DOI:10.1190/segam2013-0250.1

Shear modulus of heavy oils: Measuring at Low Frequencies

P. Rodrigues and M. Batzle, Colorado School of Mines

Summary

An alternative method used to measure the shear modulus of heavy oils at low frequencies is the rheometer. Typical rheometers are oscillating plate devises commonly used to ascertain rapidly the shear properties of liquids and polymers. However, many question how comparable are these measurements to what has been traditionally done at ultrasonic or with stress/strain measurements at intermediate frequencies. Until now no one had directly compared results done by the three techniques under similar conditions. We performed ultrasonic, stress/strain and rheometer measurements on a heavy oil sample. Through proper calibration, the shear modulus of the heavy oil from the three techniques was matched using the Cole-Cole model. The experiments covered a frequency range from 0.01 Hz to 0.8 MHz. These experiments are the first evidence of consistency between the three techniques to measure the modulus of heavy oils at different frequencies. This work unveils many aspects to consider while doing rheometer experiments to ensure results are meaningful for rock physics applications.

Introduction

Heavy oils constitute one of the largest contributors to the hydrocarbons reserves of the world, particularly in the Western hemisphere. Due to their high viscosity, heavy oil reservoirs often require some kind of Enhanced Oil Recovery (EOR) method to improve recovery. Efficiency of exploitation techniques and EOR methods are tied to a detailed reservoir characterization and monitoring which often utilize 3D and time-lapse seismic techniques. The success of seismic monitoring techniques is largely based on the ability of geophysicists to accurately model the waves propagation through real and potential rock-fluid scenarios encountered during EOR operations. It is imperative therefore, to translate production scenarios into changes in the elastic properties that govern wave propagation (density, bulk and shear modulus). Translating these scenarios into elastic properties is the goal of rock physics models, which estimate the elastic properties of the rock-fluid system from its components. Components are usually divided in rock and fluids components. Regardless of how the rock physics models divide its components, all are based on the assumption that the properties of the individual components are well known. This last assumption is far from the truth in the case of heavy oils.

True "fluids" are not capable of supporting shear stress; however, heavy oils (API < 20), and all their heavier relatives (API <10), behave acoustically different than the rest of the fluids in the reservoir system. Below a certain temperature or above a certain frequency, which varies by sample, heavy oils behave acoustically as a viscoelastic material and can support a shear wave. *Figure 1* from Han et al. (2007) shows a schematic representation of properties of an extra-heavy oil at ultrasonic frequencies. The more important aspect of this figure is that below certain temperature, the heavy oil supports a shear wave.

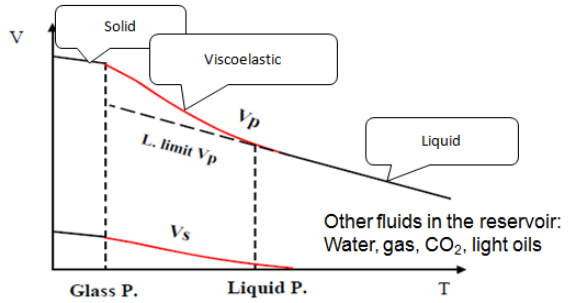

Figure 1 - Schematic representation of Vp and Vs measurements on an extra-heavy oil sample at ultrasonic frequency vs. temperature. Below the liquid point temperature a shear wave can be measured (Han et al. 2007)

Geophysicists, analyzing seismic data in heavy oil reservoirs, must face the fact that heavy oils can propagate a shear wave and consider it in their interpretation. Authors such as Ke, et al. (2010), Ciz and Shapiro (2007), Makarynska et al. (2010) and Gurevich et al. (2008) have developed rock physics models to include the viscoelastic behavior of heavy oils in their estimation of the elastic properties. Nonetheless, a large weakness in those models is the uncertainty of the magnitude of the shear modulus of heavy oils. Moreover, the question is open on how we can measure it. Wolf, et al. in 2008 raised that concern, emphasizing the authors limitation to model the shear wave due to the lack of appropriate rock physics models, and moreover the lack of experimental data to calibrate them. This constitutes a definite gap in rock physics models that estimate the elastic properties of heavy oils. The focus of this work is to provide geophysicists, with a reliable technique to measure the shear modulus of heavy oils at low frequency.

Theory

It has been demonstrated (Batzle, et al. 2004; Hasan 2010) that the shear modulus of heavy oils is highly dependent on frequency. Frequency dependency adds a major difficulty to the problem since the data we study are obtained at different frequencies. Laboratory acoustic experiments are usually done at ultrasonic frequencies (in the order of MHz) and seismic data are collected in the range of 10-100

DOI http://dx.doi.org/10.1190/segam2013-0250.1

Hz, Vertical Seismic Profiles (VSP) at ~30-120 Hz and sonic logs at 10-30 kHz. To provide a complete picture of the shear modulus of heavy oils, we need to account for the properties at different frequencies. Frequency dependency of the shear modulus of heavy oils, also called dispersion, has been measured (Batzle et al. 2006b) but lacks extensive characterization due to the difficulty of measuring shear properties at different frequencies in the lab. *Figure 2* shows the shear modulus of an extra-heavy oil (data from Batzle et al. 2006b) by two different techniques, ultrasonic and stress/strain at low frequencies. The lines represent the Cole-Cole model fit to describe the frequency dispersion.

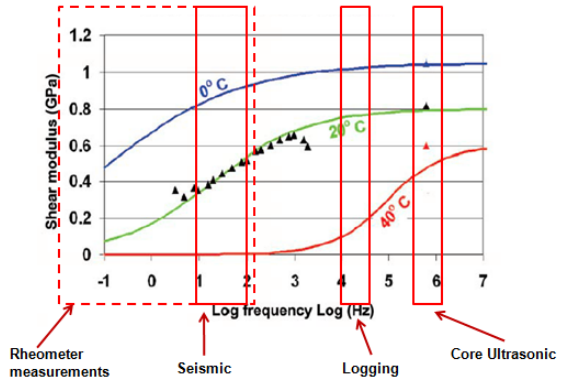

Figure 2 - Measured (triangles) shear modulus in the extra-heavy oil (API: -5) from ultrasonic and stress/strain measurements. Solid lines are from Cole-Cole model. Boxes indicate the frequency range of different geophysical techniques (modified from Batzle, et al. 2006a). Rheometer is a good alternative to measure the shear modulus at low frequency.

Measuring elastic properties at high frequencies has been done for many years and it is less complicated than measuring the same properties at low frequencies. Low frequency measurements of bulk heavy oils bring many complications. One technique used at Colorado School of Mines consists of a stress/strain system that deforms or compresses the sample at a frequency range from 3 to 3000 Hz. The equipment works well for solid or solid-like samples but cannot be used for liquid-like samples. An alternative method used to measure the shear modulus of heavy oils at low frequencies is the rheometer. Rheometers are common tools in chemical engineering and constitute a convenient way of measuring shear modulus of oil samples. The equipment is widely available and much data has been published. In Figure 2, red boxes show the frequency at which different geophysical measurements are acquired in the field. The dashed line box represents the range of frequencies that can be acquired using the rheometer.

The rheometer can measure the shear modulus in a frequency range of 0.01 to 100 Hz, providing insight into the low end of the range. Several authors such as Hinkle et al. (2008), Rojas et al. (2008), Hasan (2010), Bazyleva et al. (2010), and Behura et al. (2007) have measured the shear modulus of heavy oils with the rheometer. Although these authors have been successful in making the measurements, they have not provided insight on how comparable these measurements are to what has been measured at ultrasonic frequencies and with stress/strain measurements at seismic applications frequencies simultaneously. The lack of verification of the rheometer as an adequate technique to measure the shear modulus of heavy oils at low frequencies has greatly impeded its use.

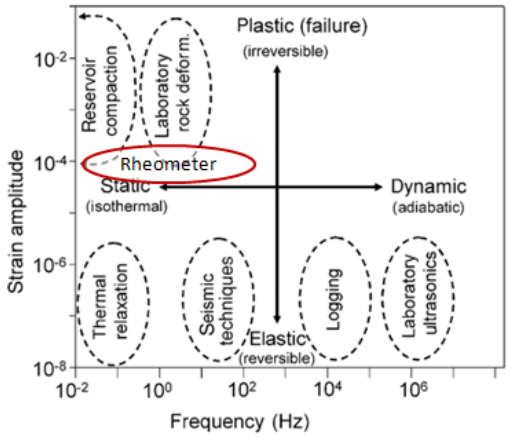

Figure 3 - Frequency and strain range of the different geophysical and geomechanical techniques. Red ellipse indicates the range of strain/frequency of the rheometer measurements. Strain amplitudes measured with the rheometer are higher (10^{-4}) than used at seismic/logging and other techniques (10^{-7}) (Batzle et al. 2006a)

An important aspect to consider when making rheometer measurements and the center of many questions about its validity can be explained in Figure 3 (from Batzle et al. 2006a). Geophysical measurements and traditional laboratory techniques, measure the elastic properties with strains amplitudes in the order of 10^{-7}, rheometer measurements are performed at strains in the order of 10^{-4}. Figure 3 shows that rheometer measurements are in between the range of elastic (reversible) and plastic (irreversible) deformation. When strains amplitudes are too high a critical strain is reached and the material structure is broken leading to unreliable measurements of the elastic properties.

A viscoelastic material will have a complex modulus consisting of both real (G') of "storage" and imaginary (G") or "loss" components. For a linear viscoelastic material the relation between stress and strain depends only on frequency and not on stress or strain magnitude (Ferry 1980). Real materials behave linearly depending, not only on the sample itself but also on the magnitude of the strain

DOI http://dx.doi.org/10.1190/segam2013-0250.1

DOI:10.1190/segam2013-0250.1

they are subjected to. In this work, strain amplitudes are tested and selected to ensure that the experiments are performed within the Linear Viscoelastic Regime (LVR). The LVR is the region in which the storage modulus stays constant with increased strains. The common behavior of the storage modulus in heavy oils is to decrease as the strain level goes beyond the critical strain in a process called strain thinning (Figure 4, modified from Hyuan et al. 2004). For strain amplitudes greater than the critical strain (γ_0), a rupture in the structure of the fluid occurs and the shear modulus decreases. Molecules in heavy oils form large aggregates due to association between components with large polarity (asphaltenes and resins). Under large strains these structures can break into smaller structures resulting in a decrease in the shear modulus.

For this reason, rheometer measurements are performed at strain levels that ensure the LVR is maintained during the length of the entire experiment (different temperatures and frequencies). This usually requires selecting the lowest strain possible that the equipment allows. When doing this, it is assumed that at lower strains, less than 10^{-4}, the same LVR persists. This assumption becomes the focus of this research. In contrast, ultrasonic and stress/strain measurements are performed at strain magnitudes in the order of 10^{-7}. If the rheometer measurements are done within the LVR, and the LVR is the same at the two different strain scales, there should be consistency between the three techniques (Figure 4).

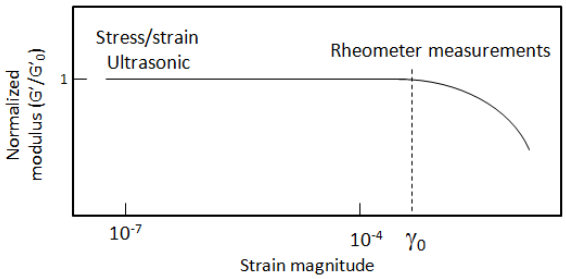

Figure 4 - Schematic representation of normalized shear modulus vs. shear strain for a shear thinning material. Above the critical strain (γ_0) the shear modulus decreases. Below the critical strain the shear modulus remains constant and this region is called the Linear Viscoelastic Regime (LVR). It is expected that the LVR covers several orders of magnitude and allows consistency between different techniques (modified from Hyun et al. 2002).

Experiments

Low frequency rheology experiments were performed in two similar ARG2 systems from TA instruments using the same sample used in the ultrasonic and stress/strain experiments by Batzle et al. (2006b). Two instruments, rheometers A and B have the same specifications and differences between them are due to periodic maintenance and calibration which are not well documented. Results shown are for an extra-heavy oil (-5 API) which is a solid-like material at the experimental temperature. This condition causes many problems during rheologic experiments performed using parallel plates. We had to perform many experiments until the appropriate settings were found for this sample. Here we will describe a subset of the experiments representative of the main learning points.

Many of the first experiments performed had issues with the experimental setting. Among the main important steps taken into consideration to ensure adequate settings were: use of zero normal force control to ensure the sample is relaxed during the experiment and to eliminate the energy effect of the compressive force coming from the upper plate (Qiu et al. 2011). Several strain sweeps were performed to ensure the measurements were kept in the LVR during the length of the experiment. We use the smaller "geometry" or end plate available (8 mm parallel plates). These smaller plates are more appropriate for stiff samples. Quality control was specially done comparing the raw vs. corrected phase calculated by the rheometer. The difference between the two should be small for high viscosity samples to be considered good quality results.

Results

After issues with the experimental settings were resolved, the comparison of the raw vs. corrected phase was very good and data considered reliable. The first test done under optimal conditions is shown as red diamonds in Figure 5. These data were obtained with Rheometer A using a 1 mm gap. As it can be seen, the data shows good quality but the results are shear modulus amplitude is lower than expected by the Cole-Cole model. The same experiment was repeated reducing the gap to 0.5 mm (yellow squares in figure 5), the amplitude is higher but still not the expected from the Cole-Cole model.

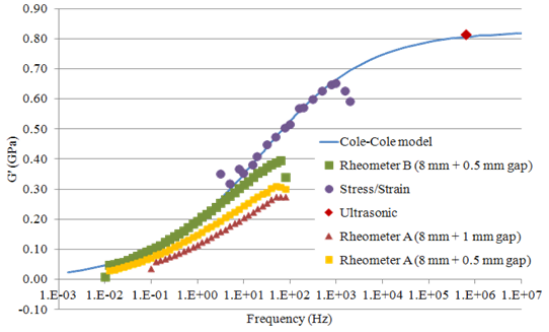

Figure 5 - Comparison of ultrasonic, stress/strain and storage modulus using Rheometer B and the 8 mm plate geometry. Gap between parallel plates = 0.5 mm. When changing the rheometer used for the measurement the data matches the expected behavior form the Cole-Cole fit.

DOI http://dx.doi.org/10.1190/segam2013-0250.1

DOI:10.1190/segam2013-0250.1

Finally, the same experiment, using 0.5 mm gap, was repeated in Rheometer B obtaining the green squares in the figure 5. This last result shows consistency with what is expected from the Cole-Cole model. Mismatch at frequencies above 30 Hz are probably due to the system reaching its performance limit as the sample becomes stiffer at higher frequencies. It is also good to point out that this work assumes that the strain/stress and ultrasonic tests are of good quality and that the Cole-Cole model is sufficient to describe the observed dispersion within the studied frequency range.

Understanding the discrepancies seen between the three experiments is key to assess the validity of the results. The main driver of the discrepancies lies with the nature of the sample. The sample at the experimental temperature is at a solid-like state below the glass point and has a shear modulus in the order of 10^8 Pa. This sample is highly sensitive to temperature and film thickness, these two variables are driving the discrepancies observed in the 8 mm plate experiments.

Temperature effects

Heavy oils and asphalts are well known for their high sensitivity to temperature. The heat source in rheometer experiments comes only from the lower plate which inevitable creates a temperature gradient within the sample.

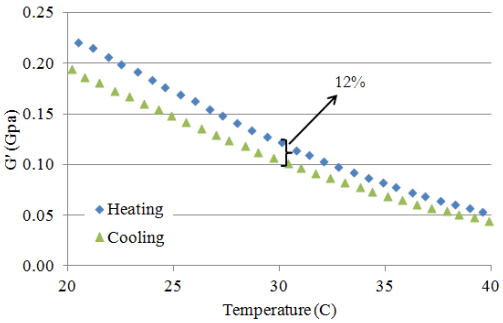

Figure 6 – Temperature ramps between 20 and 40 °C indicating a 12% differences in storage modulus at 30°C between the heating and cooling cycles at 1 Hz.

In addition, conditioning time is important to achieve an uniform temperature before performing the experiment. Petersen et al. (1994) reports variations of up to 40% in the modulus due to conditioning time. We assessed the effect of temperature changes in our sample using two different approaches. The first approach was through temperature ramps experiments. Figure 6 shows the detail of the experiment below 40 °C, a heating lag can be identified because the two curves separate. This lag occurs due to the diminished thermal conductivity of the sample at lower temperatures. This lag results in a variation of the shear modulus of 12% at 30 °C.

The second approach studied the change in modulus with temperature performed at a fixed frequency of 1.25 Hz. Results indicate that the sample experienced a change in modulus in the order of 7% per °C. Discrepancies observed between the two rheometers could be due to temperature calibration or conditioning time of the sample between experiments.

Film thickness (Gap effect)

The other main difference observed in the results was the effect of the gap or sample/film thickness. While the system was at 0.5 mm gap, we decreased the gap further and repeated the measurements to assess the effect of the film thickness. We compared these results with the measurement done at 1 mm gap. At a fixed frequency, the modulus first increases when reducing the gap and then, the modulus decreases again. A similar behavior was described by Zhai et al. (2000) in which the asphalt film stiffness increased as the thickness decreased until an optimal value was reached. Thereafter the stiffness will decrease as the gap thickness decreases. This phenomenon was attributed to the change in the orientation of the molecules during the application of the shear force Qiu et al. (2011).

Conclusions

This work confirms rheometer measurements can be a reliable tool to measure the shear modulus of heavy oils for rock physics applications. The data obtained confirms consistency between low frequency measurements done in the rheometer with higher frequency measurements done with stress/strain and ultrasonic equipments. Even though the comparison was successful, it is important to recognize the sensitivity of the measurements to experimental design and take them into account when doing the tests. In this work we identified the LVR, normal force control, geometry and the temperature as the main aspects to consider during the tests. Additionally, a detailed quality control of the data is fundamental to validate results. Review of the corrected vs. raw phase changes with frequency and temperature is an excellent tool to identify potential problems in the data.

Acknowledgments

We would like to acknowledge the contribution of Dr. Ala Batzyleva of the Dept. of Chemical Engineering for performing an important part of the experiments leading to these results and for many productive discussions. We would also like to thank Dr. Matt Liberatore from the Dept. of Chemical Engineering for allowing us to use his laboratory facilities. We also want to thank the Fluids/DHI and OCLASSH Consortiums at Colorado School of Mines for financial support.

DOI http://dx.doi.org/10.1190/segam2013-0250.1

http://dx.doi.org/10.1190/segam2013-0250.1

EDITED REFERENCES

Note: This reference list is a copy-edited version of the reference list submitted by the author. Reference lists for the 2013 SEG Technical Program Expanded Abstracts have been copy edited so that references provided with the online metadata for each paper will achieve a high degree of linking to cited sources that appear on the Web.

REFERENCES

Batzle, M., D. H. Han, and R. Hofman, 2006a, Fluid mobility and frequency-dependent seismic velocity — Direct measurements: Geophysics, **71**, no. 1, N1–N9, http://dx.doi.org/10.1190/1.2159053.

Batzle, M., R. Hofman, and D. H. Han, 2006b, Heavy oils — Seismic properties: The Leading Edge, **25**, 750–756, http://dx.doi.org/10.1190/1.2210074.

Batzle, M., B. Zadler, and R. Hofman, 2004, Heavy oils — Seismic properties: Presented at the 74th Annual International Meeting, SEG.

Bazyleva, A. B., A. Hasan, M. Fulem, M. Becerra, and J. M. Shaw, 2010, Bitumen and heavy oil rheological properties: Reconciliation with viscosity measurements: Journal of Chemical & Engineering Data, **55**, no. 3, 1389–1397, http://dx.doi.org/10.1021/je900562u.

Behura, J., M. Batzle, R. Hofman, and J. Dorgan, 2007, Heavy oils: Their shear story: Geophysics, **72**, no. 5, E175–E183, http://dx.doi.org/10.1190/1.2756600.

Ciz, R., and S. A. Shapiro, 2007, Generalization of Gassmann Equations for porous media saturated with a solid material: Geophysics, **72**, no. 6, A75–A79, http://dx.doi.org/10.1190/1.2772400.

Ferry, J. D., 1980, Viscoelastic properties of polymers: John Wiley & Sons, Inc.

Gurevich, B., K. Osypov, R. Ciz, and D. Makarynska, 2008, Modeling elastic wave velocities and attenuation in rocks saturated with heavy oil: Geophysics, **73**, no. 4, E115–E122, http://dx.doi.org/10.1190/1.2940341.

Han, D. H., J. Liu, and M. Batzle, 2007a, Shear velocity as the function of frequency in heavy oils: 77th Annual International Meeting, SEG, Expanded Abstracts, 1716–1719.

Hasan, A., 2010, Rheological behavior and nanomicrostructure of complex fluids: Biomedical and bitumen-heavy oil applications: Ph.D., University of Alberta.

Hinkle, A., E. Shin, M. Liberatore, A. Herring, and M. Batzle, 2008, Correlating the chemical and physical properties of a set of heavy oils from around the world: Fuel, **87**, no. 13–14, 3065–3070, http://dx.doi.org/10.1016/j.fuel.2008.04.018.

Hyun, K., S. H. Kim, K. H. Ahn, and S. J. Lee, 2002, Large amplitude oscillatory shear as a way to classify the complex fluids: Journal of Non-Newtonian Fluid Mechanics, **107**, no. 1–3, 51–65, http://dx.doi.org/10.1016/S0377-0257(02)00141-6.

Ke, G., H. Dong, and M. Johnston, 2010, Rock physics modeling of the frequency dispersion in bitumen saturated sands: 80th Annual International Meeting, SEG, Expanded Abstracts, 2552–2556.

Makarynska, D., B. Gurevich, J. Behura, and M. Batzle, 2010, Fluid substitution in rocks saturated with viscoelastic fluids: Geophysics, **75**, no. 2, E115–E122, http://dx.doi.org/10.1190/1.3360313.

Petersen, J., R. Robertson, J. Branthaver, P. Harnsberger, J. Duvall, S. Kim, and D. Anderson, 1994, Binder characterization and evaluation: Test Methods: Strategic Highway Research Program, National Research Council.

Qiu, J., M. F. C. van de Ven, S. P. Wu, J. Y. Yu, and A. A. A. Molenaar, 2011, Investigating self-healing behavior of pure bitumen using dynamic shear rheometer: Fuel, **90**, no. 8, 2710–2720, http://dx.doi.org/10.1016/j.fuel.2011.03.016.

Rojas, M., J. Castagna, R. Krishnamoorti, D. H. Han, and A. Tutuncu, 2008, Shear thinning behavior of heavy oil samples: Laboratory Measurements and Modeling: Presented at the 78th Annual International Meeting, SEG.

Wolf, K., T. Vanorio, and G. Mavko, 2008, Measuring and monitoring heavy-oil reservoir properties: The Leading Edge, **27**, 1138–1147, http://dx.doi.org/10.1190/1.2978976.

Zhai, H., H. Bahia, and S. Erickson. 2000, Effect of film thickness on rheological behavior of asphalt binders: Transport Res Rec 1728 Paper No. 00-1373: 7–14.