TruGap™: A new device for controlling the true gap size in real time during rheological testing.

Jörg Läuger, Patrick Heyer, Gerhard Raffer

Anton Paar Germany GmbH, D-73760 Ostfildern / Germany
Phone: +49-711-72091-0
info.de@anton-paar.com, www.anton-paar.com

ABSTRACT

The actual size of the measuring gap in rotational rheometers has been a matter of discussions for a long time. In order to overcome the limitations of existing gap adjustment procedures a new patented system called TruGap™ was developed, which directly measures the actual gap size during the running experiment.

INTRODUCTION

The determination of the exact size of the measuring gap in cone-and-plate and parallel-plate geometries for a rotational rheometer is crucial for precise rheological investigations [1]. An error in the gap size influences directly the accuracy of the measuring results in parallel-plate and cone-and-plate measurements. Nowadays modern research rheometers use electric motors to move the measuring head with the attached plate or cone. After a determination of the so-called zero gap position the measurement position can be set accurately within 1µm in a high quality rheometer. In temperature dependent experiments the change in length of the measurements systems due to thermal expansion with temperature is taken into account by using a compensating gap adjustment routine. Normally, a thermal expansion coefficient stored in the software is used by such Automatic Gap Control (AGC) methods. In order to account for the gap change by the thermal expansion the rheometer is moving the measuring geometry in axial direction and adjusts the gap by the calculated value.

However, these adjustments are based on empirically established temperature-position functions and hence use fixed expansion coefficients, which are determined under specific temperature conditions when the thermal expansion is completed. Expansion coefficients are typically on the order of 1 µm/K. Quite often temperature ramps are conducted much faster and the measurement geometries have not reached their final length making the automatic gap adjustment inaccurate. Additionally other effects like the expansion of the rheometer frame due to altering laboratory conditions change the actual gap as well and influence long lasting experiments. Therefore an adequate level of compensation cannot be achieved in practice, owing to the largely unknown nature of temperature equalization times. In addition such compensation routines need to be established individually for each measurement geometry and environmental system combination.

In order to overcome the limitations of existing gap compensation procedures TruGap™, a patented (US Patent 6,499,336) device, was developed. Unlike in existing methods the TruGap™ device does not approximate the gap size, but, directly measures and keeps constant the real gap size during the running experiment.

ERRORS DUE TO GAP SETTING

An uncertainty or an error in the gap size results directly in an error in the rheological property, which is measured, for example the viscosity. Absolute values of theses errors have been calculated for various cone angles and cone diameters. Two different models have been employed. Firstly, the error in viscosity was calculated by assuming a variable sample volume by changing the gap. This simulates a wrong gap size during the sample filling and sample trimming process, i.e. before the actual measurement starts. Secondly, an error due to a change in gap after the sample is loaded was calculated, i.e. in this case the sample volume is assumed to be constant. For both calculations a Newtonian liquid and uniform condition at the edges are assumed.

For small cone angles and small changes in the gap both calculations, i.e. with constant and with variable volume, reveal the same size of the error.

Figure 1 shows some examples of the calculations for different cone angles and cone diameters. Interestingly, for the CP25-2 (25 mm diameter, 2° cone) and the CP50-1 (50 mm diameter, 1° cone) geometry the error size is of the same order.

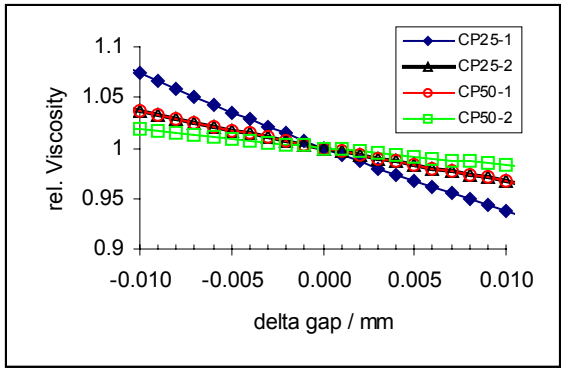

Figure 1. Calculated error for different measuring geometries.

As can be seen form Figure 1 a gap error of 5 µm leads to an error in the viscosity between 1.5% and 4%. A smaller gap reveals a higher viscosity, whereas a larger gap gives a viscosity reading, which is too low, respectively.

In order to confirm the error calculations measurements on a standard oil have been performed as shown in Figure 2.

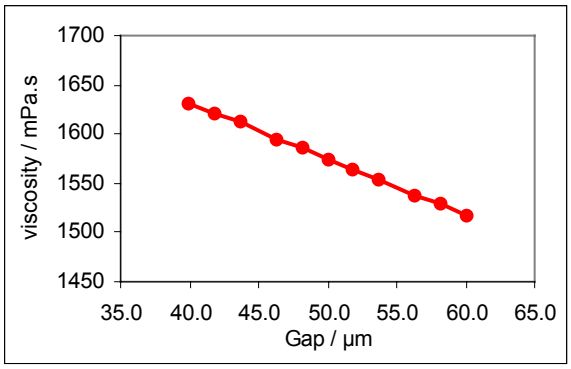

Figure 2. Viscosity at various gap sizes measured with a CP50-1 geometry.

A cone-and-plate geometry with a 50 mm diameter and a 1° cone angle has been used. The standard truncation of cone was 50 µm. The viscosity of the oil has been measured at the right gap position of 50 µm and at various positions above and below this value.

MEARUREMENT PRINCIPLE

The measuring principle of the TruGap™ system, which is depicted in Figure 3, is based on an induction position sensor. In a rotational rheometer the sample normally is placed between a fixed bottom plate and a moving upper plate or cone.

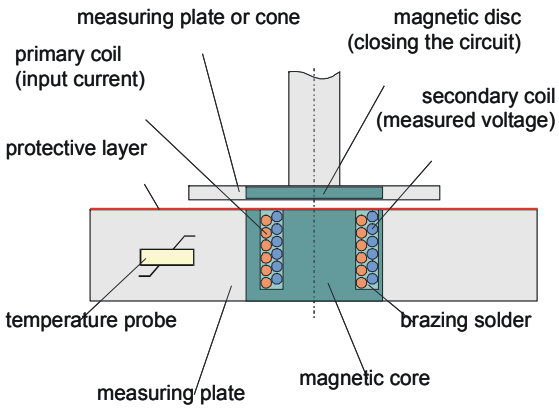

Figure 3. Measuring principle of the Anton Paar TruGap™ system.

In the fixed bottom plate a solenoid with two electric coils is embedded in a magnetic core. An AC current flows through the primary coil, which induces a voltage in the secondary coil and therefore the magnetic impedance can be calculated. A soft magnetic disc in the upper geometry closes the circuit. The magnetic impedance and the voltage at the secondary coil correlate with the distance, i.e. the gap, between the magnetic core in the lower plate and the soft magnetic disc in the upper plate.

In Figure 4 cross-sectional views of a Peltier bottom plate and an upper measurement geometry are shown, respectively.

Figure 4. Cross-sectional views of prototypes of a Peltier lower plate (bottom) and an upper geometry (top). The solenoid system and the magnetic disc can bee seen, respectively.

Figure 5 shows a typical example of the dependence of the measured voltage to the gap size. Such relations serve as calibration curves and are measured for each individual environmental system and are the basis for the real gap calculation during measurements.

Since the voltage-gap relation is temperature dependent this relation is measured at various temperatures leading to a whole set of calibration curves, which are stored on a chip located in the plug of the individual environmental chamber. As soon as the environmental chamber is connected to the rheometer the chamber is recognized by the rheometer electronics and the software and calibration data are automatically available without the need of any special setup in the software.

Similarly to the environmental chamber recognition a TruGap™ measurement geometry is recognized as well as soon as it is connected to the rheometer. However, the calibration data are only dependent on the solenoid system in the base plate and not on the measurement geometry used. The measurement geometry and environmental chamber recognition system is called Toolmaster™ and is available for all rheometers of the Physica MCR series from Anton Paar.

During a rheological test the voltage is measured and by taking into account the calibration data a feedback mechanism is used to move the measurement geometry in order to keep the gap at the desired constant value.

It has been verified that the induction based gap measurement does not produce any additional effect to the normal force signal nor to the torque measurement, respectively, due to possible eddy currents.

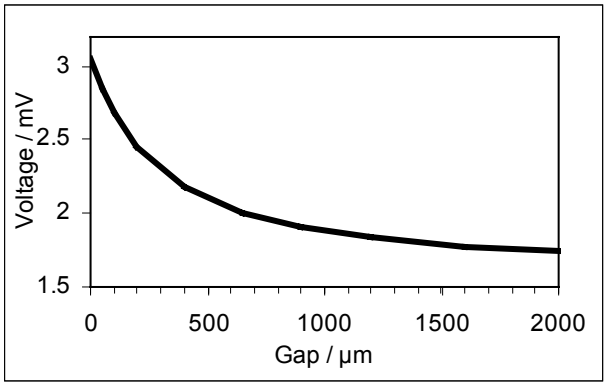

Figure 5. Typical relation between the measured voltage and the gap size.

Since the TruGap™ system does not require any mechanical interaction with the

sample the rheological experiment will not be influenced by the gap measurement at all. The method is also not limited to optical transparent samples. The only restrictions are ferromagnetic samples.

The device was found to work over an extended temperature range from -100°C to 280°C with an accuracy of < 1µm for cone-and-plate measuring systems with 50µm gap due to the truncated cone tip and < 3µm for parallel-plate measuring systems with 1mm gap size.

The TruGap™ device is an additional option for the Physica MCR 301 and MCR 501 rheometer from Anton Paar and is available for Peltier, electrical resistance, and convection based environmental systems, respectively.

EXPERIMENTAL

Instrument

The measurements were conducted using the Physica MCR 501 rheometer from Anton Paar. The MCR 501 rheometer as shown in Fig. 6 is based on an air-bearing supported synchronous motor with electrical commutation (EC-motor). This unique motor concept offers full control of the motor movements, thus allowing real controlled stress and controlled strain with the same instrument [2]. For temperature control a Peltier system with an additional actively controlled Peltier hood is used assuring uniform temperature distribution throughout the measuring gap [3]. The Peltier bottom plate and the measurement geometries used are TruGap™ systems.

Figure 6. The Physica MCR 501 rheometer from Anton Paar (left) and the PTD 200 temperature control system (right).

RESULTS AND DISCUSSION

External disturbance on the shaft

In order to show how the TruGap™ system is working the shaft of the measuring geometry was influenced by an external temperature disturbance during a running test. A silicone oil (AK 5000) was tested with a cone-and-plate measurement geometry (CP 50-1) at room temperature. The viscosity was measured by applying a constant shear rate of 10 s^{-1}. A coolant spray and a heating gun was used to cool or heat the shaft of the measuring geometry during the test, respectively.

Figure 7 shows the influence of different gap correction methods on the oil viscosity measurement after cooling the shaft with the cooling spray. Cooling the shaft leads to thermal shrinkage of the shaft.

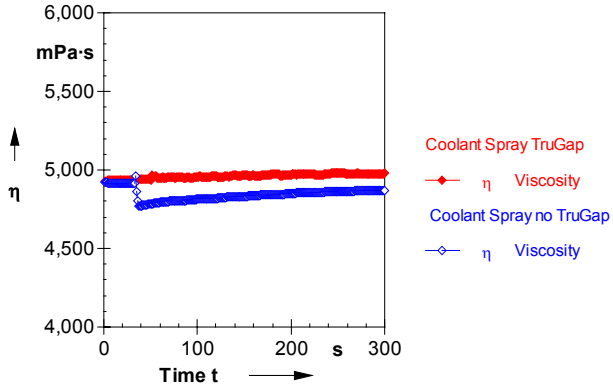

Figure 7: Viscosity curves of a silicon oil at a shear rate of 10 1/s with and without TruGap. After some time a short external disturbance with a coolant spray at the measuring shaft was applied.

When AGC as correction method is used the change in the gap size due to the shrinkage of the shaft is not noticed because AGC is just related to the temperature of the bottom plate. As soon as the shaft is cooled the measured viscosity value decreases. This viscosity change is just apparent because the gap size increases due to the thermal shrinkage of the shaft so that the calculation of the measured viscosity is based on an incorrect gap size. As discussed in Figure 1 a larger gap leads to a lower viscosity value. As the cooling was stopped the shaft starts to heat up again and expands until the initial temperature and length is reached again. As can bee seen in Figure 7 this process takes a rather long time.

In the case of performing the same test using the TruGap™ control the gap is measured and adjusted to a constant value during the whole test. If a thermal shrinkage occurs due to the application of the cooling spray on the shaft, the gap is adjusted immediately and the measured viscosity is displayed correctly as a constant value during the whole test.

A similar situation occurs if the shaft is heated shortly by a heating gun as indicated in Figure 8. With the TruGap™ system switched off and just an AGC correction on, the shaft expands, the gap becomes smaller, and the measured viscosity increases. Only after a long time the shaft shrinks back to its original length and then the viscosity value shows the initial value again. Like in the case of the coolant spray the TruGap™ system controls the gap to the right size even during a sudden length change of the measuring geometry, thus revealing a constant viscosity value during the whole test.

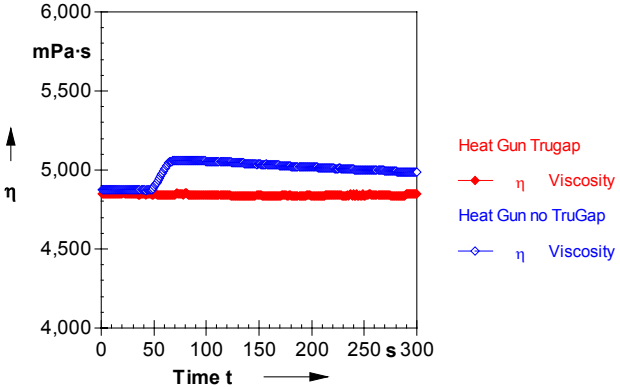

Figure 8: Viscosity curves of a silicon oil at a shear rate of 10 1/s with and without TruGap. After some time an external disturbance with a heat gun on the measuring shaft was applied.

Temperature sweep

As another example, a temperature sweep experiment from 30°C up to 90°C was performed.

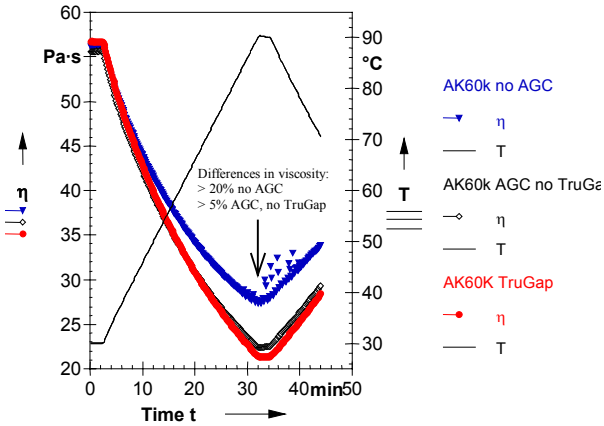

Figure 9: Effect on the viscosity during a temperature sweep with different gap control functions: a) no gap compensation, b) AGC with standard settings, c) TruGap™.

Figure 9 shows the temperature dependent viscosity function of a silicone oil AK 60000 sample measured with a cone-and-plate geometry (CP 50-1) during a temperature sweep from 30°C up to 90°C at a constant shear rate of 10 s^{-1}. The same measurement has been performed using three different gap control settings: a) No gap control, b) AGC (standard thermal expansion coefficient), and c) TruGap™ control, respectively.

The highest viscosity values are measured without any gap control or gap correction function. In this case the thermal expansion of the measurement geometry is not compensated. The gap size at 80°C is only 5 µm instead of 50 µm as indicated in Figure 10. Reaching 85°C, the cone eventually touches the bottom plate which results in a scattering of the data in the viscosity curve.

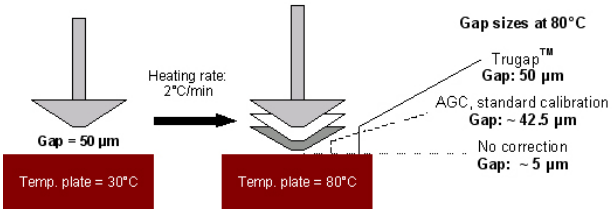

Figure 10: Gap dimensions at 80°C resulting from different gap control modes using a heating rate of 2°C/min.

The curve showing the medium viscosity values is measured using AGC as gap correction function. The instrument adjusts the gap using the expansion coefficient given in the software. For accurate results, this expansion coefficient has to be exactly determined for any given heating rate and measuring configuration. The measurement with AGC shown in Figure 9 is performed without any special determination of the AGC coefficient. Therefore the default expansion coefficient from the software program, which represents the thermal equilibrium, i.e. the final expansion of the geometry is used. Obviously the expansion coefficient does not exactly fit for the given heating rate of 2°C/min. Therefore at 80°C the gap size is 42.5 µm instead of 50 µm and thus the measured viscosity values are to high.

In order to get accurate results using AGC as correction method a time intensive calibration of the expansion coefficient for the used measuring geometry and heating rate had to be performed and repeated whenever the heating rate or the measurement geometry is changed.

CONCLUSIONS

A new patented (US 6,499,336) online gap measurement device based on an induction method was developed. The Physica TruGap™ does not require any mechanical interaction with the sample and therefore the rheological experiment will not be influenced by the gap measurement at all. The device is working over an extended temperature range and can be incorporated into Peltier, electrical resistance, and convection based temperature control systems, respectively.

REFERENCES

1. Macosko C. W., Rheology: Principles, Measurements, and Applications (VCH Publishers, New York, 1994)

2. Läuger J, Huck S., Proceedings of the XIIIth International Congress on Rheology, Cambridge, UK (2000), 3: 10-13

3. Läuger J. Bernzen M., Wollny K., Raffer G., Annual Transactions of the Nordic Rheology Society, Volume 10 (2002), 147-153