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The Effect of Electron Irradiation and Stretching on the Modification of Macromolecules of Polyimide Films

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Abstract. *The effect of the absorbed dose of 40 MGy of electron irradiation and uniaxial tension to rupture at room temperature on the Instron 5982 test breaking machine on changes in the structure of polyimide film macromolecules is studied. Irradiation was carried out on an electronic linear accelerator ELA-6, at room temperature, and the structure change was studied using the Nicolet 5700 IR spectrometer in the range of 4000-400 cm^{-1}, the ESP 300 E spectrometer from Bruker at a wavelength of 3.2 cm, and uniaxial stretching to rupture at room temperature on the Instron 5982 test breaking machine. The aim of this work was to study changes in the structure of macromolecules of polyimide films as a result of electron irradiation and mechanical loading. It was found that after irradiation, the intensity of the IR spectrum increases by ~2-6 times and the width of the bands increases, indicating an increase in the content of radicals. Irradiation of the film with subsequent mechanical loading causes a shift in the EPR line of the spectrum from 3475.0 cm^{-1} to 3512.5 cm^{-1} with a simultaneous decrease in the signal amplitude from $6 \cdot 10^3$ to $4 \cdot 10^3$, indicating a decrease in the concentration of radicals = N-H, -N-H_2 groups until their complete disappearance and the formation of new ones caused by electron irradiation.*

Keywords: *polyimide, films, electron irradiation, IR- and EPR-spectroscopy, breaking machine, Instron 5982, supramolecular structure, mechanical properties, absorbed dose, Bruker ESP 300 E spectrometer.*

Introduction

Intensive introduction of new polymer and composite materials in engineering and industry poses the task of finding ways to develop them, a detailed study of their composition, structure and physical and chemical processes occurring at the same time. These features of the polymer structure are complex formations with varying degrees of ordering of segments of macromolecular chains, forming the internal structure of the material. The structure of the material is influenced by the specifics of its supramolecular organization (SMO), which determines the spatial arrangement of individual elements. Such an organization of the structure of polymers forms their physical properties Classification of SMO is usually made according to the main discrete or fluctuation features [1, 2].

The purpose of this work is to identify the features of the modification of polyimide films as a result of changes in the defective structure of macromolecules irradiated with high-energy electrons, as well as subjected to unicose stretching.

Materials and Methods

Industrial undirected polyimide (PI) films were selected as the objects of the study.

Polyimides are widely used in the production technology of semiconductor devices. Among other polymers, they stand out for their characteristic physical properties such as: high temperature degradation, mechanical strength and radiation resistance [3].

Polyimides have a low permittivity value, which contributes to the amplification of signal propagation in electronic devices. Therefore, they are used in multilevel high-density and low-speed electronic circuits. During operation, in the designs of devices exposed to radiation exposure, the properties of polyimides are stable during operation, so they have found wide application [4-5].

Polyimides belong to a class of heat-resistant synthetic polymers whose macromolecule structure contains an imide cyclic group.The structural link of a polyimide macromolecule based on pyromelitic acid dianhydride and paraoxydiphenylenediamine is

shown in figure 1 [2].

Polyimide has good mechanical characteristics such as strength retention over a wide temperature range. Considering these properties, they are widely used in promising areas of technology, such as aerospace instrumentation, transport engineering, nuclear power engineering, etc. We made samples from polyimide films that were 5 cm long, 5 cm wide, and 130 μm thick [6, 7].

The prepared samples were irradiated using an electronic linear accelerator of the ELA-6 type. The irradiation was performed by electron beams with an average energy of 2 MeV, with an integral current of up to 1000 μA and a pulse frequency of 200 Hz with a duration of 5 μs. The absorbed dose (D) of the samples was 40 kGy, 50 kGy, and 500 kGy.

The destruction of polymers was studied by IR spectroscopy and the products of chemical transformations after electron irradiation were analyzed. As is known, IR spectroscopy is based on irradiating a sample with light in a wide range of the infrared spectrum and then measuring the absorbed wavelengths [8, 9]. The Nicolet 5700 IR spectrometer was used to measure the spectra in the range of 4000-400 cm^{-1}. The power on the line was 200 mWt with a spectral resolution of 0.09 cm^{-1}.

In polymers, as a result of irradiation, compounds with free radicals are formed that have unpaired electrons, the EPR of which is very effective. They interact between the magnetic dipole moment of the spin of an unpaired electron and the magnetic dipole moment of the spin of a molecule or radical. This interaction leads to the appearance of a multicomponent structure in the spectrum instead of a single signal splitting of the spectrum [10]. Studying the nature of this splitting of the spectrum, the number of lines and their relative intensity allows us to get an idea of the structure of the paramagnetic system and valuable information about its microdynamics. By irradiating the sample with radiation at one wavelength and collecting the resulting scattered radiation, EPR spectra are obtained. The EPR method consists in the fact that the frequency of scattered light depends on the strength of bonds within the molecule, on the mass of bound atoms, and on the intermolecular interaction, and this serves as valuable information about the structure of para-magnetic systems.

We used a Bruker ESP 300 E spectrometer at a wavelength of 3.2 cm with a resonator power of ≤ 200 mWt, a modulation frequency of 100 kHz, and a sensitivity of $7 \cdot 10^{13}$ sp/Tl. The magnetic field strength was 0.01-1.5 Tl with a field stability of 10^{-5} per hour and a field uniformity of 10^{-5} Tl/cm. The central magnetic field had 34.70 G and a magnetic field sweep of 100 G. 2 types of resonators were used: rectangular – type TE-102 and cylindrical – type TE-011.

The manufactured samples were subjected to uniaxial stretching to rupture at room temperature on the Instron 5982 test breaking machine, which met all the requirements of European and American standards. The Instron 5982 Electromechanical machine had the following technical characteristics: the maximum permissible load of 100 kN, load and strain measurement errors were +/– 0.5% of the measured value.

Discussion of the results

The effects of radiation on polyimide films cause changes in their defective structure and affect their physical and chemical properties. Therefore, establishing the nature of the formation and evolution of the defective structure, as well as an appropriate understanding of the mechanism of radiation-induced changes in the properties of materials is an urgent task of researchers. Radiation-induced defectiveness caused by exposure to powerful beams of charged particles affects a significant change in the polymer structure, affecting the spatial transformation of the polymer. As a result, the molecular weight of macromolecules changes, some types of chemical bonds accumulate and disappear, etc. The structural changes caused in the material significantly affect the restructuring of the supramolecular structure and, which are reflected in the variety of its properties.

Radiation exposure to polymer materials leads to significant changes in their physico-chemical properties and structure, which depend on the initial structure, its composition and degree of purity, characteristics of incident radiation, radiation dose, etc. This promotes the release of individual atoms or radicals and the disruption of the corresponding bonds in macromolecules. An increase in the number of labile atoms leads to a significant change in the structure, affecting the change in the physical properties of the material.

The characteristic course of chemical reactions in polymers under the influence of mechanical stresses causes a violation of the degree of ordering of the structure and the manifestation of their level. The effects of mechanical stresses cause the rupture of chemical bonds in the main chain of polymers.

In polymer materials, as a result of exposure to various types of radiation, the defective structure and, as a result, physical and chemical properties change. The degree of such changes and their nature depend on many complex mechanisms of effects on the polymer, which are associated with the initial

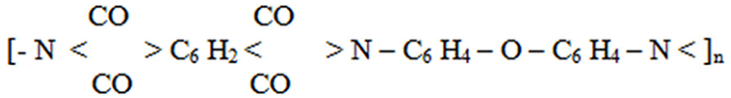

Figure 1 – The form of structural unit of the polyimide c molecule

parameters of the polymer, the characteristics of the affected radiation, etc. [2]. In this regard, when conducting such studies, the actual and main tasks of researchers are to establish the nature of the creation and evolution of the defective structure in the studied material, as well as the relationship of radiation-induced defectiveness with changes in their properties.

Such studies are primarily caused by practical interest in solving problems of improving the reliability of devices and devices operating in the radiation field, and secondly, the prospects for the development of radiation technologies that provide targeted modification of the physicochemical properties of materials.

In polymers irradiated by powerful beams of charged particles, significant structural changes occur due to the spatial transformation of macromolecules. This leads to a change in the molecular weight of the material, as well as some types of chemical bonds strengthen and disappear, etc. The structural changes caused significantly affect the re-structuring of the supramolecular structure of polymer materials. This causes them to develop various properties, consisting in improving or deteriorating technical characteristics.

Irradiation of polymer materials causes the formation of radicals formed as a result of dissociation of excited states of macromolecules, as well as due to the course of secondary reactions of radicals and ions. These factors affect the concentration of macroradicals formed, reaching up to ~ 10^{19}-10^{21} cm^{-3} [4]. As a result, significant structural changes occur in these materials, as well as modal characteristics of their properties, which are associated with irreversible processes of structuring and destruction. These processes are opposite in nature and occur simultaneously. At the same time, the structure of the polymer affects the predominance of one process over others, and also depends on the elements of the system present and the conditions of irradiation. In [7-10], regularities were established and shown that the presence of organic compounds of certain functional groups in macromolecules leads to the occurrence of radiation absorption with a characteristic frequency. Such spectra make it possible to identify with a high degree of probability that a particular absorption band belongs to a specific functional group present in the molecule. The vibrations of the rest of the molecule are no more than 5% and do not affect the nature of the frequencies of the functional groups. Therefore, the obtained spectra of functional groups allow us to accurately determine the structural features of the molecules of complex compounds.

The IR spectra of unradiated and irradiated polyimide films were measured using the Nicolet 5700 spectrometer, the results of which are shown in Figures 2 and 3. The films were irradiated with electrons with an energy of E = 2 MeV and a dose of D = 40 MGy. The analysis of polyimide spectra was based on the study of changes in the absorption bands 720 and 1380 cm^{-1} (C-N group in the imide cycle), 1775 cm^{-1} (C = O group in the imide cycle). It can be seen from Figures 2 and 3 that irradiation

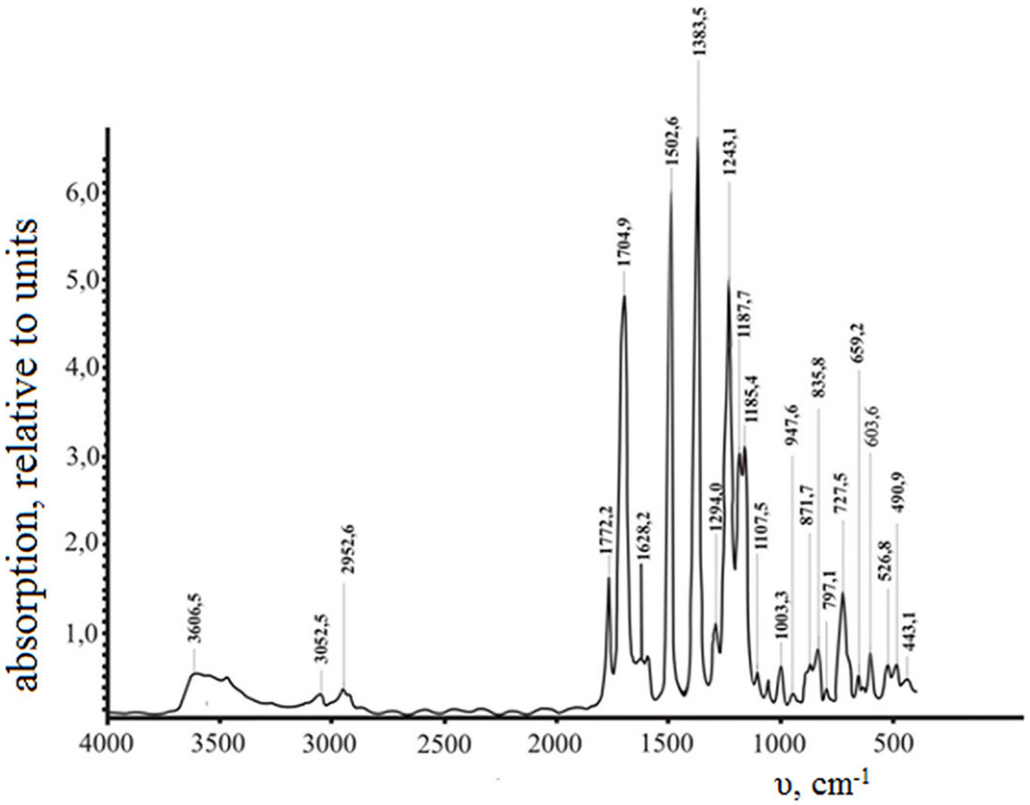

Figure 2 – IR spectrum of an unirradiated polyimide film

causes an increase in the intensity of these spectral lines by ~ 2-6 times, and also significantly increases the width of the bands.

Table 1 shows informative lines of the IR spectrum of the polyimide film, which lie in the intervals 500-1100 cm^{-1} and 1700-3500 cm^{-1}.

In the first interval, the content of substituted benzene rings of polyimide films is strongly noticeable. At the same time, in the range from 1700 to 3500 cm^{-1}, the content of associated polyimide groups is characteristic. As we can see, irradiation causes an increase in the content of radicals in the samples, which is reflected in an increase in the intensity of the spectrum and is explained by the formation of hydrogen bonds.

The change in the intensity of the absorption bands in the polymer (Figures 2, 3 and Table 1) it is explained by the fact that under the influence of irradiation, the processes of competing processes are caused: the formation of cycles involving nitrogen atoms and the formation of nitrogen oxides.

The EPR studies of these films were based on the determination of the equipment operating modes that do not distort the shape of the EPR signal.

Table 2 shows the data on measurements of the EPR spectra for one polyimide film irradiated with electrons.

For the sample, the standard mov.ave filter is

Table 1 – Comparison of the intensity of the IR spectrum of a polyimide film irradiated with electrons with energy E = 2 MeV and a dose D = 40 kGy with an unirradiated one

Chemical compounds	Frequency range cm^{-1}	The increase in intensity (times)
Overtone benzene rings PI	443,1	2
	490,9	2,8
	526,8	3,6
	603,6	3,4
	660	3,5
Aliphatic groups	726	3,3
Substituted benzene rings PI	797,1	3,3
	835,8	3,7
	871,7	3,7
	947,6	4
Lactone groups	1772,2	2,5
Associated carboxyl groups PI	3052,5	6,7
Associated groups	3361,5 and 3466,3	3,8

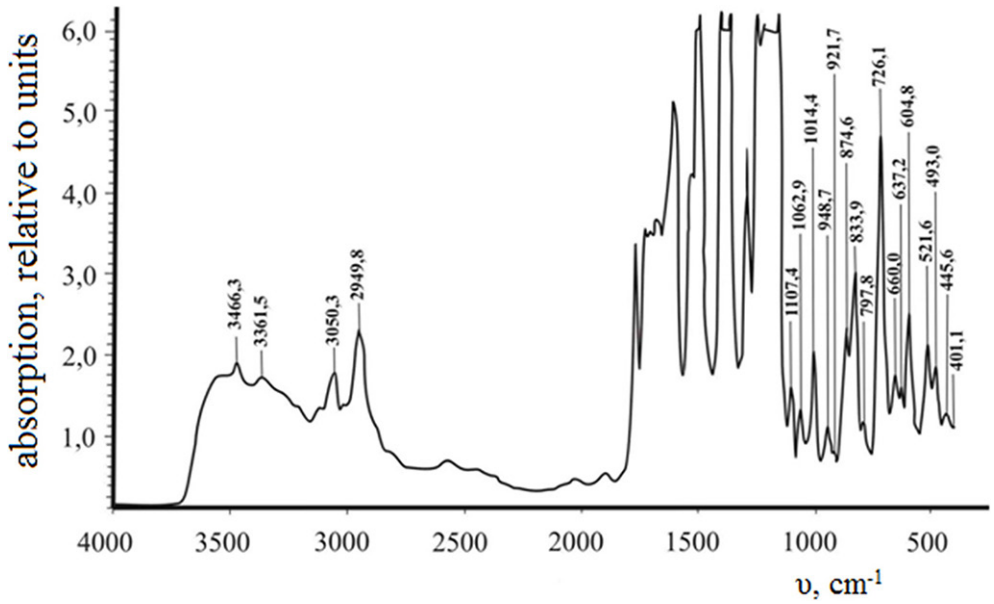

Figure 3 – IR spectrum of a polyimide film irradiated with electron energy E = 2 MeV and absorbed by a dose D = 40 kGy

applied. The data in the table are given by 15 points for the parameters of the EPR signal (width ΔH_{pp} and g-factor).

Table 2 shows that for the same sample of a polyimide film, the value of the line width ΔH_{pp} increases by 0.38 mTl, when the amplitude of the magnetic field modulation changes from 0.6 to 1.6 mTl. This indicates its modulation broadening. At the same time, this change does not affect the value of the g-factor. It remains constant. Therefore, a modulation amplitude of 6.15 mTl was chosen for serial measurements, which does not distort the shape of the EPR signal.

Table 3 shows the results of measuring the EPR spectra of polyimide films, unradiated and unbroken (initial) and irradiated with electrons and subjected to stretching to rupture (experimental).

Table 3 shows that the parameters of the EPR signal do not depend on the action of the external load after irradiation. The values: line width ΔH_{pp} are 0.925 mTl and 0.930 mTl, respectively, and the g-factor is 2.00824 and 2.00791.

Figure 4 shows the change in the intensity of the EPR signal of polyimide films from the concentration of paramagnetic centers.

It can be seen that the EPR spectrum is determined by radicals associated with associated groups = NH, -NH2 of linear type. The concentration of such radicals increases as a result of exposure to an external mechanical load until the film breaks (Figure 4a and 4b), which is reflected in an increase in the amplitude of the EPR signal from $3 \cdot 10^3$ to $5 \cdot 10^3$. The value of the relative elongation of the films at break did not exceed 6.

The effect of electron irradiation on polyimide films, followed by their mechanical loading, causes a shift of the spectrum line from 3475.0 cm^{-1} to 3512.5 cm^{-1} with a simultaneous decrease in the signal amplitude from $6 \cdot 10^3$ to $4 \cdot 10^3$ (Figure 4c and 4d), i.e. there is a decrease in the concentration of radicals of the above groups = N-H, -N-H_2 to their complete disappearance and the formation of new ones caused by the effect of electron irradiation. This means that the changes in the EPR spectrum shown in Figure 4 reflect the transition of radicals of polyimide films from one type to another, due to the action of external load and electron irradiation. Moreover, this transition is accompanied by the complete disappearance of the first type of radicals and the formation of new ones.

The results presented on the main mechanisms and patterns of changes in the mechanical properties of polyimide materials as a result of the actions of these physical factors allow us to make scientifically sound recommendations for the creation of new materials, as well as for the improvement of existing ones. They are based on the studied processes of formation of defects and radicals with high mobility, causing changes in their mechanical properties.

The peculiarity of the processes of changing the mechanical properties of polyimide films is that as a result of the external mechanical load and electron irradiation, the radicals in these materials transition from the original form to others, which is accompanied by the complete disappearance of the first type of radicals and the formation of new ones. Such changes in the polymer structure are automatically reflected in its mechanical properties. At the same time, the number of radicals in a polyimide film primarily depends on the effect of an external mechanical load and only then on the dose of radiation exposure.

The spatial arrangement of the individual structural elements of the polymer is formed by a specific supramolecular organization that cooperates with its internal structure, on the basis of which its physical properties are formed.

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Such changes in the polymer structure are automatically reflected in its mechanical properties. At the same time, the number of radicals in a polyimide film primarily depends on the effect of an external mechanical load and only then on the dose of radiation exposure. The spatial arrangement of the individual structural elements of the polymer is formed by a specific supramolecular organization

Table 2 – Parameters of the EPR signal of the PI film (width ΔH_{pp} and g-factor)

Sample Name	Sample weight, mg	Power, mW	Ampl. modulation, mtl	Width ΔH_{pp}, mT	g-factor
experimental	46	3,99	0,6	0,82	2,00788
experimental	46	3,99	1,60	1,2	2,00790

Table 3 – Effect of electron irradiation and stretching to break on the EPR parameters of the PI film signal

Sample Name	Sample weight, mg	Power, mW	Ampl. modulation, mT	Width ΔH_{pp}, mT	g-factor
initial	43	3,99	6,15	0,925	2,00824
experimental	26,3	3,99	6,15	0,930	2,00791

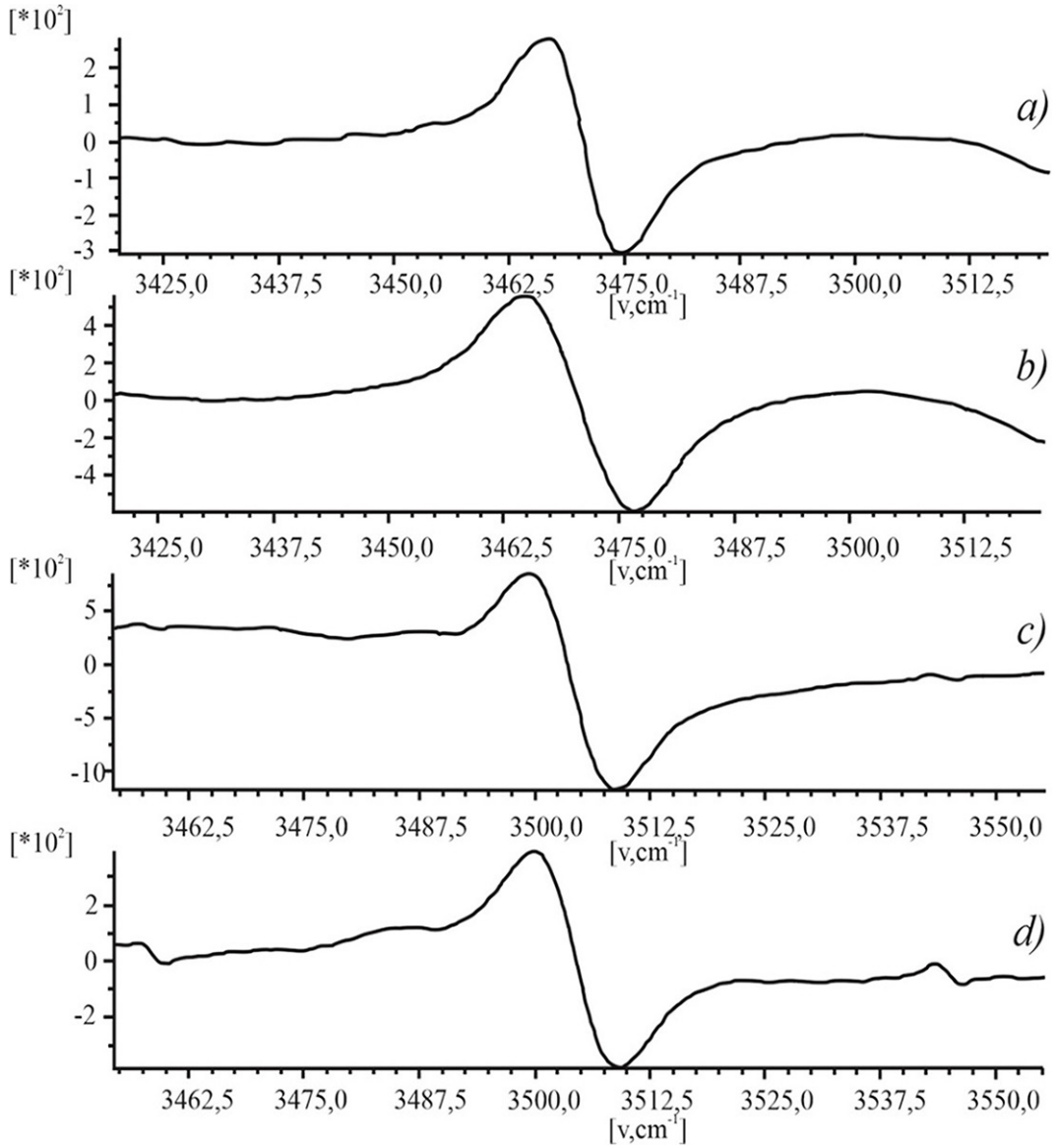

Figure 4 – EPR spectra of polyimide films: unirradiated and unbroken (a); unirradiated and stretched to break (b); electron-irradiated at a dose of 50 kGy and stretched to break (c); electron-irradiated at a dose of 500 kGy and stretched to break (d)

that cooperates with its internal structure, on the basis of which its physical properties are formed.

Conclusions

1. The experimental results obtained allow us to conclude that: electron irradiation of polyimide causes an increase in the intensity of the IR spectrum by ~ 2-6 times and significantly increases the width of its bands. The most informative lines of the spectrum lie in the intervals 500-1100 cm^{-1} and 1700-3500 cm^{-1}.

The first interval characterizes the content of substituted benzene rings of PI films. The second interval from 1700 to 3500 cm^{-1} shows the content of associated groups of polyimide macromolecules. An increase in intensity indicates an increase in the content of radicals as a result of irradiation.

This behavior of polyimide materials is associated with the formation of hydrogen bonds, and the change in the intensity of the IR spectrum of absorption bands in them (under the influence of irradiation) is caused by the course of competing processes: the formation of cycles involving nitrogen atoms and the formation of nitrogen oxides.

2. The effect of uniaxial stretching to rupture of non-irradiated polyimide films is reflected in an increase in the amplitude of the EPR signal from $3 \cdot 10^3$ to $5 \cdot 10^3$, which is caused by an increase in the number of radicals in the material as a result of mechanical action.

3. Electronic irradiation of polyimide films with their subsequent mechanical loading causes a shift of the EPR spectrum line from 3475.0 cm^{-1} to 3512.5 cm^{-1} with a simultaneous decrease in the signal amplitude from $6 \cdot 10^3$ to $4 \cdot 10^3$. This is due to a decrease in the concentration of radicals = N-H, -N-H_2 groups until their complete disappearance and the formation

of new ones caused by electron irradiation. Such changes in the EPR spectrum reflect the transition of radicals in polyimide films from one type to another caused by the action of uniaxial stretching and electron irradiation. Such a transition is accompanied by the complete disappearance of the first type of radicals and the formation of new ones.

Taking into account such features of the connection of mechanical properties with the behavior of structural elements of polymer materials, under the influence of various external physical factors, it is possible to predict the creation of new ones and improve existing ones.

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Электронды сәулелену мен созылудың полиимидті пленкалардың макромолекулаларының модификациясына әсері

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***Аңдатпа.** Электронды сәулеленудің 40 мГр сіңірілген дозасының және бөлме температурасында үзілуге дейін бір осьті созудың Instron 5982 сынақ машинасында полиимидті пленка макромолекулаларының құрылымының өзгеруіне әсері зерттелді. Сәулелендіру бөлме температурасында ELA-6 электронды сызықтық үдеткішінде жүргізілді, ал құрылымның өзгеруі Nicolet 5700 ИҚ спектрометрінің көмегімен 4000-400 $см^{-1}$ диапазонында, Bruker фирмасының ESP 300 E спектрометрінің толқын ұзындығы 3,2 см және Instron 5982 сынау машинасында бөлме температурасында үзілуге дейін бір осьті созылу арқылы зерттелді. Жұмыстың мақсаты – электронды сәулелену және механикалық жүктеме нәтижесінде полиимидті пленкалардың макромолекулаларының құрылымындағы өзгерістерді зерттеу. Сәулеленуден кейін ИҚ спектрінің қарқындылығы ~2-6 есе, ал жолақтардың ені артатыны анықталды, бұл радикалдардың көбеюін көрсетеді. Пленканы кейіннен механикалық жүктememен сәулелендіру ЭПР спектрінің сызығын 3475,0 $см^{-1}$-ден 3512,5 $см^{-1}$-ге дейін жылжытады, сигнал амплитудасының бір уақытта $6·10^3$-тен $4·10^3$-ке дейін төмендеуімен, бұл радикалдар концентрациясының төмендеуін көрсетеді = N-H, -N-H_2 топтар толығымен жойылғанға дейін және жаңаларының пайда болуы электронды сәулеленуден туындайды.*

***Кілт сөздер:** полиимид, үлбірлер, электрондық сәулелену, ИҚ- және ЭПР-спектроскопия, жару машинасы, Instron 5982, супрамолекулалық құрылымы, механикалық қасиеттері, сіңірілген дозасы, Bruker ESP300E спектрометрі.*

Влияние электронного облучения и растяжения на модификацию макромолекул полиимидных пленок

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***Аннотация.** Изучено влияние поглощенной дозы электронного облучения 40 мГр и одноосного растяжения до разрыва при комнатной температуре на испытательной разрывной машине Instron 5982 на изменения структуры макромолекул полиимидной пленки. Облучение проводили на электронном линейном ускорителе ELA-6 при комнатной температуре, а изменение структуры изучали с помощью ИК-спектрометра Nicolet 5700 в диапазоне 4000-400 $см^{-1}$, спектрометра ESP 300 E фирмы Bruker при длине волны 3,2 см и одноосного растяжения до разрыва при комнатной температуре на испытательной разрывной машине Instron 5982. Целью данной работы было изучение изменений структуры макромолекул полиимидных пленок в результате электронного облучения и механического нагружения. Было обнаружено, что после облучения интенсивность ИК-спектра увеличивается в ~2-6 раз, а ширина полос увеличивается, что свидетельствует об увеличении содержания радикалов. Облучение пленки с последующей механической нагрузкой вызывает сдвиг линии ЭПР спектра с 3475,0 $см^{-1}$ до 3512,5 $см^{-1}$ с одновременным уменьшением амплитуды сигнала с $6 \cdot 10^3$ до $4 \cdot 10^3$, что указывает на уменьшение концентрации радикалов = N-H, -N-H_2 групп до их полного исчезновения и образования новых, вызванных электронным облучением.*

***Ключевые слова:** полиимид, пленки, электронное облучение, ИК- и ЭПР-спектроскопия, разрывная машина, Instron 5982, супрамолекулярная структура, механические свойства, поглощенная доза, спектрометр Bruker ESP300E.*

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