Wall shear measurement based on ultrasonic velocimetry for process in-line rheometry

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In this paper we demonstrate that the ultrasonic velocimetry in pipe flow is a tool which can be used to measure quantitatively in-line rheological properties of polyurethane based adhesives and sealants. In such concentrated suspension, the velocity profile is only measurable in the first few millimeters at the wall of the pipe due to the high acoustic attenuation. However, we show that the wall shear rate can be quantitatively measured by using a new velocity estimation method based on the analysis of the spectral data across different depths. The resulting profiles in the wall region were not visibly affected by changes of the ultrasonic sample volume (pulse length, frequency) and allow the determination of rheological parameters of the adhesive over a large range of volume flow rates.

Keywords: in-line rheometry, shear thinning suspensions, ultrasonic velocimetry, pipe flow

1 Introduction

In many industries such as chemical, food, pharmaceutical and cosmetics the rheology is an important part of the product quality. At the same time the rheological properties are often difficult and time consuming to measure, especially for complex fluids. For Sika, producing all kinds of construction chemicals, it is important to be able to characterize the rheological properties of the products [13]. The possibility to measure the shear thinning behavior of a product directly in-line in a continuous production process would have further advantages. Dealing with highly filled, opaque suspensions such as polyurethane based adhesives and sealants, the ultrasound velocimetry based rheometer concept [1–4, 11, 12, 14, 15, 19, 20, 22] is an interesting option. The method is based on the measurement of the velocity profile in steady and laminar pipe flow and often pressure drop. With this information it is possible to obtain the viscosity by either fitting to a fluid model (e.g. power law or Herschel Bulkley) or by using directly the velocity gradient as local shear rate combined with the local shear stress derived from the pressure drop (known as gradient or point wise method).

First measurements in different types of adhesives showed that velocity profile measurements are possible but the penetration depth is limited to at most a few millimeters due to the high attenuation. Therefore we tried to optimize ultrasound transducer and signal processing for the measurement of the velocity profile close to the wall, as this contains also important rheometric information. For industrial applications it is important to have a quantitative measurement of the rheology and therefore the influence of the ultrasonic pulse parameters, such as length and frequency, were varied to investigate their effect on the velocity profile and the derived rheological properties.

2 Material and methods

2.1 Suspension

The adhesive studied in this paper is a filled polyurethane with high adhesive strength. It belongs to a class of adhesives used for making permanent elastic seals of different types of substrate materials such as wood, metal, metal primer, ceramic material, plastic and can be used to assemble windows on their frames either for the construction or for the car industry. These products must have features to suit the customers' expectations: sealing properties, strength, color, curing kinetic but should also have special rheological properties (viscosity, yield stress) for their correct application. Consequently they are the result of a complex formulation work. High strength polyurethane sealing products are composed of: (i) polyurethane (20 – 30 w%) responsible for the adhesive and reactive properties of the paste, (ii) fillers (20 – 40 w%), that, depending on the system, consist in chalk, kaolin, titan dioxide, alumino-silicates or carbon black and are used to increase the viscosity of the paste and its final strength, (iii) other materials (10 – 30 w%) which can be plasticizers, stabilizers and catalysts [5].

2.2 Flow setup

The material was pumped by a dosing unit as it is used for the robotic application of the material e.g. to windscreens in the automotive industry. The dosing unit itself has a capacity of 614 ml and is supplied with another pump directly connected to a 200 L barrel. With the used adhesive the volume flow rate can be varied between 1 and 40 ml/s thus the phases of constant flow (corresponding to one filling of the dosing unit) are between 10 minutes and 15 seconds respectively.

The flow cell for the ultrasonic measurement was attached to the end of a temperature controlled tubing of a total length of 15 m. The pipe diameter in the flow cell was 25 mm. The cell was also equipped with two temperature and two pressure sensors.

2.3 Electronics and transducer

We used an ultrasound apparatus developed in the Microelectronics Systems Design Laboratory of the University of Florence [16] that allows the control of several transmission and reception parameters. The system was connected to a home-made 5.5 MHz pencil transducer featuring a 80 % -3 dB bandwidth and a 1.2 mm beam-width in the focus region. The transducer was excited with frequencies between 2 and 8 MHz and pulse width between 1 and 32 cycles. The data received from the transducer in each pulse repetition interval were sampled at 64 MS/s, demodulated, decimated to up to 16 MS/s (corresponding to a maximum gate resolution in water (c = 1480 m/s) of 46 µm), and finally saved in a file for post processing.

2.4 Signal processing

The convolution of sample volume and the actual flow profile [9, 10] results in a significant distortion of the measured velocity profile towards the wall. Literature describes mainly two different strategies to recover the actual profile: (i) deconvolution in the frequency domain [7–10] or (ii) use of the envelope profile [17, 18]. The first method requires knowledge of the sample volume which would be difficult to obtain in our highly attenuating suspension. While the envelope profile is

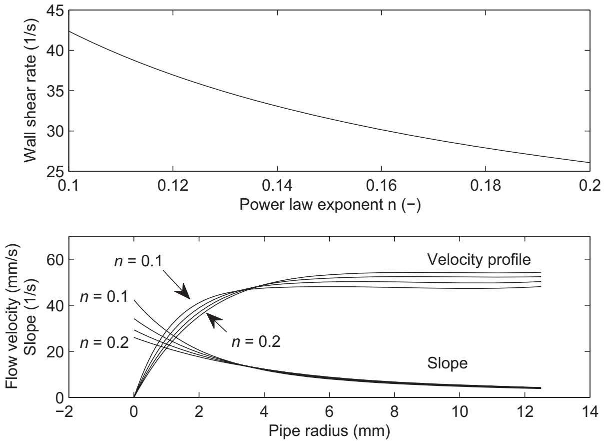

Figure 1: Theoretical calculations for power law fluids with n ranging from 0.1 to 0.2. Top: wall shear rate in function of n, bottom: velocity profiles and corresponding slopes.

easier to calculate as it only requires one correction factor describing the transducer characteristics, this method has a reduced accuracy due to the high attenuation. In this work we tested an alternative profile estimation method which was successfully used for the wall shear rate determination without requiring a sample volume dependent correction factor.

2.5 Rheometry

For most of the measured velocity profiles the available information covers only 5 to 10 % of the pipe radius. Altough the measured fluid is highly shear thinning, the onset of the velocity plateau in the central part of the pipe is not always visible.

Figure 1 shows some theoretical pipe flow calculations for a power law fluid for which viscosity η is a function of shear rate $\dot{\gamma}$, the coefficient K and the exponent n: $\eta = K\dot{\gamma}^{n-1}$. For the calculations the exponent n was varied from 0.1 to 0.2, the pipe radius R = 12.5 mm and the flow rate Q = 20 ml/s. Those parameters are typical for the presented measurements. For the power law fluid the wall shear rate $\dot{\gamma}_w$ can be calculated using:

$$\dot{\gamma}_w = \frac{3n+1}{4n}\frac{4Q}{\pi R^3} \qquad (1)$$

The resulting $\dot{\gamma}_w$ varies from 42 to 26 s^{-1} for n equal 0.1 and 0.2 respectively. Thus $\dot{\gamma}_w$ has the potential to be used for characterization of the shear thinning behavior of the fluid. The second plot of figure 1 shows the resulting velocity profiles and in addition the slope of the velocity profile calculated from the wall to the radial position. This indicates the shear rate can be determined from a linear interpolation in the corresponding region. It also shows that it would be ideal to be able to measure the shear rate in the first millimeter from the wall as this results in the most important differences.

3 Results and discussion

3.1 Off-line rheometry

Figure 2 shows the evolution of the viscosity as a function of the shear rate for different temperatures ranging from 25 to 50 ℃. The onset of the flattening of the infinite viscosity region is in the range of the expected wall shear rate which varies between 5 and 40 s^{-1}. Thus the power law model is only an approximation of the actual shear thinning characteristics of the fluid.

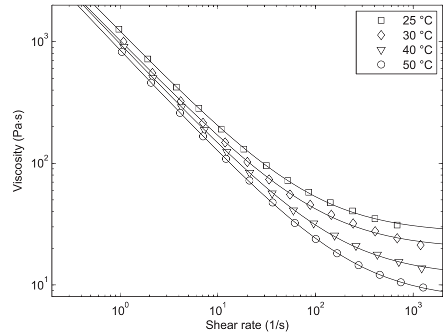

Figure 2: Viscosity curves of the adhesive used for the profile measurements obtained with a laboratory rheometer at different temperatures.

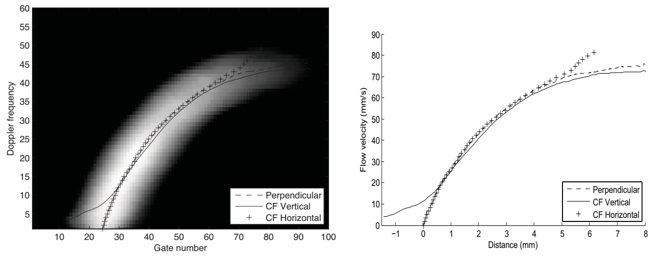

Figure 3: Example of a velocity profile measured for a flow rate of 30 ml/s with a pulse length of 4 cycles and a base frequency of 3.5 MHz. The left side shows the spectral intensities and the estimated profiles. The right side just the profiles estimated with three different methods.

3.2 Acoustic properties

The material has a relatively high attenuation coefficient of 0.4 Np/cm and a sound velocity of 1376 m/s at 20 ℃ with a temperature dependency of $-3.4\,\mathrm{m\,s^{-1}\,℃^{-1}}$.

3.3 In-line rheometry

Figure 3 shows a typical result for a profile measurement at a volume flow rate of 30 ml/s. The full line («CF Vertical») shows the conventional profile estimated from the channel-wise central frequency. It is obvious that this profile is not usable to extract the wall shear rate. The «+» symbols («CF Horizontal») indicate the central frequency determined across different depths. The resulting profile is realistic over the first 5 mm from the wall until the penetration limit of the measurement is reached. Finally a dashed line («Perpendicular» shows the result of an estimation of the central frequency analyzing the spectral information perpendicular to the velocity profile. This approach results in a profile overlapping with the horizontal central frequency over a wide radial range. Towards the end of the measured profile the result is better but not necessarily very reliable. Very close to the wall the method is not usable with the available spectral information.

3.4 Variation of physical measurement parameters

3.4.1 Pulse length

The pulse length was varied from 1 to 32 cycles for a base frequency of 3.5 MHz for a flow rate of 1 ml/s. The surface plots of the spectral intensity distribution (figure 4) show the expected broadening of the signal, the estimated velocity profile is shifted proportionally to the number of cycles away from the wall. The pulse length has no visible effect on the profile shape.

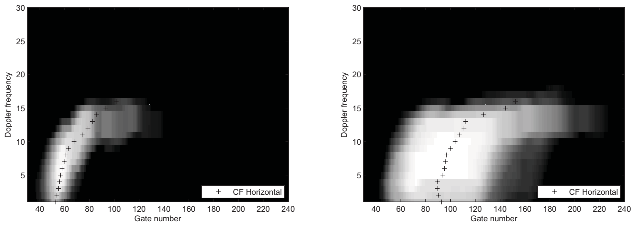

Figure 4: Spectral intensities for a pulse length of 1 (left) and 32 (right) cycles.

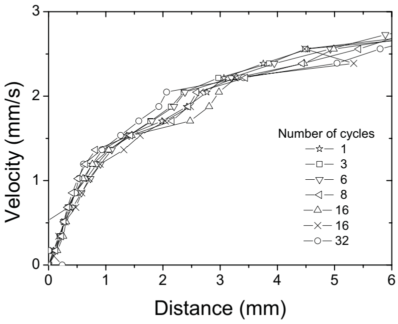

Figure 5: Profiles estimated for different pulse lengths. All curves were normed to distance zero.

3.4.2 Frequency variation

The result of a variation of base frequency from 2 to 6.5 MHz (figure 6) looks similar to the one for the pulse length variation. The profiles are just shifted in radial direction (not visible in the figure as the profile locations are normalized) while the estimated wall shear rate is not affected (within the limited available precision).

3.4.3 Gain variation

Also a variation of the analog gain (figure 7), the amplification of the received signal, does not influence the shape of the profile and the resulting wall shear rate significantly.

3.5 Variation of post processing parameters

3.5.1 Assumed Doppler angle

The transducer was not directly in contact with the fluid. Therefore the Doppler angle (angle between flow- and beam directions) in the fluid has to be calculated by Snells law ($c_2 \sin\theta_1 = c_1 \sin\theta_2$) which correlates incidence θ_1 and refraction θ_2 angles with the sound velocities (c_1 and c_2) in two different media [6]. The wall shear rate is sensitive to the Doppler angle (figure 8) but even an error of 50 m/s in the sound velocity of the fluid would only result in an error of the Doppler angle of less than 1° and accordingly 1.5 s^{-1} in the wall shear rate. Thus the relative errors in sound velocity and wall shear rate are approximately equal.

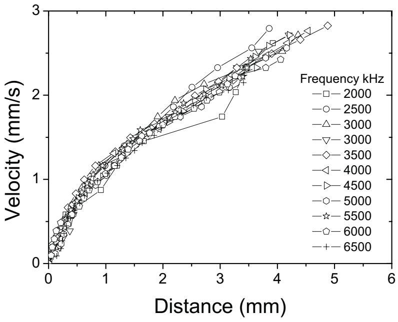

Figure 6: Profiles measured at different frequencies. All curves were normed to distance zero.

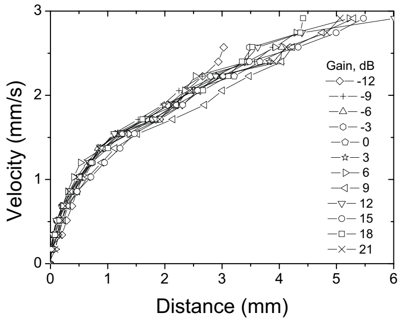

Figure 7: Profiles measured with different amplification settings.

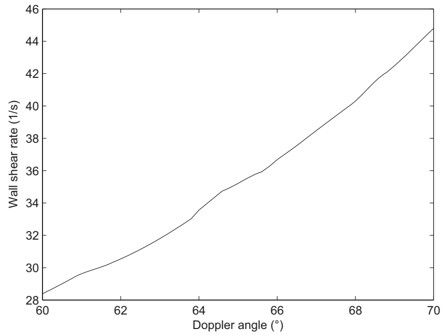

Figure 8: Influence of assumed Doppler angle on wall shear rate.

3.6 Flow rate variation

The flow rate was varied in steps of 5 ml/s from 5 ml/s to 30 ml/s and back to 5 ml/s. The wall shear rate was determined using the velocity data in the first 0.5 mm from the wall (corresponding to 4 % of the pipe radius). In a first step the velocity information was used to to fit the power law exponent n with following equation [21]:

$$v(r) = \bar{v}\frac{3n+1}{n+1}\left(1-\left(\frac{r}{R}\right)^{\frac{n+1}{n}}\right) \quad (2)$$

where $\bar{v}$ the average flow velocity ($\bar{v} = \frac{Q}{\pi R^2}$). Next the wall shear was calculated using equation 1. Fitting to the power law based velocity profile equation was chosen to obtain more realistic wall shear rates compared to a simple polynominal fit.

The resulting wall shear rates are shown as circles in figure 9. The shear rate predicted using a constant power law exponent n equal 0.2 and equation 1 is indicated with «×» symbols in the same figure. In this case the wall shear rate is simply direct proportional to the volume flow rate. But as shown in the laboratory rheometer measurements (section 3.1 and figure 2) the power law model is only an approximation, especially for the high shear rates. Therefore we used a second approach and calculated a «local» power law fit of the viscosity curves measured with the rheometer using one decade of shear rates around the approximate wall shear rate. Those calculations also consider the fluid temperature measured in the flow cell. The resulting n varies between 0.20 and 0.33 for flow rates of 5 and 30 ml/s respectively.

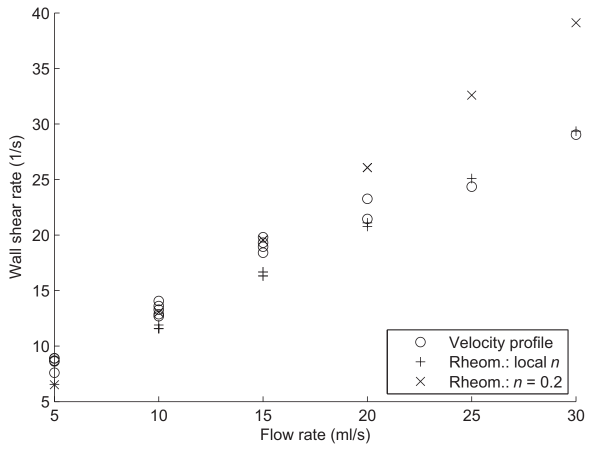

Figure 9: Wall shear rates in function of the flow rate extracted from the measured velocity profiles and predicted based on the rheometer measurements.

The resulting wall shear rates, again calculated using equation 1 are indicated with «+» symbols in figure 9. Especially for the higher flow rates those wall shear rates match the ones extracted from the velocity profiles quite well. For the lower flow rates the wall shear rate extracted from the flow profiles is slightly overestimated. This corresponds to an increase of the velocity slope very close to the wall also visible in figures 5 to 7. It is assumed that this artifact is due to the fact that in this region only a part of the sample volume is inside the fluid. More sophisticated spectral analysis methods should enable a compensation of this effect.

4 Conclusions

It was shown that an optimized measurement setup and profile estimation method allow to obtain the velocity profile in the wall region. The presented velocity estimation method has the additional advantage that the wall shear rate is insensitive to the sample volume, thus ultrasound parameters such as pulse length, base frequency and amplification. Further measurements, for example in fluids with different shear thinning characteristics at a constant flow rate, are necessary to test accuracy and precision the measurement system. Also a CFD calculation of the velocity profile based on the rheometer measurements will be of interest.

Acknowledgments

We would like to thank Dr. Stefano Ricci and Prof. Piero Tortoli from the Microelectronics Systems Design Laboratory of the University of Florence for their help optimizing the measurement setup. We are also thankful to Kurt Berli from Sika Services Widen for the help conducting the flow measurements and Patrik Kuhn from Sika Technology for the laboratory measurements.

References

[1] B. H. Birkhofer, S. A. K. Jeelani, E. J. Windhab, B. Ouriev, K.-J. Lisner, P. Braun, and Y. Zeng. Monitoring of fat crystallization process using UVP-PD technique. *Flow Measurement and Instrumentation*, 19(3-4):163–169, 2008.

[2] N. Dogan, M. J. McCarthy, and R. L. Powell. Comparison of in-line consistency measurement of tomato concentrates using ultrasonics and capillary methods. *Journal of Food Process Engineering*, 25:571–587, 2003.

[3] N. Dogan, M. J. McCarthy, and R. L. Powell. Measurement of polymer melt rheology using ultrasonics-based in-line rheometry. *Measurement Science and Technology*, 16:1684–1690, 2005.

[4] H. Fock, J. Wiklund, and A. Rasmuson. Ultrasound velocity profile (UVP) measurements of pulp suspension flow near the wall. *Journal of pulp and paper science*, 35:26–33, 2009.

[5] C. Hepburn. *Polyurethane Elastomers*. Elsevier, 2nd edition edition, 1992.

[6] C. R. Hill, J. C. Bamber, and G. R. ter Haar, editors. *Physical Principles of Medical Ultrasonics*. Wiley, 2nd edition, 2004.

[7] P. E. Hughes and T. V. How. Quantitative measurement of wall shear rate by pulsed Doppler ultrasound. *Journal of Medical Engineering & Technology*, 17(2):58–64, 1993.

[8] P. E. Hughes and T. V. How. Pulsatile velocity distribution and wall shear rate measurement using pulsed Doppler ultrasound. *Journal of Biomechanics*, 27(1):103–110, 1994.

[9] J. E. Jorgensen and J. L. Garbini. An analytical procedure of calibration for the pulsed ultrasonic Doppler flow meter. *Transactions of the ASME/Journal of Fluids Engineering*, 96:158–167, 1974.

[10] J. E. Jorgensen, D. N. Campau, and D. W. Baker. Physical characteristics and mathematical modeling of pulsed ultrasonic flowmeter. *Medical and Biological Engineering*, 11(4):404–421, 1973.

[11] R. Kotze, R. Haldenwang, and P. Slatter. Rheological characterisation of highly concentrated mineral suspensions using an ultrasonic velocity profiling with combined pressure difference method. *Applied Rheology*, 18(6):62114, 2008.

[12] T. A. Kowalewski. Velocity profiles of suspension flowing through a tube. *Archives of Mechanics*, 32(6):857–865, 1980.

[13] D. Lootens, P. Jousset, C. Dagallier, P. Hebraud, and R. Flatt. The "dog tail test": a quick and dirty measure of yield stress. application to polyurethane adhesives. *Applied Rheology*, 19(1): 13726, 2009.

[14] M. Müller, P. Brunn, and C. Harder. New rheometric technique: The gradient-ultrasound pulse Doppler method. *Applied Rheology*, 7(5):204–210, 1997.

[15] B. Ouriev and E. J. Windhab. Rheological study of concentrated suspensions in pressure-driven shear flow using a novel in-line ultrasound Doppler method. *Experiments in Fluids*, 32:204–211, 2002.

[16] S. Ricci, E. Boni, F. Guidi, T. Morganti, and P. Tortoli. A programmable real-time system for development and test of new ultrasound investigation methods. *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, 53(10):1813–1819, 2006.

[17] P. Tortoli, F. Guidi, G. Guidi, and C. Atzeni. Spectral velocity profiles for detailed ultrasound flow analysis. *IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control*, 43(4):654–659, 1996.

[18] P. Tortoli, T. Morganti, G. Bambi, C. Palombo, and K. V. Ramnarine. Noninvasive simultaneous assessment of wall shear rate and wall distension in carotid arteries. *Ultrasound in Medicine & Biology*, 32(11):1661–1670, 2006.

[19] J. Wiklund and M. Stading. Application of in-line ultrasound Doppler-based UVP-PD rheometry method to concentrated model and industrial suspensions. *Flow Measurement and Instrumentation*, 19(3-4):171–179, 2008.

[20] J. Wiklund, I. Shahram, and M. Stading. Methodology for in-line rheology by ultrasound Doppler velocity profiling and pressure difference techniques. *Chemical Engineering Science*, 62:4277–4293, 2007.

[21] W. L. Wilkinson. *Non-Newtonian fluids: fluid mechanics, mixing and heat transfer*, volume 1. Pergamon Press, 1960.

[22] T. Wunderlich and P. O. Brunn. Ultrasound pulse Doppler method as a viscometer for process monitoring. *Flow Measurement and Instrumentation*, 10(4):201–205, 1999.