

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

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

M.H.E. van der Beek, G.W.M. Peters, H.E.H. Meijer
Eindhoven University of Technology, Department of Mechanical Engineering

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.

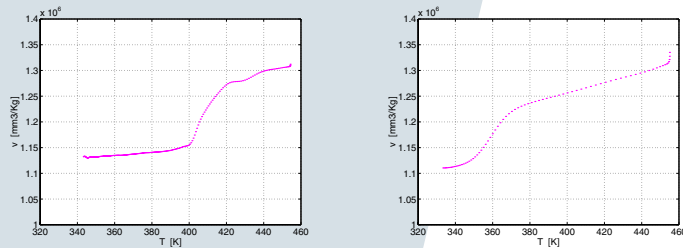


Figure 1 Specific volume of iPP (K2Xmod, Borealis) measured at cooling rates of 0.21 [K/s] (left) and 54.22 [K/s] (right) [1].

Methods

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

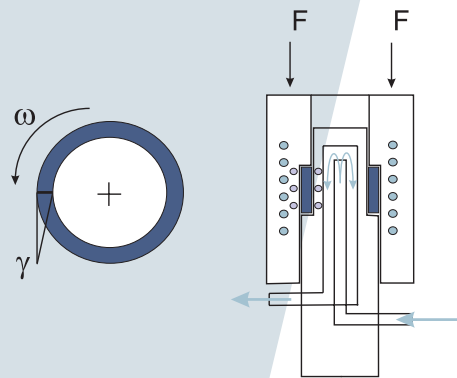


Figure 2 Schematic representation of the dilatometer showing polymer sample (blue), cooling channels (grey), and locations for temperature measurement (violet).

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.

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Design Considerations

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

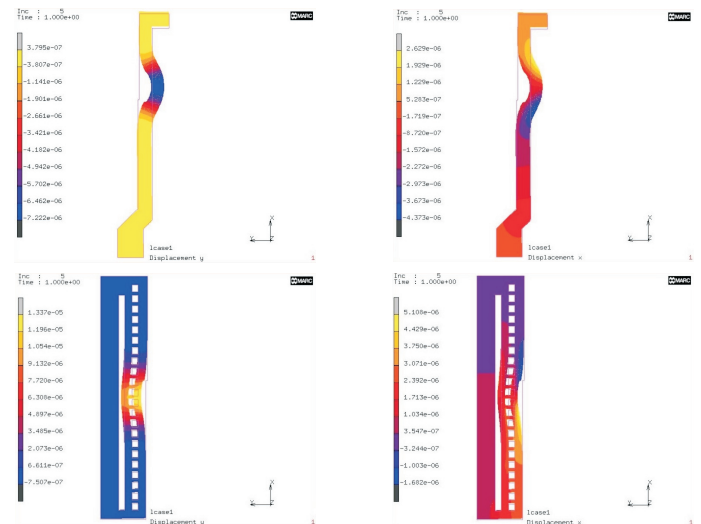


Figure 3 Radial (left) and axial (right) deformations of piston and housing at a sample pressure of 1000 [bar].

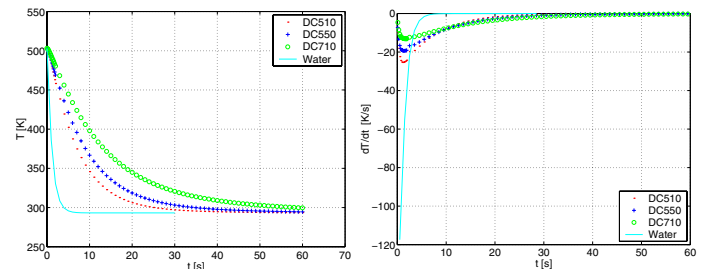


Figure 4 Temperature history (left) and cooling rate (right) of the sample core when applying varies silicon oils and water as cooling media.

Conclusions

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

- maximum applicable sample pressure $P = 10^3$ [bar]
- cooling rates can be reached to $\dot{T} = O(10^2)$ [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)