A novel dilatometer for the investigation of PVT-T-γ behavior of semi-crystalline polymers

A Novel Dilatometer for the Investigation of PVT-$\dot{T}$-$\dot{\gamma}$ Behavior of Semi-Crystalline Polymers

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Introduction

The heterogeneous microstructure of semi-crystalline polymers strongly depends on the thermal-mechanical history experienced during processing. For the prediction of material properties that are closely related to this microstructure, such as specific volume (figure 1), a realistic computational model is required. Therefore, a novel experimental set-up is developed that provides the input data for this model as a function of the thermal-mechanical history.

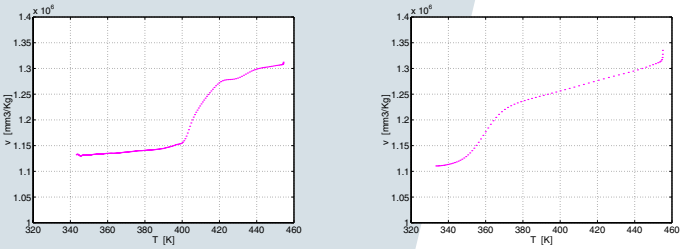

Figure 1 *Specific volume of iPP (K2Xmod, Borealis) measured at cooling rates of 0.21 [K/s] (left) and 54.22 [K/s] (right) [1].*

Methods

A dilatometer based on the principle of confined compression is designed to study the influence of the thermal-mechanical history on specific volume.

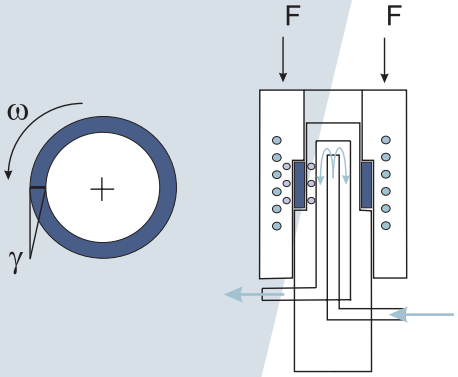

Figure 2 *Schematic representation of the dilatometer showing polymer sample (blue), cooling channels (grey), and locations for temperature measurement (violet).*

The annular shape of the sample, with a radial thickness of 0.25 mm, enables rapid cooling and applying a uniform shear deformation. Temperatures are measured at 6 locations. The axial displacement of the outer cylinder is a measure for volumetric shrinkage of the sample. To compensate for the thermal-mechanical expansion of the set-up, the displacement signal taken from a calibration measurement is used for correction. Limitations to the use of the confined compression technique [2] are investigated by comparing measurements performed on a confining fluid set-up.

Design Considerations

A thermal-mechanical analysis performed with the finite element package MARC served as a basis for the detailed design of the dilatometer.

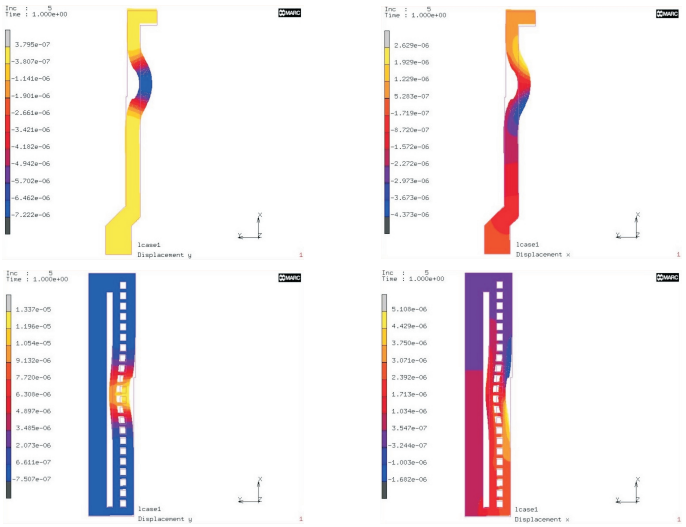

Figure 3 *Radial (left) and axial (right) deformations of piston and housing at a sample pressure of 1000 [bar].*

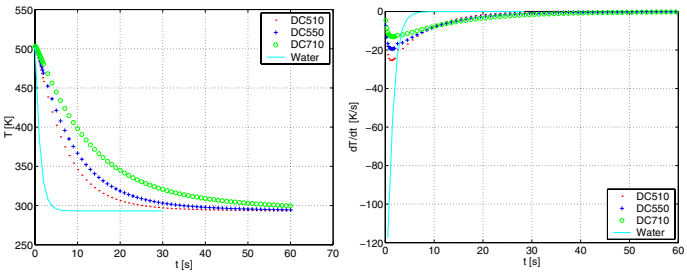

Figure 4 *Temperature history (left) and cooling rate (right) of the sample core when applying varies silicon oils and water as cooling media.*

Conclusions

A dilatometer is designed to measure specific volume as a function of thermal-mechanical history that is characterized by:

- maximum applicable sample pressure $\mathrm{P} = 10^3$ [bar]
- cooling rates can be reached to $\dot{\mathrm{T}} = O(10^2)$ [K/s]
- uniform sample deformation with $\dot{\gamma} = O(10^3)$ [1/s]

References:

[1] Zuidema, H.: *Flow Induced Crystallization of Polymers* ISBN 90-386-3021-2 , (2000)

[2] Lei, M., Reid, C.G., and Zoller, P.: *Stresses and Volume Changes in a Polymer Loaded Axially in a Rigid Die* Polymer, 29, pp. 1784 - 1788 (1988)

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