

# Shear History Extensional Rheology Experiment (SHERE)

The resistance of a fluid to an imposed flow is termed ‘viscosity’, and it is a fundamental material parameter by which manufacturers and end-users characterize a material. Normally, researchers place a material in a commercial instrument that imposes a simple rotational shearing flow and obtains a rate-dependent shear viscosity. While this level of characterization is sufficient for some processes, in typical industrial polymer processing operations the material experiences a complex flow history with both shear and extensional characteristics. For example, in fiber spinning, the fluid experiences a complex rotational shear flow as it flows through the spinneret head before entering a region of dominant axial elongation in the spinline.



Polymer behavior under these conditions is process-dependent and stems from their long chain structure. Polymers are typically hydrocarbon-based molecules composed of repeated molecular units and can contain hundreds to tens of thousands of these repeat units. The resulting long molecular chain is usually very flexible, allowing the polymer to coil, extend, and entangle with neighboring polymer chains. In its rest state, a typical polymer chain will assume a random coiled configuration. When exposed to a rotational shearing flow, this coil will align 45° to the flow direction and flip over and over again to coil the polymer chain. When exposed to an extensional flow, the coil extends axially and can be pulled taut if the flow is strong enough. Because polymers act like springs, more stress is required to stretch them to higher strains. This relationship between stress and extensional deformation rate (i.e., strain rate) is expressed as an extensional viscosity and is a fundamental material parameter independent of shear viscosity.

## Science Overview

Due to the coiling effect of rotational shear flow on the polymer chains, shearing on the fluid immediately before extension will influence the extensional behavior of the fluid. Therefore, the main objective of SHERE is to study the effect of rotational preshear on the extensional behavior of the fluid. Of specific interest is the transient evolution of the microstructure and viscoelastic tensile stresses that are present during the extension of the fluid.

The combination of both shearing and extensional flows is common in many polymer-processing operations such as extrusion, blow-molding and fiber spinning. Therefore, knowledge of the complete rheological properties of the polymer fluid is required to accurately predict and account for its flow behavior. In addition, if numerical simulations are to serve as a priori design tools for optimizing polymer processing operations, then it is critical to have an accurate knowledge of the extensional viscosity and its variation with temperature, concentration, molecular weight, and strain rate.

Unlike common Newtonian fluids, complex fluids such as polymers cannot be characterized by a single material parameter such as the Newtonian (shear) viscosity. Instead, they exhibit nonlinear responses to imposed deformations. The extensional function of non-Newtonian fluids is not constant but depends on both the rate of deformation and the total strain experienced by a fluid element.



A class of dilute polymer solutions, collectively referred to as ‘Boger fluids,’ has become a popular choice for rheological studies of non-Newtonian fluids and will be used in this experiment. These ideal elastic fluids exhibit a nearly constant shear viscosity, which allows a direct comparison of Boger fluids with Newtonian fluids having similar viscosities. The high viscosity of the suspending solvent results in long relaxation times and substantial normal stresses, and the low concentration of high molecular weight polymers facilitates modeling analysis.

### **Experiment Operations**

SHERE is designed to fly in the Microgravity Science Glovebox (MSG) on the International Space Station (ISS). The main SHERE hardware consists of the interface box, rheometer, camera arm, cabling, keyboard, and toolbox as shown in Figure 1. In addition, there are 25 fluid modules in a stowage tray (not shown). The interface box contains all power distribution, controllers, and data acquisition and storage. It also contains the video system that combines the camera view and data display onto one video signal for recording and downlink. The rheometer (see Figure 2) contains the rotational preshear motor, translation slider, sensitive force transducer, electroluminescent backlight panel, laser micrometer, and thermistors. During testing it will also contain one of the 25 fluid modules. The camera arm attaches to rheometer for video recording of the stretched fluid’s shape. The toolbox contains miscellaneous tools used during setup and operation of the experiment. The keyboard is used to control the experiment with the help of the MSG video monitor. The 25 fluid modules contain the fluid that will be sheared and stretched during the experiment. Each fluid module contains prepackaged Boger fluid, and all samples are identical.

The fluid modules are stored at 20°C at least 24 hours prior to testing. After the hardware is installed in the MSG, the experiment goes through a set of initial check-out and calibration procedures. Once these procedures are complete, one fluid module is installed in the rheometer, the outside shells of the fluid module are removed, and the inner shroud is slid back to expose the Boger fluid. The preshear motor is then rotated at a slow 100 rpm, and a stable fluid column is verified. Horizontal and vertical position controls are available as necessary to achieve a stable column. A test point is then selected (preshear and strain rates), and the experiment automatically executes. The fluid is presheared and stretched according to a preprogrammed exponential velocity profile. The stretch is stopped abruptly at 194 mm in length, and the fluid is allowed to relax. Each experiment lasts approximately five minutes, most of which is spent waiting for the fluid column to drain to the end plates and break in the middle. See Figure 2 – SHERE Rheometer

Afterwards, the translation slider is repositioned to the starting position, and the fluid column is recombined. If it is reusable due to criteria based on temperature, bubble contamination, and previous strain encountered, then another test can be performed with the same fluid module. Otherwise, it is removed, and the next one is installed for the next test.

### **Measurements**

During each experiment, a smooth, bubble-free cylindrical liquid bridge is generated between two flat endplates. The liquid bridge will initially be 5 mm long and 10 mm in diameter. A rotational shear rate will be imposed on the fluid by rotating one endplate from 0 to 500 rpm for a predetermined time while holding the other endplate stationary. As soon as the rotation has stopped, an elongational deformation will be imposed by axially translating one of the endplates in an



exponential manner to generate constant strain rates from 0.1 to 5.0 s<sup>-1</sup>. The tensile force and filament shape will be monitored during the elongation, and the elongation will stop at a length of approximately 194 mm. The position of the moving plate is recorded to verify the imposed velocity profile. By backlighting the test fluid with an electroluminescent panel and viewing the fluid column from above with a video camera, half of the fluid column's shape can be recorded for use in later analyses and simulations. Additionally, the fluid diameter is recorded via a laser micrometer at the column's midpoint. Fluid and air temperatures are digitally recorded from thermistors. Once the elongated bridge has been allowed to stabilize, the experiment will monitor subsequent evolution of the midpoint radius, filament shape, and tensile force in the column. Eventually, the filament necks down and breaks under the combined action of elastic and viscous capillary stresses. From measurements of force and radius during the stretch and relaxation of the fluid, we can compute the extensional viscosity as a function of strain rate and a function of the amount of preshearing.

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## **Shear History Extensional Rheology Experiment-II (SHERE-II)**

Following the successful completion of the SHERE Increment 18 operations, the newly redesigned 25 FM was returned to earth. These Fluid Modules will be emptied, cleaned and inspected. This will allow them to be refilled to permit reflight and re-use of the extensional rheometer that is currently in storage aboard ISS.



The natural next step is to use the SHERE hardware to probe the extensional rheology of a different non-Newtonian fluid. In principle this is straightforward, however stringent ISS safety requirements would require ground-based testing of seal compatibility, sample cleanup, offgassing if a completely new fluid was selected. However, we propose a much simpler approach: we seek to extend our knowledge by augmenting the existing polystyrene flight fluid with a dilute concentration (5%) of rigid inert filler.

Filled polymeric suspensions form the bulk of the engineering plastic market; by replacing a fraction of the (relatively expensive) synthetic polymer in a plastic product with a cheap mineral filler (e.g., titania (TiO<sub>2</sub>), silica (SiO<sub>2</sub>) or carbon black) it is possible to lower the cost, and enhance properties such as environmental sensitivity to UV degradation, water-swelling etc. However, as we describe in detail below, the systematic understanding of such fluids is in its infancy because of the



nonlinear interactions between the matrix viscoelasticity and the rigid filler. The no-slip boundary condition on the surface of the filler results in additional shear stresses, in all processing flows and enhances the total viscoelasticity. In the past four years, theoretical advances have led to a proper asymptotic constitutive theory for dilute suspensions of rigid spherical fillers dispersed in dilute polymer solutions [3,4]. This theory was validated in shear by comparison with ground-based shear rheology experiments performed in the P.I.'s lab [10] and makes explicit predictions (See Eq. (1) below) of the change in the extensional viscosity induced by the additional shearing kinematics induced by the rigid particles. The SHERE rheometer provides a perfect platform for testing this new theory because of the ability to independently control the shear rate and the extension rate in the rheometer.

We thus propose to formulate an enhanced fluid consisting of a dilute suspension (5 vol%) of rigid inert polymethylmethacrylate (PMMA or 'Plexiglas') microspheres (diameter  $d = 6$  or  $15\ \mu\text{m}$ ) dispersed in the same Polystyrene Boger fluid used for Increment 17 and 18 (consisting of a dilute solution (0.025 wt.%) of a narrow polydispersity high molecular weight polystyrene dissolved in oligomeric styrene oil). The specific formulation is discussed in the next section; however, it is important to note that this fluid can be rapidly formulated and readily characterized on the ground in the P.I.'s laboratory because (i) an excess of base fluid was originally formulated for the SHERE experiments, and (ii) the inert filler (spherical plexiglass beads) are also already available in the P.I.'s laboratory. The base fluid that has been stored in cold dark conditions will comprise 95% of the new test fluid and is ready to use immediately.

We propose to clean and fill the 25 FM that have been returned following Increment 18 with this new test fluid. Because the rheometer hardware is already in orbit, it is only necessary to launch new fluid samples, and the resulting upmass is minimal. The redesigned fluid modules used in Increment 18 performed flawlessly and there is no necessary additional redesign work for the hardware or the fluid modules. The new design enables a better deployment of the fluid column using a new chamfered lip design that prevents the fluid from wetting the force transducer endplate. As a result, a nice cylindrical fluid column pinned at the edges of the transducer plate is obtained, which guarantees well-defined initial conditions for subsequent numerical modeling. The only ground-based testing required before reflight will be a deployment test to ensure that the dilute concentration of inert microspheres do not interfere with the performance of the seals.

### **Theoretical Background and Objectives**

The goal of the SHERE II experiment will be to investigate the effects of a pre-shear history on the transient extensional viscosity in a uniaxial stretching flow for a model filled viscoelastic suspension (consisting of inert rigid non-Brownian spheres dispersed in a dilute polymer solution). The role of internal stresses produced by shearing between the rigid filler and the viscoelastic matrix can be explored systematically using the pre-shear capabilities of the SHERE platform. Access to extended microgravity also allows the subsequent relaxation behavior to be measured after cessation of the extensional deformation during the extended range of time scales that can be accessed because of the absence of gravitational sagging. As with the original SHERE experiment, SHERE II will be operated inside the Microgravity Science Glovebox (MSG) aboard the International Space Station (ISS).



**Rapid Turn-Around:** The major advantage of the proposed SHERE II experiment is that it will only require us to clean and refill the existing (redesigned) 25 fluid modules from Increment 18 once they have been returned to earth. No additional engineering and optimization will be required for these fluid modules that already proved their performance during Increment 18.

In addition, the base fluid (or ‘matrix fluid’ in the language of suspension mechanics) will remain unchanged, namely a dilute solution (0.025 wt.%) of a narrow polydispersity high molecular weight polystyrene dissolved in oligomeric styrene oil. This fluid has already been fully characterized in our lab and sufficient fluid remains to form the matrix fluid for the proposed SHERE II test matrix. The ISS experiments already performed without suspended microspheres correspond to a particle volume fraction  $f = 0$  and thus serve as a reference or baseline; these tests will not have to be repeated for SHERE II. This is another benefit of using the same base Boger fluid.

Also, the hard microspheres of diameter 15  $\mu\text{m}$  that will be used as filler particles do not have to be synthesized from scratch but are now readily commercially available under the brand name “spheromer” [3] from Microbeads SA (<http://www.micro-beads.com>). A range of sizes are available from 1 – 50  $\mu\text{m}$ . The smallest sizes  $d \leq 1 \mu\text{m}$  cannot be used because the existing theory is for non-Brownian spheres (i.e. Brownian motion is negligible); the larger sizes are increasing prone to sedimentation effects (under 1 g) which become worse as  $\sim d^2$ . These sedimentation effects can be mitigated using an available rotisserie system, but preliminary calculations show they can also be designed to be of minimal importance by reducing the particle size to  $d = 6$  or  $15 \mu\text{m}$ . A SEM image of the 15  $\mu\text{m}$  microbeads is shown in Fig. 2 (c). According to the supplier, the bead size distribution is very narrow, and the microspheres are almost monodisperse, which is suitable for subsequent numerical modeling.

Understanding the rheological properties of highly viscoelastic suspensions may be of paramount importance for lunar in-situ resource utilization and for the future construction of a permanent lunar base. Indeed, the lunar soil, referred to as regolith, is composed of loose dust and broken rocks as shown in Fig. 3 that could be used as a high-volume fraction filler when mixed with a polymeric binder or matrix for building foundation or roadbed materials. The viscoelastic dilute suspension used for SHERE II thus serves as a highly idealized model system amenable to theoretical analysis which can shed light on future development of lunar regolith-based construction materials.

Suspensions of particles in viscoelastic liquids are used in many earth processing operations: polymer melts with fillers, ceramic pastes, biomedical materials, food, cosmetics or detergents. The final properties of the suspensions are greatly determined by the shape, concentration and size of the filler. In particular, the fillers can range from nanoscopic to microscopic characteristic dimensions, which leads to very different types of flow behaviors, filler/matrix interactions and dynamics.

### **Relevance to NASA Research Mission**

The SHERE II experiment will allow us to measure, for the first time, the transient extensional viscosity in a uniaxial stretching flow for dilute polymer suspensions. In addition, SHERE II will enable us to study the subsequent fluid relaxation behavior after cessation of the extensional deformation, which is not possible in a 1-g environment because of sagging issues [13]. This



research has direct applications in many industrial processes involving filled viscoelastic systems, ceramic pastes, biomedical materials, food, cosmetics or detergents.

Secondly, as noted above, the possibility of using lunar regolith as high-volume fraction filler when mixed with a polymeric binder or matrix has application for future lunar exploration; e.g., the regolith may be blended with a viscoelastic binder (e.g., bitumen or other resin-like binder) for augmenting the volume and compressive strength of building foundation or roadbed materials. We have recently become aware of a regolith simulant (JSC-1A) that could also be used in future rheometric investigations. The additional rheological challenges associated with this material relate to the broad polydispersity of sizes and the high aspect ratio/angularity of the filler. In parallel with preparation of the SHERE II flight fluid (0.025wt%PS/5% PMMA spheres/94.975% oligomer), the PI will also begin ground-based experiments using a viscoelastic regolith suspension.

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