Magneto-Optical Properties of Dy³⁺ in Oxide Glasses: The Origin of the Magneto-Optical Activity of *f–f* Transitions and Its Anomalous Temperature Dependence

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Abstract—The temperature dependence of the absorption spectra and magnetic circular dichroism due to *f–f* transitions from the $^6H_{15/2}$ to $^6F_{5/2}$ and $^6(F_{7/2} + H_{5/2})$ states in the Dy³⁺ ion in (Dy₂O₃–P₂O₅–SiO₂–GeO₂) and (Dy₂O₃–La₂O₃–Al₂O₃–B₂O₃–SiO₂–GeO₂) glasses and the temperature dependence of the Faraday effect were studied. The temperature dependence of the Faraday effect caused by *f–d* transitions was found to differ from that of the magnetic circular dichroism due to *f–f* transitions. It was shown that *f–f* transitions occur preferentially in Dy³⁺ ions associated into clusters. The origin of the paramagnetic magneto-optical activity of *f–f* transitions was analyzed. It was shown that the contributions to this activity can differ in value and sign and that the ratio between these contributions depends on the transition type. In some cases, this difference results in an anomalous temperature dependence of the magneto-optical activity.

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1. INTRODUCTION

Over the past several decades, glasses doped with rare-earth (RE) elements have attracted the attention of researchers. Such glasses are widely used in optical quantum generators and amplifiers. In particular, this also applies to glasses containing Dy³⁺ ions [1, 2]. A huge number of studies have been devoted to the absorption spectra of RE ions in glassy matrices; in particular, glasses with various compositions containing dysprosium were studied, e.g., in [3–6]. Since glasses doped with RE elements are used in magneto-optical devices, particular attention has also been paid to the Faraday effect (FE) (see, e.g., [7–9] and references therein) associated with allowed *f–d* transitions. The magnetic circular dichroism (MCD) observed only within absorption bands (in particular, *f–f* bands) offers additional opportunities for studying degenerate electronic levels split by the crystal field, as well as levels that are not resolved in the optical absorption spectra. A number of studies have been devoted to the spectral dependences of the MCD associated with *f–f* transitions in the Er³⁺, Pr³⁺, and Ho³⁺ ions in soda glasses [10, 11]; the Er³⁺ ion in phosphate glasses [12, 13]; and the Ho³⁺, Nd³⁺, and Pr³⁺ ions in fluoride–zirconate glasses [14–

16]. In [17], the MCD spectra of Dy³⁺ in oxide glasses were presented for the first time simultaneously with the absorption spectra for a number of *f–f* transitions. These transitions were found to differ significantly in terms of paramagnetic magneto-optical activity (MOA). To clarify the mechanism of this phenomenon, we studied the temperature dependence of the MOA of *f–f* transitions in Dy³⁺ in two types of oxide glasses, namely, phosphosilicate–germanate and alumina-borosilicate–germanate glasses. The high Dy concentration in the samples made it possible to study the influence of Dy³⁺ clustering on the MOA. Previously, association of these ions into clusters in glasses containing a significant amount of RE ions was observed using magnetic measurements [18–21].

2. EXPERIMENTAL

The absorption spectra were measured by the two-beam technique using an automated spectrophotometer designed based on an MDR-2 monochromator. The spectral resolution was ~20 cm⁻¹. The MCD was measured by the method of light-polarization modulation using a piezoelectric modulator. The MCD measurement accuracy was 10⁻⁴, and the spectral resolution was ~50 cm⁻¹. The FE was measured by the compensation
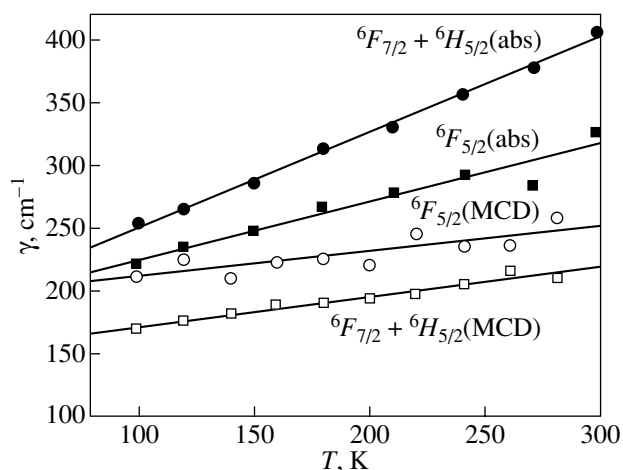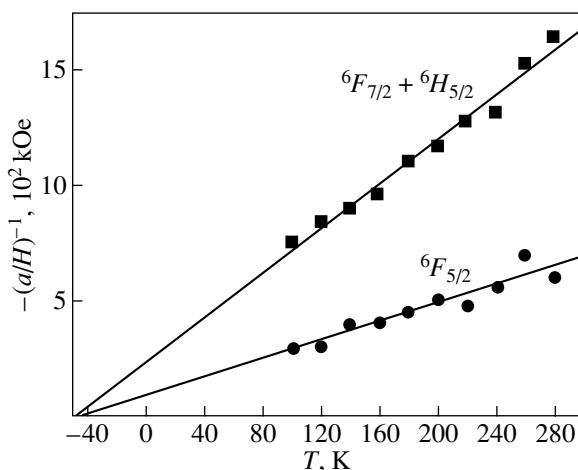
† Deceased.
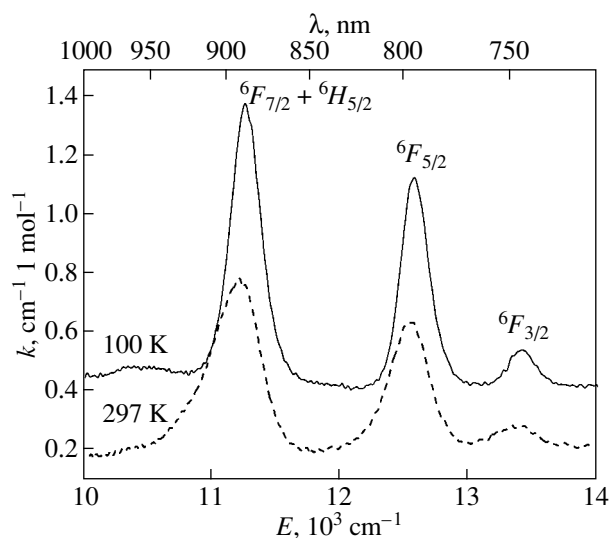
Fig. 1. Optical absorption spectra of the Dy2 glass at temperatures of 100 and 297 K.
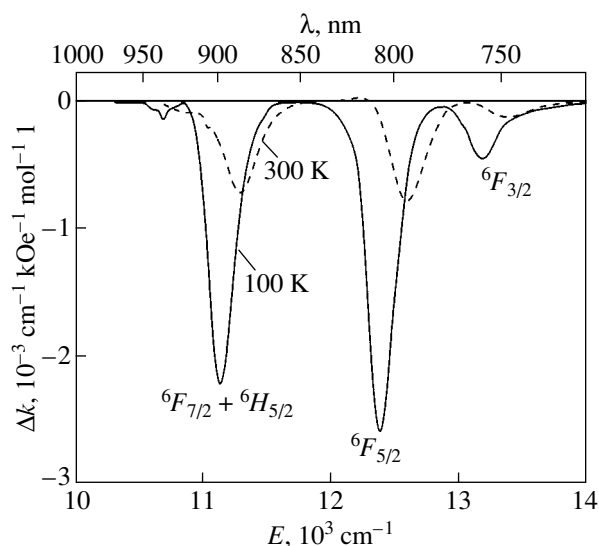
Fig. 2. MCD spectra of the Dy2 glass at temperatures of 100 and 300 K.

Fig. 3. Temperature dependences of the optical-absorption and MCD bandwidths for the Dy2 glass.

Fig. 4. Temperature dependences of the inverse paramagnetic MOA of f–f transitions in Dy^{3+} ions in the Dy2 glass.

technique. The temperature was maintained with an accuracy of ~±1 K. We studied glasses of two compositions: Dy_2O_3–P_2O_5–SiO_2–GeO_2 (Dy2 glass) and Dy_2O_3–La_2O_3–Al_2O_3–B_2O_3–SiO_2–GeO_2 (Dy3 glass), with the Dy_2O_3 mass concentration being 42.3 and 61.8%, respectively. The sample numbers are the same as those in [19]. The technology used to prepare these glasses is described in [7].

Figures 1 and 2 show the absorption and MCD spectra, respectively, of the Dy2 glass at two different temperatures. The spectra of the Dy3 glass differ from those of Dy2 only in terms of the band intensities. By fitting the absorption and MCD bands with Gaussians, we found the temperature dependences of their position, width (Fig. 3), and intensity for transitions from

the $^6H_{15/2}$ to $^6F_{5/2}$ and $^6(F_{7/2} + H_{5/2})$ states. Based on these data, we determined the temperature dependences of the paramagnetic MOA of these transitions, i.e., the ratio of the zeroth moments of the MCD and absorption bands (Figs. 4, 5 show the inverse values of the MOA). The temperature dependences of the inverse FE measured at a wavelength $\lambda = 520$ nm are shown in Fig. 6. These experimental results raise several questions. How can the experimental MOA values and their difference for various transitions be explained? Why do the temperature dependences of the FE and MOA of f–f transitions differ from each other? What is the nature of the deviation of the temperature dependence of the MOA of f–f transitions in the Dy3 glass from the Curie–Weiss law?
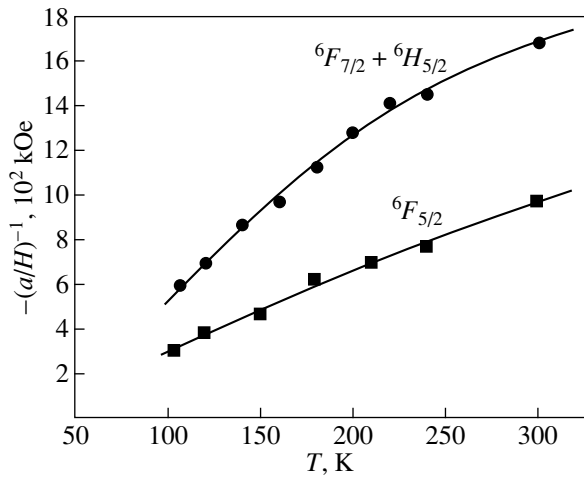
Fig. 5. Temperature dependences of the inverse paramagnetic MOA of f–f transitions in Dy^{3+} ions in the Dy3 glass.
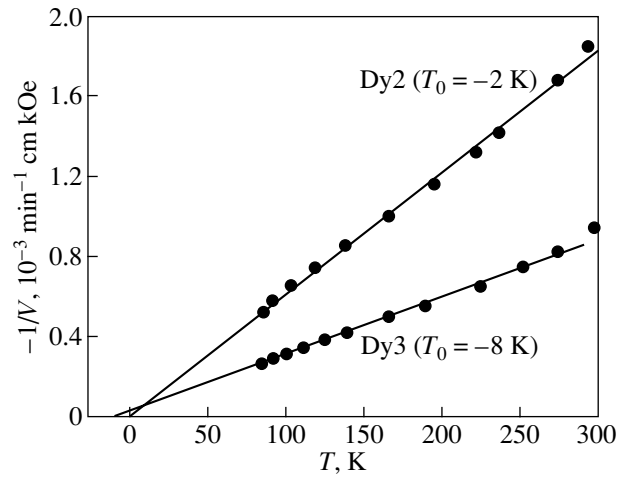

Fig. 6. Temperature dependences of the inverse Faraday effect at a wavelength $\lambda = 520$ nm for the Dy2 and Dy3 glasses.

3. RESULTS AND DISCUSSION

The Dy^{3+} ion states with half-integer total angular momenta are split in the crystal field (CF) (which presumably has low symmetry without an inversion center in glass) into a number of doublet states. A large number of transitions are possible between these states (see, e.g., [22]). In glass, the corresponding narrow absorption lines overlap and form a continuous unstructured absorption band due to the spatial nonuniformity of the local CF. The width of this band is roughly equal to the maximum distance between elementary absorption lines. The split of the ground state of the Dy^{3+} ion in the CF can be rather large (several hundreds of cm^{-1} [23]). Therefore, the observed broadening of the absorption bands with temperature (Fig. 3) is mainly associated with the increased population of the upper components of the split ground state and with the involvement of new transitions into the absorption band. The shape of

the MCD bands (Fig. 2) is qualitatively similar to that of the absorption bands (Fig. 1). However, the MCD bands are narrower than the absorption bands (Fig. 3), which is especially pronounced for the transition to the $^6(F_{7/2} + H_{5/2})$ state. This means that the elementary transitions composing the absorption band differ in terms of their MOA values.

The table shows that the experimentally measured MOA values of the transitions studied differ significantly. Let us analyze the cause of this phenomenon.

In the Judd–Ofelt theory [24], the line strength for a forbidden $i \longrightarrow f$ transition is given by

$$D = |D_{if}|^2 = e^2[3(2J_i + 1)]^{-1}\sum_{\lambda}\Omega_{\lambda}\Gamma_{\lambda}^2 \equiv \sum_{t\lambda}D(t\lambda),\,(1)$$

Experimental and theoretical values of the paramagnetic MOA of transitions at $T = 300$ K

Multiplet	C/D (experiment with Dy2)	C/D (experiment with Dy3)	λ	t	C/D (theory)
$^6F_{7/2}+$	-2.2 ± 0.3	-2.6 ± 0.3	4	3	+5.66
				5	−4.53
			6	5	+5
				7	−4.28
$+^6H_{5/2}$			6	5	+5.38
				7	−4.61
$^6F_{5/2}$	-5.2 ± 0.3	-4.6 ± 0.3	6	5	+5.38
				7	−4.61
$^6F_{3/2}$	-6 ± 1	-6 ± 1	6	5	+5.66
				7	−4.88

Note: C/D is the integrated MOA of transitions. The parameters λ and t introduced in formula (1) are explained in the text.

where $\Omega_\lambda = \sum_t \Omega_\lambda(t)$ are the Judd–Ofelt parameters; Γ_λ are theoretically calculated parameters, which depend only on the transition type and the environment symmetry; and $D(t\lambda)$ are the partial line strengths describing the contributions of terms with various values of t and λ to the line strength. The numbers λ are even integers. The maximum value $\lambda_{max} = 2l$, where l is the electron orbital quantum number. Thus, $\lambda = 2, 4,$ and 6 for the f shell. The numbers λ characterize the selection rules for parity-forbidden transitions:

$$|J_f - J_i| \le \lambda. \tag{2}$$

The parameter t is the tensor rank of a spherical harmonic in the CF expansion in terms of spherical harmonics and takes on values

$$t = \lambda \pm 1. \tag{3}$$

The zeroth moment of the absorbance in the region of the transition under consideration is proportional to the line strength:

$$\langle k(\omega) \rangle_0 \sim D. \tag{4}$$

There are three contributions to the MCD: (i) from the diamagnetic effect, (ii) from the temperature-dependent paramagnetic effect, and (iii) from the temperature-independent paramagnetic effect or the mixing effect. The zeroth moment of an MCD absorption band characterizes the total paramagnetic contribution to dichroism (the contribution of the diamagnetic effect to the zeroth moment is identically zero). Extrapolation of the MOA as a function of $1/T$ to $1/T = 0$ ($T = \infty$) yields the mixing effect. Such a procedure for Dy2 yields a zero mixing effect to within experimental error. Thus, we can write

$$\langle \Delta k(\omega) \rangle_0 \sim C\mu_B H/kT, \tag{5}$$

where H is the magnetic field, T is the temperature, and C is a constant that is dependent on the electronic transition type. Therefore, the integrated paramagnetic MOA of a transition is given by

$$a = \frac{\langle \Delta k(\omega) \rangle_0}{\langle k(\omega) \rangle_0} = \frac{C}{D}\frac{\mu_B H}{kT}. \tag{6}$$

In glass, the parameters of both even and odd CF components are spatially inhomogeneous. In [13], statistical averaging over all possible orientations of the axes of RE centers was carried out. (It should be noted that, as shown in [3], the distortions of the RE-ion nearest environment themselves are not random.) This made it possible to simplify the problem and find the paramagnetic MOA of the $J_i \longrightarrow J_f$ transition corresponding to particular values of λ and t to be [13]

$$A = \frac{C(t, \lambda)}{D(t, \lambda)} = \frac{g_i}{8\lambda(\lambda + 1)}[t(t + 1) - \lambda(\lambda + 1) - 2]$$
$$\times [J_f(J_f + 1) - J_i(J_i + 1) - \lambda(\lambda + 1)], \tag{7}$$

where g_i is the Landé factor of the initial state. It is interesting that this expression does not involve the parameters Ω_λ, which characterize the integrated intensity of the $J_i \longrightarrow J_f$ transition and enter into Eq. (1). However, in order to calculate the MOA of the transition summed over all possible values of the parameter λ, it is necessary to know the parameters Ω_λ even for a certain value of t [13]. These parameters for glass are practically impossible to determine, since prior theoretically calculation of the parameters Γ_λ in Eq. (1) would be required and these parameters are dependent on the CF symmetry, which is unknown for glass. However, the parameters Γ_λ are generally considered independent of the environment. It follows from Eqs. (2) and (3) that the possible parameter values are as follows: $\lambda = 6$ ($t = 5, 7$) for the ($J = 15/2$) $\longrightarrow$ ($J = 5/2$) and ($J = 15/2$) $\longrightarrow$ ($J = 3/2$) transitions and $\lambda = 4$ ($t = 3, 5$) and $\lambda = 6$ ($t = 5, 7$) for the ($J = 15/2$) $\longrightarrow$ ($J = 7/2$) transition. The table lists the values of the MOA of the transitions under discussion calculated from formula (7) at all possible values of the parameters λ and t, as well as the experimental values of the MOA at $T = 300$ K. The uncertainty in the theoretical predictions is smallest for the transitions to $^6F_{5/2}$ and $^6F_{3/2}$. The experimental paramagnetic MOA of these transitions corresponds (in sign and magnitude) to the activity of the seventh harmonic in the CF. The transition to the $^6(F_{7/2} + H_{5/2})$ state has a significantly lower paramagnetic MOA. This indicates that the transition intensity is also due to other CF harmonics or terms in Eq. (1) with other values of λ, whose contribution to the MCD is of the opposite sign (see table).

We can also use another approach to the problem of the MOA of the f–f transitions. For an allowed transition in a free atom, the paramagnetic MOA of a pair of transitions from the components of the ground state splitting in a magnetic field according to the magnetic quantum number $\pm m$ can be easily shown to be $A_m = g|m|p_m$ (in units of $\mu_B H/kT$), where g is the Landé factor of the ground state and p_m is the degree of circular polarization of the transition from the $\langle -|m| \rangle$ state, taking into account the polarization sign. Then, the integrated MOA of the $J_i \longrightarrow J_f$ transition is given by

$$A = g \sum_{|m|} |m| p_m V_m, \tag{8}$$

where V_m is the weight of the pair intensity in the total transition intensity. According to the Wigner–Eckart theorem, the Zeeman component intensities are proportional to the squared 3j symbol:

$$V \sim \begin{pmatrix} J_f & 1 & J_i \\ -m_f & \mp 1 & m_i \end{pmatrix}^2. \tag{9}$$

These quantities are given, e.g., in [25, p. 201]. Using Eq. (7), we find that

for transition $J \longrightarrow (J-1), \quad A = -g(J+1)/2,$ (10)

for transition $J \longrightarrow J, \quad A = -g/2,$ (11)

for transition $J \longrightarrow (J+1), \quad A = +gJ/2.$ (12)

These relations are valid for both integer and half-integer angular momenta. We can see that the paramagnetic MOA is strongly dependent on the excited state, whereas for the $J \longrightarrow J$ transition the MOA depends only on the Landé factor of the ground state.

The f–f transitions become allowed due to the admixture of states with opposite parity. These states should also provide allowance by the total-angular-momentum selection rules. Let us consider the admixture of states to only an excited state, since it is closer to the odd states of the $4f^8 5d$ configuration. In this case, the transition will be allowed if the following total-angular-momentum selection rule is satisfied for the admixed J_f' state:

$$\left| J_f' - J_i \right| \le 1.$$ (13)

Thus, the f–f transitions from the $J = 15/2$ ground state can become allowed due to an admixture of a state with $J = 13/2, 15/2,$ or $17/2$. Using Eqs. (10)–(12) and taking into account that the Landé factor of the ground state is $g = 4/3$, we find that the MOA of transitions to these states is $-5.66, -0.66,$ and 5, respectively. Comparing these values with the experimental data (see table), we can conclude that only the $J = 13/2$ state is admixed to the $^6F_{5/2}$ and $^6F_{3/2}$ states and that the states with $J = 15/2$ and/or $J = 17/2$ can likewise be admixed to the $^6F_{7/2} + {}^6H_{5/2}$ states.

According to Eqs. (13) and (2), the admixed states should satisfy the condition

$$\left| J_f - J_f' \right| \le \lambda - 1.$$ (14)

It follows from Eqs. (13) and (14) that only the $J = 13/2$ state can be admixed to the $^6F_{3/2}$ state, which is confirmed experimentally. The states with $J = 15/2$ and $13/2$ can be admixed to the $^6F_{5/2}$ and $^6H_{5/2}$ states. However, it follows from the experiment that, even if the $J = 15/2$ state is admixed, its weight is negligible. The states with $J = 17/2, 15/2,$ and $13/2$ can be admixed to the $^6F_{7/2}$ state. Indeed, the small value of the MOA of the transition to the $\longrightarrow {}^6F_{7/2} + {}^6H_{5/2}$ state (see table) suggests the presence of the $J = 17/2$ state giving a positive MOA. The admixed states should have a parity opposite to that of the ground state; i.e., they should correspond to the $4f^8 5d$ configuration. The ground state of the $4f^8$ core is $^7F_6$; i.e., it has $L = 3$ and $S = 3$. Therefore, the $4f^8 5d$ configuration can have $L = 1$ to 5, $S = 5/2$ or $7/2$, and the maximum total angular momentum $J = 17/2$. Thus, all the abovementioned admixed states indeed exist.

A comparison of the values of the MOA calculated for a free ion and the experimental values shows that the CF causes almost no suppression of the MOAs of transitions, which is characteristic of ions with half-integer angular momenta. Indeed, a low-symmetry CF does not completely remove the degeneracy of the Dy^{3+} states. They split into Kramers doublets, which are sometimes considered quasi-spins with various Landé factors. The experimentally measured g factors of the ground-state components are of the order of 10 [26]. Then, under the assumption that the transitions between these doublets are of the J–J type, relation (11) yields MOA values close to the experimental values, but not for all transitions; i.e., this approach is very rough.

Since the degeneracy of the states of Dy^{3+} is not removed completely even in a low-symmetry CF, the magnetic susceptibility of isolated Dy^{3+} ions in glasses approximately obeys the Curie law [18]. The magnetic susceptibility of oxide Dy_2O_3 follows the Curie–Weiss law with a Weiss constant $\theta = -18$ K [27]. Glass with a high dysprosium concentration can contain both isolated dysprosium ions and clusters similar to the oxide. In this case, the paramagnetic susceptibility of glass can be written as

$$\chi = C_i/T + C_c/(T - \theta),$$ (15)

where

$$C_i = n_i m_i^2/3k, \quad C_c = n_c m_c^2/3k$$ (16)

are the Curie constants for isolated and cluster ions; m_i and m_c are the high-temperature $(T \gg |\theta|)$ values of the magnetic moments of these ions; and n_i and n_c are the numbers of isolated and cluster ions, respectively. The diamagnetic susceptibility is disregarded in Eq. (15), since its contribution is not large at a high concentration of paramagnetic ions. Linear extrapolation of the temperature dependence inverse to susceptibility (15) in the high-temperature region to zero in the presence of clusters yields a temperature T_0 intermediate between zero and θ. We shall determine this temperature in the following way. Equation (15) can be written as

$$\frac{1}{\chi} = \frac{T(T-\theta)}{C_i(T-\theta) + C_c T}$$

$$= \frac{T-\theta}{(C_i + C_c)\left(1 - \dfrac{C_i}{C_i + C_c}\dfrac{\theta}{T}\right)}.$$ (17)

At $T \gg |\theta|$, we have

$$\frac{1}{\chi} \approx \frac{(T-\theta)\left(1 + \dfrac{C_i}{C_i + C_c}\dfrac{\theta}{T}\right)}{(C_i + C_c)} \approx \frac{T - \dfrac{\theta C_c}{C_i + C_c}}{C_i + C_c}.$$ (18)

Thus, the plot of function (17) asymptotically approaches a straight line. Equating expression (18) to

zero, we determine the intersection point of this straight line with the temperature axis:

$$\frac{T_0}{\theta} = \frac{C_c}{C_i + C_c} \equiv K. \tag{19}$$

With sufficient accuracy, we can set $m_i = m_c$. Then, according to Eq. (16), the parameter K characterizes the degree of clustering of Dy^{3+} ions. Extrapolation of dependence (17) in a bounded high-temperature region obviously yields a value of T_0 that is underestimated in comparison with the accurate value given by Eq. (19).

A magnetic field splits Kramers doublets, and a temperature-dependent paramagnetic MCD occurs. Van Vleck and Hebb [28] showed that the paramagnetic FE is proportional to the paramagnetic susceptibility,

$$V = k\chi. \tag{20}$$

Experiments confirm that, as a rule, the temperature dependences of these quantities are indeed identical. However, the proportionality factor k depends not only on the ion type and wavelength of measurement but also on the property of the electronic transition responsible for the observed magneto-optical effect [28]. It is this situation that occurs in the case under consideration. We measured the temperature dependence of the FE at a wavelength $\lambda = 520$ nm (Fig. 6), which is far from f–f transitions. In this case, the FE is caused by allowed f–d transitions in the ultraviolet spectral region. The intensity of allowed f–d transitions is almost independent of the ligand environment symmetry. Therefore, the FE equally reflects the contributions of both isolated and cluster ions and its temperature dependence is indeed identical to that of the paramagnetic susceptibility in glasses as well [19]. The value $T_0 = -8$ K obtained for Dy3 from the FE is close to the value obtained by linear extrapolation of the magnetization of similar glasses [19]. The observed MCD and corresponding MOA are caused by f–f transitions (see the temperature dependences of the inverse MOA in Figs. 4, 5). The intensity of f–f transitions is nonzero because the ligand environment is not centrosymmetric. Therefore, the contributions of isolated ions and cluster ions to the MOA of f–f transitions can differ significantly. The large value of the Weiss constant determined for the Dy2 glass from f–f transitions, $\theta = -43 \pm 3$ K (Fig. 4), suggests that f–f transitions occur almost exclusively in clusters. This fact, in turn, suggests that the ligand environment of isolated Dy^{3+} ions is much more symmetric than that in clusters. Indeed, at least the environment of ions on the cluster surface is a priori asymmetric. The smaller the cluster, the larger the surface ion fraction. Close values of the Weiss constant for clusters were obtained previously from magnetic measurements on other glasses containing Dy^{3+} [19], and these values significantly exceed (in magnitude) the Weiss constant of pure dysprosium oxide. As is known, the nearest neighbor atoms of transition-element ions have a decisive effect on the interaction between these ions.

Hence, the difference between the Weiss constants means that the symmetry of the nearest neighbor atoms of dysprosium ions in glass differs significantly from that in the crystalline oxide. Thus, assuming that the measured MOA of f–f transitions yields the Weiss constant of clusters and that the FE yields the temperature T_0 (Fig. 6), we can approximately estimate the degree of clustering from formula (19). The value of T_0 is determined with an accuracy of ± 0.5 K. Then, we obtain $K > (5.0 \pm 1.2)\%$ for the Dy2 glass. The situation with the Dy3 glass is significantly different and requires additional discussion.

The values of the MOA of f–f transitions differ from each other (see table), since they depend not only on the ground state but also on excited states. Moreover, as was shown above, the MOA of f–f transitions can contain independent contributions that are opposite in sign. It is clear that the ratio of these contributions can depend on the population of the components of the CF-split ground state. This means that there is an additional source of the temperature dependence of the MOA. This dependence can be different for different f–f transitions and can fundamentally differ from that of the magnetic susceptibility, in contrast to f–d transitions. The temperature dependences of the MOA of the f–f transitions studied in the Dy2 glass differ only slightly and follow the Curie–Weiss law. Therefore, we can conclude that the temperature dependence of the MOA in this case indeed correlates with that of the magnetic susceptibility of clusters in the Dy2 glass. In the Dy3 glass, the temperature dependences of the inverse MOA of f–f transitions have no linear portions in the temperature range studied. This behavior suggests competition of the above-described contributions to the MOA of f–f transitions. As the temperature increases further and, hence, all the components of the CF-split ground state are populated, the temperature dependences of a^{-1} for Dy3 will probably likewise become linear. We are planning to perform further experiments in the future.

4. CONCLUSIONS

The paramagnetic MOA of f–f transitions consists of several contributions that differ in magnitude and sign, with the ratios between them being dependent on the transition type. The ratio of the contributions also depends on the population of the crystal-field-split ground state components, which results in an additional temperature dependence of the integrated MOA. This explains the anomalous temperature dependence of the MOA of one of the glasses studied. The temperature dependences of the FE and MOA of f–f transitions differ because the FE is controlled by allowed transitions, which occur in a single ion and in a cluster with equal probability. This is not the case for forbidden f–f transitions, since they are caused by distortions of the ligand environment. The results of the experiments discussed above suggest that f–f transitions occur mostly in clusters.

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