Measurements on selected (semi)-solids in a wide temperature range using new solid clamps

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ABSTRACT

Thermo Electron is launching a new temperature control module for the HAAKE MARS. The controlled test chamber (CTC) for measurements on polymer melts and (semi-) solids combines state of the art technology with easy handling.

For the CTC a unique solids clamp tool has been developed. The concept of the HAAKE MARS with the new CTC and some measurements results are shown.

INTRODUCTION

During rheological measurements on polymer melts high normal forces can be observed. These forces are valuable information but can also cause trouble because they might slightly change the measuring setup. Following that thought, compared to its predecessors like the HAAKE RheoStress 600, the HAAKE MARS has been given a different frame design. Apart from more space for sample handling or optional modules this "H"-frame has all forces aligned in one plane and thus offers an approximately 10 times higher stiffness compared to the standard "C"-frame. This makes the HAAKE MARS the perfect instrument to measure polymer melts and semi-solids.

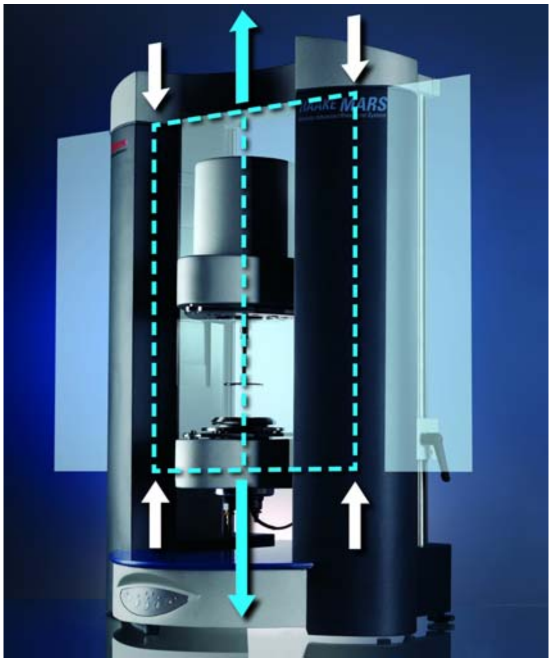

Figure 1. Optimized force balance with the "H"-frame.

CONTROLLED TEST CHAMBER

For measuring polymer melts and semi-solids, the HAAKE MARS is completed by the newly designed Controlled Test Chamber (CTC). It consists of 2 separate halves, each mounted on sliding rails, which allow moving them independently sideward and backwards and forwards. These halves can be moved backwards and closed in a so-called parking position. This allows free access to the sample fixtures for cleaning or loading a new sample without having to work between the cold or hot inner surfaces of the CTC. In the mean time the parked and closed CTC can already be brought to the desired temperature. It is even possible to leave the CTC in its parking position while using another temperature control unit like e.g. a Peltier, which saves a lot of time for mounting or dismounting the whole unit.

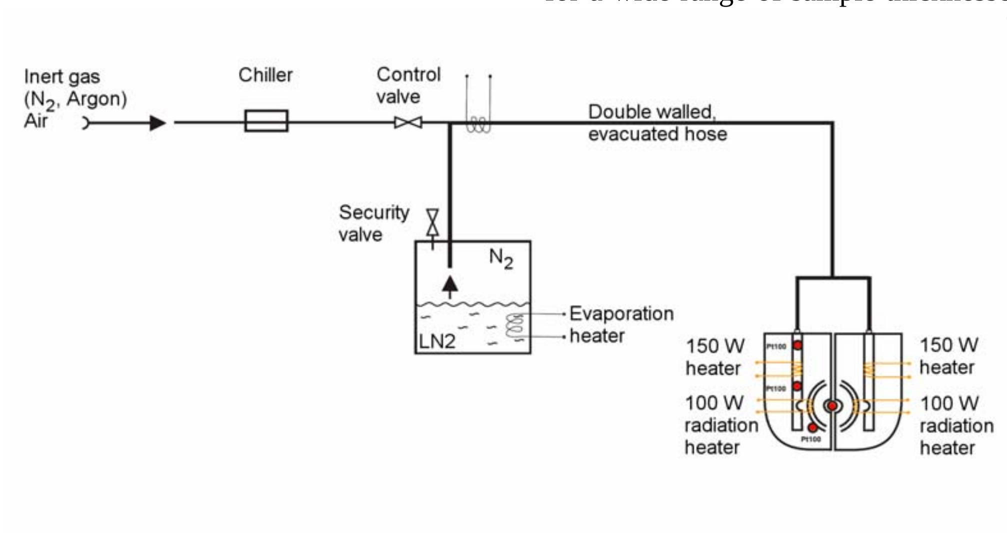

Figure 2. Schematic diagram of the CTC with its Low Temperature Option

The CTC combines the advantages of radiant heat transfer (fast temperature changes) with those of convection heat transfer (uniform temperature distribution). The two heat transfer systems are controlled by a state of the art “model predictive” digital temperature control loop. The temperature range spans from 30 °C to 600 °C and can be expanded down to -150 °C with the low temperature option using liquid nitrogen and a nitrogen evaporator. The maximum heating and cooling rates are 20 K/min. Thanks to the flexible, double walled, vacuum insulated tubing connecting the liquid nitrogen vessel to the CTC, the forming of ice on the outer surfaces of the unit is negligible, even at the lowest measuring temperature. Both halves are equipped with a window to observe the sample during measurements.

SOLIDS CLAMPS

The entirely new solids clamping tools according to DIN/ISO 7621-1 consist of two moving jaws, which guarantee an automatic centring of the sample relative to the rheometers axis. The automatic clamping force adaptation for the sample as well as a very simple semi-automatic gap adjustment for a wide range of sample thicknesses with just one fixture allow to measure over a wide range of temperatures in one go without ever loosing the grip on the sample. The jaws are easily removable for cleaning. Also jaws with various profiles for different sample types (soft, medium, hard) are available.

Figure 3. Solids Clamps set (left) and some details (right)

The sample can be 5.0 – 12.7 mm wide, 0.15 – 4.0 mm thick and have a maximum length of 68 mm. The bottom clamp can be adjusted in height. Samples of different lengths can all be fixed with their middle part being close to the centre of the CTC. Thus all samples of different lengths are exposed to the same extremely low temperature gradient. Using a flexible temperature sensor the temperature is measured very close to the sample.

MEASUREMENTS AND RESULTS

With any type of analytical equipment the ease of use is most impressively demonstrated by the speed with which users come to the point of generating good results with it. We prepared a set of solid samples of polyphenyleneether-polyamide (PPE-PA) compound by mould injection with the HAAKE MiniJet.

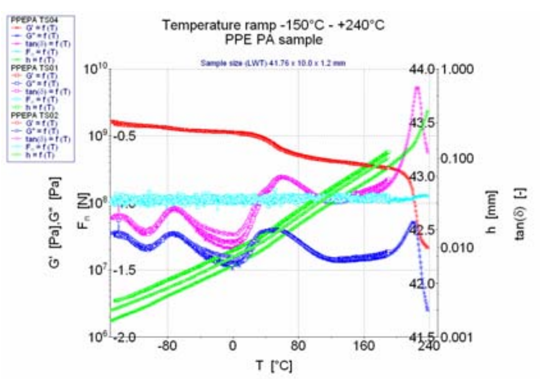

Figure 4. Good reproducibility of results done by first time users

During a training session different users took a fresh sample each, fixed it with the solids clamps and performed a temperature sweep between -150 °C and +240 °C. The curves shown in Figure 4 demonstrate the very good reproducibility of the results.

Figure 5 shows the measurement of a polystyrene (PS) sample under difficult conditions. The sample had also been prepared by mould injection but not given enough time to temper, which led to inner tensions. As a result, the sample shrank significantly above 105 °C. The quality of the rheological results is not affected because the lift of the HAAKE MARS compensated the change in length.

Since the lift of the HAAKE MARS is capable of determining its position with an accuracy of 0.5 µm, it is not simply “a lift” but in combination with the normal force sensor it is suitable as a measuring device as well.

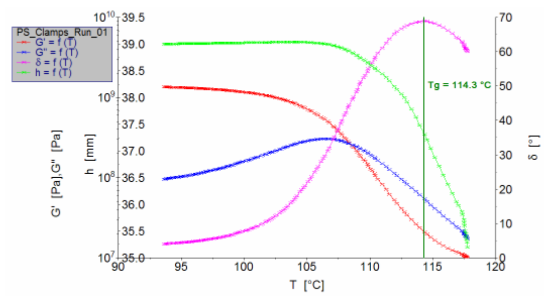

Figure 5. Tg of a shrinking PS sample

Although polymer samples soften above a certain temperature the solids clamps do not let go of the sample due to their self adjusting grip. Figure 6 shows an extreme example where the measurement of a thermoplastic polymer has been performed until the polymer started to melt.

Figure 6. Molten sample still showing the good grip of the clamps

Although the measurement itself had to be stopped due to the drastic change in diameter the clamps still had a good grip on the remaining polymer.

The very good grip of the solids clamps also nicely shows when testing very thin samples like e.g. a plastic coated copper wire of 1.5 mm diameter (Figure 7).

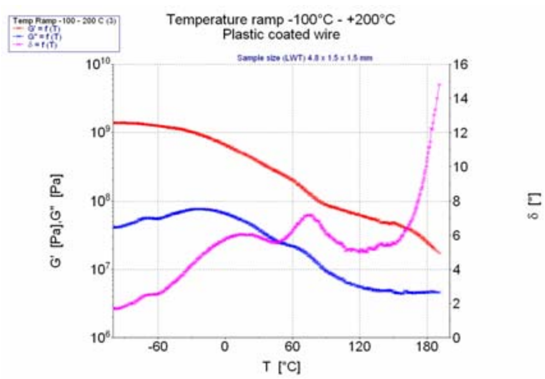

Figure 7. Plastic coated copper wire, diameter 1.5 mm

The capabilities of the solids clamps and the accuracy of the lift combine very well for a completely different kind of sample. Figure 8 shows the oscillation data of a piece of wood between room temperature and 200 °C. First the wood shrinks due to drying. Beyond a temperature of 100 °C the wood softens and shows thermal expansion.

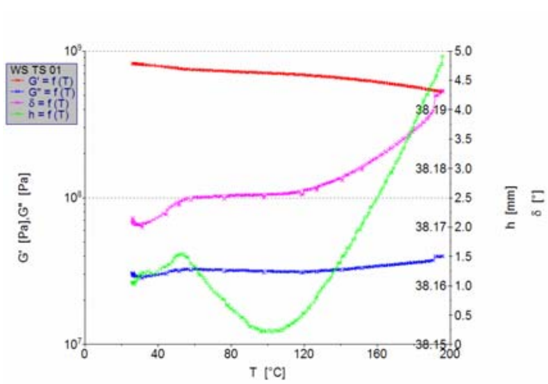

Figure 8. Shrinking and softening of wood measured with CTC and solids clamps

CONCLUSIONS

The newly designed CTC and the also newly designed unique solids clamps are robust, easy to use and allow measurements on polymer melts and semi-solids in one run between -150 °C and +600 °C. The samples are automatically centred relative to the rheometers axis. The clamps are self adjusting and thus always maintain a good grip on the sample. All these features combine to the good quality and reproducibility of the measured data.