

A MULTIFUNCTIONAL PVT DEVICE FOR THE CHARACTERIZATION OF THERMOPHYSICAL PROPERTIES OF THERMOPLASTICS IN EXTREME THERMAL CONDITIONS

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Introduction

Quality and mechanical properties of thermoplastic composite parts are sensitive to the thermal history during their processing. The heat transfer modelling requires accurate knowledge of thermo-physical properties and crystallization kinetics of the matrix. It includes specific volume, thermal conductivity and specific heat in amorphous and semi-crystalline states determined ideally in conditions in terms of temperature, pressure and possibly shear similar to those existing in the process. Depending on the process and the polymer these properties must be characterized over a wide range of pressure (up to 200MPa), of temperature (up to 400°C) and for high cooling rates (more than 100K/min). Finally, heat transfer and crystallization being intimately coupled, crystallization kinetics has also to be determined in the same conditions. The characterization of all properties is obviously very time-consuming since several apparatuses have to be used and is even sometimes impossible in the range of cooling rate or pressure. Within this framework, we designed a new device named PvT-xT with the associated experimental protocol and identification methodology to estimate thermal properties as function of temperature and pressure in both molten and solid states as well as crystallization kinetics. These parameters are obtained from volume variation of a sample submitted to controlled temperate and pressure cycles. .

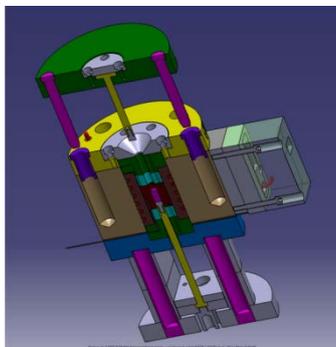


Figure 1: Sketch of the PvT-xT device

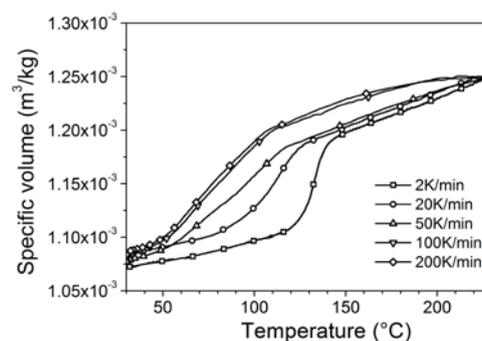


Figure 2: iPP specific volume for different cooling rates at 60 MPa

Device design and instrumentation

The PvT-xT device (Figure 1) consists in a cylindrical stainless steel moulding cavity with a mobile piston in which the polymer sample is placed. The mould is positioned between two platens of an electric press that adjusts, in real time, the position of the piston to keep applied pressure constant. The heating of the sample is ensured by induction (Roctool© 3iTech) and the cooling by a continuous circulation of cold water. PID controllers allow reaching cooling rates from 1K/min to 150K/min in the surface of the sample. Volume variations of the sample are recorded by a LVDT-type displacement sensor with a precision of 1µm.

The device has been designed to get only 1D radial heat transfers through the sample. The apparatus is instrumented with a heat flux sensor which measures lateral wall heat flux density and mould surface temperature. Three additional thermocouples positioned at 0.25mm from the mould surface verify *a posteriori* the assumption of 1D heat transfers.

Experimental set-up and heat transfer modelling

The material used for the experiment is isotactic polypropylene commercial named Eltex PP HV252 from Solvay. All thermophysical properties and crystallization kinetics of this polymer have been already published in previous papers [1, 2]. These data will be useful to validate our results. The polymer (around 0.5g) is introduced in the mould cavity, then melted at 250°C for several minutes to erase the thermal history. The cavity surface is cooled at a constant cooling rate and the piston's displacement and thermocouples responses are recorded. Heat transfer in the sample is modelled using the heat conduction equation, taking into account volume variations, coupled to a crystallization kinetic model (Eq (1)).

$$(\rho \cdot C_p)(P, T, \alpha) \cdot \frac{\partial T}{\partial t} = \frac{1}{r} \cdot \frac{\partial}{\partial r} \left[k(T, \alpha) \cdot r \cdot \frac{\partial T}{\partial r} \right] + \rho(P, T, \alpha) \Delta H \cdot \frac{\partial \alpha}{\partial t}, \quad r \in]0, R[\text{ and } t \in]0, t_f[\quad (1)$$

The spatial domain is discretized in a 2D axisymmetric scheme with moving boundaries. The mass in the mesh is constant so its thickness varies. Volume of the domain can be thus computed.

Results

Specific volume measurements have been first performed at 60 MPa for different cooling rates: 2, 20, 50, 100, 200 K/min to have a significant gradient which allows the identification of thermal conductivity and crystallization kinetics (Figure 2). When the specific volume is known, the thermal conductivity of amorphous PP can be estimated by inverse method if thermal gradient exist along the radius of the PP cylinder. The results ($\approx 0.14 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$) are in very good agreement with those presented in [1]. Following the same method, the kinetic function $K_{nak}(T)$ of the Nakamura kinetic model has been estimated for different cooling rates and the results have been validated with [2]. Measurements with PA and PEEK are currently in progress. As a conclusion, the PvT-xT mould is a multifunctional apparatus which allows with few experiments to determine with a non-invasive method the specific volume, the thermal conductivity and the kinetic parameters of thermoplastic materials.

Acknowledgements

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