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GaSe<β-CD<J_2>> Architecture Supramolecular Clathrate: Properties and Application

V. Maksymych[1], Dariusz Calus[2], R. Shvets[1], P. Chabecki[2], N. Pokladok[1], F. Ivashchyshyn[1,2]

[1] *Institute of Applied Mathematics and Fundamental Sciences, Lviv Polytechnic National University, 12, Bandera St., 79013 Lviv, Ukraine*
[2] *Faculty of Electrical Engineering, Czestochowa University of Technology, 17, Al. Armii Krajowej, 42-200 Częstochowa, Poland*

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Modern nanotechnologies are designed to create functionally hybrid inorganic/organic materials with extraordinary properties. Great interest in this direction was gained by clathrates – inclusion compounds built on the host<guest> principle. Applying intercalation techniques, the supramolecular structures thus formed can be ordered in a certain way using inorganic matrices, forming clathrates of subhost<host <guest>> type. According to this principle, we formed GaSe<β-CD<J_2>> clathrate. By forming this structure using weak interactions, it was possible to observe a giant magnetocapacitance effect at room temperature. Its obtained values indicate the prospects of using GaSe<β-CD<J_2>> clathrate as a material for creating capacitive analogues of resistive storage devices. The observed effect is related to a special state of the impurity energy subsystem, which at room temperature will have a decisive influence. Thus, when <β-CD<J_2>> supramolecular complex is introduced into the GaSe semiconductor matrix, impurity levels are split into bands and a quasi-continuous spectrum is formed in two temperature ranges: 248-252 and 298-332 K. The formation in this case also of deep quantum wells at the semiconductor matrix/supramolecular complex interface led to the effect of negative capacitance, which, in turn, can be used to form non-gyratory delay lines that can be directly incorporated into the structure of micro- and nanoelectronics. The magnitude of this effect, according to the research results, can be controlled by illumination and an applied constant magnetic field. The theoretical calculations of the impurity energy spectrum, carried out based on impedance spectroscopy data according to the Geballe-Pollack theory, show good agreement with the experimental data.

Keywords: GaSe, Impedance spectroscopy, Magnetoresistivity, Photo- and magnetocapacity, Inductivity.

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1. INTRODUCTION

The modern development of nanotechnology and nanodevices has brought to the fore the task of creating new functional materials and nanosystems based on them. The use of nanosized structural elements for their construction opens up completely new properties and new areas of their practical application. Due to their functional hybridity and extraordinary properties, organic molecules and substances are increasingly used as relevant components. Therefore, scientific research is increasingly focused on the creation and study of new nanohybrid materials obtained by intercalation of guest components into the cavity of the host materials. This technological approach to the synthesis of heterostructured materials allows creating supramolecular complexes on the lock-and-key principle, according to which the host-guest bond is characterized by weak interaction, which allows maintaining the identity of their properties [1-3].

Currently, the closest to practical application are the results of the research of semiconductor clathrates, phonon glasses, which belong to the most promising thermoelectric materials [4-6]. In this case, the solution to Slack's hypothesis [7, 8] on the formation of structures, in which weakly bound guest atoms or molecules may oscillate in a limited volume at a frequency that provides resonant scattering of phonons, ensuring low thermal conductivity at high electrical conductivity in the host bonds, is almost found [9]. As for other physical aspects of host-guest supramolecular ensembles, the papers devoted to the electronic structure calculations (for instance, [10, 11]) or systems with excitation energy transfer [12] should be particularly mentioned.

The construction of clathrates with a hierarchical structure of supramolecular subhost<host<guest>> ensembles enables the formation of inorganic/organic structures with the desired functional hybridity. For instance, in the clathrates that we recently synthesized at ambient temperature and weak magnetic fields, the colossal magnetoresistivity and magnetocapacitance effects [13, 14], negative capacitance phenomenon and the possibility of accumulating electrical energy at the quantum level [15, 16] were detected. Quantum mechanisms will allow to accumulate electricity of much higher density, which is predicted [17, 18] to give impetus to the significant development of renewable energy systems. These extraordinary properties of hierarchical clathrates undoubtedly actualize further research, in particular, the study of the dependence of physical processes on the type of the subhost and guest matrix, as well as clarification of the influence of the architecture hierarchy on them. This paper focuses on these issues.

2. CONCEPTUAL PROVISIONS AND EXPERIMENTAL PROCEDURE

The purpose of this paper was to obtain and study an intercalated nanocomposite with the subhost<host <guest>> hierarchical configuration. A gallium selenide (GaSe) semiconductor single crystal was used as a subhost material in the experiments. The single crystal grown by the Bridgman-Stockbarger method has a pro-

nounced layered structure and *p*-type conductivity. The band gap (according to the optical data) is 2.02 eV. As is well-known, it is characterized by the presence of the so-called guest positions, i.e., areas of the effect of weak van der Waals forces oriented perpendicular to the crystallographic *C*-axis [19].

For the construction of the supramolecular host<guest> complex, *β*-cyclodextrin (*β*-CD) and molecular iodine (J_2) were selected, respectively. As is known, *β*-CD has the ability to bind molecular iodine in its internal molecular void by molecular recognition, forming host-guest inclusion complexes [20]. Therefore, *β*-CD<J_2> supramolecular cavitate was the guest fractal content of the hierarchical architecture.

The three-stage intercalation/deintercalation technology described in our paper [21] was used to form GaSe<*β*-CD<J_2>> clathrate. The GaSe single crystal reached a 5-fold expansion at the third stage of this technological operation. The so adapted *n*-step-ordered GaSe matrix was placed in a relevant aqueous solution for intercalation prior to the insertion of the *β*-CD<J_2> supramolecular complex. The aqueous solution was prepared by the following technology: 230 mg of KI and 150 mg of I_2 were dissolved in 25 cm^3 of water and dropped into a conical flask containing a solution of 1.5 g of *β*-CD in 40 cm^3 of water. The conical flask was then sealed with wax, stirred for 3 h with a magnetic stirrer and kept for 12 h in an ice bath for complete iodine encapsulation by *β*-cyclodextrin. After the completion of intercalation, the sample was removed from the solution and washed with deionized water (100 cm^3) and KI solution (1.2 mM, 100 cm^3). Finally, the test sample of GaSe<*β*-CD<J_2>> clathrate was dried in a vacuum drying oven at 45 °C for 24 h.

Studies using impedance spectroscopy were carried out along the crystallographic *C*-axis in 10^{-3}-10^6 Hz frequency range using the AUTOLAB measurement system (ECO CHEMIE, the Netherlands) equipped with FRA-2 and GPES software packages. The uncertain points were removed using Dirichlet filter [22, 23]. The impedance measurements were performed under normal conditions, in a constant magnetic field of 2.75 kOe illuminated by a 65 W solar simulator. External fields were applied along the impedance spectra measurements. This geometry of measurements was chosen in order to dismiss the Lorentz force.

The studies were also carried out using a thermally stimulated discharge in the temperature range – 25-70 °C with a constant heating rate of 5 degrees/min.

Based on the obtained impedance spectroscopy data according to the Geballe-Pollack theory [24], the following parameters of the impurity energy spectrum were calculated: density of states at the Fermi level N_F, hopping radius R, spread of traps near the Fermi level J and real density of deep traps N_t.

3. RESULTS AND DISCUSSION

Fig. 1 shows the frequency dependences of the real component of the complex specific impedance (Re$Z(\omega)$) measured along the crystallographic *C*-axis of the 5-fold expanded GaSe single crystal and GaSe<*β*-CD<J_2>> clathrate formed on its basis. Based on the presented dependences of Re$Z(\omega)$ measured under normal conditions for both samples under study, a conclusion can be made that the insertion of the guest component does not lead to significant changes in the real component of the complex resistance, as the magnitude and nature of the behavior of the dependence remain almost unchanged (curves 1 and 2 in Fig. 1). As can be seen from Fig. 1, Re$Z(\omega)$ dependence behaves as usual for the initial 5-fold expanded GaSe matrix and GaSe<*β*-CD<J_2>> clathrate: the quasihorisontal low-frequency branch transforms to a decreasing one at higher frequencies due to the contribution of charge carriers' hopping on localized states near the Fermi level or the processes of their excitation and capture in the band tails or bands of delocalized states.

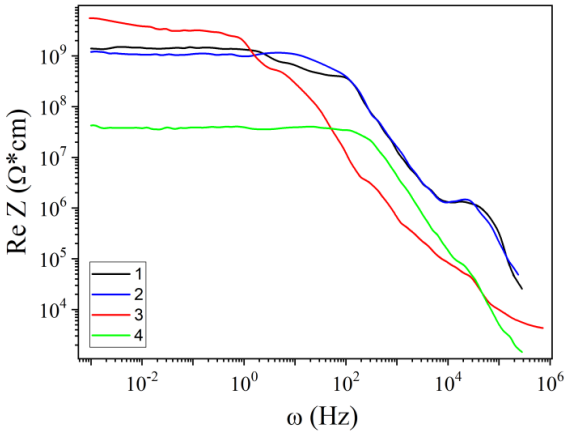

Fig. 1 – Frequency dependences of the real component of the specific impedance for GaSe<*β*-CD<J_2>> clathrate measured under normal conditions (2), in a magnetic field (3) and light (4). (1) 5-fold expanded GaSe matrix

For a more detailed analysis in order to identify the contribution to the overall electrical conductivity of the guest subsystem, impedance studies were conducted applying a constant magnetic field and light. The effect of a constant magnetic field (curve 3 in Fig. 1) leads to the occurrence of the positive magnetoresistance effect in the frequency range 10^{-3}-1 Hz, the value of which reaches $\rho_H/\rho_0 = 4.6$ times, and in the frequency range 1-10^6 Hz to the negative magnetoresistance effect, the value of which reaches $\rho_0/\rho_H = 50$ times, which is not observed for the initial 5-fold expanded GaSe matrix. This effect is most likely caused by the Zeeman redistribution of electronic subsystem states, as was shown earlier in [25]. As known, a magnetic field hinders interband tunnelling and thus reduces conductivity of the material. At the corresponding parameters, the transition of carriers from the valence band or impurity minibands to the conduction band can be significantly hindered. Besides, a magnetic field reduces mobility of the main carriers. This can be behind a significant increase in the real component of the impedance in the low-frequency region (10^{-3}-1 Hz) (curve 3 in Fig. 1). As the frequency increases, this effect starts competing with frequency-stimulated interband tunnelling, and at a frequency of 1 Hz the latter prevails. The obtained result is important in terms of practical use of this clathrate as a highly sensitive magnetic field sensor in a wide frequency range.

As expected, the influence of light causes a significant decrease in the real component of the complex resistance due to photosensitivity of the GaSe semiconductor matrix. The insertion of β-CD<J_2> supramolecular cavitate does not lead to a significant increase in photosensitivity of GaSe<β-CD<J_2>> clathrate, it remains at almost the same level as for the 5-fold expanded GaSe matrix and is $\rho_D/\rho_L \approx 31.6$ times. This result indicates the predominant contribution exclusively of the subhost, the 5-fold expanded GaSe matrix, to the photoconductivity.

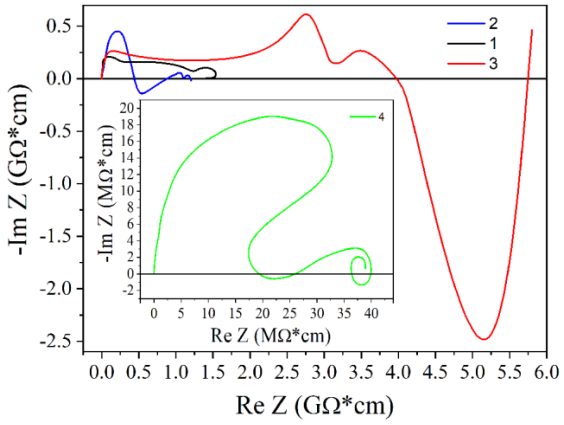

Fig. 2 – Nyquist diagrams for GaSe<β-CD<J_2>> clathrate measured under normal conditions (2), in a magnetic field (3) and light (4). (1) 5-fold expanded GaSe matrix

The above mechanisms of electrical conductivity are confirmed when displaying the Nyquist diagrams shown in Fig. 2. As can be seen, the formation of a <β-CD<J_2>> nanolayer between the GaSe layers transforms the impedance hodograph of the initial expanded crystal so that it goes into the 4th inductive quadrant of the complex hodograph plane in the mid and high frequency regions of the spectrum. Such behavior of electrical conductivity can occur when current carriers are captured by traps and held by them for a period equal to a half cycle of the sinusoidal signal. This effect in general is quite well-known to us for structures of the class under study and is quite fully described in the papers [26, 27]. It should be noted that this effect makes it possible to create non-gyratory nanosized delay lines, which can be directly incorporated into the structure of micro and nanoelectronic devices. An important point is that light does not interfere with the visualization of the negative capacitance effect in the midrange. A constant magnetic field, on the other hand, leads to levelling of the midrange region of the negative capacitance visualization and to its significant amplification in the low-frequency region. The control of the inductance value in this structure by a magnetic field allows creating a new type of magnetically controlled delay lines.

Based on the obtained results for the electrical conductivity, one should expect extraordinary polarization properties of GaSe<β-CD<J_2>>. Therefore, the next step was to study the behavior of the dielectric permittivity $\varepsilon(\omega)$ and dielectric loss tangent tgω for a 5-fold expanded GaSe single crystal and GaSe<β-CD<J_2>> clathrate. The results are shown in Fig. 3. In terms of the possibility of practical application of the obtained results, they were analyzed in the frequency range satisfying the condition tg$\delta < 1$. In general, analyzing the dependences $\varepsilon(\omega)$ shown in Fig. 3, their abnormality and non-monotonicity for both samples under study should be noted. As in the case of the electrical conductivity, the insertion of <β-CD<J_2>> supramolecular cavitate between the layers of the 5-fold expanded GaSe matrix does not lead to significant changes in the behavior of $\varepsilon(\omega)$. However, the situation changes significantly when applying a constant magnetic field and light: colossal magneto- and photodielectric effects are visible. The magnetodielectric effect is visible starting from 20 Hz and takes the maximum value of $\varepsilon_H/\varepsilon_0 = 300$ at 200 Hz, and the photodielectric effect starts at 300 Hz and takes the maximum value of $\varepsilon_L/\varepsilon_D = 125$ at 20 kHz. It should also be pointed out that when applying a constant magnetic field and light, tgδ takes values much less than 1. The observed photodielectric effect is most likely due to the direct interaction of light with the GaSe semiconductor matrix, while the magnetodielectric effect is of more complex nature. It can be caused by the Zeeman redistribution of electronic subsystem states, namely, the redistribution of charge carriers between the semiconductor matrix and the guest component, so that adjacent pairs of different phases will be charged with the opposite charge and can then be

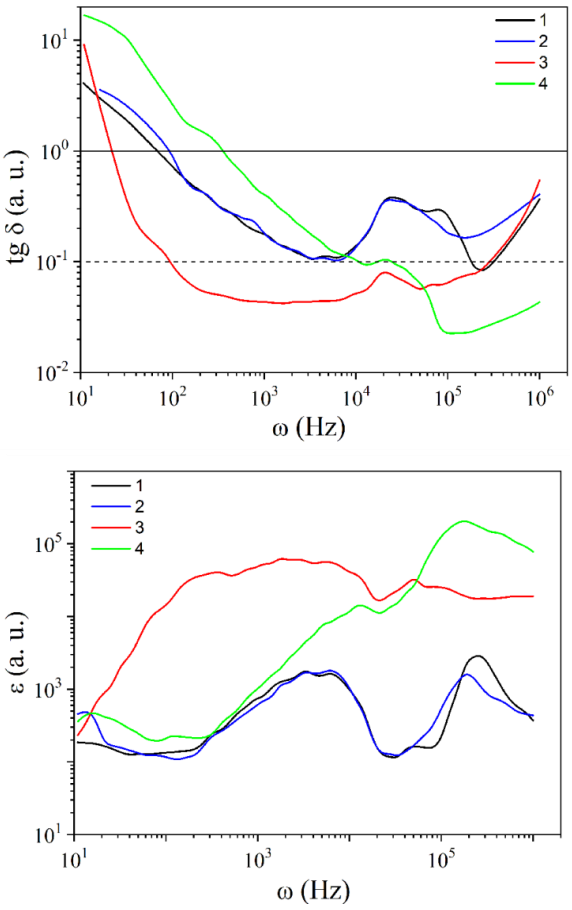

Fig. 3 – Frequency dependences of the electric loss tangent and dielectric constant for GaSe<β-CD<J_2>> clathrate measured under normal conditions (2,) in a magnetic field (3) and light (4). (1) 5-fold expanded GaSe matrix

considered as dipoles, which, due to charge polarization, make a significant contribution to the dielectric permittivity. The distribution mechanism between the phases can be associated with different values of their chemical potential or tunnelling, upon which the lifetime of an electron at an appropriate level can be quite long, as a result of which the system will be charged. The fact that such a mechanism is possible is evidenced by a change in the Nyquist diagram when a constant magnetic field is applied (curve 3 Fig. 2), the low-frequency branch of which goes into the 4th quadrant of the complex plane, indicating that current carriers are captured and held for a period equivalent to a half cycle of the sinusoidal signal.

The practical significance of the obtained results is obvious. The appropriate clathrate can be used in the manufacture of sensitive magnetic field sensors of both resistive and capacitive types. The second option is promising in terms of replacing resistive sensors, being the active elements in an oscillating circuit, with their reactive counterparts. This will allow, at the very least, avoiding the dissipation of electrical energy in the form of heat in such devices.

For the purpose of a more detailed examination of the structure of the impurity energy spectrum of GaSe<β-CD<J_2>> clathrate under study, the spectra of a thermally stimulated discharge were measured (Fig. 4). In the case of the initial 5-fold expanded GaSe matrix, we observe a miniband nature of the energy spectrum with deep trap centers, which is typical of expanded GaSe crystals. In this case, homocharge relaxation occurs, which is also expected. Homocharge relaxation is due to the injection of charge carriers and their localization at trap centers. After the formation of a <β-CD<J_2>> nanolayer between the GaSe layers, the spectrum acquires a quasi-continuous nature in two temperature ranges 248-252 K and 298-332 K, and heterocharge relaxation occurs. Heterocharge relaxation is caused by the redistribution of charge carriers between the semiconductor matrix and the guest component so that adjacent pairs of different phases will be charged with opposite charges and can then be considered as dipoles. The result obtained confirms the above assumptions regarding the nature of the current flow and polarization in GaSe<β-CD<J_2>> clathrate under study.

The results of a theoretical analysis of the impurity energy spectrum based on impedance spectroscopy data carried out according to the Geballe-Pollack theory are given in Table 1. Calculations show that the insertion of <β-CD<J_2>> supramolecular cavitate between the layers of the 5-fold expanded GaSe matrix leads to a 2-fold increase in the density of states near the Fermi level and a 20 % increase in the real density of deep traps. At the same time, the hopping radius slightly decreased (7 %) and the spread of trap centers near the Fermi level dropped by 44 %. The obtained results of calculations correlate well with the measurement data of the thermally stimulated discharge shown in Fig. 4.

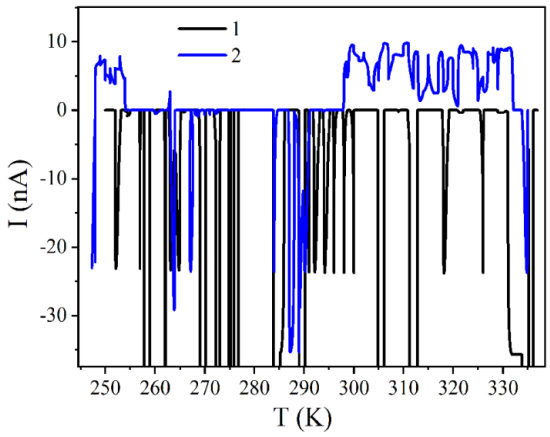

Fig. 4 – Thermally stimulated discharge current measured for the 5-fold expanded GaSe matrix (1) and GaSe<β-CD<J_2>> clathrate (2)

Table 1 – Band spectrum parameters before and after insertion of <β-CD<J_2>> into GaSe

Structure	$N_F \cdot 10^{43}$, $J^{-1}m^{-1}$	$R \cdot 10^{-8}$, m	$J \cdot 10^{-22}$, J	$N_t \cdot 10^{22}$, m^{-3}
5-fold expanded GaSe matrix	5.76	2.58	4.83	2.78
GaSe<β-CD<J_2>> clathrate	12.93	2.39	2.71	3.50

4. CONCLUSIONS

A hierarchical GaSe<β-CD<J_2>> clathrate structure was formed. β-CD<J_2> supramolecular cavitate was synthesized on the lock-and-key principle and inserted between the layers of the 5-fold expanded GaSe matrix using the intercalation technique.

The insertion of β-CD<J_2> supramolecular cavitate between the layers of the 5-fold expanded GaSe matrix leads to a change in the impurity energy spectrum: from a narrow miniband to a quasi-continuous one; from homocharge relaxation to heterocharge relaxation. Theoretical calculations also indicate a 2-fold increase in the density of states and a 44 % decrease in the spread of trap centers near the Fermi level.

GaSe<β-CD<J_2>> clathrate under study exhibits inductive behavior in the mid and high frequency spectral regions. A constant magnetic field leads to levelling of the midrange inductive region and significant amplification in the low-frequency region. This effect makes it possible to create non-gyratory nanosized delay lines, which can be directly incorporated into the structure of micro and nanoelectronic devices, and the control of the inductance value in this structure by a magnetic field allows creating a new type of magnetically controlled delay lines.

For GaSe<β-CD<J_2>> clathrate, the positive magnetoresistance effect is recorded in the frequency range 10^{-3}-1 Hz, the value of which reaches $\rho_H/\rho_0 = 4.6$ times, the negative magnetoresistance effect in the frequency range 1-10^6 Hz, the value of which reaches $\rho_H/\rho_0 = 50$ times, and the magnetodielectric effect in the frequency range 300-10^6 Hz, the value of which reaches $\varepsilon_H/\varepsilon_0 = 300$ times, while tgδ takes a value much less than 1. The relevant clathrate can be used in the manufacture of sensitive magnetic field sensors of both resistive and capacitive types.

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Супрамолекулярний клатрат GaSe<β-CD<J2>> ієрархічної архітектури: властивості та застосування

V. Maksymych[1], Dariusz Calus[2], R. Shvets[1], P. Chabecki[2], I. Bordun[1,2], N. Pokladok[1], F. Ivashchyshyn[1,2]

[1] *Institute of Applied Mathematics and Fundamental Sciences, Lviv Polytechnic National University, 12, Bandera St., 79013 Lviv, Ukraine*

[2] *Faculty of Electrical Engineering, Czestochowa University of Technology, 17, Al. Armii Krajowej, 42200 Częstochowa, Poland*

Сучасні нанотехнології покликані створювати функціонально гібридні неорганічно/органічні матеріали з неординарними властивостями. Великого зацікавлення в даному напрямку набули клатрати – сполуки включення, побудовані за принципом господар<гість>. Застосовуючи інтеркаляційні технології, сформовані таким чином супрамолекулярні структури можна певним чином впорядковувати, використовуючи неорганічні матриці, формуючи клатрати типу субгосподар<господар<гість>>. За таким принципом нами був сформований клатрат GaSe<β-CD<J2>>. Формуючи дану структуру за допомогою слабких взаємодій, вдалося отримати гігантський магнітоємнісний ефект за кімнатної температури. Отримані його значення свідчать про перспективність застосування клатрату GaSe<β-CD<J2>> як матеріалу для створення емнісних аналогів резистивним запам'ятовуючим пристроям. Спостережуваний ефект пов'язаний із особливим станом домішкової енергетичної підсистеми, яка за кімнатної температури буде мати визначальний вплив. Так, при впровадженні супрамолекулярного комплексу β-CD<J2> у напівпровідникову матрицю GaSe відбувається розщеплення домішкових рівнів у смуги та формування квазінеперервного спектру у двох температурних інтервалах: 248-252 та 298-332 К. Формування в даному випадку також глибоких квантових ям на межі розділу напівпровідникова матриця/супрамолекулярний комплекс призвело до появи ефекту "від'ємної ємності", що, в свою чергу, може бути використано для формування безгіраторних ліній затримки, які можуть бути безпосередньо інкорпоровані в структуру пристроїв мікро- та наноелектроніки. Величиною даного ефекту, як свідчать результати досліджень, можна керувати освітленням та прикладеним постійним магнітним полем. Проведені теоретичні розрахунки домішкового енергетичного спектру на підставі даних імпедансної спектроскопії за теорією Джебола-Поллака демонструють добре співпадіння з експериментальними даними.

Ключові слова: GaSe, Імпедансна спектроскопія, Магнітоопір, Фото- та магнітоємність, Індуктивність.