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Radiation-Induced Modification of Structure and One-Electron Elementary Excitations of C_{60} Fullerene Films

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The influence of the beyond threshold electron irradiation ($E_e = 1.8$ MeV) on the processes of the destruction of C_{60} fullerenes due to their polymerization and the amorphization is studied. The crystal structure, vibrational spectra, photoluminescence, interband molecular electron transitions for the condensed state of C_{60} fullerene with different fluences are investigated. It is shown that the electron irradiation with an increase in the radiation dose differently affects the appearance of polymerization and amorphization with their simultaneous taking in C_{60} fullerite. The creation of radiation defects and polymerized structures it is necessary to consider as the formation of local capture centers of charge carriers and excitons, which introduce the changes in the electronic states both in the energy gap and beyond its limits.

Keywords: C_{60} fullerite, electron irradiation, elementary electronic excitations, polymerization and amorphization, Raman scattering, photoluminescence, optical conductivity.

INTRODUCTION

The unique special features of the fullerene structure predetermine the presence of the uncommon properties caused by the intermediate state of sp-hybridization as a result of the bending of graphite surface [1]. The spectra of the elementary excitations of fullerites, which are the organic semiconductor molecular crystals, testify about the presence not only π, $\pi + \sigma$ collective vibrational and one-electron states, but also the local deep levels in the gap of the forbidden energies [2 – 5]. The presence of these deep traps, which are the capture centers of charge carriers and facilitate the recombination of excitons, is connected with the defects of crystal lattice [6 – 8]. It is obvious that changing the defective structure of fullerite, it is possible to influence the distribution of local levels inside the gap of the forbidden energies and, correspondingly, on the capture of the charge carriers and excitons. However, in spite of the simple determination of existence of local capture centers, their nature frequently proves to be unknown. Therefore it is important to establish the correspondence between the given types of defects and obtained values of energies of local and one-electron excitations. In this connection the creation of radiation defects under the action of the ionizing irradiation is of interest. Despite the fact that the mechanisms of the formation of radiation defects differ significantly from the type of the bombarding particles, the selection of sub- or beyond the threshold energies, and also the nature of the substance being investigated, the number of such mechanisms is limited in the specific cases of radiation exposure. During the studies of radiation-stimulated phenomena, which influence the kinetics of activation processes, it is necessary to consider the energy loss on the formation of defects with respect to the percussion mechanism, the long-life electronic excitations, and also the microscopic imbalance, caused by the deviation of the distribution function of atoms on the vibrational energy from the thermodynamically equilibrium one [9]. An especially essential effect of the presence of the unbalanced distribution function has to the stationary concentration of point defects under the beyond threshold influences in the case of temperature close to the room one.

It should be noted that the creation of elementary excitations during irradiation is accompanied by phase transformations in the structure of fullerite. With the small radiation doses the intermediate metastable phase of low-molecular, in essence of dimeric $(C_{60})_2$ polymers appears, which with an increase in the fluence is capable of being converted into the rhombic, tetragonal, rhombohedral polymeric compounds or their mixtures [1]. Besides the polymerization with the carbon atom displacement from the shell of fullerenes or their ionization it is possible the amorphization and the fragmentation of molecules [10 – 12]. In the case of ion irradiation the fraction of polymeric phase is characterized by the presence of maximum with an increase in the radiation dose, that is the consequence of one-way transformation of the polymer-amorphous phase. The share of amorphous structure continuously increases with the growth of fluence [10]. At the same time one should consider that the ion irradiation

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significantly affects the X-ray subsystem of the crystal through the creation of the cascades of atomic displacements and therefore under its influence the fragmentation of the fullerene molecules during the dissociation and amorphization of crystals are more noticeable. The role of electron irradiation in the creation of radiation defects and their influence on the phase transformations, the electronic excitations with the formation of local capture centers of the charge carriers and excitons in the C_{60} fullerite films is studied insufficiently, although it is known that even in the case of subthreshold energies of electrons the stimulation of the transformation of electronic and plasma structures occurs [2 – 4]. With an increase in the radiation dose with the energy of primary electrons (500 – 1200) eV the decrease of π-electrons concentration and an increase in the dose of sp^3-hybridized electrons is observed, that is caused by the formation of covalent intermolecular bonds and, as a result, by the polymerization of fullerites. It is obvious that with the beyond threshold energies of electrons the mechanisms of interaction of the bombarding particles with the C_{60} molecules will be more complex.

The purpose of this paper was to study the presence of vibrational transformations in the case of irradiation by the fast (beyond threshold) electrons (E_e = 1.8 MeV) and their manifestations in the spectra of one-electron elementary excitations for the C_{60} fullerite films depending on the dose of absorption.

EXPERIMENTAL

The condensed state of fullerite was obtained by putting of the C_{60} molecule layers during their evaporation in the vacuum from the sublimation camera to the clean surface of silicon with a crystallographic (100) orientation at room temperature [13]. Sublimation temperature was 750 K. Silicon surface was cleaned preliminarily by the argon ions. Sublimation camera together with the powder of the C_{60} microcrystals was degassed by prolonged annealing at 500 K. Thickness of layers was 2.0 μm and determined by quantum oscillator.

Crystal structure was studied by the method of crystalline diffraction with the use of the automated installation DRON-3M. The photoluminescence spectra were investigated at the temperatures of liquid helium and nitrogen with the use of an argon laser (λ = 514.5 nm) and a monochromator MDR-3. Raman spectra were studied by the X-ray spectrometer with the use of a line 514.5 nm of argon laser [14]. The resolution of spectrometer was 2 cm^{-1}. The laser power did not exceed the value of 0.5 W/cm^2.

The optical constants such as the real part of refractive index, the extinction coefficient, and also the principal angle of incidence were determined by the method of zero-ellipsometry with the application of a laser ellipsometer LEF-3M. Furthermore, using an incandescent lamp with the nonmonochromatic radiation and the nonzero method of Bittie-Kohn, the spectral dependences of optical constants, and also the real and imaginary parts of the dielectric constant, the optical conductivity were measured.

Irradiation at a temperature of 220 K was carried out using a linear electron accelerator ILU-6. The dose of absorption is varied from 1 MGy to 16 MGy.

RESULTS AND DISCUSSION

Since in the case of the C_{60} fullerite polymerization, the creation of covalent molecular bonds can be achieved along one of the directions or in the plane, the nature of the distortions of initial fcc lattice will be different. As it was noted, in the first case on the fcc phase basis the orthorhombic structure is formed. In the case of the exchange interactions in (100) plane the fcc phase transfers to the tetragonal structure, and when the covalent bond between the molecules in (111) plane appears, the transformation to the rhombohedral structure takes place. Depending on the conversion of fcc phase to one of the above structures, the nature of splitting of different interference lines will differ, that contributes to the determination of the polymerized phases. Fig. 1 gives the diffraction pattern of the nearest interference lines for the initial C_{60} fullerite film and irradiated by electrons one with different doses of absorption.

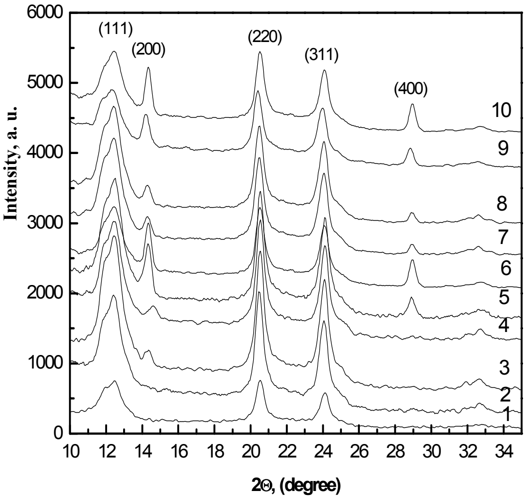

Fig. 1. X-ray diffraction patterns of the nearest interference reflections for the C_{60} films after electron irradiation with different doses of the absorption: 1 – initial (nonirradiated) sample; 2 – 1.5; 3 – 3.0; 4 – 4.0; 5 – 6.0; 7 – 10.0; 8 – 12.0; 9 – 13.0; 10 – 16.0 MGy ($d \approx 2.0$ μm, Si(100), E_e = 1.8 MeV, λ_{Co} = 1.7902 Å)

The attention should be focused on the fact that with an increase in the dose of absorption the reflections (200) and (400) appear, which are not characteristic to the fcc phase of fullerite. Several explanations of the appearance of these diffraction peaks are possible. From one side, as a result of amorphizing of the C_{60} molecules with the use of the beyond threshold energies the formation of the Frenkel pairs with the introduction to the interstitial positions of the displaced carbon atoms occurs [12]. That leads to possible the charge transfer between the C_{60} molecules and the carbon atoms as well as to the appearance of Coulomb interaction [15]. From other side, it is possible the formation of dimers due to cycloaddition of molecules [16] without a change in the crystal structure, that, as the presence of Coulomb interaction, is capable of influencing the rotational freedom degrees of fullerenes, and, correspondingly, to lead to the nonzero value of the structure amplitude of reflection (200). With further increase in the dose of absorption the intensity of absorption (200) becomes comparable with the intensities of adjacent peaks. All diffraction maximum peaks are widened and they become asymmetric, that indicate their splitting, and respectively, the conversion of the crystalline phases. The obvious predominance of one of the polymerized phases is not observed, i. e., the mixture of different phase structures, created due to the appearance of covalent intermolecular interaction occurs. Thus, even with the significant radiation doses, the polymerization, which arose in the initial stages of radiation exposure, does not disappear. Simultaneously, beginning from the small doses of absorption, the amorphization of crystal structure takes place, which is accompanied by an increase in the background at small angles, tailing peaks and decreasing their intensity. From other side, beginning from the doses of 8 MGy, an increase in the intensity of reflection (200) becomes slower, i. e. the amorphization affects not only molecules in the initial, but also in the polymerized phase. Nevertheless, the obvious suppression of polymerization with an increase in the amorphous phase it is not observed in contrast to the results of ion irradiation [10].

It is possible to expect that the structural transformations during the electron irradiation and the radiation damages of molecules due to their amorphization must influence their vibrational spectra. However, as one can see from Fig. 2, the radical change in the positions of the Raman lines, which it would be possible due to substantial change of interatomic interactions in the molecules, does not occur. First of all this relates only to the insignificant displacement of vibrational $A_g(2)$ pinch-mode (1469 cm^{-1}) to the side of smaller frequencies. With the obvious polymerization of C_{60} fullerites, including the photopolymerization, the softening of the vibrational $A_g(2)$ mode and fall in the intensity of the Raman line corresponding to it takes place.

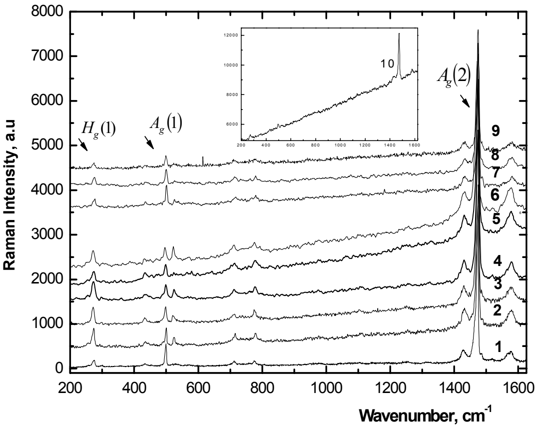

Fig. 2. Raman scattering for the C_{60} films, irradiated by electrons with different doses of the absorption: 1 – initial (nonirradiated) sample; 2 – 1.5; 3 – 3.0; 4 – 4.0; 5 – 6.0; 7 – 10.0; 8 – 12.0; 9 – 13.0; 10 – 16.0 MGy (on the inset) ($d \approx 2.0$ μm, Si(100), $E_e = 1.8$ MeV, $\lambda = 514.5$ nm)

However, the other spectral lines are transformed with an increase in the radiation dose, that corresponds to the modification of Raman spectrum with the polymerization of C_{60} fullerite under the pressure [17 –18]. In particular, the fall in the normalized intensity of $H_g(1)$ peak and its splitting is observed. Analogously, with an increase in the absorption dose the vibrational H_g modes are splited and transformed.

As in the case of polymerization with the photoexcitation and pressure the peak intensity of the breathing $A_g(1)$ mode decreases with an increase in the radiation dose. At the same time with the absorption doses of (6 – 8) MGy and especially in the case of the greatest dose of 16 MGy the growth of the background of Raman scattering occurs. The intensity of all lines, including vibrational $A_g(1)$ mode, sharply decreases. Last effect testifies about the flow of the essential processes of amorphization and, possibly, the fragmentation of the fullerene molecules. Thus, the modification of the Raman scattering spectra during the beyond threshold irradiation indicates several stages of the simultaneous realization of polymerization and amorphization of fullerites. If in the initial stage to the doses of absorption 6 MGy – 8 MGy the polymerization is mainly manifested, then, beginning from these doses, the amorphization of molecules is noticeable, which strongly grows already with the doses of absorption close to 16 MGy, although the polymerization do not cease. It should also be noted that the rise of the polymerization of structure against the background of its amorphization leads to the special features of the vibrational spectra, which are differed from their form in the case of creating the polymerized phases by pressure.

It is possible to expect that in the case of polymerization and amorphizing of fullerites, i. e. with the formation of new local states within the limits of energy gap, the width of the forbidden band, position and the density of one-electron states, and also physical properties, caused by elementary excitations are changed. Actually, as follows from Fig. 3, the photoexcitation of C_{60} fullerites, irradiated by electrons, leads to a change in the recombination both of the singlet and triplet Frenkel excitons with an increase in the dose of absorption. If proceed from the assumption that the radiative recombination of the self-localized singlet Frenkel excitons near the energy 1.69 eV corresponds to their capture by dimeric traps [19], and the emission near the energy of 1.45 eV is caused by triplet excitons [7], then it is evident that the photoluminescence spectra in this range of energies noticeably change.

First, with an increase in the radiation dose the intensity of photoluminescence decreases. Beginning from the absorption dose of 1.5 MGy the emission bands near the energies of 1.65 eV and 1.5 eV are formed. Both bands in the initial state are weak and they are displaced to the side of smaller energies. With an increase in the radiation dose the intensity of the emission peak near the value of energy 1.65 eV grows and is compared with respect to the value with the basic emission peak. Moreover, with the dose of absorption 13.0 MGy the bifurcation of peaks in the energy range of (1.6 – 1.7) eV is observed. Although with the subsequent fluence this splitting disappears, but the broad emission band remains. It should be noted that the wide unstructured emission band with the maximum near the energy of 2.05 eV appears with the greatest absorption dose of 16.0 MGy. The formation of this band becomes noticeable with the smaller absorption dose of 13.0 MGy.

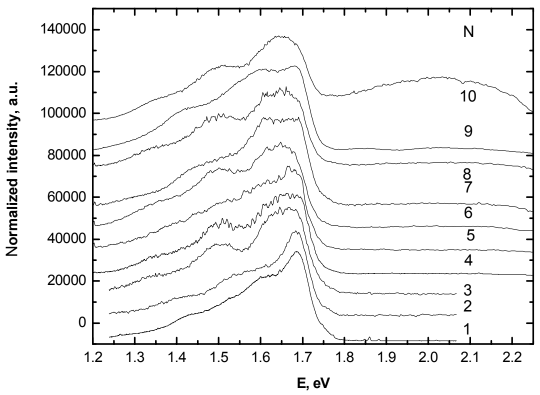

Fig. 3. Photoluminescence spectra of the C_{60} films, irradiated by electrons with different doses of the absorption: 1 – initial (nonirradiated) sample; 2 – 1.5; 3 – 3.0; 4 – 4.0; 5 – 6.0; 7 – 10.0; 8 – 12.0; 9 – 13.0; 10 – 16.0 MGy ($d \approx 2.0$ μm, Si(100), $E_e = 1.8$ MeV, $\lambda = 514.5$ nm).

It is obvious that this complex behavior of the intensity of photoluminescence is the result of the imposition of several reasons, which influence localization of neutral excitations (excitons). This is caused by the presence of weak π-bonds, which are responsible for the double bonds between the carbon atoms in the C_{60} molecule. One of such reasons can be the association of C_{60} fullerites by the π-bonds, which are responsible for the emitting transitions in the case of their consumption for the σ-bonds formation with the dimerization, polymerization and amorphization of molecules in the condensed state. This association, as in the case of photopolymerization [20–21], leads to a fall in the integrated intensity of luminescence. From other side, the luminescence can appear on the radiation defects, which destructively influence on the fullerites and leads to the local distortions of molecules. Appearance in this case within the limits of the forbidden energies of the local states, which belong to the capture centers of excitons, can contribute to the recombination of singlet and triplet excitons. Furthermore, as a result of interaction of fast accelerating electrons with the C_{60} crystal the plasmons are formed, whose disintegration leads to the generation of the singlet excitons, whose part with their interaction with the radiation defects converts to triplet state, i. e. it leads to the accumulation of triplet excitons [22].

In connection with this, the existence of the polymerization and destruction of fullerites during irradiation can be considered as the creation of the radiation defects, which facilitate the recombination of singlet excitons, in spite of elimination of glow centers, caused by the destroyed of the double bonds of molecules. With an increase in the irradiation dose the band of radiative recombination near the energy of 1.65 eV, which is responsible for the radiation-induced modification of the fullerite structure and the destruction of molecules, grows, testifying about an increase in the concentration of radiation damages, including the polymerization parts. It is possible that the polymerization of structure occurs also in the case of the absorption dose of 16.0 MGy, About that the noticeable decrease of intensity near the energy of emission 1.69 eV testifies, although, apparently, the processes of amorphization are predominated. It is seen that they are accompanied by the appearance of a high-energy band, which it is possible, is correlated to the excitons with the transfer of charge [23].

One should to assume that with the destructive radiation effects, not only the local electronic states change. That must influence the nature of interband transitions in C_{60} fullerites with the photoexcitation. Fig. 4 shows the spectrum of optical conductivity after the electron irradiation.

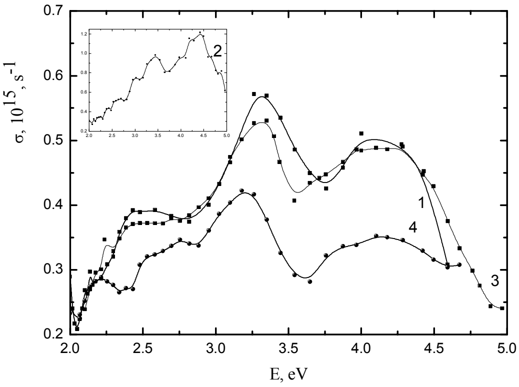

Fig. 4. Optical conductivity spectra of C_{60} fullerite, irradiated by electrons with different doses of the absorption: 1 – initial (nonirradiated) sample; 2 – 1.0; 3 – 6.0; 4 – 10 MGy (on the inset) ($d \approx 2.0$ μm, Si(100), $E_e = 1.8$ MeV)

It is seen that the optical conductivity substantial increases with the initial radiation dose, that is characteristic for the polymerized phase. Therefore, it is possible to assume that even with the small fluences of the beyond threshold electron irradiation, the active processes of structure polymerization are possible. Molecular transitions displace to the side of higher energies with a noticeable increase in the density of electronic states. With further increase in the fluence the optical conductivity value falls, i. e. the polymerization ceases to make the predominant contribution to the destruction of initial fullerites.

Since the band of the interband transitions HOMO-LUMO ($1\text{-}1^{/}$) is smeared, one should assume that the appearance of Urbach tails testifies about the amorphization of structure. The observed increase in the tailing of ($1\text{-}0{,}5^{/}$, $0{,}5\text{-}1^{/}$) and HOMO-LUMO bands with the subsequent growth of the absorption dose of 10.0 MGy indicates that an increase in the fluence after a certain of its value first of all contributes to the amorphization of structure due to the change of *sp*-hybridization of electronic states.

CONCLUSIONS

The formation of radiation defects and long-life electronic excitations in the C_{60} fullerites during the beyond threshold electron irradiation leads to the simultaneous flow of several processes, which influence structure and degeneration of the C_{60} molecules in the condensed state. Polymerization and amorphization are most essential of them. With an increase in the radiation dose these processes make contributions to a change of sp^2-hybridization state of carbon atoms in the C_{60} molecules. If with the small doses of electrons absorption the structure polymerization is noticeable with an fluence increase the amorphization is manifested with the conservation of the polymerized phases formation. Both destructive phenomena during irradiation introduce noticeable changes in the crystal structure, vibrational spectra, photoluminescence and interband molecular one-electron transitions of C_{60} fullerites, that make it possible to reveal the possible mechanisms of the influence of radiation damages on the intermolecular C_{60} fullerene interactions in the carbon nanomaterials.

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