively. These values correspond to the ones frequently used when looking to characterize complex systems using optical rheometry. We chose to create motion by translation for several reasons. First of all, it is technologically quite easy to implement. Second, the motors and translation stages make it possible to achieve the specifications in terms of the displacement stress needed to deform the range of materials designated for study. Last but not least, we sought to render sampling as user friendly as possible. Indeed, readily available commercial microscopy slides—rather than time-consuming, lab-made glasses or tubes—are used to contain the samples. Since these microscopy slides are rectangular in shape, it is necessary to use the translation mode. In the case of a geometry based on parallel plates moving by means of translation, the strain and shear rates are given by Eqs. (1) and (2), respectively,

\[
\gamma = \frac{A_T}{e}, \quad (1)
\]

\[
\gamma = \frac{v_T}{e}. \quad (2)
\]

Here \(\gamma\) is the strain rate, \(\gamma\) is the shear rate (s\(^{-1}\)), \(A_T\) and \(v_T\) are the displacement (mm) and the velocity (mm s\(^{-1}\)) of the plates, respectively, and \(e\) is the gap width (mm). Due to the fact that the RheOptiCAD\(^\circ\) is mounted on an inverted microscope, the objectives under it determine the distance the plates can travel: the bottom plate no farther than 12 mm while the top plate can travel as far as 20 mm.

### 2. Load force of the motors and the generated stress

Since the systems falling under study present various rheological properties and textures, the choice of the motors depended on ensuring a sufficient load force to deform the samples in a highly consistent and precise way. The resolution of motor motion had to be precise enough to obtain a high resolution at low strain and shear rates. Given the various food products under study, within the targeted range of shear rates, the maximum shear stress had to be around 100 Pa. Linear stages, FB-075 coupled to a piezo-electric motor HR4 (NanoMotion, USA) were selected. An encoder which renders the resolution of motor motion as precise as 10 nm was selected. Table I compares target values, specifications, and the real values of the motorized stages. Finally, three motorized stages were acquired: two are dedicated to the motion of the parallel plates (top and bottom) and the third one, fixed to the top plate, is used to control gap width.

### 3. Gap width

The gap width between the two parallel plates forming the shear geometry needs to be adjustable given the variety of samples. Generally speaking, the gap should be 10-fold bigger than the objects under observation. For example, food systems contain particles varying in size from 0.1 (lipid droplets) to 50 \(\mu\)m (swollen starch granules). Moreover, for semi-solid products with high viscoelastic properties such as dough, a gap measured in millimeters renders them easier to sample. Thus, the width of the gap had to range from 100 \(\mu\)m up to 5 mm.

Planarity and parallelism of the microscopy slides are essential criteria for a fine-tuned control of the deformation. They also play a role in the quality of observation. In order to ensure these two properties, several technical solutions were experimented with. The solution opted for is based on the creation of a vacuum thanks to a pump. The microscopy slides are thereby bound to the aluminum cassette designed and made for the purposes of this study. A path for the air was etched into the planar surface of each plate and an out-flow duct was attached to connect it to the vacuum pump. A vacuum pump produced by LaboPort (KNF, France) ensures this suction system between the plate and the glass. It can work at a minimum pressure of 160 mbars. Once the pump is turned on, the microscopy glass and the cassette become one to form the moving plate. This technique applies to both the top and

<table>
<thead>
<tr>
<th></th>
<th>Target values</th>
<th>Specific.</th>
<th>Real values</th>
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<tbody>
<tr>
<td><strong>Rheology</strong></td>
<td></td>
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<tr>
<td>Gap (mm)</td>
<td>0.1–5</td>
<td>...</td>
<td>0.1–5</td>
</tr>
<tr>
<td>Strain</td>
<td>0.01–10</td>
<td>...</td>
<td>0.02–320</td>
</tr>
<tr>
<td>Shear rate (s(^{-1}))</td>
<td>0.1–100</td>
<td>...</td>
<td>0.01–400</td>
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<tr>
<td><strong>Motors’ motion</strong></td>
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<tr>
<td>Travel range (mm)</td>
<td>0.005–20</td>
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<td>0–32</td>
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<td>Resolution ((\mu)m)</td>
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<td>1</td>
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<td>Velocity (mm s(^{-1}))</td>
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<td>0–250</td>
<td>0–40</td>
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<td>Load force (N)</td>
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<td>16</td>
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<td>Frequency (Hz)</td>
<td>0.01–5</td>
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<td>Temperature</td>
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<td>Rate (°C min(^{-1}))</td>
<td>0–10</td>
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<td>0–10</td>
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bottom plates. The advantages are numerous. First, the preparations before the observation of the samples become very time efficient, mainly by sidestepping the more standard gluing procedure. Second, only a single, perfectly plane pair of aluminum cassettes has to be manufactured. Third, the samples can be quickly positioned while maintaining a high level of reproducibility in terms of planarity and parallelism. In addition, commercial microscopy slides can be used. Otherwise, a set of three micro-step screws per plate help to adjust tilt and fluctuation along the path the plates travel.

4. Zero velocity plane

In order to single out and follow the dynamics of an object inside the observation window, once that object is submitted to a strain, it is necessary that the object be in a zero velocity plane (ZVP), producible when two plates are moved in opposite directions. The location of the ZVP in the gap between the two plates can be varied by adjusting the ratio of the plate velocity (see Figure 2). Indeed, the opacity of a sample will determine where the user wishes the ZVP to be located. In the case of fairly clear samples, the laser of the CLSM can penetrate deeply enough, thereby shedding sufficient amounts of light for observation. The ZVP can therefore be located right in the middle of the gap, obtained by having both plates move in opposite directions at the same speed of motion. In the case of a fairly opaque sample, the laser of the CLSM cannot penetrate deeply enough due to attenuation and turbidity. Thus, the ZVP should be located closer to the bottom plate. This can be achieved by increasing the velocity of the top plate while decreasing the velocity of the bottom one. The location of the ZVP according to the velocities of the plates is given by the following equation (Eq. (3)),

$$z_{ZVP} = \frac{e}{V_1^2 + 1}$$  (3)

with $z_{ZVP}$ the position of ZVP in the $z$-axis direction, $V_1$ and $V_2$ respectively, the velocities of the top and bottom plates (mm s$^{-1}$), and $e$ the gap width (mm).

C. Observation of a complex system

The dynamic observation of the structure of a complex system in the shear cell was carried out using a CLSM TCS SP2 AOBs (Leica, Germany) mounted on an inverted microscope. The rheo-optical device proposed in these pages was adapted to this microscope, and geometric restrictions (for example, weight, dimensions, and security systems for lasers) were taken into consideration. However, the RheoOptiCAD® was designed such that it can be adapted to most of the inverted microscopes available on the market. The dimensions and characteristics of the objectives were also taken into account, especially in terms of getting the free working distance to fit the dimensions of the RheoOptiCAD®. With this in mind, an opening in the bottom plate—to let the laser shed light on the sample—was designed in the shape of an oval, 18 mm × 8 mm or 144 mm², defining thereby the dimensions of the observation window. The geometry of the objectives was also taken into account when designing the opening through the bottom plate, in order to make it possible for users to work with both immersion (water or oil) or air objectives. Objectives are able to come closer to the glass on the bottom plate to create a good contact in the presence of an immersion medium. Finally, we kept in mind the cost of the microscope and its accessories (i.e., objectives, confocal head and computer system). We also put into place some safety measures to protect them. For example, the length of the oval shape limits the travel range of the bottom plate, preventing any contact with the objective and therefore avoiding any damage to it.

D. Temperature control

Temperature can be controlled within a range varying from 10 to 80 °C. This range is suited to many applications and systems. A 30 mm × 30 mm Peltier system was installed on the top plate and coupled to a controller TC-XX-PR59 (SuperCool, Sweden) which has its own power supply 15V/7A (Omron, France). Water circulating in a copper part attached to the top of the Peltier system evacuates heat calories, playing an important role in regulating temperature. A thermocouple was inserted into the thickness of the top aluminum cassette. Heating and cooling rates were optimized, and they typically fall within a range from 1 to 20 °C min$^{-1}$. Calibration was carried out by using a thermocouple placed in a gap set at a width of 1 mm and filled with a PDMS oil. When heated, the maximum difference between the temperature of the top plate and the sample was 0.8 °C. When temperature was set at a constant 20 °C, this difference was less than 0.2 °C.

E. Summary table and detailed views of the shear cell

Table I shows the different parameters used for the specifications and the conception of the shear cell. By putting the prototype to the test, the target values were compared to those obtained under trial conditions. Note that for the real rheological values, the mechanical and observation limits of the plates were accounted for in the calculation, i.e., total travel ranges are, respectively, 20 mm for the top plate and 12 mm for the bottom plate, and minimal motion time 1 s. This table provides a good overview of motor characteristics and the dynamics of motion. The real values of the rheological parameters in the case of the RheoOptiCAD® are also given. These
values are in good agreement with the expected ones as well as with standard rheological values in the case of commercial tools. Figure 3 presents an image sequence of the main parts of the cell, from the gap to the complete device.

FIG. 3. 3D-images of the RheOptiCAD®.

F. Piloting software

To render the shear cell user friendly, a software, consisting of a graphic interface and a tuning configuration of the three motors (Figure 4), was developed (A2V, Rambouillet, France). It is possible to control and define all the parameters related to the positioning of the plates and the deformation mode (i.e., amplitude, velocity, the length of the experience or frequency) depending on the three given modes of motion: strain jump, constant shear rate, and oscillation shear. Also, thanks to this software, a user can easily control the positions of the top and bottom plates as well as set references as needed. The limits of the travel range are automatically defined as a function of the set reference. The position of the gap was also motorized. The gap can be easily positioned before each experiment, no matter the thickness of the microscopy slides which is known to vary from glass to glass. A maximum position button for the top plate was programmed to provide more space and thereby render sampling an easier task. Once both plates are set and the gap width is defined, the device can be set in motion. The parameters of the three modes of motion are defined as follows:

- Strain-jump: instantaneous deformation (experimental time tends towards 0) with the amplitude of each plate as variables—the 2 mobile plates move in opposite directions;
- Constant shear rate: a linear deformation over time with the amplitude of each plate and experimental time as variables—the 2 mobile plates move in opposite directions;
- Oscillation: deformation following a sinus signal with amplitude and frequency as variables—the 1 mobile plate (either the bottom or top one).

After each deformation, a data file is automatically created. It contains the data series provided by the encoders of the motorized stages relative to each experiment. Several variables are recorded: time (s), position (mm), and velocity (mm s\(^{-1}\)) of, respectively, the gap, the top plate and the bottom one. The acquisition system Galil DMC-4030 allows the simultaneous recording of around 15 000 data. More or less variables can be recorded, and sampling is automatically provided by the acquisition system. The data are dispatched equally in function of the number of variables and the length of the experiment. These data were used to validate motor configurations and achieve a suitable coherence between target and real values.

FIG. 4. Functional scheme of the piloting software.

III. MOTOR CONFIGURATION AND VALIDATION

A. Gap planarity and parallelism

The parallelism of both plates along the entire length of their x-axis traveling range was validated by using a laser reflective method. Thanks to the above-described suction mechanism, two #1 microscopy slides were made to adhere to the plates and the gap was initialized. A gap width of 450 μm was set. Using an air objective ×10, 0.3 NA (Leica), and a
543 nm He/Ne laser, the position of the laser reflection at the air-glass interface was measured. Four reflections were observed, corresponding to the two faces of the microscopy slides (Figure 5). The distance between the first and second reflections as well as the third and fourth ones represents the thickness of the microscopy slides. For three different positions \((x = -6; 0 \text{ or } 6 \text{ mm})\), a series of images \((512 \times 32\) pixels\) in the \(z\)-axis direction \((\text{velocity-gradient plane})\) were recorded every \(1 \mu m\). By adjoining all images and plotting the intensity profile, four reflections were clearly detected. The number of images between the second and the third reflection, i.e., between the maximum of intensity, corresponds to the gap width. At \(x = -6\) mm, the width was \(448.3 \mu m\), at \(x = 0\) mm the width was \(445.8 \mu m\) and when \(x = 6\) mm, the width was \(447.4 \mu m\), i.e., a difference of less than \(3 \mu m\).

This value confirms that the plates (with the attached slides as designed here) are parallel. For an \(x\)-position, a tilt seems to appear. In fact, this tilt appears due to the scanning speed in the \(z\)-axis direction of the 800 images. This apparent tilt is the same for the three different \(x\)-axis positions meaning that scanning speed was always the same. A faster confocal scanner would decrease this apparent tilt. Moreover, over a total lateral traveling distance of \(20\) mm for the top plate and \(12\) mm for the bottom plate, their reflective \(z\)-position varied less than \(5 \mu m\), ensuring a high level of planarity of the microscopy slides. The reproducibility of this gap width set-up—based on the suction system making the microscopy slides one with the aluminum plates—is shown in Figure 6. The small difference (\(<1\%\)) with the perfect planarity is mainly due to the commercial slides which are not always the same thickness. Using a similar method, the planarity of both plates was verified in the \(y\)-axis direction \((\text{velocity-vorticity plane})\). An initialization function in the software allows users to free themselves from fluctuations in glass thickness. These two conditions were also verified later in the presence of a sample in the gap width, either a gel or bread dough. Perfect parallelism was obtained in all cases. The planarity and parallelism settings can be modified and adjusted by using three micro-screws on each plate. However, over time, we observed a very good reproducibility of these two parameters, and the use of a simple laser reflective method was enough to validate them before any experiment.

### B. Validation of motor specifications in the three modes of motion

In Figures 7 and 8, positive curves correspond to the top plate position and negative ones to the bottom plate position. Before running a real experiment, the mechanical behavior of our system needed to be validated. Translation motions were first carried out with the shear cell not mounted on the microscope. The goals were: to verify the behavior of the cell and the software (tuning configuration); to validate real motions compared to targeted ones; and to ensure security settings before using the cell. The weight, and therefore the inertia of each axis being different, a specific tuning was required for each stage. On top of that, both stages had to be...
synchronized in motion. A servo controller was used in conjunction with motor drivers to adjust the proportional, integral, derivative (PID) gains of the closed loop including three coefficients involving static command and three others involving dynamic command. The feedback signal was provided thanks to an integrated high resolution encoder located within each stage. Foreseeing a general use of RheOptiCAD® under an extremely wide range of motor dynamics, we adopted three sets of different gains. It is mainly the dynamic coefficients which differentiate the three configurations. The servo controller automatically activates the proper parameters depending on the amplitude and velocities selected by the user.

By applying a strain jump from 0.1 to 5 mm for each plate, in the case of the greatest deformation (γ = 10), it takes around 0.21 s to reach the set point (Figure 7(a)). The reaction time of the device proposed here is of the same magnitude as standard rheometers or other shearing systems. By plotting these strain-jump curves between 0 and 0.02 s, it is clearly shown that the top and bottom plates are starting simultaneously (Figure 7(b)). This configuration is the means to a perfect synchronization of both plates. In the case of continuous deformation, a 4-decade range of deformation (γ = 0.01; 0.1; 1 or 10) was tested for three shear rates (γ = 0.1; 1 or 10 s⁻¹). Figures 8(a)–8(c) show the behavior of the corresponding plates. For low and medium shear rates (γ = 0.1 s⁻¹, Figure 8(a); γ = 1 s⁻¹, Figure 8(b)), both plates followed the imposed signal. When shear rate increased (γ = 10 s⁻¹, Figure 8(c)), a static error—due to a lag just after motors started—was observed between the theoretical and the actual positions of the plates. This static error could be corrected by adjusting the PID coefficients. Using the prototype without any sample ensured a good agreement between specifications and the actual behavior of the plates. As no problem was encountered at this step, the next step was to test the cell using model systems.

C. First experiments

1. Microgel model system

The optical validation of the shear cell was carried out with a microgel containing fluorescent microspheres (φ = 1 μm). For all in-time observations, the confocal microscope was equipped with a ×40 water objective (with 0.8 NA and free working distance = 3.3 mm). Gap width was set to 500 μm and a 488 nm Ar laser was used. Images (512 × 64 pixels) were recorded either in the velocity-vorticity plane (xy) or in the velocity-gradient plane (xz). Films were reconstituted with ImageJ (version 1.45, National Institute of Health, Bethesda, Md, USA). First, the non-motion of particles (not shown) was verified in the ZVP using a same velocity for both plates moving in opposite directions in the case of different strains and shear rates. Second, the motion of particles uniquely in the x-axis direction during this continuous strain confirmed the parallelism of the plates. It also indicated that in the case of the proposed system, there was no slipping between the sample and the microscopy slides during the deformation. Afterwards, the ratio of the velocities between top and bottom plates was modified. This validation was necessary for the later observation of consistency and non-transparent systems, cases in which the laser cannot penetrate deep enough into the matrix. By increasing the top velocity and decreasing the bottom one, the ZVP position can be shifted closer to the bottom plate. In these images (Figure 9), particles followed a parabola motion in the velocity-gradient plane. The location of the ZVP corresponded to the plane where particles were not moving, that is to say at the top of the parabola. When top and bottom plate velocities were the same in opposite directions, the ZVP was located in the middle of the gap (Figure 9(a)). The location of the ZVP was shifted closer to the bottom plate when the
ratio decreased, that is to say in the case of a higher top velocity (Figures 9(b) and 9(c)). For a same gap width of 500 μm, the ZVP was positioned 50 μm above the bottom plate. These three images are similar to the ones obtained by Wu et al. who investigated the velocity-gradient plane of the dispersion for a constant shear but under different velocities ratios. Oscillation motions were carried out using several frequencies. An example of an oscillating motion of the top plate (A₀ = 0.2 mm, f = 0.5 Hz) for a gap width of 700 μm is shown in Figure 10. The amplitude was chosen large enough to ensure that a centered particle remained over time in the given size of the image. By plotting the theoretical sinus and the one recorded by the Galil system on the adjoining images over the time of deformation, a perfect superimposition was observed. Neither a lag nor noisy signal appeared.

2. Bread dough

One of the targeted specifications was to be able to deform high-consistency products, which, for rewarding observations, require a force of up to around 20 N. In order to test this limit of the cell, we chose as a fitting example bread dough undergoing shear. Two recipes of bread dough were prepared with standard ingredients: flour, salt, and water. The flour was the base for determining the amount of the two other ingredients. In recipe A, the amount of water was 55 wt. % of the flour and in recipe B it was 60 wt. %. As for the salt (previously dissolved in water), in both recipes it was 2 wt. % of the flour. Using a Mixograph (National MFG Co., Lincoln, Nebraska, USA), 10 g of wheat flour was premixed with 25 mg of gluten stained by rhodamine B for 1 min at 88 rpm. Then, we added the water and salt and mixed for 4 min at 88 rpm. After placing the bread dough sample on the bottom plate of the rheo-optical device, a gap width of 500 μm was set. The sample was submitted to oscillating shearing of the bottom plate at f = 0.3 Hz for an amplitude A₀ = 0.4 mm. The images are presented in Figure 11. For recipe A, at t = 0 s, no orientation of the gluten network was observed (Figure 11(a)). After 3 s of deformation, which corresponds to an amplitude of 0.4 mm, we can clearly see in Figure 11(b) that the sample dough underwent structural changes, that is to say that the gluten network evolved into long fibers oriented more diagonally. When changing the formulation, for example, simply adding more water, as is the case of recipe B, thicker, stiffer fibers were obtained before and during the deformation (Figures 11(c) and 11(d)).

To sum up, we have seen that the evolution in the gluten network, in function of the degree of deformation and time, can be investigated by varying the formulation of bread dough. It served to prove how well the developed shear cell works with the optical and mechanical inconveniences of this high consistency product, a solid indication that the device can be used for a wide range of sample materials. More specifically, qualitative elements can be easily extracted from the images this device produces. From a quantitative point of view, image analysis methods would need to be developed and used in function of the sample chosen to come under study. In our case, as a next step, and depending on the formulation of the bread dough, the structuring or orientation of the network could come under investigation.

Finally, this set of experiments with bread dough confirms the suitability of the technical solutions chosen for shearing. No slip or motion of the slides during the shearing of bread dough was observed. The focal plane was kept constant during observation thereby validating planarity and parallelism criteria, even for samples such as bread dough which exerts a strong force during deformation. Opting to use suction with a vacuum pump to hold both slides is a real advantage for sampling and reproducibility: it is easy and fast; commercial microscopy slides can be used no matter the glass thickness; and perfect planarity and parallelism are ensured.

IV. CONCLUSIONS AND PERSPECTIVES

This paper presents a novel rheo-optical device which can be used to investigate a wide range of complex fluids—from foams to gels and doughs—under a variety of shear flow conditions, including strain-jump, constant shear rate, and oscillation. Referred to as the RheOptiCAD®, it was patented under FR 11/03828. From a technical point of view, the

FIG. 9. Images in the velocity-gradient plane for different velocity ratios ω₀/ω₀: 1 (a), 0.36 (b), and 0.13 (c). Gap width was 500 μm, and height of the image is the same as the width of the gap.

FIG. 10. Example of oscillating motion (A = 0.2 mm, f = 0.5 Hz) of microgel containing fluorescent microspheres (ϕ = 1 μm) at T = 20 °C. Gap width was 700 μm. Accordance between images over time and theoretical signal (white line).

FIG. 11. Images of bread dough submitted to an oscillation motion at t = 0 s (a, c) and t = 3 s (b, d) for 2 water content recipes: Recipe A = 55 wt. % and Recipe B = 60 wt. %. White pixels represent the gluten-protein network and black pixels correspond to air bubbles and starch granules.
principle innovation lies in the fact that standard microscopy slides are attached with a depression vacuum pump, rendering the slides one with the plate. The advantages are numerous. First, this system ensures the perfect planarity and parallelism of the plates. Second, in comparison to existing devices, the time to prepare samples for observation is dramatically shortened. Indeed, putting the slides in place by using this technique, followed by initializing the gap and setting its width, plus verifying the planarity and parallelism, takes a maximum of 20 min (and not roughly the 3 h in the case of other known devices). Third, and most importantly, this system ensures reproducible sampling. In addition, temperature can be adjusted between 10 and 80 °C using a Peltier element mounted on the top plate.

Not to be overlooked, the use of the proposed device reduces the cost of using a rheo-optical approach to investigate the structural evolution of complex material as a single pair of cassettes can be used over and over again, rather than buying or making a pair per sample, and readily available commercial slides can be used instead of fabricating them. The proposed device is therefore more readily accessible to the scientific community, either as a prototype or later as a tool that can be purchased on the market. To optimize the developed shear cell, the following technical enhancements are currently under discussion: measuring the stress produced by the deformation; increasing the range of translation; designing a bottom plate suitable for higher magnification objectives; and, integrating data analysis software. In more detail, we are seeking to measure the stress produced by the deformation by placing a surface piezo-electric sensor; however, its position may influence criteria such as planarity and parallelism. Another option might be to calculate the stress from the current intensity of the motors during deformation.

The experiments carried out to validate the technical features of the shear cell, with and without samples, revealed that real values agreed well with the targeted ones. Moreover, insight into the potential use of the device was gained by initialising an investigation of bread dough, a very high-consistency material. The images of bread dough submitted to an oscillation motion showed that qualitatively speaking the material underwent structural changes in function of shear flow. Further research will lead the way towards quantitative findings through the development of image analysis methods in function of the sample falling under study. Overall, our results show that the RheOptiCAD® promises to be a useful tool to better understand, from both a fundamental and an industrial point of view, the rheological behavior of the microstructure of complex fluids under controlled thermo-mechanical parameters in the case of food and non-food systems.

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