PHOTODYNAMIC PROCESSES IN $LiCaAlF_6$, $LiY_xLu_{1-x}F_4$ AND $SrAlF_5$ CRYSTALS DOPED WITH Ce^{3+} IONS

by

Pavlov Vitaly Vyacheslavovich

ABSTRACT OF PhD THESIS

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Investigations have been carried out at Kazan Federal University (Department of quantum electronics and radiospectroscopy)

Thesis advisor:	Doctor of science, principal research fellow of Kazan Federal University **Semashko Vadim Vladimirovich**
Official opponents:	Doctor of science, professor of Ogarev Mordovia State University **Ryabochkina Polina Anatol’evna**
	Candidate of physical and mathematical sciences, senior research fellow of Kazan National Research Technical University **Gerasimov Konstantin Igorevich**
Organization:	Kazan E.K. Zavoisky Physical-Technical Institute

The defense of the thesis will take place on September 15, 2015 at Kazan Federal University. Address: 18 Kremlyovskaya St. Kazan 420008 Russian Federation. The thesis can be found in the Nikolay Lobachevsky Scientific Library of Kazan Federal University. The on-line version of the thesis (in Russian) is on the websites of Ministry of Education and Science of the Russian Federation (vak2.ed.gov.ru) and Kazan Federal University (kpfu.ru).

GENERAL CHARACTERISTIC OF THESIS

Actuality. There is a necessity of simple compact tunable lasers of the ultraviolet (UV) spectral region, providing high beam homogeneity and low beam divergence in combination with controllable time and energy characteristics. These lasers find an application for the environmental monitoring system, security systems, nanotechnologies and biomedical application.

Widely used gas lasers and lasers with nonlinear frequency conversion do not satisfy the practical requirements in many subjects. For example, the excimer laser radiation, having high energetic characteristics, is not tunable over a wide spectral range, and has poor spatial characteristics (high divergence and heterogeneity of beam). In turn, the lasers with nonlinear frequency conversion are extremely complicated to use.

At the same time in 1977 it was shown that one of the possible ways to the fix problems is the use of solid-state UV lasers operating on the $4f^{n-1}5d$-$4f^{n}$ (5d-4f) transitions of trivalent rare earth ions in a wide-dielectric crystals [1]. Unfortunately, the majority of solid-state active media demonstrates a degradation of optical and laser properties under UV excitation and nowadays there are only a few solid-state active medium that are available for practical use in UV lasers.

Numerous studies (see, for example [2]) have showed that the main cause of such degradation is an one- or multistep photoionization of impurity ions, leading to the formation of free charge carriers in the relevant energy bands of crystal host followed by their localization on the lattice defects and further color centers formation. These processes lead to the extra losses in active medium, which increase the oscillation threshold and deteriorate the energy characteristics of UV lasers. Moreover, they may be a reason why the laser oscillation is observed only during the first few pulses followed for the pumping and sometime it is even prevent any lasing.

Therefore, the study of photoionization spectra of the impurity ions in crystals is relevant not only in terms of fundamental science, but also in terms of practice. On the one hand, the study of impurity photoionization and followed photodynamic processes expands our understanding of the interaction of optical radiation with the system "activator ion - host lattice". On the other hand, it helps us to develop practical recommendations to properly choice an optimal chemical composition of materials, methods and conditions of the pump that would help minimize the harmful effects of photodynamic processes.

The aim of the thesis:

1. To study photoelectric effects and spectral-kinetic characteristics of photodynamic processes in wide-band gap insulator $LiCaAlF_6$, $LiLuF_4$, $LiYF_4$, $LiY_{0,5}Lu_{0,5}F_4$, $SrAlF_5$, doped Ce^{3+} and Yb^{3+}, when they are excited by radiation resonant with the 4f-5d transitions of Ce^{3+} ions (wavelength range 240 - 310 nm);

2. To elaborate recommendations on optimization of chemical composition and optical pumping of cerium-doped active media for tunable solid-state UV lasers.

The objects of research are the promising solid-state laser material of quantum electronics of UV spectral range - fluoride crystals $LiCaAlF_6$, $LiLuF_4$, $LiYF_4$, $LiY_{0,5}Lu_{0,5}F_4$, $SrAlF_5$ doped with Ce^{3+} and Yb^{3+} ions . Crystal $Y_3Al_5O_{12}:Ce^{3+}$ was also investigated as the reference sample in order to test new methods of photoconductivity measurement.

The objectives of the PhD thesis were as follows:

1. Design of the experimental setup that implements two methods of photoconductivity investigation in activated dielectric crystals - a technique with blocking electrodes and a microwave resonant technique with broadband quadrature balance mixer;

2. Carrying out experiments to study the photoelectric effects in the investigated cerium-activated crystals arising out of resonant 4f-5d excitation of Ce^{3+} ions, and determination of the nature of photoconductivity in the excitation wavelength of 240 - 310 nm;

3. Determination of the optimal pump wavelength for the investigated crystals and the influence of co-activating Yb^{3+} and Yb^{2+} ions on photodynamic processes.

Scientific novelty consists of the following:

1. The comprehensive studies of photoelectric phenomena and related photodynamic processes in crystals $LiCaAlF_6$, $LiLuF_4$, $LiYF_4$, $LiY_{0,5}Lu_{0,5}F_4$, $SrAlF_5$ doped Ce^{3+} and Yb^{3+}, using a combination of methods of optical, laser and dielectric spectroscopy, were carried out for the first time;

2. The lifetime of electrons in the conduction band, formed as a result of photoionization of Ce^{3+} ions in investigated crystals, has been estimated, and variations of the polarizability of crystals during resonant 4f-5d excitation of Ce^{3+} ions have been studied for the first time;

3. For the first time the basic parameters of photodynamic processes (the photoionization cross section of ions Ce^{3+} from the excited 5d-state photoionization cross section of color centers, the section of the recombination of charge carriers through the excited 5d-state of cerium ions Ce^{3+}, the electron capture cross section of lattice defects) and their spectral dependences in the range of 240 - 310 nm were determined by analysis of 5d-4f luminescence decay of Ce^{3+} ions and nonlinear absorption of cerium-doped $LiLuF_4$, $LiYF_4$, $LiY_{0,5}Lu_{0,5}F_4$, $SrAlF_5$ crystals;

4. It has been shown that nonmonotonic behavior of obtained spectra of one- and multiphoton photoionization of Ce^{3+} ions in crystals $Y_3Al_5O_{12}:Ce^{3+}$, $LiCaAlF_6:Ce^{3+}$ and $Li_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) is due to electronic transitions to the state of mixed configurations of activator ions, localized near or within the conduction band of the host material.

The scientific importance and substance of the work:

1. It has been designed a microwave resonant setup with a broadband quadrature balanced mixer, which implements a contactless method of research of photodielectric effect in activated crystals and allows us to study the dynamics of photoinduced processes with a 5-nanosecond time resolution;

2. It has been developed an original technique to study photoinduced processes in the cerium-doped materials using analysis of the spectral and energy dependences of the 5d-4f luminescence decay of Ce^{3+} ions;

3. Recommendations on the choice of pumping conditions for the investigated crystals have been proposed in order to enhance their photochemical stability and improve the energy and operating characteristics of UV lasers.

Research techniques. In this thesis the traditional methods of optical spectroscopy for the study of the absorption and luminescence spectra of crystals, luminescence decay of impurity ions; methods of laser spectroscopy for the registration of non-linear absorption of activated crystals and methods of dielectric spectroscopy to study the photoconductivity (such as a microwave resonant technique and conventional technique with blocking electrodes) have been used.

Main provisions for the defense:

1. Studies of recombination contribution to the 5d-4f luminescence decay of Ce^{3+} ions allow us to evaluate numerical values of basic parameters of photodynamic processes,

such as photoionization cross section of Ce^{3+} ions from the excited 5d-states, the photoionization cross section of color centers, the recombination cross section of free electrons through the excited 5d-states of cerium ions Ce^{3+}, cross section of electron capture by lattice defects;

2. Variations of polarizability of investigated cerium-doped crystals under the irradiation in spectral range 240 - 310 nm are due to interconfiguration 4f-5d transitions of Ce^{3+} and it are not associated with color centers absorption processes;

3. The nonmonotonic behavior of obtained spectra of one- and multiphoton photoionization of Ce^{3+} ions in crystals $Y_3Al_5O_{12}:Ce^{3+}$, $LiCaAlF_6:Ce^{3+}$ and $Li_xLu_{1-x}F_4:Ce^{3+}$ (x = 0; 0,5; 1) is caused by the electronic transitions of activator ions to the state of mixed configurations, localized near or within the conduction band of the host material;

4. Co-activation of investigated crystals by Yb^{3+} ions does not lead to a reduction in the lifetime of free charge carriers in the relevant energy bands, and only reduces the concentration and the average lifetime of long-lived color centers induced by UV excitation.

The reliability of the results was achieved by careful planning experiments, using modern experimental equipment, well-proven techniques, correct approbation of the newly created experimental setup, as well as correspondence between the obtained results and the independent studies published in the scientific literature. In addition, the results of study were presented and discussed at various conferences, and published in leading peer-reviewed Russian and foreign journals.

The results of work receive approval on 11 International and 1 Russian conferences, the titles and locations of which are shown in the list of published works [A7 - A18].

Publications. The main results of the thesis were published in six papers [A1 - A6] in journals recommended by State Commission for Academic Degrees and Titles and in twelve abstracts and conference proceedings [A7 - A18].

Personal contribution of the author is analyzing the literature data on the topic of the dissertation; participation in the discussion and formulation of the aims and objectives of the study; in realization of experimental setup, allowing to explore the photodielectric effect in activated dielectric crystals by means of the traditional method with contacting electrodes and the microwave resonance method; in experimental studies of the samples by optical, laser and dielectric spectroscopy methods and analysis of experimental data using mathematical simulation; in interpreting of the results and elaboration of the recommendations on the choice of pumping conditions for the investigated crystals.

Invaluable assistance in conducting dissertation studies was provided by the thesis advisor Semashko V.V., who participated in the formulation of research problems and the discussion of the experimental results. Co-authors of publications - Korableva S.L., Marisa M.A. and Gordeev E.Yu., who have synthesized investigated fluoride crystals. Collaborators - Efimov V.N. and Rahmatullin R.M., who participated in the creation of the resonant microwave setup. Collaborators - Gorieva V.G. and Ivoilov N.G., who have assisted in carrying out experiments on X-ray fluorescence analysis of the grown samples. Co-author - Yunusova A.N., who has carried out experiments to study the spectroscopic properties of $SrAlF_5$: Ce^{3+}, $SrAlF_5$: Ce^{3+}, Yb^{3+} crystals. Co-author - Nurtdinova L.A., who has studied the absorption spectra of double-doped fluorides crystals with scheelite structure in the VUV region and assisted in the interpretation of experimental results.

The structure of the thesis. The thesis consists of an introduction, four chapters, conclusion, a list of author's publications and a list of references, including 131 items. The size of the thesis is 153 pages, including 66 figures and 5 tables.

THE MAIN RESULTS

The *first chapter* is a literature review, summarizing information about the photodynamic processes induced in activated crystals under external optical exposure. Their effect on the optical, laser and the dielectric properties of activated crystals is described. In the conclusion of chapter the basic methods of optical and dielectric spectroscopy, which successfully applied in the study of impurity photoionization processes in dielectric crystals are listed. It is shown that the methods of optical spectroscopy cannot directly investigate the appearance of free electrons in the conduction band. Therefore, an adequate description of UV radiation-induced photodynamic processes in cerium-activated crystals, especially the processes of impurity photoionization, requires using a combination of methods of optical and dielectric spectroscopy.

The applicability of dielectric spectroscopy methods for the study of photodynamic processes in activated crystals is caused by photodielectric effect, which consists in a variation of permittivity of crystal ($\varepsilon^* = \varepsilon_1 - j\varepsilon_2$) under the action of electromagnetic radiation. The variation of imaginary part of permittivity $\delta\varepsilon_2$ is due to the photoconductivity (formation of free charge carriers in the energy bands of host). In turn, the change of real part of the permittivity $\delta\varepsilon_1$ of crystal under electromagnetic irradiation is caused by the variation of the polarizability of crystal.

In the *second chapter* an overview of the crystal-chemical properties of Ce,Yb-doped $LiLuF_4$, $LiYF_4$, $LiY_{0.5}Lu_{0.5}F_4$, $SrAlF_5$, $LiCaAlF_6$ crystals, the features of their growth and prepare for investigation are described. The chapter also describes the technical features of the used methods of optical and dielectric spectroscopy. It has been given special attention to the Q-band microwave resonance setup realized in the performance of the thesis.

A feature of the microwave setup compared to existing analogs is that it allows us to separate and simultaneously register the signal from the variation of polarizability of crystal $\delta\varepsilon_1$ and the photoconductivity signal $\delta\varepsilon_2$ with the 5-ns time resolution.

The results of testing of the realized microwave resonant setup (by example of photoconductivity investigations of $Y_3Al_5O_{12}:Ce^{3+}$ crystal) are shown in the *third chapter*. Here the results which were obtained using the microwave resonant setup are compared with the results of photoconductivity measurements got by means of conventional method, consisting in the direct measurement of the photocurrent flowing through the measuring cell with sample.

As a result of successful testing of the experimental microwave setup, the photoconductivity spectrum of $Y_3Al_5O_{12}:Ce^{3+}$ crystal in the range spectral 240 - 280 nm was recorded. This spectrum corresponds to the single electronic transition of impurity Ce^{3+} ions from ground 4f-state to the excited 5d-states localized in the conduction band of the host. It was also shown that the microwave resonant setup enables to avoid the artifacts that occur in case of photoconductivity measurements using contacting electrodes.

The *fourth chapter* contains the results of photodynamic processes studies in the fluoride crystals obtained by optical, laser and dielectric spectroscopy. Description of research is constructed in such a way that the study of each subsequent sample include into account more and more new processes.

$LiCaAlF_6$ crystal doped with Ce^{3+} was the first object of study. According to the data of previous studies [3] in this crystal under resonant 4f-5d excitation of Ce^{3+} ions solarization process (formation of color centers) are almost absent. Therefore, the photoconductivity spectrum of this crystal should be caused only by processes of stepwise photoionization of the impurity ions.

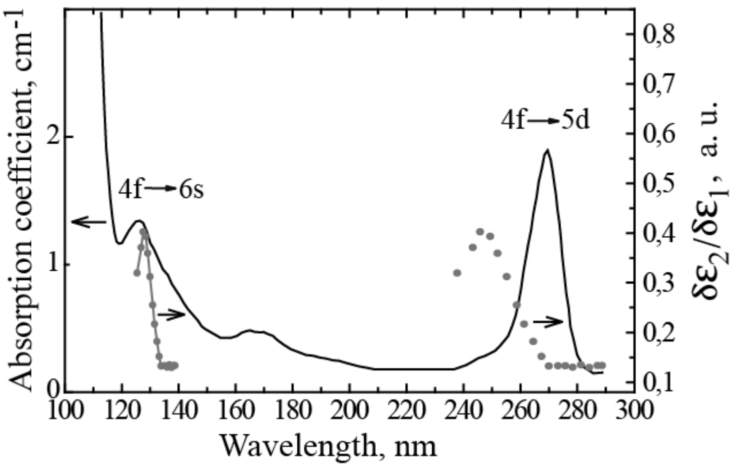

Figure 1 – Comparison of unpolarized absorption spectrum of $LiCaAlF_6:Ce^{3+}$ crystal [3] with the spectrum of $\delta\varepsilon_2(\lambda)/\delta\varepsilon_1(\lambda)$ shifted to shorter wavelengths by the energy of low-energy 5d-state Ce^{3+} ions

The study of $LiCaAlF_6:Ce^{3+}$ crystal under irradiation in spectral range 240 - 290 nm by means of the microwave resonant set-up is showed that the average lifetime of free electrons in the conduction band is about 10 ns, and the recovery time of initial polarizability of crystal after exposure to laser radiation is of the order of several hundred nanoseconds. It is also found that the spectral dependence of the real part of permittivity $\delta\varepsilon_1(\lambda)$ of crystal under laser irradiation replicates the absorption spectrum of Ce^{3+} ions. At this point, it was concluded that interconfigurational 4f-5d transition of cerium ions makes the main contribution to the variation of polarization of the crystal. Photoionization spectrum of Ce^{3+} ions from the excited 5d-state has been determined from the ratio of the photoconductivity spectrum $\delta\varepsilon_2(\lambda)$ to the spectrum $\delta\varepsilon_1(\lambda)$. Based on a comparison of the absorption spectrum of $LiCaAlF_6:Ce^{3+}$ crystal with the spectrum of $\delta\varepsilon_2(\lambda)/\delta\varepsilon_1(\lambda)$ (Figure 1), it was found that the irradiation of $LiCaAlF_6:Ce^{3+}$ crystal in spectral range 240 - 290 nm leads to a two-step photoionization of Ce^{3+} ions by successive electron transitions 4f → 5d → 6s, where the state of 6s-configuration is localized in the conduction band of the host.

To identify the contribution of photoionization of color centers to the formation of photoconductivity signal the $LiY_xLu_{1-x}F_4$ (x = 0, 0.5, 1) crystals doped with Ce^{3+} and Yb^{3+} were investigated. It is known that UV irradiation of these crystals leads to the processes of color centers formation [4].

Using of microwave resonance setup it was found that the average lifetime of free electrons in $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) and $LiY_xLu_{1-x}F_4:Ce^{3+}$, Yb^{3+} (x = 0, 0.5) crystals induced by irradiation in spectral range 240 - 310 nm is about 10 ns. In the other hand, the decay of signal from the variation of polarizability of crystal $\delta\varepsilon_1$ has two components. The most intensive short-lived component is characterized by the decay time of the order of several hundred nanoseconds, and is not dependent on the ytterbium ions. Time decay of long-lived components is reduced by about half, with additional co-activation of crystal by Yb^{3+} ions. It is associated with a decrease of the concentration and the lifetime of long-lived color centers. . As it was found for $LiCaAlF_6:Ce^{3+}$ crystal, the interconfigurational 4f-5d transitions of cerium ions in $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) crystals make the main contribution to the change of the polarization of crystal, which is greater than the contribution from the processes of color centers absorption.

To determine the nature of photoconductivity in $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) and $LiY_xLu_{1-x}F_4:Ce^{3+},Yb^{3+}$ (x = 0, 0.5) crystals which are excited by laser radiation resonant to the 4f-5d transitions of Ce^{3+} ions, a newly developed method, based on mathematical analysis of the 5d-4f luminescence decay of Ce^{3+} ions was applied [A1, A2]. The main idea of the method is to study the contribution of recombination component in non-exponential 5d-4f luminescence decay of Ce^{3+} ions.

The spectral dependences of photoionization cross-section of Ce^{3+} ions from the excited 5d-states and ionization cross-sections of color centers in $LiLuF_4:Ce^{3+}$ and $LiYF_4:Ce^{3+}$ crystals have been determined by mathematical modeling of the 5d-4f luminescence decay kinetics of Ce^{3+} ions, which were recorded at different wavelengths of the exciting radiation. Analysis of the results shows that the band in the recorded photoconductivity spectra with a maximum at 265 nm is due to the two-step photoionization of Ce^{3+} ions as a result of successive transitions 4f → 5d → 6s, where the state of 6s-configuration of Ce^{3+} ions localized in the conduction band (Figure 2). The increase of photoconductivity signal in the short-wave region is interpreted as the result of two processes: photoionization of Ce^{3+} ions directly into the conduction band and the destruction (photoionization) of color centers. It was also found that co-activation of $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) crystals by Yb^{3+} ions, which increases their photochemical stability, does not affect the photoionization spectra of Ce^{3+} ions in investigated spectral range.

However, the method based on co-activation by Yb^{3+} ions is not effective for all the cerium-activated fluoride crystals. For example, in certain fluoride crystals ytterbium ions may enter the crystal lattice not only in trivalent state, but also in divalent state. In this work the influence of Yb^{2+} ions on photodynamic processes studied by the example of $SrAlF_5$ crystals, doped with Ce^{3+} and $Yb^{3+/2+}$ ions.

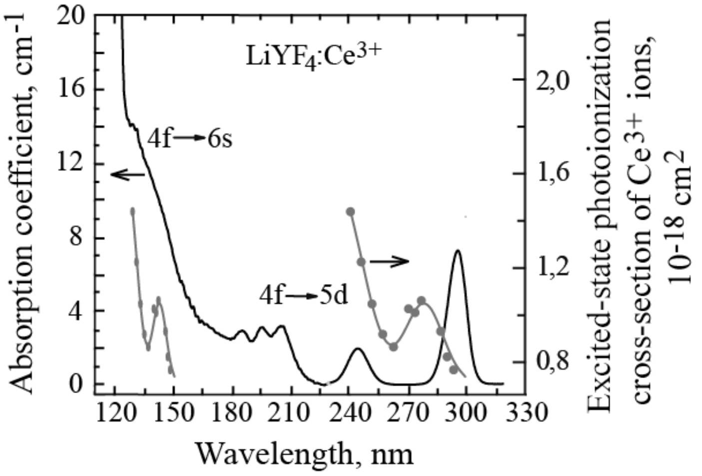

Figure 2 – Comparison of absorption spectrum of $LiYF_4:Ce^{3+}$ crystal with the photoionization spectrum of Ce^{3+} from excited 5d-state, which is shifted to shorter wavelengths by the energy of low-energy 5d-state Ce^{3+} ions

Experiments were carried out in the spectral range 240 - 280 nm. It was found that in $SrAlF_5:Ce^{3+}$ and $SrAlF_5:Ce^{3+},Yb^{3+}$ crystals the average lifetime of free carriers is about 8 ns, and the recovery time of initial polarizability of crystal is of the order of several hundred nanoseconds. The registered spectrum $\delta\varepsilon_1(\lambda)$ is in qualitative agreement with the absorption spectrum of the crystals. Therefore, as before, it can be argued that the main contribution to the change of polarizability of crystal makes the interconfigurational transitions of Ce^{3+} ions and Yb^{2+}.

The method based on mathematical analysis of nonlinear absorption of radiation was used to interpret the photoconductivity spectrum $\delta\varepsilon_2(\lambda)$. The mathematical simulation of dependences of the absorption coefficient on the excitation energy density allows us to determine the spectra of photoionization cross-section of Ce^{3+} ions from the excited 5d-states and ionization cross-sections of color centers in $SrAlF_5:Ce^{3+}$ and $SrAlF_5:Ce^{3+}$, Yb^{3+} crystals. The results of modeling have revealed that the photoconductivity spectrum of $SrAlF_5:Ce^{3+}$ and $SrAlF_5:Ce^{3+},Yb^{3+}$ crystals in 240 - 280 nm spectral range is due to two processes: the two-step photoionization of Ce^{3+} ions and the single-step destruction (photoionization) of color centers. In turn the photoionization processes of Yb^{2+} ions do not contribute to the photoconductivity of samples in the investigated spectral range. Based on studies of photoconductivity of CaF_2, BaF_2 and SrF_2, doped with Yb^{2+} crystals [5], we can assume that in $SrAlF_5:Ce^{3+}$, $Yb3^{+}$ crystal the photoionization of Yb^{2+} ions is observed at shorter excitation wavelength.

In *conclusion*, the following results were formulated:

1. The results obtained by the methods of optical and dielectric spectroscopy are complement each other and allow us to adequately describe the photodynamic processes in activated dielectric crystals under intense UV irradiation and estimate the numerical values of basic parameters;

2. The nonmonotonic behavior of spectra of one- and multiphoton photoionization of Ce^{3+} ions in crystals $Y_3Al_5O_{12}:Ce^{3+}$, $LiCaAlF_6:Ce^{3+}$ and $Li_xLu_{1-x}F_4:Ce^{3+}$ (x = 0; 0,5; 1) is caused by the electronic transitions of activator ions to the state of mixed configurations, localized near or within the conduction band of the host material;

3. Co-activation of $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) and $SrAlF_6:Ce^{3+}$ crystals by Yb^{3+} ions reduces the concentration and the average lifetime of long-lived color centers; at the same time the significant changes of the lifetime of free electrons in the conduction band are not observed;

4. To increase the efficiency of lasing for $LiCaAlF_6:Ce^{3+}$ crystal it is desirable to use the pumping radiation with of wavelength greater than 270 nm and for $LiY_xLu_{1-x}F_4:Ce^{3+}$ (x = 0, 0.5, 1) - in the spectral range 290 - 300 nm.

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