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VERY HIGH SHEAR RATE VISCOMETRY

Theodore W. SELBY[1] and Greg C. MIILLER[2]

[1]*Savant Inc., Midland, Michigan, U.S.A.*
[2]*Tannas Co., Midland, Michigan, U.S.A.*

Very high shear rate viscometry has been a subject of long interest but limited data particularly in its association with hydrodynamic lubrication of automotive engine bearings and the control of wear and friction. Shear rates of one million reciprocal seconds has relatively recently become a standard specification for engine oils and studies at two million reciprocal seconds have been reported. However, these levels are considerably less than the seven to ten million reciprocal seconds associated with the operating automotive engine. Recent work by the authors has been focused on extending the range of shear rate. Information has been collected with a multiple speed instrument which now approaches the very high shear rate associated with automotive bearings. This paper presents initial findings and the associated instrument and technique.

Keywords: viscometry, very high shear rate, viscous grip, shear-thinning, engine oils, non-Newtonian behavior, VI Improvers

INTRODUCTION

Importance of Viscosity in Lubrication

The viscosity of engine oils (and lubricants in general) has always been their most important property. By itself viscosity controls hydrodynamic lubrication and, in concert with with other lubricant factors, affects wear, friction, efficiency, transport of chemicals as well as products of wear, corrosion, and other contaminants through the engine.

Focus of the Paper

Of the various ways in which viscosity can affect the engine, this paper will focus on a techniques of very high shear rate measurement of viscosity. Such measurement is closely related to the effectiveness of hydrodynamic lubrication in the automotive journal bearing.

Non-Newtonian Behavior of Multigrade Engine Oils

High molecular weight polymer dilute solutions have long been a subject of rheological interest and theoretical speculation. Engine oils with their high polymer VI Improvers are but a sub-set of that class of liquids and are expected to behave reasonably the same. One difference between engine oil and other dilute high polymer solutions is found in the wide heterogeneity of the mineral oils' molecular composition and the tight unimolecularity of many relatively pure solvents used to study high polymer physics. Another associated property is the considerably higher collective viscosity of a base oil which can be viewed as a product of its heterogeneity.

Flow Dynamics of Polymer-Containing Oils - A number of experimental and theoretical studies over the last 40 years have shown that a polymer-containing liquid exhibits a so-called first and second Newtonian region. The first exists over a low shearing force range where a low level of 'viscous grip' (see Footnote 1 for definition) of the small solubilizing molecules is shown. The second Newtonian region is found in the very high shear stress range where the 'viscous grip' of the polymer-solubilizing liquid molecules is very high. Under these conditions the domain of the polymer molecule is distorted to the limit of its elasticity and its contribution to the liquid's viscosity becomes constant. Between these two regions exists a region in which the polymer-containing liquid is markedly non-Newtonian in flow. These three regions are shown in Figure 1.

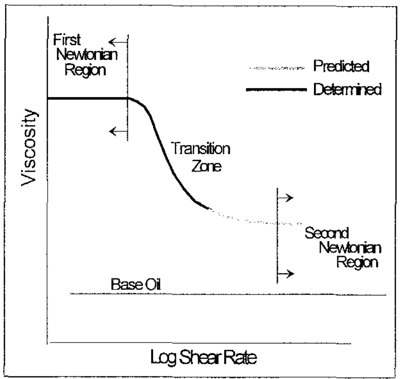

Fig. 1 Schematic representation of non-Newtonian shear curve of polymer-containing base oil

Footnote 1: The term 'viscous grip' has been coined by one of the authors (Selby) to define the influence of the polymer-solubilizing oil (or other) molecules on the polymer coil surrounded and permeated by the smaller molecules. Both influence one another; the polymer by interfering in the streamline flow of the smaller molecules (absorbing some of their energy) and thereby increasing the viscosity of the mixture; the smaller molecules by the effects of their cohesive forces in distorting the polymer coil when at either sufficiently high velocity or high cohesive energy levels (lower temperatures) thereby lowering the viscosity of the mixture.

The importance of obtaining such information led some investigators (1) to extrapolate high shear capillary viscometry by mathematical techniques. While interesting, the problem with such efforts is that, in one way or another, it presumes some knowledge about the behavior of the expectedly complex flow of polymeric solutions in this experimentally unknown region. Further, high shear capillary viscometry with its sensitivity to heat effects and its non-linear flow profile (particularly with non-Newtonian liquids such as polymer-modified engine oils), seems hardly the best platform from which to extend theory.

In addition, some VI Improvers increase their domain or degree of solubility in, and, thus, their viscous contribution to, the base oil with increasing temperatures (and for this reason have been called VT Improvers (2)). This behavior of some polymer coils would be expected to further reduce confidence in values of predicted viscosity at higher shear forces.

The obvious recourse is to actually measure viscosity at these very high shear rate conditions above $5 \bullet 10^6 sec^{-1}$ and this was the focus of the present work. Concerns by other investigators that such shear rates could not be attained (3) was an additional stimulus to the effort as was the desire of making any instrumental approach simple and straightforward for use in the laboratory. On the basis of the prior experience with high shear rate, tapered rotational viscometry (4-6) there seemed to be no obvious difficulty other than the control of operating temperature at these extraordinarily high shear rates. In the latter case, the frictional heat produced by the viscosity of a fluid increases exponentially with shear rate and linearly with viscosity according to the textbook equation

$$\mathbf{H} = \mathbf{R}_s^{2} \bullet \eta \qquad \text{Eq. 1}$$

in which **H** is heat, $\mathbf{R}_s$ is shear rate, and η is dynamic viscosity. Fortunately, this question could be simply resolved by determining the linearity of the torque-speed relationship using a Newtonian liquid (6). If the torque-speed relationship is linear then three conditions are met: 1) the fluid is proven truly Newtonian, 2) the viscometer is capable of maintaining constant temperature, and 3) the viscometer is capable of mechanically functioning as a true viscometer under the operating conditions presented.

BACKGROUND - HIGH SHEAR VISCOMETRY

Tapered Geometry Viscometry

From 1940 through 1978 efforts to reach high shear rates were made by a number of investigators using rotational and capillary equipment. Both approaches had their limitations. Temperature effects modified the concentric cylinder viscometer at operating temperatures and gave somewhat uncertain dimensions of the rotor-stator gap (and thus somewhat uncertain shear rate). Equally difficult, particularly with non-Newtonian viscometry, the capillary has a variable shear rate across the annulus as well as serious heat effects to overcome. Despite these limitations and uncertainties, moderate to fairly high shear rates were obtained with both instruments.

In particular, interesting work with a rotational, slightly **tapered** geometry was published in 1978 in which Pike and his associates (7) reached approximately 400,000 sec^{-1}. While they could only estimate the shear rate, it could be adjusted by moving the tapered rotor up or down in the matching tapered stator as illustrated in Figure 2.

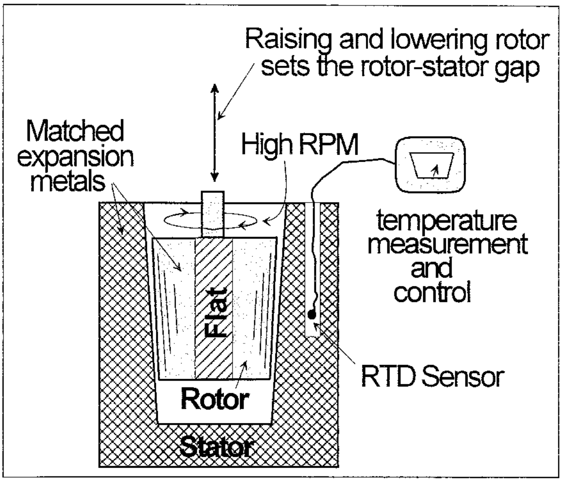

Fig. 2 Sketch of the rotor-stator configuration and adjustment to change shear rate.

Reaching 1,000,000 Sec^{-1} - This adjustable shear rate approach seemed to hold promise and in 1980 a viscometer was exhibited which was shown capable of reaching shear rates of a million or more reciprocal seconds. As in Pike's work (7), the instrument had a slightly tapered rotor/stator geometry of 164/1 (vertical to horizontal). Using a hysteresis motor for close control of a rotational speed of 3600 RPM, it was found possible to make rotor/stator pairs with sufficient precision to form a gap of 3.5 μm. Thus, not only was the viscometer found capable of reaching 1,000,000 sec^{-1} and more shear rate but to exhibit the linear torque-speed relationship and thus the temperature control required for acceptable viscometry.

From its geometry and projected application to automotive engines and engine oils, the viscometer was called the Tapered Bearing Simulator (TBS) viscometer. The slightly tapered geometry was combined with a relatively easily adjustable and measurable rotor position (which was found very important in use of the instrument as an absolute viscometer).

The development of the tapered rotational instrumental approach led to its application in SAE engine oil specifications in 1993. However, the challenge and the need remained to extend this high shear viscometry into the considerably higher levels experienced by operating automotive bearings (8-11).

First Extension to Very High Shear Rate Viscometry

Use of Absolute Viscometry - The TBS was indicated to be an absolute viscometer in 1983 when, using the readily adjustable rotor height, it was shown that the operating shear rate could be determined during operation (5). This finding had high implications for using the instrument at different shear rates and at different temperatures since the gap and the operating shear rate could be directly determined during viscometric analysis using a Newtonian oil.

ASTM Multi-Shear, Multi-Temperature Data - On the basis of the above findings, multi-shear/temperature TBS data was requested and supplied (11) during efforts within ASTM to resolve the question of correlation between high shear viscometry and bearing oil film thickness (BOFT) (8-10). The multiple operating shear rates were determined using the TBS as an absolute viscometer. Ultimately, these studies were used among other data to support development of the high shear rate engine oil viscosity classification.

In this ASTM work it was necessary to show that the heat generated by shearing the liquid at shear rates up to 2,000,000 sec^{-1} was controlled. Accordingly, using four Newtonian reference oils, a series of eight selected rotor-stator gaps between 35 and 1.75 µm was made to give shear rates ranging from 100,000 to 2,000,000 sec^{-1} at 150°C. If the viscometer were capable of operating free of heat and temperature effects over that range, linear plots of the indicated torque versus the shear rate would be produced with slopes proportional to the viscosity of the oils and a common intercept (normally near zero). The results are shown in Figure 4.

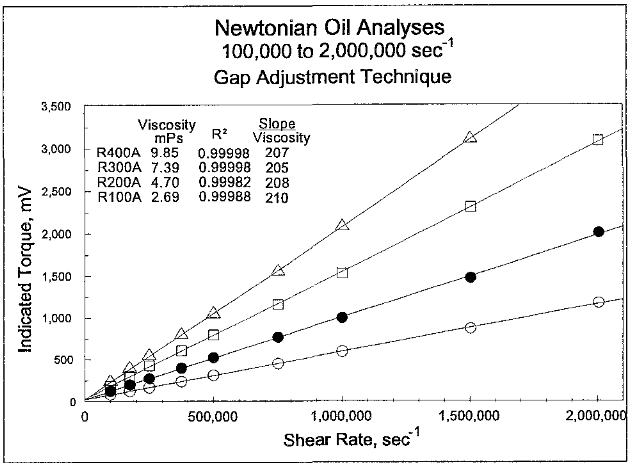

Fig. 4 Demonstration of wide shear rate range results with variable-gap instrumentation using Newtonian oils.

The four straight lines having common intersection close to zero on the Y-axis in Figure 4 give reasonable evidence that 1) the four oils are Newtonian behavior and 2) the viscometer does not show temperature effects -- at least up to 2,000,000 sec^{-1}.

This work of determining the viscosity at different shear rates and temperatures permitted extension of the Cross Equation (12) to very high shear rates of 10,000,000 (10,11). However, the empirical nature of that equation plus its assumption that the viscosity of the polymer-containing oil would be reduced to the viscosity of the base oil at infinite shear stress, was not satisfying. Far better would seem to be the measurement of viscosities of polymer-containing engine oils at these very high shear rates.

From the experience of the foregoing and other work from 100,000 to 2,000,000 sec^{-1} it was apparent to the authors that, to obtain shear rates above 4,000,000 to 5,000,000, it would be necessary to change rotor speeds rather than gap adjustment for varying shear rate. This has led to development and studies with a multispeed adaptation of rotational tapered geometry.

EQUIPMENT AND TECHNICAL APPROACH

The equipment used is a commercially available viscometer called the Super Shear TBS (SSTBS) Viscometer. This instrument has an overall speed adjustment range of one order of magnitude -- 800 to 8,000 RPM through twelve selectable speeds of 800, 1500, 2000, 2500, 3000, 3500, 4000, 4500, 5000, 6000, 7000, and 8,000 RPM.

Although the SSTBS instrument is capable of operating at shear rates up to about 3,000,000 sec^{-1} with no cooling assistance, dry air was used to assist in the heat removal necessary at higher shear rates and viscosities. At very high shear rates, a special cold bath was developed to cool the dry air to considerably lower temperatures than ambient which used a length of copper coil immersed in a mixture of solid CO_2 and methanol.

RESULTS AND DISCUSSION

Newtonian Viscometry

Initial results to determine the utility of the multi-speed approach in extending high shear rate viscometry into very high speed viscometry showed that linearity similar to that obtained by variation of gap could be obtained. This is shown in Figure 5.

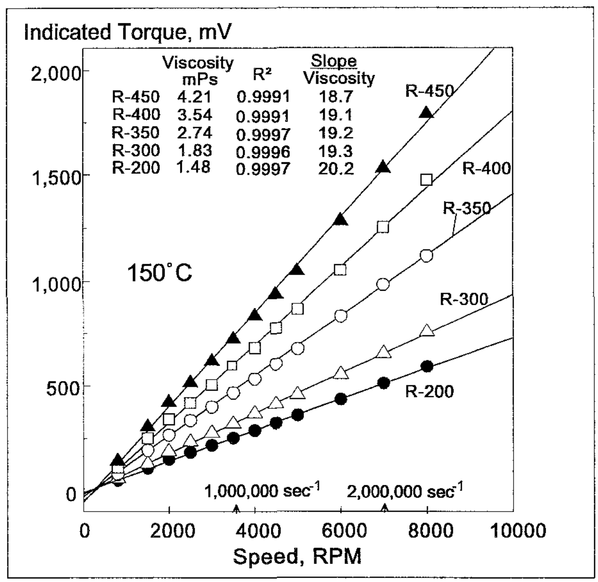

Fig. 5 Demonstration of wide shear rate range results with multi-speed instrumentation using Newtonian oils.

Figure 5 shows that over the shear rate range covered, the correlation of torque and speed is high for any of the Newtonian reference oils. Moreover, the ratio of slope to viscosity (which should be constant) is reasonably so and the intercepts are relatively closely gathered (theoretically, they should be identical).

With this information and using the solid CO_2-methanol air-cooling bath, the SSTBS viscometer was then required to work at shear rates using a Newtonian reference oil somewhat above 5,000,000 sec^{-1} to test linearity of the torque/speed (shear rate) relationship. For improved platform stability of the SSTBS a 10-g pre-load was attached using the load-cell

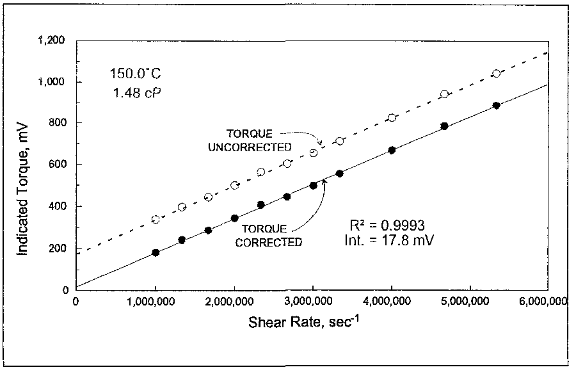

Fig. 6 Examination of linearity of torque/shear-rate relationship using Newtonian oil at shear rates from 1,000,000 to over 5,000,000 sec^{-1}.

calibration pulley. This 10-g load was equivalent to 157 mV reading of the indicated torque meter.

The results are shown in Figure 6.

Linearity is good with a Coefficient of Determination, R^2, of 0.9995. The intercept -- which reflects other influences on the load cell and should be near zero -- is 173 mV. However, correcting this value by that of the 10-g pre-load, 157 mV, gives a value of 17.8 mV -- a reasonable value for small, uncorrected influences on the load cell.

With this information, it is apparent that the SSTBS can operate at more than 5,000,000 sec^{-1} shear rate with control of rotor speed and heat influences.

Non-Newtonian Viscometry

The technique and instrumentation was then applied to a non-Newtonian reference oil, NNR-03, used in calibrating the TBS and the SSTBS viscometers. In this case the shear rate

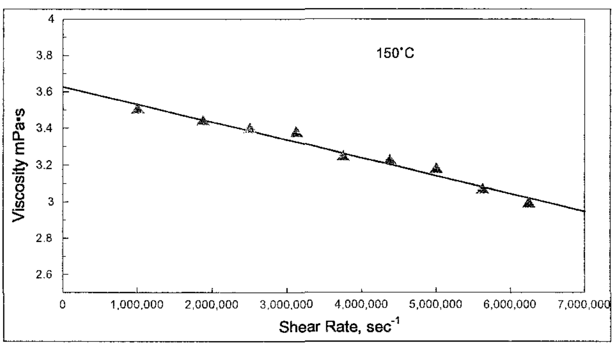

Fig. 7 Study of non-Newtonian oil over a shear rate range extending to over 6,000,000 sec^{-1}.

was extended to over 6,000,000 sec^{-1}. Results gathered at 150°C are shown in Figure 7.

It is evident from the virtually linear slope of the data in Figure 7 that the onset of the so-called 'second Newtonian' region (see Figure 1) has not begun. Over the shear rate range from 1,000,000 to 6,000,000 sec^{-1} a decrease in viscosity of more than 15% is shown -- a considerable change for an engine oil.

In contrast, a second, much more shear susceptible, engine oil was analyzed over a more limited range of shear rate but at

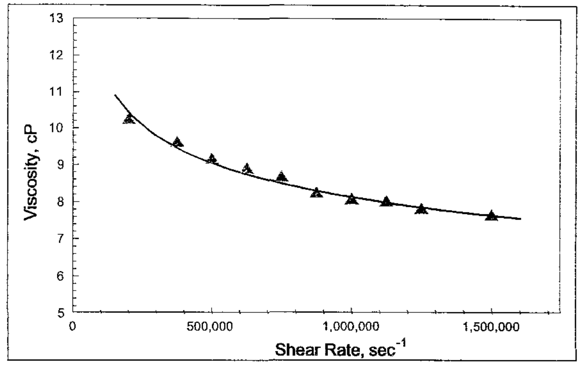

Fig. 8 Shear force susceptibility of a multigrade engine oil over a shear rate range of 2,000,000 to 1,500,000 sec^{-1}.

100°C where the 'viscous grip' of the surrounding oil molecules are more effective in creating temporary viscosity loss of the polymeric VI Improver.

This information is shown in Figure 8. Even with the more limited shear rate range, it is evident that, under the 'viscous grip' of the base oil, this highly susceptible oil is tending to reach the 'second Newtonian' region. Over the shear rate range used, the oil has lost 34% of its viscosity

On the basis of these data on non-Newtonian engine oils, super-shear viscometry seems to hold promise in evaluating the properties and types of VI Improvers on the basis of their response to very high shear rate viscometry.

CONCLUSIONS

This work with a very high shear rate viscometer using Newtonian oils has shown that it is possible to measure the viscosity of engine oils at shear rates above 5,000,000 sec^{-1} without loss of either temperature control or viscometer speed. In the process of gathering this data, good correlation of speed and torque was shown as well as a low intercept value.

When the super-shear TBS viscometer in these studies was applied to non-Newtonian engine oils significant shear thinning occurred. Although this data is from preliminary studies, the degree to which shear thinning occurred seemed related to the type of polymeric VI Improver and the degree to which the surrounding oil matrix could exert its 'viscous grip' in exerting distortional forces on the macromolecular envelope of solubility.

FUTURE WORK

Higher shear rate studies are now underway using the super-shear TBS viscometer.

The authors of this paper believe that considerably higher shear rates can be attained -- shear rates equivalent to levels in the automotive bearings.

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