EXPERIMENTAL INVESTIGATION AND IDENTIFICATION OF MATERIAL PARAMETERS FOR RUBBER BLENDS

Herbert W. Müllner [1], Josef Eberhardsteiner [1], André Wieczorek [2]

[1] Institute for Mechanics of Materials and Structures, Vienna University of Technology, Vienna, Austria
[2] Semperit Technische Produkte Ges.m.b.H. & Co KG, Wimpassing im Schwarzatale, Austria

1. Introduction

Capillary rheometry simulates polymer extrusion in a simplified way. It allows the characterisation of polymers by means of determination of the viscosity function and extrudate swell. As regards viscous properties, there is still a lack of constitutive characterisation of rubber blends. In this contribution new concepts are presented how the viscous properties are computed without using common correction methods.

The die swell is the determinant criterion for the production of rubber profiles by means of extrusion. Therefore its experimental investigation and numerical treatment is of high interest. This was one of the motivations for starting a research project in the field of rubber blend technologies. The consideration of the die swell for the production of rubber profiles is necessary. Thus, the final goal of this project is the numerical prognosis concerning tools for the extrusion of rubber.

The task of the present material characterisation is to determine all required state variables which describe the flow situation of a die through a circular capillary. For the description of the pseudo-plastic material behaviour of rubber blends the power law by OSTWALD [3] is used. Its application to the investigated rubber blends is possible for a common interval of the shear strain rate $\dot{\gamma}$. The coupled state variables are:

– material parameters of the power law,
– pressure loss according to viscoelastic properties,
– shear stress at the wall of the capillary,
– shear strain rate at the wall of the capillary and
– wall slippage velocity along the capillary.

If these state variables are known the determination of the shear rate dependent viscosity η is possible. Various types of rubber blends are used for the material characterisation, with Ethylene propylene diene monomer (EPDM) as the main component.

In order to determine viscous properties of rubber blends experiments with a capillary-viscometer are necessary. Due to the nonlinear coupling of the viscosity and the shear strain rate, correction methods are used to determine these material properties. First, Newtonian material properties are assumed. The resulting physical values are called apparent and marked with a subscript "*ap*". Second, the apparent values will be transformed into true values using correction methods.

2. Experimental Investigation

In order to characterise the investigated materials various experiments by means of a capillary-viscometer with different capillary lengths L, capillary radii R and melt temperatures T were performed. With one experiment the investigation of different stamp velocities is possible.

All tests with the capillary-viscometer were performed on a macroscopic level and are divided into two categories:

a) *Experiments with circular capillaries:* The common method has the disadvantage, that the measurement of the pressure is performed before the capillary entry. Therefore, entrance and outlet pressure losses are not considered by such a measurement.
b) *Experiments with slit capillaries:* With this type of experiment the determination of the melt pressure within the capillary is possible. Therefore, statements about the pressure losses can be performed.

In addition to the melt pressure p and the appropriate stamp velocity v, the die swell area-ratio χ of all experiments was determined, too. Three different methods for measuring die swell were used:

a) *Adaptation with high speed camera:* Because the exit speed of a die through a capillary is up to 1000 mm/s, the shooting of the exit of capillaries have to be investigated with a high speed camera (see Fig. 1).
b) *Microscopical investigations:* Because no time dependent properties are considered this method is only for validation and not for characterisation purposes.
c) *Swell value measuring unit:* With this method the diameter is measured by means of a laser beam with a wave-length of approximately 700 nm. The influence of the dead weight of a die on the swell diameter is considered, too. By cutting the strand directly below the measuring point, the influence of the dead weight is minimized for the measured swell value (see Fig. 2).

Comparison of the results of these three methods leads to a successful validation of the swell value measuring tool. Furthermore, in the contemplated intervals of the shear rate the dependency of the die swell on the shear rate has not been observed. Moreover, dependencies of the die swell on the capillary length L, the capillary radius R and the melt temperature T were established.

As a consequence the material characterisation has to be extended for the determination of the die swell area-ratio of the investigated rubber blends.

Fig. 1: Capillary-viscometer with high speed camera.

Fig. 2: Position of the swell value measuring unit.

3. Identification of Material Parameter

According to the experiments, the two different nonlinear solution algorithms were developed.

3.1 Generalised Newton-Raphson procedure

Because of the consideration of wall slippage and the coupling between viscosity and shear rate the resulting characterisation is represented by a coupled system of nonlinear equations. Describing their solution requires a numerical integration algorithm. For this purpose the NEWTON-RAPHSON-iteration method has been adopted. It requires for a certain number of unknown variables the same number of conditional equations. The variables are the coupled state variables listed in Chapter 1. A disadvantage of this method is the fact that a starting state near the unknown solution is required.

3.2 Genetic Algorithm

Genetic algorithms are non-deterministic stochastic optimisation methods that utilize the theories of evolution and natural selection to solve a problem within a complex solution space. A complete description of genetic algorithms can be found in GOLDBERG [1]. The obtained material parameters describe all coupled state variables listed in Chapter 1 for the investigated experiments. Further details of this material characterisation method of rubber blends can be found in MÜLLNER et al. [2].

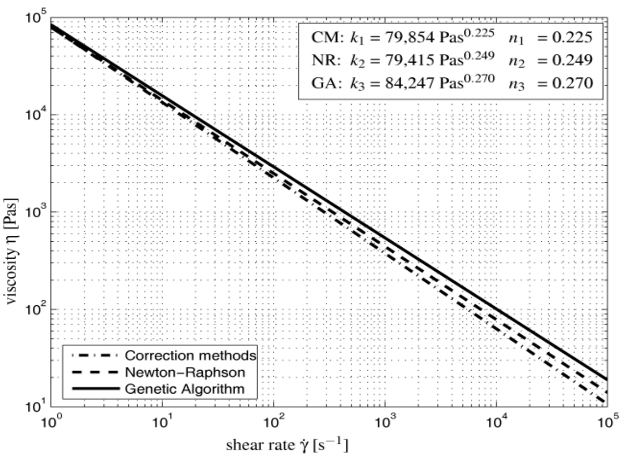

Fig. 3: Comparison of velocity profiles within a capillary for the used characterisation methods.

4. Validation of the new Methods

The different characterisation methods lead to comparable viscosity curves for the investigated rubber blends (see Fig. 3) but different velocity profiles within the contemplated capillaries (see Fig. 4). This results from the fact that the wall slippage velocity is not considered for the former characterisation method. Furthermore, the average velocities are equal for both new material characterisation methods. Further research activities concern the consideration of the die swell in the presented algorithms in form of the dimensionless swell area-ratio. After that, experiments with single-screw extruders are planned, to validate the developed material characterisation methods.

5. References

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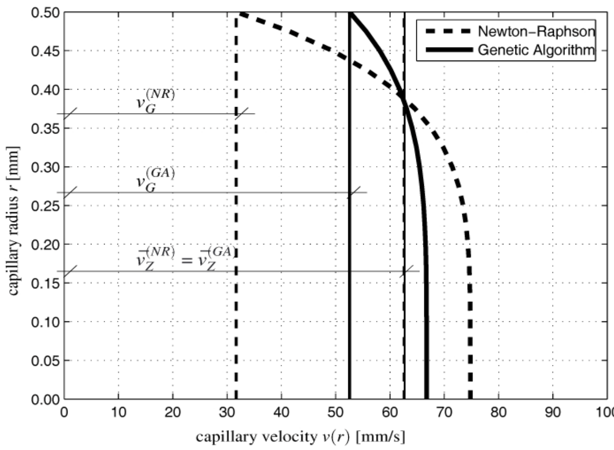

Fig. 4: Comparison of velocity profiles within a capillary for the used characterisation methods.