Measurement of Elongational Viscosity of Polymer Melts Using SER Universal Testing Platform

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Abstract:
The measurement of elongational viscosity still evokes a series of problems in comparison with the relatively well-established measurement of shear viscosity. Recently new techniques have appeared enabling measurement of elongational viscosity with the samples for which the aspect ratios of their geometrical shapes (i.e. length vs. width (diameter)) can attain moderate values, i.e. not necessarily of a longitudinal character as in the case of earlier techniques. The aim of this contribution is to experimentally demonstrate the invariantness of transient uniaxial elongational viscosity measured with respect to a rectangular shape and thickness of LDPE samples using a SER Universal Testing Platform fixed in an Anton Paar MCR 501 host system. The width of the samples was varied within the range 2.1-12.7 mm and thickness altered within 0.1-1 mm. An advantage of fixing polymer samples directly to both drums (if possible) over the application of clamps is documented.

Zusammenfassung:
Die Messung der Dehnviskosität ist weiterhin mit einer Reihe von Fragestellungen verbunden im Vergleich mit den sehr gut etablierten Messungen der Scherviskosität. Mit Hilfe von kürzlich entwickelten neuen Methoden kann die Dehnviskosität ermittelt werden, bei denen das Aspektverhältnis (Länge zu Breite bzw. Durchmesser) der Probe moderate Werte annehmen kann, d. h. bei denen nicht notwendigerweise sehr lange Proben verwendet werden müssen. In dieser Arbeit wird die Unabhängigkeit der transienten Dehnviskosität von Proben mit einem rechteckigen Querschnitt in einfacher Dehnung mit dem SER in Kombination mit dem MCR 501 gemessen. Die Breite der Probe wurde variiert und lag im Bereich von 2.1 bis 12.7 mm. Ihre Dicke variierte zwischen 0.1 und 1.0 mm. Der Vorteil der Befestigung der Polymerprobe direkt auf den Trommeln im Gegensatz zu der Verwendung von Klemmen wird gezeigt.

Résumé:
Mesurer la viscosité extensionnelle présente encore une série de problèmes qui contrastent avec la mesure relativement bien établie de la viscosité de cisaillement. Récemment, de nouvelles techniques ont vu le jour, permettant la mesure de la viscosité extensionnelle d'échantillons dont les formats géométriques (c-à-d longueur sur largeur (ou diamètre)) peuvent atteindre des valeurs modérées, c-à-d ne présentant pas un caractère longitudinal, comme dans le cas des techniques précédentes. Le but de cette contribution est de démontrer expérimentalement l'invariance de la viscosité extensionnelle uniaxiale transitoire d'échantillons de LDPE par rapport à leur forme rectangulaire et à leur épaisseur, en utilisant une Plateforme de Test Universel SER fixée à un Anton Paar MCR 501. La largeur des échantillons varie entre 2.1 et 12.7 mm et l'épaisseur entre 0.1 et 1 mm. L'avantage de fixer les échantillons polymères directement sur les deux tambours (si possible) relativement à l'utilisation de clips de fixation est démontré.

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

1 INTRODUCTION

When using the 'classical' devices for measurement of elongational viscosity of polymer melts (Meissner [1], Meissner and Hostettler [2], Münstedt [3]) it is necessary to prepare longitudinal samples (length considerably exceeds width (diameter)) of polymeric materials. Necessity of this requirement has been eliminated with the appearance of novel experimental devices such as the filament stretching rheometer (McKinley and Sridhar [4], modification for high-temperature measurements of polymer melts Bach et al. [5], Chellamuthu et al. [6]) or

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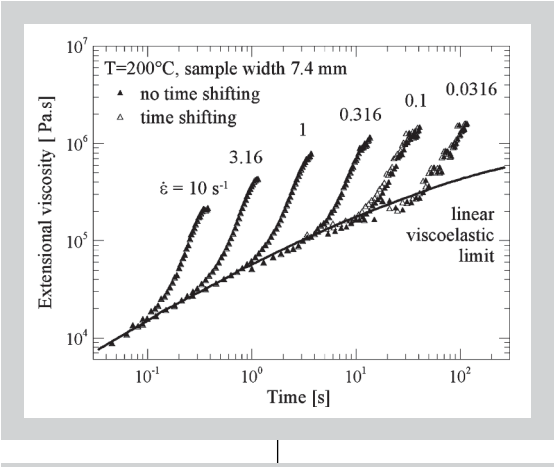

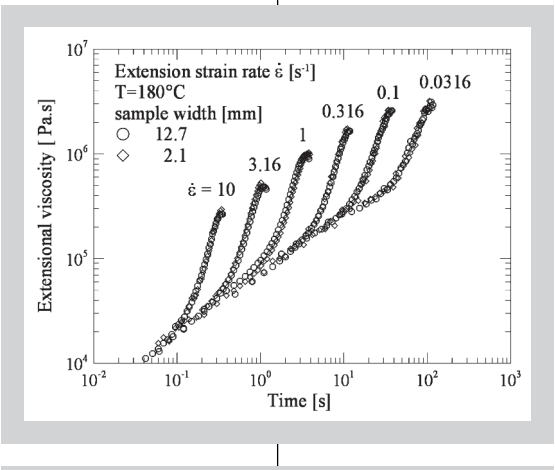

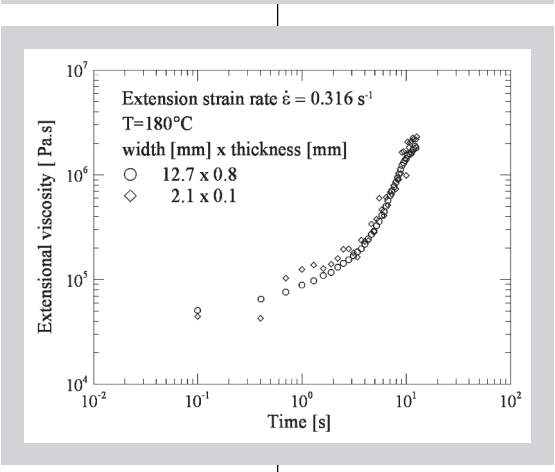

Figure 9:
Comparison of uniaxial extensional viscosity with and without time shifting at a temperature of 200°C.

Figure 10:
Comparison of uniaxial extensional viscosity measured with the samples of two widths (2.1 and 12.7 mm) at a temperature of 180°C.

Figure 11:
Comparison of uniaxial extensional viscosity measured with the samples of two distinctive dimensions at a temperature of 180 °C.

individual parameters (strain rate, temperature, sample width) was carried out at least three times, prior to each temperature run the oven was pre-heated for at least 1 hour. The temperature of each sample after fixing was approached from below to prevent the material from possible degradation and more pronounced sagging.

The same conclusion is valid even for distinctly different widths of rectangular samples, i.e. 2.1 and 12.7 mm (the respective widths differ by a multiplicative factor of 6) as depicted in Figure 10. Figure 11 presents the result obtained for two samples that were remarkably different not only in width (2.1 versus 12.7 mm) but also in thickness (0.1 versus 0.8 mm). These experimental results varies from the theoretical conclusions by Yu et al. [16] in which they restricted the dimensions of the samples applicable for measurements using the SER U.T.P.

The homogeneity of the samples to be measured always plays a crucial role. For the Münstedt-type rheometer this issue has recently been discussed in Burghelea et al. [15]. For the SER unit adequate homogeneity of the material itself and also the more or less constant thickness of the sample are very important. In our experience accurate and reproducible measurements can be achieved if the thickness across the whole prepared sample only varies by up to 3 % (i.e. not ±3 %, but up to 3 %).

Sometimes the material being measured ruptures during passage over a clamp. The exact location of rupture can easily be determined with the help of the camera system. Based on the results reported above this singularity can be removed by changing the dimensions of the rectangular sample. The use of a thinner sample also leads to more rapid heating of the whole sample to the temperature required, and thus reduces the time during which the sample is in the oven at rest, and consequently the degree of sagging.

Sagging is also very much influenced by the time interval during which a sample is fixed to both drums, as longer fixing means a higher decrease in the temperature in the oven. For some materials there is the possibility of eliminating the use of clamps for fixing the rectangular samples. This results not only in a substantial time reduction during sample fixing but also in the elimination of possible singularities accompanying the presence of the clamps. For better passage of the material that has been stretched after one revolution it is possible to fix both sample ends to the drums in such a way that their thickness reduces 'continuously to zero' at both lateral ends [17] (Figure 12). Figure 13 depicts and compares both possibilities of fixing (when no rupture appears during passage over a clamp) and justifies the measurements made without the clamps. Impossibility of using the Sentmanat U.T.P. when samples rupture during passage over a clamp is documented in Figure 14. This substantiates the measurement with the samples directly fixed to both drums.

4 CONCLUSIONS

Measurements of the extensional viscosity of LDPE Escorene were carried out using a broad range of widths and thicknesses of rectangular polymer samples in the SER Universal Testing Platform using the Anton Paar MCR501 rotational rheometer host system. The measurements were carried out at three different temperatures: 180, 190, and 200°C. Based on the experimental results the following statements about LDPE can be made:

- the measurement of elongational viscosity is invariant with respect to the dimensions of rectangular samples
- the use of thinner and narrower samples leads to better heating and less sagging
- in the absence of skilled personnel (carrying out regular experimental work), the use of wider samples facilitates their horizontal fixing

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- possible singularities caused by the presence of the fixing clamps can be removed by choosing rectangular samples of different dimensions or removing the clamps, i.e. fixing the samples directly to the drums

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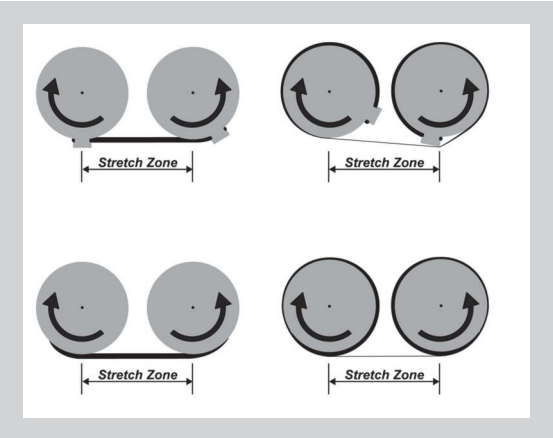

Figure 12:
Comparison of geometries of stretched samples fixed with clamps or directly to the drums.

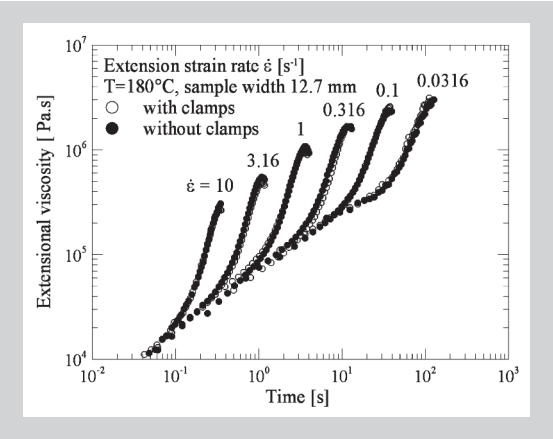

Figure 13:
Comparison of uniaxial extensional viscosity measured with and without the clamps at a temperature of 180°C (sample width 12.7 mm).

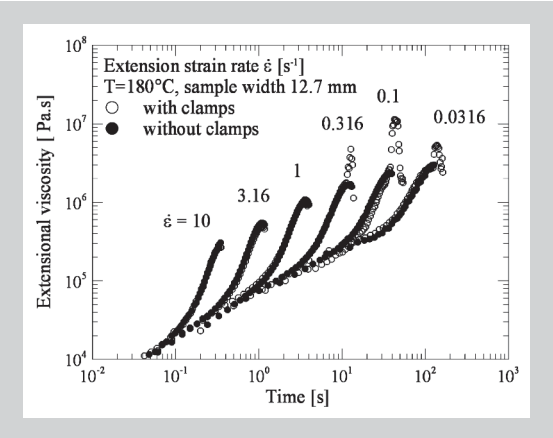

Figure 14:
Comparison of uniaxial extensional viscosity measured with and without the clamps at a temperature of 180°C (sample width 12.7 mm, with ruptures during passage over a clamp for lower values of extension strain rates).

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