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Low Temperature Luminescence Behavior of Trace Cr and Fe Impurities in $Gd_3Ga_5O_{12}$ Single Crystals

This article examines the influence of unintentional Cr^{3+} and Fe^{3+} impurity ions on the luminescent properties of $Gd_3Ga_5O_{12}$ (GGG) single crystals. The characteristic features of the spectra excited by high-energy synchrotron radiation in the temperature range of 10–300 K are analyzed. It is shown that at 10 K the luminescence is dominated by a narrow-band emission of Cr^{3+} ions arising from the spin-forbidden $^2E \rightarrow {}^4A_2$ transition, which indicates weak electron–phonon coupling and high crystalline homogeneity. It is revealed that with increasing temperature the intensity of this transition decreases significantly, while a broadband luminescence emerges, associated with the spin-allowed $^4T_2 \rightarrow {}^4A_2$ transition and the contribution of Fe^{3+} ion emission. The temperature evolution of the spectra is shown to result from thermal redistribution of the Cr^{3+} excited-state populations, interlevel state mixing, and partial removal of the spin-forbidden nature of Fe^{3+} transitions due to lattice vibrations. Based on the study, conclusions are drawn regarding the role of impurity centers in energy transfer and nonradiative relaxation processes. The results are of interest both for fundamental photonics and for the development of efficient luminescent materials and optical devices designed to operate over a wide temperature range.

Keywords: gadolinium gallium garnet (GGG), synchrotron radiation, Cr^{3+} ions, Fe^{3+} ions, luminescence, excitation spectra, temperature dependence, energy transfer, electron–phonon coupling, nonradiative relaxation, impurity centers, optical properties

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Introduction

Garnet crystals are widely used in laser technologies, optical sensors, and other photonic applications due to their unique luminescent properties [1–10]. Similar studies of luminescent materials with varying compositions have been reported, for example, for YAGG phosphors with different Al/Ga ratios, demonstrating how modification of crystal composition affects spectral and luminescent characteristics [11]. Chromium and iron are typical impurities that penetrate the lattice at various stages of synthesis. Even small amounts of Cr^{3+} or Fe^{3+} can significantly affect the spectroscopic and luminescent characteristics of the material, as confirmed by similar temperature-dependent luminescence studies in other garnet systems [12]. In GGG, Cr^{3+} exhibit a rich spectroscopic behavior determined by their $3d^3$ configuration in an octahedral field [13]. At the same time, the luminescent properties of Cr^{3+} are sensitive to a number of factors: temperature, concentration, local environment, and the presence of other impurities. In GGG, Cr^{3+} substitutes for Gd^{3+} in octahedral sites, forming characteristic spectra with sharp ($^2E \rightarrow {}^4A_2$) and broad ($^4T_2 \rightarrow {}^4A_2$) bands [14]. The authors of [14] demonstrated a sharp decrease in intensity upon heating (by 90–99 % at 300 K) due to the increased probability of nonradiative relaxations.

When Fe^{3+} and rare-earth ions are simultaneously present in the GGG structure, excitation transfer becomes possible, which can be enhanced with increasing temperature, thereby influencing the selectivity and luminescent properties [15]. In [16], it was shown that the spectral position of the deep-red emission band of Fe^{3+} can be modified by adjusting the crystal field strength (CFS). Moreover, it was demonstrated that the thermal response of Fe^{3+} luminescence can be significantly improved by incorporating Cr^{3+} ions, thus enabling thermally activated nonradiative energy transfer $Fe^{3+} \rightarrow Cr^{3+}$. The authors of [16] showed that reducing the CFS has a favorable effect on the performance of contactless temperature sensing in Fe^{3+}-based luminescent thermometers.

The study of the temperature dependence of the luminescence spectra of these materials is of crucial importance for understanding their optical characteristics and optimizing their applications. The aim of this work was to quantitatively separate and compare the contributions of unintentional impurity luminescence centers Cr^{3+} and Fe^{3+} to the integral luminescence of a $Gd_3Ga_5O_{12}$ single crystal under VUV excitation (160–250 nm) at temperatures of 10–300 K, as well as to determine the mechanisms of thermally controlled redistribution of impurity ion level populations and the energy transfer channels from the host lattice to the impurities.

Methods

The $Gd_3Ga_5O_{12}$ single crystals were grown by the Czochralski method using an iridium crucible. A mixture of argon (98 %) and oxygen (2 %) was used as the growth atmosphere. High-purity oxides Gd_2O_3 and Ga_2O_3 (99.99 wt. %) served as the starting materials. It should also be noted that GGG crystals contain a certain amount (10^{-3}–10^{-4} wt.%) of uncontrolled impurity ions, which is practically difficult to avoid. The samples were prepared in the form of flat plates 0.48 mm thick, oriented in the (111) plane with polished surfaces.

The luminescence studies were carried out using synchrotron radiation at the Superlumi/P66 beamline of the PETRA III synchrotron facility at DESY in Hamburg. This setup has proven to be highly effective for investigating luminescence processes excited in the VUV region [17–21].

The synchrotron radiation provided high-intensity, tunable excitation in the required spectral range, which was selected using a 2-meter monochromator with a spectral resolution of 0.4 nm. This ensured precise excitation of luminescent states in the single crystals. The emitted luminescence from the single crystals was detected using an ANDOR Kymera monochromator, which provided a spectral resolution of 0.2 nm, allowing for accurate detection of luminescence peaks. For sensitive detection over a wide spectral range, a Newton 920 CCD camera (Oxford Instruments, UK) was employed, while photon counting was performed using a Hamamatsu R6358 photomultiplier tube (Hamamatsu Photonics, Japan), particularly effective in the ultraviolet range.

Experiments were conducted at a temperature of 10 K using a helium-cooled cryostat to minimize thermal noise and nonradiative losses. The excitation spectra were corrected against the sodium salicylate signal, ensuring the acquisition of accurate and reliable data across the entire spectral range.

Results and Discussion

The excitation spectra of $Gd_3Ga_5O_{12}$ crystals measured at room temperature for emission wavelengths of 780, 754, and 694 nm (Fig. 1) exhibit characteristic features indicating the influence of unintentional Cr^{3+} and Fe^{3+} impurity ions in the crystal. For the 694 nm luminescence band, starting from approximately 5.5 eV, a sharp increase is observed in the excitation spectrum along with a relatively broad and smooth structure. This energy corresponds to the excitonic transition ($E_g = 5.66$ eV) [22], which is also associated with the excitation of unintentional impurity centers, primarily Cr^{3+} ions, through exciton migration.

In the region around 5 eV, when detecting at 754 nm and 780 nm, an additional peak is clearly visible, which can be unambiguously attributed to the presence of Fe^{3+} ions — a typical unintentional impurity often found in gallium garnets grown under standard conditions.

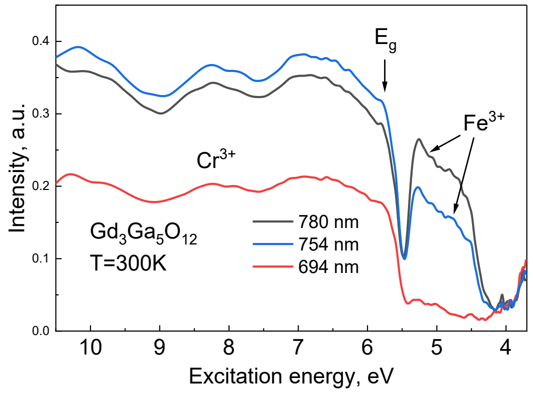

Figure 1. Excitation spectra of the $Gd_3Ga_5O_{12}$ crystal
measured at T = 300 K. λ_{reg} = 780 nm, λ_{reg} = 754 nm, and λ_{reg} = 694 nm

Analysis of the excitation spectra reveals a pronounced influence of Cr^{3+} and Fe^{3+} impurity ions, which manifest themselves as broad excitation bands in the 6–9 eV region and as a distinct peak around 5 eV, respectively. These impurities form additional levels within the band gap, facilitating uncontrolled energy trapping and affecting the luminescence characteristics.

The luminescence spectra of the GGG crystal excited by synchrotron radiation were studied at different temperatures. The emission spectra in the temperature range 300–10 K consist of a broad band spanning 650–870 nm (Fig. 2), which arises from the combined emission of two types of unintentional impurity centers. The component with a maximum around 730 nm is unambiguously associated with the ${}^4T_2 \rightarrow {}^4A_2$ transition of Cr^{3+} ions, whereas the band peaking at ≈ 775 nm is attributed to Fe^{3+} luminescence [23, 24]. To confirm this, luminescence spectra were recorded at 210 K under excitation at wavelengths $\lambda = 160$ nm and $\lambda = 250$ nm. In Figure 3, the band with a maximum at 775 nm is clearly distinguished, confirming the contribution of Fe^{3+}.

The temperature evolution of the luminescence spectra excited by high-energy quanta of 7.75 eV (160 nm) unambiguously reflects the different behavior of the two unintentional impurity centers — Cr^{3+} and Fe^{3+}. At temperatures below 210 K, the characteristic R-lines of Cr^{3+} begin to appear. At 10 K, the spectrum is characterized by structured lines in the 690–710 nm range, corresponding to the spin-forbidden ${}^2E \rightarrow {}^4A_2$ transition. As the temperature increases up to 300 K, the narrow structure is suppressed, and a broad symmetric luminescence band of 650–870 nm develops. This is associated with the dominance of the spin-allowed ${}^4T_2 \rightarrow {}^4A_2$ transition of Cr^{3+} ions and enhanced Fe^{3+} emission, indicating stronger thermal mixing of states and an increased contribution of electron–phonon interaction at higher temperatures.

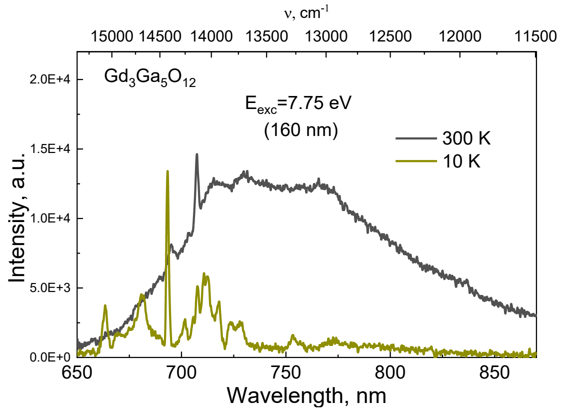

Figure 2. Temperature-dependent luminescence spectra of the GGG crystal

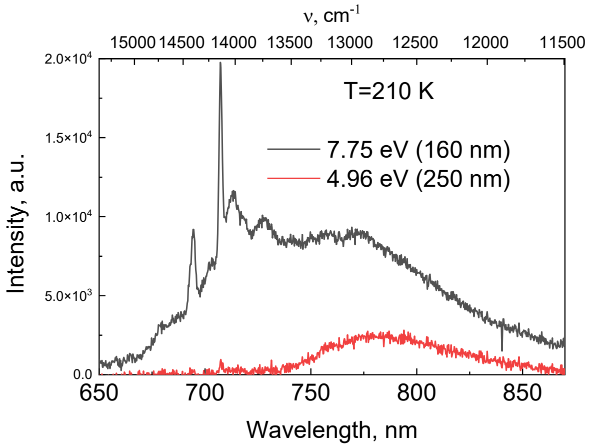

Figure 3. Luminescence spectra of GGG under 160 nm and 250 nm excitation

The experimental results can be explained as follows. Cr^{3+} ions in GGG are characterized by energy levels typical for d^3 ions: ${}^4A_2$, ${}^2E$, and ${}^4T_2$. Under excitation with 160 nm radiation in the VUV region, electronic excitations in the crystal matrix are captured by Cr^{3+} and Fe^{3+} impurity ions. Relaxation of Cr^{3+} occurs via two dominant channels: the narrow-band $^2E \rightarrow {}^4A_2$ and the broadband $^4T_2 \rightarrow {}^4A_2$. Their relative contribution depends on temperature and crystal parameters [25].

Subsequent energy transfer processes occur through phonon-assisted mechanisms and interlevel relaxation, leading to population of the excited states ^2E and $^4T_2$. At 10 K, the spectrum is characterized by structured lines in the 690–710 nm range, corresponding to the spin-forbidden $^2E \rightarrow {}^4A_2$ transition of Cr^{3+} ions occupying octahedral sites of the garnet lattice. The observed linewidth of the R-lines is only a few cm^{-1}, indicating high crystalline homogeneity of the material and weak electron–phonon coupling at low temperature. At cryogenic temperatures, relaxation predominantly terminates at ^2E, resulting in emission with a characteristic narrow spectral structure. Increasing temperature promotes a larger contribution from transitions through $^4T_2$, since thermal lattice expansion reduces the energy gap ΔE between the ^2E and $^4T_2$ levels and enhances their mixing.

In the excitation spectrum of luminescence monitored at 780 nm, a clear band with a maximum at ~5 eV (250 nm) is observed (Fig. 1). However, photons of this energy cannot directly produce emission at 750–780 nm; instead, the energy can be absorbed by defect centers and subsequently transferred to Fe^{3+} ions. Emission at 780 nm was excited only by the 250 nm excitation band, which may be related to charge-transfer transitions involving Fe^{3+} ions at different sites. The increase in luminescence intensity with rising temperature can be explained as follows. The $^4T_1 \rightarrow {}^6A_1$ transition in Fe^{3+} ions is spin-forbidden, since $\Delta S \neq 0$. The broad emission band in the 740–820 nm region has been shown in [26] to correspond to this internal transition of Fe^{3+}. The enhancement of intensity with increasing temperature is explained by the standard mechanism of vibrational and spin-orbit mixing, which grows with the amplitude of lattice vibrations and local symmetry distortions. As temperature increases, these vibrations become stronger, disrupting the local symmetry of Fe^{3+} ions. As a result, the efficiency of orbital mixing increases, leading to an enhanced quantum yield of the Fe^{3+} band. An additional contribution arises from competition with trap states: higher temperatures promote thermal ionization of defects, making Fe^{3+} a more effective acceptor of migrating excitonic energy.

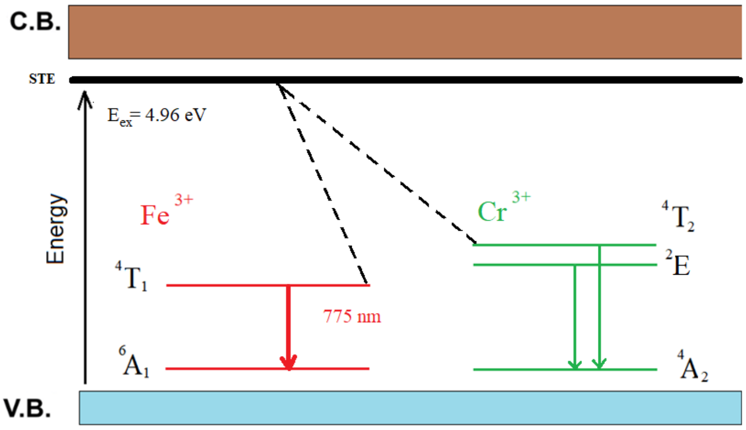

Figure 3. Cr^{3+} and Fe^{3+} centers schematic energy-level diagram in single crystal GGG

Conclusion

The luminescence spectra of unintentional Cr^{3+} and Fe^{3+} impurity ions in GGG under excitation by high-energy synchrotron radiation at 160 nm exhibit a pronounced and systematic temperature evolution. At cryogenic temperatures (~10 K), luminescence is dominated by narrow-band emission associated with the $^2E \rightarrow {}^4A_2$ transition of Cr^{3+} ions, characterized by high spectral resolution. With increasing temperature up to 300 K, a transition to broadband luminescence is observed, governed by the spin-allowed $^4T_2 \rightarrow {}^4A_2$ transition, accompanied by spectral broadening and faster dynamics. These changes are explained by mechanisms of energy transfer from defect centers, thermal redistribution of level populations, and enhanced electron–phonon coupling. The results are important both for understanding fundamental processes in luminescent materials and for the development of highly efficient optical devices operating under varying temperature conditions.

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$Gd_3Ga_5O_{12}$ монокристалдарындағы Cr және Fe қоспаларының төмен температуралы люминесценциялық қасиеттері

Мақалада $Gd_3Ga_5O_{12}$ (GGG) монокристалдарының люминесценциялық қасиеттеріне Cr^{3+} және Fe^{3+} иондарының еріксіз қоспаларының әсері қарастырылды. 10–300 K температура диапазонында жоғары энергиялы синхротрондық сәулелену мен қоздырылған спектрлердің сипаттық ерекшеліктері талданды. 10 K температурада люминесценция негізінен Cr^{3+} иондарының спинге тыйым салынған $^2E \rightarrow {}^4A_2$ ауысуынан туындайтын сызықты сәулелену мен анықталатыны көрсетілді, бұл электрон–фонондық байланысының әлсіздігін және кристалдың жоғары біртектілігін көрсетеді. Температура артқан сайын бұл ауысудың интенсивтілігі айтарлықтай төмендеп, кеңжолақты люминесценция пайда болады. Ол спинге рұқсат етілген $^4T_2 \rightarrow {}^4A_2$ ауысуымен және Fe^{3+} иондарының сәулеленуімен байланысты. Спектрлердің температуралық эволюциясы Cr^{3+} иондарының қозған күйлерінің жылулық қайта бөлінуімен, деңгей аралық күйлердің араласуы мен және тор тербелістерінің әсерінен Fe^{3+} ауысуларының спиндік тыйымының ішінара жойылуымен түсіндіріледі. Зерттеу негізінде энергияны тасымалдау және сәулеленбейтін релаксация процестеріндегі қоспа орталықтарының рөлі туралы қорытындылар жасалды. Нәтижелер іргелі фотоника үшін де, кең температура диапазонында жұмыс істейтін тиімді люминесценттік материалдар мен оптикалық құрылғыларды әзірлеу үшін де маңызды.

Кілт сөздер: гадолиний галлий гранаты (GGG), синхротрондық сәулелену, Cr^{3+} иондары, Fe^{3+} иондары, люминесценция, қозуспектрлері, температураға тәуелділік, энергия тасымалдау, электрон-фонондық байланыс, сәулеленбейтін релаксация, қоспаорталықтары, оптикалық қасиеттер

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Низкотемпературное поведение люминесценции примесей Cr и Fe в монокристаллах $Gd_3Ga_5O_{12}$

В данной статье рассматривается влияние непреднамеренных ионов-примесей Cr^{3+} и Fe^{3+} на люминесцентные свойства монокристаллов $Gd_3Ga_5O_{12}$ (GGG). Проанализированы характерные особенности спектров, возбуждённых высокоэнергетическим синхротронным излучением в температурном диапазоне 10–300 K. Показано, что при 10 K люминесценция определяется узкополосным излучением ионов Cr^{3+}, обусловленным спин-запрещённым переходом $^2E \rightarrow {}^4A_2$, что свидетельствует о слабой электрон-фононной связи и высокой однородности кристалла. Выявлено, что с повышением температуры интенсивность данного перехода существенно снижается, тогда как проявляется широкополосная люминесценция, связанная со спин-разрешённым переходом $^4T_2 \rightarrow {}^4A_2$ и вкладом излучения ионов Fe^{3+}. Доказано, что температурная эволюция спектров обусловлена тепловым перераспределением населённостей уровней Cr^{3+}, межуровневым смешением состояний, а также частичным снятием спинового запрета переходов Fe^{3+} за счёт колебаний решётки. На основе проведённых исследований сформулированы выводы о роли центров-примесей в процессах переноса энергии и безызлучательной релаксации. Полученные результаты представляют интерес как для фундаментальной фотоники, так и для разработки эффективных люминесцентных материалов и оптических устройств, функционирующих в широком температурном диапазоне.

Ключевые слова: гадолиний-галлиевый гранат (GGG), синхротронное излучение, ионы Cr^{3+}, ионы Fe^{3+}, люминесценция, спектры возбуждения, температурная зависимость, перенос энергии, электрон-фононное взаимодействие, безызлучательная релаксация, центры-примеси, оптические свойства

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