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STRUCTURE OF POLYTETRAFLUOROETHYLENE FILM AFFECTED BY ELECTRON RADIATION AND TEMPERATURES

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Abstract. *Introduction***.** The thermal conductivity of the polytetrafluoroethylene film in the temperature range of 80–330 K and radiation doses from 5 to 30 kGy was investigated. *Methodology*. Thermal conductivity studies were carried out at the TAU–5 thermophysical facility. *Results.* The temperature dependence of the thermal conductivity of polytetrafluoroethylene at different irradiation doses was obtained, and the resulting thermal conductivity curves demonstrate phase transitions at temperatures T_1 = 293 K and T_2 = 303 K. The irradiation of polytetrafluoroethylene films with low doses resulted in a shift of phase transition temperatures to the low temperature region, with a decrease in peak amplitude. Irradiation with a dose of D = 30 kGy resulted in a 2 % decrease in thermal conductivity λ, a significant decrease in the intensity of the phase transition peak at T_1 and a complete disappearance of the second peak at T_2. As the temperature and irradiation dose increased, there was a notable rise in interplanar distances, degree of crystallinity, and unit cell volume. Conversely, there was a reduction in crystal size, a rearrangement of the crystal structure, and a smoothing of phase transitions. *Conclusion*. It was determined that the thermal conductivity of the polymer at low temperatures is attributed to skeletal vibrations of the main chain, while at high temperatures and irradiation doses, it can be attributed not only to the rearrangement of the crystal structure, but also to the disappearance of pores and the ordering of the crystalline phase of polytetrafluoroethylene.

Keywords: thermal conductivity, polytetrafluoroethylene, electron radiation, high temperature, phase transition, crystallization, crosslinking, pores, structure

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1. Introduction

Polytetrafluoroethylene belongs to mass-consumption polymers due to its unique complex of physico-chemical properties. Polymers used in space must meet certain requirements, including, in some cases, sufficient resistance to ionizing radiation and temperature. A large number of monographs and articles have been devoted to the study of radiation resistance of various types of polymers, in particular, fluoroplastic (F–4) [1–3]. However, the issue related to the behavior of these polymers under irradiation with intense electron beams is currently insufficiently covered. The objective of this study was to examine the impact of temperature and electron irradiation on structural-phase transformations in poly(tetrafluoroethylene) (PTFE) films.

2. Methods and experiments

An industrial fluoroplastic film (F4) was used as the test material. The samples were disks, 30 mm in diameter and 50 μm thick, cut from a fluoroplastic film with a special knife. The irradiation of PTFE films was carried out on the linear electron accelerator ELU–6 at the Physics and Technology Center of Abai University. The main parameters of the accelerated electron beam at the accelerator output are: maximum electron energy – E = 2 MeV, pulse current – I = 0.3 mA / cm^2, pulse duration – 10 ns, repetition rate – 400 Hz, at irradiation doses D = 5, 10 and 30 kGy. The research objects were installed at a distance of 30 cm from the exit window of the accelerator. The irradiation temperature was T = 25 ° C. X-ray diffractometric studies were performed on an automatic Bruker D8 Advance diffractometer with digital recording of measurement results. The study of the structures of the initial and irradiated films was carried out on an Infrared spectrophotometer with a Fourier converter "FTIR Mattson Satellite 3000" in the frequency range 4000 – 400 cm^{-1}, on the XP-146JB optical microscope and on the Quanta 3D 200i scanning electron microscope in the nanolaboratory of the engineering profile of Al-Farabi Kazakh National University.

3. Results and Discussion

The temperature dependence of the thermal conductivity of polytetrafluoroethylene at different radiation doses was obtained (see Fig.1). Phase transitions at temperatures T_1 = 293 K and T_2 = 303 K were revealed on the curves of the dependence of thermal conductivity. Irradiation of polytetrafluoroethylene films with small doses led to a shift in the temperatures of phase transitions to the low temperature range by 1 K for a dose of D = 5 kGy and by 13 K at D = 10 kGy and a decrease in peak amplitude. The irradiation of polytetrafluoroethylene films with low doses resulted in a shift of phase transition temperatures to the low temperature region, with a decrease in peak amplitude. Irradiation with a dose of D = 30 kGy resulted in a 2 % decrease in thermal conductivity λ, a significant decrease in the intensity of the phase transition peak at T_1 and a complete disappearance of the second peak at T_2 [6].

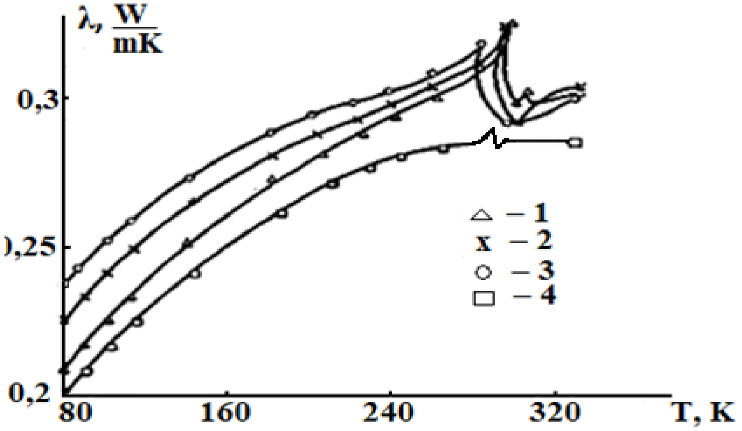


Figure 1 - Experimental curves of temperature dependence of PTFE thermal conductivity at irradiation doses D = 5, 10 and 30 kGy

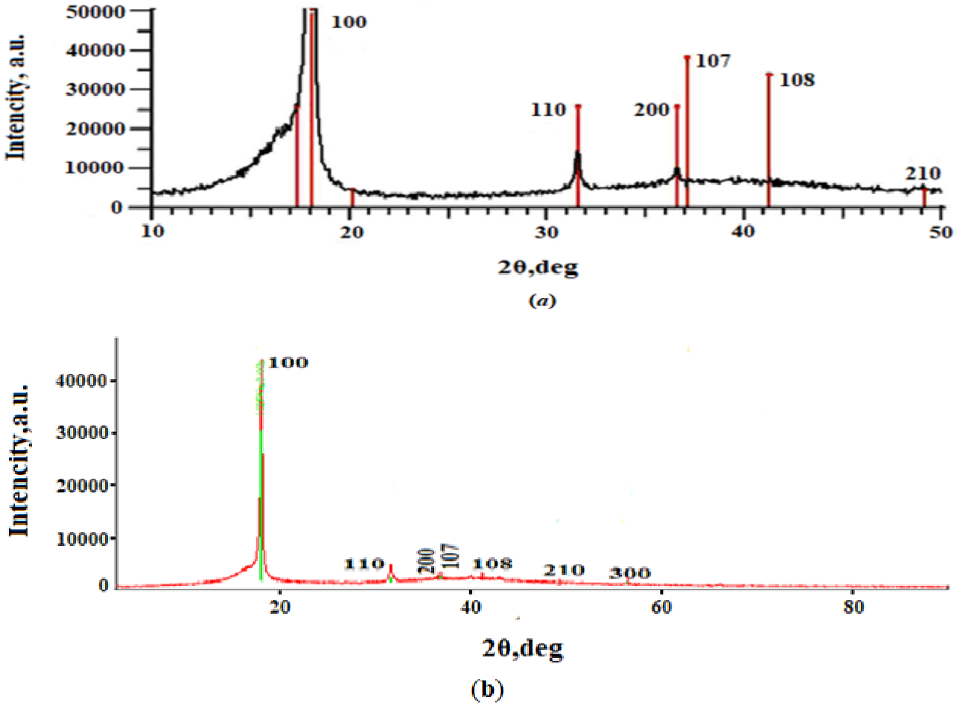
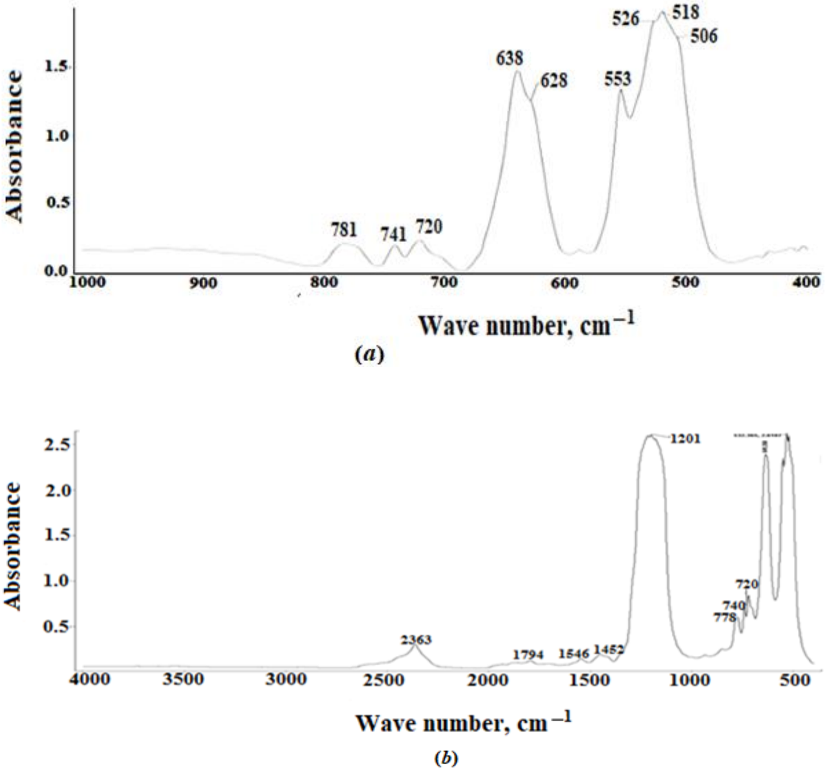


Figure 2 - Diffractogram of a fluoroplastic film (a) at a temperature of 15 ° C (b) at a dose of 30 kGy

Figure 2, a, b shows diffractograms of fluoroplastic film obtained at temperature of 15^0 C and irradiation with a dose of 30 kGy. All diffractograms contain a diffraction maximum at $2\theta = 18^0$ with index *hkl* = 100, which dominates in the spectra of the original polymer and the irradiated one; besides this line, a broad diffuse halo is observed. In addition to the appearance of a number of additional reflections splitting with index 100 and a superposition of lines 200 and 107 are observed at 278 K [4]. As thermal conductivity studies have shown [6], the most important structural changes occur at the temperatures of phase transitions. We studied how fluoroplastic changed using IR spectroscopy.

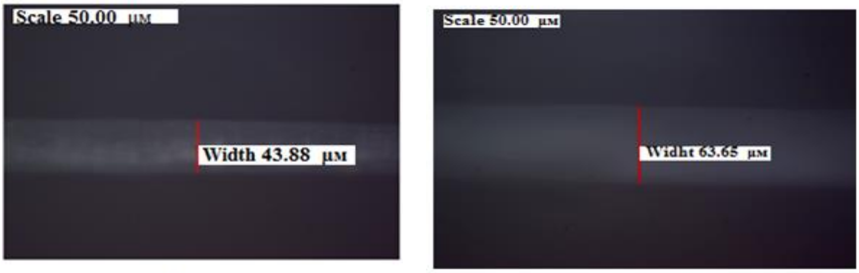


(*a*)

(*b*)

Figure 3 - IR spectra of PTFE a) initial and b) irradiated with electrons at a dose of 30 kGy at a temperature of T= 19 °C

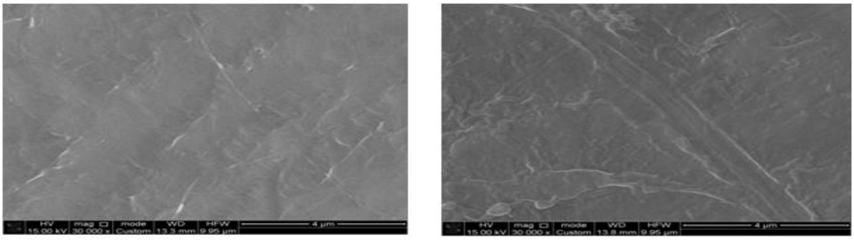
We looked at frequencies between 1000 and 400 cm^{-1} (Fig. 3). Fig. 3 shows the IR spectrum of PTFE is simple. This is due to the simple structure of its polymer chains. The IR spectrum of the original PTFE (Fig. 3a) at 19^o C shows intense absorption bands at 518, 553 and a doublet at 638 cm^{-1}, as well as three weaker bands at 720, 740 and 778 cm^{-1}. In addition to the above bands, two other bands at 526 and 506 cm^{-1} are observed as a shoulder at 518 cm^{-1}. A peculiarity of the IR spectra of this frequency interval is the appearance of the 628 cm^{-1} band of the doublet component at the 638 cm^{-1} band, expressed as a shoulder, and additional bands at 526 and 506 cm^{-1} of low intensity at the 518 cm^{-1} band. As illustrated in Figure 3b, a broad and intense band at 1201 cm^{-1} emerges in the infrared (IR) spectrum of irradiated polytetrafluoroethylene (PTFE) within the frequency range of 4000–500 cm^{-1}. The most intense bands are related to the valence vibrations of the CF_2 groups (1211 and 1154 cm^{-1}) and the ν(CC) vibration, which appears as a bend at ~1233 cm^{-1}. The thicknesses of the original and irradiated PTFE films measured with an optical microscope are shown in Figure 4.



(*a*) (*b*)

Figure 4– Micrographs of PTFE films a) initial and b) irradiated with electron at a dose of 30 kGy

The micrographs clearly show that irradiation results in a twofold increase in film thickness. Scanning electron microscope examination of the chipping of the original and irradiated PTFE films is shown in Figure 5.

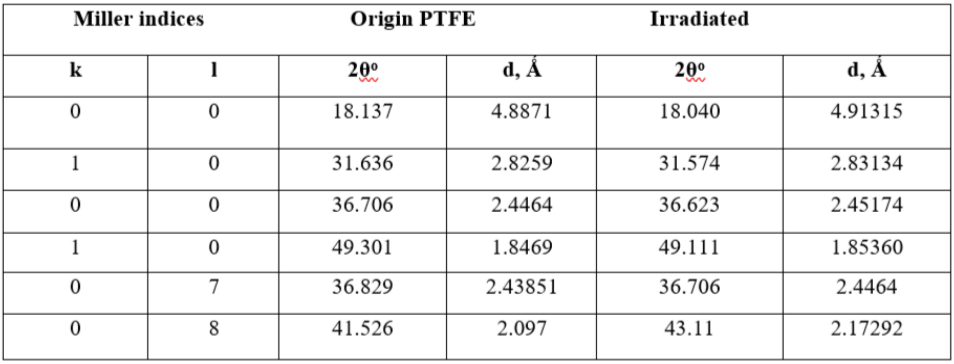


(*a*) (**b**)

Figure 5– SEM images of PTFE films a) initial and b) irradiated with electrons at a dose of 30 kGy

The SEM images show that there is a significant change in the morphology of the polymer matrix. With increasing irradiation dose, a significant disruption of the polymer crystal structure is observed with the appearance of individual irregularly shaped formations. Additionally, the matrix pores are clearly visible on the chipping surfaces of the studied samples. The crystalline region is represented by lamellae (plates) and consists of folded polymer structures separated by interfacial boundaries. The amorphous (disordered) region of PTFE is situated between crystallites and comprises randomly oriented and traversable polymer chains that connect the lamellae (Figure 5b). The linear increase of the thermal conductivity λ with temperature can be explained on the basis of the two-phase model proposed in [6].

Table1 - Characteristics of X-ray diffraction spectra of initial and electron-irradiated polytetrafluoroethylene samples



Miller indices		Origin PTFE		Irradiated	
k	l	2θ°	d, Å	2θ°	d, Å
0	0	18.137	4.8871	18.040	4.91315
1	0	31.636	2.8259	31.574	2.83134
0	0	36.706	2.4464	36.623	2.45174
1	0	49.301	1.8469	49.111	1.85360
0	7	36.829	2.43851	36.706	2.4464
0	8	41.526	2.097	43.11	2.17292

Since the polymer is considered in terms of the vibrational motion of a segment rather than the molecule as a whole, the vibrational motion (mobility) of the segments is retarded with decreasing temperature. The value of λ for the crystalline phase increases with increasing temperature [7] because for complex crystals, such as PTFE crystals, the phonon free path length is comparable to that of the amorphous phase. Since $\lambda_k > \lambda_a$, the λ values for PTFE should increase with increasing degree of crystallinity, which is consistent with our data. The decrease of λ with irradiation up to dose D = 30 kGy is probably due to the destruction processes leading to a disordered PTFE structure. Irradiation of PTFE by electrons at 30 kGy (Fig. 2b) shifted all diffraction maxima toward smaller angles (Table 1). The irradiated film's diffraction maximum at 2θ = 18^0, *hkl* = 100, is 10% smaller than the original. The shift and decrease in the diffraction maximum at 2θ = 18^0 are due to the diffraction maximum *hkl* = 100 consisting of two components with different interplanar distances [4]. An irradiation result in an increase in the interplanar distances (d_{hkl}) of both types of PTFE crystals, yet the extent of this increase differs between them. The expansion of the unit cell volume in irradiated polytetrafluoroethylene (PTFE) is accompanied by a reduction in the density, ρ_c, despite the enhancement in the degree of crystallinity of PTFE.

This conclusion is corroborated by the data obtained through the measurement of the film thickness, which is illustrated in Figure 4. PTFE macromolecules in the amorphous phase have greater mobility and experience greater thermal expansion. This is confirmed by the fact that as the temperature increases from 20 to 320^{o}C, the molecular packing factor decreases from 0.66 to 0.60 in the crystalline region and from 0.58 to 0.46 in the amorphous region [10]. Therefore, the crystalline phase of PTFE contains crystals in both hexagonal and triclinic conformations. Triclinic conformation crystals in the crystalline phase of

PTFE define its unit cell at 15°C. This increases PTFE density. PTFE-based composites filled with silica particles show that PTFE phase transitions are "smoothed out"[4]. It can be inferred that the crystalline phase of PTFE at room temperature may consist of both "triclinic" and "hexagonal" crystals, which could potentially give rise to two components of the observed diffraction maximum *hkl* = 100 in the X-ray spectra of PTFE. Consequently, the irradiation of PTFE can result in a reduction in the quantity of triclinic crystals and an increase in the proportion of hexagonal crystals within the crystalline phase of the polymer. This is corroborated by the correlation between thermal conductivity and temperature and irradiation, as illustrated in Figure 1, as well as by scanning electron microscopy (SEM) images, as depicted in Figure 5. The formation of the halo can be attributed to the phenomenon of intramolecular scattering, which occurs when disordered polymer chains exhibit a conformation that is similar to that observed in PTFE crystals It is presumed that these macromolecules are present within the smectic phase of the polymer [10]. The halo's spectral shape can be approximated by two Gaussians, indicating two noncrystalline components in the smectic phase of PTFE. The first contains partially ordered macromolecules and the second contains hexagons. [4,10]. Analysis of the IR spectrum of the initial film in the range of 1000–400 cm^{-1} showed that the bands 518 and 553 cm^{-1}, according to [5], characterize the deformation and pendulum vibrations of CF_2 groups, respectively. The IR spectrum shows a well-defined doublet at 638 and 628 cm^{-1} (see Fig. 3), which are characterized as γω (CF_2) fan vibrations. The bands at 638, 628 and 518 cm^{-1}are considered to be ordering bands and crystallinity bands [5]. The bands at 720, 741 and 781 cm^{-1} characterize the amorphous phase of PTFE. The appearance of the doublet band at 628 cm^{-1} and the triplet bands at 524, 526 and 506 cm^{-1}in the IR spectra of PTFE at 638 and 518 cm^{-1}is related to the fact that these absorption bands represent phase transitions observed in PTFE at room temperature. The change in the amplitude of the bands at 628 cm^{-1} and 638 cm^{-1} is due to changes in the shape of macromolecules. It is assumed that the band at 628 cm^{-1}characterizes the defectivity of the structure, i.e., chain regions where transitions between left- and right-handed helices occur, and the band at 638 cm^{-1} reflects the presence of a regular helix in the structure of polytetrafluoroethylene. Analysis of the IR spectrum of the irradiated sample of polytetrafluoroethylene in the region below 1000 cm^{-1}shows that the bands characterizing valence vibrations of C–F and C–C, deformation vibrations of CCC and CF_2 groups, out-of-plane vibrations of CF_2 groups, individual fragments and the whole chain [5,7,10] are preserved even at high irradiation doses. This indicates that external irradiation does not lead to complete destruction of the PTFE molecular chain. The most intense band at 1201 cm^{-1}, which appears as a bend, belongs to the valence vibrations of CF_2 groups and ν(CC) vibrations. Moreover, the IR spectrum of irradiated PTFE samples exhibits a band at 1794 cm^{-1}, which is attributed to the vibrations of $–CF=CF_2$ end groups according to quantum chemical calculations. On this basis, it was concluded that the number of these groups is sufficient to register the band corresponding to them, indicating the presence of shorter macromolecules in the structure of the samples than in the original PTFE. The IR spectra of samples irradiated at high doses show bands at 1546 and 1452 cm^{-1}. According to quantum chemical calculations [5], they can be attributed to the –CF = CF– (1546 cm^{-1}) and – CF= C < (1452 cm^{-1}) vibrations. In the IR spectra of air-irradiated PTFE samples at high doses (~ 30kGy), a band at 2363 cm^{-1}appears in addition to the band at 1794 cm^{-1}[8, 9]. It is natural to assume that at high irradiation doses the interaction of formed radicals with air oxygen occurs, especially in the presence of water vapor. The band at 2363 $cm^{-1}$is attributed to vibrations of – COOH – groups [5]. The analysis of PTFE film chips, shown in Figure 5, based on X-ray diffraction studies, shows that at high irradiation doses the transition from the helical structure of polytetrafluoroethylene to the planar structure is realized. The process of irradiation of PTFE has been observed to result in a reduction in the quantity of triclinic crystals present within the crystalline phase of the polymer, accompanied by an increase in the proportion of hexagonal crystals.

4. Conclusion

The study of the changes in thermal conductivity of PTFE film induced by temperature and electron irradiation led to the following conclusions:

1. At low temperatures, the phonon free path length increases due to the retardation of segment motion. Heat transfer in PTFE is due to skeletal vibrations of the main chain of PTFE. In this temperature range, the thermal conductivity varies linearly with temperature.

2. An increase in temperature leads to a smooth behavior initially and then to an abrupt change in thermal conductivity at 19^0C. The smooth behavior of the thermal conductivity is due to the unfreezing of the segmental mobility of the polymer chain, and the abrupt one is caused by a phase transition of the first type, namely the rearrangement of the crystalline triclinic conformation into a hexagonal conformation.

3. Above the temperature of 19^0C, the value of the thermal conductivity returns to the initial value, which is due to the ordering of the crystal structure.

4. Low dose irradiation of polytetrafluoroethylene results in an increase in thermal conductivity associated with an increase in crystallinity.

5. An increase in the irradiation dose results in a reduction in thermal conductivity and the cessation of phase transitions associated with alterations in the polymer structure. These changes are not solely the result of bond rupture, the formation of pores, and the generation of short macromolecular chains; they also stem from the emergence of branched structures and the crossing of chains. The absence of phase transitions can be attributed to the "smoothing" of phase transitions during irradiation, which results in a reduction in the triclinic crystal content and an increase in hexagonal crystal content within the polymer's crystalline phase.

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Conflict of interests: The authors declare that there is no conflict of interest between the authors that requires disclosure in this article.

ЭЛЕКТРОНДЫ СӘУЛЕЛЕНУ МЕН ТЕМПЕРАТУРАҒА ҰШЫРАҒАН ПОЛИТЕТРАФТОРЭТИЛЕННІҢ ҚАБЫҚШАНЫҢ ҚҰРЫЛЫМЫ

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Түйіндеме. *Кіріспе.* Зерттеу жұмысында 80-330 К температура аралығындағы политетрафторэтилен пленканың жылу өткізгіштігі және 5-тен 30 кГр-ға дейінгі сәулелену дозалары зерттелді. *Әдістеме.* Жылу өткізгіштігін зерттеу ТАУ-5 жылу-физикалық қондырғысында жүргізілді. *Нәтижелер және талқылау.* Әр түрлі сәулелену дозаларында политетрафторэтиленнің жылу өткізгіштігінің температураға тәуелділігі алынды. Жылу өткізгіштікке тәуелділік қисықтарында T_1 = 293К және T_2 =303 К температураларында фазалық ауысулар анықталды. D = 30 кГр дозасымен сәулелену λ жылу өткізгіштігінің 2% - ға төмендеуіне, T_1-де фазалық ауысу шыңының қарқындылығының айтарлықтай төмендеуіне және T_2 -де екіншісінің толық жоғалуына әкелді. Температура мен сәулелену дозасының жоғарылауы планетааралық арақашықтықтың, кристалдық дәреженің және бірлік ұяшығының көлемінің ұлғаюына, сондай-ақ кристалдар мөлшерінің азаюына, кристалдық құрылымның қайта құрылуына және фазалық ауысулардың тегістелуіне әкеледі. *Қорытынды.* Төмен температурада полимердің жылу өткізгіштігі негізгі тізбектің қаңқа тербелістеріне байланысты екендігі анықталды, ал жоғары температура мен сәулелену дозаларында кристалдық құрылымның қайта құрылуына ғана емес, сонымен қатар кеуектердің жоғалуына және политетрафторэтиленнің кристалдық фазасының реттелуіне байланысты болуы мүмкін.

Түйін сөздер: Жылу өткізгіштік; политетрафторэтилен; электронды сәулелену; жоғары температура; фазалық ауысу; кристалдану; айқаспалы байланыс; кеуектер; құрылым

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СТРУКТУРА ПЛЕНКИ ПОЛИТЕТРАФТОРЭТИЛЕНА, ПОДВЕРЖЕННАЯ ВОЗДЕЙСТВИЮ ЭЛЕКТРОННОГО ИЗЛУЧЕНИЯ И ТЕМПЕРАТУР

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Резюме. *Введение* В работе исследована теплопроводность пленки политетрафторэтилена в интервале температур 80-330 К и доз облучения от 5 до 30 кГр. *Методика*. Исследования теплопроводности проводили на теплофизической установке ТАУ-5. *Результаты и обсуждение*. Получена температурная зависимость теплопроводности политетрафторэтилена при различных дозах облучения. На кривых зависимости теплопроводности выявлены фазовые переходы при температурах T_1 = 293К и T_2 =303 К. Облучение дозой D = 30 кГр привело к уменьшению теплопроводности λ на 2 %, к значительному снижению интенсивности пика фазового перехода при T_1 и полному исчезновению второго при T_2. Повышение температуры и дозы облучения приводят к увеличению межплоскостных расстояний, степени кристалличности и объема элементарной ячейки, а также к уменьшению размера кристаллов, перестройке кристаллической структуры и сглаживанию фазовых переходов. *Заключение*. Установлено, что теплопроводность полимера при низких температурах обусловлена скелетными колебаниями основной цепи, а при больших температурах и дозах облучения могут быть обусловлены не только перестройкой кристаллической структуры, но также исчезновением пор и упорядочением кристаллической фазы политетрафторэтилена.

Ключевые слова. Теплопроводность; политетрафторэтилен; электронное излучение; высокие температуры; фазовый переход; кристаллизация; сшивание; поры; структура

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