

## RHEOLOGY SOLUTIONS

### EVALUATION OF GREASES BY CONTROLLED STRESS RHEOLOGY

#### PROBLEM

Determination of the rheological or flow properties of greases under a variety of shear conditions is important for predicting the greases' suitability for specific end-uses. An instrument called the Lincoln Ventmeter is often used for evaluating the flow properties of greases. That device is based on a pressurized (typical pressure 12,400 kPa) capillary which gives an index of pumpability by measuring the pressure drop across a specific distance. Although the measurement is controlled stress in nature, the shear rate in the device is not constant for the entire sample during measurement because the capillary measurement geometry has a parabolic velocity profile with the shear rate approaching zero at the centerline of the tube and a maximum at the tube wall. Hence, calculation of a relative or absolute viscosity from the experimental results is difficult.

#### SOLUTION

A controlled stress rheometer, however, provides an easy-to-use quantitative alternative to the Lincoln Ventmeter. In the controlled stress rheometer (CSR), a precisely controlled, operator-selected torque is applied to a rotating shaft and measurement geometry in contact with the sample material, and the resultant strain properties are measured. The applied torque (stress) can be ramped up or down or held steady to evaluate flow and viscosity under steady (unidirectional) shear as well as to evaluate the effects of oscillatory dynamic motion and creep. Controlled stress rheology is particularly useful for probing the region of flow behavior where flow actually starts. Furthermore, quantitative viscosity can be obtained after calibration with standard Newtonian oils.

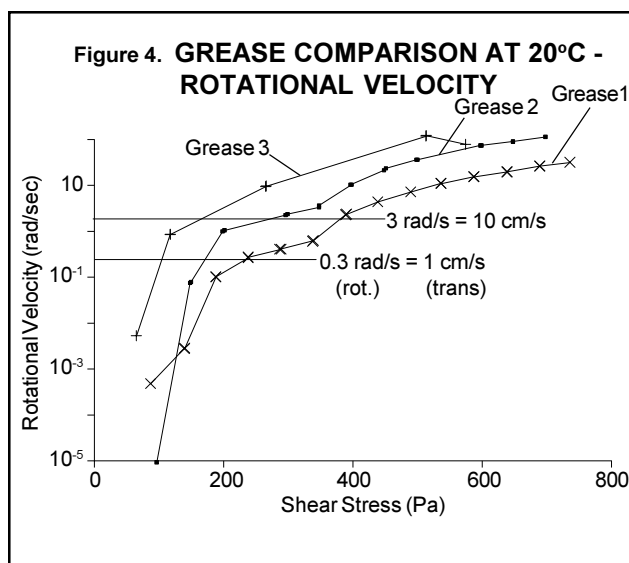
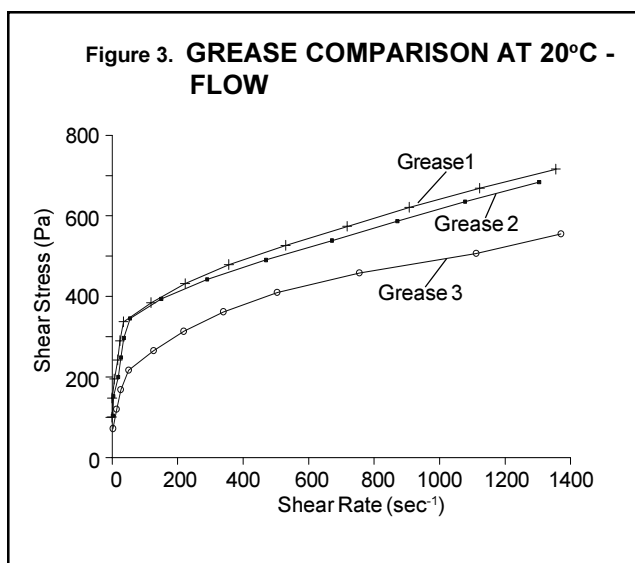
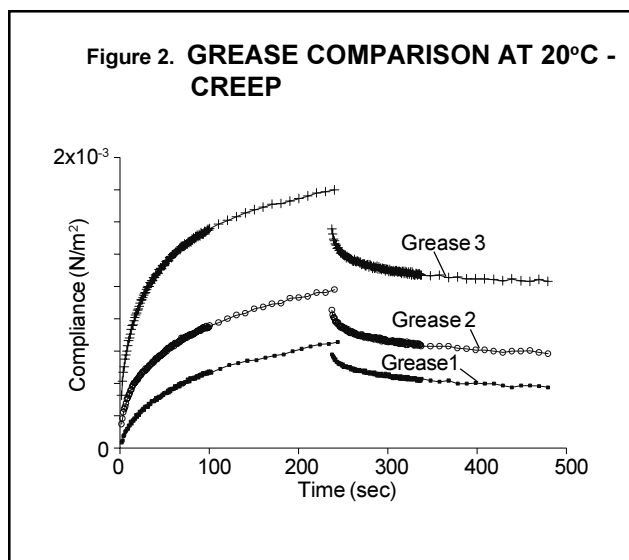
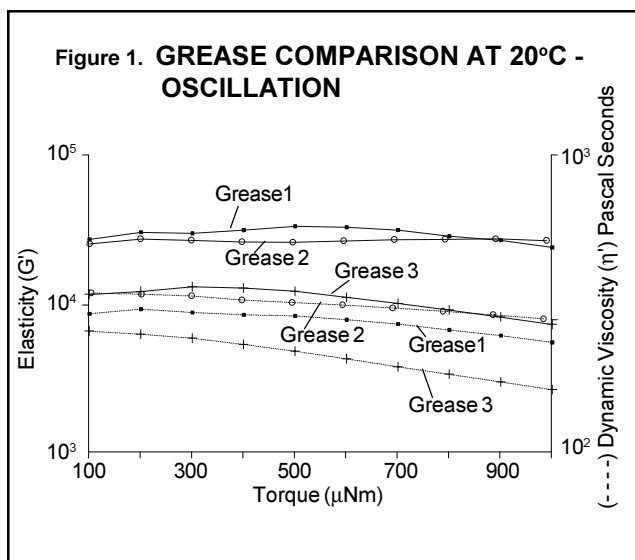
A series of three different greases were evaluated using a controlled stress rheometer under oscillation, creep, and flow conditions. A 2cm, 2° cone & plate geometry was selected for all three evaluation conditions because it provided a uniform but high shear rate across the sample gap, required only a small volume of material, and achieved rapid temperature equilibrium.

In oscillation, a small sinusoidal stress is applied to the sample to perturb it without irreversible damage to its

structure (flow). By analyzing the sinusoidal strain output and comparing it to the input, the phase angle  $\delta$  can be measured and the in-phase and out-of-phase components can be calculated. Thus, viscoelastic samples can be evaluated and the relative proportions of viscosity and elasticity can be assigned. If an increasing series of stress waves is applied, the stress/strain relationship can be examined and the point at which the material begins to deform can be seen. Figure 1 shows the  $G'$  (dynamic rigidity or elasticity) and  $\eta'$  (dynamic viscosity) results from a torque (stress) sweep for the three greases where a fixed frequency (1Hz) was applied with increasing stress amplitude. The order of stiffness and hence reverse order of pumping ease was 1>2>3.

Creep measurements evaluate a material's deformation under a steady stress. Generally, the stress applied is low so that the material can partially recover (if it is viscoelastic) once the stress is removed. The strain rates achieved are low which allows an equilibrium (Newtonian) viscosity  $\eta_0$  to be measured. By using a combination of standard flow (with the application of stress ramp) and equilibrium flow or creep, a flow curve can be generated over a huge range of shear rates. The curve allows the flow resistance in a number of situations, e.g., at rest, in mixing, in pumping and ultimately in lubrication, to be predicted. Figure 2 shows the creep results for the three greases. Grease 1 had the lowest compliance (highest elasticity).

Figure 3 shows the conventional shear stress versus shear rate flow data for the three greases. The slope of the line obtained is defined as the viscosity, and was consistent with the dynamic and creep data. This flow data was generated by an equilibrium flow technique where each data point was a creep experiment in its own right. Figure 4 shows the flow data replotted as rotational velocity versus applied stress. Based on the known measurement geometry, the translational velocity of the sample can be calculated from the rotational or measured velocity. Lines have been added to the plot to show which points correspond to a translational velocity 1cm/s and 10cm/s. Base on curves like these, the applied stress from the pressure stroke of a pump can be used to predict the flow velocity of the material and subjectively the velocity can be categorized as acceptable or not. Then, either a stronger pump can be utilized or the sample warmed to improve the situation.



**ACKNOWLEDGEMENT:** This brief is based on studies by Peter Whittingstall in TA Instruments Applications Lab (US)

For more information or to place an order, contact:

**TA Instruments, Inc.**, 109 Lukens Drive, New Castle, DE 19720, Telephone: (302) 427-4000, Fax: (302) 427-4001

**TA Instruments S.A.R.L.**, Paris, France, Telephone: 33-01-30489460, Fax: 33-01-30489451

**TA Instruments N.V./S.A.**, Gent, Belgium, Telephone: 32-9-220-79-89, Fax: 32-9-220-83-21

**TA Instruments GmbH**, Alzenau, Germany, Telephone: 49-6023-30044, Fax: 49-6023-30823

**TA Instruments, Ltd.**, Leatherhead, England, Telephone: 44-1-372-360363, Fax: 44-1-372-360135

**TA Instruments Japan K.K.**, Tokyo, Japan, Telephone: 813-5434-2771, Fax: 813-5434-2770

Internet: <http://www.tainst.com>

**TA Instruments**  
**Thermal Analysis & Rheology**  
 A SUBSIDIARY OF WATERS CORPORATION