
SEMICONDUCTORS
AND DIELECTRICS

Intensities of f – f Transitions in Pr^{3+} and Dy^{3+} in Glasses in the Near-IR Spectral Range

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Abstract—The absorption spectra of Pr^{3+} and Dy^{3+} ions in three glass matrices (SiO_2 – P_2O_5 – GeO_2 , Al_2O_3 – B_2O_3 – SiO_2 , and LiB_3O_5) are thoroughly studied in the near-IR spectral range (4600–14 300 cm^{-1}). The temperature dependences of the intensity, the width, and the location of the absorption bands observed are investigated for the first time. It is shown that the f – f transitions in the studied glasses are allowed by static odd distortions in the environment of the rare-earth ions and these distortions decrease with an increase in temperature. A comparative analysis of the absorption band parameters and their temperature behavior in different materials makes it possible to determine the differences in magnitudes, symmetries, and dispersions of the distortions in the nearest environment of rare-earth ions in different glasses. © 2001 MAIK “Nauka/Interperiodica”.

1. INTRODUCTION

Investigations of the f – f transitions in rare-earth ions in different compounds have attracted the attention of many researchers (see [1–5] and references therein). Considerable interest in the optical properties of rare-earth materials is motivated by their diverse technical applications (for example, practical use of optical laser glasses).

The intensity of the f – f transitions is usually analyzed in terms of the Judd–Ofelt theory [6, 7], which allows one to describe all the f – f transitions in a particular ion with the use of three parameters. The majority of the f – f transitions are consistent with this theory. However, Jørgensen and Judd [8] revealed the so-called supersensitive transitions, which cannot be described by the above three parameters. These transitions turned out to be especially sensitive to the ligand environment of rare-earth ions. In free atoms, the f – f transitions are parity-forbidden in the electric dipole approximation. The f – f transitions become partly allowed in a condensed state of the material due to the static and dynamical (vibrational) odd distortions of the ligand environment. Therefore, the intensities of all f – f transitions, and not just the supersensitive transitions, should depend on the symmetry of the ligand environment and the magnitude of odd distortions. Consequently, a comparative investigation of the f – f transitions in rare-earth ions in different matrices can provide information on the environment of the rare-earth ions. This analysis was the main objective of the present work.

In this work, we investigated the temperature behavior of the f – f transitions in Pr^{3+} and Dy^{3+} ions in a num-

ber of previously unexplored glasses in the spectral range 4600–14 300 cm^{-1} . Analysis of the temperature dependences of the intensities of the f – f transitions made it possible to differentiate the contributions of the static and vibronic mechanisms of allowing these transitions to the absorption intensity and to trace the evolution in certain properties of the ligand environment in glasses with a change in temperature. To our knowledge, there is only one work concerned with the investigation into the properties of the f – f transitions at different temperatures of the material [9].

2. EXPERIMENTAL TECHNIQUE

The optical absorption spectra in the near-IR range at temperatures of 78–300 K were recorded on an automated setup based on DMR-4 and MDR-12 monochromators. The instrumental resolution was equal to 20 cm^{-1} . The dynamic range of the measured optical densities was limited to 4.5. The temperature was controlled to within ± 1 K. Moreover, the absorption spectra for a number of samples were measured on UVICON 943 and SF-20 spectrophotometers (at a temperature of 300 K).

We used three glass matrices: SiO_2 – P_2O_5 – GeO_2 , Al_2O_3 – B_2O_3 – SiO_2 , and LiB_3O_5 . Praseodymium and dysprosium oxides at different concentrations were added to the melt. The procedure of glass synthesis was described in more detail in [10, 11]. The glass compositions and rare-earth ion concentrations (as-batched) are presented in Table 1.

Table 1. Glass compositions and concentrations of rare-earth ions

Sample designation	Rare-earth ion	Matrix composition	C, 10^{21} cm^{-3}	C, mol %
Pr1	Pr^{3+}	$\text{SiO}_2\text{-P}_2\text{O}_5\text{-GeO}_2$	5.02	20 (Pr_2O_3)
Pr2	Pr^{3+}	$\text{Al}_2\text{O}_3\text{-B}_2\text{O}_3\text{-SiO}_2$	2.26	15 (Pr_2O_3)
Pr3	Pr^{3+}	LiB_3O_5	1.37	6 (Pr_2O_3)
Pr4	Pr^{3+}	LiB_3O_5	3.27	13.4 (Pr_2O_3)
Dy1	Dy^{3+}	$\text{SiO}_2\text{-P}_2\text{O}_5\text{-GeO}_2$	3.55	15 (Dy_2O_3)
Dy2	Dy^{3+}	$\text{SiO}_2\text{-P}_2\text{O}_5\text{-GeO}_2$	4.52	20 (Dy_2O_3)
Dy3	Dy^{3+}	$\text{Al}_2\text{O}_3\text{-B}_2\text{O}_3\text{-SiO}_2$	8.60	25 (Dy_2O_3)

As follows from the magnetic susceptibility measurements, all the glasses under investigation are paramagnets in the temperature range covered.

3. RESULTS AND DISCUSSION

Figures 1 and 2 show the absorption spectra of two glasses containing praseodymium and dysprosium. The absorption bands were identified according to [12]. The ${}^6H_{15/2} \rightarrow {}^6(F_{11/2} + H_{9/2})$ transition in Dy^{3+} and the ${}^3H_4 \rightarrow {}^3F_2$ transition in Pr^{3+} are considered supersensitive transitions [2]. However, Hormadaly and Reisfeld [12] argued that the supersensitive transition in Pr^{3+} is the ${}^3H_4 \rightarrow {}^3P_2$ transition. The absorption spectra of all glasses containing the same rare-earth ions are qualitatively identical. The spectra of the Dy1 and Dy2 glasses with similar glass matrices (Table 1) virtually coincide. The spectra of the Pr3 and Pr4 glasses are also identical to each other, even though the praseodymium concentrations differ substantially (Table 1). This means that the interaction between Pr^{3+} ions either is insignificant or does not affect the f - f absorption.

The absorption spectra were decomposed into Gaussian components. The Gaussian function is best suited for describing the inhomogeneously broadened

absorption bands which are characteristic of glasses. Indeed, the experimental spectra are adequately represented by the spectral decomposition. As can be seen from the spectrum of Dy^{3+} (Fig. 2), whose lines are well resolved, the absorption bands are nearly symmetric at the lowest temperature studied. The number of components for the decomposition of the spectra was chosen from theoretical considerations and the observed spectral features.

The oscillator strengths of the transitions were calculated by the relationship [12]

$$f = 4.318 \times 10^{-9} \int \epsilon(k) dk \equiv 4.318 \times 10^{-9} I, \quad (1)$$

where $\epsilon(k)$ is the molar extinction coefficient at a wave number k (cm^{-1}) and I is the line intensity. The oscillator strengths of the f - f transitions in the studied glasses and the experimental data available in the literature are given in Tables 2 and 3. The temperature dependences of the absorption band parameters (intensities, widths, and locations) for the praseodymium-containing glasses investigated are depicted in Figs. 3–5.

3.1. Intensities of the absorption bands. The intensity of the f - f transitions in a material in a condensed state depends on the following factors: (1) the

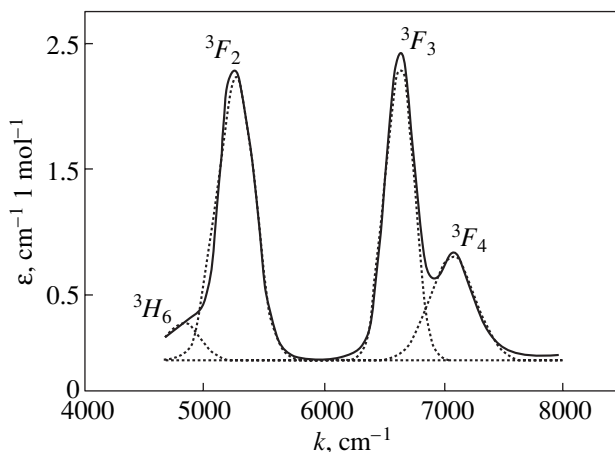


Fig. 1. Absorption spectrum of the Pr1 glass at $T = 93 \text{ K}$. Dotted lines show the Gaussian components of the spectral decomposition.

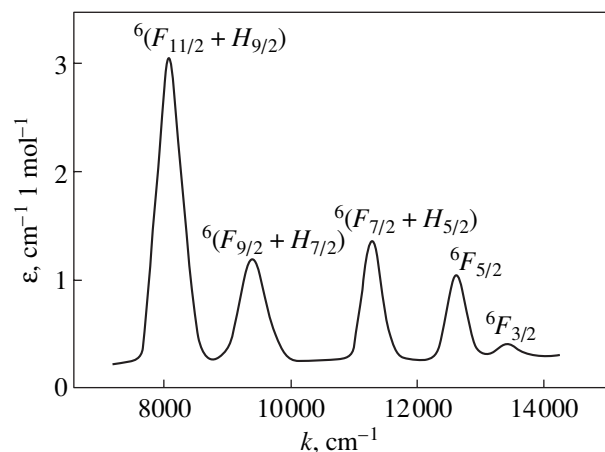


Fig. 2. Absorption spectrum of the Dy3 glass at $T = 93 \text{ K}$.

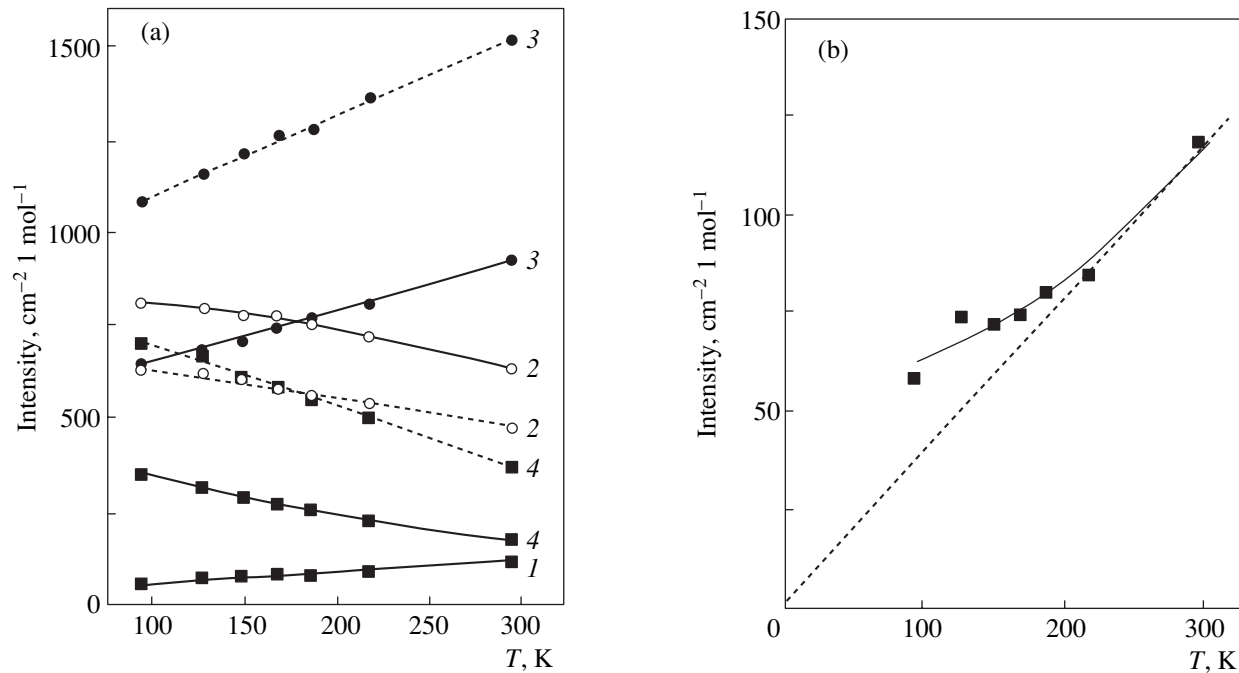


Fig. 3. (a) Temperature dependences of the absorption band intensity for (1) ${}^3H_4 \rightarrow {}^3H_6$, (2) ${}^3H_4 \rightarrow {}^3F_2$, (3) ${}^3H_4 \rightarrow {}^3F_3$, and (4) ${}^3H_4 \rightarrow {}^3F_4$ transitions in the Pr1 (solid lines) and Pr4 (dashed lines) glasses. (b) Temperature dependence of the absorption band intensity for the ${}^3H_4 \rightarrow {}^3H_6$ transition in the Pr1 glass on a different scale.

symmetry and the magnitude of static and vibrational odd distortions; (2) the ligand type, i.e., the ratio between the ionic and covalent bonds of the required symmetry; and (3) the type of the transition involved.

All the Pr^{3+} -containing compounds presented in Table 2, except for those studied in [14, 16], contain the same ligands, namely, oxygen ions. Therefore, the transition intensities should depend only on the magnitude and the symmetry of distortions and the type of the transition. First and foremost, it is clearly seen that the majority of the compounds under consideration can be separated into two groups in which the total intensities of the transitions to the 3F states differ by a factor of two. It is of interest that this intensity for a number of different glasses is equal to one-half the intensity for $\text{Pr}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ garnet. Garnet is a cubic crystal, but the rare-earth ions occupy the D_2 orthorhombic sites [1]. Consequently, the magnitude of the odd distortions in the aforementioned glasses is less than that in the garnet.

The total intensity is maximum in a $\text{Pr}^{3+}:\text{Y}_2\text{O}_3$ cubic crystal (Table 2), in which the Pr^{3+} ions predominantly occupy the C_2 noncentrosymmetric sites [15]. The replacement of oxygen by fluorine as a ligand only slightly affects the intensity of $f-f$ transitions, including the ${}^3H_4 \rightarrow {}^3F_2$ transition (Table 2). A change in the matrix has a substantially stronger effect.

Despite the fact that, in some cases, the total intensities in particular groups of absorption bands for the studied glasses are approximately identical, the intensity ratios of individual bands can differ significantly

(Table 2), which indicates different symmetries of the odd distortions. The reverse is also true: at the different total intensities, the intensity ratios of the individual bands involved in a particular group can be close to

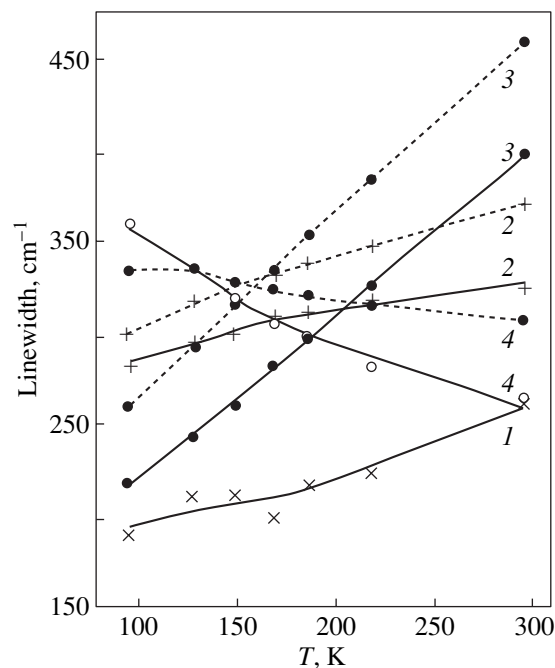


Fig. 4. Temperature dependences of the linewidth in absorption spectra for (1) ${}^3H_4 \rightarrow {}^3H_6$, (2) ${}^3H_4 \rightarrow {}^3F_2$, (3) ${}^3H_4 \rightarrow {}^3F_3$, and (4) ${}^3H_4 \rightarrow {}^3F_4$ transitions in the Pr1 (solid lines) and Pr4 (dashed lines) glasses.

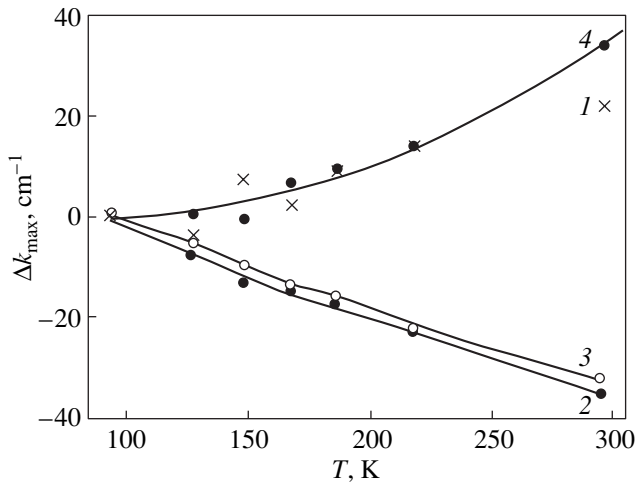


Fig. 5. Temperature dependences of the displacement of the absorption band maxima with respect to their position at $T = 93$ K for (1) ${}^3H_4 \rightarrow {}^3H_6$, (2) ${}^3H_4 \rightarrow {}^3F_2$, (3) ${}^3H_4 \rightarrow {}^3F_3$, and (4) ${}^3H_4 \rightarrow {}^3F_4$ transitions in the Pr1 glass.

each other. For example, the Pr1 and Pr2 glasses are virtually identical with respect to the symmetry of odd distortions, even though their matrices differ. The Pr4 (Pr3) glass essentially differs from the above glasses but is similar to the $\text{Pr}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ crystal and the fluoride glass studied in [16] (Table 2). This implies that the symmetry of odd distortions in the Pr4 (Pr3) glass is similar to that in the $\text{Pr}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ crystal and the fluoride glass [16], even though the total intensities of the transitions and, hence, the magnitudes of the distortions in these materials differ substantially.

The Dy^{3+} -containing compounds presented in Table 3, except for the material studied in [18], contain oxygen ions as ligands. Correspondingly, the intensity of the supersensitive transition ${}^6H_{15/2} \rightarrow {}^6(F_{11/2} + H_{9/2})$ in $\text{Dy}^{3+}(\text{aq})\text{HClO}_4\text{-DClO}_4$ [18] drastically differs from the intensity of this transition in these materials. As in the praseodymium-containing glasses (Table 2), the

total intensity of the studied transitions is maximum in the 35ZnO-65TeO_2 glass. The total intensity of the transitions in the glasses studied in the present work is less by approximately half. The intensity ratios of individual bands in the Dy1 (Dy2) and Dy3 glasses with different matrices differ considerably (Table 3); i.e., the symmetries of the odd distortions of Dy sites in these matrices are also different. By contrast, the intensity ratios of individual bands in the Pr1 and Pr2 glasses (Table 2) are the same, even though their matrices are identical to those in the Dy1 and Dy3 glasses, respectively. This implies that the symmetry of odd distortions around a rare-earth ion depends not only on the matrix but also on the rare-earth ion type. Actually, the radius of Pr^{3+} ions (1.013 Å) is larger than that of Dy^{3+} ions (0.908 Å) [1]. Furthermore, the $4f$ electrons participate in the formation of bonds [19], and, hence, these bonds depend on the number of $4f$ electrons. Therefore, the ions involving $4f$ electrons can affect the shape of ligand polyhedra, the coordination numbers, and the structure-sensitive properties, including the intensity of f - f transitions.

If the static odd distortions of the environment of rare-earth ions in a material were temperature independent, the corresponding intensities of f - f transitions would also be temperature independent. In the case when an increase in the temperature leads only to a change in the magnitude of the static odd distortions (due to the thermal expansion) but not in their symmetry, the intensities of the f - f transitions should slowly decrease with an increase in the temperature. On the other hand, the intensity of the transition which is allowed only by the vibronic mechanism should increase with a rise in the temperature according to the relationship [20]

$$f = 0.5 \sum f_{0s} \coth(h\nu_s/2kT). \quad (2)$$

Here, s is the number of an active odd vibration, ν_s is the frequency of the s th vibration, and f_{0s} is the oscillator strength of the electron transition which is allowed

Table 2. Oscillator strengths ($f \times 10^6$) of the transitions in Pr^{3+} in different matrices at room temperature

Transition	Pr1	Pr2	Pr4	[12] ₁	[12] ₂	$\text{Pr}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ [13]	[14]	[16]	$\text{Pr}^{3+}:\text{Y}_2\text{O}_3$ [15]
${}^3H_4 \rightarrow {}^3F_2$	2.75 (1)	4.0 (1)	2.05 (1)	6.15 (1)	6.36 (1)	5.63 (1)	3.12 (1)	1.25 (1)	
${}^3H_4 \rightarrow {}^3F_3$	4.0 (1.45)	5.59 (1.4)	6.55 (3.2)	11.36 (1.85)	11.06 (1.74)	14.91 (2.65)	5.48 (1.76)	3.83 (3.06)	
${}^3H_4 \rightarrow {}^3F_4$	0.75 (0.27)	1.16 (0.29)	1.6 (0.78)	3.92 (0.64)	3.41 (0.54)	5.68 (1.01)	2.89 (0.93)	1.38 (1.1)	
$\Sigma({}^3F)$	7.5	10.7	10.2	21.4	20.8	26.22	11.49	6.46	46.25

Note: [12]₁: 35ZnO-65TeO_2 , [12]₂: $20\text{Na}_2\text{O-80TeO}_2$, [14]: $60\text{ZrF}_4\text{-31BaF}_2\text{-4YF}_3\text{-2PbF}_2\text{-2AlF}_3\text{-LaF}_3$, and [16]: $36\text{InF}_3\text{-20ZnF}_2\text{-15BaF}_2\text{-20SrF}_2\text{-5CaF}_2\text{-2GaF}_3\text{-2PbF}_2$. Numbers in parentheses indicate the oscillator strength ratios for the given transition and the ${}^3H_4 \rightarrow {}^3F_2$ transition.

Table 3. Oscillator strengths ($f \times 10^6$) of the transitions in Dy^{3+} in different matrices at room temperature

	Transition	Dy1	Dy3	[12] ₁	[12] ₂	[17]	[18]
1	${}^6H_{15/2} \longrightarrow {}^6(F_{11/2} + H_{9/2})$	6.75 (1)	5.56 (1)	10.63 (1)	7.11 (1)	10.7 (1)	1.1
2	${}^6H_{15/2} \longrightarrow {}^6(F_{9/2} + H_{7/2})$	0.94 (0.14)	2.31 (0.41)	3.27 (0.31)	2.59 (0.36)	2.57 (0.24)	2.97
3	${}^6H_{15/2} \longrightarrow {}^6(F_{7/2} + H_{5/2})$	0.75 (0.11)	1.85 (0.33)	2.81 (0.26)	2.09 (0.29)	2.26 (0.21)	2.85
4	${}^6H_{15/2} \longrightarrow {}^6F_{5/2}$	0.44 (0.065)	1.12 (0.2)	1.53 (0.14)	1.27 (0.18)	1.21 (0.11)	1.55
	Σ	8.88	10.8	18.2	13.0	16.75	8.47

Note: [12]₁: 35ZnO–65TeO₂, [12]₂: 20Na₂O–80TeO₂, [17]: the Dy₂O₃ solution in the LiNO₃–KNO₃ melt, and [18]: Dy³⁺(aq)HClO₄–DClO₄. Numbers in parentheses indicate the oscillator strength ratios for the given transition and the ${}^6H_{15/2} \longrightarrow {}^6(F_{11/2} + H_{9/2})$ transition.

by the s th vibration at $T = 0$ K. The summation is carried out over all odd vibrations. At sufficiently high temperatures ($2kT > hv$), function (2) tends to a straight line passing through the origin of the coordinates.

The experimental temperature dependences of the intensity of f - f transitions are due to the competition of the aforementioned mechanisms. As the temperature increases, the transition intensity can increase or decrease depending on the ratio between the contributions of two mechanisms of the allowed transition. In particular, the intensity of the ${}^3H_4 \longrightarrow {}^3H_6$ transition in the Pr1 glass approximately follows relationship (2) for the vibronic mechanism (Fig. 3b). The contribution of the vibronic mechanism to the intensity of the ${}^3H_4 \longrightarrow {}^3F_3$ transition in Pr³⁺ is rather large for the studied glasses (Fig. 3a). The ${}^3H_4 \longrightarrow {}^3F_2$ transition is allowed primarily by static distortions. It should be noted that the temperature dependences of the intensities of transitions, for example, in Pr³⁺ (Fig. 3a), do not depend on the matrix type, even though the matrix strongly affects the magnitudes of these intensities. This means that the character of the temperature dependences and, hence, the ratio of the contributions to the transition intensity are essentially the features of a particular transition rather than of the glass matrix.

The intensity of the ${}^3H_4 \longrightarrow {}^3F_4$ transition in Pr³⁺ in the Pr1 and Pr4 (Pr3) glasses is approximately halved with an increase in the temperature from 93 K to room temperature. Similar effects were observed by Bell *et al.* [9] for the absorption bands of Pr³⁺ in the visible spectral range in the temperature range 21–360°C. The severe decrease in the intensity cannot be explained by the thermal expansion of the glass matrices. This implies that the odd distortions responsible for the intensity of a particular transition substantially decrease with an increase in the temperature. It is quite possible that the same situation, but to a smaller degree, occurs for other transitions and, hence, for other odd distortions. As is known, a decrease in the temperature

often leads to the lowering of the crystal symmetry; i.e., the structural phase transition takes place. In glasses, similar phenomena can be observed at certain temperatures in microscopic regions without macroscopic phase transitions.

The temperature dependences of the intensity of the f - f transitions in Dy³⁺ in the studied glasses are considerably weaker than those of the praseodymium-containing glasses. No specific features of the supersensitive transition are observed. Thus, we can conclude that the odd distortions around rare-earth ions in the dysprosium-containing glasses are weakly temperature dependent as compared to those in the praseodymium-containing glasses.

3.2. Locations and widths of the absorption bands. The location of absorption bands for a material in a condensed state depends primarily on the even components of the interaction between the absorbing ion and the environment (the crystal field and covalence). These interactions vary with an increase in the temperature due to thermal expansion of the material. Hence, the observed temperature dependences of the absorption band location (Fig. 5) reflect the thermal expansion of glasses. The presumably symmetric dispersion of even distortions does not affect the location of the absorption maximum but leads to the broadening of the absorption bands. In glasses, this inhomogeneous broadening makes the main contribution to the linewidth. If the dispersion of even distortions increases with a rise in the temperature, the inhomogeneous broadening should also increase. In crystals, the shape of the f - f absorption bands reflects the density of states of acoustic phonons in the neighborhood of the excited ion (see, for example, [21]). An increase in the temperature results in an increase in the width of these bands due to different relaxation processes. This suggests that the widths of the observed bands should increase with a rise in the temperature. Actually, the widths of the majority of the absorption bands for the praseodymium-containing glasses (Fig. 4) and the widths of all

the absorption bands for the dysprosium-containing glasses increase. The linewidths for the Dy1 (Dy2) glass are less than those for the Dy3 glass, and the linewidths for the Pr1 glass are smaller than those for the Pr4 (Pr3) glass. Consequently, the dispersion of even distortions in the Dy1 (Dy2) glass is smaller than that in the Dy3 glass, and this dispersion in the Pr1 glass is less than that in the Pr4 (Pr3) glass. In both pairs, the smaller dispersion of even distortions is observed for the $\text{SiO}_2\text{-P}_5\text{O}_2\text{-GeO}_2$ glass matrix.

In the Pr1 and Pr4 (Pr3) glasses, the width of the ${}^3H_4 \rightarrow {}^3F_4$ (Pr^{3+}) band exhibits a specific behavior: its value decreases with an increase in the temperature. This is the same transition whose intensity drastically decreases with an increase in the temperature. However, this can be only a coincidence, because the distortions responsible for the intensity and the linewidth differ radically: these are the odd and even distortions, respectively. The observed decrease in the linewidth can be explained by the decrease in the particular even distortion and, as a consequence, by the decrease in its dispersion.

4. CONCLUSION

Thus, in the present work, it was shown that the f - f transitions in the studied glasses are allowed primarily by the static odd distortions, which decrease with an increase in the temperature. The temperature dependences of the intensity of the f - f transitions are not affected by the matrix and depend on the type of electron transitions. The intensity of the ${}^3H_4 \rightarrow {}^3F_4$ transition in Pr^{3+} considerably decreases with an increase in the temperature. This indicates the severe decrease in the particular odd distortions. The same transition is characterized by the specific behavior of the linewidth: it decreases with an increase in the temperature. This effect was explained by the decrease in the particular even distortions.

The comparative analysis of the absorption band parameters for different materials made it possible to determine the differences in the magnitudes, symmetries, and dispersions of the distortions in the nearest environment of rare-earth ions in different glasses. In particular, it was proved that (i) the symmetry of odd distortions depends on the matrix and the rare-earth ion type; (ii) the symmetry of the odd distortions in the Pr4 (Pr3) glass with the LiB_3O_5 matrix is similar to that in the $\text{Pr}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ crystal, but the distortions in the glass are substantially smaller; and (iii) the dispersion of the even distortions is minimum in the $\text{SiO}_2\text{-P}_5\text{O}_2\text{-GeO}_2$ glass matrix.

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