Modeling the Pressure Decay Curve of a Capillary Rheometer

Russ Warley

Lord Corp., Mechanical Product Division, 2000 W. Grandview Blvd., Erie, PA 16514, USA

Email: russ_warley@lord.com
Fax: x1. 814.868.0640

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Abstract:
A mathematical model for the transient pressure response in a capillary rheometer is implemented and validated with experimental data for both a natural rubber compound and a silicone rubber compound. The pressure decay curve after the cessation of motion of the instrument piston is shown to be consistent with the extrapolation of the power law model to shear rates two decades lower than experimentally attainable in the instrument employed. The model is useful for extending the range of the instrument in question by approximating material properties at shear rates lower than attainable in a steady flow experiment.

Zusammenfassung:
Ein mathematisches Modell für die zeitabhängige Druckantwortfunktion in einem Kapillarrheometer wurde implementiert und an Hand experimenteller Daten für einen Naturkautschuk und einen Silikonkautschuk validiert. Es wird gezeigt, dass der Druckabfall nach dem Abklingen der Bewegung des Gerätekolbens mit der Extrapolation eines Power-Law Ansatzes zu solchen Scherraten konsistent ist, welche zwei Dekaden unterhalb der in unserer Apparatur realisierbaren Raten liegen. Das Modell kann verwendet werden um den Scherratenbereich der Apparatur zu erweitern, indem Materialeigenschaften für solche Raten angenähert werden, welche in einem Experiment mit konstantem Materialfluss nicht realisiert werden können.

Résumé:
Un modèle mathématique pour la réponse transitoire de la pression dans un rhéomètre capillaire est éxécuté et validé par des données expérimentales obtenues avec un caoutchouc naturel et aussi un caoutchouc siliconé. La courbe de chute de pression consécutive à l'arrêt du mouvement du piston de l'instrument se révèle être consistente avec l'extrapolation d'un modèle de loi de puissance à des vitesses de cisaillement qui sont deux décades plus petites que celles atteintes expérimentalement par l'instrument employé. Le modèle est utile afin d'étendre les capacités de l'instrument en question au moyen d'une approximation des propriétés du matériau à des vitesses de cisaillement plus petites que celles accessibles par une expérience d'écoulement permanent.

Key words: rubber, elastomer, capillary, silicone, natural rubber

1 INTRODUCTION

The pressure in a capillary rheometer does not change instantaneously to the steady value upon initiation of constant speed piston motion. This is true even though the driving piston attains the target speed almost instantaneously. The reason for the sluggish transient response is that the entire barrel of material is compressed as the pressure increases due to the piston movement. The initial flow rate out of the capillary die is lower than the target value until the pressure transient dies out and steady state conditions are attained. It is only when the pressure has achieved a steady value that the flow rate of elastomer from the die exit is the desired value. Hatzikiriakos and Dealy [1] have shown that the time required to achieve steady state pressure depends on the volume of material in the barrel, the speed of the piston, the compressibility and rheology of the material, and the resistance of the capillary die. Recent work by Pérez-Trejo et. al. [2] is in agreement with the original work. The pressure decay curve for HDPE in a multipass capillary rheometer was found by Ranganathan et. al. [3] to be in good agreement with predictions based on flow driven by the compressibility of the material. A capillary pulse viscometer has been developed by Kokal et. al. [4] that is based on the pressure decay curve of a fluid that is suddenly pressurized.

The motivation for this work came from the desire to check the validity of the power law model at shear rates lower than attainable in the instrument in our laboratory. It is well known that the power law model is an approximation to

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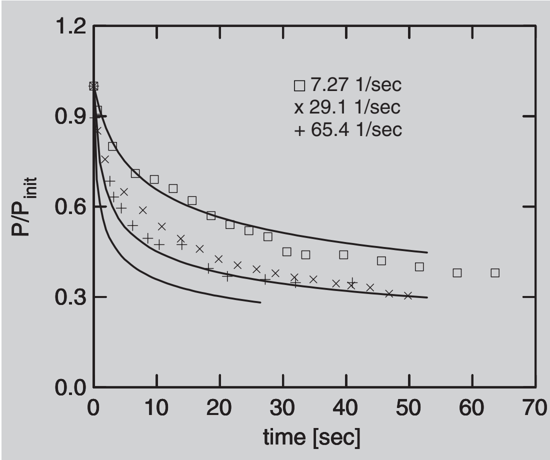

Figure 9 (left above): Decreasing pressure transient for NR compound in 10:1 die at 110°C

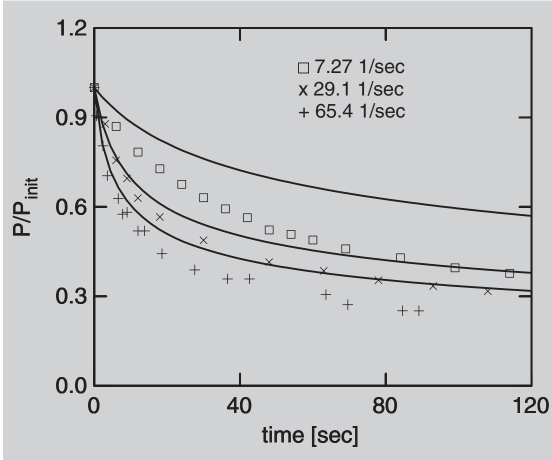

Figure 10 (right above): Decreasing pressure transient for NR compound in 30:1 die at 110°C

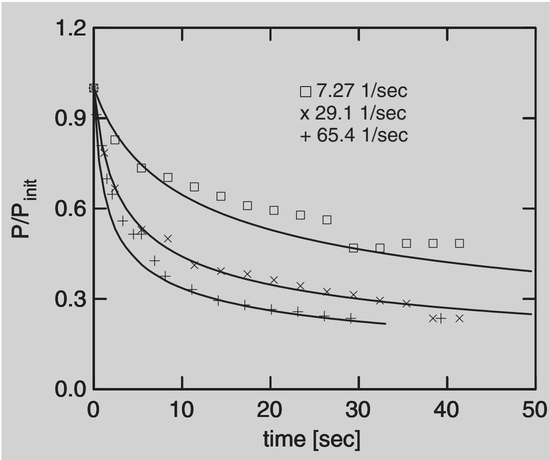

Figure 11 (left below): Decreasing pressure transient for PVMQ compound in 10:1 die at 120°C

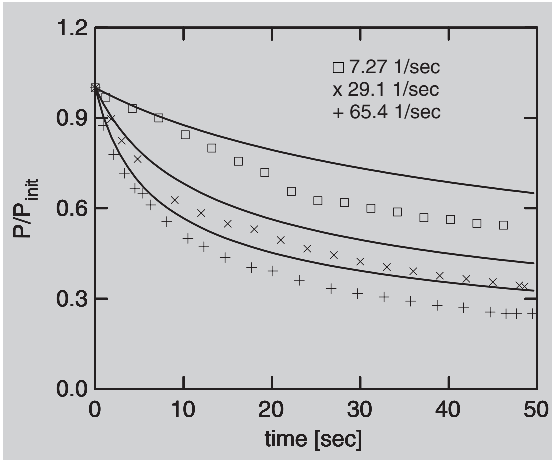

Figure 12 (right below): Decreasing pressure transient for PVMQ compound in 30:1 die at 120°C

main purpose of this work, however, is to predict the pressure decay curve which occurs after cessation of piston motion. This predicted curve is necessarily based on an extrapolation of the material model (in this particular case the power law model) to shear rates below those attainable in our instrument. These curves are then compared with experimental pressure decay data to check the validity of the power law model in the range of shear rates occurring during leak down.

This model was solved for the case of the pressure decay curves from the same capillary rheometer runs by setting v_p = 0 and using the measured steady pressure that was observed in the rising pressure part of the experiment as the starting pressure (P_i). The measured rather than the predicted steady pressure was used to test the temporal prediction of the leak down curve in the absence of the deviation of the predicted steady pressure value from the experimental data. This was done to focus the analysis on the temporal prediction of the leak down model rather than a test of the fit of the power law expression to the steady flow data. The shear rates indicated in the legends of Figs. 9 to 12 indicate the steady flow conditions used to generate the starting pressure for the leak down and hence the degree of compression of the material in the barrel at the start of the experiment. At t = 0, the piston was stopped and held in place for the remainder of the experiment. Note that the amount of material remaining in the barrel is indicated by the value of L_{bo} in the model. This was done by observing the flow time required to achieve the steady flow pressure followed by application of Eq. 3. The predictions for NR are shown in Figs. 9 and 10 and the predictions for PVMQ are shown in Figs. 11 and 12.

The correspondence of the prediction with the data is fairly good for NR at 10:1 (Fig. 9) and for the higher initial pressures for the 30:1 die data (Fig. 10). The pressure data for the 30:1 die with the lowest initial pressure is significantly lower than that predicted by the model. Deviation of the material flow from power law behavior would be expected to affect the 30:1 die more than the 10:1 die data since the viscous component of the total die pressure is more significant in the case of the 30:1 die. The observed deviation is not expected to be due to wall slip since the pressures are higher in the case of the 30:1 die which would tend to decrease wall slip. The P_{ent} value at the lowest shear rate in Fig. 2 does appear to be lower than the rest of the data would indicate. This would not seem to be the reason for the deviation of the 30:1 data since P_{ent} would exert the most influence on the 10:1 die data. The reason for the large deviation of the

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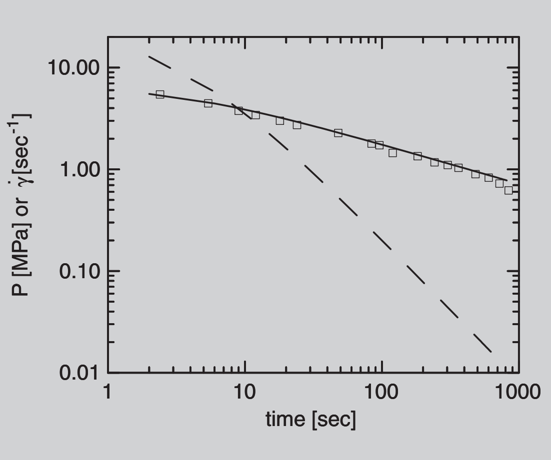

Figure 13: Long-time pressure decay trend for PVMQ compound in 20:1 die at 120°C. Solid line: P [MPa], dashed line: [s^{-1}].

data generated from the low initial pressure condition is not known.

The correspondence of the data for the PVMQ material in Figs. 11 and 12 is better. The same qualitative trend for the prediction to yield higher values than the experimental data for the 30:1 die is evident. The deviation is much less severe in this case, however. Particularly with PVMQ the extrapolation of the power law model seems to provide acceptable accuracy in the range investigated.

In order to provide an expanded test of the model, an additional experiment was carried out with PVMQ at 120°C using a 20:1 die for an extended time period. The results of this experiment, along with the model prediction, are shown in Fig.13.

It is clear that the model captures the essence of the phenomena occurring in the die during leak down. Superimposed on Fig.13 is the shear rate that exists in the capillary at any given time. Since the lowest practical steady-flow shear rate on our instrument is 3.6 s^{-1} this represents an indirect way of verifying the power law and entrance pressure models at shear rates unattainable in steady state experiments. The power law and entrance pressure models for PVMQ apparently hold to apparent shear rates as low as 0.01 s^{-1}. The experiments were not extended to lower shear rates due to limitations of the pressure transducer.

It may be possible to extend this idea further to recast the problem as one of fitting the pressure decay curve to a more realistic constitutive equation such as the Cross model. In principle this should allow an extrapolation into the region where the viscosity approaches the limiting steady-state value. The data that were gathered in these experiments did not require the use of a more complex material model, however.

A limitation of using this type of data for verification of low shear rate behavior is the tolerance of the material to thermal history. Rubber compounds are often characterized in the fully compounded condition where crosslinking agents are present. If the test temperature and test time result in appreciable crosslinking of the elastomer while in the barrel, then the analysis is no longer valid.

4 CONCLUSION

The analysis method and material property data used are sufficient to represent the pressure trace on the capillary rheometer under the range of conditions employed in this work. The model gives implicit verification of the material property data at shear rates well below those attainable in our steady flow experiments with the instrumentation in our laboratory. The model can be used to interpret capillary rheometer data, as well as to infer the material rheology at shear rates that are lower than those attainable with the same instrument under steady flow conditions.

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