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#### **Abstract**

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#### Disciplines

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#### Comments

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### Oscillatory enhancement of the squeezing flow of yield stress fluids: a novel experimental result

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The extrusion of a yield stress fluid from the space between two parallel plates is investigated experimentally. Oscillating the magnitude of the squeezing force about a mean value ( $F = f[1 + \alpha \cos(\omega t)]$ ) was observed to significantly enhance the flow rate of yield stress fluids, while having no effect on the flow rate of Newtonian fluids. This is a novel result. The enhancement depends on the magnitude of the force, the oscillatory frequency and amplitude, the fluid being squeezed, and the thickness of the fluid layer. Non-dimensional results for the various flow quantities have been presented by using the flow predicted for the constant-force squeezing of a Herschel–Bulkley yield stress fluid as the reference. In the limit of constant-force squeezing, the present experimental results compare very well with those of our earlier theoretical model for this situation (Zwick, Ayyaswamy & Cohen 1996). The results presented in this paper have significance, among many applications, for injection moulding, in the adhesive bonding of microelectronic chips, and in surgical procedures employed in health care.

#### 1. Introduction

Squeeze film flow is produced when two rigid parallel plates are brought together, extruding a fluid contained between them. Such flows have important applications in injection moulding and adhesive assembly. Recently, such flows have also found application in surgical procedures employed in hospitals. Often, the adhesive or the plastic being squeezed is a non-Newtonian particulate suspension and can be characterized as a yield stress fluid. Squeezing of such a fluid proceeds very slowly as the film approaches a limiting thickness where the squeezing force exactly balances the yield stress of the fluid. It is often desirable to speed up the flow without increasing the squeezing force. This particular consideration is important in the adhesive bonding of thin and fragile microelectronic chips (5 mm  $\times$  5 mm  $\times$  0.5 mm) to substrates. It is desirable to quickly squeeze the epoxy from underneath the chip using only a very small force. We have found that sinusoidally varying the magnitude of the squeezing force about a mean value has the following novel effects on yield stress fluids: (i) increasing the flow rate, and (ii) lowering the limiting (minimum) film thickness compared to films subject to a constant squeezing force. However, oscillations do not affect the squeezing flow of Newtonian fluids.

A careful review of the literature reveals that oscillatory squeezing flow with a yield stress fluid, such as the one described in the present paper, has not been analysed or experimentally studied. There are a number of published studies of squeezing

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flows with yield stress fluids, but these involve constant force conditions. For a review of constant-force studies, see Adams *et al.* (1994), and Zwick, Ayyaswamy & Cohen (1996). There are also a number of studies related to: (i) non-yield stress squeeze films under oscillatory forcing (see for example Hashimoto 1994), and (ii) flow enhancement due to oscillatory (non-squeezing) driving forces with shear thinning fluids (see, for example Mena, Manero & Binding 1979) and yield stress fluids (see, for example Kajiuchi & Saito 1984). It may be noted in this regard that many of the theoretical models described in the literature predict results which do not compare favourably with experimental observations for corresponding conditions. To reiterate, there are no studies available at present whose predictions may be compared with the experimental observations described herein. (See Zwick 1996 for a detailed discussion of this literature.)

In the following, we describe our experimental observations of the flow enhancement in yield stress fluids caused by oscillatory squeezing.

#### 2. Experimental study

#### 2.1. Apparatus

A modified plastometer (also known as a squeeze film rheometer) was constructed for the purpose of measuring squeezing flow rates while simultaneously applying an oscillatory squeezing force. The device consists of a cupped bottom plate for holding the specimen, a weighted upper plate, a Lucas Schaevitz model 050 MP linear displacement transducer (LVDT) for measurement of the motion of the top plate, and a 3.5 in. diameter dual cone loudspeaker for production of the oscillations. This set-up is shown in figure 1. A rigid rod connects the upper plate to the LVDT, so that the plate position may be measured. The output from the LVDT is processed by a Lucas Schaevitz CAS series amplifier and then stored and read by a Hewlett Packard model 54600B 100 MHz Digital Oscilloscope. The output from the amplifier is in volts. The output voltage is correlated to the actual film thickness measurements on the plastometer by measuring a number of known heights and plotting them against voltage.

The lower plate is attached by a single central bolt to a 0.3 mm thick stainless steel mounting plate (see figure 1). This plate is mounted on top of the speaker by two bolts at either side of the speaker. The mounting plate is free to flex in the centre, thus lifting the lower plate. The speaker is connected to a BK Precision 2 MHz function generator through a Panasonic amplifier. When the speaker is activated, the mounting plate, lower plate, fluid, upper plate, and the connection to the LVDT are all forced to oscillate in a direction normal to the faces of the upper and lower squeezing plates by the acoustic pressure created between the loudspeaker and the mounting plate. The oscillation is measured by the LVDT. The oscillation amplitude is controlled through the amplifier, and the frequency is controlled through the function generator. Oscillations with amplitudes up to 20  $\mu$ m with frequencies up to  $\sim$  250 Hz can be produced in this manner. The function generator can produce sinusoidal, triangular, or square waveforms. We used the sinusoidal waveform for all measurements because it produced a smooth sinusoidal displacement of the plates as measured by the LVDT. The oscillatory squeezing force is a result of accelerating the weight of the upper plate, and it can be determined by twice differentiating the measured displacements of the upper plate. The displacements are described by  $x = a \cos(\omega t)$  and the total force experienced by the film is then  $F = f\{1 + \alpha \cos(\omega t)\}\$ , where f = Mg and  $\alpha = -a\omega^2/g$ .

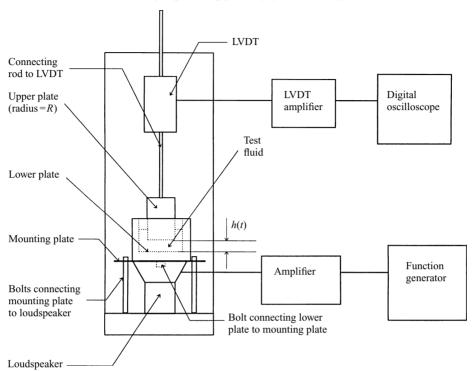


FIGURE 1. Schematic of modified plastometer for the measurement of oscillatory squeeze film flow.

Here M is the mass of the upper plate, a is the displacement amplitude,  $\omega$  is the oscillatory frequency, and g is the gravitational acceleration.

The entire device stands about 30 cm in height. The upper plate is 15 mm in radius, and two different upper plate weights were used, 1.45 N and 0.68 N, including the mass of the connections to the LVDT. The lower plate is flat with cupped sides. The cup inner diameter is 72 mm, and large enough that it should not interfere with the exiting flow from the squeeze film. The upper plate is guided by a narrow plate with a running fit hole of just over 30 mm in diameter. The guide is lubricated with a small amount of light oil. Assuming a Couette flow between the upper plate and the guide, and with a typical velocity of 1 mm s<sup>-1</sup> for the upper plate, we can estimate the drag on the plate to be about 0.0005 N. This magnitude is less than 0.1% of the squeezing force, and is taken to be negligible.

The test fluids were: SAE 140 gear oil, glycerin (99.7% pure), French's mustard, 1.5% by weight solution of Cab-O-Sil fumed silica and Fischer light paraffin oil, and Ablebond 84 1LMI-S die bonding epoxy. The Newtonian SAE 140 gear oil and the glycerin were used to test the device because their constitutive behaviours are known. The mustard and the fumed silica/paraffin oil solutions are suspensions with small and relatively uniform flocculating particles. The die bonding epoxy used is a mixture of epoxy resin, about 20% by volume 20  $\mu$ m silver particles for improved heat conduction, and  $\approx$  3% by weight fumed silica to keep the silver in suspension. The fumed silica forms a space-filling network, giving the epoxy a yield stress.

#### 2.2. Procedure

The cup/lower plate assembly is filled up to a level of approximately 5 mm with the test fluid. The upper plate is lowered to about 1 mm below the fluid surface and then released by hand. The plate travels under its own weight, and squeezes the fluid out as it travels. The measurements commence at a film thickness of approximately 0.5 mm. The fluid samples were stored at room temperature, which was noted at the beginning and end of each set of experiments. Viscous heating of the samples is not expected with the low velocities and forces used in the experiment.

In this study, each sample was preconditioned by running the test approximately ten times before storing the data for that sample. This was done to ensure that the effects of the strain history of the fluid developed during loading into the viscometer did not affect the outcome of the measurements. For a discussion of strain history dependence in fumed silica see Raghavan & Khan (1996). Tests were generally run successively with an approximately 60 s interval between each test. This rest time seemed to have little influence on the results. The film thickness versus time measurements were found to be very repeatable with very little sensitivity to the initial film thickness from which the upper plate was released  $(h_i)$ . This indicates that inertia does not greatly influence the flow under these conditions.

For each test fluid, we first measured the film thickness versus time with zero oscillatory amplitude, and stored the trace. We then measured a series of film thickness versus time traces with a constant displacement amplitude of 5  $\mu$ m and frequencies ranging from 50 to 250 Hz. The oscillatory displacement amplitude and frequency were fixed for each individual measurement. Changes in frequency and amplitude were carried out between measurements. We then measured a series of film thickness versus time traces for a frequency of 100 Hz and displacement amplitudes ranging from 5 to 20  $\mu$ m. Finally, we measured the zero-oscillation case again, and compared it to the stored trace from the beginning of the measurement series to ensure that no changes occurred during the course of the measurements. The beginning and end traces were identical, and they serve as the baseline for all comparisons.

#### 3. Physical quantities

It is convenient to present our experimental observations for the flow enhancement  $\Delta Q$  in terms of (i) input quantities: frequency of oscillation  $\omega$ , initial film thickness  $h_i$ , plate radius R, oscillatory amplitude a, and squeezing force f, and (ii) measured quantities: instantaneous film thickness h, limiting film thickness under constant-force squeezing  $h_0$ , and plate approach velocity U. An expression for  $\Delta Q$  is given subsequently. We also introduce dimensionless quantities: velocity  $U^* = (U/U_r)$ , film thickness  $h^* = (h/h_0)$ , force amplitude  $\alpha = (-a\omega^2/g)$ , and aspect ratio  $\epsilon = (h_i/R)$ . The reference velocity  $U_r$  corresponds to constant-force squeezing and is explained subsequently. In our experiments, we measure h and  $h_0$  for each fluid of interest. The limiting film thickness,  $h_0$ , is found by letting the film squeeze down under constant-force conditions until the film thickness no longer changes. This may be a matter of seconds with a highly loaded film, or several hours or days with a weakly loaded film. Generally, we would measure the film thickness every minute after a test run was made. If the film thickness changed by less than 2 µm between measurements, we considered the film thickness to be  $h_0$ . The instantaneous plate velocity U is determined by taking central differences on the film thickness measurement versus time data. The film thickness versus time data are sampled at varying

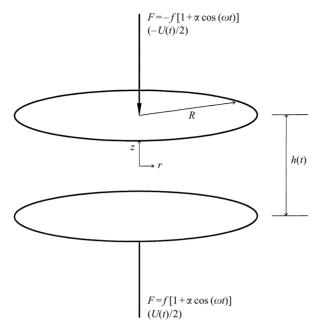


FIGURE 2. Schematic of oscillatory squeezing flow.

intervals (short intervals for large film thicknesses, and longer intervals for small film thicknesses).

We now provide a rationale for the choice of the reference velocity,  $U_r$ . For simplicity, consider the flow to be axisymmetric as in figure 2 with  $u_\theta=0$ , and  $\partial/\partial\theta=0$ . Let location  $r^*=r/R$ ,  $z^*=z/h_i$ , flow velocity components  $u_z^*=u_z/U$ ,  $u_r^*=u_rh_i/(UR)$ , pressure  $p^*=ph_i^3/(\eta_0UR^2)$ , deviatoric stress tensor  $\tau^*=\tau h_i^2/(\eta_0UR)$ , and non-dimensional time  $t^*=t\omega$ . Here,  $\eta_0$  is the nominal viscosity defined by  $\eta_0=m(UR/h_i^2)^{n-1}$ , in which m is the viscosity coefficient with units of Pa  $s^n$ , and n is a power-law exponent. We have chosen m and n appropriate to the Herschel–Bulkley fluid. Let the squeezing Reynolds number be  $Re=\rho Uh_i/\eta_0$ , the oscillatory Reynolds number be  $Re_\omega=\rho\omega h_i^2/\eta_0$ , and the body force parameter be  $B=h_i^3\rho g/(\eta_0RU)$ . The equations of motion governing the flow may be written as

$$Re_{\omega}\frac{\partial u_{r}^{*}}{\partial t^{*}} + Re\left(u_{r}^{*}\frac{\partial u_{r}^{*}}{\partial r^{*}} + u_{z}^{*}\frac{\partial u_{r}^{*}}{\partial z^{*}}\right) = \frac{\partial p^{*}}{\partial r^{*}} + \frac{\partial \tau_{rz}^{*}}{\partial z^{*}} + \epsilon\left[\frac{1}{r^{*}}\frac{\partial (r^{*}\tau_{rr}^{*})}{\partial r^{*}} - \frac{\tau_{\theta\theta}^{*}}{r^{*}}\right], \quad (3.1)$$

and

$$\epsilon^{2}Re_{\omega}\frac{\partial u_{z}^{*}}{\partial t^{*}} + \epsilon^{2}Re\left(u_{r}^{*}\frac{\partial u_{z}^{*}}{\partial r^{*}} + u_{z}^{*}\frac{\partial u_{z}^{*}}{\partial z^{*}}\right) = \frac{\partial p^{*}}{\partial z^{*}} + \epsilon\frac{\partial \tau_{zz}^{*}}{\partial z^{*}} + \frac{\epsilon^{2}}{r^{*}}\frac{\partial (r^{*}\tau_{rz}^{*})}{\partial r^{*}} + B.$$
(3.2)

The above system of equations has been examined for a low-Reynolds-number constant-squeezing-force situation in Zwick *et al.* (1996), and an analytical expression for  $U_r$  has been developed. We have used that expression for  $U_r$  as the reference velocity to describe our present experimental measurements:

$$U_r = \left\{ \frac{(n+3)Fh_0}{2\pi mR^3} \right\}^{1/n} \frac{nh_0^2}{(2n+1)R}.$$
 (3.3)

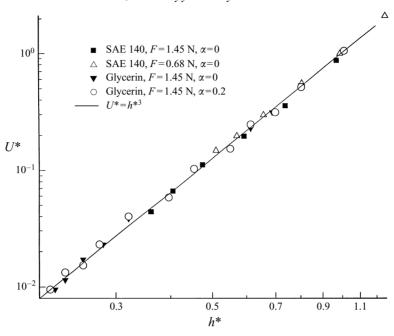


FIGURE 3. Dimensionless velocity versus film thickness for Newtonian fluids SAE 140 gear oil, and glycerin.

Also.

$$\frac{U}{U_r} = U^* = h^{*2} (h^* - 1)^{1/n}. \tag{3.4}$$

From the slope of a log-log plot of  $U/h^2$  versus  $(h-h_0)$  we may determine the value of the Herschel-Bulkley power-law exponent n. The value of the Herschel-Bulkley viscosity parameter m may then be determined from the intercept of this plot. This data reduction method is similar to that used by Covey & Stanmore (1981) for non-oscillatory squeeze films.

Finally, we define the flow enhancement by

$$\Delta Q = U^* - U_0^*, \tag{3.5}$$

where  $U_0^*$  is the squeezing velocity for the same fluid and film thickness under conditions of constant-force squeezing ( $\alpha = 0$ ).

#### 4. Results and discussion

We tested the accuracy of our plastometer and the data reduction method by squeezing the Newtonian fluids glycerin (99.7% pure) and SAE 140 gear oil and comparing the results to viscosity measurements obtained using a Brookfield Couette viscometer. The power-law indices were unity, and the limiting film thicknesses were zero, as expected for Newtonian fluids. The squeeze film viscosity measurements were within  $\pm 1\%$  of the Brookfield measurement for the SAE 140 oil, indicating an acceptable accuracy. The SAE 140 oil and the glycerin were also squeezed under a variety of oscillatory frequencies and amplitudes. No changes in average velocity of squeezing versus film thickness were discernible under any condition, indicating lack of any flow enhancement. For Newtonian fluids the limiting film height,  $h_0$ , is

Fluid	Measurement method	T (°C)	Squeezing force (N)	n	m (Pa s <sup>n</sup> )	$ \tau_0 = K_n \tau_y  \text{(Pa)} $	h <sub>0</sub> (mm)
SAE 140 Oil*	Plastometer	22	0.68	1	2.28	0	0
SAE 140 Oil*	Plastometer	22	1.45	1	2.33	0	0
SAE 140 Oil*	Brookfield	22	NA	1	2.30	0	0
Glycerin*	Plastometer	25	1.45	1	0.36	0	0
Mustard	Plastometer	24	1.45	0.72	1.41	89.4	0.208
1.5% FS/Oil	Plastometer	25	0.68	0.89	0.117	20.15	0.100
Epoxy	Plastometer	25	1.45	1.0	52.9	90.22	0.210

<sup>\*</sup>Newtonian Fluid

TABLE 1. Experimental parameters

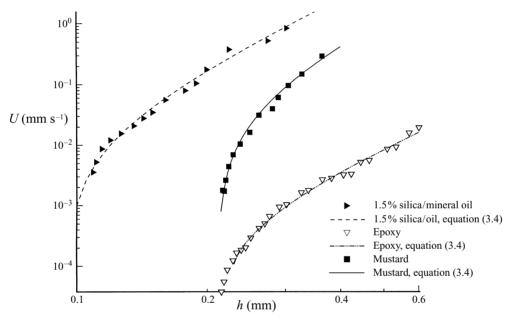


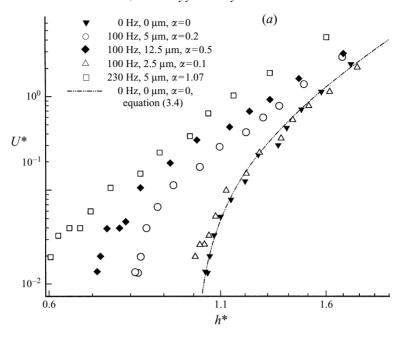
FIGURE 4. Velocity versus film thickness for the fumed silica/paraffin oil solution, mustard, and epoxy. The solid and dashed lines are from a fit of equation (3.4) to the experimental data.

zero. We substitute the initial film height  $h_i$  everywhere for  $h_0$  in our dimensionless parameters in this case. Figure 3 shows the variation of dimensionless velocity  $U^*$  versus dimensionless film thickness  $h^*$  for the Newtonian fluids, SAE 140 and glycerin, displayed on a log-log scale. The solid line corresponds to

$$U^* = \left(\frac{h}{h_i}\right)^3,\tag{4.1}$$

which may be derived from equation (3.4). For SAE 140 and glycerin, the values of m, n, and  $h_0$  are presented in table 1.

The plate velocity U versus film thickness h for the yield stress fluids fumed silica/mineral oil, mustard, and epoxy under constant-force squeezing are plotted on a log-log scale in figure 4. We note that for each of these fluids, the plate velocity approaches zero at a finite film thickness. This is the limiting film thickness  $h_0$  for the



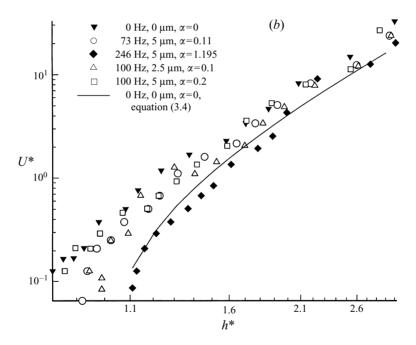


FIGURE 5 (a, b). For caption see facing page.

yield stress fluid. The fumed silica/mineral oil suspension has a lower limiting film thickness than that for the epoxy or the mustard. The yield stress under constant-force squeezing has been shown to be directly proportional to the limiting film thickness in Zwick *et al.* (1996), implying that the yield stress for the fumed silica/oil suspension is the smallest among the three fluids examined. The solid and dotted lines are obtained

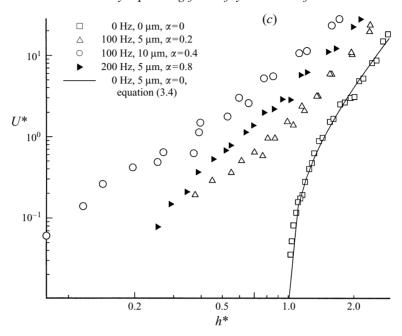


FIGURE 5. Dimensionless velocity versus dimensionless film thickness for oscillatory squeezing of (a) mustard, with  $\alpha = 0, 0.1, 0.2, 0.5$  and 1.07; (b) a 1.5% fumed silica/paraffin oil suspension, with  $\alpha = 0, 0.1, 0.11, 0.2$  and 1.195; (c) die bonding epoxy, with  $\alpha = 0, 0.2, 0.4$  and 0.8.

by fitting equation (3.4) to the data. The resulting values of m, n, and  $h_0$  are presented in table 1. The agreement between equation (3.4) and the measured data is excellent.

The variation of  $U^*$  versus  $h^*$  for various values of oscillatory frequencies  $\omega$  and amplitudes a and  $\alpha$  is plotted for mustard in figure 5(a). Equation (3.4) fits the  $\alpha=0$  curve well. The values of  $Re_{\omega}$ , Re, B, and  $\epsilon$  are given in table 2 for mustard with film thickness  $h_i=0.3$  mm and frequencies of 100 and 230 Hz. The velocity used in determining the value of the dimensionless groups is the actual measured plate velocity at film thickness  $h_i$ . The quantities B, Re,  $Re_{\omega}$ , and  $\epsilon$  are all small, so that we expect the predicted behaviour from equation (3.4) to be a reasonable approximation of the non-oscillatory squeezing flow. This is evident in figure 5(a). Oscillating the force enhances the squeezing velocity at all film thicknesses. It is apparent from the figure that the flow enhancement increases with increasing values of  $\omega$ , a, and  $\alpha$ . The limiting film thickness  $h_0$  decreases with increasing values of  $\omega$ , a, and  $\alpha$ . In fact, at 230 Hz and a=5 µm ( $\alpha=1.07$ ) the limiting film thickness approaches about 50% of its value at 0 Hz, and 0 µm ( $\alpha=0$ ). This indicates that the apparent yield stress is a function of the amplitude and the frequency of the squeezing force, and it dramatically decreases with increasing values of  $\omega$ , a, and  $\alpha$ .

The dimensionless velocity is plotted versus dimensionless film thickness for the fumed silica/oil suspension and the die bonding epoxy in figures 5(b) and 5(c), respectively. The effects of oscillation are qualitatively similar to those described for mustard. However, for epoxy, the effect of doubling the displacement amplitude a (hollow triangles to circles) is larger than the effect of doubling oscillatory frequency  $\omega$  (hollow triangles to filled triangles). The greatest flow enhancement (circles) does not correspond to the largest value of  $\alpha$  (filled triangles). For the fumed silica suspension, the flow enhancement is smaller than with mustard or epoxy, and it does

Fluid	Frequency	Film thickness $h_i$ (mm)	Velocity U (mm s <sup>-1</sup> )	$\epsilon$	Re	$Re_{\omega}$	В
Glycerin	0 Hz (0 rad s <sup>-1</sup> )	0.3	0.454	0.02	0.0005	0.0	0.14
Mustard	230 Hz (1445 rad s <sup>-1</sup> )	0.3	0.66	0.02	0.0006	0.35	0.08
Mustard	100 Hz (628 rad s <sup>-1</sup> )	0.3	0.26	0.02	0.0002	0.12	0.015
Epoxy	200 Hz (1257 rad s <sup>-1</sup> )	0.3	0.005	0.02	0.00006	0.004	0.13

TABLE 2. Fluid properties

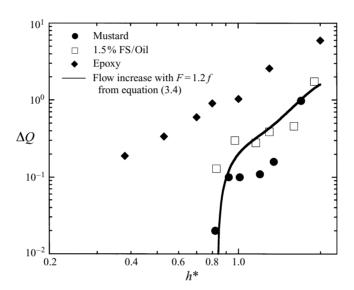


FIGURE 6. Flow enhancement measured for 1.5% fumed silica/paraffin oil, mustard, and epoxy versus dimensionless film thickness with  $\alpha = 0.2$ . The predicted flow increase obtained by squeezing with a constant force equivalent to the peak value in the oscillatory squeezing F = f(1+0.2) is also plotted.

not greatly increase with increasing  $\alpha$ . There is little difference between the  $\alpha=0.2$  and the  $\alpha=1.195$  results. In both instances, however, there is flow enhancement and a distinct reduction in limiting film thickness. The different responses of the three yield stress fluids in figure 5 shows that the flow enhancement and the change in limiting film thickness are highly dependent on the particular nature of the fluid.

Figure 6 shows the measured flow enhancement,  $\Delta Q = (U_a^* - U_0^*)$ , for the yield stress fluids studied at a frequency  $\omega = 100$  Hz ( $200\pi$  rad s<sup>-1</sup>), a displacement amplitude a = 5 µm, and a force amplitude  $\alpha = 0.2$  on a log-log scale. The flow enhancements for the various fluids are all qualitatively similar, with the enhancement increasing with increasing  $h^*$ . The solid line denotes the flow increase as calculated from equation (3.4) with a constant force F = 1.2f. This value of F corresponds to the peak force applied during the actual measurements reported in the figure. The flow increase with F = 1.2f goes to zero at  $h^* = 0.83$ . This is where the plate velocity would go to zero for the film under the larger load. The flow enhancements for the fumed silica/oil suspension and the mustard are reasonably approximated by this line, with  $\Delta Q$  approaching zero at about the same value for  $h^*$ . This suggests that the fumed silica/oil suspension and the mustard primarily respond to the peak force applied during oscillatory squeezing. However, the flow enhancement with the epoxy

is significantly larger at all film thicknesses, and it does not appear to approach zero at any finite film thickness. It is interesting to note that oscillating the squeezing force with a given amplitude increases the flow rate of the epoxy beyond what would be obtained under a constant force equal in value to the peak force. This suggests that the oscillations fundamentally modify the flow behaviour of the die bonding epoxy, although the identification of the exact mechanism(s) warrants more study. It now appears that the epoxy can be quickly and efficiently squeezed from under the chips by oscillating the chip with a lesser force (lesser than both mean and peak) than would be required under constant-force squeezing. This observation has important implications for the bonding of thin fragile microelectronic chips.

#### 5. Conclusion

Experiments have shown that the superposition of an oscillatory force upon a mean squeezing force significantly increases the flow rate and reduces the limiting film thickness in the squeezing flow of yield stress fluids. This is a novel result. In the limit of constant-force squeezing, the experimental results obtained here compare well with an earlier theoretical model developed by the authors for that situation. All yield stress fluids studied show similar trends for flow enhancement under oscillatory squeezing. This flow enhancement has important applications in microelectronic manufacturing and possibly in injection moulding.

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