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Test method

Analytical review of some relevant methods and devices for the determination of the specific volume on thermoplastic polymers under processing conditions

Sergio A. Suárez*, Alberto Naranjo, Iván D. López, Juan C. Ortiz

Instituto de Capacitación e Investigación del Plástico y del Caucho (ICIPC), Carrera 49 #5 Sur – 190 Bl. 37, Medellín, Colombia

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ABSTRACT

This article reviews some of the most representative methods and devices to measure the specific volume in polymeric materials found in literature. Each method is described and a comparison between the experimental conditions and typical injection molding processing conditions is discussed. In general, the main difficulty with specific volume measurements is determination of the temperature distribution through sample thickness, particularly when achieving typical processing conditions is intended. Therefore, the necessity of developing a new device which allows direct measurements of pressure and temperature at various positions along the sample thickness in order to calculate the cooling condition was identified.

1. Introduction

1.1. Aim of the work

Around 30% of plastic materials are processed by injection molding [1]. Production sectors such as automotive, health and electronics use injection molding to produce most of their plastic components, which are manufactured tor high dimensional standards. Quality features are achieved by well-tuned processes and well-designed molds, both as the result of correct prediction of deformation and shrinkage associated with material thermodynamic properties. Predictions are obtained by mathematical models that rely on experimental measurements of these properties. However, shortcomings have been identified in the material characterization process. Therefore, there is still low reliability on software performance when it comes to mold design for injection molding.

Nowadays, several procedures and devices are available to determine the specific volume of thermoplastic polymers. However these procedures can be improved in order to obtain more exact mathematical models that can be used as a starting point for mold design. A critical review of the state of the art about existing methods and devices is presented. The comparison between experimental and processing conditions is established for each device.

1.2. Background

The injection cycle is divided into three main material transformation stages: plasticizing the material, filling the cavity and cooling the plastic. In the first stage, the material is melted in the plasticizing unit. In the second stage, the melt is rapidly pressed to fill the mold cavity and, finally, the part is cooled within the mold until it reaches room temperature. Thus, the plastic material is initially at temperatures above 473 K, then a high pressure is exerted to fill the cavity (around 1000 bar). During filling, the material undergoes shear rates over 1000 s^{-1}, and it is rapidly compressed, as will be discussed further in this article [2]. Cooling rate is not homogeneous throughout the sample, reaching values up to 3000 K/min close to the mold walls and 60 K/min near the center of the thickness. Fig. 1 exhibits the results obtained by Naranjo et al. [3] for the temperature and cooling rate at different points (from the wall to the center) for a semi-crystalline material.

Luyé et al. [4] presented the influence of the temperature gradient on specific volume by an experiment in which the specimen is cooled at a rate of 5, 10 and 20 K/min, starting from 493 K to 453 K. After reaching 453 K, specific volume is registered while the

* Corresponding author.
E-mail addresses: icipc@icipc.org (S.A. Suárez), anaranjo@icipc.org (A. Naranjo), ilopez@icipc.org (I.D. López), jortiz@icipc.org (J.C. Ortiz).

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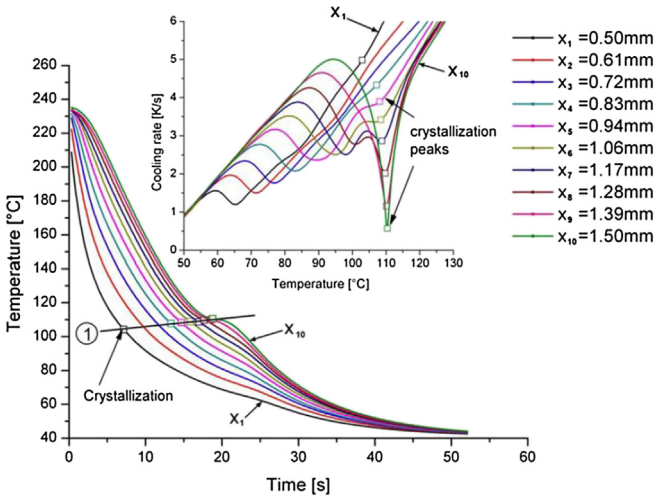

Fig. 1. Cooling rate and temperature variation versus time and part thickness, measured by Naranjo [3].

temperature is kept constant. The results show that the volume becomes constant as temperature stabilizes across the sample. Fig. 2 displays the effect of the temperature gradient on the specific volume measurement for 5 and 20 K/min. The impact of cooling rate on specific volume was studied by Kowalska and Sikora [5,6], whose work will be discussed later in the article.

From the above, an isobaric cooling condition can be stated as the appropriate way to determine the specific volume reproducing the typical injection molding processing conditions. Some of the existing devices to measure specific volume involve direct temperature measurements over the specimen, while others carry out calculations by using heat transfer models.

In this article, several devices found in the literature for the determination of the specific volume of polymers are presented. In each section, the measuring method is described. Furthermore, the advantages and disadvantages of each device with respect to temperature, pressure, cooling rate and density are discussed.

2. Confining – fluid method

The confining fluid measuring method of PVT involves the immersion of a specimen in a fluid, usually mercury. This fluid is heated and pressurized. The volume change is recorded by a bellows, which is connected to the cavity in which the sample is immersed. Zoller et al. [7] developed a commercial device to measure pvT known as GNOMIX, which has been used as a starting point for the design of new devices.

2.1. Eindhoven University of Technology (confining – fluid)

2.1.1. Description

Zuidema et al. [8] recently developed a device to measure

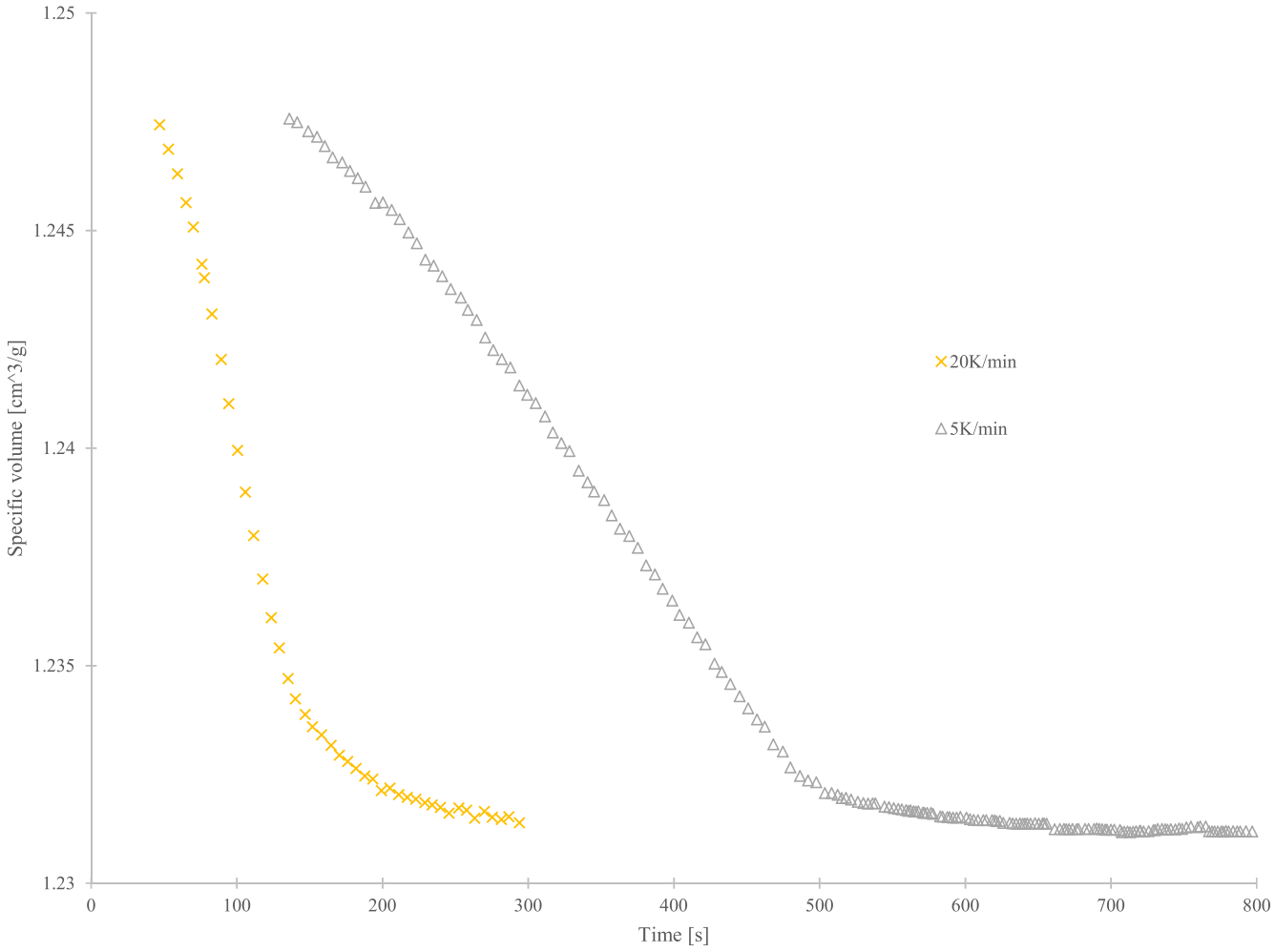

Fig. 2. Effect of temperature gradient in measuring the specific volume at different cooling rate in a temperature range of 493 K – 453 K. Data taken from Ref. [4].