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Visualization of Elongation Measurements Using an SER Universal Testing Platform

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Abstract:
A Sentmanat Extension Rheometer represents one out of a few experimental devices for the measurement of elongational viscosity of polymer melts. However, the appropriateness of this technique for individual polymer materials is not sufficiently apparent and in some case is disregarded or ignored. The proposed visualization technique is based on imprinting painted pattern from the inner surface of the studied polymer samples onto the counter-rotating drums. Digitization of the imprinted pattern gives a possibility to evaluate a degree of sagging, incorrect fixing of rectangular polymer samples to the drums, possible appearance of sample inhomogeneity (variance in thickness, bubbles, etc.). The presented visualization technique is demonstrated using branched LDPE Escorene. Two various imprinted patterns are applied. First, the upper and lower contours are charted on a prepared sample with the aim to determine the sample shapes during stretching and to compare them with the theoretical ones. Second, the inclined rectangular grid pattern is charted for evaluating possible inhomogeneity of the sample.

Key words:
elongational viscosity, SER Universal Testing Platform, polymer melts, LDPE

1 INTRODUCTION

The onset of more elaborated and sophisticated devices for the measurement of uniaxial elongational viscosity of polymer melts dates back to the second half of the 70s' when two different principles were projected into the Meissner [1, 2] and Münstedt-type [3] extension rheometers. Recent trend is to substitute the two 'classical' ingenious extensional rheometers in many rheometric laboratories by a new generation of devices such as the Filament Stretching Rheometer (FSR) [4], its modification for high-temperature measurements of polymer melts by Bach et al. [5] and Chellamuthu et al. [6], or the Sentmanat Extension Rheometer Universal Testing Platform (SER) [7–10]. Analysis of acceptability of these new devices for the measurement of elongational viscosity is very scarce as well as their mutual comparison, and their comparison with the Meissner- and Münstedt-type rheometers. This need is especially evoked by the fact that usually not all attributes characterizing the 'classical' devices are fulfilled by newly developed ones. In the context of these attributes it is possible to mention the problem of the FSR rheometer, where a feed-back loop is applied to keep a constant strain rate at the mid-filament diameter [11], or possible variability in temperature and appearance of sagging when the SER rheometer is used. This stimulates a question how to classify (and according to which criteria) materials from the viewpoint of their measurability in the individual extension rheometers. There is a series of factors significantly influencing a proper measurement of viscosity starting with a suppression of shear traces (dominantly generated by a way of material fixing), sample preparation, temperature uniformness, reduction of sagging, etc.

The focus of this contribution will be paid to an applicability of the SER rheometer (Figure 1). Recently, a series of papers (prevailingly based on numerical analysis) appeared questionning an applicability of this device, see for example Yu et al. [12] and references therein. Based on experimental findings, Hoyle et al. [13] compared efficiency of the SER, FSR and Cross-Slot Extensional Rheometer (CSER) using three different PE materials. However, samples of thickness 1 mm were used in the SER rheometer, which is the upmost pre-

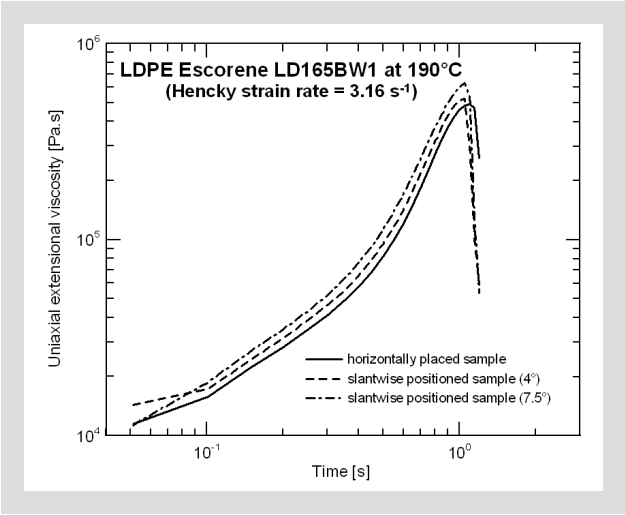

Figure 12: Difference in elongational viscosities for the samples placed horizontally and in slantwise positions.

decisive factor for which the materials are a priori excluded. This ever-presenting factor can be partially reduced by a combination of two measures: (i) acceleration of sample fixing and (ii) reduction of sagging effect as discussed above. However, sagging always results in uneven distribution of material in a vertical cross-sectional area, in other words, in a non-constant thickness of a sample along its width. The degree of unevenness then restricts the possibility of a determination of uniaxial elongational viscosity using theoretically derived formula. This degree can be evaluated by the visualization technique as presented in the preceding Section. It is not possible to expect the same degree of sagging even for the equally prepared samples as the temperature field cannot be precisely replicated in the rheometer oven (starting temperature after insertion a sample into the oven varies).

Second factor influencing quality of measurement of uniaxial elongational viscosity, is a presence of shear flow caused by way of fixing the sample to both drums (Section 5). This factor can be overcome by elimination of an initial stage of every measurement (pre-elongation region). Shear flow is also present due to asymmetrical stretching of a sample across its thickness. The tensile force initiated by both drums has an immediate impact to a lamina adjacent to the drums. However, the impact of the tensile force mildly attenuates for exterior lamina where stretching is not in an exact parallel direction due to the drums' curvature. Third factor is represented by inhomogeneous distribution of temperature field in an oven of the rotational rheometer. This factor subjects to geometry of the individual ovens and a location of air inlet(s) and outlet(s). In this case a usage of thermocouples for temperature settings seems to be inevitable [15]. Fourth factor represents a proper preparation of the rectangular samples. Variance in thickness should be reduced as much as possible (not exceeding 3 % [16]) and appearance of bubbles should be suppressed. 'Frozen' stresses should be also taken into consideration during sample preparation.

Influence of each of the four factors should be minimized when uniaxial elongational viscosity is measured. The above presented visualization technique gives a hint of how to evaluate the materials with respect to their measurability using the SER rheometer. This technique reveals a slantwise position of fixed sample, a degree of sagging, unevenness of sample thickness along the vertical cross-sectional area, possible appearance of sample inhomogeneity. The imprinted traces of the upper and lower sample edges on both drums determine a deviation from a theoretically expected course of sample exponential reduction in its width. According to this deviation and the deviation from a 'vertical' symmetry, it is possible to evaluate an appropriateness of the SER rheometer for measurement of uniaxial elongational viscosity of a chosen material. Mere reproducibility of the repeated measurements has no relation to the discussed appropriateness. For the proper determination of elongational viscosity, adequate check of basic factors (introduced above) participating in asymmetric elongation of the polymeric samples is inevitable as well as an analysis of extent of the pre-elongation region. For non-moderate extents an applicability of the SER rheometer is questionable.

The present method requires approximately five more minutes in addition to a classical time period necessary for an evaluation of single elongational measurement supposing that the evaluation software package is already installed and a drawing utensil for grid chart is prepared. The method should be used in two steps: (i) primary test for chosen common temperature and extension rate indicating (im)possibility to measure elongational viscosity of a studied material using the SER rheometer and (ii) in the case of a positive result in the step 1, the method should be applied for setting the limiting values of the measurements if necessary. This means to evaluate a limiting maximal value of temperature (with respect to sagging in combination with possible degradation) and a minimal extension rate corresponding to a given temperature value. The maximal extension rate is given by the construction of the SER rheometer as discussed in Sentmanat [8]. A criterion responsibly categorizing materials with respect to their applicability to the SER rheometer would represent an optimal solution. However, due to complexity of the whole problem and an actual unavailability of the experimental data covering a broad range of polymer materials, this is beyond the scope of the present contribution. For more detailed analysis there is a necessity of application of more polymer materials including those that are a priori unsuitable for an application of the SER rheometer.

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