

Short communication: Material behaviour

## Probing solidification kinetics of high-density polyethylene during injection molding using an in-situ measurement technique



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### ABSTRACT

The largest part of an injection molding cycle is occupied by melt cooling, which significantly influences the properties of the final parts, especially for crystalline polymers which undergo both solidification and crystallization processes simultaneously. Based on non-isothermal crystallization characterization, solidification kinetics of high density polyethylene was investigated via an *in-situ* measurement of in-cavity temperature profiles throughout the injection molding process. Existence of the turning point in the cooling curve of  $\ln \theta$  vs.  $\ln t$ , which can be employed for estimation of the minimum cooling time, was for the first time validated experimentally. Good agreement was achieved through the comparison between experimental observations and theoretical predictions using the enthalpy transformation method (ETM). The present study will be instructive for the optimization of processing variables, and gives insight into the formation of various crystalline structures in injection molded articles, as well as forecast of cooling time of injection moldings of crystalline polymers.

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### 1. Introduction

Transient heat transfer problems involving solidification (melting) are also known as “moving boundary problems” or “phase change problems”, which were initially raised by Stefan in 1890s [1]. They are still practically significant in modern engineering areas, such as the in-mold solidification of metal or polymer, the cooling of large masses, the freezing of food, and so forth. However, owing to the very nature of non-linearity caused by the fact that the interface between liquid and solid is always moving, with latent heat liberated (absorbed) at the phase interface, few exact closed-form solutions are currently available, except for some simplified or idealized systems [2,3]. Among the

many numerical solution methods (e.g., variational, enthalpy, nodal integral, boundary immobilization, etc.), the enthalpy transformation method (ETM) has recently proved to be an efficient method to treat the phase-change heat conduction issues of crystalline polymers [2,4].

Injection molding, one of the most widely employed fabrication techniques for thermoplastic polymers, presently accounts for over 1/3 of all plastics processed. During injection molding, the material is subject to high pressure, high shear rate as well as high cooling rate, etc [5–7]. All these factors influence the final property distribution across the injection molded article. For crystalline polymers, crystallization always takes place during the melt cooling (solidification) process, which has considerable influence on both productivity and quality of the final products [8–10]. In addition, the crystallization kinetics will determine the time needed for melt cooling as well as the total cycle

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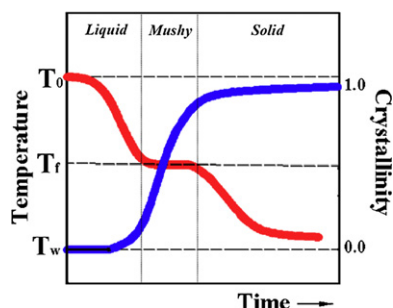
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time [11]. In this respect, optimization of the processing variables should also be concentrated on the reduction of cooling time, which is of practical importance for injection molded parts during industrial processes typically characterized by relatively large shot volumes and part thickness.

A typical cooling curve of crystalline polymer, consisting of three stages, is schematically illustrated in Fig. 1. The temperature falls drastically from the initial temperature ( $T_0$ ) in the liquid cooling stage, and subsequently drops to the phase-change temperature range, where melt crystallization occurs. Recent investigation suggests that the liberated latent heat during crystallization could lead to a remarkable increase in the melt temperature [3]. After the phase transition, the temperature continues to drop until reaching an equilibrium temperature, which is primarily determined by the mold temperature ( $T_w$ ). Unlike the first and the third stages, the phase-change stage is composed of both liquid and solid states (namely, mushy zone), with the phase interface varying with time [10]. Meanwhile, there is a significant increase in crystallinity at the occurrence of the phase transformation.

Considering the variant factors that simultaneously affect the morphology and final properties [12], it is still difficult to understand the global phenomenology involved during various stages of injection molding. Investigation on the solidification phenomena and crystallization kinetics under processing conditions will be crucial to a more complete understanding of molecular orientation and microstructure evolution, as well as the optimization of thermal or pressure history during molding [13–17].

To date, the use of in-line and on-line techniques turns out to be an effective way to monitor solidification or crystallization phenomena during real processing operations, which offers deeper insight into the investigation of structural development. The methods for experimental determination of in-cavity transient temperature profiles within the injection molded parts are commonly implemented via optical [18], infrared [19,20] and ultrasound [21] sensors or detection equipment. Carrubba et al. [5] recently proposed an in-line method called “indentation test”, which was designed to obtain information on the crystallization process (esp. for slowly crystallizing polymers) during injection molding. It was found that the disappearance of the liquid phase is primarily due to the transient heat conduction through the thickness of the mold, and



**Fig. 1.** Schematic representation of a typical cooling curve (in Red), and correlation of crystallinity versus elapsed time for crystalline polymers (in Blue). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

difference in cooling rates is a key factor dictating the formation of hierarchical structures in the injection molded parts of crystalline polymers [5–7,13,16]. However, studies on the transient heat transfer with phase-change effects (esp. for crystalline polymers) are still inadequate.

The objective of the present work is to explore the solidification kinetics of HDPE during injection molding using in-situ measurement of the in-cavity temperature decay, and experimentally elucidate the existence of a turning point on the cooling curves of crystalline polymers. The experimental results were compared with theoretical data obtained using ETM [1,2,4]. This work will be of technological significance to the optimization of processing variables, and supplies good insight into the formation of various crystalline structures in injection molded articles, as well as the forecasting of cooling time of injection moldings of crystalline polymers.

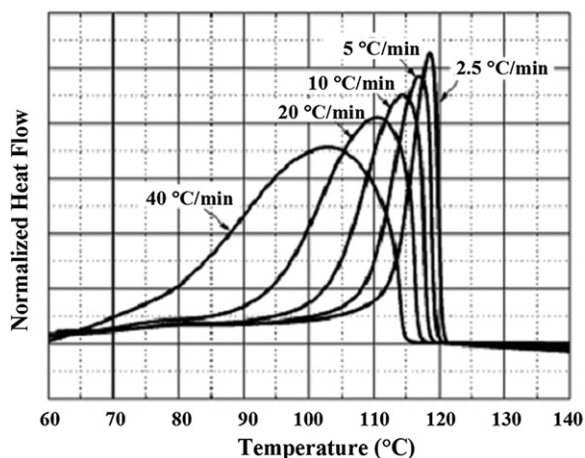
## 2. Experimental

### 2.1. Material

The material used in the present study was high density polyethylene (HDPE, Model: DGDA-6098), kindly provided by Qilu Petrochemical Co. Ltd, China, with a melt flow rate (MFR) of 0.9 g/10 min (2.16 kg, ASTM-D1238). The material has a weight-averaged molecular weight ( $M_w$ ) of  $5.63 \times 10^5$  kg/mol, and polydispersity index (PDI) of 14.7, as determined by gel permeation chromatography (GPC).

### 2.2. Differential scanning calorimetry (DSC) measurement

Measurement of non-isothermal crystallization behavior was carried out using a differential scanning calorimeter (DSC), Model: Q-200, supplied by TA Instrument Inc., U.S.A. A sample weighing 5–7 mg was initially heated to 210 °C at 10 °C/min, and kept for 4.5 min to erase the thermal history. Then, it was cooled to ambient temperature at various cooling rates of 2.5, 5, 10, 20 and 40 °C/min. The phase-transition temperature range coupled with the enthalpy of crystallization [4] was thus obtained (cf. Fig. 2).



**Fig. 2.** Normalized non-isothermal crystallization thermograms of HDPE scanned at various cooling rates.

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