NONRADIATIVE TRANSITIONS BETWEEN LEVELS OF TRIVALENT RARE-EARTH IONS IN YTTRIUM-ALUMINUM GARNET CRYSTALS

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Nonradiative transitions between levels of Nd³⁺, Er³⁺, Ho³⁺, and Tm³⁺ ions in yttrium-aluminum garnet crystals are studied. The temperature dependences of the probabilities for nonradiative transitions are studied and it is shown that in the single-frequency model approximation the optical phonons with energy ~700 cm⁻¹ play the predominant role. The spontaneous nonradiative transition probabilities for various energy gaps between the levels and the dependence of the nonradiative transition probability on the energy gap are determined by measuring the quantum yield, lifetime, and kinetics of luminescence at 77°K. For gaps lying between 1200 and 5000 cm⁻¹, the dependence can be satisfactorily approximated by an exponential law. The relaxation times from the major excitation levels to the ⁴F_{3/2} level are measured for Nd³⁺ ions excited by radiation from a ruby or rhodamine-6G laser or by the second harmonic from a neodymium laser. The measurements are based on the kinetics of the luminescence emitted by the Nd³⁺ ions from the ⁴F_{3/2} level. The relaxation time from levels with an energy ~18 800 cm⁻¹ is 5×10^{-7} sec. For levels lower than 18 800 cm⁻¹, the time does not exceed 5×10^{-8} sec. On the basis of the dependence of the nonradiative transition probabilities on the energy gap between the levels, the relaxation time between the ⁴I_{11/2} and ⁴I_{9/2} levels of the Nd³⁺ ions is estimated at 5×10^{-7} sec.

NONRADIATIVE transitions between levels of impurity ions in crystals are one of the manifestations of the interaction between the ion and the crystal lattice. Such transitions can lead to a decrease of the intensity and to a reduction of the lifetime of the luminescence of the ions, and can also exert a noticeable influence on the operation of a laser using activated crystals.

The theory of nonradiative transitions between levels of impurity ions in crystals was developed in^[1,2]. Nonradiative transitions between levels of trivalent rare-earth ions (TR-ions) were investigated experimentally in detail for the crystals LaBr₃, LaCl₃, LaF₃, SrF₂, and $Y_3O_3^{[3-10]}$. It was shown^[10] that for TR ions the rates of nonradiative transitions are determined by the structure of the phonon spectrum, namely, by the limiting frequencies of the optical phonons in the vibration spectrum of the crystals. It turned out that in the region of low temperatures $(4.2-77^{\circ}K)$ in a given crystal lattice the dependence of the probability of nonradiative transitions on the energy gap between the levels can be well approximated by an exponential law and the value of the probability for the given energy gap remains practically unchanged when the TR ion is replaced. It should be noted that the crystal having the highest-frequency oscillations was Y_2O_3 (550 cm⁻¹)^[7]. For these crystals, the values of the probabilities of nonradiative transitions between the levels of TR ions turned out to be higher than in LaCl₃, LaBr₃, LaF₃, and $SrF_2^{[10]}$.

We have investigated the nonradiative transitions between levels of the ions Nd^{3+} , Er^{3+} , Ho^{3+} , and Tm^{3+} in crystals of yttrium aluminum garnet $Y_3Al_5O_{12}$ (YAG), which is widely used as a laser active material^[11]: Experiments on the temperature quenching of luminescence of the TR ions yielded the temperature dependence of the nonradiative transition probabilities and showed that optical phonons with energy ~ 700 cm⁻¹ play the decisive role in processes of nonradiative transitions between the levels of the TR ions in the YAG crystals in the single-particle-model approximation.

From measurements of the quantum yield, lifetime, and the flareup of the luminescence, we determined the values of the probabilities of nonradiative transitions of TR ions for different energy gaps between the levels. In the time measurements, the luminescence was excited by a ruby laser, the second harmonic of a neodymium laser, and by lasers based on organic-dye solutions operating in the Q-switched regime. Using excitation of the Nd³⁺ ions by the second harmonic of a neodymium laser at levels with energy $\sim 18\ 800\ {\rm cm}^{-1}$, the flare-up of the luminescence from the ${}^{4}F_{3/2}$ level was used to measure the relaxation time from one of the pump ground levels to the upper laser level, which amounts to 5×10^{-7} sec. The times of relaxation from other pump levels lying below $\sim 18\ 800\ {\rm cm}^{-1}$ turned out to be shorter than 5×10^{-8} sec.

The investigations yielded the dependence of the probability of nonradiative transitions on the energy gap between the levels of the TR ions in the YAG crystals. For gaps in the interval $1200-5000 \text{ cm}^{-1}$, this dependence is well approximated by an exponential law. The obtained dependence has made it possible to estimate the time of relaxation between the levels ${}^{4}I_{11/2}$ (the lower laser level) and ${}^{4}I_{9/2}$ (the ground state) of the Nd³⁺ at ~ 5 × 10⁻⁷ sec.

INVESTIGATION PROCEDURE AND EXPERIMENTAL RESULTS

The measurements of the probabilities of the nonradiative transitions and the investigation of the temperature quenching of the luminescence were carried out on YAG crystals activated with the ions Er^{3+} , Nd^{3+} , Ho^{3+} , and Tm^{3+} , grown by spontaneous crystallization from the solution in the melt and by the Czochralski method from raw material of high degree of purity. The concentration of the active ions in the crystals was 0.5 at.%. As shown earlier^[12], at such activator concentrations the concentration quenching of the luminescence can be neglected.

1. Temperature Quenching of the Luminescence of the Ions Nd³⁺, Er³⁺, Ho³⁺, and Tm³⁺

To determine the temperature dependence of the probability of nonradiative transitions between levels of the TR ions in the YAG crystals, we measured the luminescence lifetime τ of the most characteristic transitions from the level ${}^{4}F_{3/2}$ of the Nd³⁺ from the level ${}^{4}I_{13/2}$ of the ions Er³⁺, from the ${}^{5}I_{7}$ level of Ho³⁺, and from the ${}^{3}H_{4}$ level of Tm³⁺ (Fig. 1) in the temperature interval 77–900°K. The measurements of τ were carried out with continuous variation of the temperature, using the procedure described in^[12].

Figure 2 shows plots of the luminescence lifetime of the ions Nd^{3+} , Er^{3+} , Ho^{3+} , and Tm^{3+} against the temperature. We see that the τ of Nd^{3+} and Er^{3+} remain unchanged with increasing temperature. The luminescence lifetimes of Ho^{3+} and Tm^{3+} remain unchanged in the temperature interval $77-450^{\circ}$ K, and with further increase in temperature they begin to shorten and change from 8.5 msec (Tm^{3+}) and 6.5 msec (Ho^{3+}) to 2.5 and 3 msec, respectively, at 900°K.

Dependence of the Probability of Nonradiative Transitions Between the Levels of the Ions Nd³⁺, Er³⁺, Ho³⁺, and Tm³⁺ on the Energy Gap Between the Levels

At 77° K, we obtained the values of the probabilities of the spontaneous nonradiative transitions between several levels of TR ions with a set of different energy gaps ΔE between them.

The ions Tm^{3+} in YAG (transition ${}^{3}\text{H}_{4}$ -- ${}^{3}\text{H}_{6}$, ΔE = 4947 cm⁻¹) and the ions Ho³⁺ in YAG (transition ${}^{5}\text{I}_{7}$ - ${}^{5}\text{I}_{8}$, ΔE = 4695 cm⁻¹).

Radiative transitions from the levels ${}^{3}H_{4}$ of Tm³⁺ and from the levels ${}^{5}I_{7}$ of Ho³⁺ in YAG crystals are observed to the levels ${}^{3}H_{6}$ and ${}^{5}I_{8}$ (luminescence in the region of $\sim 2\mu$)^[13]. The luminescence lifetimes τ of the ions Tm³⁺ and Ho³⁺ can be shortened by the nonradiative transitions and are connected with the proba-



FIG. 1. Energy level schemes of the ions Nd^{3+} , Er^{3+} , Ho^{3+} , and Tm^{3+} . FIG. 2. Temperature dependence of the luminescence lifetime of the ions $Tm^{3+}(\bullet)$, $Er^{3+}(O)$, $Ho^{3+}(\Delta)$ and $Nd^{3+}(X)$, right-hand scale) in YAG crystals.

bilities of nonradiative transitions ${\rm W}_{nr}$ and radiative transitions ${\rm W}_{r}$ by the relation

$$\tau^{-1} = W_r + W_{nr}. \tag{1}$$

We measured the luminescence lifetimes of the ions Tm^{3+} and Ho^{3+} at $77^{\circ}K$, which turned out to equal 8.5 and 6.5 msec, respectively. To determine the probabilities of the radiative transitions between the levels ${}^{3}H_{4}$ and ${}^{3}H_{6}$ of Tm^{3+} and between ${}^{5}I_{7}$ and ${}^{5}I_{8}$ of Ho^{3+} , we calculated the oscillator strengths $F_{1,2}$ of these transitions. The calculation was based on the Kravetz formula

$$f_{1,2} = \frac{c^2 m}{\pi N e^2} \int k\left(\frac{\nu}{c}\right) d\left(\frac{\nu}{c}\right), \qquad (2)$$

where N is the activator concentration and $k(\nu/c)$ is the absorption coefficient. The quantity $\int k(\nu/c) d(\nu/c)$ was estimated from the absorption spectra obtained with the aid of a two-beam spectrophotometer SP-700 at room temperature. The resolution in the registration of the spectra was not worse than 5 cm⁻¹.

The levels of the ions Tm^{3*} and Ho^{3*} consist of a number of Stark components, and the total probability of the radiative transitions is determined by the sum of the probabilities of the transitions between the individual Stark components. At the calculation of the probabilities of the radiative transitions on the basis of the values of the oscillator strengths obtained from the absorption spectra at $T = 300^{\circ}$ K, we took into account the probability of finding the particles at the corresponding Stark sublevels of the levels ${}^{3}H_{6}$ and ${}^{5}I_{8}$.

The values obtained by us for the probabilities of the radiative transitions were $\sim 130 \text{ sec}^{-1}$ for Ho³⁺ and $\sim 100 \text{ sec}^{-1}$ for Tm³⁺. The probability of the nonradiative transitions determined from formula (1) is 20 sec⁻¹ for both ions. The accuracy with which W_{nr} was determined does not exceed 50%.

 $= \frac{\text{The ions } Er^{3+} \text{ in YAG (transition } {}^{4}S_{3/2} - {}^{4}F_{9/2}, \Delta E}{2870 \text{ cm}^{-1}}.$

Radiative transitions are observed in YAG crystals from the ${}^{4}S_{3/2}$ level of the Er^{3+} ions to the levels ${}^{4}I_{13/2}$ (luminescence in the region of 0.86 μ) and ${}^{4}I_{15/2}$ (luminescence in the region of 0.54 μ)^[14]. The luminescence from the level ${}^{4}S_{3/2}$ in the YAG crystals is strongly quenched by the nonradiative transitions to the ${}^{4}F_{9/2}$ level, and their quantum yield is small. Therefore the observed lifetime of the level ${}^{4}S_{3/2}$ is determined by the probability of the nonradiative transitions to the level ${}^{4}F_{9/2}$.

In measuring the luminescence lifetime, the excitation source was a laser using an organic dye solution (sodium eosin) with radiation wavelength 0.542 μ and a pulse duration ~ 10 nsec. The energy in the pulse was ~ 0.02 J. The luminescence in the 0.86 μ region was registered with the aid of a photomultiplier on an oscilloscope screen. The time resolution of the receiving apparatus was 50 nsec. The lifetime of the ${}^{4}S_{3/2}$ level, measured at T = 77°K, was 100 ± 20 μ sec.

We estimated the quantum yield of the luminescence of the Er^{3+} ions from the ${}^{4}\mathrm{S}_{3/2}$ level. In the region of 0.86 μ it was determined from the ratio to the quantum yield of the luminescence of Nd³⁺ and YAG crystals in the region of 0.9 μ , which according to^[15] amounts to 0.25. In the case of luminescence of the Er^{3^+} ions in the 0.54 μ region, the comparison was made with rhodamine-6G, the quantum yield of which is close to unity. As shown by estimates, the total quantum yield of the luminescence of the Er^{3^+} ions from the ${}^4\mathrm{S}_{3/2}$ level in YAG crystals does not exceed 0.1. From the level ${}^4\mathrm{S}_{3/2}$ of the Er^{3^+} ions there is observed also luminescence to the level ${}^4\mathrm{I}_{13/2}$ (1.2 μ). In YAG crystals, however, the intensity of this luminescence is much lower than the intensity of the luminescence in the regions of 0.54 and 0.86 μ .

The probability of nonradiative transitions, determined from the values of the lifetimes and from the quantum yield of the luminescence from the ${}^{4}S_{3/2}$ level, is $(9 \pm 2) \times 10^{3}$ sec⁻¹.

The ions Nd³⁺ in YAG (transition ${}^{4}G_{7/2} - {}^{2}G_{7/2}$ + ${}^{4}G_{5/2} \Delta E = 1200 \text{ cm}^{-1}$)

In the case of Nd³⁺ in YAG, radiative transitions are observed from the level ${}^{4}F_{3/2}$ to the levels ${}^{4}I_{9/2--15/2}$ (luminescence in the region 0.9-1.8 μ)^[16]. When excited to levels lying above ${}^{4}F_{3/2}$, the neodymium ions experience nonradiative relaxation to the level ${}^{4}F_{3/2}$.

At temperatures 77 and 300°K, we measured the times of relaxation from the levels ${}^{4}G_{7/2}$ (~18 800 cm⁻¹), ${}^{2}G_{7/2} + {}^{4}G_{5/2}$ (~17 300 cm⁻¹), and ${}^{4}F_{9/2}$ (~14 400 cm⁻¹) to the level ${}^{4}F_{3/2}$ on the basis of the flare-up of luminescence from the level ${}^{4}F_{3/2}$ (Fig. 1).

The luminescence of the Nd³⁺ ions at the indicated level was excited by the second harmonic of a neodymium laser (${}^{4}G_{7/2}$), by a rhodamine-6G laser (${}^{2}G_{7/2} + {}^{4}G_{5/2}$), and by a ruby laser (${}^{4}F_{9/2}$) with a pulse duration not exceeding 20 nsec. The luminescence signal in the region of 0.9 μ was registered with a photo-multiplier on an oscilloscope screen. The time resolution of the receiving apparatus was 50 nsec. The luminescence decay of the level ${}^{4}F_{3/2}$ of Nd³⁺ introduced no errors in the measurement of the luminescence flare-up time, since the lifetime of the ${}^{4}F_{3/2}$ level is 240 μ sec.

Measurements of the relaxation time were carried out on YAG crystals with a neodymium content 0.5 at.%, length 20 mm, and thickness 3 mm. When the Nd³⁺ ions were excited to the ${}^{4}G_{4/7}$ level, flare-up of luminescence was observed from the ${}^{4}F_{3/2}$ level (Fig. 3). The flare-up time was 5×10^{-7} sec¹⁾.

To verify the reproducibility of the results on going from sample to sample, similar measurements were performed on YAG containing, besides the Nd³⁺ ions, also 0.5 at.% Cr³⁺ or 1 at.% Ce³⁺. The time of luminescence flare-up from the level ${}^{4}F_{3/2}$ when excited to the ${}^{4}G_{7/2}$ level remained unchanged.

When the Nd³⁺ ions were excited to the levels ${}^{2}G_{7/2}$ + ${}^{4}G_{5/2}$ and ${}^{4}F_{9/2}$, no flare-up of luminescence from the ${}^{4}F_{3/2}$ level was observed within the limits of the time resolution of the receiving apparatus. This indicates that the time of relaxation from these levels does not exceed 50 nsec. Thus, when Nd³⁺ ions are excited to the ${}^{4}G_{7/2}$ level, the time of luminescence flare-up from

the level ${}^{4}F_{3/2}$, which equals 5×10^{-7} sec at room and nitrogen temperatures, is determined by the time of relaxation between the levels ${}^{4}G_{7/2}$ and ${}^{2}G_{7/2}$ + ${}^{4}G_{5/2}(\Delta E = 1200 \text{ cm}^{-1})^{[18]}$.

The maximum energy gap in the group of levels lying below ${}^{4}G_{7/2}$ is ~1000 cm⁻¹. The probability of spontaneous nonradiative transitions is in this case not less than 2×10^{7} sec⁻¹.

DISCUSSION OF RESULTS

 Temperature Quenching of the Luminescence of the Ions Nd³⁺, Er³⁺, Ho³⁺, and Tm³⁺

When the temperature is raised from 300 to 900°K, a shortening of the lifetime of the luminescence of the ions Tm^{3+} and Ho^{3+} is observed. This shortening can be due to the change in the probability of the radiative transitions or to the increase of the probability of the non-radiative transitions. However, the fact that no reduction in the lifetime of the luminescence of the ions Nd^{3+} and Er^{3+} is observed in this temperature interval indicates that the observed reduction of the lifetime of the luminescence of the Tm^{3+} and Ho^{3+} ions is not connected with a change in the probability of the radiative transitions and is due to the increase of the probability of nonradiative transitions with increasing temperature. This increase is due to an increase in the contribution of the stimulated nonradiative transitions.

From the shortening of the luminescence lifetimes of the ions Tm^{3+} and Ho^{3+} we calculated the probabilities of the temperature quenching W(T). Figure 4 shows the obtained W(T) plots. According to^[10], the temperature dependence of the probability of nonradiative transitions, which are realized with emission of phonons of one frequency, can be represented in the form

$$W(T) = W_0 (1 + \bar{n})^p,$$
 (3)

where $n = (e^{\hbar\omega/kT} - 1)^{-1}$ are the occupation numbers of the phonons, p is the number of phonons with energy $\hbar\omega$ participating in the process, and W₀ the probability of spontaneous nonradiative transitions.

At a given energy gap between the levels, the value of W_0 in (3) is larger the higher the frequency of the



FIG. 3. Oscillogram of the flare-up of luminescence of the Nd³⁺ ions from the $4_{F_{3/2}}$ level when excited by the second harmonic of a neodymium laser: a-flare-up of luminescence, b-generation pulse.

 $^{^{1)}}No$ flare-up of luminescence of the Nd³⁺ ions from the $4_{F_{3/2}}$ level was observed in [17] for YAG crystals excited to the $4_{G_{7/2}}$ level.



FIG. 4. Plots of the probability of nonradiative transitions between the levels 5_{I_2} and 5_{I_8} of the Ho³⁺ ions (a) and between the levels 3_{H_4} and 3_{H_6} of Tm³⁺ (b). Points-experimental results, curves-calculated for phonons with energies 600 (1), 700 (2), and 800 cm⁻¹ (3).

phonons participating in the process, for in this case the order of the process is lowered.

To calculate W(T) in (3) it is necessary to know the probability of spontaneous nonradiative transitions W_0 and the frequencies of the optical vibrations of the YAG lattice. The values of W_0 for the transitions ${}^{3}H_4 - {}^{3}H_6$ of the Tm³⁺ ions and of the transitions ${}^{5}I_7 {}^{5}I_8$ of the Ho³⁺ ions was determined by us and amounts to ~ 20 sec⁻¹ for both ions. The frequencies of the optical oscillations for the YAG crystals were obtained in^[19]. According to these data, in the phonon spectrum of the YAG crystals there is a whole series of optical vibrations with frequencies up to 860 cm⁻¹.

Comparing the experimentally-observed temperature dependences of the nonradiative-transition probabilities with the calculated ones obtained from expression (3), we can estimate the frequencies of the optical vibrations, which play the principal role in the processes of temperature quenching of the luminescence of Tm^{3+} and Ho³⁺. Figure 4 shows, besides the experimental data, also calculated curves of the probabilities of temperature quenching of luminescence for photons with frequencies 600, 700, and 800 cm⁻¹. The best agreement with experiment is obtained for the curves describing the process with emission of seven phonons of frequency ~700 cm⁻¹.

Our investigations of the electron-vibrational spectra of the ions Nd^{3+} , Er^{3+} , and Cr^{3+} in YAG crystals have shown that the intensity of the oscillations with frequencies 800-860 cm⁻¹ is very low, whereas those with frequency ~700 cm⁻¹ are quite intense. Apparently, oscillations with frequencies 800-860 cm⁻¹ are weakly coupled with the active TR ion and exert no significant influence on the nonradiative transitions between the levels of the TR ions in the YAG crystals.

In the case of the ions Nd³⁺ and Er³⁺ in YAG crystals, the lifetimes of the luminescence from the level ${}^{4}F_{3/2}(Nd^{3+})$ and ${}^{4}I_{13/2}(Er^{3+})$ do not change with increasing temperature from 77 to 900°K (Fig. 2). For the Nd³⁺ ions the distance from the level ${}^{4}F_{3/2}$ to the nextlower-lying level ${}^{4}I_{15/2}$ is 4360 cm^{-1 [16]} and is close to the analogous distances for the ions Tm³⁺ and Ho³⁺. It should be expected that the value of the probability of the temperature quenching of the luminescence of the Nd³⁺ ions will be close to those observed in the case of Tm³⁺ and Ho³⁺. However, the probability of the radiative transition for the ions Nd³⁺ amounts to $\sim 4 \times 10^{3} \sec^{-1}$ and is much higher than the probability of the temperature quenching (300 sec⁻¹ at maximum



FIG. 5. Dependence of the probability of spontaneous nonradiative transitions on the energy gap between the levels.

temperature), whereas in the case of the ions Tm^{3+} and Ho³⁺ these probabilities are comparable in magnitude. Therefore no shortening of the lifetime of the ${}^{4}F_{3/2}$ level of the Nd³⁺ ion is observed in the investigated temperature interval.

In the case of the Er^{3^+} ions there is likewise no shortening of the lifetime of the ${}^{4}\mathrm{I}_{13/2}$ level in this temperature interval, although the probability of the radiative transition in the case of Er^{3^+} is close to the probabilities for Tm^{3^+} and Ho^{3^+} . This is connected with the much larger distance (~ 6000 cm⁻¹) between the radiative level ${}^{4}\mathrm{I}_{13/2}$ and the ground state ${}^{4}\mathrm{I}_{15/2}$ than in the case of the Tm^{3^+} and Ho^{3^+} ions^[13]. An increase of the energy gap between the levels leads to an increase in the number of the phonons participating in the process and to a decrease of the probability of the temperature quenching of the luminescence.

 Dependence of the Probability of Nonradiative Transitions Between the Levels of the Ions Nd³⁺, Er³⁺, Ho³⁺, and Tm³⁺ on the Energy Gap Between the Levels

As a result of our investigations we obtained the probabilities of the spontaneous nonradiative transitions for different energy gaps between the levels of a number of TR ions in YAG crystals (Fig. 5). This dependence is well approximated by an exponential law, just as for other crystals^[5-10]. The values of the probabilities of the nonradiative transitions between the levels of the TR ions in the YAG crystals slightly exceed the values of these probabilities in Y_2O_3 crystals^[10]. Thus, for the energy gap 2750 cm⁻¹ in Y_2O_3 crystals^[10] the value of W_0 is $6.5 \times 10^3 \text{ sec}^{-1}$, whereas in YAG crystals for $\Delta E = 2870 \text{ cm}^{-1}$ it is equal to $W_0 = 9 \times 10^3 \text{ sec}^{-1}$. This is apparently due to the larger spread of the phonon spectrum in YAG crystals.

The obtained dependence of W_0 on the energy gap between the levels makes it possible to estimate the probability of nonradiative relaxation between the levels ${}^{4}I_{11/2}$ (lower laser level) and ${}^{4}I_{9/2}$ (ground state) of the ions Nd³⁺ in YAG crystals. The minimum energy gap between these levels is 1200 cm⁻¹ [¹⁶]. The probability of the spontaneous nonradiative transitions corresponding to this gap (Fig. 5) is equal to 2×10^{6} sec⁻¹. This value coincides with the value of the probability of the nonradiative transitions between the levels ${}^{4}G_{7/2}$ and ${}^{2}G_{7/2} + {}^{4}G_{5/2}$ of the Nd³⁺ ions. The relaxation time between these levels does not depend on the temperature in the interval 77-300°K. One can expect the probability of the nonradiative relaxation between the levels ${}^{4}I_{11/2}$ and ${}^{4}I_{9/2}$ of the Nd³⁺ ions likewise not to change strongly on going from 77 to 300°K.

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