True Gap Control: Direct measurement of the real gap size during parallel-plate and cone-and-plate rheological experiments

Jörg Läuger, Anton Ziegler, Gerhard Raffer

Anton Paar Germany GmbH
Helmuth-Hirth-Str. 6, D-73760 Ostfildern / Germany
Phone: +49-711-72091-0, Fax:+49-711-72091-50
info@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. An error in the gap size influences directly the measuring results in parallel-plate and cone-and-plate measurements. In modern rheometer the length change of the measurement systems due to thermal expansion is taken into account by using gap correction routines. However, these adjustments are based on fixed expansion coefficients, which do not describe transient behavior during temperature ramps and due to changing laboratory conditions.
In order to overcome the limitations of existing gap adjustment procedures Physica TruGap™ (US Patent 6,499,336) was developed, which directly measures the actual gap size during the running experiment. The measuring principle is based on an induction method. Since it does not require any mechanical interaction with the sample the rheological experiment will not be influenced by the gap measurement at all. The described Physica TruGap™ device is working over an extended temperature range and can be incorporated into Peltier, electrical resistance, and convection based environmental systems, respectively.

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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.
However, these adjustments are based on empirically established temperature-position functions and hence mostly fixed expansion coefficients, which are determined under specific temperature conditions when the thermal expansion is completed. 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 Physica TruGap^{TM}, a patented based (US Patent 6,499,336) devices was developed. Unlike in existing methods, the Physica TruGap^{TM} device does not approximate the gap size, but, directly measures and keeps constant the real gap size during the running experiment.

ERROR DUE TO GAP

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 and the CP50-1 geometry the error size is of the same order.

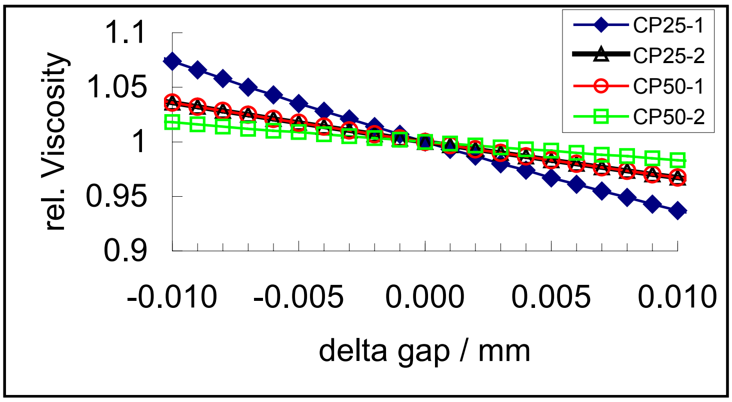

Figure 1. Calculated error for different measuring geometries.

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

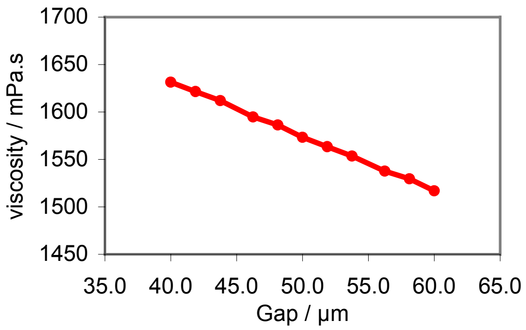

Figure 2. Viscosity at various gap sizes measured with a CP50-1 geometry.

A cone-and-plate geometry with a 50mm 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.

MEASUREMENT PRINCIPLE

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

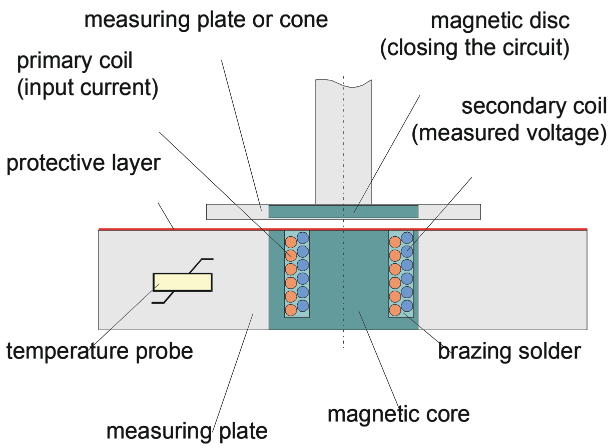

Figure 3. Measuring principle of the Physica TruGap^{TM} system.

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.
Figure 4 shows a typical example of the dependence of the measured voltage to the gap size. Such relations are measured for each individual environmental system and are the basis for the real gap calculation during measurements. A feedback mechanism is used to keep the gap at a constant value during the rheological tests.
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.

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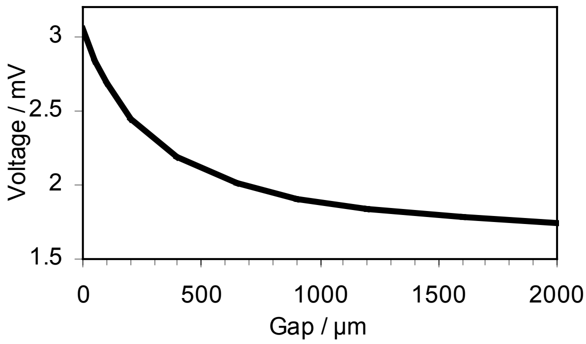

Figure 4. Typical relation between the measured voltage and the gap size.

Since the Physica 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 not limited to optical transparent samples. The only restrictions are ferromagnetic samples.
The device was found to work over an extended temperature range of -150°C – 350°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 Physica TruGap™ device is available for Peltier, electrical resistance, and convection based environmental systems, respectively.

CONCLUSIONS

A new online gap measurements device (US Patent 6,499,336) 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 environmental systems, respectively.

REFERENCES

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