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### Squeeze Flow Rheometry for Rheological Characterization of Energetic Formulations

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## Squeeze Flow Rheometry for Rheological Characterization of Energetic Formulations

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*The rheological characterization and the determination of the parameters describing the shear viscosity and wall slip behavior of energetic materials is a challenge. Some of the conventional rheometers including various rotational rheometers are not capable of deforming typical energetic formulations with their gel binders and high degrees of particulate fill. Other available rheometers are not conducive to rheological characterization of energetic formulations in the vicinity of the manufacturing operation with the data to be used immediately for quality control. Squeeze flow provides significant advantages in safety of materials handling and exposure as well as providing easy data generation for routine quality control of energetic formulations being processed. Here the basic hardware is reviewed along with the methods for the analysis of raw data to determine the parameters of the shear viscosity and the wall slip of energetic formulations. It is suggested that appropriate analytical and numerical analyses can indeed provide the basic wherewithal necessary for the solution of the inverse problem of squeeze flows to characterize the shear viscosity and the wall slip parameters provided that the issues of uniqueness and stability are properly addressed.*

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## Introduction

Energetic formulations are complex fluids. Such complex fluids present challenges for the characterization of their rheological behavior [1–6]. This challenge stems from their viscoplasticity and concomitant wall slip behavior. Specialized techniques and multiple viscometers are employed to simultaneously characterize the parameters of the shear viscosity material function and wall slip versus the shear stress relationship [1–3]. Generally, the procedure for the characterization of the shear viscosity and wall slip involves systematic changes in the surface to volume ratio of the sample followed by the analysis of the flow curves [1–5]. When capillary flow is employed to generate flow rate versus pressure drop data, the procedure for wall slip corrected shear viscosity determination requires the use of multiple capillary dies involving systematically varied capillary lengths at constant diameter and different capillary diameters at constant length over the diameter ratio (for example, 12 capillaries were used in the study of the behavior of concentrated suspensions by Yilmazer and Kalyon [1]). In steady torsional flow between two parallel disks the procedure requires the systematic change of the gap of the rheometer or the imaging of the velocity distribution at the free surface of the fluid [1,2].

The squeeze flow involves the unbounded compression of the energetic formulation (the energetic material is free to flow in the radial direction upon compression in the axial direction, i.e., an important positive safety aspect) that is kept under isothermal conditions and partially or completely filling the space between two parallel and rigid circular disks. One or both of the circular disks can move in the axial direction at constant relative velocity, while the time-dependent force is being measured, or under constant normal force while the time-dependent relative velocity of the plate is measured [7]. In the analysis of the transport problem to derive the parameters of the shear viscosity

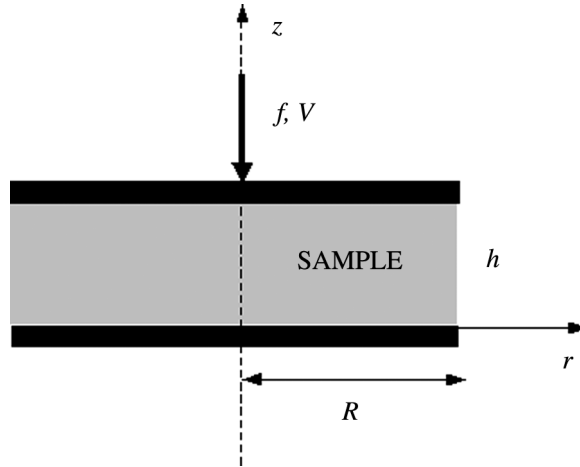
and the wall slip of the energetic formulation, approximate solutions to this unsteady state problem can be obtained by assuming that the speed of travel of the disk is sufficiently slow so that the time derivatives in the conservation equations can be neglected, i.e., the “quasi-steady state assumption” [7,8]. A number of studies have been made on the squeeze flow for generalized Newtonian fluids [9–16]. Nevertheless, although its literature is rich, the squeeze flow has not been fully exploited to facilitate the identification of parameters of constitutive equations and wall slip behavior of energetic formulations. In this paper, first typical hardware suitable for the rheological characterization of energetic fluids is presented followed by the analysis of the transport equations which represent the dynamics of the squeeze flow for the determination of the parameters of the shear viscosity and wall slip behavior of energetic formulation. Analytical as well as Finite Element Method (FEM) based numerical methods are used in our methodologies in conjunction with the solution of the inverse problem for the determination of the rheological parameters.

### Squeeze Flow Hardware for Rheological Characterization of Energetic Materials

A schematic representation of the squeeze flow is shown in Fig. 1. The energetic fluid sample is placed in between the two circular disks with a radius of  $R$ . Although different experimental configurations are possible, including the use of a constant normal force, our current configuration involves the top disk moving down at a constant speed of  $V$ , and the bottom plate being stationary. The time-dependent gap between the plates is designated as  $h$ , and the total force acting on the top plate is  $f$ .

The squeeze flow rheometer for energetics applications needs to have the following features:

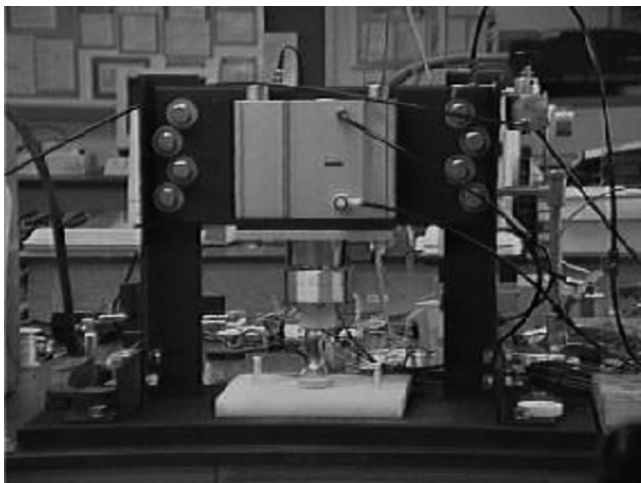
- For safety the squeeze flow rheometer should be explosion proof.
- The unit needs to be run remotely, with no operator present during the characterization.



**Figure 1.** Schematic representation of the squeeze flow.

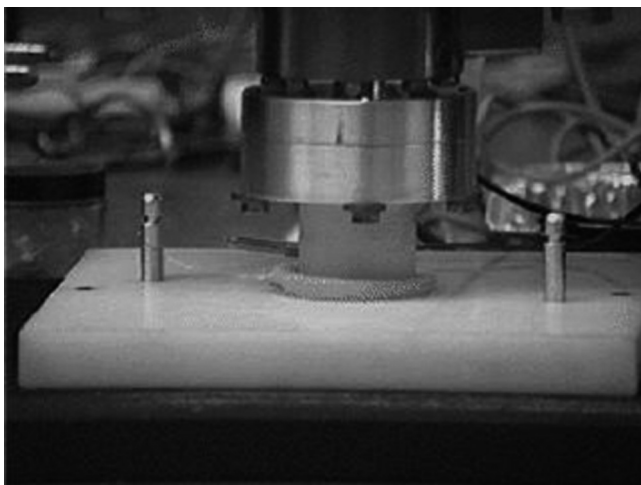
- The squeeze flow unit needs to be designed to be mobile and configured as a bench top unit.
- The rheometer needs to be sufficiently easy to use so that the plant operators can collect the rheological characterization data to be used for process and product quality control.
- The data analysis should be part of the data acquisition unit of the rheometer so that the operator/engineer need not be involved with data analysis.
- The parameters of constitutive equations and wall slip need to be generated immediately upon the testing of the sample to allow the use of the data in product quality control.
- The unit needs to be easy to clean and the surfaces that come into contact with the energetic material can be easily replaced.

The squeeze flow rheometer, which satisfies all of these objectives, is shown in Figs. 2–4 (available from Material Processing & Research, Inc. of Hackensack, NJ [17]). The mechanical displacement to achieve the squeeze motion is generated by compressed air pressure (90 psi is sufficient).

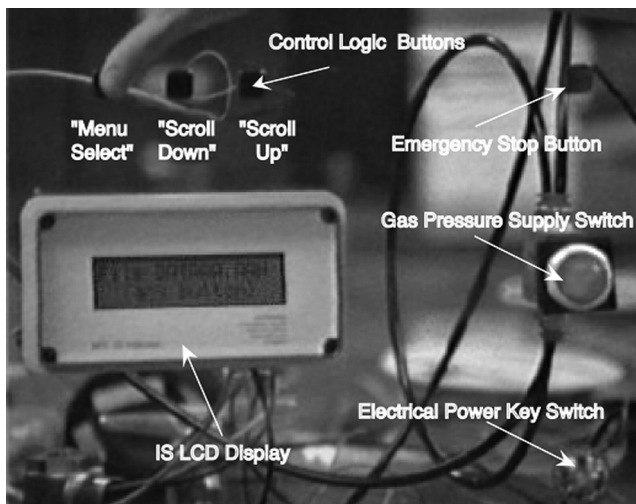


**Figure 2.** Squeeze flow rheometer hardware. Overall view.

The squeeze flow rheometer is incorporated with an embedded computer, which allows real-time data to be collected and concomitantly analyzed. There are two sensors on the unit, a



**Figure 3.** Squeeze flow rheometer hardware. Squeeze of the sample.



**Figure 4.** An explosion-proof version used in the gun propellant industry.

pressure transducer and a linear variable displacement transducer. The rheometer is designed to press specimens, which are collected from mixers and processors that are located in the immediate vicinity of the rheometer so that solvent loss and temperature control are not issues. Especially with viscoplastic and structured fluids, the resting of the specimens during relatively long periods associated with thermal stabilization can alter their rheological properties by building up a yield stress.

The source code for the data analysis is burnt into the chip so that the data analysis is immediate. The data can be transferred or stored on a flash card, which can then be immediately downloaded to another PC or using field point technology, the rheometer can be run wireless, or through the internet. A web camera is integrated so that the squeeze test not only can be run remotely but also can be monitored remotely. One explosion-proof version, which is being used in the gun propellant industry, is driven only through three buttons and the parameters are displayed on an intrinsically safe LCD display (Fig. 4). In the following section the analysis of the data emanating from

the squeeze flow rheometer is discussed in conjunction with the data emanating from the unit.

### Squeeze Flow and Method to Determine Rheological Parameters of Energetic Materials

Energetic suspensions and gels generally exhibit relatively high shear viscosity values to give rise to creeping flow conditions upon being subjected to squeeze flow. Under the resulting typical relatively low Reynolds number conditions the inertial terms in the equations of motion can be neglected. The governing equations for axisymmetric flow of incompressible fluids and under quasi-steady state and isothermal conditions become:

$$\frac{1}{r} \frac{\partial r v_r}{\partial r} + \frac{\partial v_z}{\partial z} = 0, \quad (1)$$

$$\frac{1}{r} \frac{\partial r \tau_{rr}}{\partial r} + \frac{\partial \tau_{rz}}{\partial z} - \frac{\tau_{\theta\theta}}{r} + \frac{\partial p}{\partial r} = 0, \quad (2)$$

$$\frac{1}{r} \frac{\partial r \tau_{rz}}{\partial r} + \frac{\partial \tau_{zz}}{\partial z} + \frac{\partial p}{\partial z} = 0. \quad (3)$$

Here  $v_r$  and  $v_z$  are the velocity components in the radial and axial directions, i.e.,  $r$  and  $z$ , respectively,  $p$  is the pressure, and  $\tau_{rr}$ ,  $\tau_{zz}$ ,  $\tau_{\theta\theta}$  and  $\tau_{rz}$  are components of the deviatoric stress tensor. If the rate of movement of the plate,  $V$ , is sufficiently slow to allow the viscoelastic effects to be considered to be negligible, generalized Newtonian fluid constitutive behavior is applicable, i.e.,  $\underline{\underline{\tau}} = -\eta(II)\underline{\underline{\Delta}}$ , where  $\underline{\underline{\tau}}$  and  $\underline{\underline{\Delta}}$  are the stress and rate of deformation tensors.  $\eta(II)$  is the shear viscosity material function varying as a function of the second scalar invariant of the magnitude of the rate of deformation tensor,  $\dot{\gamma}$ ; i.e.,  $\dot{\gamma} = \sqrt{II}/2$ . The squeeze flow is subject to the following non-linear wall slip condition, i.e., slip velocity,  $U_s$ , versus the shear stress,  $\tau_{rz}$ :

$$U_s = \beta |\tau_{rz}|^{sb}, \quad (4)$$

where the Navier slip coefficient,  $\beta$ , and the slip exponent,  $sb$ , are parameters of the wall slip behavior, which are related to



the shear viscosity material function of the binder of the suspension and the apparent slip layer thickness for concentrated suspensions [5]. The slip coefficients for the top and bottom disks, i.e.,  $\beta_t$  and  $\beta_b$  respectively, can be different due to differences in roughness or the materials of construction of the two plates. For 1-D flow the shear stress for the Herschel-Bulkley type viscoplastic constitutive equation becomes  $\tau_{rz} = \pm\tau_y - m|dv_r/dz|^{n-1}(dv_r/dz)$  for  $|\tau_{rz}| \geq \tau_y$  (– sign is used for negative shear stress,  $\tau_{rz}$ ) and the shear rate  $(dv_r/dz) = 0$  for  $|\tau_{rz}| < \tau_y$  (Herschel and Bulkley [18]). For complex flows the second invariant of the rate of deformation tensor needs to be used instead of the velocity gradient in the Herschel and Bulkley equation. Here,  $m$  is the consistency index,  $n$  is the power-law index, and  $\tau_y$  is the yield stress. Thus, for energetic suspensions the shear viscosity of which is represented by the Herschel-Bulkley fluid subject to wall slip there are five parameters  $\{m, n, \tau_y, \beta, sb\}$  that need to be determined for representing the shear viscosity along with the wall slip condition. For energetic suspensions,  $sb$  can be estimated from the shear viscosity material function of the binder and the concentration and properties of the rigid particles [5]. The solution of the squeeze flow problem for the Herschel-Bulkley fluid also provides the solutions for the Newtonian fluid with  $n = 1$  and  $\tau = 0$ ; the Bingham fluid with  $n = 1$ ; the power-law behavior with  $\tau_y = 0$ ; all subject to either no slip or slip at the wall.

The force acting on the top plate is:

$$\begin{aligned} f &= \int_0^R 2\pi(p + \tau_{zz})|_{z=h} r dr \\ &= \int_0^R \pi \left( \left( -\frac{\partial p}{\partial z} \right) r^2 + 2\tau_{zz} r \right) \Big|_{z=h} dr. \end{aligned} \quad (5)$$

The problem (1)–(5) can be solved either numerically or analytically to determine the normal force,  $f$ , acting on the moving plate (i.e., [8–16]).

If one starts with the Herschel-Bulkley type constitutive equation under a quasi-steady state and lubrication flow conditions ( $h \ll R$ ), and upon assuming that all three normal

stress terms and  $(-\partial p/\partial z)$  are negligible, the solution of the continuity and momentum equations, subject to non-linear wall slip, generates the velocity and shear stress distributions [the continuity equation remains the same, i.e., Equation (1) and the momentum conservation equations reduce to Equation (2) without the normal stress terms]. What is the typical error involved in the use of the lubrication assumption? Lawal and Kalyon [13] have carried out an FEM-based analysis of the squeeze flow for viscoplastic fluids under the wall slip condition and compared the FEM results versus analytical predictions of the squeeze flow of Hershel-Bulkley fluid subject to the Navier wall slip condition under the lubrication assumption. A reasonable agreement between FEM [13] and the analytical solution [11] of the squeeze flow subject to the lubrication assumption was found under certain conditions. For example, for the modified Bingham number  $(\tau_y/m(V/R)^n)$  range of 0 to 100, the percentage deviation of the total normal force,  $f$ , calculated on the basis of the lubrication assumption differed by less than 10% of the total normal force determined with the FEM analysis for the no-slip condition. However, the agreement between the total normal force values determined with FEM versus the analytical solution using the lubrication assumption was shown to deteriorate with the inclusion of the wall slip versus the no-slip condition, with increasing wall slip coefficient and upon increasing the yield stress value of the fluid [13]. It was suggested that the FEM analysis or experimental data are necessary to determine the conditions under which the lubrication assumption can be assumed to be valid for the squeeze flow.

In the following section inverse problem approaches are demonstrated first using a power-law fluid model under the no-slip condition in conjunction with an analytical solution of the squeeze flow problem. Also, a FEM based numerical solution of the squeeze flow is used in conjunction with inverse problem solution methodologies to enable the identification of four rheological parameters  $\{m, n, \tau_y, \beta\}$  in conjunction with the Herschel-Bulkley viscoplastic model and non-linear wall slip behavior.

## Squeeze Flow and Inverse Problem Solutions for Determination of Rheological Parameters

### Power-law Fluid without Slip

If the shear viscosity of the energetic material is assumed to obey the behavior of a power-law fluid without the wall slip, i.e.,  $\tau_y, \beta = 0$ , the pressure gradient can be given as:

$$-\frac{dp}{dr} = \frac{2m}{h^{1+2n}} \left( \frac{2n+1}{n} \right)^n V^n r^n, \quad (6)$$

Upon assuming that the normal stress is negligible, Equations (5) and (6) give rise to the force,  $f$ , as a function of squeeze flow geometry (radius,  $R$  and gap,  $h$ ), operating condition (velocity of ram travel,  $V$ ) and the parameters of the power-law fluid:

$$f = \frac{2\pi m R^{n+3}}{(n+3)h^{2n+1}} \left( \frac{2n+1}{n} \right)^n V^n, \quad (7)$$

which is the Scott equation [19].

Starting with the Scott equation (7) and considering that  $R, V = \text{const}$ , the objective function defined in Appendix A becomes:

$$J(F, F^e) = \frac{1}{M} \sum_{i=1}^M \left( 1 - \frac{m h_i^{-(2n+1)} g(n)}{f_i^e} \right)^2, \quad (8)$$

where

$$g(n) = \frac{2\pi R^{n+3}}{(n+3)} \left( \frac{2n+1}{n} \right)^n V^n. \quad (9)$$

At minimum points of the objective function,

$$0 = \frac{\partial J}{\partial m} = \frac{2}{M} \sum_{i=1}^M \left( 1 - \frac{m h_i^{-(2n+1)} g(n)}{f_i^e} \right) \left( -\frac{h_i^{-(2n+1)} g(n)}{f_i^e} \right), \quad (10)$$

$$0 = \frac{\partial J}{\partial n} = \frac{2}{M} \sum_{i=1}^M \left( 1 - \frac{mh_i^{-(2n+1)} g(n)}{f_i^e} \right) \times \left( \frac{-mh_i^{-(2n+1)} (-2 \ln(h_i)) g(n) + mh_i^{-(2n+1)} dg(n)/dn}{f_i^e} \right), \tag{11}$$

which yield:

$$m = \frac{\sum_{i=1}^M h_i^{-(2n+1)} / f_i^e}{\sum_{i=1}^M h_i^{-(4n+2)} g(n) / f_i^{e^2}}. \tag{12}$$

If  $n$  exists uniquely, then  $m$  will be uniquely determined and the minimizer  $\{m, n\}$  is unique. From Equations (10) and (11), and upon rearrangement the following is obtained:

$$\sum_{\substack{i=1, j=2 \\ i < j}}^{M-1, M} \frac{h_i^{-(4n+2)} h_j^{-(2n+1)}}{f_i^{e^2} f_j^e} (\ln(h_i) - \ln(h_j)) \left( 1 - \frac{f_i^e}{f_j^e} \left( \frac{h_i}{h_j} \right)^{2n+1} \right) = 0, \tag{13}$$

which provides the value of  $n$ . The question of whether  $n$  has a solution depends on whether Equation (13) has a solution. Let

$$G = \sum_{\substack{i=1, j=2 \\ i < j}}^{M-1, M} \frac{h_i^{-(4n+2)} h_j^{-(2n+1)}}{f_i^{e^2} f_j^e} (\ln(h_i) - \ln(h_j)) \left( 1 - \frac{f_i^e}{f_j^e} \left( \frac{h_i}{h_j} \right)^{2n+1} \right), \tag{14}$$

then Equation (13) will have a solution if  $G$  has a zero point. Notice that  $i < j$ ,  $0 < f_i^e / f_j^e < 1$ , and  $h_i / h_j > 1$ , it can be shown that:

$$G < 0, \quad q_{ij} \equiv 1 - \frac{f_i^e}{f_j^e} \left( \frac{h_i}{h_j} \right)^{2n+1} < 0, \quad \text{when} \\ n > n_{upper} \equiv \frac{1}{2} \left( \frac{\ln(f_M^e / f_1^e)}{\ln(h_i / h_j)_{\min}} - 1 \right), \tag{15}$$

$$G > 0, \quad q_{ij} \equiv 1 - \frac{f_i^e}{f_j^e} \left( \frac{h_j}{h_i} \right)^{2n+1} > 0, \quad \text{when}$$

$$n < n_{lower} \equiv \frac{1}{2} \left( \frac{\ln(f_j^e/f_i^e)_{\min}}{\ln(h_1/h_M)} - 1 \right). \quad (16)$$

Therefore,  $G$  must have at least one zero point  $n \in (n_{lower}, n_{upper})$  for Equation (13) to have a solution. A simple but helpful way to check the existence and uniqueness of the solution for Equation (13) is to plot out  $G$  on the  $G$ - $n$  plane and examine how many times curve  $G$  crosses the  $n$ -axis. The number of times it crosses the  $n$ -axis is the number of the roots of Equation (13). When  $G$  has multiple solutions, one needs to eliminate unrealistic solutions. For instance, those with  $n > 1$  and  $n < 1$  must be excluded for shear-thinning and thickening materials, respectively. For the case of  $M=2$ , i.e., when only two experimental data are used,  $n$  exists uniquely and the minimizer becomes:

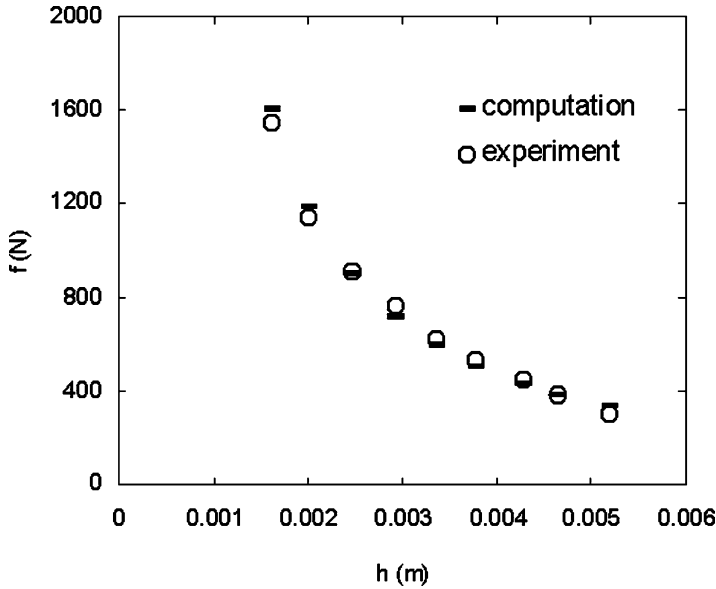
$$m = \frac{h_1^{-(2n+1)} f_1^e f_2^{e^2} + h_2^{-(2n+1)} f_1^{e^2} f_2^e}{(h_1^{-(4n+2)} f_2^{e^2} + h_2^{-(4n+2)} f_1^{e^2}) g(n)}, \quad (17)$$

$$n = n_{lower} = n_{upper} = \frac{1}{2} \left( \frac{\ln(f_2^e/f_1^e)}{\ln(h_1/h_2)} - 1 \right). \quad (18)$$

As an example let us consider one set of experimental squeeze flow data (force versus gap) collected by Zhang et al. [9], reproduced in Fig. 5. Solving numerically the Equations (12) and (13) give  $m=48,700 \text{ Pa}\cdot\text{s}^{0.2}$ ,  $n=0.2$ . The forces determined by the estimated parameters are also plotted in Fig. 5. Additional numerical test runs show that, in the range of  $10,000 < m < 500,000$  and  $0.05 < n < 2$ , the estimate is unique. Zhang et al. [9] report the results of their rheological characterization as:  $m=44,900 \text{ Pa}\cdot\text{s}^{0.25}$  and  $n=0.25$ , which are close to the estimates of these parameters given above.

### ***Herschel-Bulkley Fluid with the Wall Slip***

As indicated earlier the inverse problem solution of the isothermal squeeze flow for the Herschel-Bulkley fluid subject to the



**Figure 5.** Force versus gap for a power-law fluid obtained from experiment [9] and computation.  $R = 0.0254$  m and  $V = 2.18 \times 10^{-4}$  m/s.

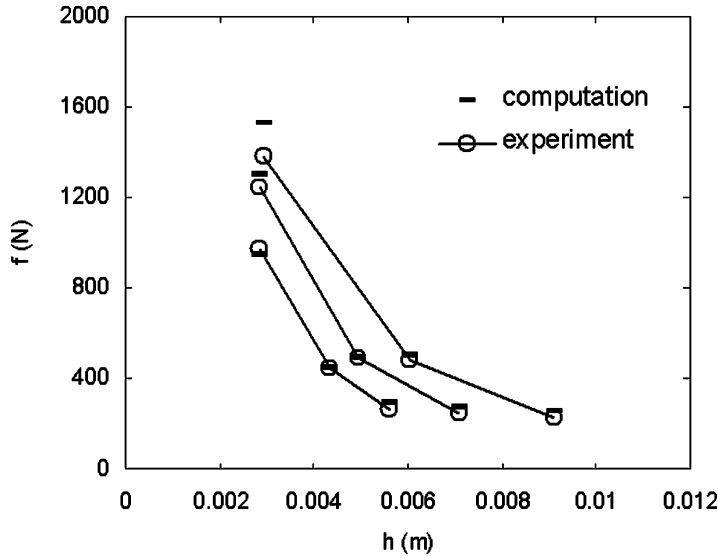
wall slip outside of the lubrication flow region requires a numerical solution. Here, the FEM approach given by Lawal and Kalyon [13] is followed to solve the governing equations, and a combination of the steepest descent and the conjugate gradient methods [20] is employed to carry out the minimization required for the solution of the inverse problem. Starting from an initial guess, the minimization begins with the steepest descent method and, after a certain number of steps in searching, switches to the conjugate gradient method. The FEM code was validated using experimental data, and the effectiveness of the minimization program has been tested (see Tang and Kalyon [21]).

The parameter domain was subdivided into multiple subdomains and initial points pertaining to each subdomain were taken to arrive at the global minimum, which was considered to represent the solution of the problem for all four parameters.

This approach of parameter domain division is based on the observations from extensive numerical experimentation, which suggested that when the number of parameters sought is equal to or greater than three, reasonable estimates could only be made if the initial guesses for parameters sought are relatively close to the true values. However, how do we know where the true solution lies, prior to the characterization of the fluid, so that the initial guesses are made to approach the true values of parameters? The method involves the division of the parameter space spanned by  $\{m, n, \tau_y, \beta\}$  into subdomains and starting the minimization with multiple initial conditions belonging to each subdomain. In conjunction with this method the objective function arising from each subdomain can be compared with the others and a global minimum can be determined. Provided that the division is sufficiently fine and the objective function is continuous and smooth, it is anticipated that the procedure will find the global minimum. In the following example to this approach four parameters of a suspension of a poly (dimethyl siloxane) (PDMS) fluid (GE Silicones SE 30), a processing simulant for an energetics suspension, were sought starting from the experimental squeeze flow versus time data.

In the experiments a suspension consisting of PDMS incorporated with 40% by volume of hollow glass spheres (Potters Industry, mean size of 12 micron) was subjected to squeeze flow. The experimental normal force versus time data for this suspension are shown in Fig. 6. The radius of the disks,  $R$ , was 0.0286 m and the plate velocity,  $V$ , was varied as  $2.1 \times 10^{-4}$ ,  $4.2 \times 10^{-4}$  and  $8.4 \times 10^{-4}$  m/s. The value of the slip exponent,  $sb$ , could be apriori specified as 2.165 on the basis of the characterized shear viscosity of the PDMS binder [5].

The entire parameter domain was subdivided to one hundred parameter subdomains, each of which contained a different set of initial parameters. The values of the objective function,  $J$  defined by Equation (A1), were determined for all one hundred sets of initial conditions from which the global minimum could be determined. This procedure gave rise to the four parameters of the suspension represented as a



**Figure 6.** Experimental and computed forces for PDMS incorporated with 40% (vol.) of glass spheres.  $R = 0.0286$  m and  $V = 2.1 \times 10^{-4}$ ,  $4.2 \times 10^{-4}$  and  $8.4 \times 10^{-4}$  m/s.

Herschel-Bulkley fluid subject to a non-linear wall slip as:  $m = 42,500 \text{ Pa} \cdot \text{s}^{0.50}$ ,  $n = 0.50$ ,  $\tau_y = 177 \text{ Pa}$ ,  $\beta = 1.55 \times 10^{-14} \text{ m} \cdot \text{s}^{-1} \cdot \text{Pa}^{-2.165}$ . The forces given by the estimate are also plotted in Fig. 6. At  $\tau_{rz} = 102,000 \text{ Pa}$  these parameters define the slip velocity,  $U_s$ , as  $0.0012 \text{ m/s}$ . The utilization of capillary flows, and corrections of the data following the procedures given in [4] provided  $m = 36,000 \text{ Pa} \cdot \text{s}^{0.49}$ ,  $n = 0.49$ , and  $U_s = 0.0017 \text{ m/s}$  at  $\tau_{rz} = 102,000 \text{ Pa}$ , suggesting acceptable agreement with the capillary flow results. Therefore, these typical results suggest that adequate estimates can indeed be obtained from the inverse problem solution of the squeeze flow using FEM if the solution domain is broken into multiple parameter subdomains and systematically varied sets of initial conditions to coincide with all subdomains are used to allow the global minimum and hence the solution of the inverse problem for four parameters to be obtained at reasonable accuracy.



## Concluding Remarks

The squeeze flow rheometer is introduced as a convenient method for the rheological characterization of energetic formulations. The hardware needs to be used in conjunction with software and source codes developed for the solution of inverse problem for the determination of the parameters of viscoplastic constitutive equations and the wall slip of the energetic formulation. If the parameter space is divided into multiple subdomains so that the initial conditions would coincide with each subdomain, the reliability of the search method increases and reasonable estimates for obtaining up to four parameters can be obtained. The advantage of the squeeze flow in conjunction with inverse problem solution for the determination of the parameters becomes apparent upon considering that the squeeze flow test takes only a few minutes versus the weeks of work generally involved in collecting conventional rheological characterization data, for example, capillary flow using multiple capillaries and multiple apparent shear rates run for each capillary for the characterization of energetic formulations.

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