Effect of thermomechanical impacts on the deformation of thin polyimide films at uniaxial tension

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Abstract. Thermomechanical studies of the dependence of deformation ($\epsilon$) on time ($t$) and temperature ($T$) for various static stresses in polyimide under uniaxial tension were carried out. Temporal deformation curves for static stresses in the range from 75 to 110 MPa consist of two stages: 1 – increase; 2 – exit to saturation, which is associated with a change in the nature of structural transformations of macromolecules, due to the action of the static stresses. An increase in temperature and voltage leads to an increase in the dependence $\epsilon(T)$. This is due to the intensification of the processes of structuring macromolecules with increasing temperature and are supplemented by processes of breaking bonds between macromolecules, which increase the strain rate. Experimental data are described in terms of exponential and linear models.

1. Introduction
The most close attention among polymer systems of various classes is caused by materials based on aromatic polyimides (PI) [1, 2], which have a unique combination of extremely high heat and heat resistance, excellent mechanical and electrical characteristics combined with high chemical and radiation resistance [3, 4].

PI matrices retain the ability to crystallize after melting and subsequent cooling [5, 6]. In this regard, it is relevant to obtain new fundamental results in the field of deformation and thermomechanical properties of materials, to determine the safe strength life of products under the combined effect of external loads and operating parameters, and to assess the hazard of technological and temporal factors [7 – 11].

Polyimides, including the alicyclic structure, are the most important polymers for the successful solution of problems in materials science. In this area, research is carried out in two directions – along the path to creating polymers with electrical insulating and electrically conductive properties. The improvement of individual physicomechanical properties leads to an improvement in the characteristics of polyacrylates, in particular, elasticity, which also expands the field of application of alicyclic polyimides [4].

In [12, 13], it was found that polyfunctional amines, in particular tetraamines, bis (o-phenylenediamines) and bis-[3-amino-4 (p-aminophenoxy)] arylenes, are widely used to modify PIs. When polyanilines are used as polyfunctional amines, PIs with good performance properties associated with the formation of block film-forming materials of spatial structure were obtained [14].
The effect of moisture on charge relaxation processes in modified polyimide films with one-sided and two-sided teflon coatings was established in [15]. This paper is devoted to the study of the complex effects of various static loads and temperatures on the mechanical properties of polyimide.

2. Experimental

Industrial polyimide with a thickness of 45 μm was selected as the test material. Film samples were cut using a special device. The length of the test material was 70 mm, the working part 50 mm, width 5 mm.

Studies of the dependence of ε on t carried out on the developed experimental setup, consisting of the following blocks: case, bracket, upper clamping device, sample under study, lower clamp, displacement sensor, load system, force (voltage) sensor, and video camera. The body is a tripod with a variety of mounts, on which all parts of the installation are located. When measuring the relative elongation (ε) and stress (σ), a special interface with various measurement sensors (Science Cube) was used. To conduct experimental studies, we determined the maximum load with the help of special cargoes, i.e. the maximum value of the force at which the sample was torn. For this material σ = 112 MPa. Then the voltage was calculated, which constitutes a certain part of the limit voltage. After that, the sample was fixed in clamps, and the dependence of ε on t was recorded. The time of testing the sample for uniaxial tension was about 50 seconds. The data was recorded on a surveillance camera for further processing.

When conducting research on the dependence of ε on temperature (T), an installation has been designed and manufactured that makes it possible to measure parameters using motion and force sensors at different loads and their change with time. Figure 1 shows the temperature as a function of time in the upper part of the installation when it is heated to 180 °C. It was obtained that the error lies within 5 %.

![Figure 1](image-url)

**Figure 1.** Dependence of temperature on time in the upper part of the temperature unit when it is heated to 180 °C

3. Results and discussion

A series of experiments was carried out to study the dependence of ε on t under various static loads. The graphs are presented in Figure 2. The patterns of strain change allow to study and evaluate the nature of the structural changes due to the effect of static load. Exposure to a polyimide film with a voltage of 60 MPa does not lead to a change in the relative elongation (Figure 2, curve 1).

The deformation-time curves for static loads in the range of 75 – 110 MPa have a similar character, consisting of two stages: 1 – in the range of 0 – 12 seconds, the relative elongation of the material
increases; 2 – at \( t = 12 \sim 50 \) seconds \( \varepsilon \) comes to saturation. Curve 1 is described by a linear function \( \varepsilon = at \) \((a = 0)\), and curves 2 – 5 by the formula:

\[
\varepsilon = \varepsilon_0 \left( 1 - \exp\left( -\frac{t}{t_0} \right) \right).
\]

The values of \( \varepsilon_0 \) and \( t_0 \) are listed in the table.

Table 1 – Dependence of \( \varepsilon_0 \) and \( t_0 \) on \( \sigma \)

| \( \sigma \) [MPa] | 75  | 85  | 100 | 110 |
|-------------------|-----|-----|-----|-----|
| \( \varepsilon_0 \) [%] | 29  | 34  | 42  | 47  |
| \( t_0 \) [sec.] | 4   | 5   | 7   | 8   |

It is seen that with increasing \( \sigma \), \( \varepsilon_0 \) and \( t_0 \) grow.

![Figure 2](image_url)

Figure 2. Dependence of deformation on the time of the polyimide film at various static stresses

Then, the dependences of \( \varepsilon \) on \( T \) were studied experimentally for various values of the static loads \( \sigma_1 = 60; \sigma_2 = 75; \sigma_3 = 85; \sigma_4 = 100 \) and \( \sigma_5 = 110 \) MPa, shown in Figure 3. As can be seen from the figure, the relative elongation increases with increasing \( T \) and \( \sigma \). As the temperature rises, the destruction processes are intensified.
Figure 3. The dependence of the relative elongation of the temperature of the polyimide film at different static loads

The first three curves indicate the intensification of the processes of structuring macromolecules with increasing temperature, and curves 4 and 5 indicate a change in the nature of the transformation of the polymer structure, which are complemented by the processes of breaking the bonds between macromolecules, which increase the deformation. The calculation according to [16] leads to a change in the tangent of the angle of inclination from 0.04 to 0.64.

The dependence of the maximum relative elongation ($\varepsilon_{\text{max}}$) on the static load of the polyimide film during thermomechanical testing is presented in Figure 4. The analysis shows that this dependence is characterized by a monotonic increase with saturation. Such behavior of $\varepsilon$ indicates a change in the nature of the structural transformations of macromolecules, due to the specificity of the action of static load and temperature. The dependence of $\varepsilon$ on $\sigma$ is described by the expression:

$$\varepsilon_{\text{max}} = \varepsilon_0 \left(1 - \exp\left(-\frac{\sigma}{\sigma_0}\right)\right),$$

where for this case $\varepsilon_0 = 57\%$ and $\sigma_0 = 60$ MPa.
Figure 4. Dependence of maximum relative elongation of polyimide film on static load during thermomechanical testing

Further, the dependence of the residual deformation $\varepsilon_{\text{res}}$ on $\sigma$ was investigated (Figure 5). As can be seen from figure 5, the dependence is linear.

![Figure 5](image-url)

Figure 5. Dependence of the residual deformation on the static load of the polyimide film during thermomechanical testing

4. Conclusion

1. Experimental work has been carried out to study the dependence of deformation on time for various values of static stresses. The deformation-time curves for static stresses in the range from 75 to 110 MPa have a similar character, consisting of two stages: 1 – in the range of 0 – 12 seconds, the relative elongation of the material increases; 2 – at $t = 12 – 50$ sec it reaches saturation. Such behavior indicates the change in the nature of the structural transformations of macromolecules due to the action of the static stresses.

2. It was found that the dependence of $\varepsilon$ on $T$ increases with both increasing $T$ and $\sigma$. This is due to the intensification of structuring of macromolecules with increasing temperature and it is complemented by the processes of breaking bonds between macromolecules, which increase the rate of deformation.

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