



Heat transfer analysis at high cooling rate on the surface of thermoplastic parts



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ABSTRACT

Heat transfer simulation in thermoplastics processing is crucial to predict the quality of parts in terms of geometry and mechanical properties. The results accuracy depends on the knowledge of thermo-physical properties, crystallization kinetics and boundary conditions, which have to be determined in representative process conditions. In this work, we focus on the impact of high cooling rate on heat transfer and crystallization at the surface of a thermoplastic part. For this purpose, a home-made device named “Lagardère apparatus” is presented. It can reproduce cooling rate encountered in injection moulding by the sudden contact between a cold surface and a molten polymer. The first objective of this paper is the direct measurement of the thermal contact resistance used to model the imperfect contact between the polymer and the mould. The mould surface temperature and the heat flux at the interface were provided by a heat flux sensor. The specificity of this methodology was to obtain the polymer surface temperature with an optical fibre associated with a photodetector. A particular attention was made to avoid the invasiveness of the sensors on heat transfer. The second objective of the paper is the crystallization study at the surface of the polypropylene part. The experimental heat flux was compared with computed ones for which crystallization kinetics comes from Fast Scanning Calorimetry measurement and a standard model commonly used in the literature. It seemed that a very fast crystallization occurred at the surface of the part with a lower released enthalpy compared to the bulk.

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1. Introduction

Semi-crystalline thermoplastic polymers are widely used in numerous industrial sectors. They enable mass production of complex geometry parts. The most used process is injection moulding where a molten polymer is injected under pressure into a cold mould. During the cooling, heat transfer is crucial, because it influences the crystallization (kinetics, crystallinity degree and microstructure) and thus shrinkage and residual stresses. These parameters directly impact the quality of the part in terms of mechanical properties, surface aspects and geometric tolerance. The simulation is essential for the development of this process to avoid numerous trial/error tests which are costly and time-consuming. The aim of this work is to predict the polymer behaviour, to optimize cycle times and tool design.

A detailed description of heat transfer in injection moulding requires the accurate characterization of both crystallization and

thermophysical properties such as specific volume, specific heat and thermal conductivity. They are intimately coupled since the phase change is fast and exothermic. Indeed, crystallization kinetics strongly depends on the temperature and mechanical stresses such as shearing (not considered in this paper), while the temperature distribution is influenced by the crystallization enthalpy released and by the dependence of thermophysical properties to temperature and crystallinity. The main difficulty is to create characterization apparatuses working in operating conditions encountered in injection moulding process and namely in terms of pressure and cooling rate.

The crystallization kinetics is commonly investigated by standard Differential Scanning Calorimetry (DSC) to obtain parameters of overall kinetics models. However, crystallization temperature range with this apparatus is restricted by the cooling rate (<200 K/min). The latter is limited by thermal capacity of sample of several milligrams, the thermal contact resistances furnace/pan and pan/sample, and the thermal capacity of the apparatus. Consequently the crystallization cannot be described in the temperature range encountered in injection moulding, where the order of magnitude of cooling rates is few thousands Kelvin per minute at the part surface. To address this issue, the first version of Fast Scanning

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Calorimeter (FSC) has been presented by Adamovsky et al. [1], where a sample of few hundreds or even few dozen nanograms is cooled at 10,000 K/s. Nevertheless, this calorimeter had some difficulty to accurately regulate the temperature during transformations. This problem has been overcome by a power compensation apparatus developed by Zhuravlev et al. [2,3] which can reach 100,000 K/s. With the development of FSC, the crystallization can now be measured in the whole temperature range between the glass transition temperature and the melting temperature [4]. However, the question is raised about the representativeness of samples of several nanograms compared to bulk material. Is it possible that for some polymers the confinement in a very small volume caused modifications of the crystallization kinetics? Furthermore in injection moulding, the crystallization occurs also under very high pressure and shear and these factors cannot be evaluated by calorimetry. The shear dependence is classically studied with rheometers, specific hot-stages under optical microscopes [5–8], while the pressure is investigated through the specific volume variation with the crystallization [9,10]. Some apparatuses combined these two factors such as the PvT apparatus developed at TU Eindhoven [11,12].

Another requirement for an accurate simulation of the injection moulding process is the knowledge of boundary conditions. During the filling of the cavity, heat transfer at the interface between the melted polymer and the mould is large due to the wide temperature difference. This transfer is impeded by the imperfect contact that comes from roughness of the surface. An interstitial gas is trapped between asperities and its volume evolves during the cooling phase due to shrinkage. The constriction field induced by the imperfect contact is modelled by the thermal contact resistance (TCR), characterized by the temperature drop between the polymer and the mould surface ($T_{polymer}$ and T_{mould}) and the heat flux density φ crossing the interface.

$$TCR = \frac{T_{polymer} - T_{mould}}{\varphi} \quad (1)$$

The thermal contact resistance is not constant along the injection cycle due to dependences with temperature, crystallization and pressure. Some authors developed numerical models for changing solid–liquid interface [13,14]. In this paper, we will focus on the measurement of the TCR. Previous authors [15–17] estimated the mould surface temperature T_{mould} and the heat flux density φ at the interface by inverse method [18]. The main difficulty comes from the polymer surface temperature measurement $T_{polymer}$, because the direct measurement using standard thermocouples, even if sometimes used, is not reliable for a surface temperature measurement. To avoid this issue, an indirect method was proposed by Delaunay et al. [15] and Le Goff et al. [16] where the polymer surface temperature was obtained from the computation of heat transfer in the part. The direct measurement of the TCR was presented for the first time by Bendada et al. [17], where $T_{polymer}$ was measured with an infrared hollow waveguide pyrometer. We will see in this paper that even with this methodology some precautions have to be made to avoid the intrusiveness.

Within this framework, the first objective of this paper is the direct measurement of the thermal contact resistance at the interface polymer/mould. For this purpose, the cooling rate and the heat flux density at the interface polymer/mould representative to those observed in injection moulding process can be reproduced by the sudden contact between a cold surface and a molten polymer. It is possible using a home-made device named “Lagardère apparatus”, which allows the study of heat transfer at the surface a bulk thermoplastic part. The additional information of the polymer surface temperature is obtained with an optical fibre associated to a photodetector. A particular attention is made to avoid

the invasiveness of measurements. The second objective of this paper is to study the crystallization at the surface of a polypropylene part. The experimental heat flux at the polymer/mould interface is thus compared with computed ones, considering either the crystallization kinetics obtained by FSC and the already existing standard model.

2. Description of the apparatus

2.1. Measuring principle

The objective of the “Lagardère apparatus” is to cool a molten polymer with a cooling rate representative of the injection moulding process, without taking into account pressure and shear effects. For this purpose, the molten polymer is put into contact with a cold metallic surface.

As presented in Fig. 1, the polymer sample was a cylinder, 8 mm thick and 50 mm in diameter, positioned in an elastomer ring with a low Young’s modulus. It allowed the thermal insulation of the sample lateral side and prevented leakage of the polymer after its melting. The sample was melted by two heaters, which were regulated with PID controllers. On the other side, the piston was cooled and laterally isolated. A Zinc Selenide window was used as thermal screen to avoid the thermal invasiveness of the instrumentation as detailed in Section 2.2. After the removal of the thermally regulated upper plate, the cold piston impacted the molten polymer using a pneumatic actuator, as presented in Fig. 2.

The contact was detected by a LVDT displacement sensor. At this time heat transfer at the interface between the polymer sample and the piston was very high. A heat flux sensor, positioned in the piston, provided the piston surface temperature and the heat flux density at the interface. However, as the contact was imperfect, the polymer surface temperature was required to measure explicitly the thermal contact resistance. This information was provided by an optical fibre associated to a photodetector. A K-type thermocouple was also positioned at the half thickness of the sample, to get a boundary condition for the heat transfer model presented in Section 4. All sensors responses were recorded on a Yokogawa© DL 750 multichannel acquisition system.

2.2. Polymer surface temperature measurement

2.2.1. Principle and positioning of the optical fibre

The polymer surface temperature measurement was achieved by an optical fibre connected with a photo-detector. The fibre was polycrystalline infra-red fibre, with a core in silver bromide (AgBr), chosen for its transmission range between 4 and 18 μm , corresponding to the temperature range. It was mechanically protected by a PEEK sheath and its ends were framed by stainless steel. The optical fibre was connected to a MCT photodetector (Mercury Cadmium Telluride) from IR associate©, selected to be sensitive to the same range of wavelengths. It was cooled with liquid nitrogen.

However, positioning of the optical fibre at the piston surface raised the problem of invasiveness on the measurement of the polymer surface temperature. Indeed the temperature in front of the fibre is different from the one in front of the steel piston due to large thermophysical properties differences (Table 1). To quantify this deviation, the contact between a molten polymer at 220 °C and the piston at 30 °C, corresponding with the temperature range for the polypropylene used in this study, was simulated with the Comsol© FEM software. The model used a 2D axisymmetric scheme. Lateral heat losses were neglected due to insulation and a standard thermal contact resistance of $5 \cdot 10^{-4} \text{ m}^2 \text{ K/W}$ was considered between the polymer and the piston often encountered

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