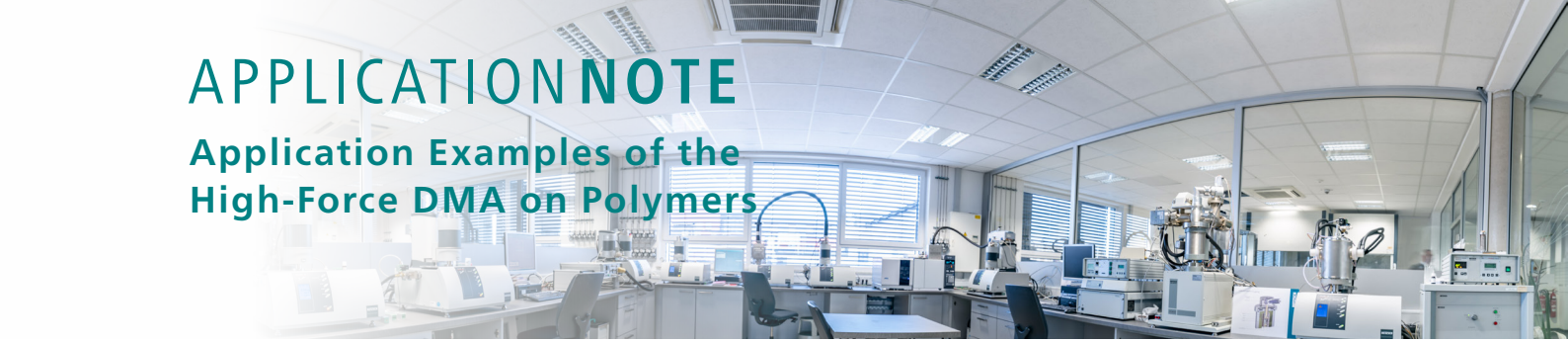

APPLICATION**NOTE**

Application Examples of the High-Force DMA on Polymers

Creep Behavior of PE-HD under Different Loads and Temperatures

Dr. Wiebold Wurpts

Introduction

When a metal is subjected to a force, it usually deforms immediately and then remains in the same shape even after a long period of time. If the load was not too high, the metal will also return elastically to its original state when the load is removed. When polymers are loaded with a force, they also deform immediately; after a longer period of time, however, it is often found that the body has deformed even further. This behavior is called creep. Basically, metals also creep, but with polymers, this behavior is much more pronounced and must be kept in consideration when describing mechanical behavior. For this reason, a quasi-static stress-strain diagram is often sufficient for metals; for polymers, however, the time-dependent deformation should also be taken into account.

Here, it is important to basically distinguish between creep and relaxation: In creep, a constant load acts on the body, which consequently deforms. In relaxation, deformation of a body remains constant, but over time, the required force is reduced. Relaxation is of great interest for certain applications, such as for seals; but for many components, it is rather the load that is constant and the time behavior of the deformation that is of interest.

In materials testing, the actual creep measurement is often combined with a recovery phase (creep recovery) in which the material can obtain its original shape again. This way, a distinction can be made between elastic and and irreversible creep. The irreversible deformation depends to a large extent on temperature and level of load. These relationships will be investigated in more detail in this publication.

Creep Recovery Measurements on PE-HD

The creep behavior of polymers is investigated here using the example of semi-crystalline high-density polyethylene (PE-HD). The samples with dimensions of 55 x 5 x 2 mm are tested with the help of the dynamic-mechanical high-load NETZSCH DMA GABO EPLEXOR® 500 N in tensile mode (figure 1).

With the EPLEXOR®, static forces up to 1500 N can be applied in the temperature range from -160°C to +500°C.

1 PE-HD in standard tension holder

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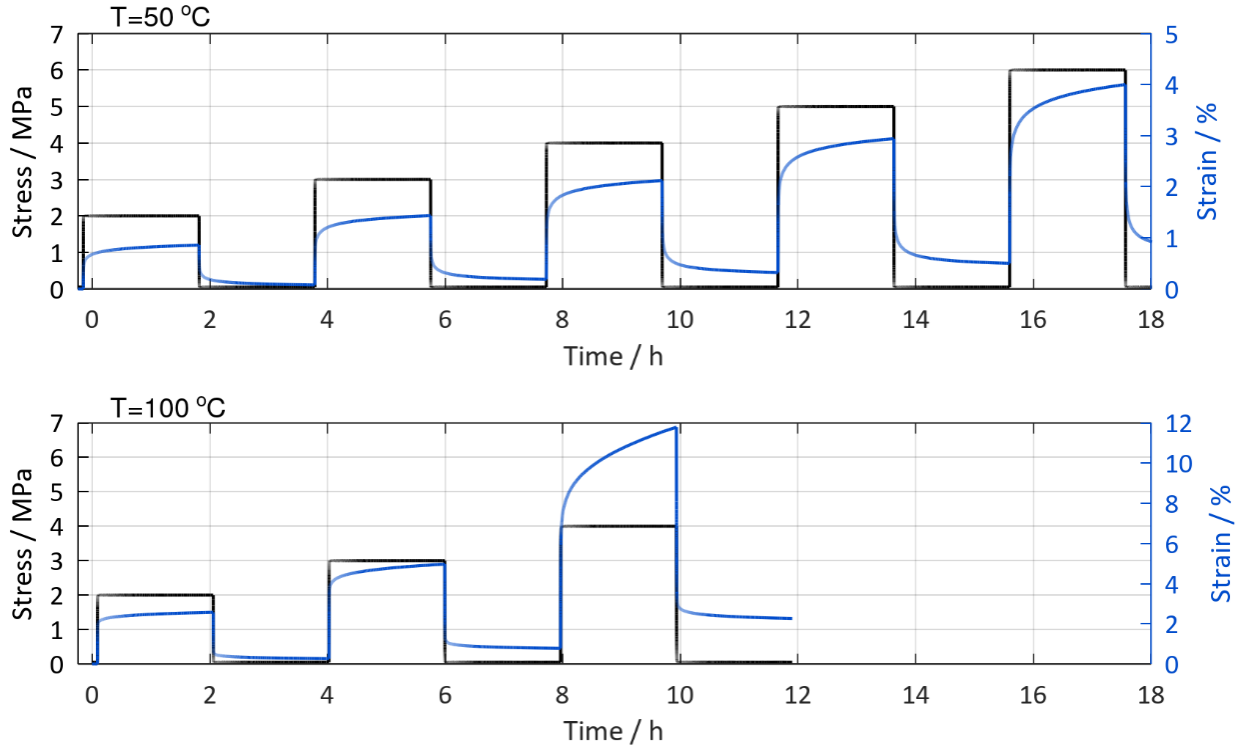
Depending on the application range, different tensile sample holders are available: With the standard tensile sample holder, up to 700 N can be applied, depending on the sample. For higher forces, a stronger version up to 1500 N is available.

Since the dependence of creep on force is to be investigated in particular, the individual measurements are compared under increasing loads. This way, different load levels can be investigated in a single measurement series without the need for reclamping.

With this procedure, however, the sample may in principle be deformed before the actual loading step. In order to prevent deviations from the reference geometry from becoming too extensive, no further load increase is carried out here once a strain of 10% is reached. The measurements are carried out each at a defined sample temperature. At 50°C, five load steps are carried out from 2 to 6 MPa, with 2 waiting hours in order to guarantee that a stable condition can be established in each case.

At an elevated temperature of 100°C, the load is only increased to 4 MPa as the maximum strain is reached.

As shown in figure 2, creep typically consists of three phases for each loading step. First, the sample is stretched relatively abruptly, followed by visco-elastic creep. These two processes are typically reversible. Thereafter, the sample rather transforms into a viscous flow (constant strain rate) and it can clearly be seen that this flow is more pronounced at higher stresses and temperatures. Since this viscous flow is not reversible, a remanent deformation remains even after the subsequent unloading phase. This visco-plastic behavior occurs with increased intensity at higher temperatures and stresses.



2 Creep-recovery experiments under different loads and temperatures

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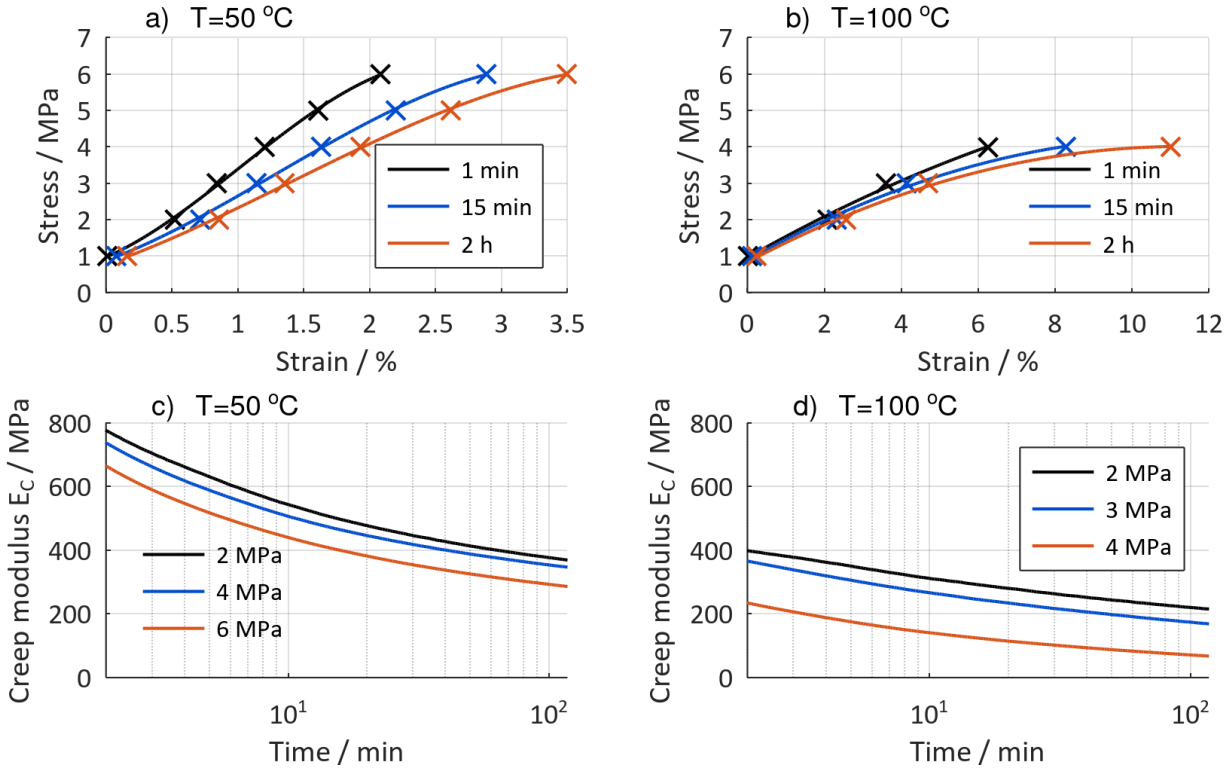
In DIN ISO 899 [4], the tensile creep test for determination of the creep behavior is described. Although is does not specifically address the creep-recovery experiments employed here, typical evaluations are presented that can also be used for the respective creep phases. Figures 3 a) and b) thus show the isochronous stress-strain diagrams associated with the above measurements. The strain is noted for each stress after a fixed time and entered in the diagram. Since different loads are applied to a sample in this test series, the strain refers in each case to the state directly before the load step. This presentation is of particular interest for the design of components because the resulting strain can be read completely analogous to the classical stress-strain diagram for a given load. Typically, the strains are also of interest after much longer time periods than those recorded here. As seen above, mainly the viscous behavior dominates for longer time periods, which will later be discussed in more detail.

As another typical presentation, DIN ISO 899 describes the time-dependent creep modulus (figures 3 c and d). The reciprocal value of the modulus, i.e., the creep compliance, is often used instead, but here, the creep modulus is shown in accordance with the standard. Presentation of the creep modulus is particularly suited for investigation of the non-linearity of the material. It becomes clear that the higher stresses generally lead to a lower creep modulus and thus, to higher compliance.



3 a) and b): Corresponding isochronous stress-strain diagrams
c) and d): Creep moduli of PE-HD at different temperatures and stresses

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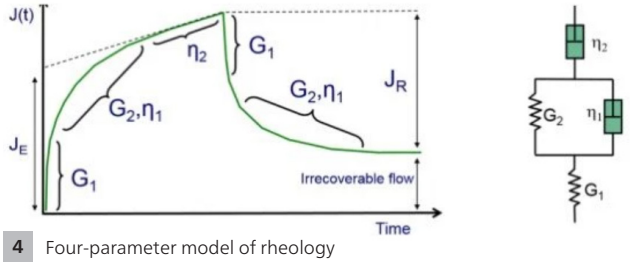
Description of Creep Rates According to Eyring

Polymer creep is often described by the four-parameter rheological model (figure 4). The model consists of a spring and damping element (Maxwell element) connected in series. The spring can be used to illustrate instantaneous strain jump and the damper to model the viscous flow. The visco-elastic behavior is described by the parallel spring-damping element. Thus, for each previously performed creep-recovery experiment, a corresponding model can be identified.

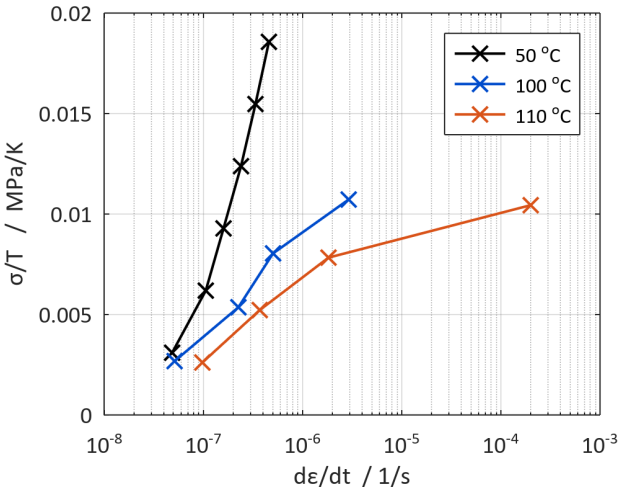


4 Four-parameter model of rheology

As shown above, the visco-plastic component relevant for long-term creep is mainly caused by viscous flow. The dependence of viscous flow on temperature and stress can be derived, model-based, from the probabilities that a molecule will overcome a certain obstacle. Details can be found, for example, in [2]. Here, it is stated as a result that, according to this model, the relationship between stress and temperature depends linearly on the logarithm of the strain rate. Accordingly, an increase in stress leads to an exponential increase in strain rate.

Shown in figure 5 are the strain rates determined for the respective stresses. Along with the measurements already presented above, the experiment was additionally carried out at 110°C. At 50°C, the behavior between the strain rate and stress is very well described by the model, i.e., there is a largely linear relationship between the stress and logarithmic strain rate. At higher temperatures and stresses, further molecular processes are possible, then leading to a bend in the logarithmic strain rate.

In the Eyring plot [1], a separate line is recorded for each temperature. In this respect, the plot allows for presenting the extrapolation of the strain rate for other stresses. It should be noted, however, that there are also more advanced approaches for including an additional time-temperature superposition; see, for example, [3].



5 Eyring plot for the creep of PE-HD

Conclusion

The creep behavior is heavily dependent on temperature and load level. While elastic creep components can even be measured at smaller forces, higher forces and stresses occur in many applications. The DMA GABO EPLEXOR® allows for the characterization of load-dependent plastic creep in many cases relevant in practice. It is thereby shown that the long-term creep behavior is mainly determined by the viscous flow of the polymer. Exactly this dependence of the strain rate on the acting stress can clearly be illustrated in an Eyring plot.

Literature

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