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Correspondence of the luminescence enhancement of Er^{3+} ions and the local electric field of Ag nanoparticles aggregates in oxide glasses

© V.V. Srabionyan, D.S. Rubanik, V.A. Durymanov, I.A. Viklenko, L.A. Avakyan, L.A. Bugaev

Faculty of Physics, Southern Federal University, Rostov-on-Don, Russia

e-mail: bugaev@sfedu.ru

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Using the oxide glasses doped with Er^{3+} ions with different refractive indices containing Ag nanoparticles (NPs) with widely variable average sizes and degree of agglomeration of NPs, a quantitative correspondence has been established between the average enhancement of the intensity of experimental photoluminescence (PL) of Er^{3+} ions and the calculated local electric field (LEF) in the vicinity of silver NPs agglomerates. It is shown that for Ag NPs with sizes $\gtrsim 5$ nm, the LEF enhancement is the dominant mechanism for enhancement of PL of RE ions. It has been established that the value of the average enhancement of the PL intensity of the RE ions in the studied glass can be obtained by calculation of LEF intensity for a „representative aggregate“consisting of rather small number of plasmonic NPs, with the structural parameters of the aggregate determined by transmission electron microscopy. The possibility of using such an aggregate for quantitative estimation of the PL of RE ions allowed to suggest that the main effect of the PL enhancement was due to those of the RE ions that were located in the areas of high density of NPs, or in the vicinity of particles agglomerates. To characterize an average agglomerate, a representative aggregate of NPs in the studied glass was introduced, which was characterized by its natural wavelength, determined by the average particle size, the minimum distance between them, and the refractive index of the glass. This wavelength is in good agreement with the position of the maximum in the experimental optical absorption spectrum of this glass. Using of the natural wavelength of a representative aggregate as an optical characteristic of the doped glass makes it possible to formulate optimal requirements for glass synthesis and for the choice of the exciting radiation wavelength, providing the most effective enhancement of the intensity of RE ions PL due to an increase in the intensity of LEF of plasmonic NPs.

Keywords: luminescence enhancement of Er^{3+} ions, local electric field of Ag nanoparticles aggregates, surface plasmon resonance, oxide glasses.

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1. Introduction

Enhancement of photoluminescence (PL) of rare-earth (RE) ions as a result of their interaction with nanoclusters (NCs) and nanoparticles (NPs) of plasmonic metals in glasses of various compositions has been studied extensively and remained a topical problem in photonics and optoelectronics in the past few years [1–3]. Studies of various mechanisms for enhancing the PL of RE ions interacting with NPs [4,5] have revealed that the enhancement of the local electric field (LEF) [6], which is caused by the localized surface plasmon resonance (LSPR) of particles that induces a multiple field enhancement in their vicinity where RE ions are located, is the dominant effect for particles ≥ 3 nm in size. The efficiency of this PL enhancement mechanism is determined by a number of factors, among which the composition and size distribution of nanoparticles, the degree of their agglomeration, the concentration of NPs and RE ions, and the correspondence between the energy range of LEF enhancement and the range of RE ion pumping deserve a mention [7]. The results of examination of the dependence of LEF intensity enhancement in the vicinity of a group of single-component (Ag, Au, and Rb) and bimetallic (AgAu and AgRb) NPs on their compositions and degrees of agglomeration were presented in [8] together with the proposed approach to calculating the LEF enhancement. Notably, LEF in the vicinity of a group of closely located particles was calculated with the use of structural units or aggregates of such NPs (the minimum number of interacting particles needed for theoretical description of their optical absorption spectrum) [9].

At the same time, the issue of quantitative correspondence between the calculated enhancement of intensity of LEF of plasmonic NPs and the experimentally observed enhancement of RE ion PL in glass remains open. The importance of establishing such a correspondence or the reasons for its violation stems, among other things, from the fact that, according to available estimates, the LEF intensity in the vicinity of Ag NP aggregates in oxide glasses may increase dozens of times depending on the listed factors [8], whereas just a several-fold enhancement of PL of ions (e.g., Er^{3+}) located in the vicinity of Ag NP aggregates in a number of oxide glasses was observed in experiments [10–12]. An understanding of the reasons for such discrepancies is an important and necessary condition for (1) formulating

the synthesis conditions needed to obtain NPs of the required size and degree of agglomeration in glass doped with plasmonic metals and RE ions and (2) choosing the optimum wavelength of exciting radiation that provides the maximum enhancement of both the LEF intensity in the vicinity of NP aggregates and the PL intensity of RE ions.

In the present study, we propose an approach to quantitative assessment of the average enhancement of PL intensity of RE ions in glass doped with RE ions and plasmonic metals based on the average level of LEF enhancement in the vicinity of plasmonic NPs calculated in accordance with the procedure detailed in [8]. The proposed approach is used to analyze the quantitative correspondence between the average enhancement of intensities of experimental Er^{3+} ion PL and the calculated LEF for aggregates of Ag NPs $\gtrsim 5$ nm in size in a number of oxide glasses. Dependences of the average enhancement of LEF intensity for aggregates of silver NPs in the glasses under consideration on the excitation wavelength are calculated. The obtained dependences are used to formulate the condition for choosing the most efficient excitation wavelength that provides the maximum enhancement of PL intensity of RE ions through the mechanism of LEF enhancement by plasmonic particles.

2. Average enhancement of intensity of LEF of NP aggregates and RE ion PL

The relation between the increase in intensity of luminescence of rare earth ions and the LEF enhancement by plasmonic particles is specified by Fermi's golden rule. According to this rule, the probability of photoabsorption by an atom or ion per unit time ($P_{init\to fin}$), which is accompanied by the excitation of an electron from the initial state ($init$) to the final one (fin), or the probability of pumping of an RE ion surrounded by a local field of N photons with frequency ω corresponding to LEF intensity $I_{LEF} = N\hbar\omega$ is written as

$$P_{init\to fin} = \frac{2\pi}{\hbar}|\langle\Psi_{fin}|\hat{V}|\Psi_{init}\rangle|^2 \times \rho(E_{init\to fin}), \quad (1)$$

where $\rho(E_{init\to fin})$ — density of photon states with energy $E_{init\to fin}$ corresponding to the electron transition in an RE ion from the initial core level with wave function Ψ_{init} to the final state — Ψ_{fin}, $\langle\Psi_{fin}|\hat{V}|\Psi_{init}\rangle$ — matrix element of such a transition induced by perturbation potential V of exciting photons. Using the expression

$$\langle\Psi_{fin}|\hat{V}|\Psi_{init}\rangle = CE_{init\to fin}\left(\frac{N}{\omega}\right)^{1/2}\langle\Psi_{fin}|\mathbf{ur}|\Psi_{init}\rangle$$

in the dipole approximation for the matrix element of the electron transition in an atom under the influence of the surrounding LEF, which is characterized by photon number N (C is a constant factor and $\mathbf{u}$ is a unit vector of polarization direction of exciting photons that may be used for averaging in accordance with the experimental conditions), one may determine that photoluminescence intensity $I \sim P_{init\to fin} \sim N$ or is proportional to the LEF intensity around the absorbing RE ion.

If the enhancement of absorption or pumping of RE ions through the enhancement of LEF of plasmonic NPs is dominant compared to other mechanisms of interaction of plasmonic NPs with RE ions in glass, it is fair to assume that the enhancement of PL intensity of an individual ion located in the vicinity of an NP aggregate at pump energy $E_{init\to fin}$ may be expressed as

$$\frac{I_{REI+NPs}(E_{init\to fin},\mathbf{R})}{I_{REI}(E_{init\to fin},\mathbf{R})} \approx \frac{I_{LEF(NPs)}(E_{init\to fin},\mathbf{R})}{I_{Incident}(E_{init\to fin})} = \frac{N(E_{init\to fin},\mathbf{R})}{N_{incident}(E_{init\to fin})}, \quad (2)$$

where $E = h\omega = 1239.8/\lambda$ is the energy of exciting photons with wavelength ($E \geq E_{init\to fin}$), $\mathbf{R}$ specifies the possible position of an RE ion in the vicinity of a plasmonic NP aggregate, $N_{incident}(E)$ is the number of photons with energy E incident on glass in unit time ($I_{incident} = N_{incident}\hbar\omega$ is the incident light intensity), and $N(E,\mathbf{R})/N_{incident}(E) = I_{LEF}(E,\mathbf{R})/I_{incident}(E)$ is the amplification of intensity of LEF with energy E at point $\mathbf{R}$.

The LEF in the vicinity of plasmonic NP aggregates was calculated on the basis of Maxwell's wave equations solved by the finite difference method [13]. Size-corrected corrections were introduced in accordance with [14] into the calculation of dielectric functions for NPs with sizes $D \leq 10$ nm. LEF calculations were performed for spherical particle aggregates, and the influence of the matrix of specific oxide glasses was introduced via experimental values of refraction index n.

According to expression (2), the LEF intensity is determined at points $\mathbf{R}$ (possible positions of RE ions); as a result, the obtained LEF distribution in the vicinity of plasmonic NP aggregates is poorly suited for quantitative assessment of the enhancement of PL of RE ions located in the vicinity of such aggregates. This is a byproduct of the common representation of the spatial LEF distribution in flat sections of particle localization regions (see the left panel of Fig. 1). Within this context, a method for quantitative assessment of the average LEF intensity enhancement (one of the most important characteristics for evaluating the efficiency of luminescent media), where the spatial distribution of LEF intensity amplification (ratio $N(E,\mathbf{R})/N_{incident}(E)$) was analyzed with the use of one-dimensional dependence $N(E,R_i)/N_{incident}(E)$ instead of flat sections, was proposed in [8]. Here, R_i is the distance corresponding to all points equidistant from the surfaces of particles closest to these points (i is the number of a set of such points with the same R_i), which is shown schematically in the left panel of Fig. 1. This ratio was obtained by averaging over all spatial points R_i the possible positions

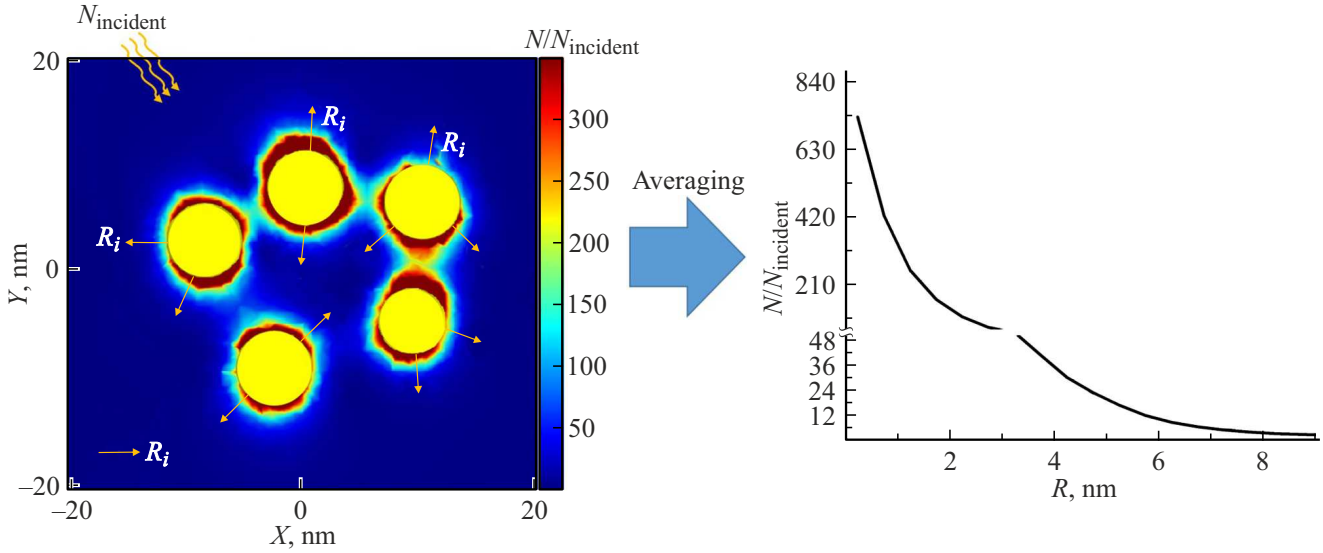

Figure 1. Distribution of the calculated enhancement of LEF intensity in the vicinity of an aggregate of Ag nanoparticles $D = 10$ nm in size in the form of flat sections (left panel) and in the form of averaged dependence $N(R_i)/N_{incident}$, where R_i is the distance from the possible positions of a hypothetical RE ion to the surfaces of the nearest particles [8].

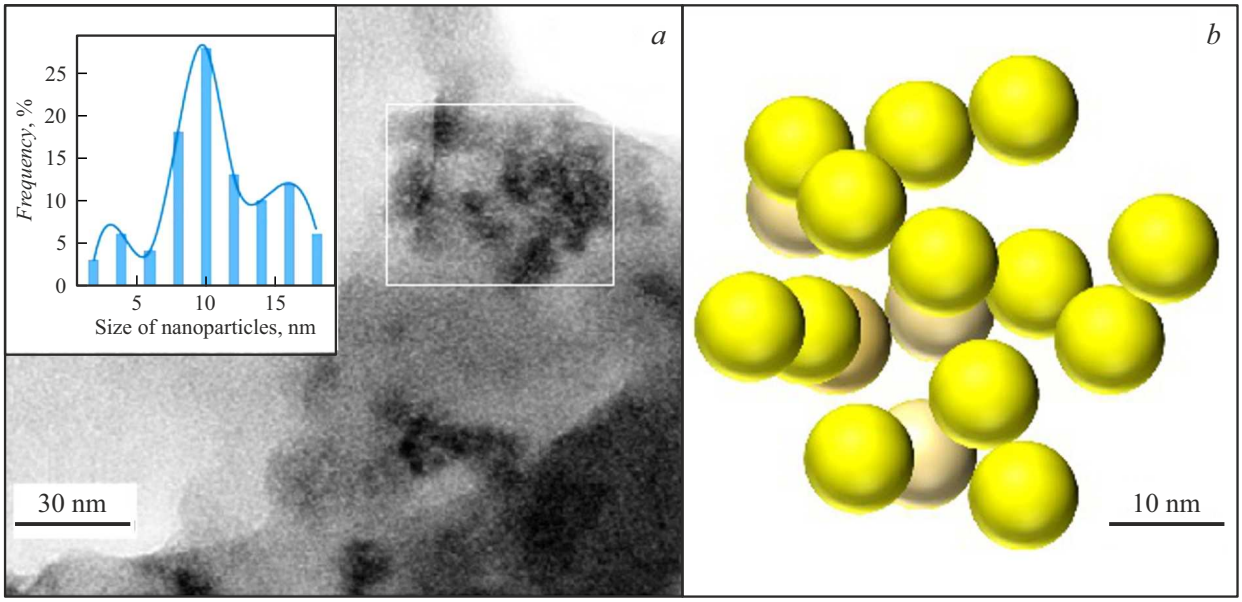

Figure 2. (a) TEM image of zinc-containing oxide glass with single Ag nanoparticles and their agglomerates; the area used for agglomerate modeling is highlighted by a white rectangle. (b) Ag NP aggregate model (aggregate 1). The particle size distribution is also shown in the inset in the upper left corner (a).

of hypothetical RE ions located at the same distance $R_i \pm 0.25$ nm from the surface of plasmonic particles closest to such points, where 0 on the R_i distance axis corresponds to the surface of each particle in the aggregate (right panel of Fig. 1).

Probability density function $w(R_i)$ of finding RE ions at point R_i was obtained via Monte Carlo simulations under the assumption of an uncorrelated uniform distribution of RE ions in the vicinity of particle aggregates. In this scheme, the number of points with a certain distance R_i from the particle surface was calculated using a fixed-width histogram step $\Delta R_i \sim 0.5$ nm, and $w(R_i)$ was estimated as the fraction of sites of hypothetical positioning of RE ions at a certain distance R_i in region ΔR_i in the periodicity bin. To estimate the average enhancement of the experimental PL intensity of RE ions based on the average enhancement of LEF intensity for plasmonic NP aggregates calculated in accordance with the procedure described above, expression (2) was

generalized and rewritten as

$$\frac{I_{REI+NPs}(E_{init\to fin})}{I_{REI}(E_{init\to fin})} \approx \frac{I_{LEF(NPs)}(E_{init\to fin})}{(I_{incident}(E_{init\to fin})} = \frac{\sum_i \left(\frac{N(E,R_i)}{N_{incident}(E)}\right)\cdot w(R_i)\cdot \Delta R_i}{\sum_i w(E,R_i)\Delta R_i}. \quad (3)$$

3. Construction of a representative NP aggregate for calculating the average LEF enhancement

A 3D model of a representative aggregate of NPs consisting of a minimum number of them (on the order of 10–15) sufficient for correct assessment of LEF in the vicinity of NPs was used to obtain the spatial LEF distribution in glass [9]. Dependences of the average increase in LEF intensity on the characteristics of aggregates (particle sizes, interparticle distances) were determined this way for various configurations of NPs in the aggregates [8], and it was established that a more than 1.5-fold LEF intensity enhancement is observed in regions with an increased density of NPs.

Representative aggregates were obtained from transmission electron microscopy (TEM) data for the samples. It appears extremely important in this context to develop a methodological approach aimed at the construction of representative models of aggregates that have the capacity to reproduce reliably the structural features of the observed agglomerates. This approach should combine sufficient simplicity of implementation and a high degree of correspondence to experimental TEM data.

A technique for constructing an aggregate of at least 10 particles of the same size D, which are distributed uniformly in space in such a way that the distance between the centers of particles is no shorter than R_{ij}, was proposed for this purpose. The mentioned interparticle distance was set via parameter $\alpha > 1$ in such a manner that $R_{ij} = \alpha D$, particle size D corresponded to the average size of an NP set determined by analyzing TEM images, and minimum distance R_{ij} was estimated by visual analysis of the agglomerates present in these images.

An agglomerate of Ag NPs in zinc-tellurite glass with $n = 2.36$ [10], which is the most characteristic of this sample, is shown in the upper right corner of Fig. 2, *a*. Figure 2, *b* presents the corresponding model of the Ag NP aggregate (aggregate 1) constructed using the proposed method. The average LEF intensity amplification factor calculated with this aggregate for each possible position of a hypothetical RE ion was 4.6 ± 0.4 (see the first row of the table).

To analyze the influence of errors associated with the selection of minimum distance R_{ij} on the calculated LEF intensity in the vicinity of NP aggregates, the particle distribution was altered by varying parameter α by ± 0.1 with respect to the values of α determined from the TEM micrographs of the corresponding NP agglomerates. For example, the stability of the calculated LEF intensity enhancement against the α parameter choice for silver NP aggregate 1 shown in Fig. 2, which was constructed with $\alpha = 1.2$, was checked at α values varying from 1.1 to 1.3. The results of calculations demonstrated that the corresponding changes in the average increase in LEF intensity for the studied particle aggregates do not exceed 10%, which is indicative of stability of the model to α variations.

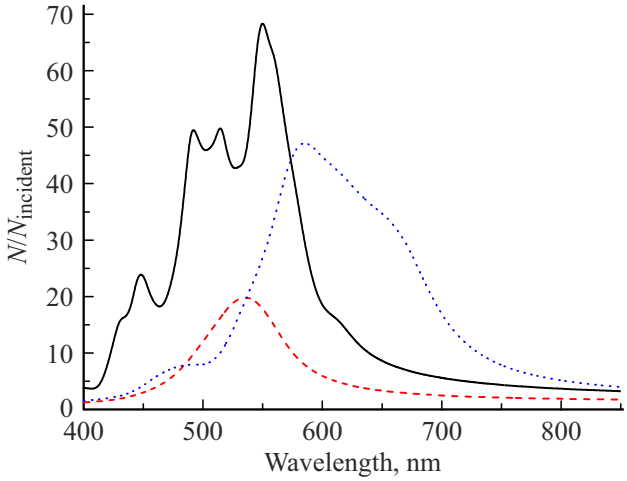

Figure 3. Calculated dependences of the average enhancement of intensity of LEF corresponding to a hypothetical RE ion in the vicinity of aggregates of Ag nanoparticles on exciting wavelength λ_{exc} varying from 400 to 850 nm: for aggregate 1 in glass with $n = 2.36$ (black solid curve), aggregate 2 with $n = 2.03$ (red dashed curve), and aggregate 3 with $n = 1.60$ (blue dotted curve).

4. Average enhancements of the experimental PL intensities of Er^{3+} ions and the calculated LEF in the vicinity of Ag NP aggregates in glasses

The method for obtaining a model of NP aggregates from TEM micrographs for the calculation of LEF enhancement in the vicinity of NPs was tested using experimental PL data for Er^{3+} in various glasses with the (Er^{3+}+ Ag nanoparticles)/glass configuration and TEM data. In these tests, we estimated the enhancement of PL of RE ions due to aggregates of Ag NPs with size $D > 5$ nm. According to [1,8,12], the mechanism of LEF in the vicinity of NPs should be dominant at such sizes. This study confirmed that the enhancement of LEF intensity in the vicinity of NP aggregates has a dominant role in the enhancement of PL of RE ions and that the proposed approach to calculating LEF in the vicinity of Ag NPs is applicable. In addition, it provided an opportunity to establish the excitation conditions for the optimum enhancement of PL

Experimental average increase in PL intensity for glasses containing Er^{3+} ions with refractive index n and average size D of Ag nanoparticles in composite glasses according to the data from [10–12] and average increase in LEF intensity for these glasses calculated in accordance with [8] for different exciting wavelengths λ_{exc}

Number	Composition (Er^{3+}+Ag NPs)/glass, Designation of an aggregate of Ag NPs	Experimental data			Calculated average LEF intensity enhancement $I_{LEF(NPs)}/I_{incident}(\pm 7\%)$		
		Refraction index n	Average NP size, D, nm	Average PL intensification transition Exciting	$\lambda_{exc} = \lambda_{exc}(exper.)$,	$\lambda_{exc} = \lambda_{exc}(Er^{3+}({}^4I_{15/2} \rightarrow {}^4F_{7/2})$ (488 nm)	$\lambda_{exc} = \lambda_{max}$
1	(Er^{3+}+Ag/(zinc-) /tellurite glass) [10] Aggregate-1	2.36	10	4.5 (786 nm) ${}^4I_{15/2} \rightarrow {}^4I_{9/2}$	4.6 (786 nm)	8.4	47.2 580 mm
2	(Er^{3+}+Ag)/ (tellurite glass) [12], Aggregate-2	2.03	10	15 (980 mm) ${}^4I_{15/2} \rightarrow {}^4I_{11/2}$	1.6 (980 mm	8.8	19/7
3	(Er^{3+}+Ag)/ (phosphate glass) [11], Aggregate-3	1.60	35	2.1 (797 mm)	2.3 (797 mm)	36.7	56.1 (528 nm)

of RE ions and identify the limitations on applicability of the quantitative correspondence between the enhancement of LEF of plasmonic NP aggregates and the enhancement of PL of RE ions located near them.

The results of comparison of average enhancements of the experimental PL intensity ($I_{REI+NPs}/I_{REI}$) of Er^{3+} ions in the examined glasses [10–12] obtained at exciting wavelength $\lambda_{exc}(exper)$ with the average enhancements of LEF intensity ($I_{LEF(NPs)}/I_{incident}$) calculated using formula (3) at exciting wavelengths $\lambda_{exc} = \lambda_{exc}(exper)$ from reference experiments, $\lambda_{exc} = \lambda_{Er^{3+}}({}^4I_{15/2} \rightarrow {}^4F_{7/2}) = 488$ nm (corresponds to the $4f-4f$ transition in Er^{3+} ions and Ar^+ laser radiation), and $\lambda_{exc} = \lambda_{max}$ (the position of the maximum of average LEF intensity enhancement) are presented in the table. The experimental data used to determine particle size D and refraction index n are also listed in the left part of the table.

According to the results presented in the table, the calculated average values of LEF intensity enhancement correspond to the average values of enhancement of Er^{3+} ion PL at the exciting wavelengths used in [10–12] with an error less than $\sim 10\%$.

The results of theoretical calculations listed in the middle column of the right part of the table demonstrate that the excitation of (Er^{3+}+Ag)/glass samples at a wavelength of 488 nm (Ar laser radiation) should lead to a significant increase (varying with particle size to a maximum of ~ 37-fold enhancement) increase in LEF intensity near Ag NP aggregates. This effect is made possible by the fact $\lambda_{exc} = 488$ nm falls within the wavelength range of significant LEF enhancement near Ag NP aggregates for the examined samples (Fig. 3). Therefore, almost the same enhancement of PL of Er^{3+} ions in the corresponding glasses is to be expected, since $\lambda_{exc} = 488$ nm matches the exciting wavelength for the ${}^4I_{15/2} \rightarrow {}^4F_{7/2}$ pump transition of an Er^{3+} ion.

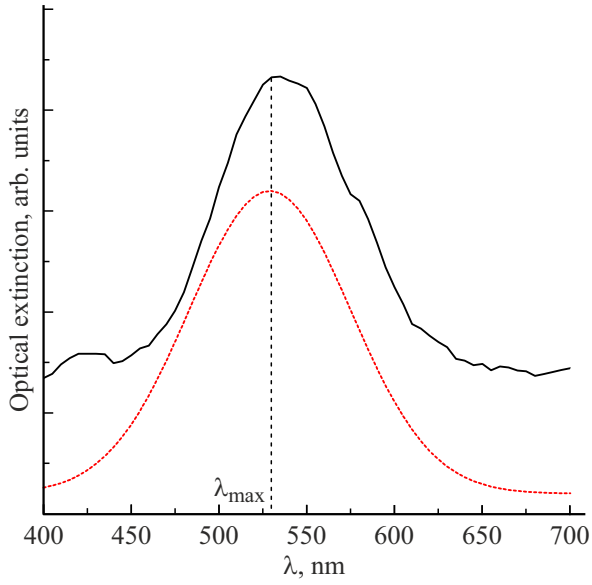

Figure 4. Comparison of the experimental extinction spectrum of the (Er^{3+}+Ag)/(phosphate glass) sample [11] (black solid curve) with the theoretical extinction spectrum calculated using representative Ag NP aggregate 3 (red dotted curve) characterized by intrinsic wavelength λ_{max}, which is indicated on axis λ.

The last column of the table presents the maxima of average LEF intensity enhancement (Fig. 3) for the used representative NP Ag aggregates and the wavelengths corresponding to them. It follows from these results and our earlier calculations [8] that the indicated LEF characteristics in the studied composite glasses are determined by the average values of parameters D, R_{ij} of particle agglomerates and refraction index n of glass, which are controlled by adjusting the parameters of material synthesis. Wavelength $\lambda_{\max}$ corresponding to the maximum amplification is also a function of these parameters D, R_{ij}, and n.

It should be emphasized that the $\lambda_{\max}$ values (Ag NP aggregate) for the curves in Fig. 3 are consistent with the spectral position of the absorption maxima of the corresponding composite glasses [10–12]. This is illustrated in Fig. 4, where the experimental absorption spectrum of the $(Er^{3+}+Ag)$/(phosphate glass) sample [11] is compared with the theoretical spectrum calculated using representative aggregate 3 of Ag NPs. The obtained correspondence confirms that the proposed method is applicable in construction of a representative aggregate of interacting NPs in glasses. Specifically, this agreement verifies the used scheme for selecting the minimum distance between particles R_{ij} and provide an opportunity to fine-tune its value (within $\pm 10\%$ of R_{ij}) for constructing an aggregate, since the other two parameters (D and n) affecting $\lambda_{\max}$ are determined quite accurately.

The results of calculations and comparison with the experimental data in the table suggest that the relatively small increase in Er^{3+} PL intensity in the experiments under consideration is attributable to significant differences in the positions of LEF intensity maxima in the vicinity of representative aggregates $\lambda_{\max}$ (Ag NP aggregate) for the studied glasses, which were determined based on the results of synthesis [10–12], and the λ_{exc} values of exciting radiation used in these studies. The latter were chosen in accordance with the main pump wavelengths $\lambda_{exc}(Er^{3+})$ without account for the spectral features of the representative aggregate of nanoparticles in a specific glass. As a result, they turned out to be far from the values of $\lambda_{\max}$ (Ag NP aggregate) for each glass. The obtained results allow us to conclude that the following condition must be fulfilled in order to maximize the enhancement of the average intensity of RE ion PL by the mechanism of LEF amplification in the vicinity of aggregates of plasmonic NPs in which these ions are positioned:

$$\lambda_{exc} \approx \lambda_{\max} \approx \lambda_{exc}(\text{RE ions}). \quad (4)$$

This condition may be satisfied by combining the proper choice of exciting wavelength λ_{exc}, the synthesis conditions leading to changes in the composition and agglomeration of particles in glass, and the use of additional temperature and laser processing techniques that ensure the production of glasses containing plasmonic NPs with the required average size and degree of agglomeration.

The established quantitative correspondence between the average enhancements of intensities of the experimental PL of an RE ion and the calculated LEF for a representative aggregate of plasmonic NPs in the studied glass may be disrupted by the effects of PL quenching with an increase in concentration of both plasmonic NPs and RE ions. The latter effect has been examined experimentally in detail and may be neglected if one chooses RE ion concentrations up to ~ 1 mol% [15], whereas the factors influencing the quenching of PL of RE ions due to an increased concentration of plasmonic NPs warrant further study.

5. Conclusion

A series of Er-doped oxide glasses with different refraction indices containing Ag NPs with widely varying average sizes and degrees of agglomeration was used as an example to reveal a quantitative correspondence between the average enhancements of intensity of the experimental PL of Er^{3+} ions and the calculated LEF in the vicinity of silver NP agglomerates. On the one hand, this correspondence confirms the assumption that the LEF amplification is the main mechanism for enhancing RE ion PL for NPs ≥ 5 nm in size. On the other hand, it allows one to estimate quantitatively the average enhancement of PL intensity of RE ions by performing a relatively simple electrodynamic calculation of LEF amplification in the vicinity of plasmonic NP agglomerates. The established possibility of reproducing the value of average PL enhancement of RE ions by calculating the LEF intensity for a „representative aggregate“ consisting of a small number of plasmonic NPs with its structural parameters chosen based on the results of TEM glass measurements suggests that the primary contribution to PL enhancement is produced by those RE ions that are located in the regions of increased NP density or in the vicinity of their agglomerates. The representative NP aggregate used for the examined glass is characterized by intrinsic wavelength $\lambda_{\max}$, which is determined by the average particle size, the minimum distance between them, and the refraction index of glass, and agrees closely with the position of the maximum in the experimental optical absorption spectrum of this glass. The use of such an intrinsic wavelength as an optical characteristic of doped glass allows one to formulate the optimum requirements for (1) the synthesis of glass and its targeted adjustment aimed at obtaining the closest match between $\lambda_{\max}$ and the pumping wavelength of RE ions and (2) the selection of exciting wavelength λ_{exc} providing the most efficient enhancement of PL intensity of RE ions due to the enhancement of LEF intensity by plasmonic NPs.

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Conflict of interest

The authors declare that they have no conflict of interest.

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