VIP-DROP$^2$ – A module for characterization of the spread of complex fluids droplets under various gravitational accelerations

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(Dated: 18 April 2022)

Axisymmetric droplets provide a demonstration system to study the spread of complex fluids, which are an essential component of industrial and technological processes, among which additive manufacturing (AM). Tweaking the gravitational environment provides access to different regimes, quantified through the Bond number; in particular, microgravity allows to form large droplets while remaining in the regime where the surface tension effects and internal driving stresses are predominant over hydrostatic forces. The VIP-DROP$^2$ module offers a versatile platform to study a wide range of complex fluids through the deposition of initially axisymmetric droplets. It offers the possibility to deposit the droplets on a precursor layer: a thin, homogeneous layer, which can be made of the same or of a different fluid. Besides, it allows to deposit simultaneously 4 droplets, while conducting shadowgraphy on all of them, and observing either the flow field through particle image velocimetry (PIV), or the stress distribution within the droplet in the case of stress birefringent materials. Developed for a drop tower catapult system, it is designed to withstand a vertical acceleration of up to 30 times the gravity of Earth in the downwards direction, and can operate remotely, under microgravity conditions.

I. INTRODUCTION

Many industrial applications involve the deposition and spreading of complex fluids on solid surfaces. Examples include various methods of coatings$^{1,2}$, inkjet printing$^{3,4}$, and additive manufacturing (AM)$^{5-8}$, where usually a thin film, a droplet, or a filament of a complex fluid spreads on a surface. Hence, the spreading of films and droplets (from Newtonian to polymeric fluids) has been studied extensively to improve and optimize the current technologies$^{9-13}$. However, given that most of the previous studies have been performed under the Earth’s gravitational conditions, our knowledge of spreading in low and zero gravity is limited. This lack of knowledge is even more pronounced for fluids with complex rheological properties such as viscoelasticity and viscoplasticity.

The spreading of droplets is typically a function of surface tension, the interaction with the solid surface, inertia, body forces (including gravity), and the rheological properties of the material. In this context, the importance of gravity is typically measured by comparing the hydrostatic pressure $\rho g L$ (where $\rho$ is the fluid’s density, $g$ the gravitational acceleration, and $L$ a typical length scale of the droplet) and the capillary pressure $\sigma/L$ (where $\sigma$ is the surface tension of the fluid). The relevant dimensionless number is then the Bond number,

$$B = \frac{\rho g L^2}{\sigma}. \quad (1)$$

A plethora of experimental work addresses the regime of large $B$, which represents the gravity-dominated regime applicable to large-scale phenomena on the ground, such as landslides or lava flows$^{14,15}$. The focus of this study is, however the limit of $B \to 0$ in a regime where $L$ is finite and relatively large, i.e., in the low-gravity regime.

Investigations of the spreading of complex fluids under various levels of gravity have important implications for at least two primary reasons. First, such studies are crucial for developing space technologies that rely on the spreading of complex fluids. For instance, these include the AM of thermo-plastics (e.g., for on-site printing tools in space stations), 2D and 3D printing soft materials (e.g., high precision electronics and bio-printing organs), and large-scale 3D printing of cement-like materials for possible habitats on the moon (and other planets)$^{16-19}$. Second, experiments under low gravity conditions allow exploring limits that are difficult to study under the Earth’s gravity. For instance, on ground, the limit of negligible gravitational effects is mainly achievable by significantly reducing the characteristic size of the droplets. This,

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Gravity-related experimental platforms such as drop towers, parabolic flights or sounding rockets, among others, are great candidates to provide insight into the problem of spreading under different levels of gravity. Here we describe a setup that has been designed and built to allow for the investigation of the spreading of droplets (with controlled volume and deposition speed) in weightlessness ($g \to 0$). The experimental hardware was developed for the Center of Applied Space Technology and Microgravity (ZARM) drop tower (Bremen, Germany) under the name VIP-DROP$^2$ (Visco-Plastic DROPlents on the DROP tower).

Axisymmetric spreading droplets provide an excellent demonstration system towards understanding and modeling the spread of complex fluids. On the one hand, they are closely related to applications where single droplets or filaments are deposited on the surface (e.g., printing). On the other hand, despite its geometrical simplicity, axisymmetric spreading remains a complex problem because it involves dynamic phenomena happening simultaneously on multiple length scales.

Our experimental setup can be used for materials with various rheological properties. A main focus of our work is to study viscoplastic (yield-stress) fluids. Such materials behave like an elastic solid at low stresses, but above a critical stress (the yield stress), the material flows like a viscous fluid. Viscoplastic fluids are widely used in industrial units and studying their spreading mechanisms is essential to understand and optimize technological processes. The spreading of viscoplastic fluids features a finite spreading time, when stress everywhere inside the droplet is below the yield stress. Hence, the droplet will arrive at a final shape that depends on many parameters, including the surface tension, properties of the fluid, and the physicochemical properties of the surface. Another class of fluids that we study are shear-thinning viscoelastic fluids that are characterized by a large structural relaxation time, and hence behave as fluids with an apparent yield stress on time scales that are short compared to their relaxation time.

Surface-specific interactions complicate matters for the spreading of droplets. Here, we simplify the problem by studying droplets on a pre-wetted surface that is covered by a thin film of the same fluid as the one composing the droplet. Under these conditions, a droplet of a Newtonian fluid would evolve from the initial shape toward a completely flat state. Droplets of yield-stress fluids, on the other hand, evolve with time towards a flattened dome-like shape of finite size, where a balance between surface tension, the material’s yield stress and hydrostatic pressure (in the presence of gravity) is attained. The application of such a precursor layer can also produce a more appropriate representation of the system studied. For example, in AM and paint spray applications, liquid droplets or filaments are deposited on an existing layer of the fluid. Hence, understanding the underlying fluid mechanics of droplet spreading on a thin film helps to improve the design of such systems as well.

While the precursor layer simplifies the physics of the problem and the associated theory by providing abstraction from surface-chemistry effects, it poses an experimental challenge: a thin film of the yield-stress fluid needs to be created reproducibly before each experiment. In the case of experiments in weightlessness, this typically needs to be attained by an automated procedure just at the beginning of the microgravity phase, since the thin films cannot be expected to be stable over the prolonged preparation time of a microgravity experiment, nor with respect to the strong forces that act, e.g., during the catapult launch at the drop tower or during the launch of a sounding rocket.

Our experimental setup provides the possibility for extensive characterization of the spreading behavior of complex fluids during and after their deposition. The deposition can be done directly on a substrate of a chosen material, but can also be preceded by the deposition of a pre-wetting film of variable height, constituted of either the same or a different fluid. The setup is inherently modular, in that it allows for a wide variety of fluids to be tested, with no predetermined limitation in yield stress or viscosity of the experimental fluid. The module allows to study up to four droplets in parallel, in order to maximize the utilization of limited drop-tower time. Besides, the module offers the possibility to simultaneously conduct three types of measurement on all or some of the droplets: simple shadowgraphy provides basic information on the temporal evolution of the spreading; particle image velocimetry (PIV) on fluids with embedded tracer particles provides simultaneous information on the internal flow fields; internal stresses can be visualized by a rheo-optical method for fluids that show flow-induced birefringence.

In the following, we describe the design and implementation of the VIP-DROP$^2$ module, and first reference measurements that have been obtained during a drop-tower campaign at ZARM. We first detail the available measurement techniques that are implemented in the module (Sec. II), and after that we describe in detail the specifications and implementation-specific details of the module (Sec. III). Initial experimental results are presented in Sec. IV, after which a summary and outlook are presented in Sec. V.
II. MEASUREMENT METHODS

We start with a brief summary of the principles of the experimental techniques that are implemented in the VIP-DROP$^2$ module.

A. Shadowgraphy

The simplest but also one of the most important parameters to describe the spreading of a droplet, is its height profile $h(t)$ as a function of time. Shadowgraphy is a robust and easy experimental technique to gather this information: Illuminating the droplet from one side with a homogeneous light source, and observing by a camera from the opposite side, images are obtained where the radial extent of the droplet is visible. In principle, since inhomogeneities in optical media change the light refraction, shadowgraphy gives access to more detailed information, but in the present situation the binary information encoding the droplet cross-section is sufficient. A simple edge-detection algorithm can thus be employed to process the images in order to extract the time-dependent droplet profile under assumption of axial symmetry around the depositing nozzle.

From the profile $h(t)$, we can in particular extract the droplet radius $R(t)$. In the case of yield-stress fluids whose spreading comes to a halt at a finite radius, the long-time behavior of $R(t)$ allows to determine the asymptotic radius $R_f$ for which theoretical predictions in the form of separate scaling laws for the regimes $B = 0$ and $B \gg 1$ exist$^{13}$.

Shadowgraphy is also possible from below the droplet, which gives access to the time-dependent shape of the droplet’s rim, and thus allows to explicitly check the assumption of axial symmetry.

B. Particle image velocimetry

Of specific interest beyond the information on the macroscopic shape of the droplet, is its internal flow field. Calculations based on the Navier-Stokes equations in the thin-film limit and supplemented with empirical material laws, suggest that the flow field of a yield-stress fluid has a rich phenomenology: owing to the fact that locally the imposed stress remains below the yield stress of the fluid, a stagnation zone appears in the center, and a plug-flow zone near the top surface of the droplet, separated by a localized shear band$^{13}$.

We employ particle image velocimetry (PIV)$^{29–31}$ to observe the flow field of the spreading droplets. PIV gives access to the Eulerian velocity field, i.e., the locally resolved instantaneous fluid velocity. For this, fluorescently labelled tracer particles (typically with sizes in the range of 1 μm to 10 μm) are embedded in the fluid, small enough to provide least possible disturbance to the flow field, and large enough to be visible, and such that they can be assumed to be advected by the fluid flow. A laser sheet illuminates one cross-section of the sample, so that the fluorescent emission of the tracers in this cross-section can be detected by a camera. From the analysis of two consecutive frames captured by a high-speed camera, the local velocity of the flow at the position of the tracers can be determined.

PIV has been employed previously for drop-spreading experiments$^{32}$, albeit observing through an inverted microscope and a confocal scanning unit placed below the droplet. In the present setup, the laser sheet is oriented such that a cross-sectional plane orthogonal to the surface of deposition is observed with a camera placed on the side of the droplet. This gives information on the height-dependence of the velocity field; assuming that the flow inside the droplet is axisymmetric to a good approximation, the full information can then be reconstructed, if the laser sheet is placed to cut through the central axis of the droplet as closely as possible.

C. Rheo-Optical Measurements

Flow in fluids in general causes internal stresses. In particular in non-Newtonian fluids, the transient evolution of these stresses as a function of time does not instantaneously follow the current flow field, but stresses build up and are released with a time delay that depends on the local structural relaxation mechanisms in the material. Spatially resolved measurements of internal stresses in the droplets thus reveal localized rearrangements in the structure of the fluid as it spreads.

A convenient method to observe internal stresses in optically transparent materials at least qualitatively, in specific cases also quantitatively, is based on the stress-optical law first formulated by Maxwell: it links the optical indices of refraction to the principal stresses inside the material. For solid materials, the observation of the resulting load-dependent birefringence patterns is a technique called photoelasticity. Since the classical work of Frocht$^{33}$, it has developed into a standard method for the assessment of internal stresses in transparent solids or transparent analog-models of mechanical parts$^{34}$.

In fluids, the momentaneous stresses, by the same physical mechanism give rise to a phenomenon called flow-induced birefringence. It is a powerful optical rheological method used to obtain information on the flow of complex fluids, especially in many polymer melts that display a large rheo-optical effect$^{35}$.

The principle of rheo-optics is the assumption that at each point within the material, the optical properties are expressed in terms of three principal refractive indices $n_i$ ($i = 1, 2, 3$) for electromagnetic waves whose polarization is aligned with the principal stresses $\sigma_i$,

$$n_i - n_j = C(\sigma_i - \sigma_j),$$  \hspace{1cm} (2)

with a material-specific constant $C$ called the stress-optic or photoelastic constant. Hence, the material becomes optically birefringent in response to stress,
such that transmitted polarized light is rotated by an angle that corresponds to the distribution of local stresses along the light-ray’s path through the material. Observing the transmitted light under crossed polarizers thus results in typical birefringence patterns that can be analyzed. In practice, the use of circular (instead of linear) polarizers helps to eliminate further spurious transmission from refraction and eliminates dark lines known as isoclinics that arise purely from the geometry of the linear polarizer setup. Taking images both from the side and from below the droplet, two different tomographic projections of the internal stresses can be obtained.

III. SPECIFICATIONS

A. Microgravity platform

The VIP-DROP\textsuperscript{2} module is designed for the ZARM drop tower (see Figure 1) situated in Bremen, Germany\textsuperscript{20,23,24}. The 146 m high tower offers the possibility to perform experiments in weightlessness by letting the experiment fall for approx. 4.6 s in an evacuated tube so that aerodynamic drag is avoided and free-falling conditions are achieved. The available time for such a “flight” can be essentially doubled by first catapulting the experiment module to the top of the tower. Under these conditions, the experiment module is effectively in microgravity for approx. 9.5 s, with an acceleration of $10^{-5} g$ (where $g \approx 9.81 \text{ m s}^{-1}$ is the gravity of Earth).

The high quality microgravity obtained in the ZARM drop tower allows to conduct experiments that are very sensitive to gravity-jitters. This high quality is important in droplet-spreading experiments, since in principle, yield-stress fluid droplets could be sensitive to vibration-induced deformations that complicate the measurements\textsuperscript{37}.

B. VIP-DROP\textsuperscript{2} module

The module, shown in Figure 1, is divided into three platforms that are arranged vertically and mounted on stringer elements forming the base structure of the drop-tower capsule. The intermediate platform 2 houses the main experiment with the central components. Platform 1 (underneath) contains the recording modules and imaging system, whereas the electronic components are mounted on platform 3 on top. The module is made to attach to the capsule base from ZARM, which contains the Capsule Control System (CCS) and additional elements such as sensor systems and the battery. Together with the outer shell, this assembly forms the final capsule that is launched in the drop tower.

The simultaneous deposition of four droplets is made possible by dividing the main experiment platform into four equal sections arranged in a cross, labeled for later reference as North (N), East (E), South (S) and West (W). Each section allows for the independent preparation and examination of one droplet on a transparent, pre-wetted glass substrate. They are each equipped with independent syringe pumps for pre-wetting on deposition (see Fig. 2), so that four different materials can be examined during one flight, optionally also pre-wetting on films of different materials. On each section, different independent measurement units are installed, and corresponding cut-outs in the platform base allow to also observe the droplets from below. (The latter option is realized only for the two sections N and S in the current setup.)

In each section, a deposition syringe is connected to a nozzle via tubing and a standard Luer-Lock connection. The nozzle unit of the deposition system can be precisely adjusted with a linear stage to be at a certain distance from and perpendicular to the glass substrate. A pre-wetting syringe is connected to a combination of a custom made multi-nozzle element and a wiper to allow to create homogeneous thin films of fluid.

The four glass substrates are placed on a rotary stage. Just after entering the microgravity phase and depositing on the glass substrates the corresponding fluids for the pre-wetting films, the rotary stage moves...
The structure of one section of the main experiment platform: The designed CAD model next to a close-up of the deposition and pre-wetting system of the final experiment, together with the glass substrate in experiment position.

The glass substrates from the pre-wetting position to underneath the droplet-deposition nozzles, thereby spreading the pre-wetting fluids into a thin homogeneous layer (see below).

The four pre-wetting and four deposition syringes are driven by an individual linear motor each. All motors are controlled individually by the experiment control computer. With this it is possible to precisely control the amount of fluid and the deposition rate of all the fluids separately. The motor control also implements a configurable amount of retraction of the syringe pistons, in order to stop the deposition of yield-stress fluids as instantaneously as possible: without retraction, the cessation of the flow out of the deposition nozzle produces some leakage that depends on the rheological properties of the fluid.

The temperature inside the capsule is recorded with a type K thermocouple, placed on the experiment platform close to the deposition nozzle (see Figure 3). The current setup is intended for fluids whose rheology is only weakly dependent on temperature around ambient conditions, so that no active temperature control is needed.

C. Image capture

On each of the four sections, two cameras can be mounted in order to provide both imaging from the side and from below the droplet (see Fig. 3). Corresponding LED panels to the side and above the droplet provide backlight for the cameras, so that shadowgraphy and rheo-optical information can be recorded. The LED panel on the top of the deposition area is pierced in its center to allow passage of the nozzle. In the current setup, the sections W and E only implement side-view cameras, so that the top LED panel has been omitted in these sections. In the case of the experiment section N that is used for PIV, no LED backlight panels are installed, as here the camera shall predominantly record the fluorescent light emitted by the PIV tracer particles. (Limited shadowgraphy information can still be extracted from these images.) For measurements of stress-induced birefringence, the LED panels are covered with circular polarizer foil, and the camera objectives are equipped with corresponding circular polarizing filters.

High speed cameras are used on all sections. In the current implementation, two Photron FastCam MC-2 recording systems available from ZARM were installed, providing four camera heads in total, each with a $512 \times 512$ CMOS chip recording at 500fps either in grayscale or, for the use for birefringence measurements on section S, 32bit color mode. Two additional USB-3 cameras (Ximea MQ013MG-ON) were installed and connected to an Intel-NUC computer; these provide recordings with a resolution of $1264 \times 1016$ px at up to 150fps in color mode (for PIV), and $640 \times 512$ px at up to 500fps for b/w shadowgraphy.

The laser used for PIV is mounted vertically on
FIG. 4. Laser setup and beam guidance. (a) Illustration of the setup, showing the vertically mounted laser module, and the light-sheet optics to the side of the deposition system. (b) Photograph of the glass substrate with a deposited droplet, illustrating the laser-light illumination. (c) Exemplary camera recording from the side of the droplet.

the main stringer structure, oriented downwards (see Fig. 4a). The model used is a 532 nm continuous-wave (CW) diode-pumped solid-state (DPSS) laser (LaserGlow LRS-0532) with optical output power of 500 mW. A laser sheet is formed from the laser beam using a cylindrical lens (Edmund 12.7 mm × 12.7 mm, −25 mm FL, Uncoated Laser Grade PCV), and oriented across the droplet using a 45° mirror (Thorlabs Broadband Dielectric Mirror, 400 nm to 750 nm). The resulting laser sheet is visible as a bright streak in Fig. 4b. An optical filter (Thorlabs 625 nm CWL, Hard Coated OD 4.0, 50 nm Bandpass Filter) is placed on the side-view camera objective to filter out the wavelengths captured besides those emitted by the fluorescent tracer particles (cf. Fig. 4c).

For additional visual inspection of the experimental procedure, three action cameras (GoPro) were installed on the main experiment platform. Two allow to assess the pre-wetting and deposition procedure on the E and W sections, while a top-view camera provides an additional overview.

D. Pre-wetting mechanism

An automated system allows to create a precursor film on the substrates, prior to the deposition of a droplet, by depositing and spreading a small quantity of material into a homogeneous film. The syringe used for the deposition of the pre-wetting fluid is distinct from that used for the deposition of the droplet or filament studied, allowing the precursor layer to be of a different fluid from that of the main droplet.

The pre-wetting mechanism, shown in Fig. 5, consists of a custom-made fixed assembly of an array of nozzles (labelled multi-nozzle), a fixed steel blade chamfered at 45° to create a defined edge (labelled wiper), and a glass substrate moving under the wiper. The multi-nozzle first deposits an array of droplets over its full length, which are then mechanically spread as the glass substrate moves under the wiper.

All of the glass substrates are fixed onto the same rotary plate, which rotates around the center of the main experiment platform. With this, it is possible to execute all pre-wetting procedures simultaneously for all four sections, as the rotary plate rotates ≈80° clockwise (see Figure 6) and moves the glass substrates under the wipers.

The full pre-wetting procedure takes approx. 1.4 s and is executed after the hypergravity phase of the capsule launch. A full sequence of images of the pre-wetting procedure is shown in Figure 7. Initially, the rotary stage is positioned such that the inner tip of the blade is just in front of the front edge of the glass substrate, ensuring that fluid is spread over the entire glass surface.

The homogeneity of the precursor layer depends on multiple factors, including the rheology of the pre-wetting material, the timing and speed of successive events (e.g., onset of deposition of pre-wetting fluid and rotation of the rotary stage), the angle between the multi-nozzle and the glass substrate, and the quantity of material deposited by the multi-nozzle.

The homogeneity of the layer was tested on an exemplary viscous fluid by optical coherence tomography (OCT) in the lab, and was assessed by visual inspection through GoPro video recordings for the flight campaigns.
FIG. 6. Movement of the rotary stage during the experiment: (a) Initial pre-wet position with the glass substrates just before the wipers; a clockwise rotation of the stage (indicated by arrows) moves the glass substrates underneath the stationary wipers. (b) Final experiment position with the glass substrates centered under the deposition nozzles.

FIG. 7. Motion of the rotary stage during the pre-wetting sequence. Left: initial pre-wetting position; center: during rotation; right: final position under the deposition nozzle. Images taken from one of the action cameras installed on the setup.

E. Microgravity Considerations

Since there are moveable masses inside the setup, an analysis of the residual acceleration and angular velocities of the capsule has been performed (Fig. 8). Note that the overall volume of fluid that is deposited (on the order of 20 mL) is small, so that no drastic influence on the $\mu g$ quality is expected. Indeed (Fig. 8a), the deviations in vertical acceleration are too small to be resolved by a standard accelerometer (with measurement error around $10^{-2}g$).

The motion of the rotary stage necessarily causes a counter-rotation of the entire capsule due to conservation of angular momentum. This is seen in the measured angular velocity around the vertical $z$-axis during the flight (Fig. 8b): a small dip is seen around 2 s after launch, corresponding to the stopping of the rotary stage. The disturbance is seen to be small on the scale of the overall rotation rate of the capsule.

IV. RESULTS

The VIP-DROP$^2$ module was used during multiple campaigns at ZARM between November 2021 and June 2022 to obtain data in microgravity, using the drop tower’s catapult system to provide sufficient experiment time for the observation of the spread of large droplets. We report here preliminary data analyses to exemplify the capabilities of the module. A more in-depth discussion of physical effects will be provided elsewhere. Data have been obtained both under microgravity conditions (labeled $\mu g$), and in corresponding ground experiments in the same setup and using the same materials and experiment parameters (labeled $1g$).

The four locations provided by the module were exploited as follows: (a) PIV is conducted on the N section. Imaging from below also allows to verify the sphericity of the droplet and confirm its radius. The PIV data is obtained at 50 fps using one of the Ximea cameras. (b) The E and W sections were exclusively used for shadowgraphy, with frame rates of respectively 500 fps and 240 fps. (c) In the S section, stress birefringence patterns were observed with 500 fps, combining imaging from the side and from below.

A. Experimental fluids

Two material classes were used for the experiments. Shadowgraphy and PIV were performed on aqueous
The simplest quantity to extract from the shadowgraphy experiment is the radius of the droplet as a function of time, $R(t)$. Figure 10 shows exemplary evolutions of $R(t)$, where $t = 0$ s is the time at which extrusion effectively starts.

In the case of Fig. 10, the radius is evaluated from the side views of droplets (similar to the ones shown in Fig. 9) of Fluid c.5, the Carbopol solution highest yield stress. The four curves correspond to two droplets sizes (characteristic lengths $L$ of 1.7 cm and 0.87 cm, respectively blue and red symbols), under Earth gravity and microgravity (respectively full and empty marks).

The data demonstrate the influence of gravity on the droplet spreading clearly: in both cases, the fluid spreads faster in the presence of gravitationally-induced hydrostatic pressure. In other words, the experiment is performed in a regime where $B > 0$ in the presence of gravity, and $B = 0$ is achieved keeping all material parameters constant but eliminating gravity.

C. Particle image velocimetry (PIV)

For PIV, some of the carbopol dispersions were seeded with fluorescent-labeled tracer particles. The trac-
ers are 10 µm diameter polystyrene (PS) particles from MicroParticles GmbH (product PS-FluoRed-10.0). Polymerized with organic fluorescent dyes, they absorb light at a wavelength around the laser excitation of 532 nm, and emit fluorescence light at a wave length of 607 nm.

An exemplary velocity-field reconstruction in a spreading droplet is shown in Fig. 11. The droplet side view was recorded with 100 fps, and to reduce noise, four consecutive frames were averaged. PIV was performed on pairs of pre-averaged images separated by two frames, using the software OpenPIV (version 0.23.8)\textsuperscript{47}.

Especially for large droplets, performing PIV from the side has the drawback that the droplet induces a “lens effect”, so that due to total inner reflection, dark regions appear in some of the PIV images. This can be mitigated by placing the laser sheet slightly less centered for large droplets.

Already from the exemplary images, one can see that there is a dramatic effect of the deposition rate for the micellar fluid, both on the shape of the droplet and on the magnitude of the internal stresses. In particular, for the faster deposition rate, we observe the formation of a high “pillar” of the viscoelastic fluid under microgravity conditions, that remains stable over the time scale of the experiment. The same material, with the same extrusion speed, on ground forms much flatter droplets, demonstrating the role of microgravity conditions to stabilize the extrusion of tall structures of a soft material.

A rough estimate of the stresses from the blue-ish color that is clearly visible in fig. 13, making use of suitable interference color charts\textsuperscript{48}, confirms that the internal stresses are comparable to the yield stress of the material, around 10 Pa to 50 Pa.

V. CONCLUSION

We have described our implementation of a drop-tower module to study the spreading of yield-stress fluids in weightlessness. The motivation to perform such studies is given by the fact that droplet spreading in the presence of gravity, i.e., at finite Bond number, is notably different from the spreading in the surface-tension dominated regime at $B = 0$. The latter regime is the one of interest of AM applications. Performing experiments under microgravity conditions, large droplets can be used to perform optical analysis of the droplet shape, its internal velocity, and also its internal stress fields, while maintaining small Bond numbers. The microgravity experiment essentially achieves to decouple the limit of small Bond number from the material and deposition parameters of the yield-stress fluids.

Further developments could include an adaptation of the experimental module for different microgravity ($\mu g$) platforms in order to provide longer time in

| Fluid | Composition | Yield stress | Surface tension |
|-------|-------------|--------------|----------------|
| c.1  | 12 PAA + 28 H2O | 6.5 Pa | 0.07 N m$^{-1}$ |
| c.2  | 14 PAA + 26 H2O | 9 Pa | 0.08 N m$^{-1}$ |
| c.3  | 16 PAA + 24 H2O | 14 Pa | 0.09 N m$^{-1}$ |
| c.4  | 18 PAA + 22 H2O | 21 Pa | 0.1 N m$^{-1}$ |
| c.5  | 20 PAA + 20 H2O | 35 Pa | 0.11 N m$^{-1}$ |

TABLE I. Experimental parameters for the Carbopol dispersions used in the experiments. Surface tension values are extrapolated from measurements by Boujlel and Coussot\textsuperscript{38}.

| Fluid | Composition (CTAB/NaSal) | Viscosity | relaxation time | apparent yield stress |
|-------|--------------------------|-----------|-----------------|----------------------|
| sb.1  | 200/120 mM               | 167 Pa s$^a$ | 1 s$^b$         | 150 Pa$^b$          |
| sb.2  | 160/96 mM                |           |                 |                      |
| sb.3  | 120/72 mM                |           |                 |                      |
| sb.4  | 100/60 mM                | 1200 Pa s$^c$ | 30 s$^c$        | 40 Pa$^c$           |

TABLE II. Parameters of the micellar solutions used as stress birefringent materials in our experiment. Values for viscosity, structural relaxation time and apparent yield stress are taken respectively estimated from literature: $^a$ Gladden and Belmonte\textsuperscript{42}; $^b$ Gladden, Skelton, and Mobley\textsuperscript{43}; $^c$ Hartmann and Cressely\textsuperscript{45}.

D. Rheo-Optical Measurement

A detailed analysis of the rheo-optical data obtained from our setup is beyond the scope of the present contribution. We only show here a set of exemplary images to allow for a qualitative assessment.

In Figures 12 and 13, droplets of the micellar fluid sb. 1 (CTAB/NaSal 200/120 mM) are shown at three representative instants in the experiment: first, shortly after the effective start of extrusion; then right before the end of extrusion; finally, in the last instant preceding impact for the microgravity case, and at the exact same time for the ground one. Figures 12 and 13 show experiments at respective extrusion speeds of $Q = 1.875 \cdot 10^{-3}$ m s$^{-1}$ and $Q = 7.5 \cdot 10^{-3}$ m s$^{-1}$. For these experiments, no pre-wetting layer was induced since the spreading of highly visco-elastic fluids into thin films proved unsatisfactory.

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weightlessness, for example to study the deposition of filaments of viscoplastic materials that are relevant for AM. Possible platforms include sounding rockets (providing several minutes of experiment time in weightlessness).

A more detailed analysis of the results provided by our drop-tower module will address the comparison with scaling laws predicted by the thin-film solutions of the Navier-Stokes equations for viscoplastic fluids. It is expected, that the case $B = 0$ is governed by different scaling exponents, and achieving this limit with large droplets will allow to verify the scaling law without being hampered by experimental artifacts arising from small droplets.

The PIV measurements can be used to determine the gravity-dependence of the yield surface inside the droplet, separating the region of plastic flow in the region with large stresses from the “plug” flow in the region where the stresses remain below the yield stress.

Future extensions of the setup could include particle tracking velocimetry (PTV), a Lagrangian method where the tracer particles are individually tracked. Suitable methods have been developed for droplets, including aigmatic PTV\(^{49}\), which allows the tracking in three dimensional (3D) with a single camera utilizing its astigmatic imaging defects to reconstruct out-of-focus plane positions, and OCT that has allowed to study the flow fields of evaporating droplets at various inclination angles (and hence various effective gravity levels)\(^{50}\).

We wish to propose the analysis of stress-induced birefringence in droplets of yield-stress fluids (or shearthinning viscoelastic fluids with a large apparent yield stress) as a qualitative, and ultimately qualitative tool to predict internal stresses in yield-stress materials. To this end, the birefringent images obtained in our experiment will be compared to computer simulation combined with ray tracing, potentially using machine learning to address the inverse problem of reconstructing the internal stress field from the experimental images.

For the present analysis, we have largely ignored the dynamics of the droplet in the moment of impact at the end of the drop-tower flight. These moments under strong hypergravity conditions could potentially be exploited further to provide data on the effect of external acceleration on the fluid’s spread, such as in centrifugal casting or density-based separation methods.

ACKNOWLEDGMENTS

We acknowledge Kasper van Nieuwland, Clint Ederveen Janssen, Daan Giesen, and Tjeerd Weijers from TC section of the University of Amsterdam for their invaluable help in designing and building the experiment. ODA acknowledges European Low Gravity Research Association (ELGRA) through the ELGRA Research Prize. We acknowledge European Space Agency (ESA) for providing the drop-tower flight opportunities at the Center of Applied Space Technology and Microgravity (ZARM) through the ESA-CORA program. We also thank Thorben Könenmann from ZARM for his support and Fred Oetken and Jan Siemer for their always friendly help during the campaigns.

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FIG. 12. Droplets of micellar fluid exhibiting stress birefringence patterns, (a) on-ground and (b) under microgravity conditions, for the fluid sb. 1 (CTAB/NaSal 200/120 mM). On both levels of gravitational acceleration, material extrusion was done at a volumetric rate of $Q = 1.875 \times 10^{-3} \text{ m s}^{-1}$. Time $t$ is given as time since the effective start of extrusion $t_0 = 0 \text{s}$. The scale bar (white, bottom left of each picture) represents 2 mm.

FIG. 13. As in Fig. 12, but for a four-fold increased volumetric deposition rate, $Q = 7.5 \times 10^{-3} \text{ m s}^{-1}$.

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