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Analysis of Stress Relaxation Processes in Polyimides at High and Low Temperatures

Paulius BANEVIČIUS, Jonas GYDAS[1*]

[1]*Faculty of Design and Technologies, Kaunas University of Technology, Studentų 56, LT-3031, Kaunas, Lithuania*

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The article presents an analysis of viscoelastic behavior of thermoplastic heat-resistant polymers (HRP) polyimides under stress relaxation conditions at high (up to 500 °C) and at low (up to –170 °C) temperatures. The main characteristic features of stress relaxation processes are considered on the basis of polyimide PI3 in comparison to other polyimides PI1 and PI2. The polyimides under consideration have different aromatic structures and various hinge-type groups in polymer macromolecules. The difference in structure of macromolecules leads to the different glass transition and relaxation solid-solid transition temperatures. According to the results obtained the relaxation processes in polyimide PI3 are rather well expressed at high as well at low temperatures. On the basis of isochronous-isothermal relationships of relaxing stress the existence of critical state is disclosed and critical parameters expressed by critical stresses and critical strains are determined. Applying limiting parameters (fracture stress, fracture strain) and critical parameters (critical stress, critical strain) the mechanical efficiency of polyimide PI3 is estimated under stress relaxation conditions at high and low temperatures.

Keywords: heat–resistant polymer, polyimides, stress relaxation, critical state, critical stress, critical strain, limiting parameters, mechanical efficiency.

INTRODUCTION

Thermoplastic polyimides (PI) represent one of the most important groups of structural heat-resistant polymers (HRP) [1 – 3]. The structure of PI differs essentially from that of conventional polymers and can be widely varied resulting in different physical properties. During the past decades a lot of various types of PI have been developed and a large variety of different materials have been created. Structure of various PI and possible ways for their development are considered in detail in special literature [4 – 7].

Aromatic structures of macromolecules define high glass transition temperatures of thermoplastic PI (210 – 520 °C) and lead to the overall balance of their thermal-mechanical properties in a wide range of temperatures [3 – 6]. Due to this fact the structural materials based on PI take a special importance in connection with wider application of high-performance materials under extreme loading conditions.

Under such conditions, when considerably high levels of strains or stresses are caused, especially in the region of elevated and high temperatures, the relaxation processes in polymers are clearly expressed [8]. The most important of relaxation processes, as stress relaxation and creep under isothermal loading conditions, have influence on mechanical behavior of polymers and require to be investigated in a more extended spectrum.

The mechanical behavior of some thermoplastic polyimides as PI1 and PI2 under stress relaxation conditions has been investigated in the wide range of temperatures and analysed in [9 – 11].

The relaxation processes in HRP change non-uniformly and in certain regions of temperature their rates differ substantially. The non-uniform change of relaxation processes in definite temperature regions of a glassy-state indicates the existence of relaxation solid-solid transitions (ss-transitions), which occur long before the glass transition temperature. Considering a wide range of glassy-state temperatures from glass transition temperature up to –170 °C only two ss-transitions for HRP were detected [12]. For some HRP ss-transition occur even in the region of low temperatures [13].

This article deals with the presentation of experimental results of viscoelastic behavior of thermoplastic polymer PI3 under isothermal conditions of stress relaxation. The analysis of stress relaxation processes of PI3 is done in the range of high and low temperatures (500 °C – –170 °C). The main features of mechanical behavior of PI3 under stress relaxation conditions is considered in comparison to polyimides PI1 and PI2 having different structures.

MATERIALS AND RESEARCH PROCEDURES

Relaxation processes and viscoelastic properties of HRP depend on such very important factors as polymer structure, temperature, level of strains, loading type and deformation rate [3, 8 – 13].

For investigation and analysis of relaxation processes of one group of HRP the polyimides having different structure (PI1, PI2 and PI3) were considered. The chemical structure of their monomers is represented as follows:

Polyimide (PI1)

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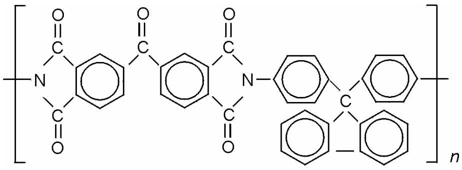
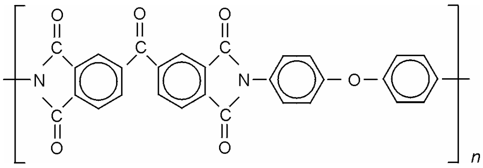
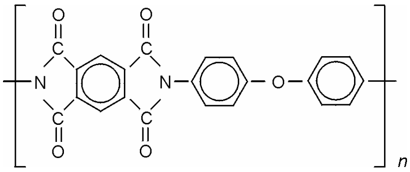
[*] Corresponding author. Tel.: + 370-37-300231; fax: + 370-37-353989.
E-mail address: *jonas.gydas@.ktu.lt* (J. Gydas)



Polyimide (PI2)



Polyimide (PI3)



According to the represented monomer structure the main chains of macromolecules of investigated polyimides contain aromatic rings, imide cycles and different number of hinge-type links. Aromatic rings or cyclic groups in the backbone reduce flexibility and add bulkiness. Flexibility and bulkiness of the monomer unit and the polymer chain that is built from it influence some important properties directly [3].

For thermoplastic HRP, as materials developed for high-temperature service, primarily the thermal characteristics as glass transition temperature T_g and temperatures of relaxation ss-transitions T_{ss} are to be evaluated. Characteristic temperatures of polyimides under consideration are represented in Table 1 [9 – 11].

Table 1. Characteristic temperatures of thermoplastic polyimides

Polymer	Characteristic temperatures T, °C		
	T_g	T_{ss1}	T_{ss2}
PI1*	380	320	–
PI2	380	110	300
PI3	500	40	300

*Note: no relaxation results at low temperatures.

The glass transition temperature for amorphous polymers reflects the transition of a material from the rubbery state to the glassy one. The importance of glass transition temperature T_g as a material property can be understood in terms of loss of rigidity that accompanies the transitions. A drop of several decades in the modulus of elasticity from a common value at T_g is usually observed above the transition temperature. The glass transition temperatures of polyimides were determined applying the temperature-scanning method with constant temperature elevation rate ($\beta_T = 4$ °C/min) [8].

Below the glass transition temperature the amorphous polymers show secondary transitions, which also have influence on viscoelastic properties of a material. The relaxation solid-solid transitions (ss-transitions) in HRP are considered as intrinsic material-dependence phenomenon and reflect increase or decrease relaxation processes with respect to temperature at the definite it regions. In general, the transition temperatures T_{ss} divide the region of a glassy-state into three sub-regions having different mechanisms of relaxation processes [12].

Hence, the temperatures T_g and T_{ss}, as the key controlling parameters in the time temperature-dependent viscoelastic behavior of a polymer, are very important factors in the determining the usefulness of a given polymer. The most impressive value of the glass transition temperature ($T_g = 500$ °C) corresponds to the polyimide PI3. The backbone of macromolecule of PI3 is created on the basis of aromatic nuclei, imides cycles and only one hinge-type group (–O–). In this case the material is rigid and brittle.

Due to the changes of the polyimide structure incurred by incorporating two hinge-type groups (–O– and –CO–) into the main chain of macromolecule making them flexible, as done in PI2, it is possible to create more plastic material. Location of those hinge-type groups in a polymer macromolecule has a significant influence on physical properties of the material. The hinge-type groups, involved only in a dianhydride component, lead to less rigid polyimide with a high glass transition temperature. When hinge-type groups are involved only in a diamine component of a macromolecule the polyimides are more deformable at low temperatures [3].

In polyimides having the same number of imide cycles, benzene nuclei and other groups, their heat resistance increases with an increase in degree of the condensed cycles. The polyimides with involved hinge-type groups in dianhydride as well as in diamine components are characterized as having less density but higher deformability in comparison to those based on rigid macromolecules. By involving various hinge-type groups the increase of deformability may be achieved but the main advantage of these polymers as their high heat resistance, characterized by value of T_g, can be lost to a great measure (Table 1).

The main results of analysis are represented on example of polyimide PI3 with the highest glass transition temperature. For investigations of mechanical behavior of polyimides the small-scale specimens with dimensions of 3×3×4.5 mm were applied. The specimens of block-shaped material were produced by the method of compression moulding at high temperatures (520 °C) and pressures (100 MPa) with further application of machining. Processing conditions of investigated polyimide PI3 and optimisation of it technology were considered in [14].

The investigations of relaxation processes of PI3 were performed under uniaxial compression in isothermal stress relaxation conditions. The levels of initial strains and stresses were varied covering regions of linear and non-linear viscoelasticity. The ranges of temperatures were varied in the limits from low of –170 °C up to the region of glass transition (softening) temperature being for PI3 of 500 °C.

Such main factors as the level of initial strain, temperature, and duration of the test time affect strongly the relaxation processes of polymer materials [8]. Taking into account the functional dependence of relaxing stress, the general features of viscoelastic behavior were obtained by changing one factor, which governs the relaxation processes, on retention of constant the others.

RELAXATION AT HIGH TEMPERATURES

Influence of Initial Strain. The value of initial strain has an essential influence on the level of stress as well as on the course of a relaxation process. Due to change of preassigned initial strain the stress relaxation curves are gradually shifted from the region of smaller stresses to the region of larger ones. As an example the stress relaxation curves for PI3, obtained at constant temperature $T = 220$ °C and various initial strains (1 – 9 %) are represented in Fig. 1.

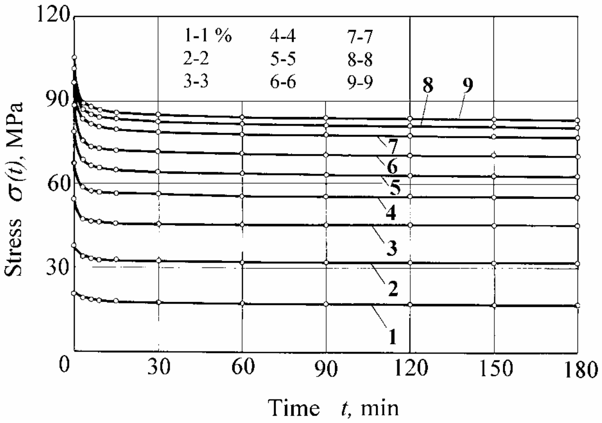


Fig. 1. Stress relaxation curves for PI3 at constant temperature $T = 220$ °C and at various initial strains

With an increase in initial strain the stress relaxation process becomes more intensive at the first stage (curves 1 and 9). The analysis of stress relaxation curves show that the effect of the initial strain on relaxation process is expressed not so distinctly for the value of relaxing stress as for the rate of stress decrease.

In the second stage, when the stress relaxation process is running at a more or less settled rate, the other view is observed. In this stage the relaxation rates are close to each other. The stress relaxation curves of the second stage are similar and usually they differ only in their relative positions with respect to the relaxing stress at that time.

At very high levels of strains the relaxation process is going with an essentially greater rate and the stress relaxation curves are closer displaced (Fig. 1, curves 7 – 9). At a further increase in strain the stress relaxation curves begin to be shifted from the region of large stresses to the region of small ones. Such phenomenon has been noticed for polyimides PI1 and PI2 [9, 11] and is also characteristic for PI3. The level of strains at which explained shifting of stress relaxation curves is noticed depends on temperature. Due to increase of initial strains the shifting of relaxation curves in the opposite direction occurs at lover levels of stresses in the region of higher temperatures.

Influence of Temperature. Due to the change of temperature the mobility of structural elements alters and the final result is reflected on the relaxation process. The influence of temperature on the stress relaxation process of polyimide PI3 is analysed at various levels of initial strains. As an illustration of viscoelastic behavior of PI3 at different temperatures the relaxation curves, obtained in the region of high temperatures and constant initial strain $\varepsilon_0 = 6$ % are represented in Fig. 2.

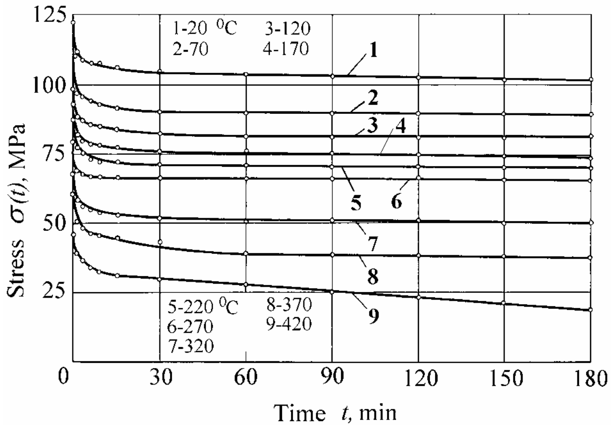


Fig. 2. Stress relaxation curves for PI3 at constant initial strain $\varepsilon_0 = 6$ % and various high temperatures

According to the results obtained the increase in temperature leads to the intensive decrease in stress at its initial stage. In this stage a noticeable change of relaxation rates is also observed with an increase in initial tensile strain. The character of stress relaxation curves of this polyimide can be divided into two sub-states in the region of a glassy-state. Up to the relaxation ss-transition temperature 300 °C (Fig. 2, curves 1 – 6) low intensity of relaxation processes is observed. At the temperature that is higher than 300 °C (curves 7 – 9) intensity of relaxation processes increases even at low levels of strains.

At quite high temperatures the stress relaxation curves are often losing their similarity and at different strain levels the relaxation rates become variable (Fig. 2, curves 8, 9). When the initial strain is much less if compared to the fracture strain, its increase leads to the greater value of relaxing stress. Polyimides PI1 and PI2 also behave in a similar way at high temperatures under stress relaxation conditions [9, 11].

Thus in the region of a glassy-state the increase in temperature is reflected by viscoelastic behavior in the following manner. First of all, with an increase the temperature the period of an initial relaxation process becomes longer and the slope of a stress relaxation curve becomes larger in the second stage.

When the temperature is increased actually but is rather far from the softening temperature, the stress relaxation process at large levels of strains may limit itself only by the first stage of a curve. It demonstrates the essential drop of relaxing stress in a material. From the engineering point of view the temperature region and the level of strains, which result in the mentioned stage of a relaxation process, are of special interest when mechanical load-bearing capability of HRP is to be evaluated [15].

The essential drop of relaxing stress for polyimides is evident at the range near to the glass transition or softening temperatures (Table 1: PI1 – 380 °C; PI2 – 380 °C and PI3 – 500 °C). For these polymers with an increase in temperature not only a great drop of relaxing stress is observed, but there is a rather stable second period of relaxation. Taking into account these features the polyimide PI3 is valuable as relaxation-stable structural material, suitable to be used under long-term loading conditions at relatively high temperatures.

By introducing in polyimide PI2 main chain additionally to hinge-type link –O– such link as –CO– it is possible to change it flexibility and create ductile material compared to the brittle polyimide PI3. According to the results obtained the increase of flexibility of polymer macromolecules leads to more intensive relaxation processes and essential decrease of stress level possible for different polyimides at the same temperature regions [11].

RELAXATION AT LOW TEMPERATURES

Influence of Initial Strain. Pronounced deformability of HRP at low temperatures is related to their expressed relaxation processes at high levels of strains [10, 13]. Thus, investigation of stress relaxation of HRP at low temperatures is a question no less important than the one in the range of high temperatures.

The level of initial strain, as it was considered in the range of high temperatures, has the essential influence on the rates of a relaxation process. These rates are in a definite manner reflected on viscoelastic behavior of polymers in the range of low temperatures. As an example, the stress relaxation curves for investigated PI3 at temperature $T = -170$ °C and various levels of initial strains are represented in Fig. 3.

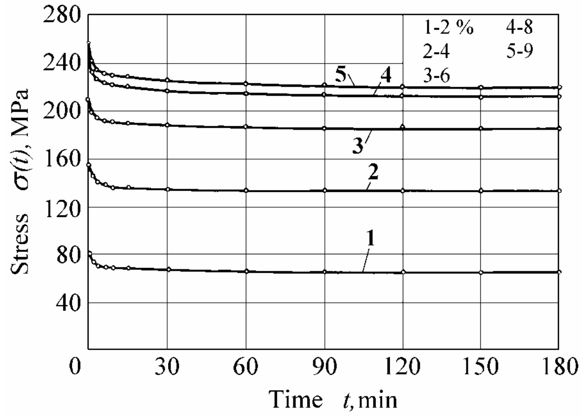


Fig. 3. Stress relaxation curves for PI3 at constant temperature $T = -170$ °C and at various initial strains

With the increase in the initial level of strains the stress relaxation curves are consistently shifted to the region of higher stresses. Such regularity is characteristic even at temperature of –170 °C. At very high levels of strains, however, the relaxation process becomes faster. As a result of it the stress relaxation curves become closer to each other (Fig. 3, curves 4, 5).

According to the results obtained the process of stress relaxation at low temperatures as at high ones may also be characterized by two stages. In the first stage a high rate of a relaxation process is observed and in the second one the rate of this process becomes essentially lower. Comparisons of relaxation curves obtained at low temperatures with the curves at high ones reveal some essential differences.

The first stage of stress relaxation process at low temperatures is more prolonged (10 – 20 min) than that at high ones. Higher intensity of these processes at low temperatures is observed in the second stage.

The process of stress relaxation runs deeper in the first stage at high temperatures and its intensity in the second stage is expressed in lower degree. Actually, with the growth of initial strain main regularities of relaxation processes for investigated polyimide PI3 remain similar to those as at high temperatures.

At the same time it should be noted that the values of stresses in the relaxation curves are essentially higher when compared with those at high temperatures. This phenomenon is related to the fact that the decreases in temperature in the range from 20 °C up to –170 °C leads to the significant increase in yield or fracture stress.

Influence of Temperature. The change of temperature is related to the change of macromolecular mobility and, consequently, to the rates of relaxation processes in polymers. With lowering of temperature the relaxation process is developed in a material not so deep. Nevertheless, it is reflected on viscoelastic behavior of HRP [13].

As an example, the stress relaxation curves for PI3 obtained at constant initial strain $\varepsilon_0 = 6$ % and at various temperatures within the limits from 20 °C up to –170 °C are represented in Fig. 4.

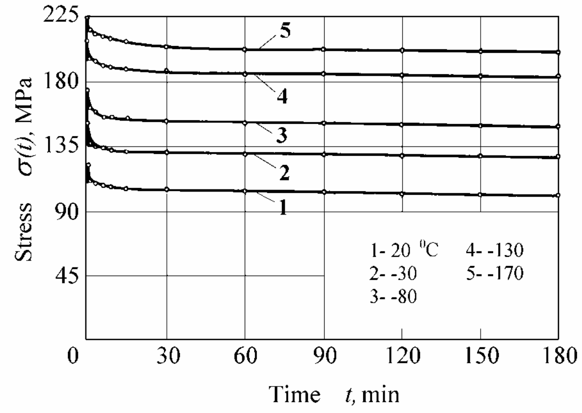


Fig. 4. Stress relaxation curves for PI3 at constant initial strain $\varepsilon_0 = 6$ % and at various low temperatures

According to the experimental results, the lowering of temperature even up to –170 °C leads to the essential slow down of relaxation processes. In spite of this fact, they are pronounced (Fig. 4, curve 5) and must be evaluated when polymer is intended to be applied under extreme loading conditions. Furthermore, it should be emphasized that relaxation processes for PI3 are more pronounced in the range of low temperatures from –50 °C up to –100 °C, especially at high levels of initial strains.

With the decrease in temperature the stress relaxation curves are regularly shifted to the level of higher stresses. As it can be seen from given results, the level of stresses at the same initial strain but at different temperatures may vary in a rather wide range of its values (Fig. 4, curves 1 and 5).

As a generalized characteristic which makes it possible to reveal the influence of temperature on relaxation processes, the relationships of $\sigma_t = f(T)$ plotted at various strains may be used (where σ_t is the stress at definite or final test time). Such relationships for some PI3 are represented in Fig. 5.

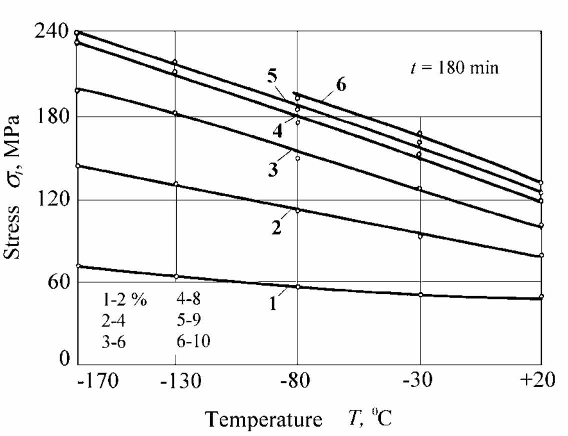


Fig. 5. Relationships of relaxing stress σ_t with respect to temperature for PI3 at constant duration t = 180 min and at various strains

For polyimide PI3 the relationships $\sigma_t = f(T)$ are close to the straight lines. It means that the rates of relaxation process with the decrease in temperature for the definite strain levels practically remain unaltered. Different slopes of considered relationships express the change of relaxation rates, which depend on the initial levels of strains.

But for another HRP polybenzoxazole (PBO), as it is considered in work [13], two stages have been determined with different relaxation rates in the relationship $\sigma_t = f(T)$ when initial strain is increased. At rather great initial strains with lowering of temperature up to –150 °C the levels of relaxing stress become much higher, but the location of a transition region does not depend on the value of initial strain.

CRITICAL STATE OF RELAXATION

On the basis of the stress relaxation data the isochronous-isothermal relationships of relaxing stress with respect to strain $\sigma(t) = f(\varepsilon_0)$ were constructed. Such relationships, obtained at the different temperatures and durations, make it possible to reveal the critical state and determine the values of critical stress and strain.

For investigated PI3 the relationships, $\sigma(t) = f(\varepsilon_0)$, constructed at high and low temperatures and different durations of the relaxation process are represented in Figs. 6 and 7.

The isochronous-isothermal relationships of relaxing stress, at the different durations of the test are also separated. The regions of these relationships shown by hatching cover the durations of relaxation processes from 0.5 up to 180 min. For PI3 the maxima on the relationships $\sigma(t) = f(\varepsilon_0)$ are expressed not so clear as for other HRP [9 – 11, 13].

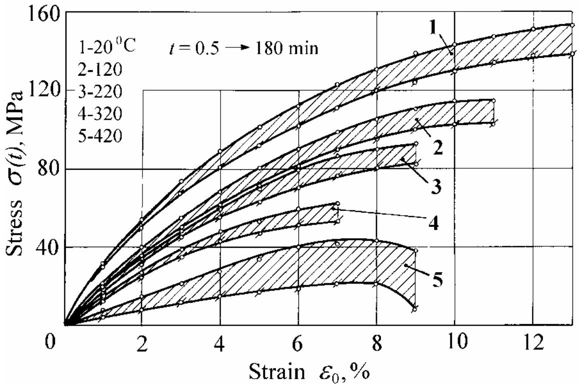


Fig. 6. Isochronous-isothermal dependences of relaxing stress (compression) for PI3 at various temperatures. Duration of stress relaxation process: ○ – 0.5 and ⌀ – 180 min

According to the results obtained the relationships $\sigma(t) = f(\varepsilon_0)$, resemble each other at different temperatures. Mentioned phenomenon is characteristic both in the range high (Fig. 6) and low (Fig. 7) temperatures.

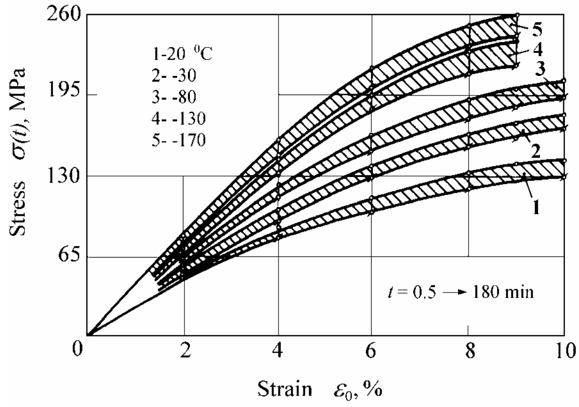


Fig. 7. Isochronous-isothermal dependences of relaxing stress for PI3 at various temperatures. Duration of stress relaxation process: ○ – 0.5 and ⌀ – 180 min

The occurrence of such maxima is related to the increase in relaxation rates at rather large levels of strains. The exceeding of stress or strain corresponding to this maximum leads to the mechanical softening of polymer material. The values of stress and strain at the maximum σ_{cr} and ε_{cr}, respectively, are assumed as critical parameters and define the critical stage in a polymer [15]. In the case as obtained for PI3 Figs. 6 and 7), when such maximum does not occur, the largest values of stress and strain at the longest possible to be determined duration are assumed as critical parameters.

According to the results of experimental investigation the values of critical strains usually do not depend on the duration of a relaxation process. But the level of relaxing stresses decreases with the growth of the duration of this process. Thus, in fact, the values of critical stresses depend on elapsed time.

The analysis of the relationships $\sigma(t) = f(\varepsilon_0)$ has shown that the location of a maximum on a curve depends on the temperature. In all cases the stresses and strains corresponding to the maximum on the curve become smaller with an increase in temperature (Figs. 6 and 7; curves 1 and 5).

Exceeding of stress and strain, conforming to the maximum, leads to an essential change of the relaxation rate in polymers. These critical parameters are used to evaluate the mechanical efficiency of HRP under stress relaxation conditions [15].

MECHANICAL EFFICIENCY

For quantitative assessment of load-bearing capability of polyimide PI3 the stress and strain determined under definite loading conditions are assumed as the criteria of mechanical efficiency [15]. These criteria are expressed by limiting and critical parameters determined by evaluating the limiting state under constant deformation rate and the critical state under stress relaxation conditions.

High Temperatures. The temperature relationships of critical and limiting parameters constructed for PI3 in the range of high temperatures are shown in Fig 8.

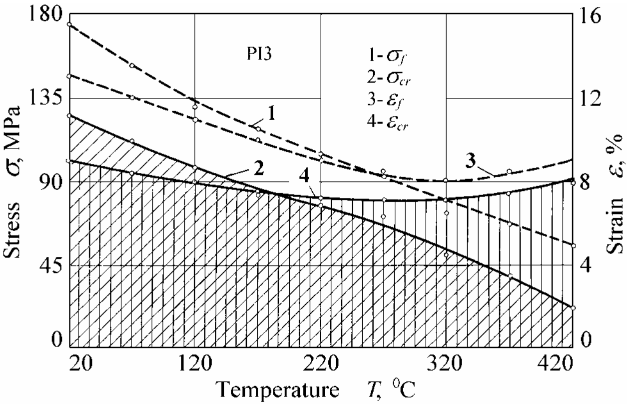


Fig. 8. Relationships of limiting (dashed lines) and critical parameters (solid lines) for PI3 with respect to temperature under static compression and stress relaxation conditions, respectively

Curves $\sigma_{cr} = f(T)$ and $\varepsilon_{cr} = f(T)$ together with the axes of coordinates confine the region of stresses or strains and temperatures in which polymer behaves like a solid material. The softening or fracture of a solid polymer does not occur at the prearranged conditions of loading and this region is usually known as the region of load-bearing capability. The load-bearing capability under the isothermal stress relaxation conditions means the ability of a polymer to resist failure and softening within the entire range of temperatures, stresses, and strains possible for that polymer [15].

At an increase in temperature the region of load-bearing capability with respect to stress substantially decreases. According to the results obtained for PI3 the change of critical stress σ_{cr} with an increase in temperature is more expressed than that of critical strain ε_{cr}. The change of the value of strain ε_{cr} is not so essential despite the fact that critical stress σ_{cr} decreases substantially. In the case of other polyimides at the temperature regions close to the glass transition temperature the essential decrease in the value of strain ε_{cr} has been observed [9 – 11].

Consequently, the level of stresses and strains, defining the region of mechanical capability of polymers, becomes lower with an increase in temperature. These features are especially distinctly marked under extreme loading conditions when the relaxation process takes place near the softening temperatures of polymers.

The regions of load-bearing capability of PI3, determined on the basis of relationships $\sigma_{cr} = f(T)$ and $\varepsilon_{cr} = f(T)$ (Fig. 8, solid lines), are smaller compared to those determined by the relationships $\sigma_f = f(T)$ and $\varepsilon_f = f(T)$ (dashed lines). In the region of lower temperatures where the relaxation processes are less expressed the smaller difference between limiting and critical parameters is observed.

Low Temperatures. Mechanical efficiency of polyimide PI3 under isothermal stress relaxation conditions at low temperatures is evaluated applying the same criteria as those used at high ones. The temperature relationships of critical parameters $\sigma_{cr} = f(T)$ and $\varepsilon_{cr} = f(T)$ compared to the relationships of limiting parameters $\sigma_f = f(T)$ and $\varepsilon_f = f(T)$ are shown in Fig. 9.

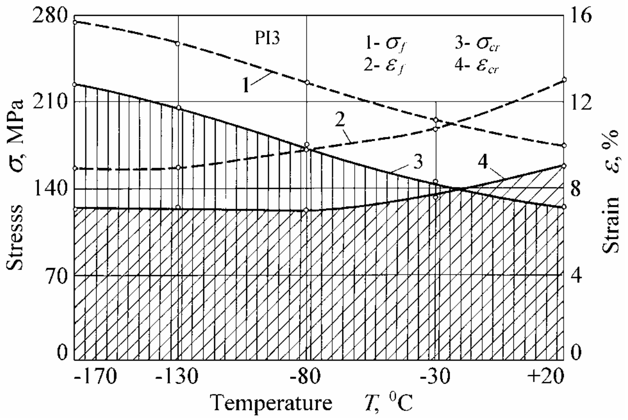


Fig. 9. Relationships of limiting (dashed lines) and critical parameters (solid lines) for PI3 with respect to temperature under static compression and stress relaxation conditions, respectively

According to the results obtained, the general features of load-bearing capability of HRP in the region of low temperatures may be summarized in the following way. At a decrease in temperature (from 20 up to –170 °C) the essential growth of critical stresses σ_{cr} is noticed (Fig. 9, curve 3). The regions of load-bearing capability of PI3 determined on the basis of relationships $\sigma_{cr} = f(T)$ and $\varepsilon_{cr} = f(T)$ (Fig. 9, solid lines) are smaller compared to those determined applying the limiting parameters expressed by relationships of $\sigma_f = f(T)$ and $\varepsilon_f = f(T)$ (dashed lines).

It is obvious that application of limiting parameters for determination of load-bearing capability of PI3 as structural material cannot be considered sufficiently enough in the wide range of temperatures. The assessment of load-bearing capability of PI3 done on the basis of temperature relationships of critical parameters obtained in the stress relaxation process enables to evaluate the possibilities of this material to be used under extreme loading conditions.

At the same time it should be noted that the critical stress and critical strain are rather convenient parameters for more rigorous comparison and evaluation of different polymers with respect to their capability to be applied under the stress relaxation conditions at various temperatures. In this connection, the investigation of mechanical efficiency of HRP in the region of high and low temperatures has a great importance from the scientific as well as the practical point of view [16, 17].

CONCLUSIONS

Due to it aromatic structure thermoplastic polyimide PI3 is characterized by an excellent balance of mechanical properties in the region of both high and low temperatures. High deformability in the range of low temperatures even up to –170 °C and pronounced relaxation processes at high levels of stresses and strains allow also to consider heat-resistant polyimide PI3 as cold-resistant polymer.

At an increase in temperature the region of load-bearing capability with respect to stress substantially decreases. According to the results obtained for PI3 the change of critical stress with an increase in temperature is more expressed than that of critical strain.

The regions of load-bearing capability of PI3, determined on the basis of critical parameters are smaller compared to those determined by the limiting ones. In the region of lower temperatures where the relaxation processes are less expressed the smaller difference between limiting and critical parameters is observed.

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