Experimental identification of viscoelastic properties of rubber compounds by means of torsional rheometry

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Abstract Rheological models are used for the description of viscoelastic properties. Such investigations are mostly based on experiments with a torsional rheometer, i.e. a Rubber Process Analyzer. The results of such experiments are storage and loss modulus, which depend on the applied angular frequency. The investigations were performed with rubber compounds used in industry, with different crystallization degrees of the used EPDM basic polymer as well as different filler degrees. Moreover, the influence of melt temperature on these dynamic moduli is considered. For the rheological description of the investigated rubber compounds three different viscoelastic models are used. After derivation of the corresponding relationships for each of the spring-dashpot models the application for rubber compounds was investigated. The best agreement between experiment and model prediction was achieved by a nonlinear viscoelastic model. With such a model the influence of different crystallization and filler degrees on the viscoelastic material properties can be covered.

Keywords Rheology · Viscoelasticity · RPA · Huet model · Mechanics of fluids

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1 Introduction

The design of injection heads for the extrusion of rubber profiles is exclusively based on empirical knowledge of the non-linear viscoelastic flow behavior of elastomers. Thus, the injection heads were designed for the employed rubber compound, where the geometry of the sealing profile is obtained by empirical adaptation of the extrusion die.

This adaptation process is mainly influenced by the swelling of rubber after extrusion. This process consists of several steps. Disadvantages of the current production process are the non-reproducible process technology and the time-consuming design of dies, which affects the production capacity. The was a strong motivation for a research project devoted to characterization of the die swell properties of rubber compounds. The research work was jointly carried out with Semperit Technische Produkte Ges.m.b.H., who provided the material and the experimental equipment.

An important task of the constitutive material characterization is the determination of viscoelastic properties which are required for the description of arbitrary parts of the tools for production of rubber profiles. Until now, torsional rheometry is one of the most important methods for the determination of such properties of plastics. In many cases this method is also used for rubber compounds. For the performance of numerical simulations of injection heads and other extrusion applications knowledge of viscoelastic material parameters is required. The latter are coupled with

rheological models, i.e. spring-dashpot models. Such viscoelastic models must cover both, optimization of correlation between experiments and rheological models as well as change of viscoelastic properties due to different contents of the used compounds.

In this paper, three models are investigated for application to rubber compounds. For explanation of the chosen procedure, the simplest rheological model, i.e. the Maxwell model is used. To improve the agreement of the results obtained by experiment and rheological model, a generalized Maxwell model is used. Because of a huge number of material parameters a nonlinear material model, i.e. the Huet model, is introduced. Applying nonlinearity allows for covering the requirements for a satisfying viscoelastic description of rubber compounds.

In Sect. 2 of this paper the principal experimental idea and the main quantities are described briefly. Section 3 contains a brief description of the investigated materials and the used experimental device. The principal way of the determination of viscoelastic material parameters is described in Sect. 4. In Sect. 5 three rheological models are applied to rubber compounds. This includes the derivation of corresponding equations for the determination of material parameters as well as the comparison of experimental results and model prediction. In Sect. 6 the influence of different constituents on viscoelastic properties is explained. Finally, conclusions are drawn.

2 Phenomenological aspects

When a viscoelastic material is subjected to a sinusoidally varying strain, a steady state will be reached in which the resulting stress is also sinusoidal, having the same angular frequency but retarded in phase by the so-called phase angle δ.

If the origin along the time axis is selected to coincide with a time at which the strain passes through its maximum, the strain and stress functions can be written as [10]:

$$\varepsilon(t) = \varepsilon_0 \cos(\omega t) \tag{1}$$

and

$$\sigma(t) = \sigma_0 \cos(\omega t + \delta). \tag{2}$$

Using algebraic transformations common in the analysis of harmonic systems, it is convenient to write
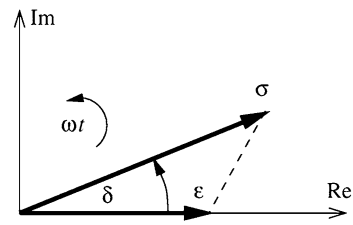

Fig. 1 Relation between harmonic stress and strain

the stress function as a complex quantity σ^*, whose real part is in phase with the strain and whose imaginary part is 90° out of phase with it:

$$\sigma^* = \sigma_0' \cos(\omega t) + i \sigma_0'' \sin(\omega t), \tag{3}$$

where the asterisk and $i = \sqrt{-1}$ denotes a complex quantity. Figure 1 visualizes the observable stress and strain as the projection on the real axis of vectors in the complex plane at a frequency ω. If the strain vector $\boldsymbol{\varepsilon}$ passes the real axis, the phase angle δ is obtained [10].

With Fig. 1, the norm of the stress vector can be expressed in terms of the corresponding axis values:

$$|\sigma^*| = \sigma_0 = \sqrt{(\sigma_0')^2 + (\sigma_0'')^2}, \tag{4}$$

where

$$\sigma_0' = \sigma_0 \cos \delta \quad \text{and} \quad \sigma_0'' = \sigma_0 \sin \delta. \tag{5}$$

Dividing (4) by the corresponding norm of the strain vector, ε_0, the complex modulus G^* is obtained as:

$$|G^*| = \frac{\sigma_0}{\varepsilon_0} = \sqrt{\left(\frac{\sigma_0'}{\varepsilon_0}\right)^2 + \left(\frac{\sigma_0''}{\varepsilon_0}\right)^2}. \tag{6}$$

This form of the complex modulus can be used for the definition of two dynamic moduli, both being ratios of stress and strain, but having different molecular interpretations and macroscopic consequences [10]. The first one is the storage modulus, defined as the ratio of the in-phase stress to strain,

$$G' = \frac{\sigma_0'}{\varepsilon_0} = \frac{\sigma_0}{\varepsilon_0} \cos \delta, \tag{7}$$

and the second one is the loss modulus, defined as the ratio of the out-of-phase stress to strain,

$$G'' = \frac{\sigma_0''}{\varepsilon_0} = \frac{\sigma_0}{\varepsilon_0} \sin \delta. \tag{8}$$

Table 1 Main properties of employed rubber compounds

Compound code	EPDM A	EPDM B	EPDM C	EPDM D	EPDM E	EPDM F
molecular structure	amorphous	crystalline	amorphous	crystalline	amorphous	crystalline
$ML(1+4)$	59.9 ME	64.3 ME	63.7 ME	62.4 ME	66.3 ME	64.3 ME
Shore-grade	67.0 ShA	78.0 ShA	64.0 ShA	76.0 ShA	59.0 ShA	72.0 ShA
density	1.294 g/cm³	1.298 g/cm³	1.209 g/cm³	1.214 g/cm³	1.098 g/cm³	1.104 g/cm³
filler degree	high	high	average	average	low	low
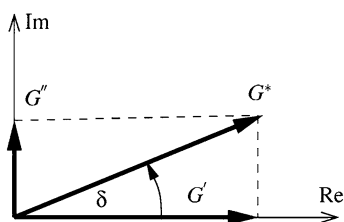
Fig. 2 Definition of storage and loss modulus

The storage and loss modulus in viscoelastic liquids measure the stored energy, representing the elastic portion, and the energy dissipated as heat, representing the viscous portion. Figure 2 shows, according to Fig. 1, a similar visualization for storage and loss modulus. The phase angle is, therefore, defined as ratio of loss to storage modulus as:

$$\tan \delta = \frac{\sigma_0''}{\sigma_0'} = \frac{G''}{G'}. \tag{9}$$

If $\delta = 0°$ the material is elastic, if $\delta = 90°$ the material behaves totally viscous. For rubber, the phase angle is always $0° < \delta < 90°$.

3 Experimental investigations

3.1 Materials

The materials considered in this paper were various unvulcanized rubber compounds used in industry containing EPDM (ethylene-propylene-diene-monomer) as basic polymer and mainly carbon black and chalk as filler material. In total six compounds were investigated, two different chemical conditions of EPDM, amorphous and crystalline, with three different filler degrees each. Such rubber compounds are used for window sealings, pipeline constructions, bridge dilatations and various parts of cars.

Fig. 3 Rubber Process Analyzer (RPA)

The main material properties of the investigated compounds are summarized in Table 1, where $ML(1+4)$ denotes the Mooney viscosity after four minutes and one minute preheating at a temperature of 100 °C. The hardness values are given in Shore A according to the German design code DIN 53505. Melt densities were determined according to DIN 53479.

3.2 Instruments and methodology

The rheological tests were performed with a torsional rheometer (manufactured by Alpha Technologies, US). Such a rheometer is used for determination of the dynamic properties of viscous and viscoelastic materials. Various types of rheometers are normally used. Figure 3 shows a cone-cone-rheometer. With this

type two cones are pressed onto the sample, it is also called Rubber Process Analyzer (RPA)

Figure 4 shows the principle of an RPA. This torsion-rheometer strains a sample in shear by oscillating the lower die sinusoidally. The upper die is fixed and connected with a measuring device, which records the applied bending moment S^*. Due to the viscoelastic properties of rubber S^* is not in-phase with the applied strain.

The RPA is designed to measure both elastic and viscous properties of elastomers and compounds. S^* is split into an elastic component S' and a viscous component S'' by means of a Fourier transformation. Af-

terwards, by applying a form factor the storage modulus G' and the loss modulus G'' are provided. More details concerning the RPA can be found in Dick et al. [2].

The result of an RPA experiment is the storage and the loss modulus for one specified angular frequency ω_r. During one experiment, more than one frequency is investigated. For rubber compounds, oscillation frequencies have been set from 0.1 to 30 Hz. In this context, r denotes the actual operating point and s the number of investigated angular frequencies per experiment.

Figure 5 shows the results of one RPA experiment for the compound EPDM 1 at a temperature of $100\,°C$. As it can be seen, the storage and loss modulus are increasing monotonously with increasing angular frequency ω.

① upper stamp
② lower oscillating stamp
③ heating
④ sealing
⑤ display
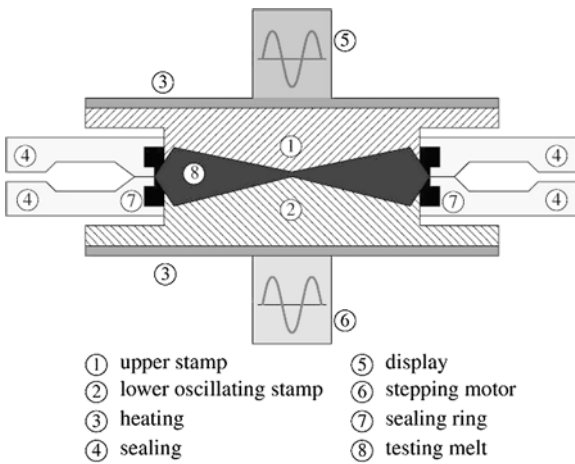⑥ stepping motor
⑦ sealing ring
⑧ testing melt

Fig. 4 Principle of a Rubber Process Analyzer

4 Modeling viscoelasticity

Viscoelasticity is used to describe the behavior of materials showing an accumulation of strains under constant stress (creep) or a reduction of stress under constant strain (relaxation). Depending on the prescribed strain, the corresponding stress is determined, using a relaxation modulus $E(t)$ and a creep compliance
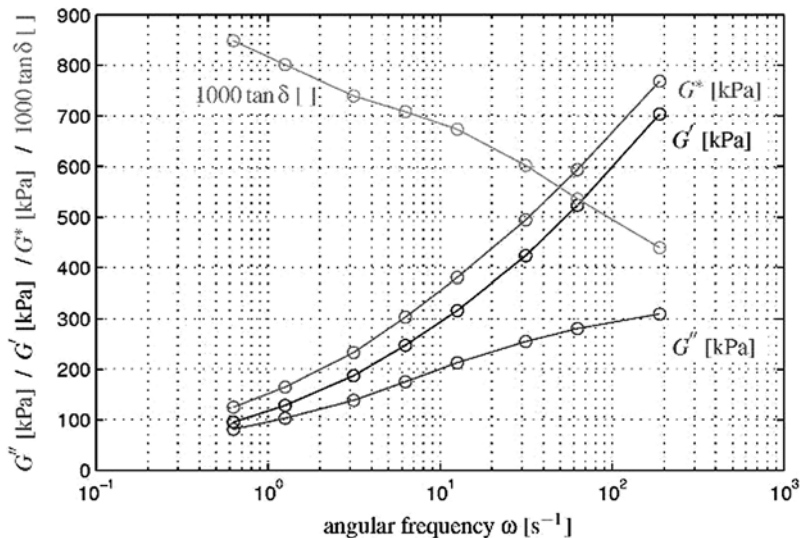

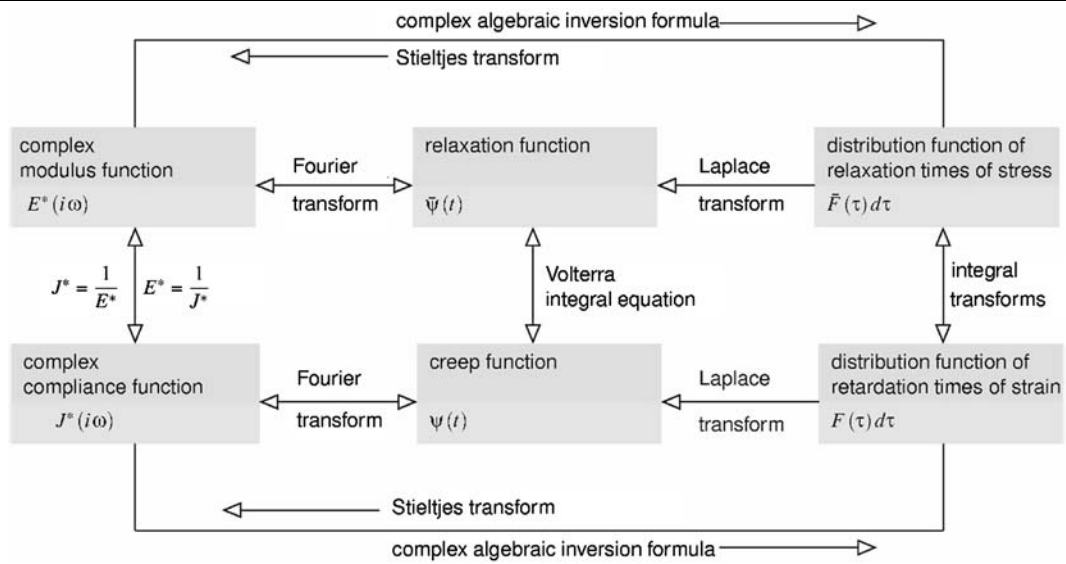**Fig. 5** Typical results with an RPA for material EPDM F

Fig. 6 Mathematical structure of viscoelasticity

$J(t)$, respectively. For a specified strain history $\varepsilon(t)$, the stress is obtained by the convolution integral as

$$\sigma(t) = \int_{\tau=0}^{t} E(t-\tau) \frac{\partial \varepsilon(\tau)}{\partial \tau} d\tau. \tag{10}$$

Analogous, the strain for a given stress history $\sigma(t)$ reads

$$\varepsilon(t) = \int_{\tau=0}^{t} J(t-\tau) \frac{\partial \sigma(\tau)}{\partial \tau} d\tau. \tag{11}$$

Since creep and stress relaxation represent two aspects of the same viscoelastic behavior of a material, $E(t)$ and $J(t)$ are related. This relation could, e.g., found by the Laplace transformation, reading as algebraic equations in the transform variable s:

$$\hat{\sigma}(s) = s\hat{E}(s)\hat{\varepsilon}(s) \quad \text{and} \quad \hat{\varepsilon}(s) = s\hat{J}(s)\hat{\sigma}(s). \tag{12}$$

Finally, the relation between E and J is obtained from (12), according to Findley et al. [3], as

$$\frac{\hat{\sigma}(s)}{\hat{\varepsilon}(s)} = s\hat{E}(s) = \frac{1}{s\hat{J}(s)} \quad \text{or} \quad \hat{E}(s)\hat{J}(s) = \frac{1}{s^2}. \tag{13}$$

Figure 6 shows a scheme for applying the relation between creep and relaxation [4], where the different procedures are related by Laplace and Fourier transformations, respectively.

4.1 Basic elements for modeling

Rheological models used to describe the viscoelastic behavior of materials are commonly composed of three basic elements, i.e. a linear spring and a linear and a nonlinear dashpot. The linear Hookean spring is characterized by a constant creep compliance

$$J = \frac{1}{E} = \text{const}, \tag{14}$$

where E represents the stiffness of the spring. The second element is the linear dashpot with a creep compliance

$$J(t) = \frac{t}{\eta'}, \tag{15}$$

where η' denotes the dynamic viscosity of the material. A third basic element is the nonlinear dashpot. The creep compliance of the latter can be described by means of a power law as

$$J(t) = J_1 \left(\frac{t}{\tau}\right)^{m_1}, \tag{16}$$

where J_1 represents the creep compliance at $t = \tau$ and m_1 a dimensionless parameter. τ is the characteristic time of the underlying viscoelastic process.
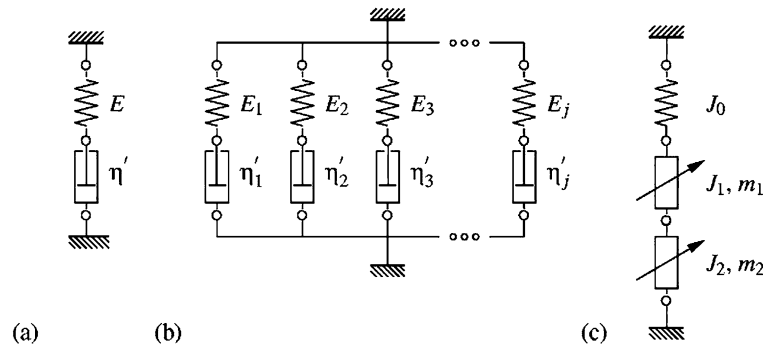
Fig. 7 Used rheological models for investigated rubber blends: **a** Maxwell model, **b** Wiechert model, **c** Huet model

4.2 Models with basic elements connected in series

Generally, in the viscoelastic material modeling a combination of the basic elements described in Sect. 4.1 is required. From the two possibilities in combining basic elements, connection in parallel and in series, the latter is more appropriate to model the viscoelastic behavior of rubber compounds.

In case of rheological representation by basic elements connected in series, the creep compliance of the respective model is simply obtained as the sum of the creep compliances of the corresponding basic elements. In this paper, three rheological models will be described for the modeling of the viscoelastic behavior of rubber:

1. *Maxwell model:* The simplest rheological model consists of a spring and a linear dashpot [7]. The creep compliance is obtained as:

$$J(t) = \frac{1}{E} + \frac{t}{\eta'}. \tag{17}$$

2. *Wiechert model:* This model corresponds to a generalized Maxwell model which combines several Maxwell models in parallel [11]. The creep compliance is obtained as:

$$J(t) = \sum_{i=1}^{j} \frac{1}{E_i} + \frac{t}{\eta'_i}, \tag{18}$$

where j denotes the number of Maxwell models.

3. *Huet model:* This rheological model consists of a spring and two nonlinear dashpots [5]. The creep compliance is obtained as:

$$J(t) = J_0 + J_1 \left(\frac{t}{\tau}\right)^{m_1} + J_2 \left(\frac{t}{\tau}\right)^{m2} \tag{19}$$

with

$$J_0 = \frac{1}{E}. \tag{20}$$

Figure 7 shows the scheme of the three considered rheological models, the unknown material parameters indicated.

5 Determination of viscoelastic material parameters

After RPA experiments at different temperatures were performed, the material parameters of the rheological models mentioned in Sect. 4.2 have to be determined. For this task, relationships for the storage and loss modulus and the material parameters have to be determined by means of the so-called Cole-Cole [1] and Black diagram. The former plots the loss modulus G'' over the storage modulus G'. The Black diagram plots the phase angle δ over the complex shear modulus G^*, which are obtained from storage and loss modulus (see Sect. 2).

Figure 8(a) shows a Cole-Cole diagram for EPDM F. In this diagram all investigated temperatures are shown, indicating a temperature independence of the function $G''(G')$. This property has been observed for all investigated rubber compounds. Figure 8(b) shows the corresponding Black diagram. The decrease
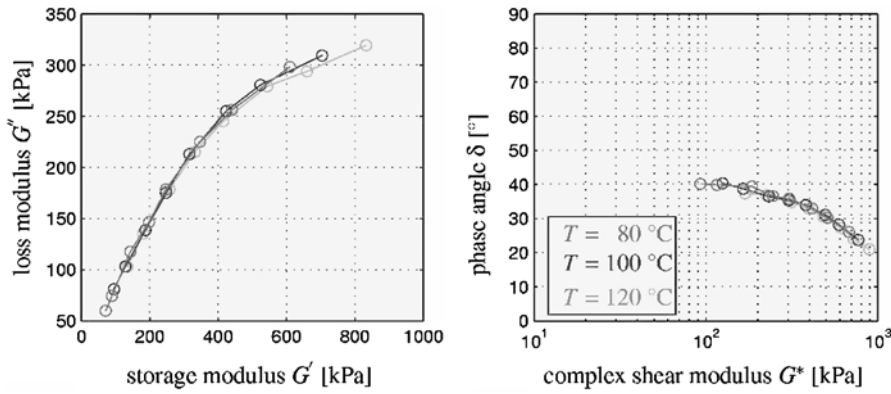
Fig. 8 Temperature independent functions: **a** Cole-Cole diagram, **b** Black diagram

of the phase angle with increasing complex modulus is characteristic for this temperature independent diagram.

5.1 Application of Maxwell model

Because of an serial connection of linear spring and dashpot the resultant shear deformation is given as sum of the shear deformations of both basic elements. The time derivative of the shear deformation provides a relationship for the shear strain rate:

$$\dot{\gamma} = \frac{\dot{\tau}}{G} + \frac{\tau}{\eta'}. \tag{21}$$

Using this relationship Pahl et al. [9] derived the following equations for storage and loss modulus as functions of spring constant E and dashpot constant η' as:

$$G'_r = E \frac{\omega_r^2 \lambda^2}{1 + \omega_r^2 \lambda^2} \quad \text{and} \quad G''_r = E \frac{\omega_r \lambda}{1 + \omega_r^2 \lambda^2}, \tag{22}$$

with

$$\lambda = \frac{\eta'}{E} \tag{23}$$

as the relaxation time. Equation (23) is only valid for the Maxwell model. Elimination of the relaxation time in (22) yields the desired relations for storage and loss modulus

$$G'_r = \frac{\omega_r^2 \eta'^2}{E} \quad \text{and} \quad G''_r = \eta' \omega_r \tag{24}$$

as functions of the unknown material parameters, i.e. the spring stiffness E and the dashpot constant η'. Application of (24) is allowed, if the following relationship is valid:

$$\omega_r \lambda = \omega_r \frac{\eta'}{E} \approx 0. \tag{25}$$

The material parameters, E and η', are determined by means of a Gaussian quadrature and usage of (24) and (25). The results are compared with the RPA measurements in a Cole-Cole as well as a Black diagram.

Figure 9 shows the obtained diagrams for the rubber compound EPDM F. The solid curve represents the result of the Maxwell model. Table 2 summarizes the values of the corresponding material parameters, exemplarily for the materials EPDM A and EPDM F.

From Fig. 9 it is clearly visible that the Maxwell model is not applicable to the investigated rubber compounds. In the Cole-Cole diagram a half circle was observed. In the Black diagram for small complex moduli a phase angle of 90° is obtained. Both properties do not occur for rubber compounds.

From (24) a relation between dynamic viscosity and loss modulus is derived as:

$$\eta' = \frac{G''_r}{\omega_r}. \tag{26}$$

The dynamic viscosity of rubber compounds is usually determined by means of a capillary-viscometer [8]. Pahl et al. [9] establish that application of the Maxwell model for rubber compounds is reasonable for small angular frequencies, only. Therefore, this model is not applicable for the frequency range occurring during the extrusion process of rubber profiles.
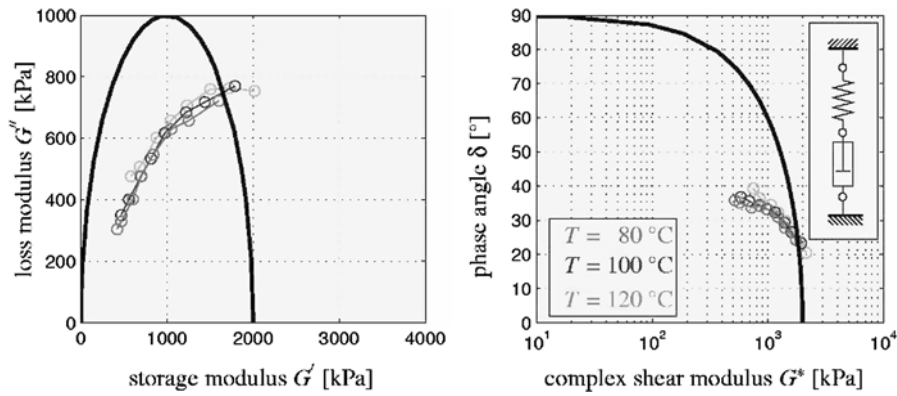
Fig. 9 Application of Maxwell model to EPDM F

Table 2 Material parameters for Maxwell model

Compound code	E [Pa]	η' [Pas]
EPDM A	800×10^3	200
EPDM F	2000×10^3	300

5.2 Application of Wiechert model

A possibility to describe the function G'' (G') more precisely, is a parallel connection of various Maxwell models. The relationships for storage and loss modulus, therefore, results as sum over all single spring-dashpot elements:

$$G'_r = E \sum_{x=1}^{y} \frac{\omega_r^2 \lambda^2}{1 + \omega_r^2 \lambda^2} \qquad (27)$$

and

$$G''_r = E \sum_{x=1}^{y} \frac{\omega_r \lambda}{1 + \omega_r^2 \lambda^2}, \qquad (28)$$

with r as the working point of the corresponding RPA experiment according to Sect. 3.2. Analogous to (23) the relaxation time for the ith Maxwell model is obtained as:

$$\lambda_i = \frac{\eta'_i}{E}. \qquad (29)$$

In (27) to (29) the stiffnesses $E_i = E$ of all linear springs are constant. This assumption has been proven to be helpful for the modeling of viscoelastic behavior. Figure 10 shows the obtained diagrams for the rubber

compound EPDM F. The solid curve represents the result of the Wiechert model. Table 3 summarizes the values of the corresponding material parameters, exemplarily for the materials EPDM A and EPDM F.

From Fig. 10 it can be seen that the Wiechert model is in principle applicable for describing the viscoelastic behavior of rubber compounds. However, the number of material parameters is increased dramatically, because a minimum of ten Maxwell models were necessary to obtain a satisfactory correlation with experimental results. Due to the huge number of material parameters, an investigation of the influence of crystallization and filler degree on the viscoelastic properties is not possible. As for the Maxwell model, in the Black diagram for small complex moduli a phase angle of 90° is obtained.

5.3 Application of Huet model

In order to reduce the number of material parameters and to improve the fitness in the Cole-Cole as well as in the Black diagram the application of the Huet model is recommended. In order to keep the proposed model as simple as possible the following assumptions are made:

- Only one nonlinear dashpot is used ($J_2 = m_2 = 0$).
- The creep compliances of the linear spring and the remaining nonlinear dashpot are equated ($J_0 = J_1$).

With these assumptions following relationships for storage and loss modulus are obtained:

$$G'_r = \frac{J_0}{\Delta_r} + \frac{J_1}{\Delta_r} \Gamma(1 + m_1) \omega_r^{-m_1} \cos \frac{m_1 \pi}{2} \qquad (30)$$
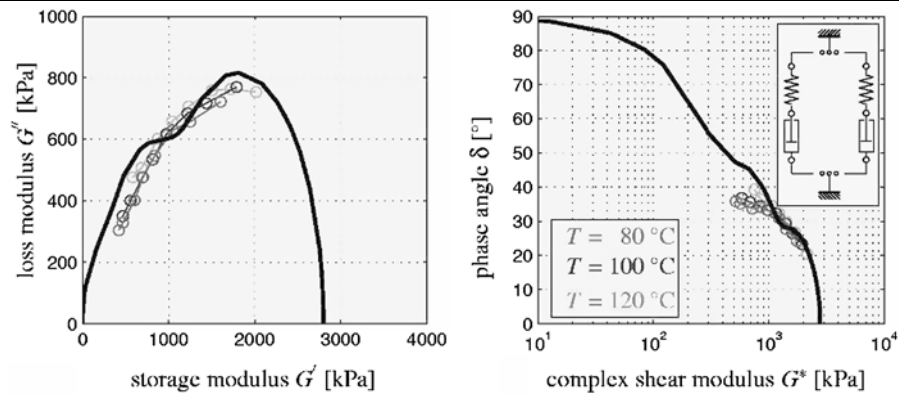
Fig. 10 Application of Wiechert model to EPDM F

Table 3 Material parameters for Wiechert model

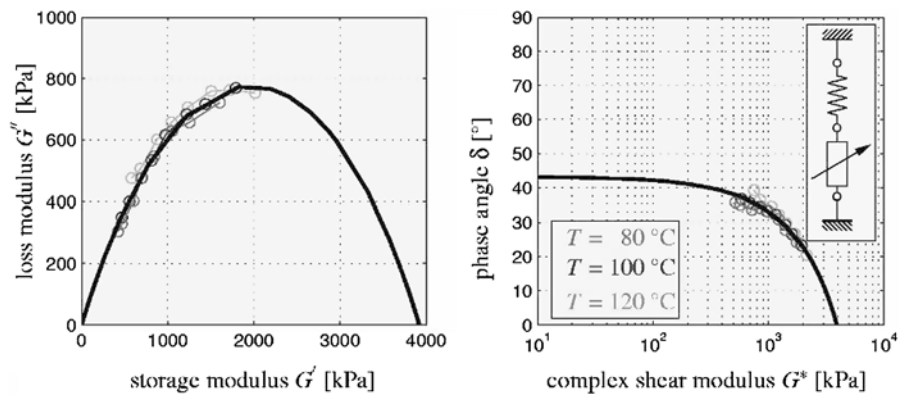| Compound code	E [Pa]	λ_1 [s]	λ_2 [s]	λ_3 [s]	λ_4 [s]	λ_5 [s]	λ_6 [s]	λ_7 [s]	λ_8 [s]	λ_9 [s]	λ_{10} [s]
EPDM A	200×10^3	0.0002	0.0010	0.0050	0.0100	0.0100	0.0500	0.0500	0.1500	0.6000	5.4000
EPDM F	280×10^3	0.0060	0.0060	0.0180	0.0180	0.0180	0.0180	0.1800	0.1800	0.1800	1.8000

Fig. 11 Application of Huet model to EPDM F

and

$$G_r'' = \frac{J_1}{\Delta_r}\Gamma(1+m_1)\omega_r^{-m_1}\sin\frac{m_1\pi}{2} \qquad (31)$$

with the abbreviation

$$\Delta_r = J_0^2 + 2J_0J_1\Gamma(1+m_1)\omega_r^{-m_1}\cos\frac{m_1\pi}{2}$$
$$+ [J_1\Gamma(1+m_1)\omega_r^{-m_1}]^2. \qquad (32)$$

The Gamma function

$$\Gamma(1+m_1) = \int_{t=0}^{\infty} e^{-t}t^{m_1}dt \qquad (33)$$

is the basis for the definition of the Gamma probability distribution. Jäger & Lackner [6] used relationships (30) to (32) for determination of viscoelastic properties of asphalt. Figure 11 shows the obtained diagrams for the rubber compound EPDM F. As for Figs. 9 and 10, the solid curve represents the model result. Table 4 summarizes the values of the corresponding material parameters for all investigated materials.

Figure 11 indicates that the simplified Huet model leads to the best agreement for rubber compounds, although only two material parameters, $J_0 = J_1$ and m_1, were used. The main reason for the good results is the
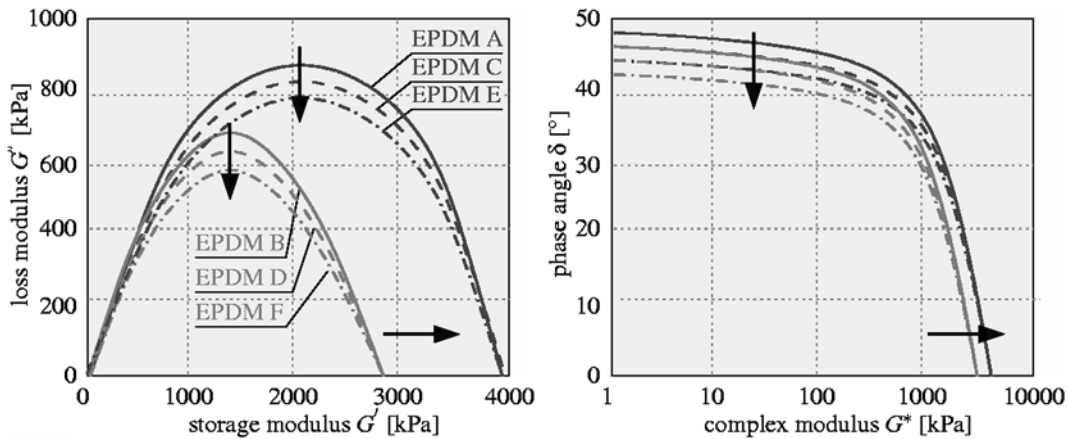
Fig. 12 Identification of influence of content variation on viscoelastic properties by means of Huet model

Table 4 Material parameters for Huet model

Compound code	$J_0 = J_1$ [Pa^{-1}]	m_1 []
EPDM A	7×10^{-10}	0.48
EPDM B	6×10^{-10}	0.50
EPDM C	7×10^{-10}	0.46
EPDM D	6×10^{-10}	0.48
EPDM E	7×10^{-10}	0.44
EPDM F	6×10^{-10}	0.46

fact that, compared to Maxwell and Wiechert model, in the Black diagram smaller phase angles for small complex shear moduli are obtained. Therefore, the simplified Huet model will be used in order to identify the influence of crystallization and filler degree on the viscoelastic properties of rubber compounds.

6 Influence of crystallization and filler degree

For realistic numerical simulations the knowledge of material parameters are required, which describe the material behavior as realistic as possible. In the present case, the simplified Huet model reproduces the viscoelastic behavior of the investigated rubber compounds satisfactory. Its application allows identification of the influence of both, the crystallization degree of the basic polymer and of the amount of used filler material, on the viscoelastic properties of the investigated rubber compounds.

Under consideration of the material parameters of Table 4, a clear trend for both properties is detected.

Using EPDM with amorphous chemical structure, i.e. a low crystallization degree, leads to an increase of the elastic behavior of the corresponding rubber compound, i.e. to an increase of the storage moduli G'. This is modeled by an increase of the compliance of the linear spring. Furthermore, an increase of the filler content of the rubber compound, i.e. an increase of the filler degree, yields a decrease of the viscous properties, i.e. a decrease of the loss moduli G''.

Both properties are depictured in Fig. 12 by means of a Cole-Cole as well as a Black diagram. In the Cole-Cole diagram, the vertical arrows mark the increase of the corresponding filler degree; the horizontal arrows mark the decrease of the crystallization degree. In the Black diagram, the same conclusions can be drawn. Thus, the nonlinear Huet model allows a decoupling of both material properties.

7 Conclusions

For determination of the viscoelastic properties of polymers and rubber compounds, respectively, experiments with a torsional rheometer are required. The choice of rheological models for description of viscoelastic properties of rubber compounds depends on the considered material. In this paper, three different rheological models were used.

The Maxwell model (connection in series of linear spring and linear dashpot) is the simplest model for application to viscoelastic materials such as rubber. Results showed that the application of this model

is possible for a small frequency range, only. The usage for modeling of extrusion tasks is, therefore, not possible.

The Wiechert model (connection in parallel of several Maxwell models) shows a better correlation between experiment and model prediction. However, a huge number of necessary material parameters the application to rubber compounds is questionable. Furthermore, this model fails for small complex moduli due to wrong prediction of phase angles.

A simplified Huet model (connection in series of linear spring and one nonlinear dashpot with equal creep compliances) shows the best agreement between experimental and modeling results. With only two material parameters the viscoelastic behavior of rubber compounds is modeled quite satisfactory. However, for the mechanical modeling of rubber, the introduction of nonlinearity is required. Finally, application of the Huet model for materials with different crystallization and filler degrees enables identification of both properties from the viscoelastic properties of rubber.

As a next step of an ongoing research project, finite element simulations of injection heads for the extrusion of rubber profiles will be done. For that purpose, the implementation of the Huet model in CFD software is required.

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