APN029

The SER2 – Universal platform for material testing

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INTRODUCTION

The roots of extensional rheometry are to be found in the very beginning of the 20th century. It was Trouton, who when experimenting with pitch and shoemaker's wax, subjected these materials to "torsion" and "traction" deformations. He discovered through his ingenious experiments, that the uniaxial extension viscosity is three times the shear viscosity."….A variety of pitch which gave by traction method λ=4.3 x 10^{10} poise was found by torsion method to have a viscosity μ=1.4 x 10^{10} poise… F.T. Trouton (1906)" [(1)]

Despite these early experiments, the developments in elongation rheometry were few until the end of the sixties. The exciting phase in elongation rheometry started in 1969 with Meissner [(2)], Cogswell[(3)] and Vinogradov et al.[(4)]. Whereas all experimental approaches up to this date were based on pulling the ends of a rod like sample apart, Meissner introduced a novel idea which changed the field of elongation rheometry. He replaced the clamps moving in space with fixed mounted rotating clamps. The sample is held between two pairs of rotating extensional viscosity of the specimen being stretched in the isolated 'stretch zone' of length L, defined by the tangent plane between the two drums.

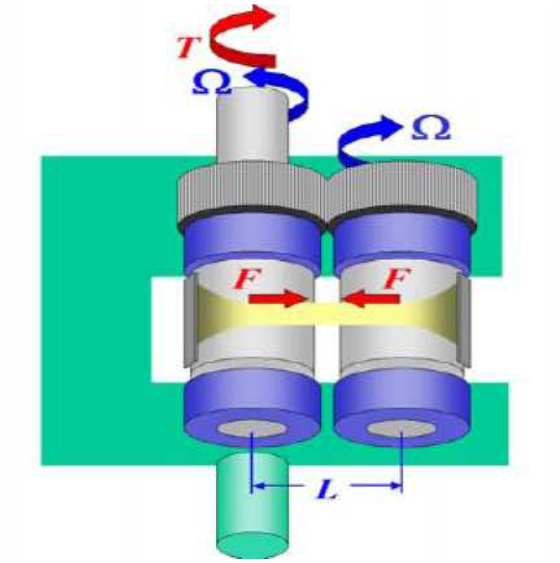
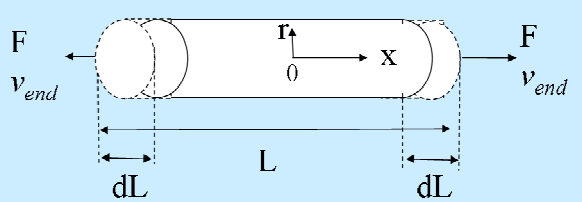


Figure 1: Schematics of the SER2 design

In addition to extensional measurements on polymer melts, the SER2 is capable of performing a range of physical property measurements such as tensile, peel, tear and friction measurements on small hard and soft solid samples

THE ELONGATION STRAIN RATE (HENCKY STRAIN RATE)

The most common elongation measure is the engineering strain or Cauchy strain, defined as the increase in length ΔL divided by the initial length L_o. The Cauchy strain is a deformation measure valid for small deformations. For large deformations ($\Delta L >> L_o$), the Cauchy strain is replaced by the Hencky strain. The Hencky rate is di-



$$v_x = \dot{\varepsilon} x \quad v_r = -\tfrac{1}{2}\dot{\varepsilon} r$$

$$v_{end} = \frac{\dot{\varepsilon} L}{2}$$

$$L(t) = L_o e^{\dot{\varepsilon} t}$$

$$\varepsilon_H(t) = \dot{\varepsilon} t = \ln \frac{L(t)}{L_o}$$

Figure 2: Hencky strain in elongation testing

rectly related to the velocity of a particle along the deformation axis $v_x=(d\varepsilon_H/dt)x$ (figure 2). In a constant rate experiment (two ends moving at the same speed), the particle in the center of the rod has a velocity zero and the particle velocity increases with the distance from the center. The rotating clamp technique deforms the material at constant Hencky rate by expelling the material at a fixed distance $x = L_o/2$ from the sample center with a constant velocity v_x imposed by the rotating clamps. In a traditional elongation experiment, with the sample volume constant, the sample ends must therefore move at a speed $v_{end}=(L/2)d\varepsilon_H/dt$. Integrating from L_o to the final length L_f, leads to an exponential increase of the sample length over time $L(t)=L_o\exp[(d\varepsilon_H/dt)t]$. The final strain thus can be expressed as $\varepsilon_H=\ln[L_f/L_o]$.

The elongation viscosity η_E is defined as the stress divided by the elongation rate. The stress is the force divided by the surface area normal to the direction of deformation. For an incompressible material, the volume is conserved and the surface area must decrease exponentially as $A(t)=A_o\exp\{-(d\varepsilon_H/dt)t\}$ with the sample length increasing (Figure 3) while the experiment proceeds.

SER2 CONCEPT

Instrument description

The Universal Testing Platform (SER2) is schematically represented in the figure 3. The SER is connected via the SER base adapter to the SER bracket, mounted to the test head of the rheometer. The SER drive shaft connects the motor spindle to the SER, and aligns the motor spindle with the primary rotating drum. The second drum is driven by the first, at the same rotation speed in opposite direction by a set of spur gears mounted to the drive shafts of the drums. The lateral offset of the center axis of the two drums is 12.7 mm. The diameter of the drums is 10.3 mm and the clearance between the drums is 2.4 mm. The drums can be removed from the drive shaft easily and exchanged. The SER2 is designed to fit into the standard rheomter furnace.

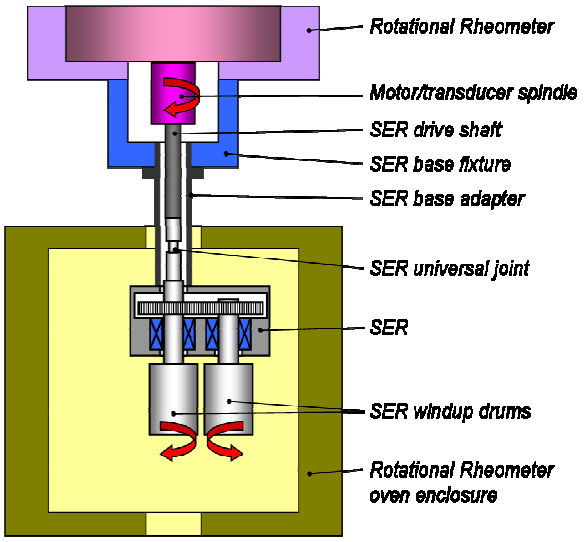


Figure 3: Universal testing platform SER2

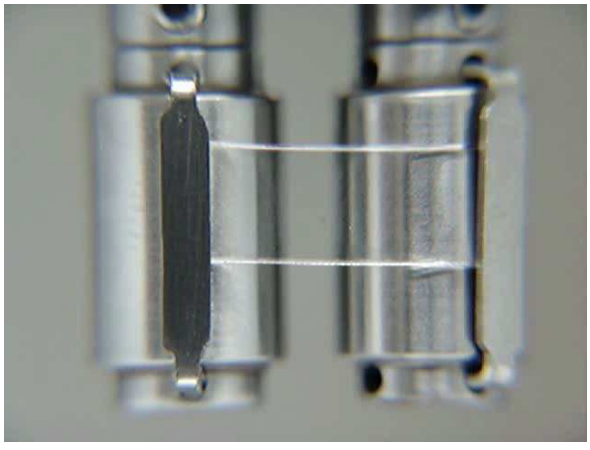


Figure 4: Rotating drums with sample loaded

The figure 4 shows the SER2 option installed on the rheometer. Since the cylindrical drums are mounted vertically, the sample is also loaded vertically onto the drums and attached with two tiny clips.

Sample Support

On the SER2, the samples are mounted vertically and the sample length L is reduced to 12.7mm. The Meissner elongation rheometer RME was using a support medium to prevent the molten sample from sagging. The sample (40mm long) was floating horizontally on an air cushion during the experiment. The reduction of the sample size and vertical orientation in the SER are responsible for the stiffness increase of a sample (same material at the same temperature) by 1 million. Materials with a shear viscosity above 1000 Pa s are therefore not significantly sagging under the effect of the gravity and can be measured with the SER2. Because of the small sample size and the fast heating rate of the furnace, the waiting time after loading the sample is reduced to less than a few minutes, thus preventing creep in the sample. The gravitational force does not contribute to the force measurements because of the horizontal alignment of the sample.

PREPARING SAMPLES

Sample preparation is an important criterion to obtain good results up to the maximum achievable Hencky strain. Sample non-uniformity and necking decrease the measured force, which results in a viscosity decrease at large deformations. Sample homogeneity is a fundamental requirement for good elongation measurements up to Hencky rates of 4 and more. The main goal of sample preparation is to prepare a homogeneous and stress free sample of the correct size. The optimum sample size for the SER2 is:

Length = 18 mm
Width = 10 mm
Thickness = 0.7 mm

The best method to prepare samples is hot compression molding. The samples have to be molded at a temperature above the test temperature and cooled slowly under pressure to obtain relaxed and stress free homogeneous samples showing minimum dimensional changes after loading into the SER2 test fixture..

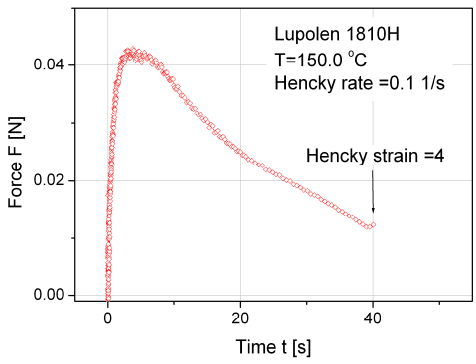


Figure 5: Force curve during a step rate extension

EXPERIMENTAL

The procedure for extensional measurements on polymers consists of 4 extensional steps. These extension steps are configured as follows: Step 1 is a pre-stretch test to make sure that the sample is not buckling at the start of the test; step 2 is a relaxation test, step 3 is the principal extension test and the results are recorded using fast data sampling; step 4 is a final relaxation test to check for eventual drifts in the force measurement.

This extension procedure can be used with polymer melt samples and other high viscosity materials. Other physical tests, such as peel/adhesion/ tear/friction etc. can be performed on the SER2. In this case standard rheometer tests such as flow tests, ramps and creep tests can be programmed. For some of these tests, the sample geometry is not relevant and the calculated material parameters not meaningful. In this case instrument based parameters such as velocity and torque are controlled and/or recorded

When the furnace is equipped with a camera, the sample deformation can be monitored and images saved with the test data. The recording speed is a function of the hardware used, typically one image can be saved every 1-2 s. Video streaming is not possible during the experiment.

TEST RESULTS FOR THE STANDARD LDPE LUPOLEN 1810H AT 150°C

Force and stress results

The response of the material to a constant deformation rate is the stress. During the extension experiment the reactive force at the drums is recorded as a function of time or deformation. The force in figure 5 grows from zero as the stress builds up in the sample with test time. After a short period, the force exhibits a maximum and then decreases continuously more or less exponentially. The reason for the force decrease is the exponentially decreasing cross section of the sample with increasing total deformation. The stress, the ratio of force F and sample cross section A(t) increases strongly at the start up and then levels off. Between 10 and 20 seconds (Hencky strain 1-2), the stress continues to increase again. This behavior, typical for LDPE is referred to as strain hardening (Figure 6).

The test results of a transient step rate experiment are represented by the viscosity as a function of time. The logarithmic representation allows a detailed analysis of the start-up behavior and shows clearly when strain hardening occurs and where the material response deviates from the linear viscoelastic behavior. The extension viscosity versus time is shown in figure 7 for a series of tests performed at a Hencky rate from 0.01 to 10 1/s. At the beginning of the experiment, the viscosity follows the linear viscoelastic start-up curve. For comparison, the linear viscoelastic response of 3 times the shear viscosity is plotted in grey. The time dependent shear results were obtained directly from a step rate experiment in a cone plate (full symbols) and recalculated from oscillation tests (open symbols) obtained using a parallel plate geometry.

The strain hardening, represented by the deviation from the linear viscoelastic stress growth curve shifts to longer time with decreasing Hencky rate. The on set of the strain hardening occurs at 0.1 s at a rate of 10, 1s at a rate of 1, 10s at a rate of 0.1 etc., which corresponds to a Hencky strain of 1. This proves that the on set of the non-linear response is controlled by the strain rather than the strain rate.

Friction test of a brake pad

In addition to elongation testing of polymer melts and similar highly viscous materials, the SER2 is capable of performing a range of physical material tests. These tests include 1: Peel/Adhesion testing, 2: Friction testing, 3: Tensile testing and 4: Tear testing.

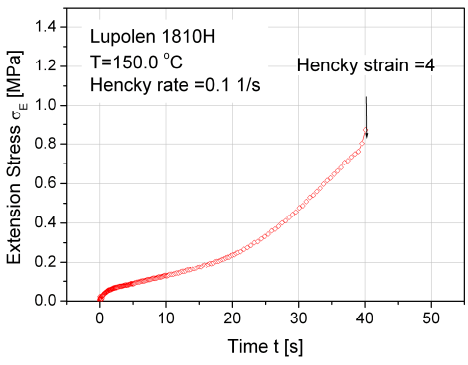


Figure 6: Stress curve during step rate extension test .

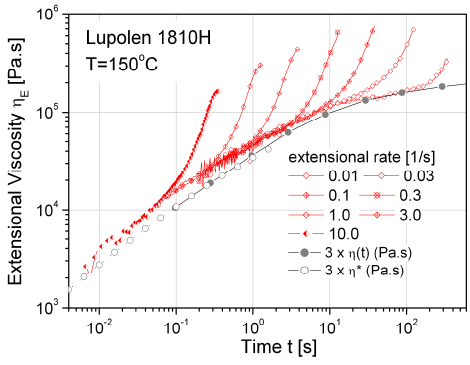


Figure 7: Viscosity function of LDPE Lupolen 1810H at different elongation rates

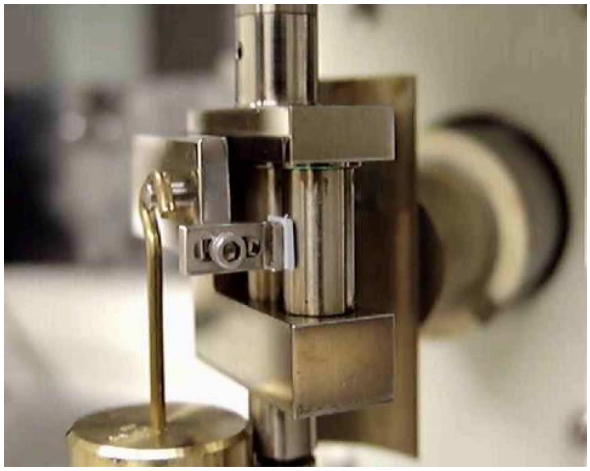


Figure 8: Friction testing

The physical material tests make use of the standard rheometer test modes such as flow, creep, etc. while controlling torque and angular displacement or angular velocity. Force and linear displacement can easily be set up using user defined equations [Force F[N]=M(t)/0.00515 and Linear displacement[m/s] = displacement [rad] *0.00515].

Figure 9 shows the results from a friction test. The sample, a brake pad, is attached with double sided tape to the support and pressed against the rotating drum using 100g & 200g weights (figure 8). The measured force is recorded as a function of time for various rotation speeds. The results show that the friction force is independent of the rotation rate, but increases linear with the applied pressure (weight). The friction coefficient is the friction force divided by the applied normal force.

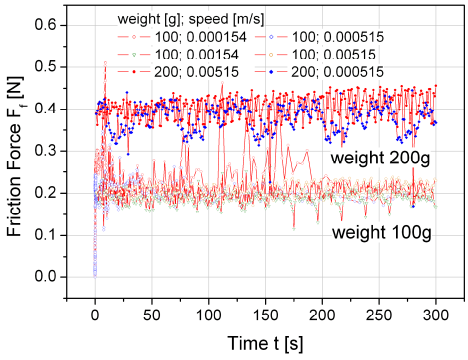


Figure 9: Friction tests performed at two loads

.REFERENCES

1. Trouton, F. T. *Proc. R. Soc*. 77, 426 (1906)
2. Meissner, J. *Rheologica Acta* 8, 78 (1968)
3. Cogsell, F.N. *Rheologica Acta* 2 187 (1969)
4. Vinogradov et al. *J. Polym. Sci. A-2* 8, 657 (1970)
5. Sentmanat, M.L. *Rheologica Acta* 43, 657 (2004)