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## RELAXATION PARAMETERS OF POLYMERS

*Ключевые слова: релаксационные процессы, диссипация механической энергии, тангенс угла механических потерь, принцип температурно-временной суперпозиции.*

*Показано, что при исследовании таких релаксационных характеристик как тангенс угла механических потерь, диссипация механической энергии в результате внутреннего трения, измеренных при периодическом воздействии одноосного сжимающего напряжения на образце полимера (ПВД) в высокоэластическом состоянии, происходит процесс развития релаксации. В связи с этим необходимо учитывать температурно-временные условия эксперимента или вводить соответствующие поправки в полученные результаты.*

*Keywords: relaxation processes, dissipation mechanical energy, a tangent of mechanical losses, a principle of temperature-time superposition.*

*It is shown, that at research such relaxation characteristics as the tangent of mechanical losses, dissipation of mechanical energy as a result of the internal friction, measured at periodic action of monoaxial compressive stress on the sample of polymer (LDPE) in the high elasticity state, occurs development relaxation processes in the sample. In this connection it is necessary to take into account the temperature-time conditions of the experiment or introduce appropriate amendments to the results obtained.*

### Introduction

Any measurement of physical system is made by means of some device (in the more general case - the measuring environment). Thus there is an interaction of the device to measured system therefore the system condition to some extent changes depending on intensity of influence from the device. At measurements of classical system quite pertinently to suppose, that the measurement doesn't change a condition of measured system at all. If describe a condition of measured system and measurement procedure make so in details that features of influence of measuring system that a situation cardinally are shown changes. It appears that owing to the quantum nature of physical systems during the measurement the conditions of measured system are changing. These changes are so more than more information received by measurement. It is necessary to pay for the information. So in the theory of measurements the information increase corresponds to reduction of entropy [1]:

$$S = - \sum_i^n p_i \ln p_i,$$

where  $p_i$  - aprioristic probabilities of various conditions of system,  $n$  - quantity of conditions.

Thus, increasing accuracy of measurement, we necessarily increase also return influence of measuring procedure by a condition of measured system.

John Neumann for quantum system has proved a background and mathematically has strictly formulated a postulate of a reduction. According to this postulate at measurement of some observable size the system condition changes in such a manner that in a new condition the measured observable has already another certain value, and it has turned out as a result of measurement. Occurrence of this condition is called as a condition reduction of a system.

In the theory of measurements it is considered two types of measuring systems: passive and active [2]. In the passive measuring system there is a comparison of the defined size with the standard without any active

influence on the system which parameters define. The feature of active measuring system is influence on characterized system, and the response of system to this influence gives the information for calculation of demanded parameters.

As the active measuring system assumes a certain influence on characterized object in the course of this influence the object can undergo changes. Therefore for reception of the most exact value of the defined parameter in the theory of measurements perform the operation of coordination between measuring system and the measured object, consisting in reduction, and at the best data dissipation, influences of entrance influence on measured object.

At the measurements concerning difficult systems or objects, the measured size often depends on set of various circumstances. Usually the nature and quantitative characteristics of these dependences are unknown. The circumstances influencing on result of measurement don't remain constants during carrying out of measurement, there fore it's impossible to correct this or that error of measurement. It means that measurement isn't selective, and the result of measurement comprises also other factors.

In the greatest measure these principles are important for sizes which assume measurements in which basis the difficult physical and mathematical models demanding a certain sort of updating according to conditions of measurements lie. In the mechanic of polymers such sizes are the parameters characterizing relaxation properties of materials. These sizes and dependences corresponding to them give the chance to judge structure of polymers, to find temperatures of structural transitions and service conditions of corresponding materials [3,4].

### Theoretical part

One of widely used methods in research elastic and relaxation properties of polymers in the block at periodic sinusoidal loadings is the method of

Aleksandrova-Lazurkina [6]. Unlike resonant this method is applied for high elasticity deformations of polymers in the field of the frequencies lying considerably below own frequency of the sample – far from resonant area. In this case, the phase relations, phase lag of deformation from stress-relaxation time is determined only by or through the appropriate range of relaxation time and elastic material. In this method phase parities don't depend on the form, the size and density of the sample that allows to find relaxation time of a material from measurements.

The method is based idea of rubbery (*high elasticity*) deformation as a reflection of the deformation of flexible macromolecules, and the appearance of the elastic forces of deformation and shape recovery after unloading - the result of thermal motion of parts of macromolecules. However, all the patterns that underlie the method, refer to the equilibrium states of the body under load. The study of temporal patterns of rubbery deformation in a regime of constant stress or strain, as well as in periodic loads confirmed the significant role of the kinetics of rubbery deformation relaxation phenomena in the behavior of polymeric materials under mechanical stress, and in the process of vitrification of polymers [5,7].

Depending on the interim regime changes impact the behavior of the material. At a constant temperature with increasing speed or increasing the frequency of impacts observed so-called effect of "hardening" of the material [8].

Total deformation of the polymer is composed of an elastic, rubbery (high elastic) flow and deformation. When considering the polymer in the rubberlike (high elasticity) state accepts that the macroscopic viscosity of the material is great and flow absent.

To obtain the dependence of rubbery component of the strain on the applied stress using the simplest model for these conditions[9]. In this case, such a model is a three-element model representing a model of Kelvin (parallel connected spring and damper), connected in series with a spring. The equation describing the relationship between stress and strain of this model is as follows:

$$\frac{d\sigma}{dt} + \frac{E_0 + E_1}{\eta} \sigma = E_0 \frac{d\varepsilon}{dt} + \frac{E_0 E_1}{\eta} \varepsilon, \quad (1)$$

where  $\sigma$  -stress acting on the system being studied;  $\varepsilon$ -deformation occurring in the system under the applied stress,  $E_0$ - the module of elasticity;  $E_1$ - high elasticity module;  $\eta$  – micro viscosity.

Then the deformation of the polymer is made up of elastic strain  $\varepsilon_0 = \sigma/E_0$  and high elasticity  $\varepsilon_1$  parts. Rewriting equation (1) relative rates of change of strain and isolating highly elastic component of deformation, we can received :

$$\frac{d\varepsilon_1}{dt} + \frac{E_1}{\eta} \varepsilon_1 = \frac{\sigma}{\eta} \quad (2)$$

If the stress varies with time harmonically with frequency  $\omega$ :

$$\sigma = \sigma_0 \cos \omega t, \quad (3)$$

full deformation is described by the equation:

$$\varepsilon = C e^{-\frac{t}{\tau}} + \sigma_0 \left\{ \left( \frac{1}{E_0} + \frac{1}{E_1} \frac{1}{1 + \omega^2 \tau^2} \right) \cos \omega t + \frac{1}{E_0} \frac{\omega \tau}{1 + \omega^2 \tau^2} \sin \omega t \right\}, \quad (4)$$

where the parameter  $\tau = \eta / E_1$  is called as *relaxation time*. In some works [10,11] this parameter is named delay time, and relaxation time is named the parameter, which proportional to it:

$$\tau_1 = \tau \frac{E_1}{E_1 + E_0} \quad (5)$$

The first exponential member of the equation (4) contains constant C, witch depending on initial conditions, and defines an unsteady part of deformation fading in due course. Therefore, if from the beginning of carrying out of measurement has passed enough time  $t \gg \tau$  (the transients which have arisen at the moment of a start of motion, have already faded and takes place the established conditions) it is possible to neglect this member and consider only that part of expression (4), which is concluded in braces. This part describes the stationary oscillations, which are studied on experience. They consist of oscillations in a phase with the pressure, described by expression in parentheses and consisting of elastic and *high elastic* components, and the oscillations, which are lagging behind pressure on a phase on  $\pi/2$ . As these two harmonious oscillations are directed along one axis (a vector of their speeds are collinear) the amplitude of deformation is expressed by the equation:

$$\varepsilon_0 = \sigma_0 \sqrt{\left( \frac{1}{E_0} + \frac{1}{E_1} \frac{1}{1 + \omega^2 \tau^2} \right)^2 + \frac{1}{E_1^2} \frac{\omega^2 \tau^2}{(1 + \omega^2 \tau^2)^2}} \quad (6)$$

Using condition  $E_0 \gg E_1$ : as the high elasticity module for polymeric materials on some orders less than the module of elasticity, it is possible to receive dependence of deformation on pressure and frequency of loading ( $\omega$ ):

$$\varepsilon_0 = \frac{\sigma_0}{E_1} \frac{1}{\sqrt{1 + \omega^2 \tau^2}} \quad (7)$$

The received expression can be transformed as

$$\frac{\varepsilon_0}{\sigma_0} = \frac{1}{E_1 \sqrt{1 + \omega^2 \tau^2}} \quad (8)$$

Parameter  $\varepsilon_0/\sigma_0$  is dynamic compliance (I) and equal to the inverse dynamic module. The compliance makes sense strain in a single strain.

Using complex representation of harmoniously changing deformation:  $\varepsilon(t) = \varepsilon_0 e^{i\omega t}$ , strain rate will have an expression  $d\varepsilon(t)/dt = \omega \varepsilon_0 e^{i(\omega t + \pi/2)}$ . Substituting this expression in the differential equation (1) and reducing on  $\varepsilon_0 e^{i\omega t}$ , we received:

$$(i\eta\omega + E)E^*(i\omega) = iE\eta\omega, \quad (9)$$

where  $E^*$  is a complex dynamic modulus, which can be represented as:  $E^*(i\omega) = \frac{\eta^2 \omega^2 E}{E^2 + \eta^2 \omega^2} + i \frac{\eta \omega E^2}{E^2 + \eta^2 \omega^2}$  (10)

The first term is a real, and the second - the imaginary part of the complex dynamic modulus ( $E^* = E' + iE''$ ), which is proportional to  $E$  and depends on the frequency.  $E''(\omega)$  determines the losses at harmonic deformation and is the module of losses.

Similarly complex dynamic module  $E^*(i\omega)$  can be represented by a complex dynamic compliance  $I$

\* as the sum of the imaginary and real  $I' + iI''$  parts. Considering that  $I^* (i\omega) E^* (i\omega) = 1$ , we can provide the relevant expressions in the form:  $I^* (i\omega) = I'(\omega) + iI''(\omega)$ , where

$$I' = \frac{1}{E_0} + \frac{1}{E_1} \frac{1}{1 + \omega^2 \tau^2} = I_0 + \frac{I_1}{1 + \omega^2 \tau^2} \quad (10)$$

$$I'' = \frac{1}{E_1} \frac{\omega \tau}{1 + \omega^2 \tau^2} = I_1 \frac{\omega \tau}{1 + \omega^2 \tau^2}, \quad (11)$$

where  $I_0 = 1/E_0$ , and  $I_1 = 1/E_1$ . Absolute measured deformation looks like:  $I = \sqrt{I'^2 + I''^2}$

From condition  $I_1 \gg I_0$  (as  $E_0 \gg E_1$ ):

$$I \approx \frac{I_1}{\sqrt{1 + \omega^2 \tau^2}} \quad (12)$$

The phase angle  $\delta$  between the  $I$  and  $I'$ , i.e. between strain and stress is defined as:

$$\operatorname{tg} \delta = \frac{I''}{I'} = \frac{I_1 \omega \tau}{I_1 + I_0 (1 + \omega^2 \tau^2)} \quad (13)$$

In essence the angle  $\delta$  describes the mechanical loss, i.e. share of mechanical energy, which came into heat, or the proportion of dissipated energy per cycle of deformation per unit volume. A measure of this transformation may be an area corresponding to the hysteresis loop formed by the dependence of deformation on the voltage in the cycle of periodic actions (between the curve of loading and unloading).

At low frequencies, when you can measure the hysteresis loop and hysteresis loss coefficient is used mechanical loss [3]:  $\chi = \Delta W/W$ , where  $W$ -total work force for a series of mechanical deformation, and  $\Delta W$  - dissipated energy per cycle of deformation, which is proportional to the square hysteresis loop.

Between  $\chi$  and  $\operatorname{tg} \delta$  there is a dependence at all frequencies in terms of linear viscoelasticity. Thus, for asymmetric vibrations from 0 to  $2\varepsilon_0$  according to work [12] such dependence is found:

$$\chi = \frac{2\pi g \delta}{4\sqrt{1 + \operatorname{tg}^2 \delta} + \pi g \delta} \quad (14)$$

The decision of this equation concerning parameter  $\operatorname{tg} \delta$  gives dependence:

$$\operatorname{tg} \delta = \frac{4\chi}{\sqrt{4\pi^2 (1 - \chi) - 6\chi^2}} \quad (15)$$

This expression can be represented as:  $\operatorname{tg} \delta = \psi$ . In expression (13) from condition  $E_0 \gg E$  and  $I_1 \gg I_0$  at low frequencies in a first approximation, we obtain:

$$\operatorname{tg} \delta = \omega \tau \quad (16)$$

Equating last two expressions, we receive:  $\psi = \omega \tau$ , whence  $\tau = \psi / \omega$ .

According to the second postulate of the Boltzmann adopted in his theory of the elastic aftereffect, and the underlying Boltzmann-Volterra model that describes the relaxation phenomena, using a function of heredity [13]: action occurred in the past few strains on the stresses caused by deformation of the body at any given time, do not depend on each other and therefore algebraically added. This position has received also the name of a principle of the Boltzmann's superposition. It should be noted that the polymer body

superposition principle holds in the upper-bounded the range of deformation, stress and rate of change.

Given this principle, considering the dissipative processes occurring during application of periodic voltage to the material in the rubberlike state for a long time, we can conclude that there is an accumulation of mechanical energy dissipation in each cycle. Then, if the energy is transformed into heat during one cycle is determined by parameter  $\chi_1$ , then in a low heat with the environment during  $N$  cycles of the energy dissipated during the time  $t$  will be:  $\chi_{\text{com}} = \chi_1 t \nu$ , where  $t \nu = N$ .

Stored energy in the sample is converted into heat, which should lead to an increase in temperature. The principle of temperature-time superposition [14], which establishes the equivalence of the effect of temperature and duration of exposure on the relaxation properties of polymers, we can assume that the increase in the impact load on the material is proportional to the action of temperature. Empirical dependence of the temperature  $\Delta T$  of the exposure time  $t$  and the intensity (frequency) exposure to  $\nu$  in the first approximation be written as:  $\Delta T = b t \nu$ , where the  $b$ -parameter, taking into account the characteristics of energy conversion, depending on the structure of the material.

Relaxation time of the supplied periodic voltage decreases with increasing temperature and obeys the Arrhenius equation

$$\tau = \tau_0 e^{U/RT} \quad (17)$$

For elastic-plastic bodies similar dependence follows from Aleksandrova-Gurevich's equation [15] and has the form

$$\tau = \tau_0 \exp[(U_0 - a\sigma)/RT], \quad (18)$$

where  $U_0$  - activation energy of relaxation process, the constant of the material.

This equation takes into account the dependence of the relaxation of the load. If we assume that  $U_0 - a\sigma \approx U$  and to determine the relative relaxation time as  $\tau_t/\tau_1$  (the ratio of the current value of the relaxation time of the initial value of you during the load application), then, on the basis of equation (18), we can represent this value as an expression:

$$\frac{\tau_t}{\tau_1} = \exp\left(\frac{U}{R(T_1 + \Delta T)} - \frac{U}{RT_1}\right) \quad (19)$$

where the temperature  $T_1$  corresponds to the beginning of load application in the relaxation time  $\tau_1$ , and the increment of the  $\Delta T$  is the temperature change in the impact load.

After application of rather simple algebraic transformations the formula (19) will become:

$$\frac{1}{\ln \frac{\tau_1}{\tau_t}} = \frac{RT_1}{U} + \frac{RT_1^2}{U} \Delta T \quad (20)$$

If instead the increment of temperature  $\Delta T$  would use the proposed higher proportion of the value of the exposure time  $t$  and frequency of the applied load  $\nu$ , then the expression (20) becomes:

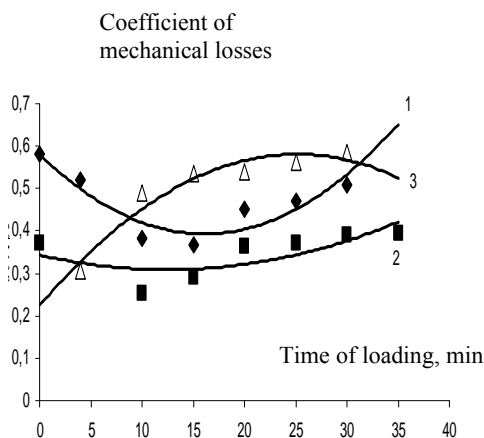
$$\left(\ln \frac{\tau_1}{\tau_t}\right)^{-1} = \frac{RT_1}{U} + \frac{RT_1^2}{U} (b \nu t)^{-1} \quad (21)$$

Using this expression and taking into account the approach adopted, it is possible to experimental data

on changes in the mechanical loss factor (tangent of mechanical losses) over time, the impact loads to find the estimates of the activation energy of relaxation process, determine the extent to which the process is stationary (steady), the degree of linearity of the relaxation processes and the range of conditions and the regime correct determination of relaxation parameters for a periodic load.

### Experimental part

In the present work as object of research polyethylene of low density (*LDPE*). Samples in the form of the cylinder: diameter (*d*) from 8 mm at a parity  $h/d = 1,5$  made pressing at temperature  $180^{\circ}\text{C}$ , pressure of  $150 \text{ kgs/sm}^2$ . To obtain a homogeneous sample produced an extract of the pressure and temperature  $180^{\circ}\text{C}$  at least 10 minutes with pre-pressing for the release of air located between the grains of the original polymer. Samples subjected to periodic monoaxial deformation of compression on the installation described in [16] at room temperature ( $293 \text{ K}$ ). As a result of the periodic action of the voltage on the sample received the stress-strain during loading and unloading of the form a hysteresis loop. The study used three discrete frequencies of loading: 0.017, 0.17 and 1.7 Hz. Under each of these frequencies is tested at least three samples for 30 min., Taking readings every 5 min. Results cheated, define the parameters of the mechanical losses as the ratio of the hysteresis loop to the area between the curve of loading and the axis of strain ( $\chi = \Delta W/W = S_{\text{loop}}/S_{\text{hole.}}$ ). The results of measurements of at least three samples were averaged and subjected to further processing in accordance with those presented in the theoretical part of the calculations. Figure 1 shows kinetic curves of variation of the mechanical loss by prolonged exposure of three frequencies: 0.017, 0.17 and 1.7 Hz.

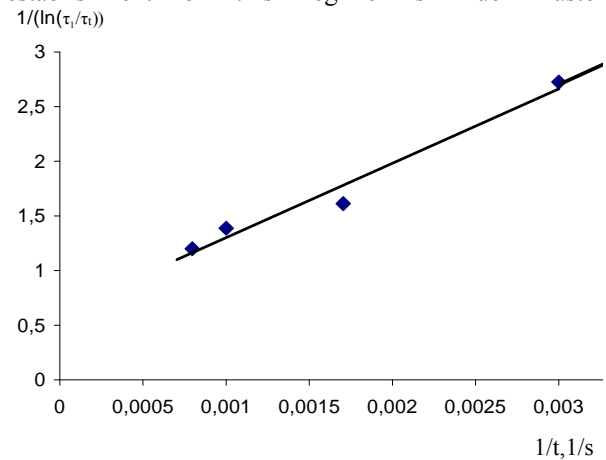


**Fig. 1 - Change of the mechanical losses coefficient eventually at influence of periodic loadings with frequency: 1- 0.017 Hz, 2 – 0.17 Hz, 3 – 1.7Hz**

It is seen that with increasing time of deformation coefficient of mechanical losses vary, but relationships have different characteristics

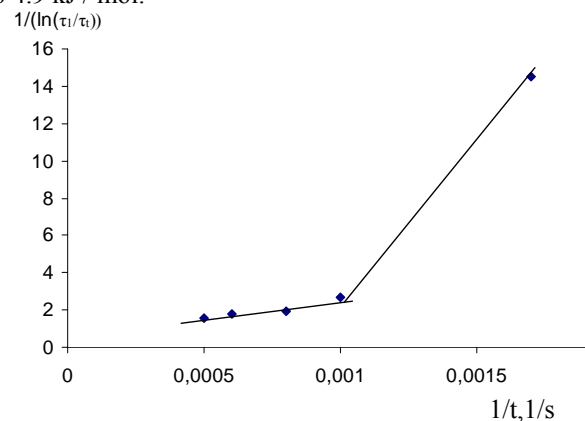
So for low frequencies 0,017Hz ( curve 1) and 0,17 Hz ( curve 2)the initial value of this parameter is higher than the next. In all probability this is due to the

fact that during the reduction of  $\chi$  is the system output at steady state, ie where the constant *C* in equation (4) becomes equal to 0. For higher frequency - 1.7 Hz (curve 3) the establishment of this regime is much faster. For higher frequency - 1.7 Hz (curve 3) the establishment of this regime is much faster.



**Fig. 2 - Dependence of relative time of a relaxation ( $\tau/\tau_0$ ) on duration of influence with frequency of 0,017 Hz**

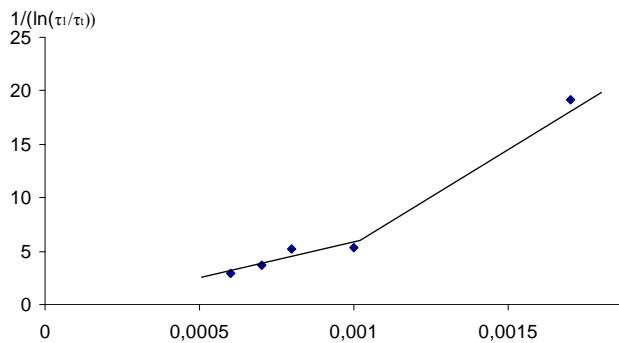
Figure 2 shows the inverse of the logarithm of the relative relaxation time in degree -1 (which corresponds to the left side of the equation (21)) the reciprocal of the time of impact load on the sample with a frequency of 0.017 Hz. Dependence is well approximated by a straight line, i.e. the found coordinates dependence of relative time of a relaxation and time of influence of loading is directly proportional. Meaningfully the value found at the intersection of this dependence with the vertical axis and bearing  $T_1$  equal to ambient temperature ( $293 \text{ K}$ ) can determine the activation energy. In these conditions (Fig. 2) it is equal to  $4.9 \text{ kJ/mol}$ .



**Fig. 3 - Dependence of relative time of a relaxation ( $\tau/\tau_0$ ) on duration of influence with frequency of 0,17Hz**

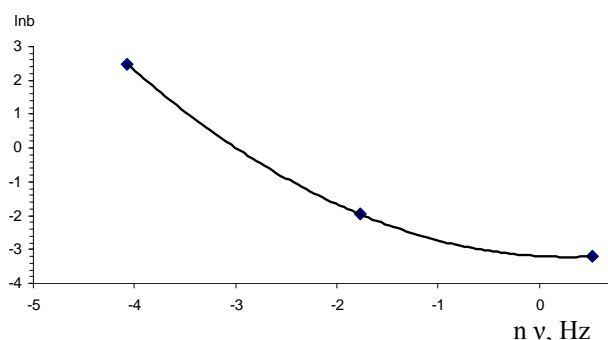
The slope in Figure 2 provides an estimate of the value of the parameter "b" in formula (21) . The calculation shows that the frequency of 0.017 Hz,  $b = 11,88$ . Since the dependence is linear in a rather wide time interval, this gives reason to conclude that the activation energy of relaxation process with periodic loading of the solid LDPE under these conditions virtually unchanged.

Using the representation of the relative relaxation time of the duration of the periodic effects in the corresponding coordinates for the frequency of 0.17 Hz (Fig. 3) makes it possible to calculate the activation energy and the parameter  $b$  for the relaxation process of solid LDPE under these conditions. With accuracy to the experimental errors (for a test frequency of 0.17 Hz), the activation energy is 4.9 kJ / mol and  $b = 0,414$ . In Fig.4 a similar dependence is shown for the frequency of 1.7 Hz. The calculated value of activation energy is the 2,4 kJ/mol. The value  $b = 0,04$ .



**Fig. 4 - Dependence of relative time of a relaxation ( $\tau/\tau_0$ ) on duration of influence with frequency of 1,7Hz**

Analyzing the obtained values, we should note a decrease of  $b$  with increasing frequency (Fig. 5), indicating that the difference in the relaxation processes occurring at different frequencies. Another interesting fact is that for frequencies 1.7 and 0.17 Hz sampling rate on the parameter  $b$  is the same and equal to 0.07, while the frequency of 0.017 Hz (three orders of magnitude smaller than the largest) is the product of three times and equals 0,202. It should be noted the difference in the nature of the drawings: Figure 2 - to  $v = 0,017$  Hz and Figures 3 and 4 - respectively for 0.17 and 1.7 Hz.



**Fig. 5 - Dependence of parameter "b" from frequency of loading**

In the Figure 2 no jumps in the dependence, in Fig1f on fig 3 and 4, the values for the initial periods of exposure time is several times higher than those in the subsequent course of dependencies. Perhaps this difference is caused by various structural transformations under mechanical loading with different frequencies.

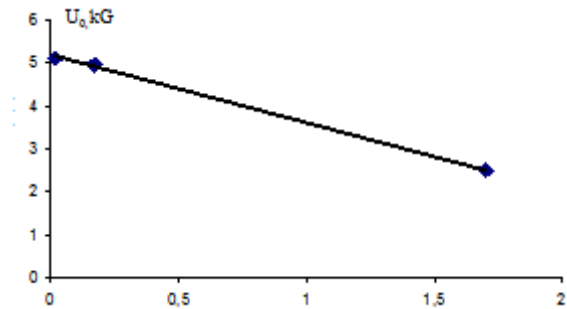
With regard to the activation energy, it is the smallest (2.4 kJ / mol) for the frequency of 1.7 Hz and for frequencies of 0.17 and 0.017 Hz, the activation energy,

calculated according to the results of these experiments, it turns out the same and equal to 4.9 kJ.

To paraphrase the equation of the Aleksandrov-Gurevich (18), where instead of the stress ( $\sigma$ ) using the frequency  $v$ , instead of the coefficient "a" use the "b", then we can define a certain characteristic value, similar to  $U_0$ , the initial activation energy of relaxation process, a constant:

$$U_0 = U + bv$$

Analysis of the dependence of the initial activation energy  $U_0$  of the frequency (Fig. 6) shows that with increasing frequency  $v$  decreases linearly energy  $U_0$  frequency of loading, Hz



**Fig. 6 - Dependence of initial energy of activation relaxation process from frequency of loading**

Thus, using the principle of temperature-time superposition and kinetic coefficient of mechanical losses can be at different intensities of loads to determine the time interval in which the measurement of relaxation parameters will be most correct. Besides using the above approximation, we can give a preliminary assessment of the relaxation parameters and analyze the nature of relaxation processes taking measurements without changing the initial temperature.

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