

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

A I Kupchishin^{1,2}, B G Taipova¹, M N Niyazov¹, V M Lisitsyn³ and N A Voronova¹

¹ Abay Kazakh National Pedagogical University, 13 Dostyk, Almaty, Kazakhstan

² Al-Farabi Kazakh National University, 71 al-Farabi ave., Almaty, Kazakhstan

³ National Research Tomsk Polytechnic University, Tomsk, Russia

E-mail: ankupchishin@mail.ru

Abstract. Thermomechanical studies of the dependence of deformation (ε) 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 $\varepsilon(T)$. 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 physicochemical 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 $\sigma = 112$ 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 %.

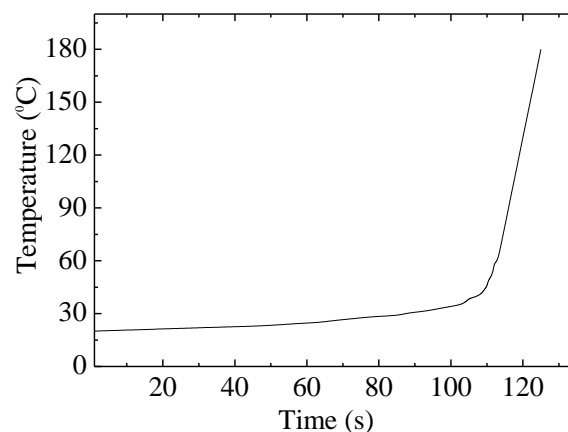


Figure 1. Dependence of temperature on time in the upper part of the temperature unit when it is heated to 180 °C

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 $t = 12 - 50$ seconds ε comes to saturation. Curve 1 is described by a linear function $\varepsilon = at$ ($a = 0$), and curves 2 – 5 by the formula:

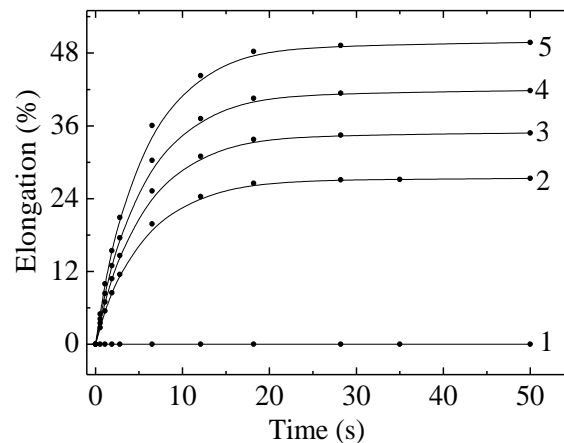
$$\varepsilon = \varepsilon_0 \left(1 - \exp\left(-\frac{t}{t_0}\right) \right).$$

The values of ε_0 and t_0 are listed in the table.

Table 1 – Dependence of ε_0 and t_0 on σ

σ , [MPa]	75	85	100	110
ε_0 , [%]	29	34	42	47
t_0 , [sec.]	4	5	7	8

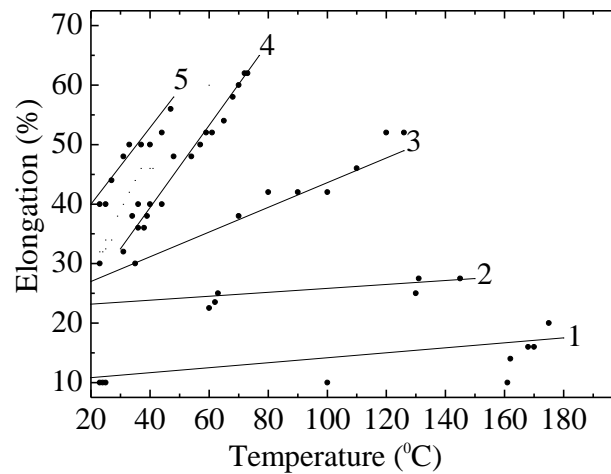
It is seen that with increasing σ , ε_0 and t_0 grow.



1 – 60; 2 – 75; 3 – 85; 4 – 100; 5 – 110 MPa;
points – experiment; solid lines – calculation

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

Then, the dependences of ε 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 σ . As the temperature rises, the destruction processes are intensified.



1 – 60; 2 – 75; 3 – 85; 4 – 100; 5 – 110 MPa

Points – experiment; solid lines – calculation according to Hooke

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 (ε_{\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 ε 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 ε on σ is described by the expression:

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

where for this case $\varepsilon_0 = 57\%$ and $\sigma_0 = 60\text{ MPa}$.

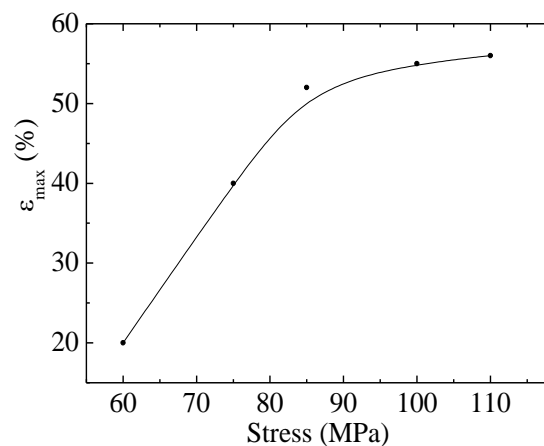


Figure 4. Dependence of maximum relative elongation of polyimide film on static load during thermomechanical testing

Further, the dependence of the residual deformation ε_{res} on σ was investigated (Figure 5). As can be seen from figure 5, the dependence is linear.

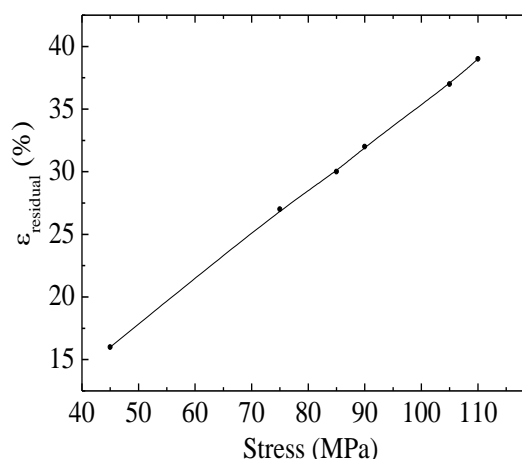


Figure 5. Dependence of the residual deformation on the static load of the polyimide film during thermomechanical testing

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 ε on T increases with both increasing T and σ . 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.

References

- [1] Krutko E T, Prokopchuk N R and Globa A I 2017 Chemical modification of polypyromellitimide *Polymeric materials and technologies* **3** 1 33-46
- [2] Liu B, Zhang B, Wang X, Chen X and Gu G 2019 The preparation and properties of polyimide films modified by octa(aminopropylsilsesquioxane) *Journal of Saudi Chemical Society* (In press. <https://doi.org/10.1016/j.jscs.2019.02.002>)
- [3] Zuev V V, Uspenskaya M V and Olekhovich A O 2010 *Physics and chemistry of polymers Textbook* (SPb.: SPbSU ITMO)
- [4] Kudaykulova S K, Iskakov R M, Kravtsova V D, Umerzakova M B, Abadie M, Zhubanov B A et al 2006 *Polymers for special purposes* (Almaty)
- [5] Lian M, Zheng F, Lu X and Lu Q 2019 Tuning the heat resistance properties of polyimides by intermolecular interaction strengthening for flexible substrate application *Polymer* **173** 205-214

- [6] Kim K, Yoo T, Kim J and Han H 2015 Effects of dianhydrides on the thermal behavior of linear and crosslinked polyimides *J. Appl. Polym. Sci.* **132** 6 44412-21
- [7] Gerasimenko N N, Voronova N A, Kovtunets V A, Taipova B G, Kupchishin A I and Omarbekova Z A 2003 Study of the mechanical properties of polymer films effecting on their by static load and temperature *Proceedings of the universities. Materials of electronic technology* 6-12
- [8] Sh.-Sh Dong, W.-Zh Shao, Li Y, Hui-Jian Ye and Liang Zhen 2018 Microstructure evolution of polyimide films induced by electron beam irradiation-load coupling treatment *Polymer Degradation and Stability* **155** 230-237
- [9] Rahnamoun A, Engelhart D P, Humagain S, Koerner H, Plis E, Kennedy W J, R Cooper, Greenbaum S G, Hoffmann R and Adri CT van Duin 2019 Chemical dynamics characteristics of Kapton polyimide damaged by electron beam irradiation *Polymer* **176** 135-145
- [10] Sorokin S V, Terentyev A V and Krotov E A 2014 *Introduction to the mechanics of a deformable solid* (SPb: SPbGMTU)
- [11] Kupchishin A I, Niyazov M N, Voronova N A, Kirdiashkin V I and Abdukhairova A T 2017 The effect of temperature, static load and electron beam irradiation on the deformation of linear polymers *IOP Conf. Series: Materials Science and Engineering* 168012016 1 – 4
- [12] Babman T, Hamid Y 2001 Preparation and properties of novel polyimides de-rived from 4-aril-2,6-bis(4-aminophenyl)pyridine *J. Polym. Sci. Part A, Polym. Chem.* **39** 21 3826-31
- [13] Lee D H et al. 2002 Preparation and properties of aromatic polyimides based on 4,4-(2,2,2-trifluoro-1-phenylethylidene)diphthalic anhydride *J. Appl. Polym. Sci.* **85** 1 38-44
- [14] Meleshko T K et al. 2009 Electrically conductive film-forming compositions based on a mixture of polyaniline and polyimides *Macromolecular compounds. Ser. A* **51** 3 447-453
- [15] Kamalov A M and Borisova M E 2016 The effect of moisture on the relaxation of electric charge in modified films based on polyimide *Scientific-technical statements SPbGPU. Physics and Mathematics* **3** (248) 33-38
- [16] Voronova N A, Kupchishin A I, Niyazov M N and Lisitsyn V M 2018 Uniaxial Stress and Electron Irradiation Effects on Nanochains Straightening in Film Polymer Materials *Key Engineering Materials* **769** 78-83