MEASUREMENT OF ELONGATIONAL VISCOSITY OF PE MATERIALS

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Summary: *This contribution aims at the measurements of elongation viscosity for polyethylene materials. The approach is analysed from the viewpoint of possible degradation of materials tested and the emphasis is paid to admissible acceleration of the whole measuring process. There is presented a hint how to simplify (and accelerate) the measurements for a suitable class of materials.*

1. Introduction

Viscosity represents one of the main characteristics of polymer melts, therefore rheological measurements focus on determination of this material property in different flow geometries. In polymer processing polymeric materials usually subject to two types of deformation: shear flow and extensional flow. This results in two corresponding types of viscosities: shear and extensional.

Shear flow is the most common type of flow discussed in rheology. Shear flow may be produced in many ways and determination of shear viscosity is relatively well documented. In some polymer processing operations as e.g. fibre spinning or injection moulding, there occurs a combined flow exerted by both shear and elongational forces. In this case mere shear experiments cannot describe real polymer flow and a proper analysis of extensional flow is inevitable.

Extensional flow measurements are very sensitive to molecular structure, crystallinity and the macrostructure effect (mainly influenced by long-chain branching) of the polymeric system being tested. The measurements face certain difficulties as necking and rupture or sagging during heating a sample. Often it is not possible to reach a steady stress before the sample ruptures or irregularly deforms. A couple of methods have been tried to solve these problems and generate purely extensional flow. There exist several instruments such as a rotating clamp or extensional rheometer with a translating clamp and vertical (or horizontal) buoyancy control bath, proposed by the leading researchers in this branch, e.g. Meissner (1969), Münstedt (1975,1979), Meissner et al. (1982).

In recent years there has been developed a new instrument, known as a SER (Sentmanat Extensional Rheometer) Universal Testing Platform. This detachable unit is used with commercially available rotational rheometer systems (TA Instruments, Physica series MCR,

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etc.). The SER unit consists of paired master and slave wind-up drums on bearings housed and mechanically coupled via intermeshing gears. Rotation of a drive shaft results in a rotation of the affixed master drum and rotation of the slave one with equal angular velocity but in the opposite direction. This causes the ends of the sample to wind up onto the drums, and thus stretches a sample over an unsupported length. The ends of the sample are attached to the drums by two securing clamps as documented in Fig.1, and described in detail by Sentmanat (2003,2004), Sentmanat & Hatzikiriakos (2004).

The present contribution aims at the measurements of transient uniaxial elongation viscosity of polyethylene material using this device including a discussion on advantages and disadvantages of this experimental method.
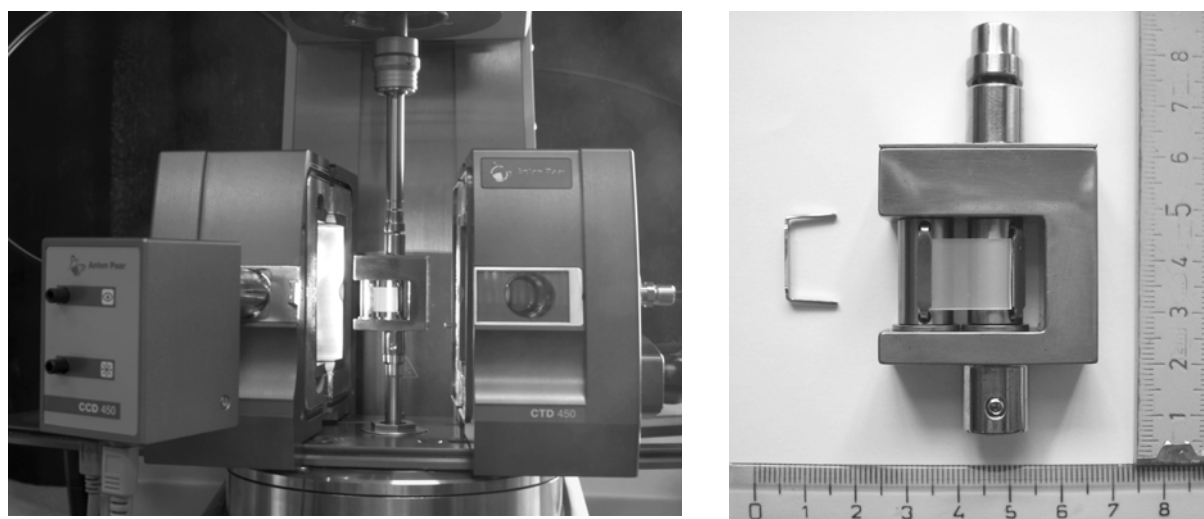

Figure 1 Experimental set-up (SER Universal Testing Platform surrounded by both halves of Convection Temperature Control Device CTD450) with a sample at room temperature (left) and a detail of SER Universal Testing Platform with an individual clamp (right).

2. Experimental

2.1 Experimental device

Measurements were carried out using the SER Universal Testing Platform from the Xpansion Instruments; the model SER-HV-P01 was applied with a Physica MCR501 rotational rheometer host system. For measurement of rheological properties of polymer melts there was used a convection heated measuring chamber CTD450 equipped with a camera system. The images and videos were recorded and displayed directly in the Rheoplus software supporting Physica MCR501.

2.2 Material

The low density polyethylene (LDPE) Escorene used in the experiments is produced by Exxon Mobil (basic characteristics in Tab.1). Sample specimens of LDPE were prepared by compression moulding in a preheated press. The final dimensions of the solid polymer rectangular specimens were approximately 12.7 mm (1/2") in height and 18 mm in width, with thickness in the range of 0.6-0.9 mm.

Table 1   The basic characteristics of the used material.

Material	MFI [g/10min]	Density [g/cm^3]	M_w [g/mol]	M_n [g/mol]
LDPE Escorene (Exxon, USA)	0.33	0.922	366,300	30,280

2.3 Measurements

Transient uniaxial extensional viscosity was measured at temperature 180°C. For characterising polymer melt there were conducted start-up experiments with constant strain rates (within the range 0.0316-10 s^{-1}); the results are shown in Fig.2. If a sample rupture appears prior to one revolution of the drums is attained, then there is no problem with determination of the magnitudes measured. A little complication occurs if a sample has to surmount the individual clamps (more than one revolution). This phenomenon is accompanied by a diversion (steep peak) of an experimental curve (very roughly resembling δ function) from the expected course and as such can be transformed to true values by continuous connection of the preceding and subsequent parts of the curve investigated. However there is no compensation if this 'δ-clamp behaviour' is exhibited at the maximum as demonstrated in Fig.2 for extension strain rate 0.0316. To eliminate this case there is a possibility to change a sample thickness.

Fig.3 depicts behaviour of extensional viscosity vs. Hencky strain for six different strain rates within the range 0.0316-10 s^{-1}.
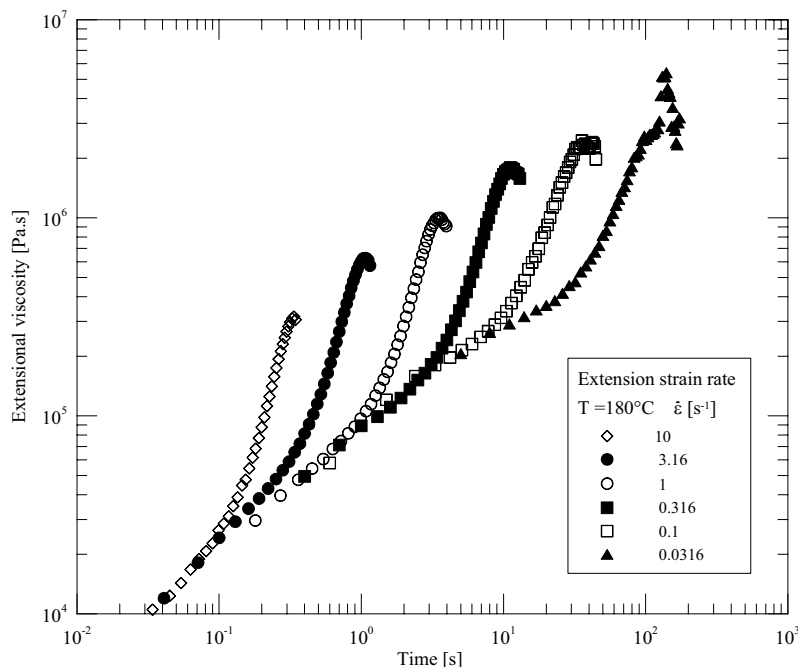

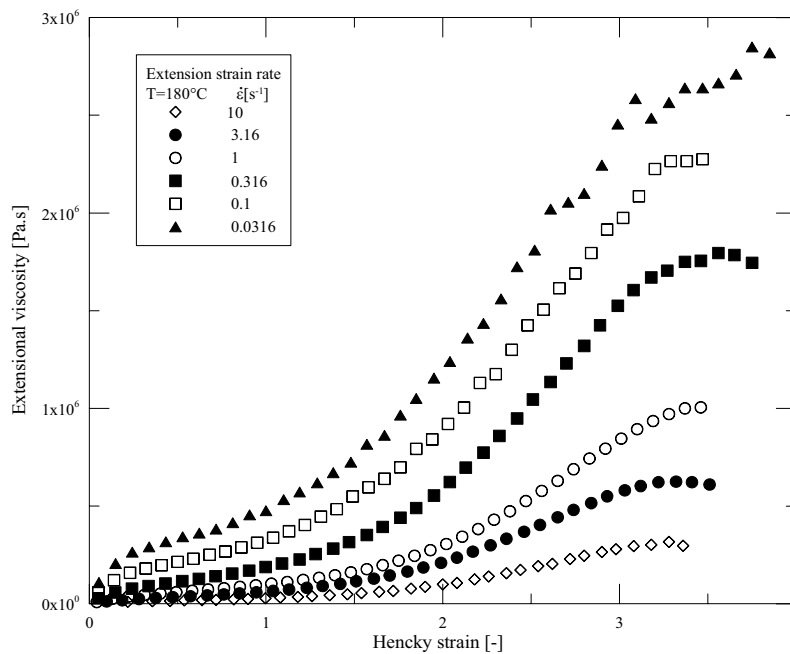Figure 2   Behaviour of transient uniaxial extensional viscosity.

Figure 3 Transient extensional viscosity as a function of Hencky strain
at six different strain rates.

3. Discussion

For proper measurement of elongational viscosity using the SER unit there should be fulfilled
the following assumptions (or at least almost fulfilled):
1) even distribution of temperature throughout a whole sample during measurement;
2) a sample should be stretched along its full height;
3) prevention from degradability.

ad 1) Due to its geometrical arrangement (including the air inlets and outlets) the oven
CTD450 exhibits a uniformly distributed temperature field even in the presence of the
asymmetric SER unit. This results in an even heating of a whole sample and a sample
thickness only influences a time period for which the sample should be exposed to reach the
pre-set temperature.

ad 2) A rectangular shape (see Fig.4) is an optimal one for starting elongational measurement
as this ensures proper stretching of the whole sample along its height just from the beginning.
To achieve this shape is not so trivial as gravitational force acting on an unsupported part of a
sample during heating causes it's sagging. For observation of this phenomenon the oven
CTD450 is equipped with a camera enabling direct checking of the whole process including
its recording. This provides a priori an elimination of the measurements for which a location
of the sample measured is unfavourable and should result in an incorrect data. Fig.5 illustrates
an application of the camera system for the selected points (however the recording is
continuous during the whole measurements).

ad 3) Time required for proper heating of the whole sample is not limited only by a possible
sagging as discussed above but by material degradation as well. This can be partially checked
using a camera (colour changes). There are two possibilities how to accelerate the
measurements and hence to suppress material degradation:
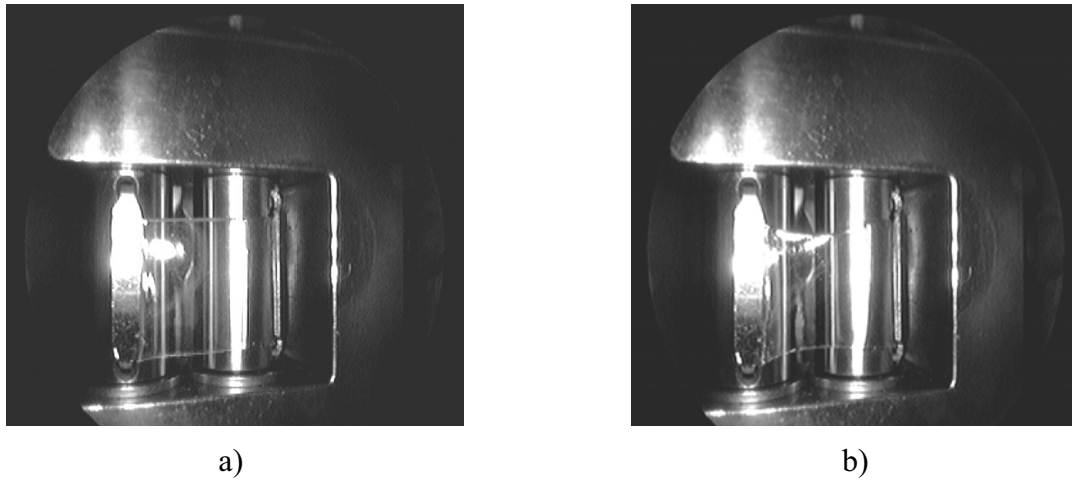
a)   b)

Figure 4 Shape of the sample a) optimal shape b) sagging of the sample.
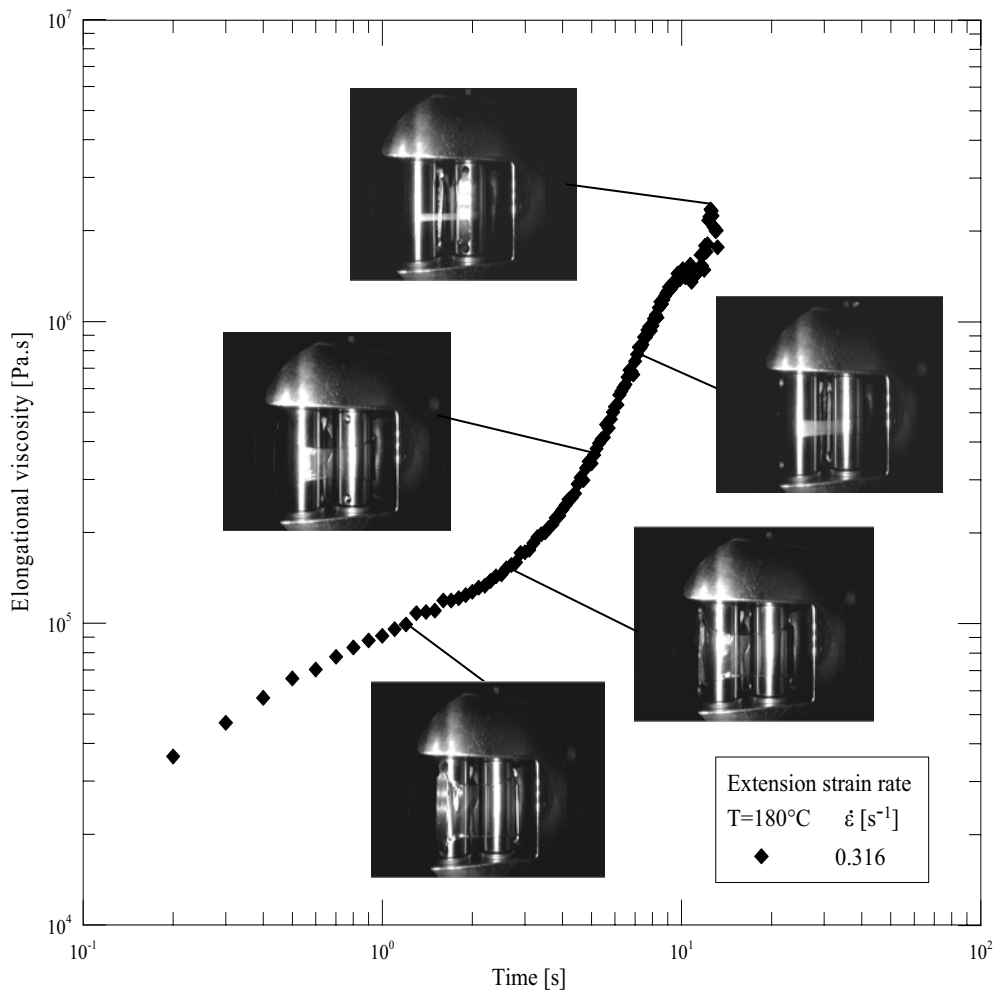

Figure 5 Geometric shapes of the sample for selected values of extension strain rate.

a) Usage of a thinner sample should shorten a time period for uniform heating to a required temperature. However due to other factors it is recommended not to reduce a sample thickness remarkably under 0.5 mm.

b) Possible elimination of the clamps used for fixing the ends of a sample measured exhibits two favourable effects - both this accelerates measurement itself (and thus postpones possible onset of degradation) and substantially eliminates the 'δ-clamp behaviour' discussed in Section 2. This removal of the clamps is not applicable for all materials, nevertheless for PE Escorene that was measured the sticking of the ends of samples directly to pre-heated drums is admissible as proved in Fig.6.
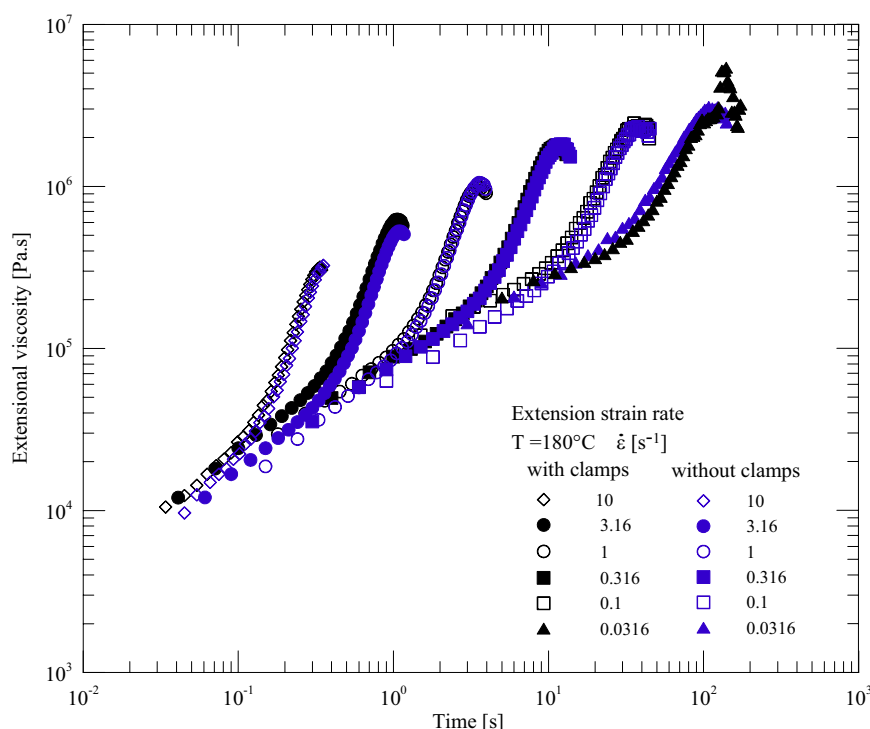

Figure 6 Comparison of uniaxial extensional viscosity measured with and without the clamps.

4. Conclusion

The experimental set-up discussed above provides more responsibility for proper evaluation of extensional measurements due to visual checking, including a movie record allocating corresponding pictures to the individual measured points and eliminating unexpected material degradation.

Nevertheless measurement of the transient elongational viscosity using the SER Universal Testing Platform has some limitations and other measurement instruments [1-4] provide wider possibilities (e.g. temperature control) and higher accuracy; for instance enable to avoid formation of the type 'δ-clamp behaviour'.

However for majority of standard materials the results measured using various instruments are fully comparable.

5. Acknowledgement

6. References

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