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High strain rate characterization of low-density low-strength materials
A study on the flow, failure, and rupture mechanisms of low-density polyethylene in controlled-stress uniaxial extensional flow

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Synopsis

The main aim of this work is to study the flow, failure, and rupture dynamics of a benchmark low-density polyethylene (BASF Lupolen 1840H) in true tensile creep conditions in both the viscoelastic and elastic deformation regimes. For this, we used a novel extensional rheometer that for the first time allows real controlled-stress conditions to be applied to the materials (as opposed to nominally controlled-stress) until physical rupture occurs (as opposed to being limited by maximum Hencky strain). We observed that constant strain rate was achieved for all flow conditions, and depending on the level of applied tensile stress, one or two states were obtained. In the former case, the observed mode of rupture was ductile, or liquid-like, and in the latter was cohesive, or elastic-like. The coupling at the molecular level between these flow and rupture mechanisms is not yet fully understood, although some recent studies in the literature may be able to offer at least partial explanations. © 2011 The Society of Rheology. [DOI: 10.1122/1.3596210]

I. INTRODUCTION

Extensional rheology of entangled polymer melts has been the subject of a relatively strong computational, theoretical, and experimental effort over the years (see, for example, the works of Meissner (1971, 1985a, 1985b); Laun and Münstedt (1976, 1978); Münstedt and Laun (1981); Petrie (1979); Maia et al. (1999); Bach et al. (2003a, 2003b); Sentmanat (2004); Münstedt et al. (2005); Rasmussen et al. (2005); and Nielsen et al. (2006), for a sample of the breadth of work performed thus far) because many industrially important processes, such as fiber and melt spinning, film blowing, and blow molding, are dominated by the fluids’ extensional properties. Also, the study of this type of flow allows an insight into the molecular structure of the materials to be gained, since extensional behavior is very dependent on the particular structure (molecular weight, molecular weight distribution, degree of branching, etc.).

In comparison with the more general work on extensional rheometry of melts, relatively little attention has been paid in to the physical mechanisms of failure and rupture of polymers under extensional flow, despite these being very relevant to a number of important issues in the processing of polymeric materials, including melt filament breakage in fiber formation and the onset of surface roughness (sharkskin) in melt extrusion from a die.

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One of the main early exceptions was the work of Vinogradov and co-workers (1972, 1975a, 1975b, 1975c, 1977, 1981), who were the first to address the problem of failure and rupture under extensional flow (in nominally controlled rate and controlled-stress modes) of monodisperse polymers of different molecular weights and at different temperatures. Vinogradov and co-workers (1975b) presented a master curve obtained for the dependence of the strain at break on stress in nominally constant stress experiments. Based on the experimental results from uniaxial stretching of monodisperse polymers in the flow regime at constant strain rate [Vinogradov 1975a; Vinogradov et al. 1975b; Vinogradov and Malkin 1980; Malkin and Vinigradov 1985], Malkin and Petrie (1997) presented a similar master curve describing the different types of behavior depending on the applied strain rate. Malkin and Petrie (1997) argued for the existence of four zones of distinct rupture behavior, as previously proposed by Vinogradov and co-workers (1975a, 1975b). In zone I (corresponding to very low strain rate), there is purely viscous flow, steady state elongation can be achieved, and the sample will break by ductile rupture due to surface tension. Zone II corresponds to the transition to the viscoelastic region and is characterized by the appearance of a non-negligible elastic component in the flow as strain rates increase; rupture is still ductile, but partial recovery of the deformation upon rupture occurs. Zone III is referred to as the rubbery zone and is reached by further increasing the strain rate. It is characterized by a completely elastic deformation in which the total strain at rupture is almost completely recovered, and rupture is cohesive and is followed by an almost complete elastic recovery. Zone IV was designated as the glassy regime and occurs at extremely high strain rates. Here, a decrease in the ultimate strain at break with strain rate is postulated, and rupture is of a brittle nature.

Other related experimental studies can be found in the literature, for controlled rate conditions, are the works of Ide and White (1977, 1978), where the authors found that high-density polyethylene (HDPE) and polypropylene (PP) would exhibit necking and would rupture in a ductile manner, while low-density polyethylene (LDPE), polystyrene, and poly(methylmethacrylate) (PMMA) would show cohesive rupture. Pearson and Connolly (1982) also performed experiments in extension with polystyrene, PMMA, and a co-polyester and found that the Hencky strain to break was almost independent of strain rate.

There is also some computational work in the literature such as, for example, Hassager et al. (1998) where ductile failure of viscoelastic fluid filaments using numerical simulations is studied. McKinley and Hassager (1999) used the Doi–Edwards (DE) model [Doi and Edwards (1979, 1986)] in order to simulate the failure behavior for linear polymer melts and for branched materials using the Pom-Pom model [McLeish and Larson (1998)].

In what regards the issue of the failure of polymers in extensional flows, the Considère criterion (1885) continues to be the most used criterion for the onset of failure. For example, it was used by McKinley and Hassager (1999). This criterion estimates the failure of elastic solids and states that for such materials nonuniform deformation (necking) will occur after the engineering stress (tensile force divided initial cross sectional area) passes through a maximum. The Considère criterion also states that from this point on, the deformation is no longer purely extensional and homogeneous. However, there are many arguments in the literature as to whether this is also a valid criterion to define failure of viscoelastic fluids. Joshi and Denn (2004) and Lee et al. (2002) argue that the Considère criterion is used improperly to predict the strain to failure in polymer melts in conditions where irreversible flow is present, because yielding and failure do not occur simultaneously in these cases. More recently, Petrie (2009) in a review on the recurring use of the Considère criterion concludes that “… the use of the Considère construction for materials
which flow (…) at best requires great care and at worst leads to confusion and misleading conclusions.” Barroso et al. (2010) in a case-study of failure of a polyisobutylene (PIB) melt in uniaxial extension also showed clearly that nonuniform deformation was only achieved well after the maximum in engineering stress had passed.

Joshi and Denn (2003, 2004) developed a scaling model that tries to predict rupture in the rubbery zone, taking into account the recoverable component of the total strain at break. The authors consider a polymer suffering elongation in the rubbery regime at a macroscopic rate and they assume an affine deformation on one test chain, which is due to the effective frictional force between the test chain and the surrounding molecules. The model assumes a catastrophic failure when the frictional force of an entangled chain can no longer balance the tension in the molecular chain. The authors also presented a prediction for rupture at constant stress, assuming that the rate of elongation is maximal initially and then decreases as the steady state is approached, which means that the instantaneous value of the elongation rate fixes the maximum permissible tension. The authors argue that “a rupture occurs when the maximum permissible tension decreases to a value equal to the tension” being experienced by the chains. Comparisons were performed with some sets of data from Vinogradov (1975a) and Vinogradov et al. (1975c), and fairly good agreement was found in the elastic regime. The authors recognize, however, that the theory is not applicable when the flow is in the viscoelastic regime.

An intensive and ongoing work is being conducted by Wang and co-workers (2007a, 2007b, 2008, 2009) to study the integrity of a polymeric material under external deformation. In their work, the authors argue that the DE [Doi and Edwards (1979)] theory is not adequate to treat a many-body phenomenon such as failure of polymers melts, since the DE theory is a single-chain mean-field treatment of chain dynamics, and that in the elastic deformation regime, the maximum engineering stress represents the network yield point, which leads to visible material failure when nonuniform extension eventually occurs. In their studies, Wang and co-workers performed studies in three different regimes. In the regime of very slow deformation rates (terminal flow regime), Wang and Wang (2008) observed that the maximum in $\sigma_{\text{eng}}$ coincides with the moment when flow takes place due to the irreversible rearrangement of the initial entangled network by molecular diffusion. In the elastic deformation regime, the authors [Wang and Wang (2008); Wang et al. (2007b)] found a series of scaling laws that they believe indicates that the maximum in tensile force has the same physical meaning in shear and extension, since similar results were found in previous work in shear [Wang et al. (2007a); Boukany and Wang (2009)]. According to Wang and Wang (2008), when the elastic retraction force equals the intermolecular gripping force and consequently the tensile force ceases to increase (and eventually reaches a point where starts decreasing since the interchain sliding), the maximum in $\sigma_{\text{eng}}$ is achieved. They argue that this represents the yield point and the uniform extension cannot be sustained afterward, leading to the break of the network entanglement and consequently to the visible failure of the sample. In the viscoelastic regime, their work is not as complete due to the inability to achieve the steady state, but they argue that the same molecular phenomena will occur and provide supporting evidence.

Tensile creep experiments are very relevant not only because steady state is more quickly achieved than constant strain rate conditions but also because, as was mentioned before, they are prime candidates to provide insights into possible rupture mechanisms, the liquid–solid transition, and into flow instabilities related with extension-dominated phenomena, such as sharkskin and melt fracture, which are essentially stress dependent and are very important in limiting the optimization of operating windows during processing sequences.
Although a wide body of work exists on controlled rate extensional rheometry for polymer melts, the more recent of which focus on ease of use and modularity [Maia et al. (1999); Sentmanat (2004); Sentmanat et al. (2005); Hodder and Franck (2005)], there have only been a few attempts that we are aware of at developing controlled-stress capabilities.

Work in this field was pioneered and carried out by Cogswell (1968), who developed an apparatus where the stress is nominally maintained constant by a weight acting on a moment arm which decreases as the extension proceeds, assuming uniform extension, \textit{i.e.}, neglecting nonhomogeneities in the deformation. Vinogradov et al. (1972b) presented a more elaborate constant stress rheometer where hydrodynamic drag was reduced and the strain was measured by a photoelectric follower without friction. However, this instrument also assumes that the deformation is homogeneous and, as such, the stress is only imposed nominally, because if the deformation is inhomogeneous, the true stress in the sample is not the calculated one. Münstedt and co-workers (1998) used a filament stretching device to control the stress via a feedback loop of the tensile force, which decreases in time in order to keep the stress in the sample constant; this solution can overcome the assumption of homogeneous deformation because of the control loop, but the maximum achievable deformation is still limited by the achievable length of the sample. More recently, Bach et al. (2003a) presented a filament stretching rheometer (FSR) technique to be applied to polymer melts at high temperatures, with the aim to address the existent problems with temperature gradients. This instrument is really a modification to the original Münstedt-type device and, as such, suffers from the same drawback of the deformation being limited by the available plate displacement. Sentmanat (2004) developed an extensional device that when working in “controlled” stress mode imposes an exponentially decreasing force to the sample in order to keep the nominal stress constant [Sentmanat et al. (2005)]. Although very flexible and accurate, if the right experimental precautions are taken, the Sentmanat extensional rheometer (SER) is still limited by the low achievable Hencky strains (~4) and the assumption of constant stress, without accounting for possible experimental sources of error or nonhomogeneities in the deformation. Recently, Hassager and co-workers (2009) used the SER, the extensional viscosity fixture by TA Instruments, extensional viscosity fixture (EVF) [Hodder and Franck (2005)], and a FSR [Bach et al. (2003b)], and confirmed the limitations in Hencky strain of the first two and, to a lesser degree, also of the FSR. Thus, no existing rheometer can perform true tensile creep experiments up until the physical rupture of the sample.

Both these limitations (the inability to control the actual tensile stress the sample is experiencing and/or the low maximum achievable strain) have been overcome for the first time in a recent controlled-stress extensional rheometer (CSER) developed by Maia and co-workers (2008), the instrument used in this work to study the flow, failure, and rupture in different viscoelasticity regimes of a standard branched (low-density) polyethylene in controlled-stress uniaxial extensional flow up to the point of physical rupture of the sample.

II. THE CONTROLLED-STRESS EXTENSIONAL RHEOMETER

This device works under the principle used by Maia et al. (1999) of what could be considered a “half Meissner” rheometer, which is composed of a fixed clamp and a rotating clamp with two counter-rotating rollers that pull the sample, which is broadly based on the fiber wind-up device of Macosko and Lorntson (1973). Despite the difference in strain conditions at both ends of the test sample, this set-up was shown to yield homogeneous flow for initial L/D (for cylindrical samples) or L/W (for rectangular samples)
ratios in excess of 12, provided (as in any other extensional rheometer) sample preparation and loading is carefully performed and samples are free of residual stresses at the onset of testing [Barroso et al. (2002)]. This set-up was applied to a Paar Physica MCR 300 rheometer.

Figure 1 shows the general outlook of the rheometer. The extensional flow cell is attached to the main body of the rotational rheometer (1); a high-temperature oil bath replaces the base plate of the rheometer and supports and heats the sample (2); a high resolution video camera measures its cross section of the sample, an adapter (3) allows the torque sensor to transmit movement to the rollers, (4) and a temperature control module controls the bath temperature.

The sample was mounted between a pair of pulling rollers and a clamp; in the latter case, the clamp is not aligned with the deformation, but rather orthogonal to it, with the initial portion of the sample wrapped around it, as seen in Fig. 1, so that end effects are minimized and there is no fresh material being fed into the sample during the deformation (which is ensured by marking the beginning of the test section with a marker and making sure this does not move during the experiment).

In order to control the stress, it is necessary to use a feedback loop that resorts to a customized software to control the torque and thus maintain the stress constant. The first known feedback loop used to control the stress in the rheometer was presented by Meissner (1972); however, this early solution again assumed uniform extension and, as the author states, is limited by the fact that “the rotational speed of synchronous motor M1 is so low that clamp Z1 performs only 3/4 of a full rotation within the duration of the whole test.” As is shown in Fig. 2, the initial parameters are input using the original software of the rheometer (1); once the experiment starts, the sample cross section is captured at the pulling rollers by a high resolution video camera (2) at an adjustable rate between 10 and 50 frames per second; this information is, in turn, fed into the torque

**FIG. 1.** CSER: Left—frontal view; right—rear view.

**FIG. 2.** Schematic of the feedback control loop. By performing data acquisition on the sample cross section in almost real time, true constant stress conditions can be achieved.
control software that adjusts the torque imposed on the rollers in real time in order to keep the tensile stress constant (3). The point of measurement of the cross section is irrelevant while the deformation is homogeneous, but after sample failure and inception of nonhomogeneous deformation, it is still possible to control the stress acting on the thinnest part of the sample, i.e., the one that is undergoing higher tensile stress, by measuring its cross section at the rollers (which is where necking is more pronounced). This solution ensures that the stress is controlled regardless of deformation homogeneity and that deformation is only limited by sample rupture.

The main initial validation step was to ensure that the feedback loop works and the stress in the sample is kept constant. Initially, empty runs were performed in order to check for instrument friction, and the measured torque values were used as a baseline for the measurements with the samples, i.e., the empty value was subtracted from the actual measured value when samples are loaded.

The evolution in time of the real stress acting on the sample was calculated from the imposed torque at each time step of image acquisition and compared with the set value. Figure 3 shows the results for an imposed tensile stress of $10^5$ Pa, and it is clear that the real stress deviates from the imposed value by less than 0.1%, which is excellent, with larger errors at long times when the sample is very thin and camera resolution starts being insufficient.

III. MATERIALS AND SAMPLE PREPARATION

This set-up was used in this work to study a benchmark LDPE, Lupolen 1840H, very well characterized in literature [see, for example, Sentmanat et al. (2005); Münstedt et al. (1998); Gabriel et al. (1998)] that was tested at 150°C. In order to minimize errors [Barroso et al. (2002)], samples were prepared by a very slow extrusion at 180°C in a
capillary rheometer, in order to produce cylindrical samples with good surface finish and constant diameter below 2 mm. Once loaded onto the rheometer, they were allowed to relax for a few minutes at the test temperature before the experiment started, in order to remove any residual stresses. Finally, any slack that developed during the relaxation process was removed. The applied tensile stress varied between $3 \times 10^3$ and $10^6$ Pa; for stresses lower than $3 \times 10^3$ Pa, there was slip at the rollers that corrupted long-time data, and for stresses above $10^6$ Pa, the experiment was too fast (typically less than 0.2 s) for accurate data to be acquired and instrument inertia began being a further source of error.

IV. RESULTS AND DISCUSSION

When performing tensile creep experiments up to physical sample rupture, two different behaviors were observed, as shown in Fig. 4. Analyzing Fig. 4(a), it is possible to observe that for a constant sample stress of $10^6$ Pa, dynamic steady state, i.e., the point when constant strain rate and extensional viscosity are achieved, is reached almost instantaneously upon flow inception, with rupture occurring after a very short time (approximately 0.12 s). However, when the stress applied decreases by an order of magnitude [see Fig. 4(b)], two distinct steady states are clearly achieved, with the second one corresponding to a much lower effective strain rate than the initial one.

At this point, it is important to point out that the experiments are quite reproducible and even for the very highest stresses we do always observe a single steady state only, so we believe instrument inertia is not playing a significant role here.

Nominally, tensile creep experiments have been performed before on this material by Münstedt et al. (1998) and Sentmanat et al. (2005). Figure 5 depicts the steady state extensional viscosity, normalized by the zero-rate extensional viscosity for experiments from both groups (in keeping with the designation used by Münstedt et al. (1998), “stressing experiments” mean experiments in controlled rate mode, while all other filament stretching and SER experiments were performed under nominally constant tensile stress) and from the present work and for high stresses excellent agreement is obtained by all three instruments in all four experimental sets. However, for lower and medium stresses, although the results for the first steady state plateau are in excellent agreement between all groups, they differ in the existence of this second steady state in our experiments, as is shown by the full circles in Fig. 4(b). This second steady state was also achieved by Münstedt et al. (1998) for a linear low density polyethylene (LLDPE) sample but not for LDPE and was also not observed in the SER for LDPE. However, the authors do not explain the phenomenon, stating simply that “two different molecular processes within the LLDPE have to be considered,” without further explanation. One likely cause for neither of the previous groups’ experiments reaching the second steady state in LDPE is the fact that neither the SER nor the filament stretching apparatus can reach the necessary Hencky strains for this second steady state to develop.

It is important to notice that two steady states were only observed when ultimate sample rupture was ductile [see Fig. 6 (left)]; when sample rupture was cohesive, with virtually 100% strain recovery [Fig. 6 (right)], only one steady state was ever observed.

Although this phenomenon was not previously observed and its origins are not immediately apparent, a possible explanation may lie with the previous observations by several researchers [Rokudai (1979); Gahleitner (2001)] of strain-induced reversible chain disentanglement in strong shear and extension-dominated flows of various polyolefins, such as LDPE, PP, and bimodal HDPE, which is also the physical phenomenon behind the failure model of Wang and co-workers [Wang and Wang (2008, 2009)]. In what regards our experiments, it is clear that at imposed stresses lower than approximately $2 \times 10^5$ Pa, the
deformation ceases to be fully elastic and flow has time to develop. Then, it is possible that at some point after the first steady state is achieved, the elastic retraction force will surpass the intermolecular gripping force and lead to partial disintegration of the initial entangled network. If this were to happen, the low molecular weight fractions and/or the side branches would be the most prone to disentangle, which would turn the material into an effectively less branched entangled polymer in which entangled long chains are the

![Graph 1](image1.png)

**FIG. 4.** Tensile creep measurements at: (a) $10^6$ Pa, one steady state is achieved; (b) $10^5$ Pa, two steady states are achieved.

![Graph 2](image2.png)

**FIG. 5.** Comparison of steady state extensional viscosity $\eta_E$ (scaled with $3\eta_0$) as a function of the imposed tensile creep stress measured with CSER and with the data taken from literature. As per the original designations, "stressing experiments" mean experiments in controlled rate mode. All other filament stretching and SER experiments were performed under nominally constant tensile stress.
effective load-bearing elements. When this phenomenon is complete, it is feasible (and this is only a conjecture) that the rheological response stabilizes again in a second steady state, but at a much lower effective rate, due to the high molecular weight of the load-bearing chains. This model is also consistent with the observed modes of rupture since it is to be expected that for higher stresses the elongation time is not enough for disentanglement (and therefore flow) to occur, and the sample will rupture like an elastic solid, i.e., cohesively. If partial disentanglement has time to occur, then a portion of the deformation will be nonrecoverable and partial recovery upon ultimate rupture is to be expected. This is exactly what is observed. Eventually, at very high strains, even this second network will disintegrate, leading to sample failure and the visual onset of inhomogeneous flow (necking) and, eventually, to ductile, i.e., liquid-like, rupture. This sequence of events is supported by our experimental observations, as exemplified in Fig. 7 for the experiment at 10^5 Pa, previously shown in Fig. 4(b).

It must be emphasized, however, that the evidence provided by this work alone is not enough to be able to prove or otherwise the molecular scenario above and, as such, until further and more systematic work with different polymers with well-controlled molecular structure, i.e., molecular weight, molecular weight distribution, and degree of long-chain branching, is performed, it remains as a working hypothesis only.

**FIG. 6.** Examples of ductile rupture (left), where there is the non-homogeneous deformation (necking) prior to rupture, and cohesive rupture (right), where total strain recovery is obtained.

**FIG. 7.** Torque evolution in time for $\sigma = 10^5$ Pa, showing ductile rupture proceeded by two dynamic steady states.
Prokunin (1978) and Prokunin and Filippova (1979) observed two force maxima in time for both LDPE and HDPE in constant strain rate experiments; in HDPE, the strain only became nonuniform after the second maximum, and in LDPE, it was uniform until rupture. The similarity with our observations in controlled-stress conditions of nonuniform flow after two consecutive steady states is apparent, but without further work, it impossible to tell if there is any relation between the phenomena in both deformation modes.

The only consistent theoretical framework specifically for constant stress experiments was developed by Malkin and Petrie (1997), with the authors arguing that in the elastic deformation regime the durability, \textit{i.e.}, the time-to-rupture, will decrease with the power $-3$ of the applied stress. Although developed for tensile creep conditions, Malkin and Petrie (1997) argue that their results should also be valid for constant rate experiments, but our group has found [Barroso et al. (2005, 2010)] that this is not the case experimentally. In tensile creep experiments, however, their prediction seems to hold, as seen in Fig. 8. In fact, from our experiments, it is clear that not only they hold in the elastic regime (except at the very highest stress, where experimental errors due to the very short times involved, such as instrument inertia, may come into play), but the behavior is maintained down to stresses for which the deformation is clearly viscoelastic (and the rupture ductile), but where the elastic component is still important.

V. CONCLUSIONS

The present work used a new rheometer able to perform very accurate controlled-stress experiments in uniaxial extension up to physical sample rupture to show that low-density polyethylene has two distinct rupture mechanisms and that these are closely related to the existence or not of a second steady state during flow. At stresses in excess of approximately $2 \times 10^5$ Pa, viscous flow is negligible, and steady state conditions are achieved almost instantaneously, with the material showing cohesive, \textit{i.e.}, solid-like, rupture. At lower stresses, the deformation has a non-negligible viscous component, and two steady state regimes are observed before visual onset nonhomogeneous flow. In this case, physical rupture is ductile, \textit{i.e.}, liquid-like.

The meaning of this second steady state plateau is not yet clear, but it is compatible with disentanglement of the polymer network, \textit{i.e.}, flow, at low and medium stresses that does not have time occur at high stresses, where the deformation is predominantly elastic;
the end of the first steady state corresponds to the onset of low molecular weight chain disentanglement, and the second plateau corresponds to the new equilibrium state of what has become effectively an entangled (quasi) linear polymer of higher molecular weight. Work is ongoing with materials of varying but controlled molecular weight, molecular weight distributions, and degree of branching to further confirm or disprove this hypothesis.

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