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Measurement and Prediction of Fluid Viscosities at High Shear Rates

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1. Introduction

Polymeric materials in general are viscoelastic in nature because they exhibit strong dependence of deformation and flow on time and temperature. Molecular structures in polymeric materials undergo rearrangements when a load is applied in order to minimize localized stresses imposed by the applied load. Performance of materials in real time applications can only be evaluated by testing the materials under conditions that the material would encounter in real time applications. This poses serious problems in evaluating materials that would need to be in service over very long periods of time or if the material undergoes deformations (rates) over a wide range. For example, it would be impossible to evaluate the flow behavior of materials in solar panels over the span of time they are designed for (20 – 30 years). Similarly, aircraft/spacecraft materials that are rated for service up to thousands of hours cannot be evaluated. Measurements taken using an instrument at a set temperature usually cover a range of three to four orders of magnitude of time or frequency [1]. This range is usually not sufficient to evaluate material viscoelastic behavior in the complete range of frequencies required, from the low frequency terminal zone to high frequency glassy region.

Viscosity measurements of materials have become a very essential part of a wide variety of industries including petroleum, food, plastics, paint and composite industries, especially in the last decade. With the advent of advanced lightweight structures like fiber reinforced plastics, that possess high specific strength and specific modulus, the development of testing techniques to evaluate the lifetimes of such materials in operating environments has become of high importance. The life times of some of these materials are a few years which makes it impossible to conduct real time experiments for that span of time. The TTS technique works

very well in such situations to predict the behavior of the material over the life time of the material in specific applications.

Time-temperature superposition (TTS) has been used to solve the kind of problems mentioned above over the last few decades. This technique is well grounded in theory and applies to a wide variety of hydrocarbon materials that are thermorheologically simple. Thermorheologically simple materials are those where all relevant relaxation and retardation mechanisms as well as stress magnitudes at all times and frequencies have the same temperature dependence.

The theory and mechanisms that form the basis for the TTS technique have been well documented in literature. However, a brief overview of the underlying theory of the TTS technique is presented to refresh the readers' memory.

The first step towards using the TTS technique is to generate data at several temperatures close to the temperature at which the flow properties/deformations of the material in question are to be evaluated (reference temperature). The flow curves at the temperatures obtained can then be superposed on to a master curve showing the material behavior at reference temperature. The TTS technique is based on the facts that the molecular rearrangements that occur due to applied stresses take place at accelerated rates at elevated temperatures and that there is an analogous relationship between time and temperature [2, 3]. These phenomena lend to the ability to conduct measurements at elevated temperatures and then superpose the data to lower temperatures. Superposing the data to lower temperatures enables the analyst to predict the material behavior over a large time scales (small deformations over long periods of time). Similarly, superposing lower temperature data to a higher temperature enables one to predict the material behavior over smaller time scales (high shear rates).

The extent of shifting along the x and y axes in the TTS technique, in order to superpose experimental data on to that at the reference temperature, is represented by the horizontal shift factor a_T and vertical shift factor b_T . Variables that have units of time or reciprocal time get subjected to a horizontal shift and variables that have units of stress or reciprocal stress are subject to a vertical shift [1]. The vertical shift factor (b_T) can be calculated using the equation 1 [4, 5]:

$$b_T = T_0 Q_0 / T Q \quad (1)$$

The horizontal shift factor is given by the Arrhenius relationship:

$$a_T(T) = \exp\left[\frac{E_a}{R}\left(\frac{1}{T} - \frac{1}{T_0}\right)\right] \quad (2)$$

Where E_a is the activation energy, R is the gas constant, T is the measurement temperature and T_0 is the reference temperature. This relationship is valid as long as the measurement temperature is well above the T_g of the material. For measurement temperatures closer to T_g , the following relationship holds well:

$$\log a_T = \frac{-c_1(T - T_0)}{[c_2 + (T - T_0)]} \quad (3)$$

Where c_1 and c_2 are empirical constants.

The application of TTS to predict the creep behavior of materials over long period of time has been in practice for a few decades. However, predicting material flow behavior and viscosity values of materials at extremely high deformation rates is a relatively uncharted territory. This study demonstrates the use of capillary rheology to measure viscosity profiles of motor oils up to high shear rates of 2,000,000 s^{-1} . Viscosity profiles of oils at six different temperatures up to shear rates of $\sim 2,000,000 s^{-1}$ are presented. The data sets at lower temperatures (100, 110, 120, 130 and 140 °C) are superposed on to the data set at 150 °C using the TTS technique (IRIS software) in order to predict the viscosity behavior of the oil at extremely high shear rates (up to ~ 15 million s^{-1}) at 150 °C. This study demonstrates that the TTS technique can be used to predict such data with reasonable accuracy.

2. Experimental

Steady shear rheological measurements of two [2] oil samples: Newtonian control oil sample and a commercially available multiweight motor oil, using a RH 2000 Dual Bore, Bench Top Capillary Rheometer with a die of dimensions 0.156 x 65 mm are presented in this study. A 69 MPa pressure transducer is used for all experiments in this study. The shear rates used in this study range from $\sim 50,000 - 2,000,000 s^{-1}$. Viscosity profiles of both oils at 100, 110, 120, 130, 140 and 150 °C are presented.

The viscosity profiles of the control oil show Newtonian behavior at all temperatures and shear rates measured. The commercial oil however, exhibits slight shear thinning at lower temperatures and Newtonian behavior at higher temperatures. Also, the viscosities of both samples decrease with increase in temperature. The dies used in this study are fitted in an in-house designed die holder with a heating sleeve heated to test temperatures in order to sustain uniform operating temperatures throughout the length of the dies.

Time temperature superposition (TTS) is used to estimate viscosity values of the two [2] oils at extremely high shear rates using the IRIS software. TTS shifting gives viscosity data at higher shear rates with reasonable accuracy while eliminating the upturn in viscosity at high shear rates due to turbulence. Viscosity profiles of respective oils at 100, 110, 120, 130 and 140 °C are shifted to superpose on to the viscosity profile at 150 °C to generate the master curve, which predicts the viscosity profile up to extremely high shear rates (~ 15 million s^{-1}).

3. Results and discussion

The reliability of any data always depends on the calibration and accurate measurement capabilities of the instrument. Therefore, proper calibration and verification of the perform-

ance of the instrument is necessary in order to generate accurate and reliable data. Figure 1 presents the viscosity profile of a NIST traceable Newtonian viscosity standard oil with nominal viscosity of 10 cP at 25 °C. The data verifies the accuracy of the measurements and demonstrates that the instrument is properly calibrated.

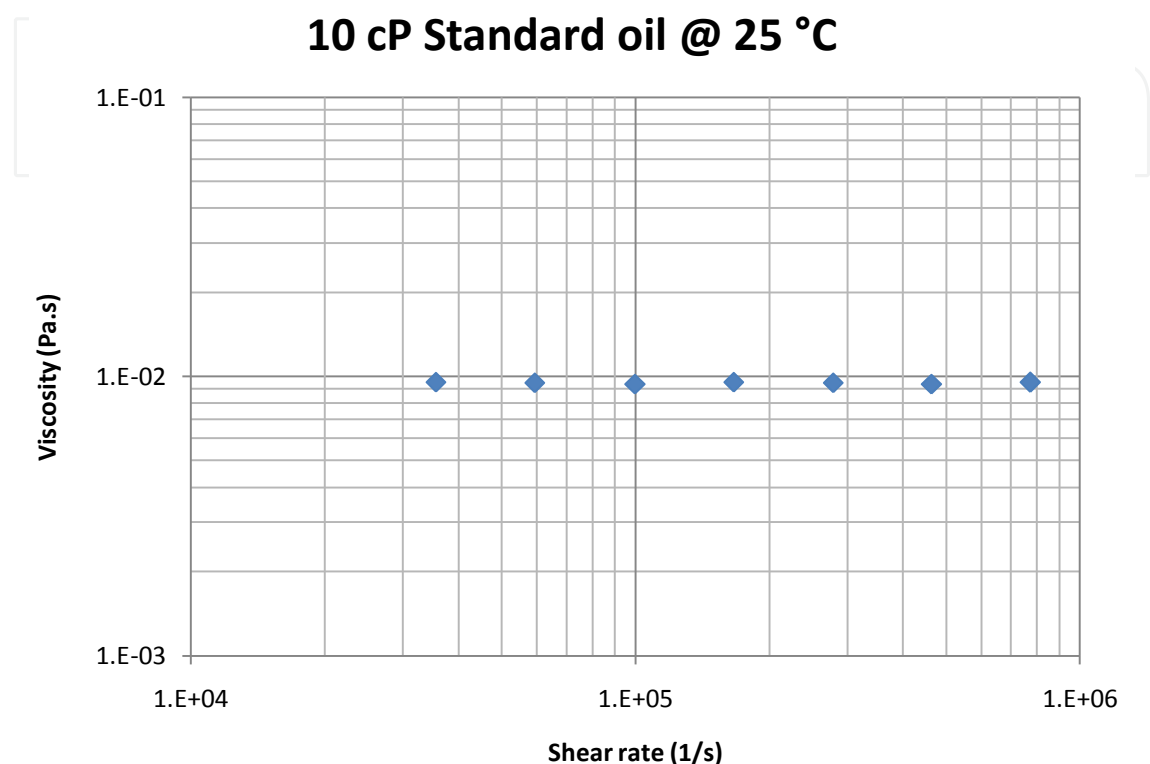


Figure 1. Steady state shear viscosity of 10 cP nominal viscosity standard oil at 25°C.

Figure 2 presents the viscosity profiles of a Newtonian oil at 100, 110, 120, 130, 140 and 150 °C. Data indicates that the oil is Newtonian in the range of shear rates and temperatures measured and is sensitive to temperature, i.e., viscosity decreases with increase in temperature. The high end shear rates measured experimentally are unique to this study. Viscosity measurements up to the high shear rates represented in this study are achievable by using a die with high L/D (length/diameter) ratio (> 400) using an in-house design. The die fits into an in-house designed die holder, which can be modified to fit any commercially available capillary rheometer. Commercially available capillary rheometers are generally not equipped to measure viscosities of low viscosity fluids at high shear rates. The temperature range used in this study is chosen due to the fact that flow behavior of the oils at temperatures below 100 °C indicates strong shear thinning at high shear rates. A shear thinning viscosity profile cannot be superposed on to a Newtonian viscosity profile to generate the master curve using the TTS technique because the relaxation and retardation mechanisms at play in shear thinning behavior are different from the mechanisms at play in Newtonian behavior. As mentioned above, only data that involves similar molecular rearrangement mechanisms can be used to conduct the time-temperature superposition.

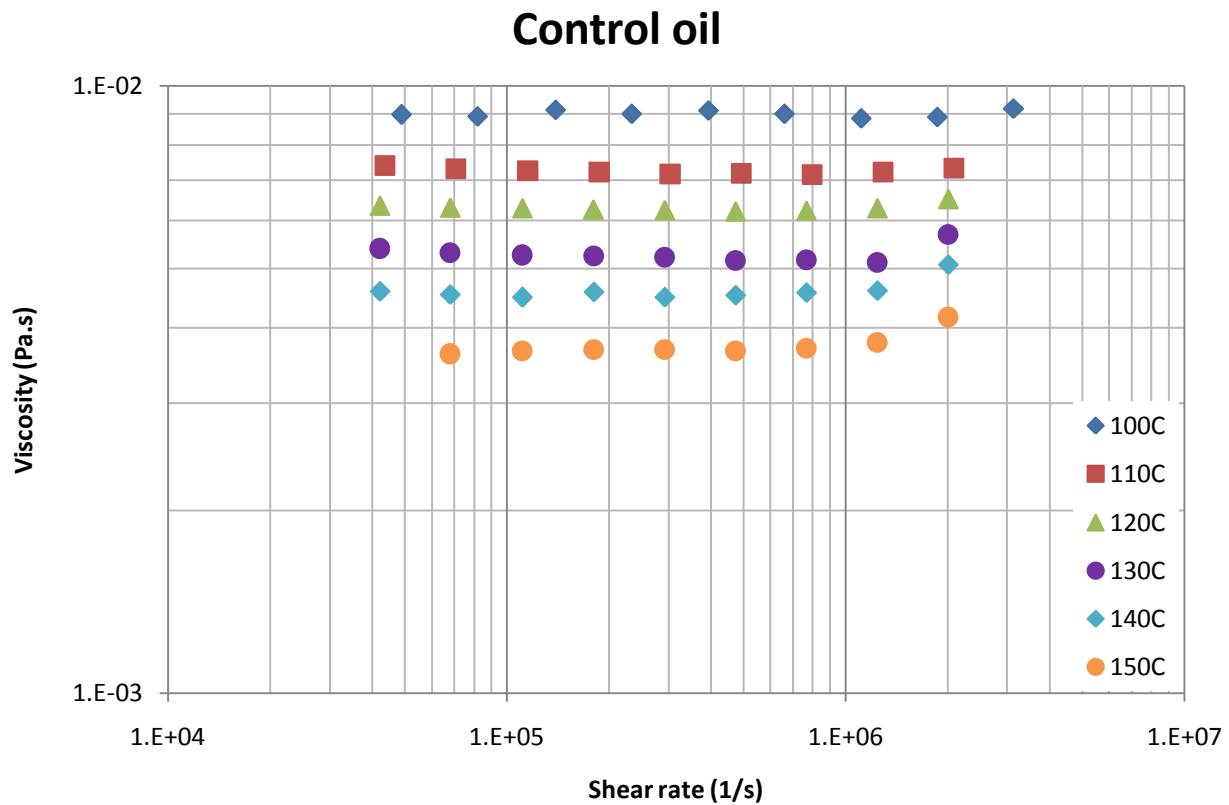


Figure 2. Steady state shear viscosity profiles of the control oil at 100, 110, 120, 130, 140 and 150 °C.

The commercial multiweight oil viscosity profiles in the temperature range of 100 – 150 °C at 10 °C increments are presented in Figure 3. Data indicates that this oil is slightly shear thinning up to 130 °C, but shows Newtonian behavior above this temperature. As expected, the oil viscosity decreases with increase in temperature. It is to be noted that in Figures 2 and 3, the viscosity profiles at 120 °C and above show an upturn at shear rates above 1,000,000 s⁻¹. This upturn is due to the onset of secondary flows (turbulence). Turbulent flow manifests itself as an upturn in viscosity due to additional resistance to flow. The use of the high L/D ratio dies pushes the onset of turbulence to shear rates of ~ 1,000,000 s⁻¹, which otherwise would set in at much lower shear rates leading to misleading viscosity values. This in turn would make it impossible to estimate viscosity values at high shear rates using TTS.

One of the applications of this study is in estimating the viscosity of motor oil in engine parts, where the oil undergoes extremely high shear rates. As there is no way of achieving true viscosity values experimentally while mimicking the shear rates present in the engine parts, the use of TTS to estimate the viscosity values at such high shear rates (~ 1,000,000 – 15,000,000 s⁻¹) is very useful. Also, the detrimental effects of turbulent flow in viscosity measurements at high shear rates can be eliminated by estimating the viscosity values using TTS.

Figure 4 presents the master curves obtained by superposing the viscosity profiles of the control sample oil and the commercial multiweight non-Newtonian oils (from Figures 2 & 3, respectively) at 100 – 140 °C on to that at 150 °C. As expected, estimated viscosity values using TTS at shear rates higher than those measured experimentally ($> 1,000,000 \text{ s}^{-1}$) show that the Newtonian oil is constant up to $\sim 15,000,000 \text{ s}^{-1}$ shear rate. However, the non-Newtonian oil exhibits slight shear thinning behavior, as would be expected from the experimental data. Figure 4 also compares the experimental data sets at 150 °C with respective master curves generated using TTS. It is demonstrated that the upturn in viscosity due to secondary flows is eliminated as well by using the TTS. The y -axis is plotted on a linear scale for clarity.

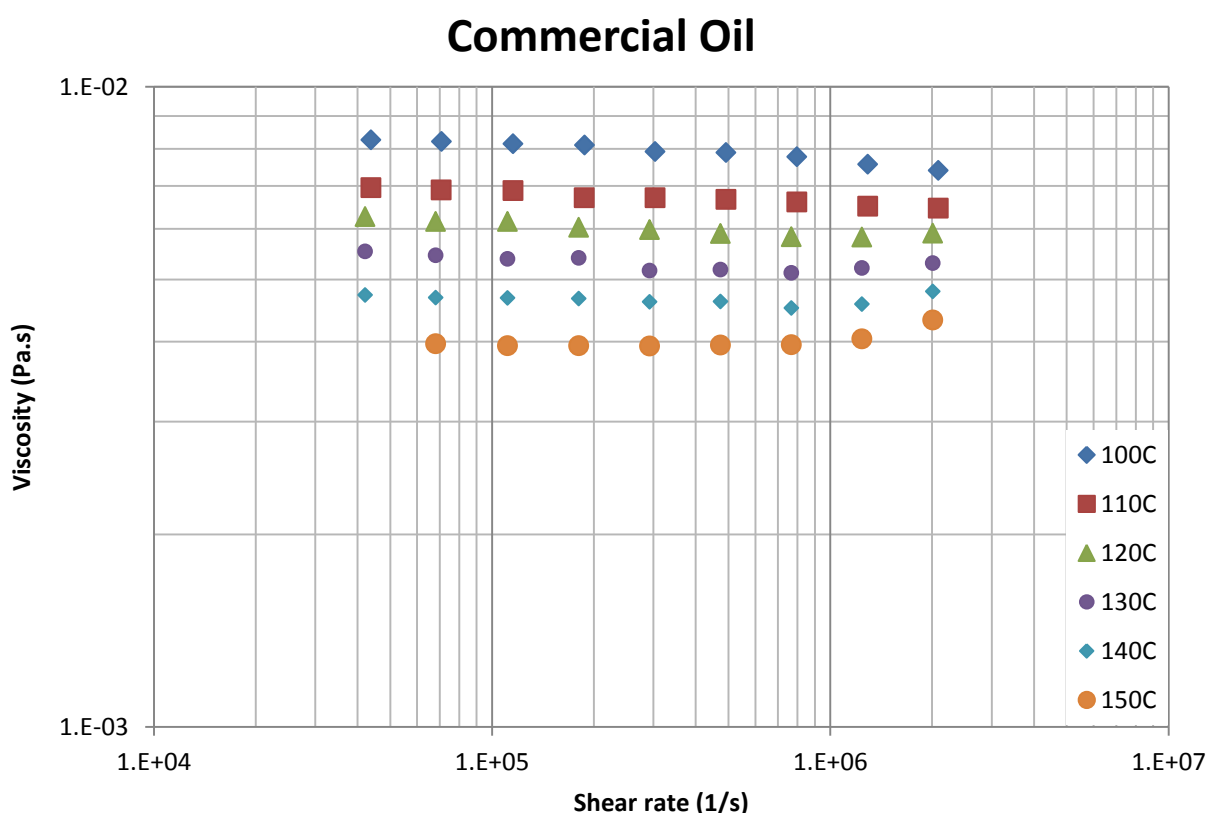


Figure 3. Steady state shear viscosity profiles of the commercial multiweight oil sample at 100, 110, 120, 130, 140 and 150 °C.

The transition from laminar to turbulent flow in capillary rheometry plays an important role in determining whether the viscosity values obtained experimentally are real. This transition can be determined by calculating the Reynolds numbers. Figure 5 presents the Reynolds numbers of the control oil as a function of pressure drop. The Reynolds numbers are calculated using the Fanning friction factor given in equation (4) [6]:

$$f = \frac{\Delta P}{L} \times \frac{D}{2Qv^2} \quad (4)$$

where ΔP is the pressure drop, D is the die diameter, L is the die length, ρ is the fluid density and v is the fluid velocity in the die. In order to check if the flow in the studied shear rate range is laminar, the Reynolds numbers are also calculated using the Hagen-Poiseuille equation given by equation (5):

$$f = \frac{16}{R_e} \text{ (Laminar flow)} \quad (5)$$

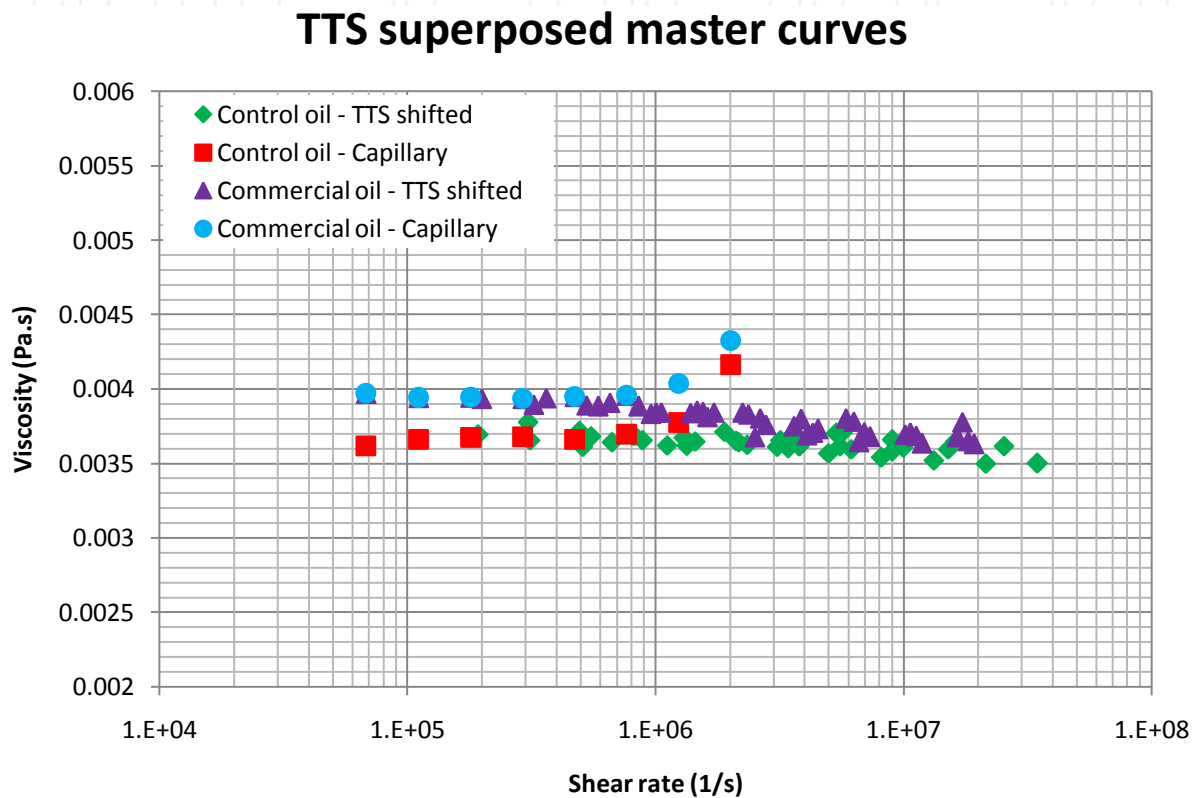


Figure 4. Estimated viscosity data of the control and commercial oils over a very large range of shear rates at 150 °C using TTS compared to original viscosity profile using capillary rheology.

For flow in the transition and turbulent regions, the Blasius formula given in equation (6) is employed:

$$f = \frac{0.079}{R_e^{\frac{1}{4}}} \text{ (Turbulent flow)} \quad (6)$$

Reynolds numbers for all samples are calculated using equations (5) and (6) to check if the flow is in the laminar region or the turbulent region. Reynolds number calculations suggest there is a transition in the flow pattern from laminar to turbulent flow in the Reynolds number range of 850 – 1000. The pressure drop data is corrected for kinetic energy contributions as well. From experience, the transition from laminar to turbulent flow commences when the graphs of laminar and turbulent flow start to converge, in this case around a Reynolds

number of ~ 850 . The onset of complete turbulent flow is assumed to set in approximately at the point where the laminar flow graph intersects the turbulent flow graph. The x -axis in the figure is plotted on a log scale for clarity.

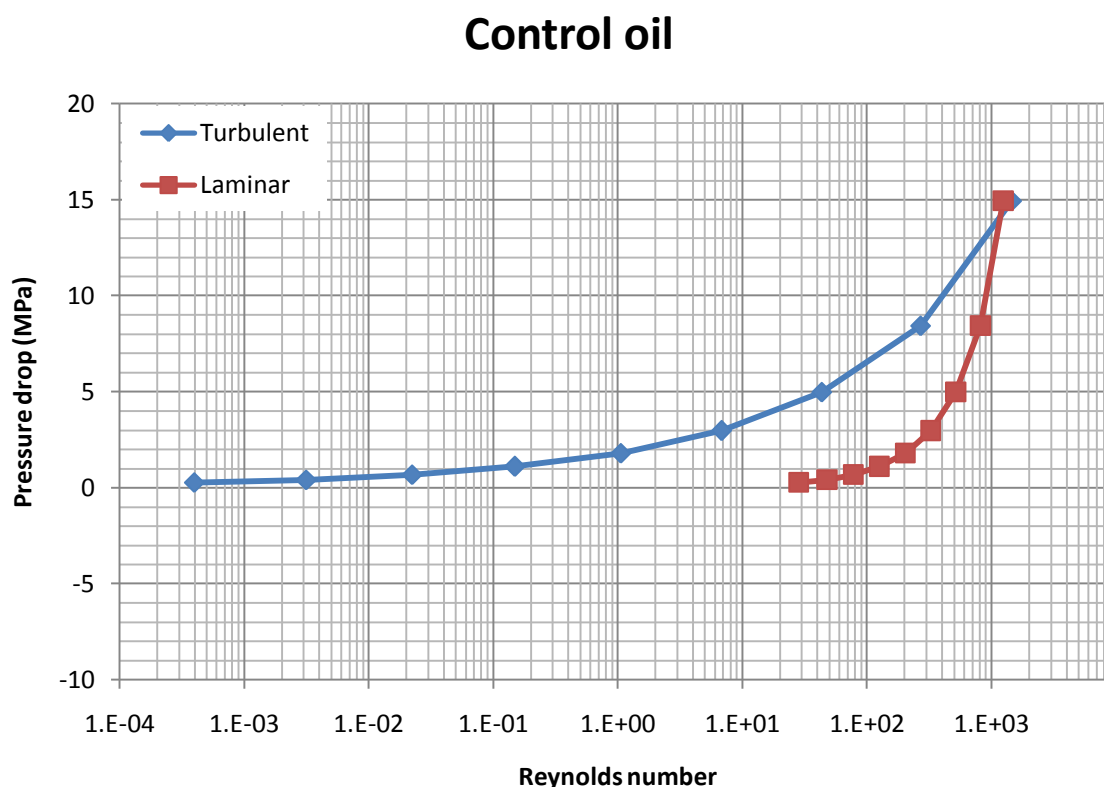


Figure 5. Reynolds numbers of the control oil as a function of pressure drop using equations (b) and (c).

Figure 6 shows the Reynolds number at the point of viscosity upturn for the control oil at high shear rates. This upturn in viscosity is attributed to the transition from laminar flow to turbulence in capillary flow for this configuration. For the purpose of this study, an increase in viscosity by $\sim 10\%$ is where the transition from laminar to turbulent flow is assumed to commence. The Reynolds number of ~ 820 presented in Figure 6 is calculated using equation (5), and the Reynolds number 1450 is calculated using equation (6).

In order to check for viscous heating effects in the capillary at high shear rates, the Nahme numbers (Na) for all the data points presented in this report are calculated. The Na number needs to be less than 1 for die viscous heating to be neglected. The Na number can be calculated by the following equation (7): where β is the temperature sensitivity of viscosity, η is the viscosity of the fluid, γ is the shear rate, R is the radius of the die, and k is the thermal conductivity of the fluid.

$$Na = \frac{\beta \eta \gamma R^2}{4k} \quad (7)$$

All Na values except at shear rates above $1,000,000 \text{ s}^{-1}$ were lower than 1 for both oils, thereby indicating that viscous heating effects can be neglected up to shear rates of $1,000,000 \text{ s}^{-1}$. Data above $1,000,000 \text{ s}^{-1}$ shear rate at any temperature is not used for TTS shifting in this study. It should be noted that the Na approaches 1 at $1,000,000 \text{ s}^{-1}$ at $100 \text{ }^\circ\text{C}$. Samples with higher viscosity and/or at lower temperatures will have Na higher than 1 below $1,000,000 \text{ s}^{-1}$ shear rates, in which case, viscous heating effects would need to be taken into account while calculating the real viscosities of such samples.

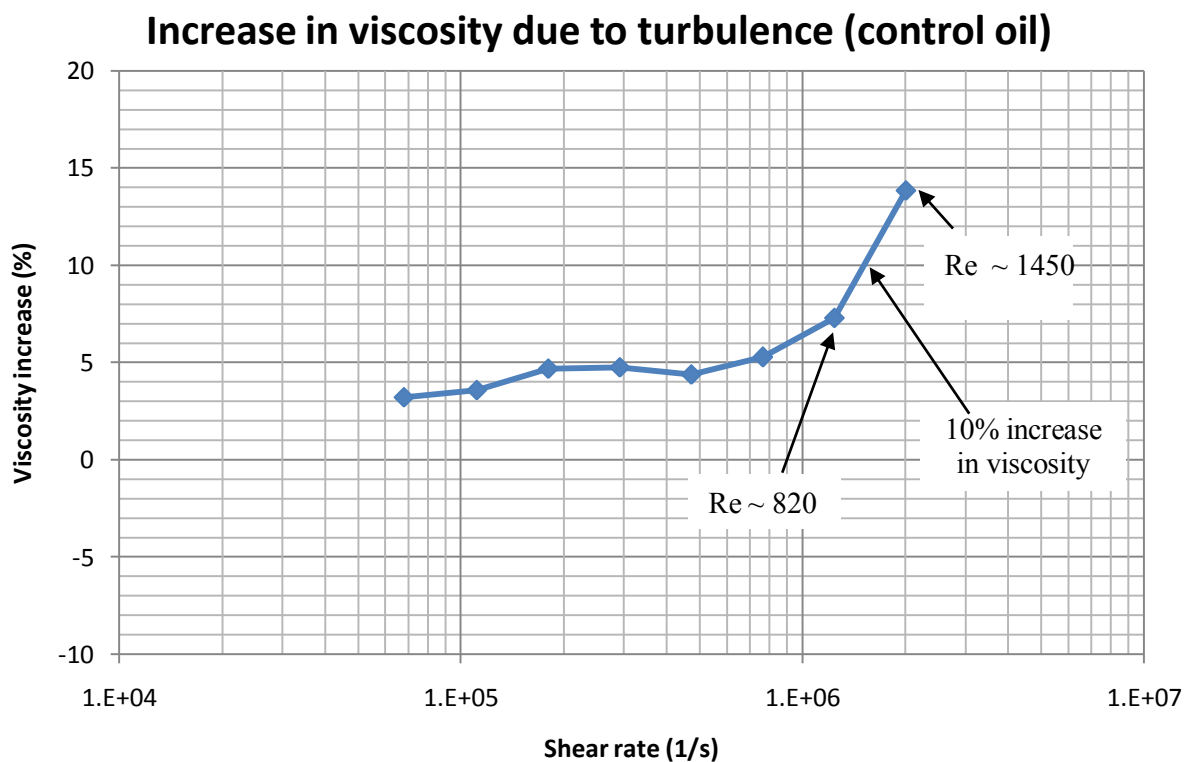


Figure 6. Increase in viscosity (percent) of the control oil due to onset of turbulence at $150 \text{ }^\circ\text{C}$.

4. Conclusions

This study emphasizes the importance and explains the applications of high shear rate viscosity measurements. A novel technique to measure low viscosity fluids at high shear rates ($\sim 50,000 - 2,000,000 \text{ s}^{-1}$) is developed using capillary rheology. The difficulty to achieve reliable viscosity measurements at shear rates above $1,000,000 \text{ s}^{-1}$ is addressed and a method (time-temperature superposition) to estimate the viscosity behavior of low viscosity fluids with reasonable accuracy up to extremely high shear rates ($\sim 15,000,000 \text{ s}^{-1}$) is established. It is demonstrated that the time-temperature superposition (TTS) technique also alleviates the

problem of errant viscosity numbers at high shear rates ($> 1,000,000 \text{ s}^{-1}$) due to onset of turbulent flow. This study is expected to have a prolific impact on low viscosity - high shear rate applications such as oil flowing through engine parts, paint spray guns, petroleum pipelines, among other applications in various industries.

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