Fluid temperature control in rotational rheometers with plate-plate measuring systems

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ABSTRACT

This study analyses the temperature differences between a top plate, a bottom Peltier and a fluid sample in a rotational rheometer during oscillation tests.

The results show that the steady state temperature difference between the fluid and the Peltier plate is of the order of several degrees centigrade when using high temperatures, and the lack of control gets even worse during rapid transient heating tests.

INTRODUCTION

Control with temperature is of paramount importance during rheological measurements since the viscosity and other rheological parameters strongly depend on temperature. It is commonly practised that rheological tests are undertaken at temperatures other than those of ambient or room temperature. The presence of thermal gradients induced by conductive heat fluxes and surface heat transfer effects are difficult to observe, these effects are particularly prevalent in systems where the sample is heated only from bellow, typically by a Peltier device.

A Non-isothermal sample will present anisotropic rheological properties. Care must be taken when measuring fluids having high temperature dependent viscosities and also fluids having low thermal conductivities.

The temperature variations in plate-plate and cone-plate geometries have previously been determined using temperature sensors, [1, 2].

In this work the temperatures are recorded by the use of IR-camera and by temperature sensors in the rheological instrument. In addition, by employing a Newtonian certified viscosity standard, Polybuthene-1, between the plates in the rheometer it is also possible to determine the effective temperature of the fluid sample (the Polybuthene-1 sample) by measuring the viscosity and back-calculate the temperature based on a known calibrated viscosity versus temperature curve.

MATERIALS AND METHODS

The basic measuring geometry consisted of a Physica UDS200 rheometer fitted with a MP31 top plate and Peltier bottom plate.

An IR camera, ThermoVision A40M Researcher (FLIR Systems AB, Danderyd, Sweden) with software was connected as shown in Fig. 1. The camera measures the long wave IR radiation between 7.5 and 13.0 μm with a focal plane array detector, displaying 76 800 pixels.

The MP31 top plate was painted opaque black with acrylic paint (OS476 Black, Duncan Enterprises, US) to obtain a good measurement of how much radiation is emitted by setting the right emissivity

(see Fig. 2). The emissivity of the surface was determined by placing the MP31 in a temperature controlled cabinet (Termaks Serie 4000[a]), at 70 °C while monitoring the probe surface. The emissivity was determined by using the software ThermaCAM Researcher Pro 2.8 (FLIR Systems AB) to be 0.83.

Experimental set-up

The internal clocks in the Physica system and the external PC running the IR software were synchronized.

The UDS200 was programmed to run at different controlled temperature gradients, and in one test the UDS200 was programmed to do a quick step change from 40 °C to 90 °C.

All the tests were performed in oscillation with an angular frequency of 1 s^{-1} at 0.2% strain in order to minimize convective fluid effects. Since the measurements were made without fluid flow, the system can be treated as stationary.

Figure 1: IR camera set-up in front of the Physica UDS200 rheometer.

Fluid temperature determination

The representative fluid temperature was determined from a back-calculation method from the viscosity of Polybuthene-1.

A calibration test was initially run on a cylindrical bob/cup system, Z3, at a slow temperature gradient; 0.01 K/minute over the temperature range 45 - 105 - 45 °C as shown in Fig. 3.

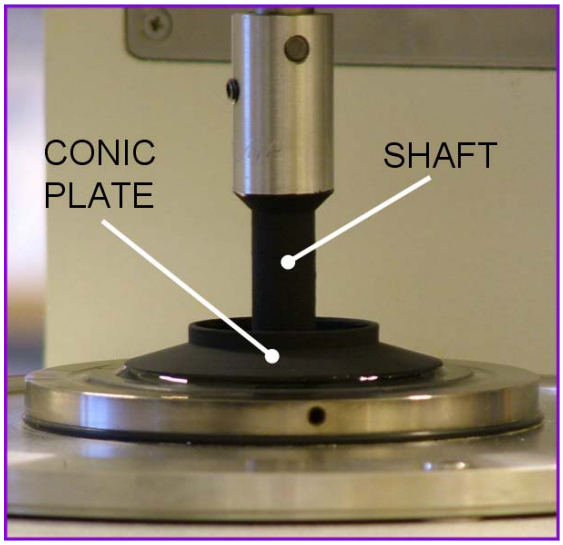

Figure 2: MP31 top plate painted opaque black for the occasion. The chosen positions for the IR temperature measurements on the shaft and conic plate are shown.

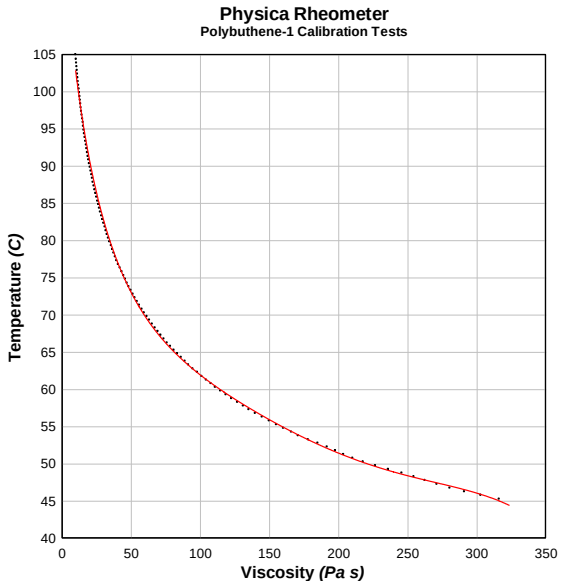

Figure 3: Results from calibration tests of Polybuthene-1 in the Physica UDS200 rheometer.

The rheometer was in this manner used as a thermometer, giving a representative average temperature of the fluid sample. Knowledge of the viscosity of the fluid

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sample is used to predict the sample temperature.

RESULTS

IR-measurements

A typical greyscale version of a thermogram is shown in Fig.4. It is also possible to generate colour diagrams from the software package. The ThermaCAM Researcher Pro software was also capable to analyse the thermograms and generate tables of temperature versus time for specified locations. These tabular results are shown in the displayed diagrams.

The results from the 0.5 mm gap tests are shown in Fig. 5. Initially the temperature of the Peltier was programmed to rise at 0.71 K/min from 40 °C to 90 °C. The temperature was then kept at 90 °C for a period of 20 minutes. The temperature was then reduced at a rate of -3.57 K/min until the temperature of the Peltier reached 40 °C. The temperature was then kept constant at 40 °C for 5 minutes. Afterwards the temperature was increased and decreased at +/- 3.57 K/min with 5 minute holding time at 90 °C and 40 °C.

The results from the 1.2 mm gap tests with different temperature gradients are shown in Fig. 6 and Fig. 7. The response to the step change in temperature is shown in Fig. 8.

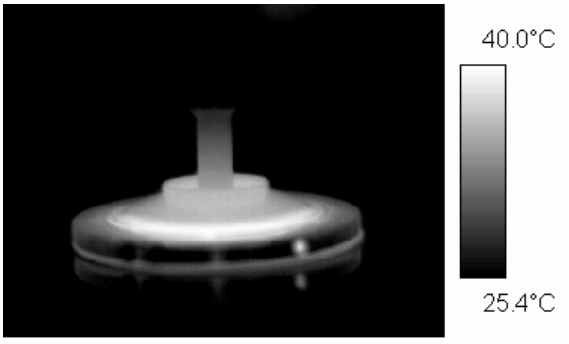

Figure 4: Thermogram in grey scale coding obtained directly from the IR camera software.

The response with temperature gradients of +/- 1.70 K/min with 7 minutes holding time at 90 °C is shown in Fig. 6. The response with temperature gradients of +/- 3.57 K/min with 3.5 minutes holding time at 90 °C is shown in Fig. 7.

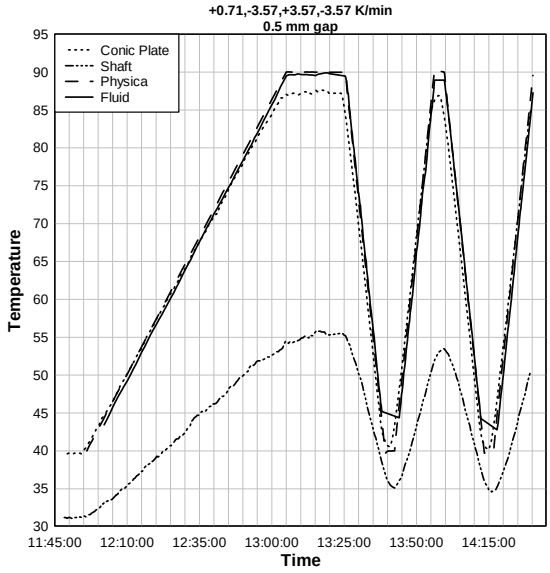

Figure 5: Recorded temperatures with 0.5 mm gap assembly with varying temperature gradients.

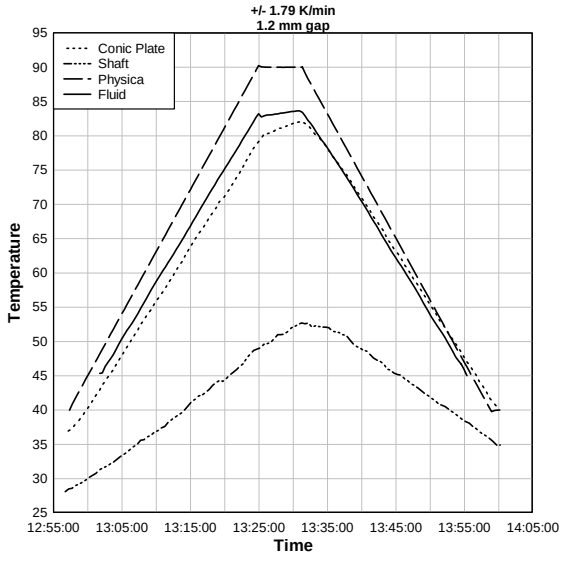

Figure 6: Recorded temperatures with 1.2 mm gap at 1.79 K/minute.

Back-calculation technique

The temperature of the fluid sample was back-calculated from the viscosity versus temperature calibration data in Fig. 3, and the fluid temperature is shown in the diagrams.

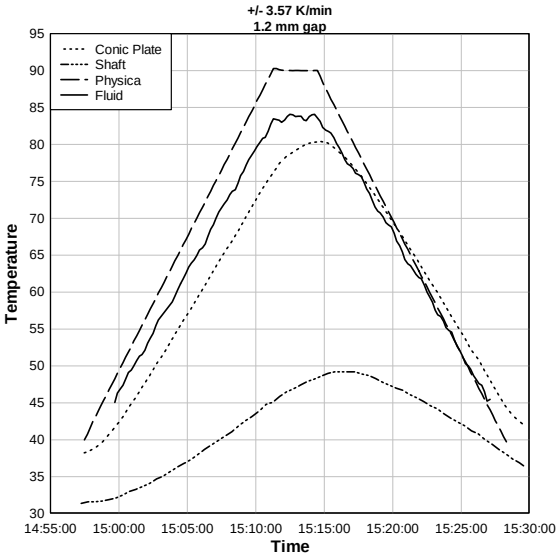

Figure 7: Recorded temperatures with 1.2 mm gap at 3.57 K/minute.

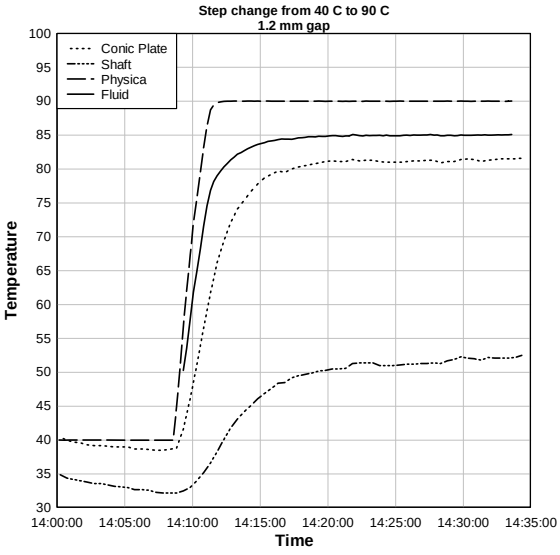

Figure 8: Recorded temperatures with 1.2 mm gap and fast change in Peltier set-point temperature.

The resulting temperature difference between the Peltier plate temperature sensor and the fluid temperature is shown in Fig. 9.

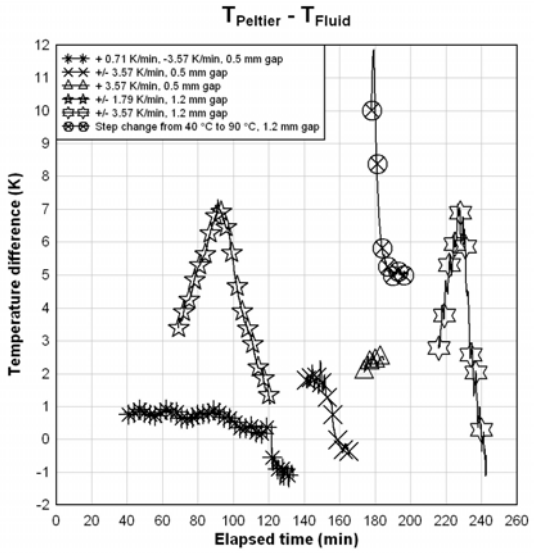

Figure 9: Temperature difference between Peltier element and fluid sample.

DISCUSSION

The temperature gradients chosen in this study are higher than what is normally advised for transient rheological measurements. The values were, however, chosen in this way to observe relatively large errors and to see if the temperature back-calculation method with the Ploybuthene-1 fluid worked satisfactorily.

The tests with 0.5 mm gap size show in general smaller temperature difference than the measurements performed with 1.2 mm gap size. The maximum observed difference was approximately 2.5 K for 0.5 mm gap size and close to 12 K with 1.2 mm gap size.

The largest error occurred with the 1.2 mm gap and was almost 12 K during the transient step change, while the maximum error was about 7 K at +3.57 K/min temperature gradient. The maximum error at 0.5 mm gap was approximately 2.5 K at 3.57 K/min and approximately 1 K at 0.71 K/min.

The results at 1.2 mm gap size do not seem to be very sensitive to the

temperature gradient. The reason for this must be that the heat transfer with the surroundings is dominating the heat transfer situation, making the MP31 temperature less sensitive to transient variations in the Peltier plate.

The search for a method that could predict problems of temperature control during rheological tests can be a great help to improve accuracies in the measurements.

The results from this study have also been compared with finite element calculations[3] (FEM), showing that finite element modelling also is a feasible method in evaluating transient rheological tests and can be used in experimental design.

CONCLUSIONS

The results from this study can be summarized as follows:

- The difference between the rheometer temperature and the fluid element temperature can be several degrees even at relatively slow temperature gradients.
- The observed temperature difference was a high almost 12 K when a step change in Peltier set point temperature.
- The back-calculating procedure using Polybuthene-1, worked well, using the rheometer as a thermometer.
- The temperature differences increase with increased gap size and during rapid transient heating.
- IR techniques are flexible and can be used to monitor the temperature variation of external surfaces during rheological measurements.
- Care must be exercised when setting tests in plate-plate rheometers if the sample is only heated from below, also special care must be considered when the sample is at several degrees above the room temperature.

REFERENCES

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