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The influence of ultraviolet radiation on electrical relaxation processes within the temperature area of stained glass films of PET

I. A. Fedorov [1], N. P. Bebenin [1], A. V. Stepchenkova [1], E. A. Volgina [1], D. E. Temnov ✉[1]

[1] Herzen State Pedagogical University of Russia, 48 Moika Emb., Saint Petersburg 191186, Russia

Authors

Ivan A. Fedorov, e-mail: ivasispb@yandex.ru

Nikita P. Bebenin, e-mail: nikita2001bebenin@gmail.com

Anastasiya V. Stepchenkova, e-mail: nastyastep7@icloud.com

Elena A. Volgina, ORCID: 0000-0002-1536-5841, e-mail: volgina.elena.1999@mail.ru

Dmitry E. Temnov, ORCID: 0000-0002-9560-4346, e-mail: detem@yandex.ru

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Abstract. The electrical relaxation processes observed in poly(ethylene terephthalate) were investigated using thermal activation spectroscopy. In the area of glass transition temperature of this polymer, two relaxation processes were detected — in the area of 60°C and 80°C. Ultraviolet radiation caused a significant change in the intensity of these processes. Relaxation around 60 °C is related to the glass transition process in the amorphous phase of poly(ethylene terephthalate). Relaxation in the region of 80 °C may be related to a part of the amorphous phase at the surface of crystals (rigid amorphous fraction) characterized by reduced mobility of molecular segments. After UV irradiation of this polymer, the relative content of this fraction increases substantially.

Keywords: poly(ethylene terephthalate), thermostimulated depolarization, glass transition temperature, UV radiation, electrical relaxation

Introduction

Poly(ethylene terephthalate) (PET) is one of the most widely used thermoplastic polymers due to its combination of mechanical, thermal and dielectric properties (Dhaka et al. 2022; Joseph et al. 2024). PET has applications in food and beverage packaging (Benyathiar et al. 2022), textile fibers (Sarioğlu, Kaynak 2017), optical and electronic films, as well as substrates for flexible electronics and energy-harvesting devices (Yakimets et al. 2010). Its stability in use, low cost and technological versatility are the reason for constant interest in studying the structure, relaxation mechanisms and durability of the material under the influence of external factors.

PET crystallization is accompanied by the formation of not only an ordered crystal phase and amorphous phase but also a limited amorphous phase with reduced segment mobility at the surface of the crystals, resulting in a three-component structure: crystalline, amorphous fraction and rigid amorphous fraction (RAF) (Heidrich, Gehde 2022). This morphology affects the segmental mobility of molecules in the amorphous phase, since molecular segments closer to the surface of the crystals have greater limitations compared to those in freer amorphous regions. This heterogeneity is clearly evident

in relaxation processes: in fully amorphous α-relaxation polymers, the glasswork-associated process can be regarded as relatively uniform; in semi-crystalline PET, relaxation dynamics are spatially heterogeneous and reflect the complex interaction between amorphous and crystalline phases through local motion distribution and relaxation times (Alves et al. 2002; Wei et al. 1994).

Such heterogeneity makes relaxation processes in PET particularly sensitive to external factors that can modify the local mobility of macromolecules and the chemical structure of chains. In particular, ultraviolet exposure leads to photo-oxidative degradation of PET, accompanied by chain rupture, forming carbonyl and hydroxyl groups (Rostampour et al. 2024). These processes mainly develop in the amorphous phase and near the interphase regions, which allows one to expect a significant influence of UV radiation on glazing and α-relaxation parameters in semi-crystalline PET.

Despite the large number of articles and researches dealing with the photodegradation of PET and the modification of its mechanical and optical properties by UV radiation, the influence of UV radiation on glass processes and segmental relaxation in semi-crystalline PET remains understudied. This determines the relevance of this work, aimed at studying the influence of UV-effects on relaxation processes in PET.

Methods and materials

Materials

The samples were made from Hostaphan RNK PTP 13-μm thickness film produced by Mitsubishi Polyester Films. Ultraviolet radiation was carried out in the air using LE30 lamps manufactured by Lisma (Saransk, Russia).

Methods

The TSC-II system of Setaram (France) was used to measure thermal depolarization currents (TDC). The process of measuring the depolarization currents of the samples under investigation involved several sequential procedures. In the electric field at E = 90 V/min over 2 min, the samples were polarized at a polarization temperature of T_p = 60 °C. Next, in the electric field, the film was cooled at a rate of 2 °C/min to a temperature of T_0 = −10 °C and then held for t = 1 min. The specimen was then heated at a rate of 9°C/min to a temperature of T_f = 110 °C. The current was recorded with a Keithley 6517E electrometer with a measurement accuracy of 10^{-14} A. All measurements were carried out in a vacuum chamber in the atmosphere of helium gas.

When there are complex overlapping processes, standard thermal current peak processing methods such as the Buchi method, heating rate variation method and initial rise method produce incorrect results. The optimization method described in the article was used to process data in this work (Volgina et al. 2026).

Results and discussion

Fig. 1 shows the TSC curves of unirradiated UV radiation from the PET film, polarized at 60 °C in an electric field of different voltages. The practically linear dependence of the current value on the electric field voltage indicates the dipole mechanism of relaxation processes occurring at this temperature interval.

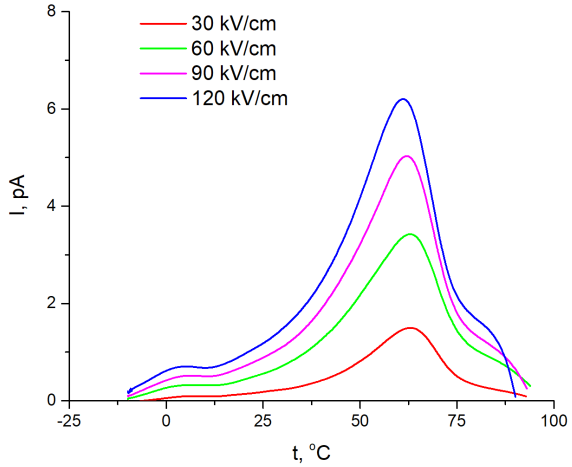

Fig. 1. The dependence of TSC for the original (unirradiated UV) PET film at different polarizing field values. Polarization temperature — 60 °C, heating rate — 9 °C/min

The data obtained clearly shows that the TSC curve is complex, indicating that several overlapping processes occur in the given temperature range as shown in Fig. 2(a). The following relaxations can be distinguished: in the areas of 10 °C (1), 40 °C (2), 60 °C (3) and 80 °C (4).

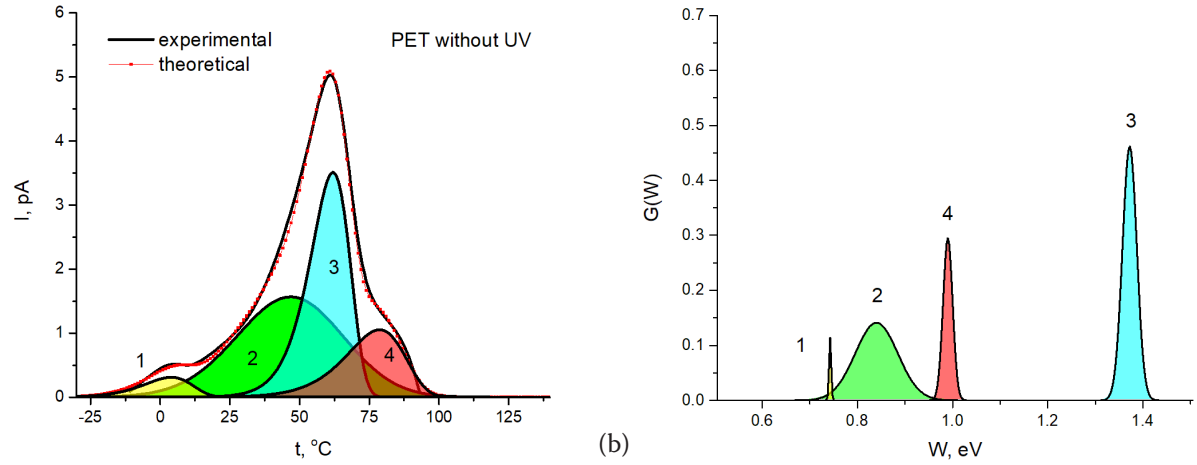

Fig. 2. a) Decomposition of the TSC curve for unirradiated PET polarized in the field 90 kV/cm at 60 °C to separate relaxation processes. b) The relaxer distribution functions for unirradiated UV polyethylene terephthalate processes as shown in Fig. 2(a)

At temperatures below the glass transition temperature T_g = 60 °C in a PET, only relaxation processes associated with the movement of macromolecular segments can occur (process 1). This is due to the fact that at temperatures below T_g the polymer is in a glass state, where the mobility of the chains is severely limited. As the temperature rises, different types of molecular motion begin to be activated: rotation of lateral groups, oscillatory movement of segments of the main chain and movement of entire macromolecule sections (process 2). At temperatures close to 60 °C, the TSC spectra show a peak corresponding to the process of glazing the amorphous part of the polymer (process 3). As previously mentioned, the presence of a crystalline part leads to the appearance of an amorphous part with limited segmental mobility and, as a consequence, to the emergence of a second relaxation process in the TSC currents associated with the glass transition process (process 4).

Fig. 2(b) presents the power distribution functions of G (W) relaxers involved in all the observed processes at temperatures ranging from -10 °C to 100 °C. The 'red' curve in Fig. 2(a) corresponds to the theoretical TSC curve calculated according to formula (1) and corresponding distribution functions in Fig. 2(b).

$$j(T) \sim \sum_{i}^{4} \int_{0}^{W_m} G_i(W) \exp\left[-\frac{W}{kT} - \int_{T_0}^{T} \frac{\omega_i}{\beta} \exp\left(-\frac{W}{kT'} \right) dT' \right] dW . \quad (1)$$

The high activation energy (W) for process 3 compared to process 4 is due to higher frequency factor values ($\omega_3 > \omega_4$).

Fig. 3 shows the TSC spectrum after the exposure of the samples to ultraviolet radiation from an unirradiated sample.

As is known, photo-oxidative degradation processes occur in PET due to the influence of UV radiation and are mainly accompanied by chain scission of macromolecules. This leads to increased mobility of the chain segments in the amorphous phase. The resulting shorter chains and fragments of macromolecules have an increased capacity for conformational relaxation. As a result, they can be regrouped and arranged in interlamellar regions, as well as near the crystal line, which is accompanied by a local increase in the degree of crystal personality.

According to the work (Falkenstein et al. 2020) performed using ATR-FTIR, NMR spectroscopy and enzymatic degradation analysis, UV treatment leads to the formation of a surface layer characterized by increased organization and relative increase in the proportion of the crystalline phase. This is related to photoinductive chain decompression and subsequent structural reorganization of the superficial regions.

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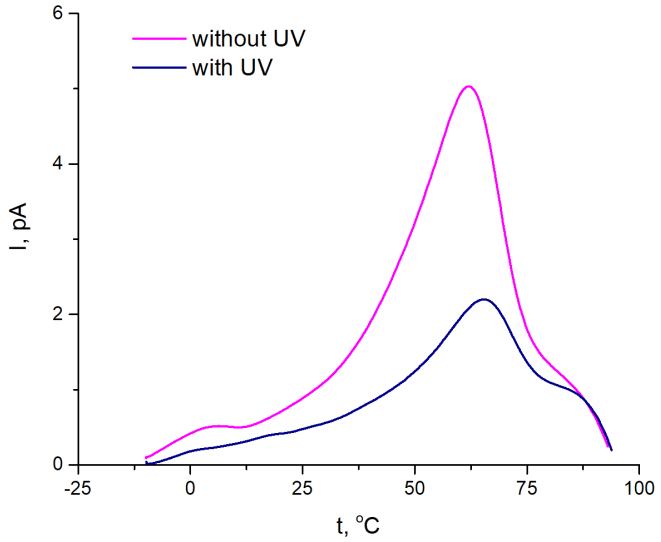

Fig. 3. TSC current dependence for the unirradiated and irradiated UV film of PET at 90 kV/cm polarizing field stress. Polarization temperature — 60 °C, heating rate — 9 °C/min

UV irradiation can lead to the redistribution of the phase composition of semi-crystalline PET: changing the ratio between a movable amorphous fraction, rigid amorphous fraction (RAF) and crystal phase, which potentially affects glazing parameters and α-relaxation characteristics.

Fig. 4(b) shows the power distribution functions of G (W) relaxants involved in all the observed processes at a temperature range from -10 °C to 100 °C. The 'red' curve in Fig. 4(a) corresponds to the theoretical TSC curve calculated according to formula (1) and corresponding distribution functions in Fig. 4b.

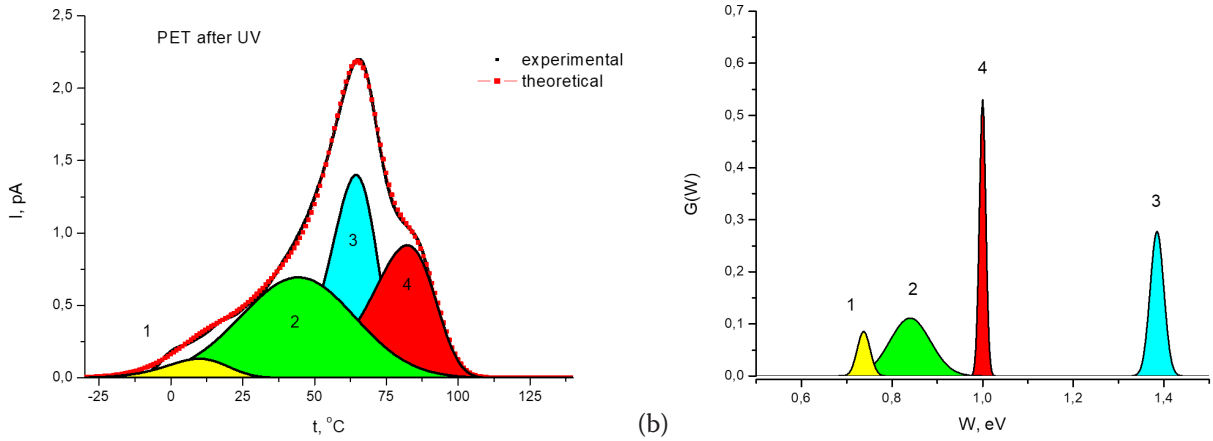

Fig. 4. a) Decomposition of the TDD curve for the irradiated UV PTEF polarized in the field E = 90 kV/cm at 60 °C for individual relaxation processes. b) The relaxer distribution functions for unirradiated UV polyethylene terephthalate processes as shown in Fig. 4(a)

For an unirradiated sample, the ratio between processes 4 and 3 is 4/3, whereas after UV irradiation, it decreases to 3/5. Therefore, the relative contribution of process 3 increases compared to process 4, indicating an increase in the RAF share in the irradiated sample.

Conclusion

Our research employed the method of thermally stimulated depolarization to study UV-irradiated and unirradiated PET films. The spectrum of TSC in the area 0 °C–100 °C demonstrates a complex character due to closely located overlapping relaxation processes. Relaxation processes in the area of 10 °C and 40 °C are observed below the glass transition temperature of this polymer and can only be related

to the movement of individual segments of macromolecules. Relaxation in the area of 60 °C and 80 °C is related to the processes of glass transition developing in the amorphous phase of the polymer and the so-called rigid amorphous fraction, which is on the border with available crystallites of the polymer. UV radiation of this polymer leads to a significant increase in the content of the limited amorphous phase, which indicates a local increase in the degree of crystallization of the polymer in the superficial regions. The functions of distribution of electrically active defects for all the relaxation processes were found by the optimization method.

Conflict of Interest

The authors declare that there is no conflict of interest, either existing or potential.

Author Contributions

All the authors discussed the final work and took part in writing the article.

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