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Relaxation and physicomechanical characteristics of polyethylenes with different molecular weights

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Objectives. *Determination of the influence of molecular weight on the modulus of elasticity, yield, strength, and retardation processes in polyethylene.*
Methods. *We used vane samples (thickness: 4 mm; length: 100 mm; width: 10 mm) made by injection molding at* $p = 60$ *MPa,* $T = 210$ *°C,* $\tau = 15$ *s from the following polyethylenes: HDPE 277-73 (Stavrolen, Lukoil, Russia); BorSafe HE3490-IM (Borealis, Austria; black); CRP 100 Hostalen (Basell Polyolefins, Netherlands; black); Stavrolen PE4PP-25B (Stavrolen, Lukoil, Russia; black). The samples were in accordance with the defined standards for the AL 7000 LA-5 tensile testing machine. The study of relaxation characteristics was carried out in two modes: relaxation and retardation.*
Results. *We obtained stress-strain diagrams at various temperatures under isothermal conditions (*T *= const) and determined the influence of polyethylene molecular weights on the modulus of elasticity, yield, and strength of polyethylenes. We have shown that under isothermal conditions, when the stress equals the yield strength, the removal of the external action results in a two-stage response. The first stage is the stress relaxation. The second stage characterizes the elastic features of the studied materials under the external action* ε *= const.*
Conclusions. *We have established that temperature increase affects the physicomechanical characteristics of polyethylenes differently, depending on their molecular weights. The experiments have shown that when the stress exceeds the yield strength, at constant deformation, there is a complex response of the polyethylenes to external action. This response is characterized by two stress stages throughout the course of time. The first stage is characterized by asymptotic decrease in stress down to a constant value; the second stage is characterized by constant stress throughout the course of time. We have determined relaxation times for the relaxation stage (stage I) and calculated activation energy. We have also established that the activation energy depends on molecular weights of the polyethylenes. It has been shown that an increase in polyethylene molecular weight leads to a decrease in relaxation time and activation energy.*

Keywords: *relaxation, inelasticity, internal friction, modulus of elasticity, yield strength, polyethylene.*

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Релаксационные и физико-механические характеристики полиэтиленов с различной молекулярной массой

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Цель. *Определение влияния молекулярной массы полиэтиленов на модуль упругости, текучесть, прочность и процессы ретардации.*

Методы. *В качестве образцов были взяты лопатки (толщиной 4 мм, длиной 100 мм, шириной 10 мм), полученные методом литья под давлением р = 60 МПа при Т = 210 °С, τ = 15 с, из полиэтилена следующих марок: ПЭВП 277-73 (Ставролен, Лукойл, Россия); BorSafe HE3490-IM (Borealis, Австрия; черный); CRP 100 Hostalen (Basell Polyolefins, Нидерланды; черный); Stavrolen PE4PP-25B (Ставролен, Лукойл, Россия; черный). Использованные образцы соответствовали стандартно-определенным образцам для исследования на разрывной машине AL 7000 LA-5. Исследование релаксационных характеристик проводилось в двух режимах: релаксационном и ретардационном.*

Результаты. *В результате проведения экспериментов были получены диаграммы «напряжение–деформация» при различных температурах в изотермических режимах (Т = const) и определено влияние молекулярной массы на модуль упругости, текучесть и прочность полиэтиленов. Показано, что при напряжениях, равных пределу текучести, в изотермических условиях при снятии внешнего деформирующего воздействия наблюдаются две стадии отклика системы полиэтилена на это воздействие. Первый отклик – процесс релаксации напряжения и второй отклик – область, характеризующая упругие характеристики исследуемого материала при внешнем воздействии ε = const.*

Заключение. *Установлено, что повышение температуры неодинаково отражается на физико-механических характеристиках полиэтиленов различной молекулярной массы. Эксперименты показали, что при напряжениях выше предела текучести при постоянстве деформации наблюдается сложная реакция исследуемых полиэтиленов на внешнее воздействие. Эта реакция характеризуется двумя областями зависимости напряжений, возникших при деформации, от времени. Первая область характеризуется асимптотическим снижением напряжения до постоянного значения, а вторая область – постоянством напряжения во времени. Рассчитаны времена релаксации для релаксационной области (участок I), а также рассчитана величина энергии активации. Показано, что увеличение молекулярной массы полиэтиленов ведет к снижению времени релаксации и уменьшению энергии активации.*

Ключевые слова: *релаксация, неупругость, внутреннее трение, модуль упругости, предел текучести, полиэтилен.*

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Introduction

Amorphous-crystalline polyethylene (PE), despite the simple structure of its polymer chain, is characterized by multiplet relaxation behavior observed in solid state, from low temperatures and up to the melting temperature T_{melt} [1–3]. This can mostly be explained by the complex structure of crystalline polymers and their molecular motion, and this is why it is also challenging to establish all possible relaxation transitions in PE and determine their nature. Consequently, there is still no clear understanding of relaxation transitions in PE and how they are influenced by the molecular weight of the polymer. Research on local dissipative losses by a dynamics method [4] shows that the PE structure consists of four structural-kinetic subsystems that respond to external action quasi-independently; this happens when such deformation destabilizes mechanical and thermodynamic equilibria of all elements of these structural-kinetic subsystems. Theoretical analysis of these physicomechanical parameters allows us to determine the degree of relaxation microheterogeneity for elements of various structural-kinetic subsystems.

The aim of this work was to determine the influence of molecular weight on the modulus of elasticity, yield, strength, and retardation processes in polyethylene.

Materials and Methods

We used vane samples (thickness: 4 mm; length: 100 mm; width: 10 mm) made by injection molding at p = 60 MPa, T = 210 °C, τ = 15 s from the following polyethylenes: HDPE 277-73 (*Stavrolen, Lukoil,* Russia); BorSafe HE3490-IM (*Borealis*, Austria; black); CRP 100 Hostalen (*Basell Polyolefins*, Netherlands; black); Stavrolen PE4PP-25B (*Stavrolen, Lukoil,* Russia; black). Our samples were in accordance with the defined standards for the AL 7000 LA-5 tensile testing machine. The stretching was performed at the strain rate of $V_{\Delta l}$ = 100 mm/min. According to the preparation method, the sprue was placed toward the mold in such a way that it was co-oriented with the polymer molecules. After shrinkage, all PE samples with different molecular weights kept their shapes. We did not evaluate the influence of shrinkage on the macromolecule orientation.

The main physicochemical and physicomechanical characteristics of polyethylenes used in this study are shown in Table 1.

We performed the evaluation of relaxation characteristics in two modes:

1. The relaxation mode:

external action

$$\varepsilon(t) = \varepsilon_0 h(t) \begin{cases} 0 \text{ when } t < t_0 \\ \varepsilon_0 \text{ when } t \geq t_0 \end{cases} \tag{1}$$

response

$$\sigma(t) = \begin{cases} 0 \text{ when } t < t_0 \\ \sigma_0 = E\varepsilon_0 \text{ when } t \geq t_0 \text{ and } \sigma_0 \leq \sigma_{pl} \\ \sigma_0 \Psi(t) \text{ when } t \geq t_0 \text{ and } \sigma_{pl} \leq \sigma_0 \leq \sigma_{el} \end{cases}, \tag{2}$$

where σ is a stress, MPa; ε is a strain, %; *E* is a modulus of elasticity, MPa; *t* is a time, s; t_0 is a moment of the external action removal; σ_{pl} is the proportionality limit (the limit of linear elasticity), MPa; σ_{el} is the elastic limit, MPa.

Table 1. Main characteristics of polyethylenes used in the study

№ п/п	PE type	HDPE 277-73	BorSafe HE3490-IM	CRP 100 Hostalen	Stavrolen PE4PP-25B
1	Molecular weight (MW), g/mol × 10^4	24	67	74	75
2	Molecular weight distribution (MWD)	Monomodal	Bimodal		Monomodal
3	Melt flow index, g per 10 min at 190 °C and 21.6 kgf at 190 °C and 5.0 kgf at 190 °C and 2.16 kgf	17–25 – 5–7	6.00 0.57 0.40	5.50 0.52 0.38	12–16 0.51 –
4	Melt stretching index, s	–	120	215	400
5	Density at 23 °C, g/cm^3	0.957	0.962	0.960	0.952
6	Tensile yield strength, not below, MPa	25.5	24	–	16.7
7	Soot content, %	–	2	2	2
8	Melting temperature, °C	125–135	125–135	125–135	125–135
9	Type of soot distribution	–	I-II	I-II	I-II
10	ΔH, J/g	157.1	142.6	132.4	107.8
11	Degree of crystallinity, α, %	53.6	48.7	45.2	36.8

2. The retardation mode:
external action

$$\sigma(t) = \sigma_0 h(t) \rightarrow \begin{cases} 0 \text{ when } t < t_0 \\ \sigma_0 \text{ when } t \geq t_0 \end{cases} \quad (3)$$

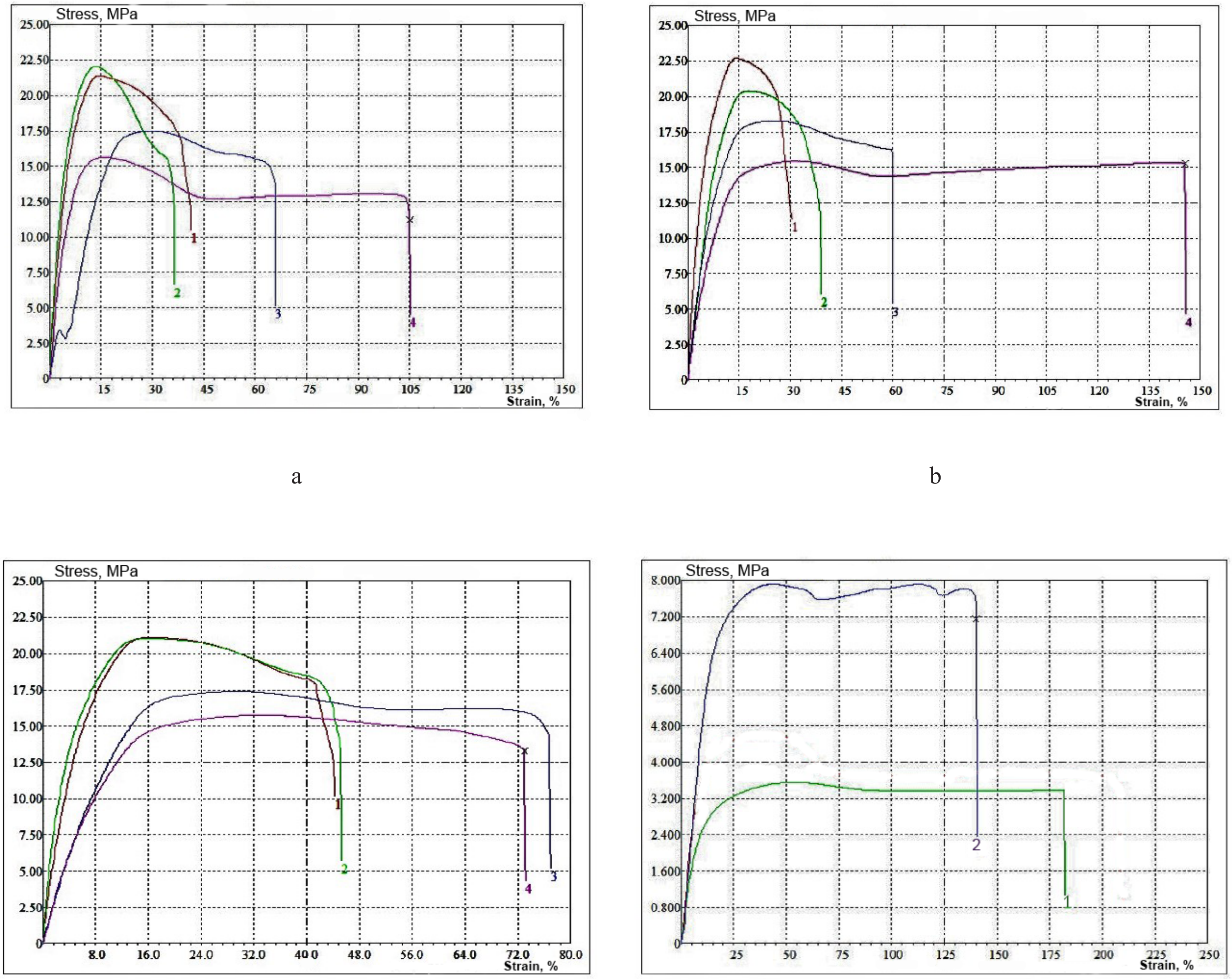
response

$$\varepsilon(t) = \begin{cases} 0 \text{ when } t < t_0 \\ \varepsilon_0 = \sigma_0 / E \text{ when } t \geq t_0 \text{ and } \sigma_0 \leq \sigma_{pl} \\ \varepsilon_0 I(t) \text{ when } t > t_0 \text{ and } \sigma_{pl} < \sigma_0 \leq \sigma_{el} \end{cases} . \quad (4)$$

This allowed us to evaluate the dependency of the modulus of elasticity and the yield strength on the molecular weight, using stress-strain diagrams. We were also able to determine creep modes and elastic aftereffect (retardation) modes. Knowing these parameters (and how they depend on molecular weights) allows us to evaluate the functions that describe the stage of inelastic response to an external action (equations 1–4), based on isothermal conditions of the hereditary theory of viscoelasticity.

Results and Discussion

We have obtained stress-strain diagrams at various temperatures under isothermal conditions (T = const) and these results are shown in Fig. 1.



a b

c d

Fig. 1. Stress–strain diagrams for BorSafe HE3490-IM (a), CRP 100 Hostalen (b), Stavrolen PE4PP-25B (c) polyethylenes at the following temperatures, °C:
1 – 21; *2* – 30; *3* – 40; *4* – 50; for HDPE 277-73 (curve 1) and Stavrolen PE4PP-25B (curve 2) polyethylenes at 100 °C (d).

We have determined the effect of molecular weight on the modulus of the elasticity, yield, and strength of polyethylene (Table 2).

Based on our experimental data, we have obtained temperature-dependency curves for the modulus of elasticity and the yield strength (Fig. 2), as well as curves showing the dependency of these parameters on the molecular weight (Fig. 3).

The experimental data show that temperature increase affects the physicomechanical characteristics of polyethylenes differently, depending on their molecular weights. In the case of HDPE 277-73, the PE with the lowest molecular weight (MW = 24×10^4 g/mol) (Table 1), we observe a gradual decrease in the modulus of elasticity (Fig. 2a) and the yield strength (Fig. 2b) upon temperature increase. In the case of PE with a higher molecular

Table 2. Physicomechanical characteristics of polyethylenes with different molecular weights (at $V_{\Delta l}$ = 100 mm/min)

Temperature of the experiment, °C	F_{max}*, kgf	Tensile strength at F_{max}, MPa	Strain at F_{max}, %	Yield strength, MPa	Strain of yield strength, %	Modulus of elasticity, MPa	Ultimate tensile strength, MPa	Strain at breakage point, %
HDPE 277-73								
21	88.65	21.71	13.18	21.71	12.35	891.74	12.4	–
30	84.50	20.67	13.83	20.67	12.73	837.26	12.73	–
40	74.36	18.21	14.42	18.21	13.60	742.89	13.6	–
50	59.85	14.67	16.14	14.67	15.47	563.77	15.47	–
BorSafe HE3490-IM								
21	87.25	21.39	14.65	21.38	14.01	731.37	10.99	41.44
30	89.83	22.02	13.62	22.02	13.15	714.99	12.43	36.47
40	71.35	17.49	30.00	17.48	28.37	169.92	13.78	65.89
50	63.86	15.66	15.97	15.66	15.75	231.77	11.18	105.2
CRP 100 Hostalen								
21	92.50	22.63	14.13	22.68	14.06	641.98	11.33	30.40
30	83.14	20.38	17.63	20.38	17.34	166.80	11.35	38.90
40	74.66	18.30	25.76	18.27	22.70	224.34	16.06	59.99
50	63.01	15.45	31.74	15.44	30.38	271.60	15.29	145.84
Stavrolen PE4PP-25B								
21	85.14	21.12	16.16	21.11	15.74	511.40	12.21	44.25
30	85.82	21.04	15.61	21.04	15.55	609.46	13.36	45.27
40	70.94	17.39	28.93	17.36	26.49	205.43	13.96	77.00
50	64.30	15.76	32.79	15.76	31.47	211.81	13.33	73.10
HDPE 277-73								
100	14.49	3.55	55.42	3.43	34.45	28.81	3.34	182.33
Stavrolen PE4PP-25B								
100	32.30	7.92	113.39	7.89	38.77	25.17	7.15	140.13

*F_{max} is the maximum stress during the tensile test.

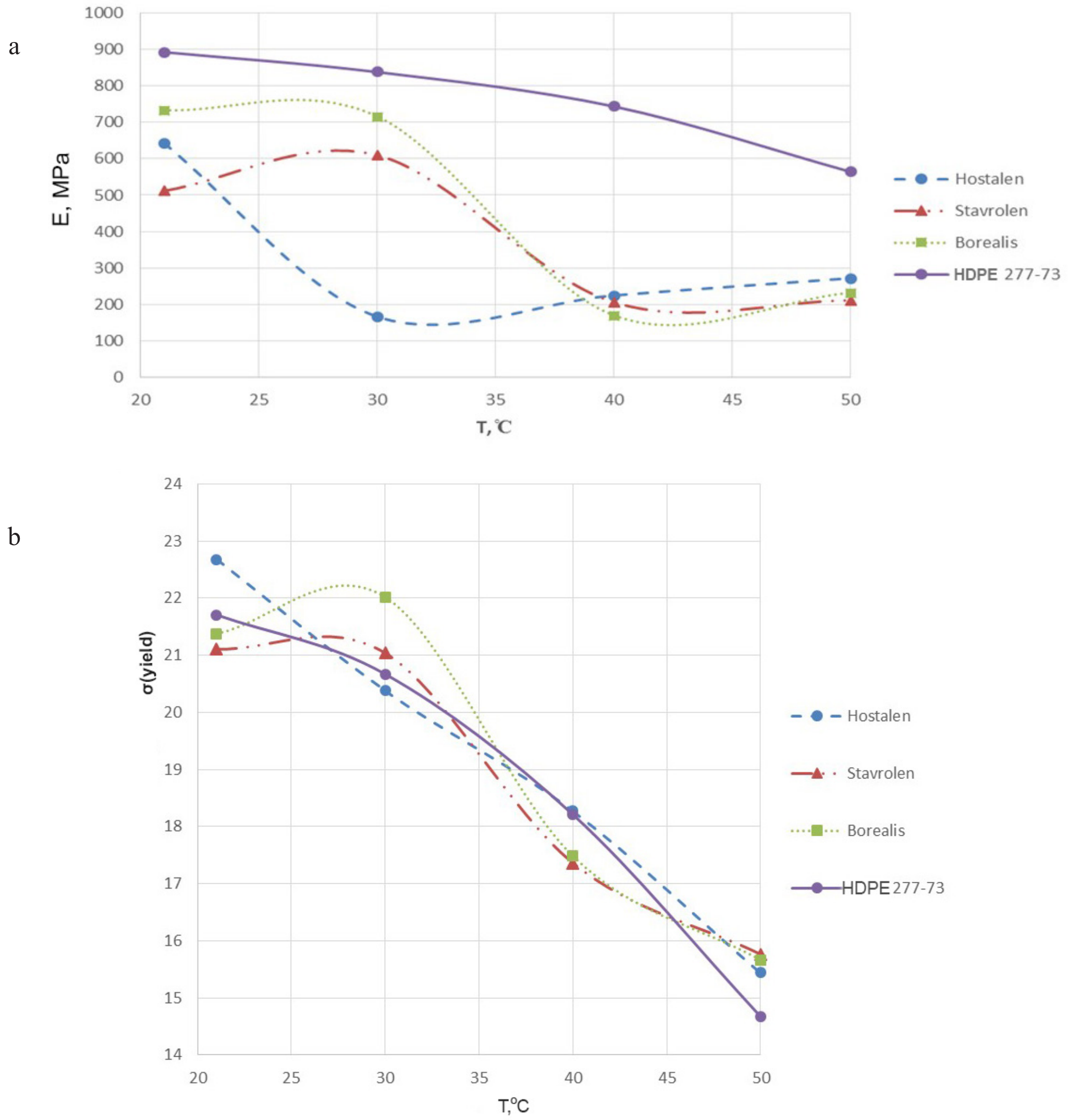


Fig. 2. Dependency of the modulus of elasticity (a) and the yield strength (b) for polyethylenes of different molecular weights on temperature.

weight, extreme changes in these parameters occur. For Stavrolen PE4PP-25B (MW = 75×10^4 g/mol) and BorSafe HE3490-IM (MW = 67×10^4 g/mol) polyethylenes (Table 1), we see that the parameters have a maximum at T = 30 °C, followed by a decrease and stabilization of the modulus of elasticity after 40 °C (Fig. 2a). Additionally, for the Hostalen polyethylene (MW = 74×10^4 g/mol) (Table 1), we observe an opposite tendency, where the modulus of elasticity decreases as the temperature increases to 30 °C, and afterwards it stabilizes, following a period of insignificant growth within the 40–50 °C range. At the same time, we observe abnormal behavior in the yield strength for Stavrolen PE4PP-25B and BorSafe HE3490-IM polyethylenes at T = 30 °C (Fig. 3b).

We have analyzed the inelasticity and stress relaxation of PEs using the same technique, at $\sigma_{el} < \sigma \approx \sigma_{yield}$ (σ_{yield} is the stress equaled the yield strength, MPa). A temperature of 100 ºC was chosen to speed up the experiment, because the increase in temperature leads to a decrease in the yield strength. Study of the dependence of relaxation times on molecular weight and temperature is a separate task and may be the object of future research. To perform these experiments, samples were subjected to a stress of $\sigma = \sigma_{yield}$ and strain of $\varepsilon = 42\%$ (Stavrolen PE4PP-25B) and $\varepsilon = 11\%$ (HDPE 277-73) (Fig. 4, stage I); after that, the external action was removed and the residual strain mode ε_{res} = const was observed (Fig. 4, stage II).

The strain that appears in the samples changes throughout the course of time, meaning that $\sigma = f(t)$. Area I in Fig. 5 – the upper part in the figure – describes the stress relaxation process, and area II describes the elastic nonrelaxing part of the stress in the PE.

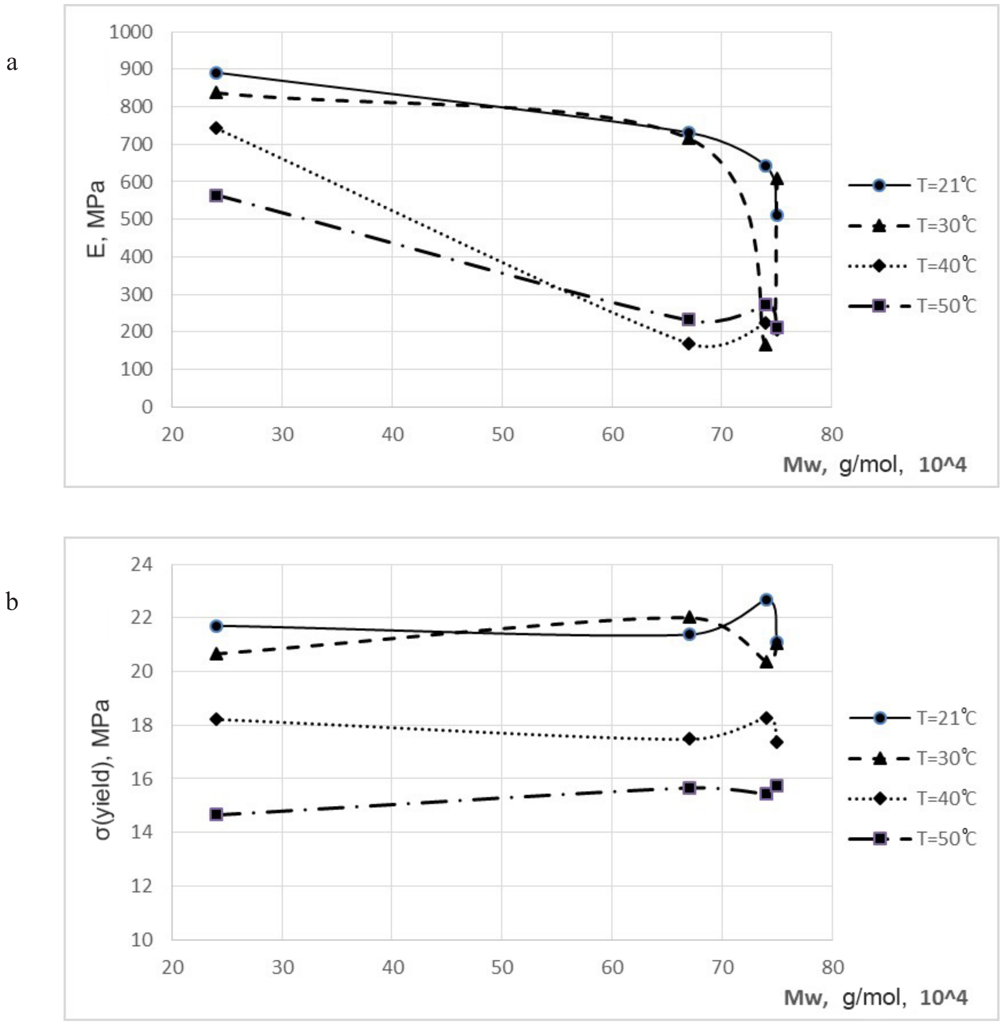


Fig. 3. Dependency of the modulus of elasticity (a) and the yield strength (b) on the molecular weight, for polyethylenes of different molecular weights; obtained under isothermal conditions.

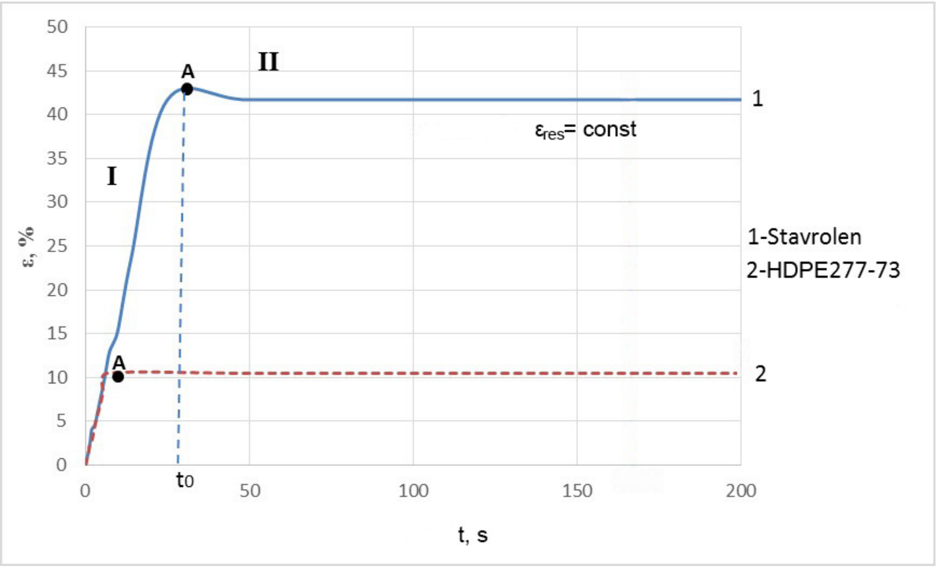


Fig. 4. Strain dependency on time, under isothermal conditions (T = 100 °C), for PEs with MW = 75×10^4 g/mol (curve 1) and MW = 24×10^4 g/mol (curve 2).

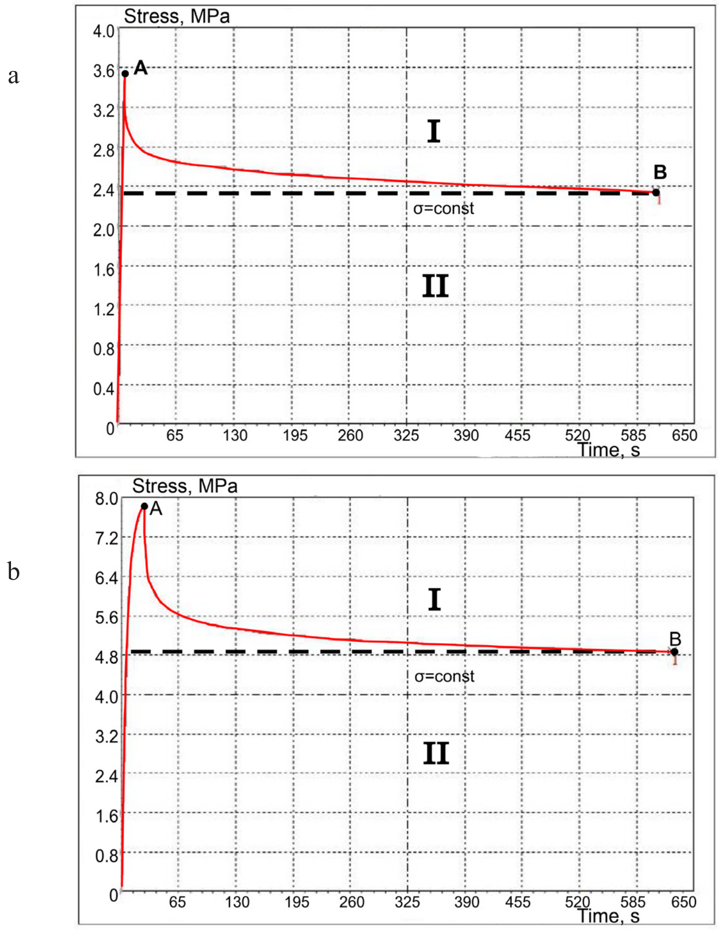


Fig. 5. Stress dependency on time for HDPE 277-73 (a) and Stavrolen PE4PP-25B (b).

Accordingly, for PEs with different molecular weights, the stress depends on time, which means that the stress does not change instantly with time, but rather stepwise, from point A to point B (Fig. 5a and 5b).

We have established that this stress asymptotically approaches a constant value σ = const (Fig. 5a and 5b). We have shown that when the stress equals the yield strength in isothermal conditions the removal of the external action results in a two-stage response. The first stage is the stress relaxation, and the second stage characterizes the elastic features of the studied materials under the external action ε = const.

In order to characterize the response of the PE during relaxation (the first stage response), we can use the Maxwell model:

$$\frac{d\sigma}{dt}+\frac{\sigma}{\tau}=E\frac{d\varepsilon}{dt}, \qquad (5)$$

where σ is a stress, MPa; ε is a strain, %; *E* is a modulus of elasticity, MPa; *t* is the time, s; and τ is the relaxation time, s.

The solution of this differential equation is the equation that connects the stress relaxation curve with relaxation time:

$$\sigma(t)=\sigma_0 \exp\left(-\frac{t}{\tau}\right), \qquad (6)$$

where σ(*t*) is the stress in the time moment *t*, MPa; and σ_0 is the stress in the time moment *t* = 0, MPa.

According to this equation, we can determine the relaxation function normalized to 1 (Fig. 6):

$$\varphi(t)=\frac{\sigma(t)}{\sigma_0}, \qquad (7)$$

where φ(*t*) is the relaxation function.

Based on the analysis of the time-dependent relaxation function φ(*t*), the relaxation time τ can be determined using the φ(*t*) curve (Fig. 6) according to the formula:

$$\tau=\frac{1}{e}, \qquad (8)$$

where *e* is the Euler number.

The τ values depend on the molecular weight of polyethylene. For HDPE 277-73 (MW = 24×10^4 g/mol) τ equals 10.1 s, and for Stavrolen PE4PP-25B (MW = 75×10^4 g/mol) τ equals 8.5 s.

The obtained values of relaxation time depend on the polyethylene molecular weight. In order to carefully characterize the function τ = *f*(MW), we have performed similar measurements for intermediate molecular weights.

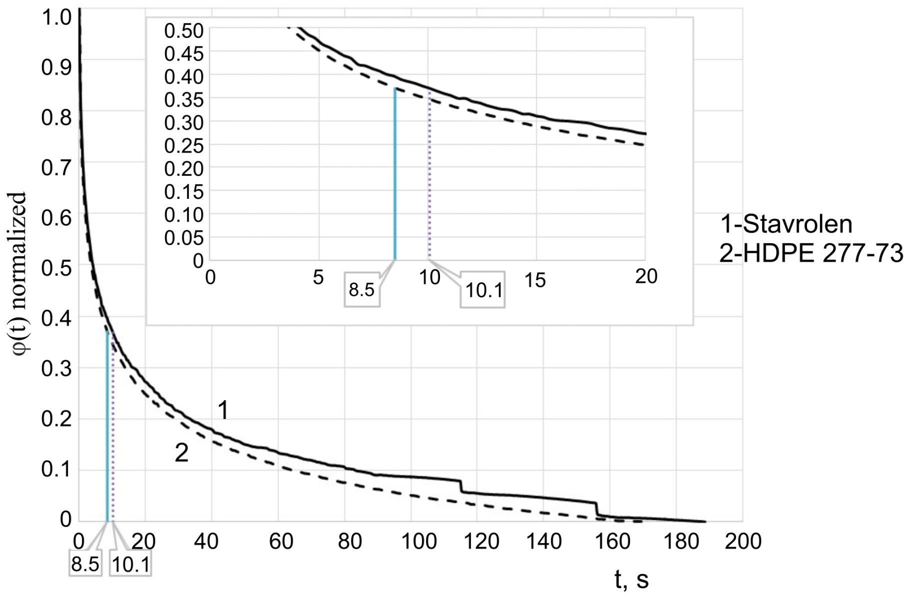


Fig. 6. Dependency of the normalized relaxation function on time, for PEs with MW = 75×10⁴ g/mol (curve 1) and MW = 24×10⁴ g/mol (curve 2).

Based on the Arrhenius equation for relaxation time

$$\tau_i = \tau_0 \exp\frac{U}{RT_i}, \tag{9}$$

and assuming that $\tau_0 = 1\times10^{-3}$ s for any polyethylene [5], we can calculate the activation energy of relaxation using the following formula:

$$U = RT_i \ln\frac{\tau_i}{\tau_0}, \tag{10}$$

where τ_i is the relaxation time, s, i.e. the time for transition of particles from one position to another with the overcoming of the potential barrier; *U* is the activation energy required to overcome the potential barrier, kJ/mol; τ_0 is the time of one attempt of a particle to pass through a barrier, s; *R* is the universal gas constant; and T_i is a temperature, K.

For example, for HDPE 277-73 (MW = 24×10⁴ g/mol) *U* equals 28.6 kJ/mol, and for Stavrolen PE4PP-25B (MW = 75×10⁴ g/mol) *U* equals 28.1 kJ/mol.

Our calculations show that the activation energy of relaxation does not depend greatly on the molecular weight. With the increase in the molecular weight of polyethylene, an insignificant decrease in activation energy occurs.

Conclusions

We have established that when the stress achieves $\sigma = \sigma_{pl}$ and ε_{res} = const, there are two response mechanisms in PE systems: mechanism I is the stage of stress relaxation (viscoelastic response); and mechanism II is the stage of elastic response that is characterized by constant stress σ = const. The value σ is a function of molecular weight. We have calculated relaxation times for the relaxation stage (stage I) and evaluated the activation energy, and established the connection *U* = *f*(MW). We have established that an increase in the molecular weight of polyethylenes leads to a decrease in the values of relaxation time and activation energy.

The experiments have demonstrated that when the stress exceeds the yield strength, the constant deformation leads to complex responses of the PE to such external action. This mode is characterized by two stress stages developed throughout time. The first stage is characterized by the asymptotical decrease in stress to a constant value, and the second stage is characterized by constant stress throughout time.

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