

# Development of a RheoDSC, an Instrument for Simultaneous Rheological and Calorimetric Measurements

L. Van Lokeren<sup>1</sup>, R. Verhelle<sup>1</sup>, C. Block<sup>1</sup>, P. Van Puyvelde<sup>2</sup>, G. Van Assche<sup>1</sup>

<sup>1</sup>Vrije Universiteit Brussel, Brussels, Belgium

<sup>2</sup>Catholic University of Leuven, Leuven, Belgium

## Abstract

During the processing of polymers, transformations go hand in hand with changes in the thermal and rheological properties. During the crystallization of thermoplastics and the crosslinking polymerization (curing) of thermosets, the heat released is a direct measure of the progress of the transformation, while at the same time the rheology of these materials changes from a (predominantly) liquid to a solid behavior. The RheoDSC developed [1] combines a TA Instruments Q2000 Tzero DSC and AR-G2 rheometer, permitting simultaneous rheological and calorimetric measurements on a sample inside a DSC cell (Fig.1). More recently, a design optimization of the RheoDSC instrument and its calibration were supported by heat transfer modeling combined with crystallization or reaction kinetics modeling [2] (Fig.2). For this purpose, we used COMSOL Multiphysics extended with the Heat Transfer and Chemical Reaction Engineering modules. Rheological and calorimetric signals were validated using measurements on reference materials. The instrument was explored for studying rheological changes during the cure of thermosetting systems [3] and during isothermal, nonisothermal, and flow-induced crystallization [4].

## Reference

[1] S. Kiewiet et al., RheoDSC: A hyphenated technique for the simultaneous measurement of calorimetric and rheological evolutions, *Rev. Sci. Instrum.* 79 (2008) 023905-1-7.

[2] C. Block et al., RheoDSC: Design optimisation by heat transfer modeling, *Thermochim Acta* 547 (2012) 130-140.

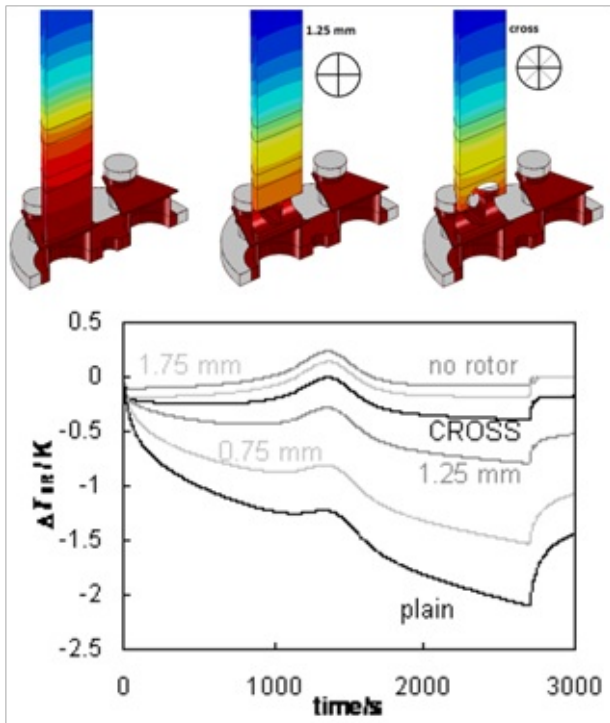
[3] C. Block et al., Time-Temperature-Transformation (TTT) and Temperature-Conversion-Transformation (TxT) cure diagrams by RheoDSC: Combined Rheometry and Calorimetry on an Epoxy-Amine Thermoset. *React Funct Polym* 73(2) 332-339 (2013).

[4] P.C. Roozmond et al., Suspension-like hardening behavior of HDPE and time-hardening superposition, *Rheol. Acta* 51 (2012) 97-109.

## Figures used in the abstract



**Figure 1:** RheoDSC setup combining a DSC cell with heat exchanger and a rheometer



**Figure 2:** RheoDSC baseline optimization by heat transfer simulations for a crosslinking thermoset