



# Rate mechanism and dislocation generation in high density polyethylene and other semicrystalline polymers



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## ABSTRACT

The aim of the paper was to use recent experimental dislocation data for the development and testing of a dislocation mediated strength model based on that introduced by Scogna and Register for semicrystalline polymers. It is shown that the model can successfully describe measured and evaluated data of high density polyethylene (PE-HD) on the flow stress as function of strain rate and temperature. Similar coincidences of model fit with experimental yield stress data from literature for polypropylene and polyethylene-ethylenemethacrylic acid co-polymers (E/MAA) were found which suggests prevailing of dislocation mediated plasticity mechanisms also in these materials. It also turned out that two parameters of the model – namely the dislocation density and the lamella size – are not determined by the molecular chemistry but by the conditions of processing and/or sample preparation. Therefore the model allows for a reliable estimation of the dependence of the yield stress on these conditions.

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## 1. Introduction

According to experimental observations the yielding of semicrystalline polymers is controlled by a thermally activated process not only in the amorphous but also in the crystalline phase. The process in the crystalline phase has been matter of discussion in the last decades as two conflicting concepts exist: The first approach by Flory and Yoon [1] argues that due to the high degree of interconnection between the crystalline lamellae, the plastic deformation of semicrystalline polymers consists of a process of localized melting and recrystallization under adiabatic conditions. The second approach is based on the concept of crystal plasticity for metallic materials – i.e. that plastic deformation of polymer crystals is governed by dislocation generation and motion. Grown-in dislocations created, for instance, due to chain ends and folds [2], are not mobile and a mechanism involving thermally activated dislocation generation is more likely [3]. Young [4] suggested a model for thermally activated generation of monolithic screw dislocations which has been extended later by Argon et al. [5] to account for rate dependence.

Recent investigations using multiple X-ray line profile analysis were able to show not only the presence but also the deformation

induced generation of dislocations in  $\alpha$ -phase isotactic polypropylene (PP) [6–8]. Also the character and specific population of dislocations in terms of slip systems (chain and transverse/screw or edge) could be derived by evaluation of their characteristic strain field [9]. These observations confirm the assumption of Young that screw dislocations prevail.

These new experimental observations suggest to revisit the subject of dislocation mediated plasticity in semicrystalline polymers by testing the related models in terms of measured dislocation data, and by applying these models to develop new semicrystalline polymers with enhanced strength.

In order to relate the temperature and rate dependence of the yield stress to the dislocation generation and the mobile dislocation density, a two phase model is proposed combining an Eyring process [10–15] for the amorphous phase with a rate dependent formulation of Young's model for dislocation generation for the crystalline phase. Data from uniaxial compression at different strain rates and temperatures as well as strain rate jump tests are used to test the model predictions.

## 2. Experimental

The samples were produced from a compression molded plate of polyethylene *ELTEX TUB 125 N2025 Orange* by *INNOVENE*. Bars with the dimensions 10·10·200 mm<sup>3</sup> were cut from the plate. In

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order to further remove preferential orientations arising from the production process and to ensure a uniform size distribution of the spherulites the samples were heat treated by melting at 200 °C for 2 h and by subsequent recrystallization at 105 °C for 15 min. Cylindrical samples (diameter: 6 mm, height: 10 mm) were machined from the recrystallized bars.

Uniaxial compression tests with and without strain rate jumps were carried out at seven different temperatures between –40 °C and 100 °C by a *Shimadzu AG50* deformation machine equipped with a *Shimadzu TCL N220* thermostatic chamber applying strain rates of  $4 \cdot 10^{-2} \text{ s}^{-1}$ ,  $4 \cdot 10^{-3} \text{ s}^{-1}$  and  $4 \cdot 10^{-4} \text{ s}^{-1}$ . Geometric stress-strain corrections of the compressed samples were achieved using a video extensometer *Messphysik 45NG*. Strain rate jumps were performed at true strains of 0.25 and 0.45 in the plastic regime.

### 3. Experimental results

The curves resulting from deformation at three strain rates and different deformation temperatures are shown in Fig. 1. The flow stresses (measured at true strains  $\varepsilon = 0.25$  and  $\varepsilon = 0.45$ ) shown in Fig. 2 decrease considerably as a function of the deformation temperature. Thermally activated processes approach a saturation stress as the process is fully thermally activated. In the present experiment this is not observed as the sample melts before saturation occurs. A considerable change is found in the temperature dependence of the strain rate sensitivity [16]

$$m = \left( \frac{\partial \log \sigma_f}{\partial \log \dot{\varepsilon}} \right)_{p,T,\varepsilon}, \quad (1)$$

with  $\sigma_f$  the flow stress,  $\dot{\varepsilon}$  the true-strain rate,  $p$  the pressure,  $T$  the temperature and  $\varepsilon$  the true strain. The temperature dependence of  $m$  is depicted in Fig. 3. The strain rate sensitivity is increasing as a function of the temperature.

The activation volume as function of the deformation temperature

$$\Delta V^* = - \left( \frac{\partial \Delta G^*}{\partial \sigma_f} \right)_{p,T,\varepsilon} = k \cdot T \cdot \left( \frac{\partial \log \dot{\varepsilon}}{\partial \sigma_f} \right)_{p,T,\varepsilon}, \quad (2)$$

is shown in Fig. 4. Here  $\Delta G^*$  is the Gibbs free energy (free enthalpy) of the activated process and  $k$  the Boltzmann constant. At lower

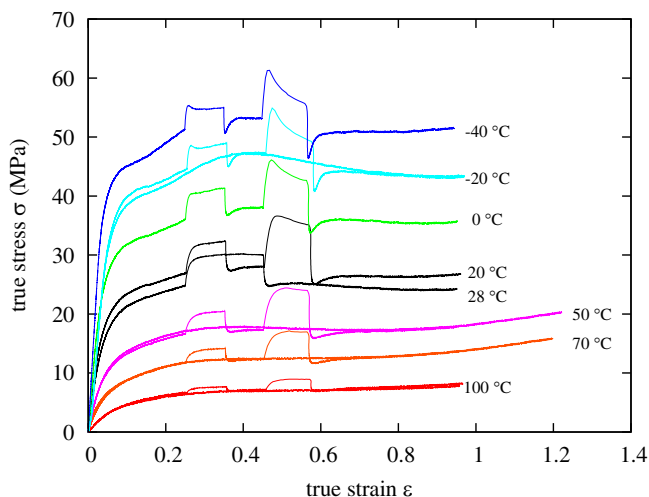


Fig. 1. Uniaxial true-stress  $\sigma$  true-strain  $\varepsilon$  curves of PE-HD with strain rate changes. For some deformation temperatures, tests without strain rate jumps were also performed.

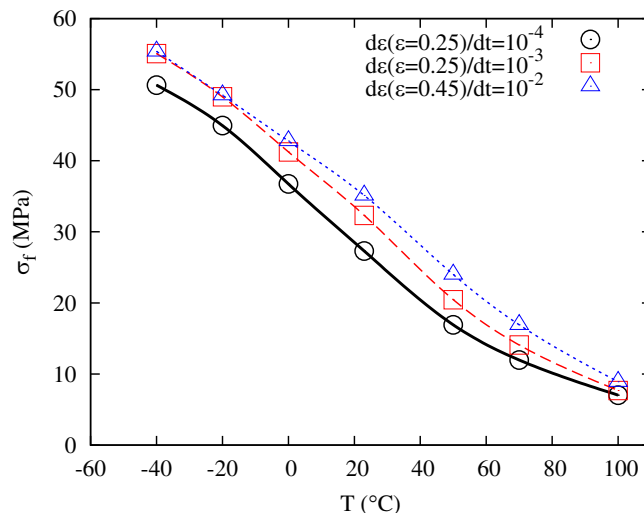


Fig. 2. Flow stress  $\sigma_f$  measured at true strains  $\varepsilon = 0.25$  and  $\varepsilon = 0.45$  as a function of the deformation temperature.

temperatures the activation volume is determined to be about  $5 \text{ nm}^3$ . This corresponds to earlier findings by Kazmierczak et al. [17]. At about 60 °C the activation volume starts to increase while the strain rate sensitivity is decreasing again.

### 4. Modeling

In order to model the strain rate sensitivity of semicrystalline polymers, at first the strain rate sensitivity of amorphous polymers has to be considered. Here several approaches have been adopted. Based on the general theory of thermal activation, Eyring [10] developed the concept of transition states i.e. the jump of segments of polymers from one equilibrium position to another. Later the generalized Ree–Eyring concept [18,19] for distributions of relaxation times was developed. Segmental motion was modeled by concepts based on conformational changes [20], kinks [21] and even one based on a dislocation analogy [22]. The Cooperative Model of Fotheringham and Cherry [11–13] was developed to take into account the meaning of the activation volume, by describing

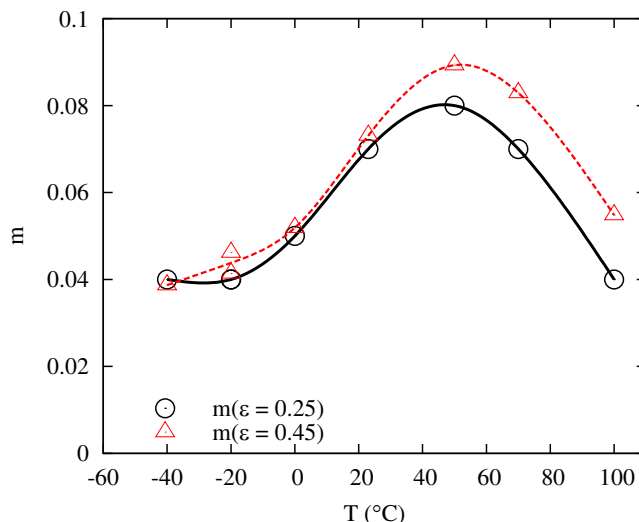


Fig. 3. Strain rate sensitivity  $m$  as a function of the deformation temperature  $T$  as determined for true-strain values  $\varepsilon = 0.25$  and  $\varepsilon = 0.45$ .

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