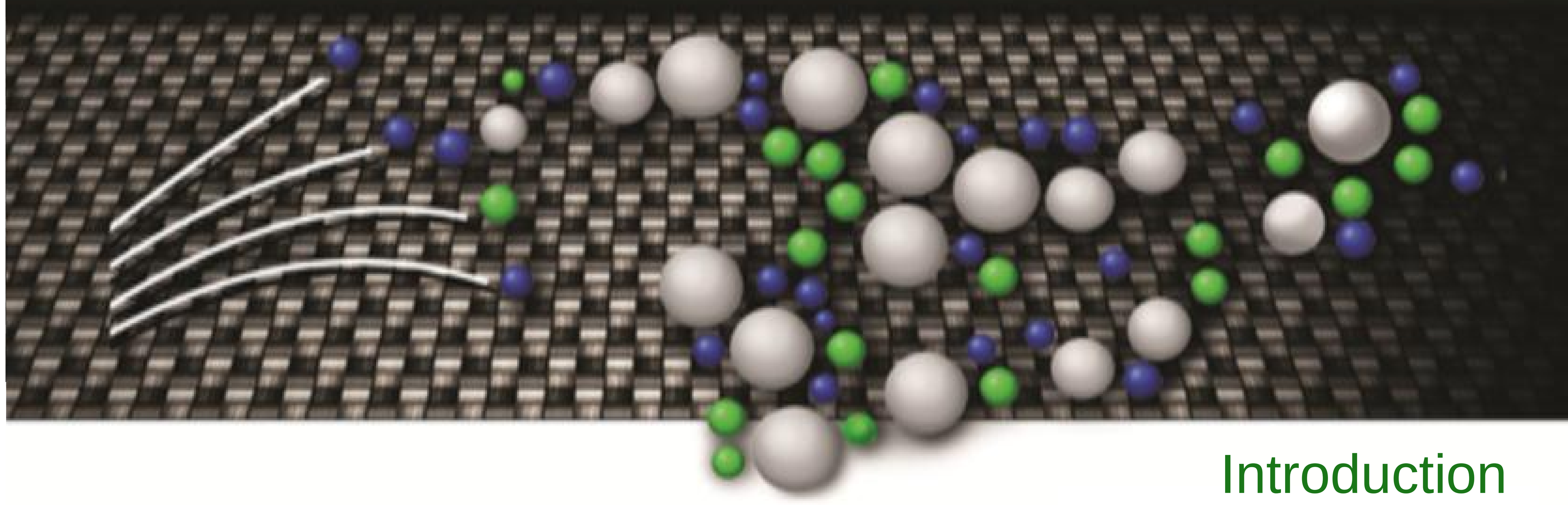
A multifunctional PvT device for the characterization of thermophysical properties of thermoplastics in extreme thermal conditions



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Introduction

ÅMechanical properties of thermoplastic composites directly depend on microstructure of the matrix, which is strongly linked to the thermal history of the forming process. For modelling purpose an accurate knowledge of the thermo-physical properties and crystallization kinetics of the thermoplastic polymer is required.

ÅThe experimental determination of these parameters involves the use of many instruments, which is time consuming. To address this issue, a home-built PvT instrumented mould, dedicated to thermoplastics (including high-performance ones), has been designed to measure and/or identify several properties from a single experiment and in thermal conditions close to the process ones.

Device design and instrumentation

ÅThe polymer sample is placed between two pistons with **pressure up to 200MPa**.

ÅThe device is heated by an induction system to **temperature up to 400°C** and allows **high cooling rate (100K/min).**

ÅThe moulding cavity is instrumented by a heat flux sensor composed of three 40εm diameter thermocouples located at 0.25, 2.5 and 6mm from the moulding cavity wall.

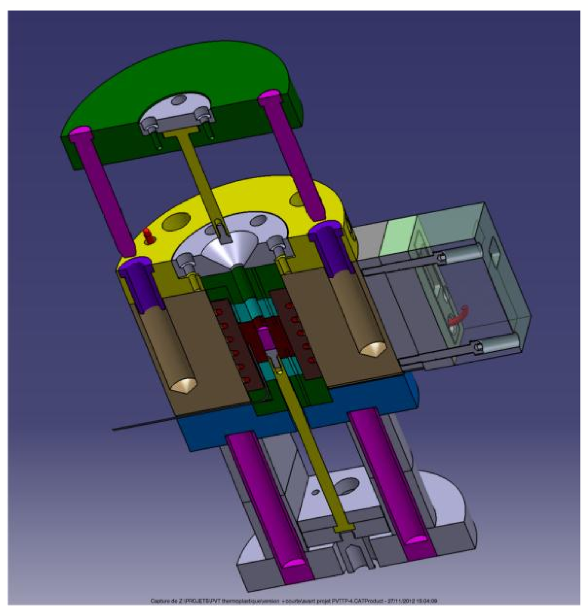

Figure 1: *Sketch of the PvT-xT device*

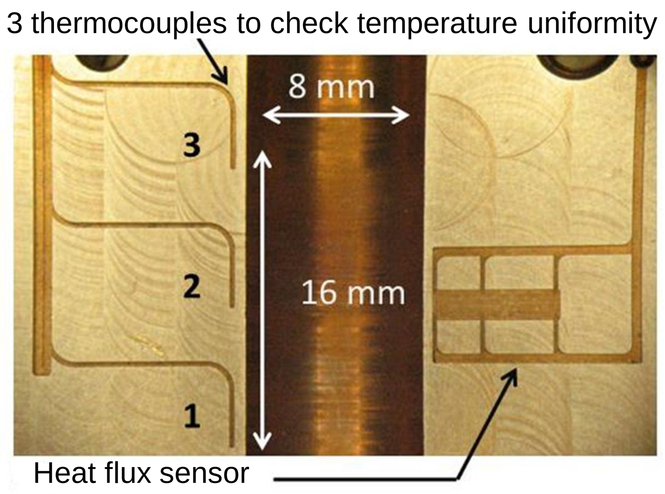

Figure 2: *Instrumentation of the moulding cavity*

Methodology and results : specific volume

ÅMaterial : isotactic polypropylene Eltex PP HV252 (MFI ¼ 11 g/10 min for 2.16 kg at 230°C) from Solvay

ÅA baseline is done with a ceramics standard cylinder to remove the impact of the mould thermal expansion.

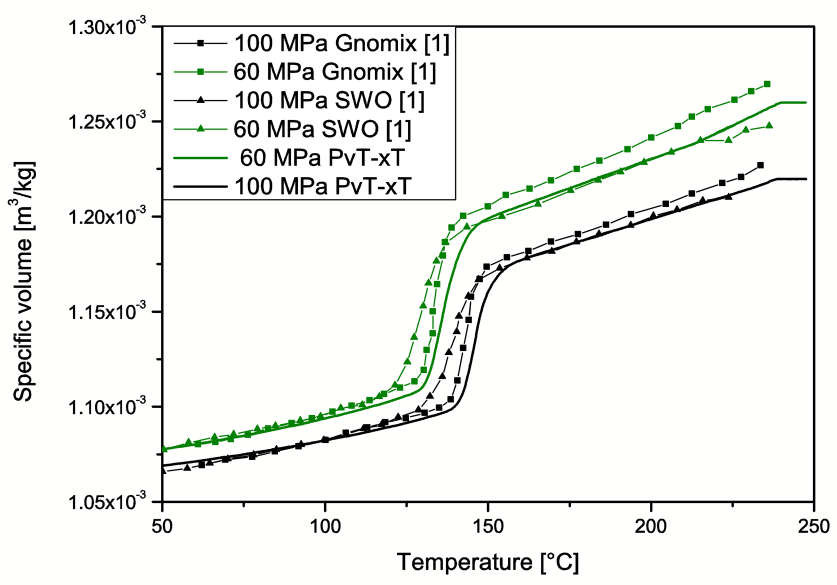

Figure 3: *iPP specific volume 2K/min, compared to Le Bot [1]*

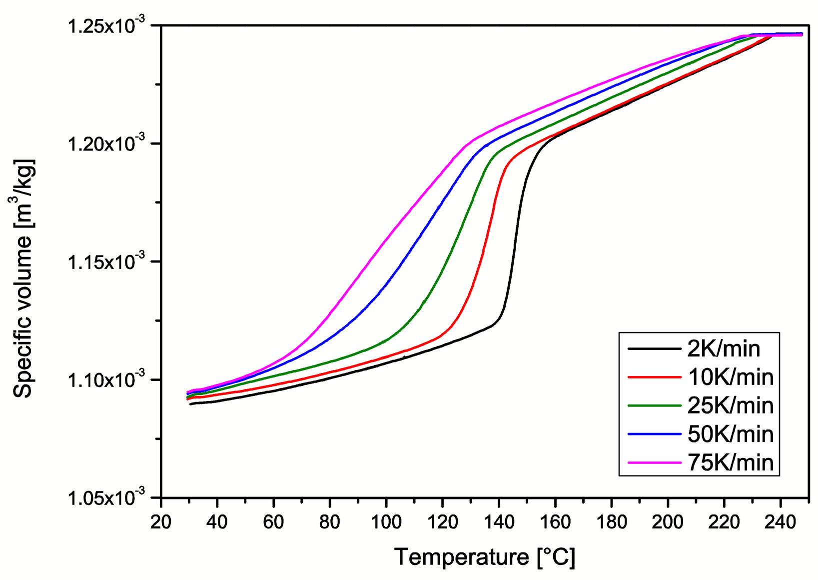

Figure 4: *iPP specific volume for different cooling rate at 60MPa*

Non-linear Heat transfer modelling

Heat transfer in the sample is modelled using the heat conduction equation, taking into account volume variations, coupled to a crystallization kinetic model.

The spatial domain is discretized in a 2D axisymmetric scheme with moving boundaries. Heat transfer is only 1D through the radius *r*.

$$\rho(P,T,\alpha).C_p(T,\alpha).\frac{\partial T}{\partial t}=\frac{1}{r}.\frac{\partial}{\partial r}\left(\lambda(T,\alpha).r.\frac{\partial T}{\partial r}\right)+\rho(P,T,\alpha).\Delta H.\frac{\partial \alpha}{\partial t}$$

Crystallization (exothermic phenomena) is modelled by Nakamura equation.

$$\frac{\partial \alpha}{\partial t}=n.K_{Nak}(T,P).(1-\alpha).\left[-\ln(1-\alpha)\right]^{\frac{n-1}{n}}$$

Thermal conductivity identification

Thermal conductivity ə(T), as shown in Figure 5, can be estimated in melted state by inverse method with a minimization criterion based on volumes. This method has the advantage to be **non-invasive**.

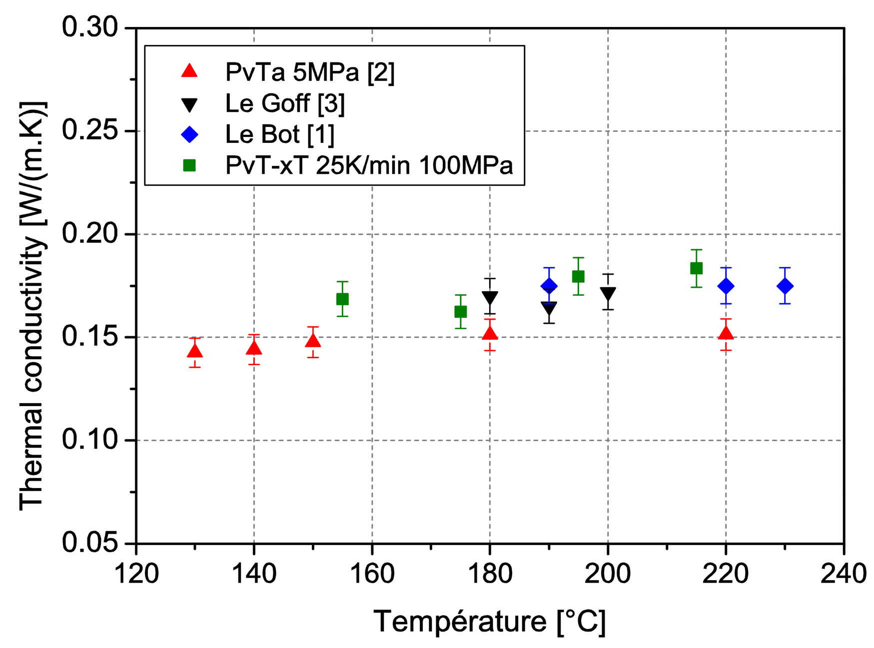

Figure 5: *Identified thermal conductivity of iPP at 50K/min 60MPa compared to previous results [1-3]*

Crystallization kinetics identification

Crystallization kinetics function $K_{nak}(T)$, as shown in Figure 6, can be estimated by inverse method following the same approach.

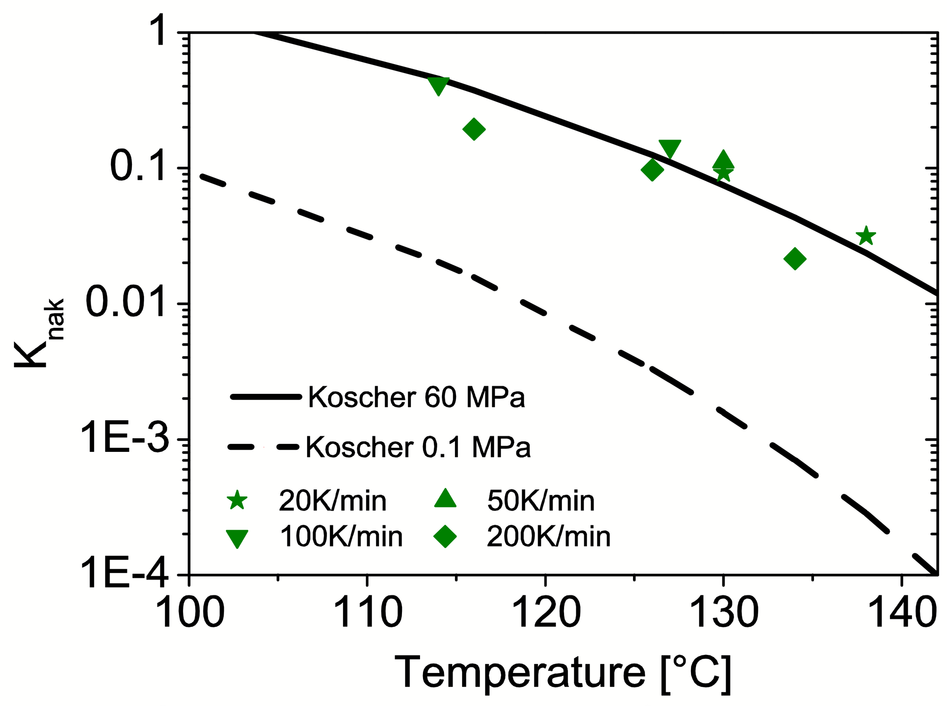

Figure 6: *Identified crystallization kinetics function and Koscher's model[4]*

After a complete identification of parameters, measured and computed volumes are well superimposed as presented in Figure 7. It highlights the strong relative crystallinity gradient through the radius of the sample at high cooling rates.

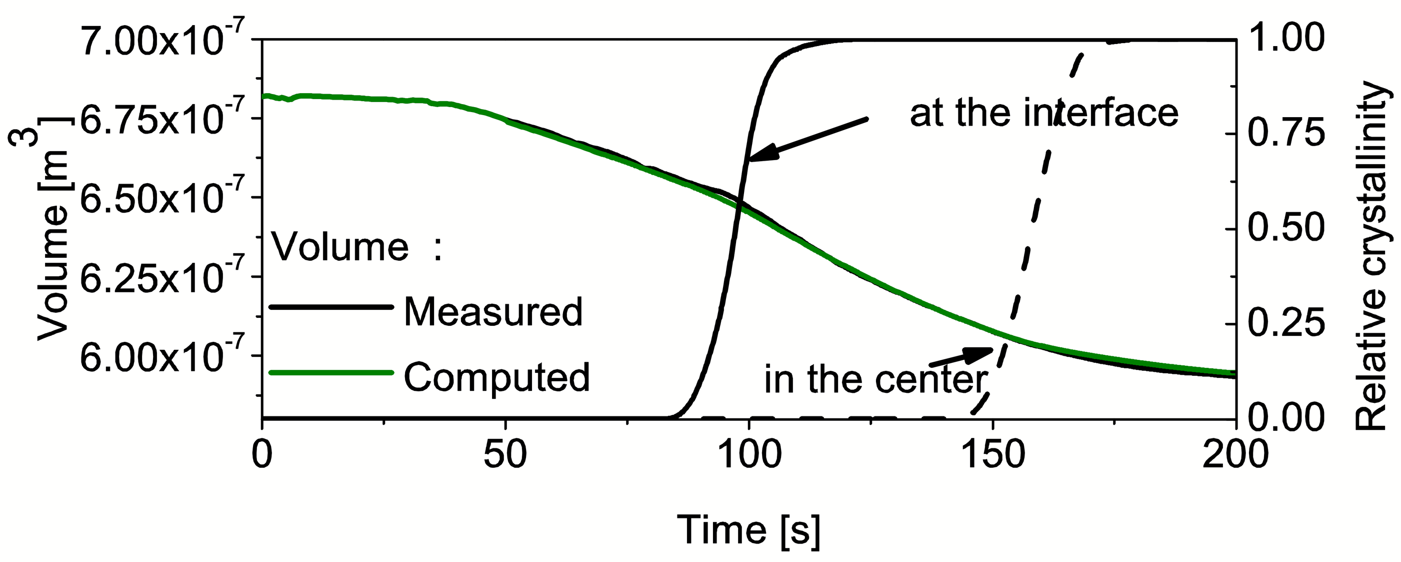

Figure 7: *Evolution of volume and relative crystallinity for 100 K/min at 60 MPa*

Conclusion and further research

ÅA new home-made PvT-xT device dedicated to high performance thermoplastic polymers is presented and validated. It can reach **temperature up to 400°C, pressure up to to 200 MPa** and **cooling rate up to 100K/min**

ÅCurrent works: the characterization of high performance polymers such as Polyamide (PA) and PolyEtherEtherKetone (PEEK).

[1] P. Le Bot, "Comportement thermique des semi-cristallins injectés. Application à la prédiction des retraits", Université de Nantes,1998
[2] X. Tardif, A. Agazzi, V. Sobotka, N. Boyard, Y. Jarny,et D. Delaunay, Polymer Testing, 2012, vol. 31, no. 6, pp. 819–827
[3] R. Le Goff, "Etude et modélisation des transferts thermiques lors de la solidification des pièces injectées en polymère semi-critsallin chargé de fibres, Université de Nantes, 2006
[4] E. Koscher, "Effets du cisaillement sur la cristallisation du polypropylène : aspects cinétiques et morphologiques," Université Claude Bernard Lyon 1, 2002.

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