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Time Variation of Nd^{3+} Ion Luminescence and an Estimation of Electron Excitation Migration Along the Ions in Glass

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The time course of luminescence quenching in the presence of excitation migration along donors is studied theoretically and experimentally. The quasidiffusion and stepwise cases of migration in a disordered medium are considered theoretically. Nd^{3+} in glass is investigated experimentally at temperatures between 20° and 600°C. Migration is found to be of a stepwise nature. The Nd^{3+}–Nd^{3+} interaction underlying the migration is assessed.

A study of the migration of electron excitation over like rare-earth ions, particularly over Nd^{3+} ions, is of interest both for a more precise analysis of the lasing process and for clarification of the physical nature of the interactions that lead to transfer of the excitation energy.

The migration effect has been usually investigated either under laser generation conditions[1-4] or else immediately after generation.[5,6] The interpretation of the experimental results is not always unique, since an important role is played, for example, by the inhomogeneity factor, the ratio of the inhomogeneous and homogeneous broadenings.

We attempt here to estimate the effect of migration in the case of weak nonlaser excitation. For such an estimate it is possible to use the plots of the time dependence of luminescence after brief excitation in the presence of a quencher.

1. THEORETICAL PART

The time dependence of luminescence in dipole-dipole quenching and in the case when the excitation migration has a diffuse character has been considered in a number of studies, for example in [1]. At low active-ion concentrations, however, when $4\pi n r_0^3/3 < 1$ (r_0 is the minimum possible distance between active ions), the excitation migration cannot be described by the diffusion equation, owing to the large fluctuations of the ion-ion transition probability.

Let us consider from somewhat different points of view, without resorting to the diffusion equation, the migration of excitation over donor ions when the excitation is simultaneously quenched by acceptors. We assume that both the donor-donor excitation transfer and the excitation quenching by the acceptor result from dipole-dipole interaction. (The reasoning can be generalized to include also other types of interaction.)

We consider a certain definite excitation and determine for it the probability of not being quenched by the instant t. Mathematically it is necessary for this purpose to find the quantity

$$\langle N(t)\rangle = \left\langle \exp\left\{-\sum_a \int_0^t W_a[R(t')]\,dt'\right\}\right\rangle, \tag{1}$$

averaged over the ensemble of the possible realizations of the excitation trajectory. Here $W_n[R(t')]$ is the probability of quenching by the a-th acceptor, and the summation is over all the acceptors.

This problem is formally similar to the problem of the line shape in gases, considered by Anderson.[8] Going over to the limit when the volume of the system $V \to \infty$ and the number of acceptors $N_a \to \infty$, under the condition $N_a/V = n_A$ = const, we obtain, in analogy with Anderson's result[8]

$$\langle N(t)\rangle = \left\langle \exp\left\{-4\pi n_A \int_0^\infty R^2 dR\left[1-\exp\left(-\int_0^t W[R(t')]\,dt'\right)\right]\right\}\right\rangle \tag{2}$$

Here n_A is the number of acceptors per cm^3, and the angle brackets denote averaging over the ensemble of the trajectory realizations.

We note that, unlike in gases, where the motion is over trajectories characterized by an average velocity $\bar{v}$,[8] in our case the migration represents a random walk of the excitation over randomly disposed donors with average concentration n_D.

Let us discuss qualitatively the character of the time variation of the luminescence. If the quenching precedes the migration, then the quenching occurs in quasistatic fashion (R in (2) does not depend on t):

$$\langle N(t)\rangle = \exp\left\{-4\pi n_A \int_0^\infty R^2 dR\left[1-e^{-W(R)t}\right]\right\}. \tag{3}$$

When $W(R) = C_{DA}/R^6$ (C_{DA} is the characteristic of the donor-acceptor interaction) we obtain from this Forster's well-known result

$$\langle N(t)\rangle = \exp\left(-\gamma\sqrt{t}\right), \qquad \gamma = {}^4/_3\pi^{3/2} n_A \sqrt{C_{DA}}.$$

The same result is obtained for the initial stage of the process at small values of t. In the final stage, the process becomes exponential, $N(t) = \exp(-\overline{W}t)$, with $\overline{W}$ determined by the migrating excitations (excitons) whose paths enter the region of strong interaction with the acceptors, where they are quenched.

We introduced the strong-quenching sphere (R_W) defined by the condition $W(R_W)\tau = 1$, where $W(R_W) = C_{DA}/R_W^6$ is the probability of quenching when enter-

The transition from the initial quasistatic part of the process to the exponential one is determined approximately by the limiting time, from the condition that the number of nondepleted quasistatic excitations $\exp(-4\pi n_A \times R_W^3/3)$ (the number of excitations that do not fall initially into the strong-quenching sphere) be equal to $\exp(-\overline{W}t)$, i.e.,

$$t_l = 4\pi n_A R_W^3 / 3\overline{W}. \tag{4}$$

The passage of the exciton through the sphere R_W differs in character, depending on the ratio between R_W and the average donor-donor distance $L = (3/4\pi n_D)^{1/3}$. When $R_W \gg L$, the passage is via a large number of jumps and has a quasi-diffuse character. When $R_W \lesssim L$, the exciton, falling on a donor located inside the sphere R_W, "sits" for some time on the donor, and then leaves R_W as a result of single jump; we call this a jump passage. Similar situations for the case when the donors make up a crystal are considered in Agranovich's book.[9]

We make a few estimates in the quasidiffusion case. We begin with R_W. By definition, $W(R_W) = C_{DA}/R_W^6$, $\tau = R_W^2/D$, (D is the diffusion coefficient), i.e.,

$$R_W = (C_{DA}/D)^{1/4}. \tag{5}$$

As already mentioned, at small values of n_D, the concept of the usual diffusion coefficient is not valid. We are more likely to have in this case a distribution of diffusion coefficients. We can, however, introduce a certain quasidiffusion coefficient D_1, which determines the average time of spreading of an exciton packet. Indeed, for the packet to spread it is necessary that the distance from the donor on which the exciton is located to the neighboring donors be approximately the same, for otherwise an exciton falling on a pair of closely-lying donors will settle on this pair, and will spread only to the extent that it interacts with the more remote donors. Therefore, to estimate D_1 it is necessary to cut off the integral in terms of which D is determined in the usual manner at a distance on the order of L, i.e.,

$$D_1 = \frac{4\pi}{6} n_D \int_L^\infty \frac{r^2 C_{DD}}{r^6} dr = \frac{1}{2}\left(\frac{4\pi}{3}\right)^{4/3} n_D^{4/3} C_{DD} \tag{6}$$

(C_{DD} is the donor-donor interaction characteristic).

To estimate the characteristic of the exponential decay $\overline{W}$, we use the result obtained in [7]: $\overline{W} = 4\pi n_A Da$, where a is the scattering length, which has the same meaning as R_W, i.e.,

$$\begin{aligned}\overline{W} &= 4\pi n_A D_1 R_W = 4\pi n_A D_1^{3/4} C_{DA}^{1/4} \\ &= \frac{(4\pi)^2}{3}\left(\frac{1}{2}\right)^{3/4} n_A n_D C_{DA}^{1/4} C_{DD}^{3/4}.\end{aligned} \tag{7}$$

With the aid of (4) we can estimate the limiting time of transition from the quasistationary decay to the exponential one:

$$t_l = R_W^2 / 3D. \tag{8}$$

As expected, this time has the same order of magnitude as the time of spreading over a distance R_W.

As indicated above, the quasidiffusion case takes place when $R_W \gg L$, so that when (5) and (6) are taken into account the condition for the applicability of the results reduces to

$$(C_{DA}/2C_{DD})^{1/4} \gg 1, \text{ i.e., } C_{DA} \gg 2C_{DD}. \tag{9}$$

τ_m is the time of "sitting" in the sphere and τ is the time of passage of the exciton through this sphere.

We emphasize once more that by virtue of the semiquantitative character of the analysis, formulas (5)–(8) should be regarded only as estimates.

We now proceed to consider the case of jump motion of an exciton ($R_W \lesssim L$). To estimate R_W we must take as the time of passage of the exciton through the strong-quantum sphere the most probable donor-donor transfer time τ_m, since the contribution of one nearby pair during the time of "sitting" does not exceed the corresponding contribution of an unpaired donor, and the number of unpaired donors is much larger than the number of pairs. Since we have in dipole-dipole interaction $\tau_m = (3/2\pi)^3 \times 1/n_D^2 C_{DD}$,[10] it follows that

$$R_W = (C_{DA}/n_D^2 C_{DD})^{1/6} (3/2\pi)^{1/2}. \tag{10}$$

To determine $\overline{W}$ we recognize that in the case of frequent jumps of the excitation, the averaging of the decay law (2) is equivalent to averaging of its rate. The latter is determined, in the case of the Poisson distribution of the lifetime of the exciton on the donors, $\exp(-\tau/\tau_0)\tau_0^{-1}d\tau$ (τ_0 is the average time), by the product of the jump frequency by the average efficiency of "sitting":

$$\overline{W} = \frac{4\pi n_A}{\tau_0} \int_0^\infty R^2 dR \int_{-\infty}^{+\infty} \varphi(\omega) d\omega \int_0^\infty \left[1 - \exp\left(-\frac{C_{DA}\tau}{R^6}\right)\right] e^{-\tau/\tau_0} \frac{d\tau}{\tau_0}. \tag{11}$$

We have introduced here $\omega = \omega_D - \omega_A$, which is the difference between the transition frequencies of the donor and the acceptor, and $\varphi(\omega)$, which is the density of the distribution of this difference; these two quantities take into account the existence of the inhomogeneous broadening. For $1/\tau_0 = \xi_m$ we use the most probable value of the donor-donor transfer rate with allowance for the inhomogeneous broadening, i.e., the donor-donor deviation ω' with distribution density $f(\omega')$. Using the Markov method and following Rozman,[10] we obtain

$$\xi_m = (2\pi/3)^3 \left[\int \sqrt{C_{DD}(\omega')} f(\omega') d\omega'\right]^2 n_D^2. \tag{12}$$

Calculation of the integrals in (11) and the use of (12) yields

$$\begin{aligned}\overline{W} &= \pi(2\pi/3)^{5/2} \left[\int \sqrt{C_{DA}(\omega)} \varphi(\omega) d\omega\right] \left[\int \sqrt{C_{DD}(\omega')} f(\omega') d\omega'\right] n_A n_D \\ &= \pi(2\pi/3)^{5/2} K_1 K_2 n_A n_D.\end{aligned} \tag{13}$$

To estimate the integrals K_1 and K_2 we assume that

$$C_{DA}(\omega) = \frac{C_{DA}(0)\gamma_1^2}{\omega^2 + \gamma_1^2}, \quad C_{DD}(\omega) = \frac{C_{DD}(0)\gamma_2^2}{\omega^2 + \gamma_2^2}$$

and we make use of the fact that

$$\sqrt{C_{DA}(\omega)} = \frac{2}{\pi} \gamma_1 \sqrt{C_{DA}(0)} \int_0^\infty \frac{dx}{\omega^2 + \gamma_1^2 + x^2}.$$

Then, breaking up the integration into regions from 0 to $\Delta_{1,2}$ and from $\Delta_{1,2}$ to ∞ ($\Delta_{1,2}$ are the widths of the distributions $\varphi(\omega)$ and $f(\omega)$) and assuming $\Delta/\gamma > 1$, we obtain

$$\begin{aligned}K_1 = \frac{2}{\pi}\gamma_1\sqrt{C_{DA}(0)} \Big\{ &\pi\varphi(0) \ln\left[\frac{\Delta_1}{\gamma_1} + \sqrt{\frac{\Delta_1^2}{\gamma_1^2} + 1}\right] \\ &+ \frac{\pi}{2\gamma_1} - \frac{1}{\gamma_1} \operatorname{arctg} \frac{\Delta_1}{\gamma_1} \Big\}.\end{aligned} \tag{14}$$

When $\Delta \gg \gamma$ we have

$$K_1 = \frac{2\gamma_1}{\pi\Delta_1}\sqrt{C_{DA}(0)}\left(1+\ln\frac{2\Delta_1}{\gamma_1}\right).$$

A similar result is obtained for K_2.

The initial quasistatic section of the time variation, with the inhomogeneous broadening accounted for in similar fashion, will be described by the expression

$$\overline{N(t)} = e^{-\gamma\sqrt{t}}, \quad \gamma = {}^4/_3\pi^{3/2}\left[\int\sqrt{C_{DA}(\omega)}\,\varphi(\omega)\,d\omega\right]n_A = {}^4/_3\pi^{3/2}K_1 n_A. \tag{15}$$

The limiting time of transition from the quasistatic section to the exponential one in the jump case is

$$t_l = \left(\frac{3}{2\pi}\right)^3\frac{1}{K_2^2 n_D^2}. \tag{16}$$

We have thus obtained expressions for the time dependence of the luminescence in terms of the characteristics of the donor-acceptor and donor-donor interactions.

2. EXPERIMENTAL PART

The time dependence of the luminescence was registered with a stroboscopic setup covering a dynamic range of intensity variation up to 40–50 dB. The error in the initial section was 1–2% and towards the end of the dynamic range not more than 10%.

The functional diagram of the setup is shown in Fig. 1. Sample 6 is excited at a frequency 120 Hz by short (~10 μsec at 0.35 level) light flashes from an ISSh-400 lamp 2. The luminescence past the monochromator 7 is registered by an FÉU-22 photomultiplier. The photocurrent from the output of the photomultiplier 8, in the form of a periodically repeating curve of the time variation of the luminescence, is fed to the photocurrent meter, which is opened by strobing pulses for 5–10 μsec in synchronism with the light flashes, but at double the frequency (240 Hz).

When the signal and the strobing pulses are simultaneously applied to the photocurrent meter 12, every second strobing pulse receives an amplitude increment proportional to the instantaneous value of the signal at the instant of coincidence. The measuring circuit suppresses all the harmonics with frequencies above 120 Hz (carried), and the fundamental amplitude of the envelope of the strobing pulses (120 Hz) is separated and measured with the aid of a synchronous detector. Such a method of strobing and separating the signal ensures high linearity and small null drift owing to the automatic cancellation of the dc component that always appears as a result of the strobing.

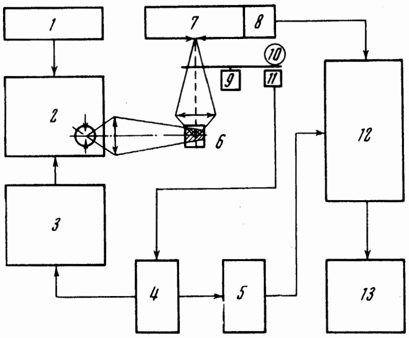

FIG. 1. Functional diagram of stroboscopic setup. 1—High-voltage rectifer, 2—block for switching the ISSh-400 lamp, 3—block for synchronizing the ISSh-400 lamp, 4—G5-4B pulse generator, 5—strobing pulse generator, 6—investigated sample, 7—ISP-51 monochromator, 8—FÉU-22 photomultiplier, 9—disc modulator (four main and eight auxiliary holes), 10—6.3V lamp, 11—photodiode, 12—strobing photocurrent meter, 13—EPP-09 potentiometer.

In the case of slow (smooth or stepwise) variation of the delay of the strobing pulses relative to the start of the light flashes, the synchronous-detector output voltage, averaged over a large number of flashes, also changes smoothly or stepwise in accordance with the time variation of the luminescence. The setup was described in detail earlier.[11]

It should be noted that if the characteristic damping time of the luminescence is comparable with or larger than the repetition period of the strobing pulses, then the reference strobing pulses also acquire increments, i.e., an error appears as a result of the fact that the luminescence intensity is no longer measured from a zero level. To eliminate this error it is necessary to interrupt the light flux from the sample at the instant when the reference strobing pulses appear. This is done with the aid of an additional disc modulator 9, against which all the units of the setup are synchronized.

The time dependence of the luminescence of Nd^{3+} was registered in the 0.9 μ region, where there is practically no reabsorption. The experimental time dependence was plotted in a semilogarithmic scale (log J vs. t). We then determined the difference between the semilogarithmic curves corresponding to the active ions with and without the quencher. The difference curve made it possible to exclude from consideration the damping constant without quenching, which is important for glasses, where the time dependence of the luminescence of the samples without the quencher is no longer exponential, apparently as a result of the presence of various types of luminescent centers in the glass. An example of the semilogarithmic curves and of the difference curve is shown in Fig. 2.

We investigated in detail the time dependence of the luminescence in a series of silicate glass samples (53.0% SiO_2, 25.9% BaO, 21.1% K_2O, 1.0% Sb_2O_3) at Nd_2O_3 concentrations from 0.5 to 8% (percentages by weight in excess of 100%). The measurements were performed at room temperature and on samples heated by a specially constructed quartz electric furnace to 200, 400, and 600°C.

The following results were obtained:

1. The time dependence of the luminescence corresponds to the formula $J \sim e^{-\gamma\sqrt{t}}$ in the initial section and $J \sim e^{-Wt}$ in the final section. This can readily be seen by plotting the difference curve in terms of the

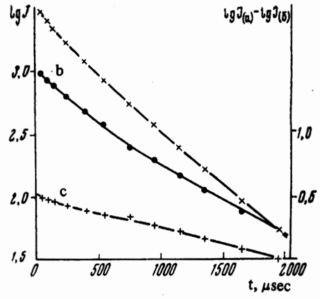

FIG. 2. Time dependence of luminescence (log J vs. t). a) 6% Nd_2O_3, b) 0.5% Nd_2O_3, c) difference curve).

coordinates log log J and log t (Fig. 3): the curve has an initial slope close to $1/2$, which subsequently approaches unity.

2. The value of γ is approximately proportional to the concentration of the quenching acceptor, which is assumed here to be equal to or proportional to the Nd_2O_3 concentration. It should be noted that, generally, speaking, the role of the quencher can be played both by the Nd^{3+} ions themselves (owing to the transitions $^4F_{3/2} \rightarrow {}^4I_{15/2}$ in the donor and $^4I_{9/2} \rightarrow {}^4I_{15/2}$ in the acceptor), and by a random uncontrollable impurity introduced with the neodymium oxide; in addition, the quenchers can be nonluminescent Nd-Nd pairs. We shall present later on certain arguments favoring the assumption that the main quenchers in the investigated glass are the Nd^{3+} ions themselves.

Table I gives the values of the ratio γ/c_A, where c_A is the concentration of Nd_2O_3 in weight percent (with account taken of subtraction of 0.5% Nd_2O_3, corresponding to the practical absence of quenching, and with recalculation of the glass composition to 100% to allow for the introduction of the Nd_2O_3).

The error is estimated from the measurements for quencher concentrations from 1.46 to 5.16%.

3. The dependence of $\overline{W}/c_A$ on the donor concentration c_D, with indication of the errors, is shown in Fig. 4. As seen from the figure, the errors in the quantities are quite large for the first two donor concentrations. This is due to the fact that the difference curve is obtained by subtracting curves that are very close to each other. For the two concentrations, the error, with the exception of one case, is less than 10%. The obtained dependence of $\overline{W}/c_A$ and c_D can be approximately regarded as linear. The slope of the lines in Fig. 4 gives the ratio $\overline{W}/c_A c_D$ for different temperatures.

4. The temperature dependence obtained for $B = \overline{W}/c_A c_D$ is shown in Table II.

Figure 5 shows the dependence of log B on the reciprocal absolute temperature. We see that up to 400°C the dependence is approximately linear, i.e., $B = \overline{W}/c_A c_D \sim \exp(-E/kT)$, where the activation energy $E \approx 140$ cm^{-1}; near 600°C, a sharp bend upwards is observed.

5. The temperature dependence of the ratio c_A has the same character (Fig. 5), and the activation energy is also close to 140 cm^{-1}.

3. DISCUSSION OF RESULTS

Since the time dependence of the luminescence attenuation due to quenching and migration (curve c on Fig. 2) turned out to be close to the theoretical one, we consider it possible, by using the results of the theoretical part, to estimate the donor-acceptor and donor-donor interaction characteristics K_1 and K_2. Indeed, by determining experimentally the values, averaged over the set of concentrations, of the quantities γ/c_A (Table I) and $\overline{W}/c_A c_D$ (Table II), we can use formulas (15) and (13), from which we see that the parameter of the initial part of the time dependence (γ/c_A) depends only on K_1, and that of the final part $(\overline{W}/c_A c_D)$ depends on K_1 and K_2.

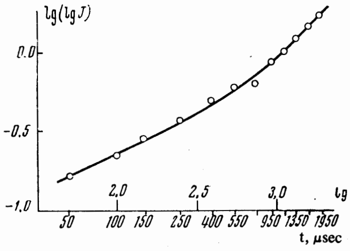

FIG. 3. Difference curve (Fig. 2c) in coordinates log log J and log t.

Table I

t, °C	20	200	400	600
γ/c_A	3.32±0.39	4.4±0.48	4.9±0.62	6.4±0.54

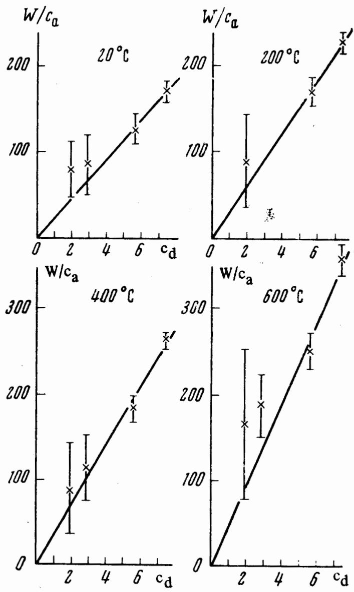

FIG. 4. Plots of W/c_A against the donor concentration at different temperatures.

Table II

t, °C	20	200	400	600
B	23	30	34	46

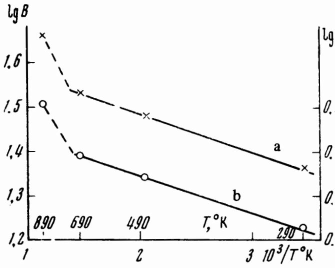

FIG. 5. Temperature dependence of $B = W/c_A c_D$ (a) and of γ/c_A (b).

Using Table I, we estimate by means of formula (15) the quantity $K_1 = \int \sqrt{C_{DA}(\omega)}\,\varphi(\omega)\,d\omega$. The result is given in Table III.

As already mentioned, we assumed that the acceptor is the Nd^{3+} ion. If the acceptor were a randomly introduced impurity, whose concentration was smaller by

Таблица 3

t, °C	20	200	400	600
$K_1 \cdot 10^{20}$	0.5	0.66	0.73	0.94

several orders of magnitudes than the concentration of the Nd^{3+}, then the value of K_1 would be much larger and the corresponding Forster radius R_0 would reach ~50 Å, which is much larger than the possible values for rare-earth ions.

To estimate now the donor-donor interaction characteristic C_{DD} or K_2 from the experimental values of $\overline{W}$, it is necessary to ascertain whether the exciton motion is jumplike or quasidiffuse, i.e., to estimate the quantity $C = \frac{4}{3}\pi n_A R_W^3$. From the drop of the difference curves at the transition time, from the $\sqrt{t}$ law to the linear law, we find that in our experiments C does not exceed 0.4, i.e., $R_W < L$, and consequently the exciton motion is jumplike.

Using formula (13) and Tables II and III, we obtain the values of K_2. We get $K_2 \approx 2.8 \times 10^{-20}$ for temperatures up to 400°C, and $K_2 \approx 3.0 \times 10^{-20}$ at 600°C. The fact that K_2 is practically independent of the temperature is the result of the parallel temperature dependences of B and γ (Fig. 5), which confirm the correctness of the assumption that the migration has a jumplike character, for in this case $\overline{W}$ and γ are proportional to K_1. Thus, the observed activation energy ~140 cm^{-1} is connected with donor-acceptor excitation transfer, and this quantity amounts to approximately one-half the energy of the third level of the multiplet $^4I_{9/2}$ of the Nd^{3+} ion.[12]. Such a result, with allowance for the empirical level scheme of the lower multiplets of Nd^{3+} in glass,[13] is evidence in favor of the assumption that the quenching is a result of the transitions $^4F_{3/2} \rightarrow {}^4I_{15/2}$ and $^4I_{9/2} \rightarrow {}^4I_{15/2}$ in the Nd^{3+} ions with participation of the third level $^4I_{9/2}$. The activation energy of B and γ corresponds to E/2, since B and γ are proportional to $\sqrt{C_{DA}}$, and $C_{DA} \sim \exp(-E/kT)$.

Using the obtained value of K_2, we can attempt to estimate the migration velocity that can appear in estimates with lasing. This velocity is apparently an integral characteristic of the type

$$F = 4\pi K_2^{\,2} n_D \int_{r_0}^{\infty} \frac{r^2\,dr}{r^6} = \frac{4\pi K_2^{\,2}}{3r_0^{\,3}} n_D,$$

where r_0 is the minimum Nd-Nd distance. We know of no experimental data on r_0, but there are x-ray investigations from which it follows that the Ba-Ba distances in silicate glass amount to approximately 4 Å.[14] Since the radius of the Ba^2 does not differ much from that of Nd^{3+}, and since we are only interested in the order of magnitude of F, we assume r_0 = 4 Å. We then obtain $F \approx 2.8 \times 10^4$ sec^{-1} for the interval from room temperature to 500°C at an Nd_2O_3 concentration of 5.66%. This estimate is close in order of magnitude to that obtained in [2,5].

In conclusion we note that the experimentally obtained values of the limiting time t_l decrease, in accordance with the theory, approximately quadratically with increasing concentration c_D (3.9% at 400 μsec, 5.7% at 250 μsec, and 7.4% at 150 μsec).

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