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THE ANOMALOUS RISE OF CAPACITANCE OF C_{60} FULLERITE FILMS AT LOW FREQUENCIES: A CAUSE ANALYSIS

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A known physical fact of the anomalous rise of dielectric permittivity ε of C_{60} fullerite films at ac low frequencies (below 1 kHz) has not had a convincing explanation up to now. Our study was aimed at elucidating the causes of that anomaly. The p -Si/ C_{60} /InGa-eutectic structure was made and a frequency dependence of its capacitance was measured. Relying on the experimental result, a versatile analysis of the phenomenon was carried out. It was shown that the anomalous rise of ε value in the low-frequency region resulted from oxygen intercalation of fullerite with formation of C_{60}/O_2 molecular groups exhibited significant dipole momenta. The presence of such groups produced a dramatic difference between dielectric permittivity of the crystallites' surface areas and that of their volumes. As a result, the difference led to an apparent increase in the dielectric permittivity ε of the structure under study.

Keywords: C_{60} fullerite, polycrystalline film, permittivity, Frohlich's equation, oxygen intercalation

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АНАЛИЗ ПРИЧИН АНОМАЛЬНОГО ПОВЫШЕНИЯ ЕМКОСТИ ПЛЕНОК ФУЛЛЕРИТА C_{60} НА НИЗКИХ ЧАСТОТАХ

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Известный экспериментальный факт аномального возрастания диэлектрической проницаемости ε пленок фуллерита C_{60} на низких частотах (ниже 1 кГц) переменного электрического тока не имеет до настоящего времени убедительного объяснения. Данное исследование было нацелено на выяснение причин указанной аномалии. Была изготовлена структура p -Si/ C_{60} /эвтектика InGa и измерена частотная зависимость ее емкости. На основании полученных экспериментальных данных проведен многосторонний анализ явления. Показано, что возможной причиной аномального повышения ε в низкочастотной области является интеркаляция фуллерита молекулами кислорода с образованием молекулярных групп C_{60}/O_2 , обладающих значительным дипольным моментом. Наличие таких групп вызывает кардинальное различие между значениями диэлектрической проницаемости поверхностных областей кристаллитов и таковой для области их объема, что, в свою очередь, приводит к кажущемуся подъему диэлектрической проницаемости исследуемой структуры.

Ключевые слова: фуллерит C_{60} , поликристаллическая пленка, диэлектрическая проницаемость, уравнение Фрелиха, интеркаляция кислорода

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Introduction

Fullerene thin films are currently believed to be promising candidates for organic electronics [1 – 3]. A major focus is investigations into the properties of fullerene C_{60} , since its molecules have the greatest symmetry and stability. A characteristic feature of fullerene in condensed phase (fullerite) is that impurity atoms can be intercalated into its crystal lattice [4]. Impurity atoms in the face-centered cubic (fcc) lattice of C_{60} fullerite fill octahedral and tetrahedral voids between the host molecules, interacting with them with a potentially pronounced effect on the physical properties of fullerite films [5].

Intercalation of fullerite by oxygen atoms is a particularly intriguing subject. The electron affinity of C_{60} molecules is significantly higher than that of oxygen molecules, estimated at about 2.67 eV [6], while for oxygen it amounts to about 0.45 eV [7]. Therefore, it can be expected that oxygen acts as an electron donor, and fullerite as an electron acceptor. It is hypothesized in [8] that partial transfer of an electron from a donor to an acceptor generates a dipole moment in the C_{60}/O_2 molecule, which is what likely causes a significant increase in the dielectric permittivity for C_{60} films at frequencies below 10^3 Hz. However, the actual physical mechanism behind this phenomenon is not considered in [8].

Electrode polarization is often the cause of the abnormal increase in capacitance at low AC frequencies, occurring in dielectrics with noticeable electrical conductivity given poor contact between the sample and the electrode. A thin layer forms at the interface in these conditions, characterized by a significant electrical impedance [9]. Obviously, this reason is not related to the physical properties of the actual fullerite films.

Other reasons for the anomalous increase in the capacitance of dielectrics in the low-frequency region are also discussed in the literature. For example, the Maxwell – Wagner polarization is observed in inhomogeneous dielectrics with con-

ducting impurities [10]; for polycrystalline dielectrics, this can be attributed to the difference between the permittivities (and conductivities) of the external and internal regions of crystallites, i.e., the grain-interlayer model [3, 10].

The goal of this study consists in understanding the potential role of the described effects and assessing the degree to which they influence the dielectric permittivity of C_{60} fullerite films.

Experimental procedure

The sample was a C_{60} fullerite film deposited on a cold substrate made of *p*-type silicon of KDB-1 grade by thermal spraying. The thickness L of the film, measured with an MII-4 interference microscope, was 250 ± 50 nm.

A close focus was on ensuring reliable contacts between the fullerite sample and the electrodes.

The C_{60} film produced by this method had a polycrystalline structure with the size D of crystallites equal to 100 – 200 nm [11]. The crystallites forming the film were randomly arranged on the silicon substrate in several layers. Before the experiments, the C_{60} films deposited on the substrates were exposed to an air atmosphere for a long time in order to ensure reliable contact of the film with the silicon substrate, since the latter was used as one of the electrodes.

The second electrode was a needle probe made of a liquid indium-gallium eutectic [12]. Such an electrode provided reliable electrical contact with the fullerite film, without mechanical damage, due to interaction of the surface tension forces of the eutectic and gravitational forces.

An E7-20 LCR meter was used to measure the capacitance C and the tangent $\text{tg}\delta$ of the dielectric loss angle in the frequency range from 25 Hz to 1 MHz. The amplitude of the test AC voltage was 0.04 V. All measurements were carried out at room temperature $T = 293$ K in a darkened measuring cell. The given section of the C_{60} film was subjected to electroforming prior to dielectric measurements: a constant voltage $U = 30$ V was applied to the electrodes for several tens of

minutes. Electroforming considerably increased the stability of the readings and the reproducibility of the results for repeated measurements in this section of the film [11].

The area S of the contact spot of the needle electrode with the fullerite film was calculated based on the data for the capacitance C of the given structure measured at a frequency of 1 MHz. It was assumed that the dielectric permittivity of the film at this frequency was close to the value of the high-frequency dielectric permittivity ϵ_∞ of C_{60} fullerite. According to the data given in literature [13], $\epsilon_\infty = 2.6$. Therefore, with a capacitance $C = 8.2$ pF, the area of the contact spot turns out to be $S \approx 0.09$ mm². This value of S was subsequently used to calculate the dielectric permittivity spectrum in the entire investigated frequency range.

Experimental results and discussion

The goal posed at the first stage of the study was to find out whether the increase in capacitance in the low-frequency region is due to the phenomenon of electrode polarization. According to the data given in [9], the correction for electrode effects in low-frequency measurements, defined as the difference between the measured capacitance C and the true capacitance C_{true} (appearing in the absence of electrode polarization), depends on the material conductivity σ and the frequency f , at which the measurement is carried out, so that

$$C - C_{true} \sim \sigma^2 / f^2. \quad (1)$$

Since the photoconductivity of C_{60} fullerite is observed in the visible spectral range, the conductivity of the given structure could be increased by exposure to light. For this purpose, we used a white LED with a color temperature of 4000 K, producing a luminous flux of 250 lm. The light was focused on the contact spot of the indium-gallium electrode with the fullerite film using a special lens. The structure's conductivity increased from $7 \cdot 10^{-7}$ S/cm (value in the dark) to $3 \cdot 10^{-5}$ S/cm upon illumination.

Fig. 1 shows the low-frequency dependences for the capacitance of the given structure in the

dark (curve 1) and under LED lighting (curve 2), i.e., at different concentrations of free charge carriers determining the conductivity in it. Apparently, the curves on the graph practically coincide, although the value of the capacitance should have increased by about four orders of magnitude, in accordance with Eq. (1). Furthermore, the assumed dependence should have been linear in the case of electrode polarization in the considered coordinates $C (1/f^2)$ but this did not happen either.

Thus, analysis of the experiment carried out at the first stage of the study allows eliminating electrode polarization as the cause of the anomalous increase in capacitance in the low-frequency region of the spectrum. In other words, the increase in capacitance we have observed should not be regarded as an artifact, that is, it is not associated with the specific experimental conditions or the peculiarities of electrical contacts.

At the second stage of the study, we considered the deep mechanisms underlying the increase in the capacitance of the structure at low frequencies.

As noted above, fullerite films obtained by thermal spraying are polycrystalline. Oxygen molecules quickly penetrate into the film, diffusing along the crystallite interfaces; as a result, the near-interface regions of the crystallites are saturated with oxygen to a greater extent than their bulk. For this reason, the conductivity and dielectric permittivity of the surface layers of crystallites and their bulk are different [3]. Consequently, the dielectric dispersion in such structures is best described by the theory of multilayer dielectric systems, characterized by an abnormally high dielectric permittivity in the low-frequency region [9].

Fig. 2, a shows the dependence of the dielectric permittivity on the frequency f . The values of ϵ' were calculated with the equation for a plane capacitor based on capacitance measurements. Evidently, the dielectric permittivity increases rapidly and monotonically with a decrease in the frequency of the applied AC voltage. For example, $\epsilon' \approx 3$ at a frequency of 10^3 Hz, reaching $\epsilon' \approx 300$ at 30 Hz.

The dependence of the dielectric loss angle

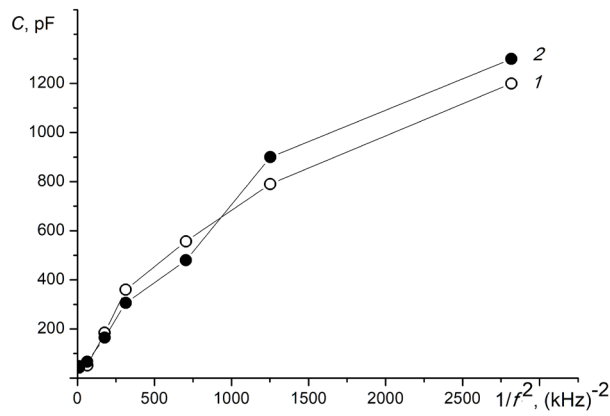


Fig. 1. Low-frequency dependences for capacitance of a $p\text{-Si}/\text{C}_{60}/\text{InGa}$ eutectic structure in the dark (1) and under LED lighting (2). Luminous flux (250 lm) focused on the contact spot of the $\text{C}_{60}/\text{InGa}$ eutectic at $T = 293 \text{ K}$

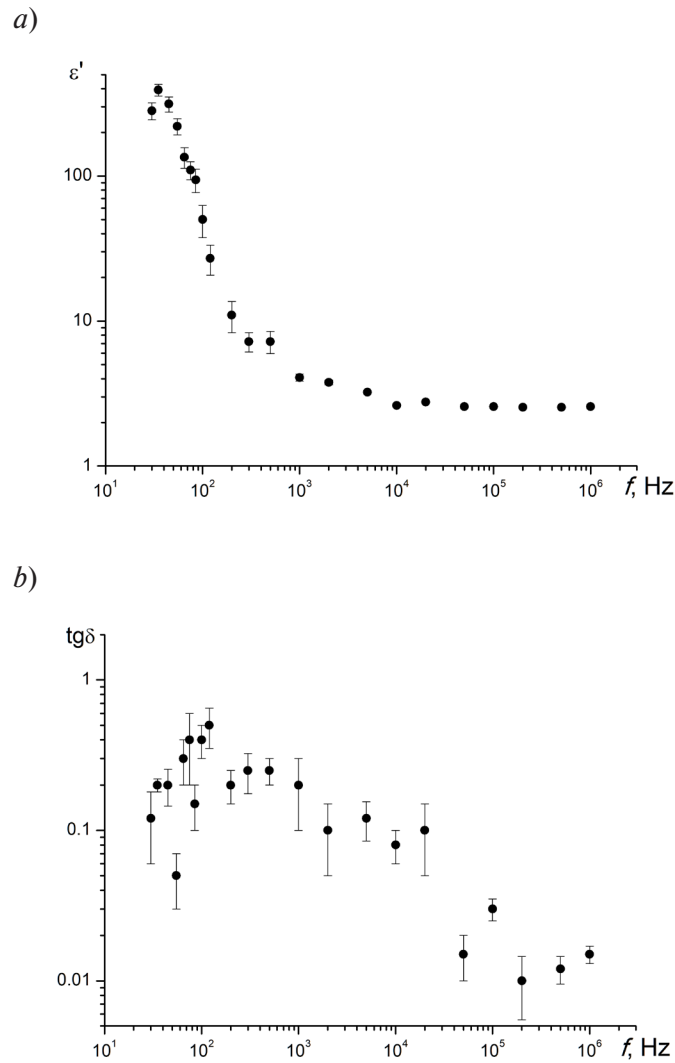


Fig. 2. Dependences of dielectric permittivity (a) and dielectric loss angle tangent (b) for $p\text{-Si}/\text{C}_{60}/\text{InGa}$ eutectic structure; $T = 293 \text{ K}$

tangent $\text{tg}\delta$ (f) (Fig. 2, b) bears a non-monotonic character. The value of $\text{tg}\delta \approx 0.01$ at frequencies above 10^5 Hz, however, it gradually increases with decreasing frequency, reaching a maximum value of about 0.8 at $f \approx 10^2$ Hz, subsequently decreasing to about 0.1. It is impossible to accurately determine the position of this maximum on the frequency scale due to the significant scatter of data in measurements of $\text{tg}\delta$. Notably, a broad maximum was also observed in [8] for the frequency curve $\tan\delta$ in the region of 1 kHz during measurements of C_{60} fullerite films. The appearance of the maximum was attributed to intercalation of fullerite with oxygen and formation of dipole groups in the C_{60}/O_2 molecule.

According to the grain-interlayer model, the set of links that are internal crystallite regions (grains) and their surface regions (interlayers) is considered as a homogeneous structure with a common relaxation time τ corresponding to the relaxation time of an individual link, while the value of τ is calculated by the Debye equations for dipole orientation polarization.

As polar molecular groups C_{60}/O_2 are accumulated in the interlayers, their dielectric permittivity turns out to be higher than that of grains, and the conductivity of these groups is lower [9]. Therefore, the low-frequency dielectric permittivity ε_1 of the oxidized near-surface crystallite layer (interlayer) can be estimated using the relation

$$\varepsilon' = \varepsilon_1 D/d, \quad (2)$$

where ε' is the dielectric permittivity of the C_{60} film, determined experimentally; d is the thickness of the oxidized near-surface crystallite layer (interlayer).

The thickness d can only be estimated approximately. According to the data in [14], the value of d should not exceed 15 nm. Consequently, given a crystallite size of $D = 150$ nm, we obtain that the dielectric permittivity ε_1 of the interlayer lies in the range from 10 to 30 at a frequency of 35 Hz with d in the range from 5 to 15 nm.

Let us estimate the values of the dipole moment of the C_{60}/O_2 molecule and the concentra-

tions of these molecules at which the given value of ε_1 is attainable. We use the Fröhlich equation relating the macroscopic dielectric permittivity to the dipole moment of the molecule

$$\frac{(\varepsilon_1 - \varepsilon_\infty)(2\varepsilon_1 + \varepsilon_\infty)}{\varepsilon_1(\varepsilon_\infty + 2)^2} = \frac{Ngp^2}{9\varepsilon_0 k_B T}, \quad (3)$$

where N is the number of polar molecules of C_{60}/O_2 (dipoles) per unit volume of the interlayer; p , D, is their dipole moment; T , K, is the temperature, k_B , JK^{-1} , is the Boltzmann constant; ε_0 , F/m, is the vacuum permittivity; g is a parameter accounting for the local ordering of molecules;

$$g = 1 + z \langle \cos \gamma \rangle.$$

Here z is the coordination number ($z = 12$ for the fcc lattice), $\langle \cos \gamma \rangle$ is the average cosine of the angle between the molecule at the point of reference and its nearest neighbors ($\langle \cos \gamma \rangle = 0.7$ was taken in the calculations).

Let us calculate the value of N bearing in mind that there are two C_{60} molecules for each fcc cell. Suppose that all C_{60} molecules in the near-surface layer of the compound are oxidized; then the number of dipoles per unit volume of this layer is equal to

$$N = 2 \frac{V_{ox}}{(a \cdot D)^3}, \quad (4)$$

where V_{ox} , nm^3 , is the volume of the oxidized layer in one crystallite, a , nm, is the edge length of the fcc cell of C_{60} ($a = 1.417$ nm [14]).

We obtain for these conditions that $N \approx 2.5 \cdot 10^{26} \text{ m}^{-3}$. This value of the concentration of polar groups seems reasonable, since, according to the data in [14], the relative oxygen content in the oxidized layer is C : O = 10 : 1.

According to the data in [8], the value of the dipole moment p of the C_{60}/O_2 molecule is 0.9 D. The authors estimated it assuming that the fraction ξ of the charge transferred from the donor (intercalated oxygen O_2) to the acceptor (C_{60} fullerite molecule) was 4%. This value of ξ was

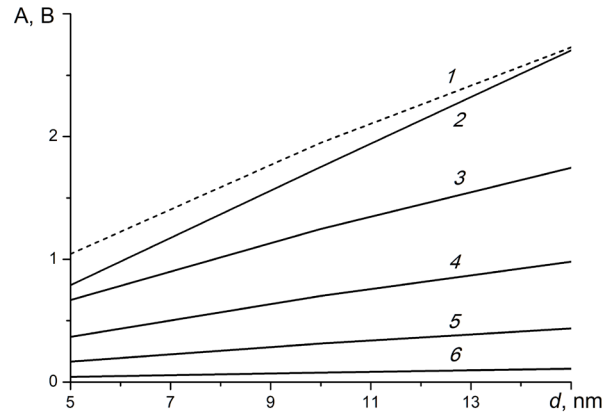


Fig. 3. Dependences of left (A) (straight line 1) and right-hand (B) (straight lines 2 – 6) sides of the Fröhlich equation (3) on the thickness of oxidized near-surface crystallite layer (interlayer) at different values of the dipole moment of the C_{60}/O_2 molecule, p , D: 5 (2), 4 (3), 3 (4), 2 (5), 1 (6)

determined by the authors from the condition of the best agreement between the calculation results and experiment. Nevertheless, it is pointed out in [15] that a substantially larger fraction of the charge can be transferred. According to the estimates in that study, the value of ζ can reach 49%.

We should note that estimating the dipole moments of the given molecular groups should account for the fact that the interaction of oxygen with fullerene molecules produces various forms of oxidized fullerene $C_{60}O_n$. For example, the so-called 'open' and 'closed' epoxides can evolve, as well as other isomers, where the oxygen atoms can be attached to different sites of the fullerene molecule. An oxygen atom in the 'open' epoxide, $C_{60}O$ (5-6, pentagon-hexagon), is attached to two carbon atoms at the border of the corresponding faces. The oxygen atom in the 'closed' one, $C_{60}O$ (6-6, hexagon-hexagon), is located above the double bond at the border of two hexagons [16]. As already mentioned, the oxygen atoms in other isomers can be attached to various sites of the fullerene molecule.

Evidently, all the formed $C_{60}O_n$ molecules are characterized by different lengths of chemical bonds and the degree of electron density transfer from donor to acceptor, and, as a consequence, different dipole moments.

In view of the above, let us find the values of

the interlayer thickness d and the dipole moment p for which relation (3) can be satisfied. Calculating its left-hand side for various values of d , we use expression (2), taking $\epsilon' = 300$ and $D = 150$ nm. We denote this left-hand side as $A(d)$, and the right-hand side, which includes the previously estimated parameters N and g , as $B(d)$. We consider the dipole moment of the C_{60}/O_2 molecule as a parameter, varying its values.

These dependences are shown in Fig. 3. Apparently, the condition $A = B$ can be fulfilled only at the thickness of the oxidized layer $d \approx 15$ nm (which is consistent with the known experimental results), but at significant dipole moments of the C_{60}/O_2 molecules, amounting to 4–5 D, when the relative fraction δ of the transferred electron charge exceeds 22%, according to the data in [8].

Conclusion

We have considered the frequency dependence of the dielectric properties of the p -Si/ C_{60} /InGa eutectic structure. We have confirmed that the molecular groups C_{60}/O_2 evolving in the external regions of the crystallites of the C_{60} fullerite possessing a substantial dipole moment can be regarded as a physical mechanism governing the anomalously high increase in the capacity of the given structure at a low frequency. The conductivity and dielectric permittivity of the surface

layers of crystallites and their bulk are different, producing an apparent increase in the dielectric permittivity of the given structure. Therefore, the dielectric permittivity in such structures can be described based on the theory of multilayer dielectric systems.

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