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Effect of hydrostatic pressure on the free-ion parameters and the crystal-field strength for YOCl:Eu^{3+}

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Abstract

The fluorescence spectra of Eu^{3+} in YOCl:Eu^{3+} were measured under pressure up to 10.3 GPa at room temperature. The pressure dependence of the crystal-field levels of the $^7F_{0-4}$ and $^5D_{0-2}$ multiplets for the Eu^{3+} ion was determined from the spectra. From the simulations of the experimental data, the free-ion parameters F_2 and ζ, the crystal-field parameters B_q^k and the crystal-field strength parameter S_t were obtained at various pressures. The result of the simulation shows that the crystal-field strength increases with pressurization, which is contrary to the results of its isomorphic compounds, e.g. LaOCl and LaOBr doped with Eu^{3+}. The electrostatic repulsion and the spin–orbit coupling interactions of 4f electrons decrease with increasing pressure. The central-field covalency mechanism and the symmetry-restricted covalency mechanism play a leading role in the relative reductions of ζ and F_2, respectively.
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1. Introduction

When a rare earth (RE) ion is incorporated in a crystal, the ion interacts with its crystal-field (CF) environment. As a result, its free-ion energy levels are shifted and split into CF levels. For the RE ion in the crystal, its energy level scheme, intraionic interaction and the interionic interaction between the RE ion and its ligand ions are very complicated, so clarification of the quantitative relation between the level scheme and the interactions is an arduous project. Still further, when we apply a hydrostatic pressure on the crystal, the intraionic interaction of the RE ion and the interionic interaction between the RE ion and its crystal environment are changed. For this reason hydrostatic pressure becomes a powerful tool for the direct study of how the CF environment affects the electronic state of the RE ion and for obtaining some information about the CF environment from the change of the electronic state under pressure.

The Eu^{3+} ion is of great advantage for studying the CF effect on electronic state because of the particular features of its energy level scheme. The RE oxyhalides (ROX: R = La, Gd, Y; X = Cl, Br) can form several isostructural host lattices by varying the host anion or the cation. Therefore ROX doped with Eu^{3+} were paid attention to [1,2]. Lots of work on ROX:Eu^{3+} have been conducted at ambient and high pressures [3–10]. According to the traditional CF theory, the interatomic distance in crystal is reduced and thus the CF strengthens with increasing pressure [11]. However, in high-pressure-luminescence study about the RE oxyhalides doped with Eu^{3+} (LaOCl:Eu^{3+}, LaOBr:Eu^{3+}, GdOCl: Eu^{3+}, GdOBr:Eu^{3+}) we have found that the CF acting on the Eu^{3+} ion in the oxyhalides decreases with the increase of pressure [3,4,9,12]. This is a kind of anomalous pressure effect of CF strength, the mechanism of which have not reached a deep understanding so far. A lot of experimental and theoretical researches are still needed. Recently, we investigated the influence of hydrostatic pressure on the intraionic and interionic interactions of Eu^{3+} in YOCl. In view of the chemical and structural similarities it was expected that its CF strength decreases with pressure as LaOCl:Eu^{3+} does, but the result turned out to be contrary

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to that expected. In this paper the result was reported in detail.

2. Experiments

The sample YOCl:Eu^{3+} used in this work is polycrystalline powder prepared by a solid reaction at high temperature. The concentration of Eu^{3+} in the sample is nominally 5.0 mol%. The fluorescence spectra from the Eu^{3+} ion in YOCl were recorded with a Spex-1403 Raman spectrometer under excitation of 457.9 nm line from an Ar^{+} laser. A gasketed diamond-anvil cell was used for generating pressure. A methanol–ethanol–water mixture with a ratio of 16:3:1 (v/v) was used as the pressure-transmitting medium. The pressure was calibrated by the shift of the ruby R$_1$ line. All the spectral measurements were carried out at room temperature.

3. Pressure dependence of fluorescence spectra and CF levels

Altogether 72 fluorescence lines within the limits of 12 900–21 500 cm^{-1} were measured at ambient pressure and assigned to the transitions between the CF components of the $^5D_{0-2}$ and $^7F_{0-5}$ multiplets of Eu^{3+} with the aid of Holsa's work [1]. Because the fluorescence is weakened under pressure, the number of the fluorescence lines available is reduced gradually with the increase of pressure. At 10 GPa only 36 stronger lines could be observed, which all shift to the red under pressure, their shift rates are between 3 and 9 cm^{-1}/GPa. Although the positions and the intensities of the spectral lines vary gradually with increasing pressure, neither abrupt change nor new spectral line occurs in the pressure range of our experiments. This shows that under pressure up to 10.3 GPa the C_{4v} site symmetry of Eu in YOCl is kept essentially, and there is no structural phase-transition. A part of the fluorescence spectra at pressures is presented in Fig. 1, from which we can see the spectral line broadening in the process of pressurization. We consider that the line broadening originates from slight structure distortion and small deviation from hydrostatic pressure in crystal under pressure. The pressure dependence of the positions for the 36 stronger spectral lines is shown in Fig. 2. The pressure dependence of the 25 CF levels is deduced from the fluorescence lines observed at various pressures and plotted in Fig. 3. Under pressure the CF levels of $^7F_{0-5}$ multiplets shift upwards or downwards and all the CF components of $^5D_{0-2}$ multiplets moved downwards. The shifts of the levels of $^5D_{0-2}$ multiplets with pressure are much more rapid than those levels of $^7F_{1-5}$ multiplets. This is the reason why all the 36 stronger lines shift to the red.

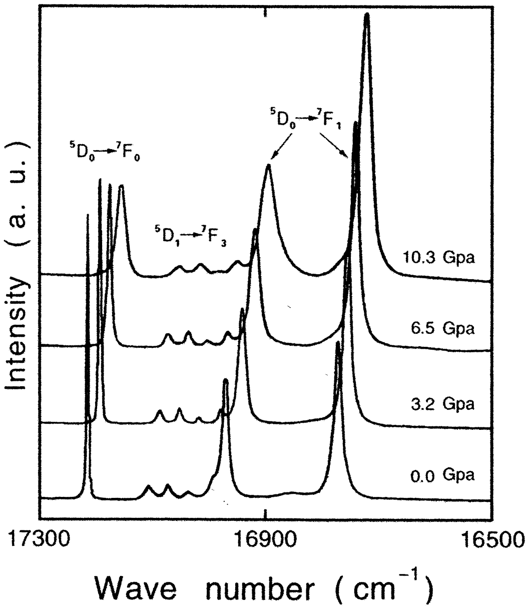

Fig. 1. A part of the fluorescence spectra for YOCl:Eu at various pressures.

4. Theory and simulating procedure

The Hamiltonian of an RE ion in a crystal is usually written as [13]:

$$H = H_F + H_C \tag{1}$$

where H_F is the free-ion Hamiltonian representing the interaction inside the RE ion.

Neglecting some weak interactions, H_F can be expressed as:

$$\begin{aligned} H_F = &\sum_k f^k F_k + \zeta \sum_i s_i \cdot l_i \\ &+ [\alpha L(L+1) + \beta G(G_2) + \gamma G(R_7)] \end{aligned} \tag{2}$$

in which the first term represents the Coulomb repulsion of 4f electrons, the second term stands for the spin–orbit coupling interaction and the others express the configuration interaction. F_k are the Slater parameters and f^k the corresponding coefficients. ζ is the spin–orbit coupling parameter, s_i and l_i are the spin angular momentum and orbital angular momentum of the ith 4f electron, respectively. α, β and γ are the configuration interaction parameters, L is the total orbital angular momentum, $G(G_2)$ and $G(R_7)$ are the eigenvalues of Casimir's operators for the groups G_2 and R_7 used to classify the states of the f^n configuration. H_C is the CF Hamiltonian, which represents the interaction between the RE ion and its crystal environment and is taken as a perturbation to H_F.

The Eu^{3+} ion in YOCl has C_{4v} site symmetry and its CF Hamiltonian can be expressed by five nonzero CF parameters B^k_q to be determined:

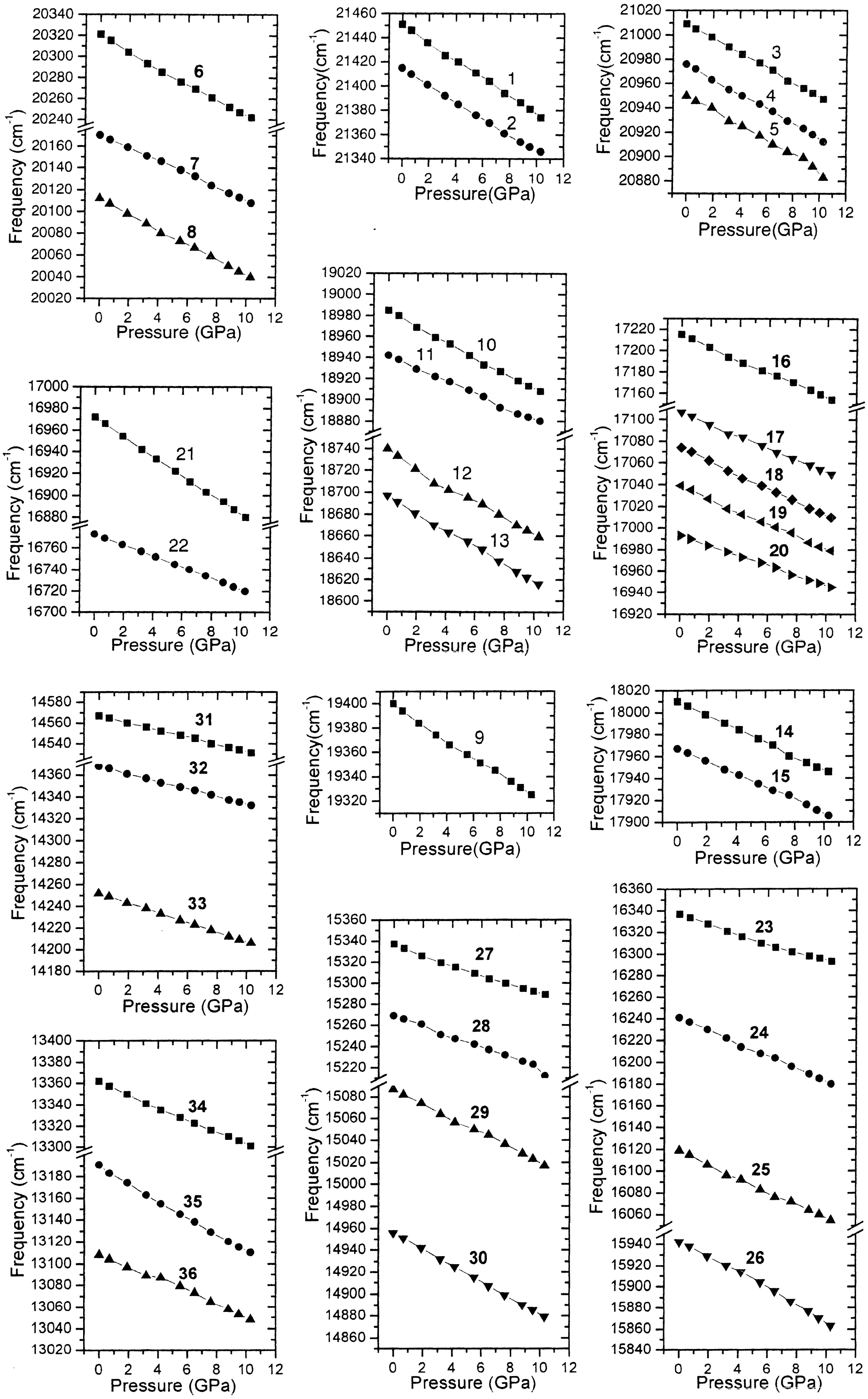

Fig. 2. The frequencies of the 36 stronger spectral lines versus pressure for YOCl:Eu. The correspondence relations between numerals and transitions in the figure are as follows: 1, $^5D_2$ (E)→$^7F_0$ (A_1); 2, $^5D_2$ (A_1)→$^7F_0$(A_1); 3, $^5D_2$ (E)→$^7F_1$ (E); 4, $^5D_2$ (A_1)→$^7F_1$ (E); 5, $^5D_2$ (B′)→$^7F_1$ (E); 6, $^5D_2$ (A_1)→$^7F_2$ (A_1); 7, $^5D_2$ (E)→$^7F_2$ (B′); 8, $^5D_2$ (B′)→$^7F_2$ (B′); 9, $^5D_2$ (B′)→$^7F_3$ (B′); 10, $^5D_1$ (E)→$^7F_0$ (A_1); 11, $^5D_1$ (A_2)→$^7F_0$ (A_1); 12, $^5D_1$ (E)→$^7F_1$ (A_2); 13, $^5D_1$ (A_2)→$^7F_1$ (A_2); 14, $^5D_1$ (E)→$^7F_2$ (E); 15, $^5D_1$ (A_2)→$^7F_2$ (E); 16, $^5D_0$ (A_1)→$^7F_0$ (A_1); 17, $^5D_1$ (E)→$^7F_3$ (E); 18, $^5D_1$ (A_2)→$^7F_3$ (A_2); 19, $^5D_1$ (E)→$^7F_3$ (E); 20, $^5D_1$ (A_2)→$^7F_3$ (E); 21, $^5D_0$ (A_1)→$^7F_1$ (A_2); 22, $^5D_0$ (A_1)→$^7F_1$ (E); 23, $^5D_1$ (E)→$^7F_4$ (A_1); 24, $^5D_0$ (A_1)→$^7F_2$ (E); 25, $^5D_0$ (A_1)→$^7F_2$ (A_1); 26, $^5D_1$ (E)→$^7F_4$ (B′); 27, $^5D_0$ (A_1)→$^7F_3$ (E); 28, $^5D_0$ (A_1)→$^7F_3$ (E); 29, $^5D_1$ (A_2)→$^7F_5$ (E); 30, $^5D_1$ (E)→$^7F_5$ (A_1); 31, $^5D_0$ (A_1)→$^7F_4$ (A_1); 32, $^5D_0$ (A_1)→$^7F_4$ (E); 33, $^5D_0$ (A_1)→$^7F_4$ (A_1); 34, $^5D_0$ (A_1)→$^7F_5$ (E); 35, $^5D_0$ (A_1)→$^7F_5$ (A_1); 36, $^5D_0$ (A_1)→$^7F_5$ (E).

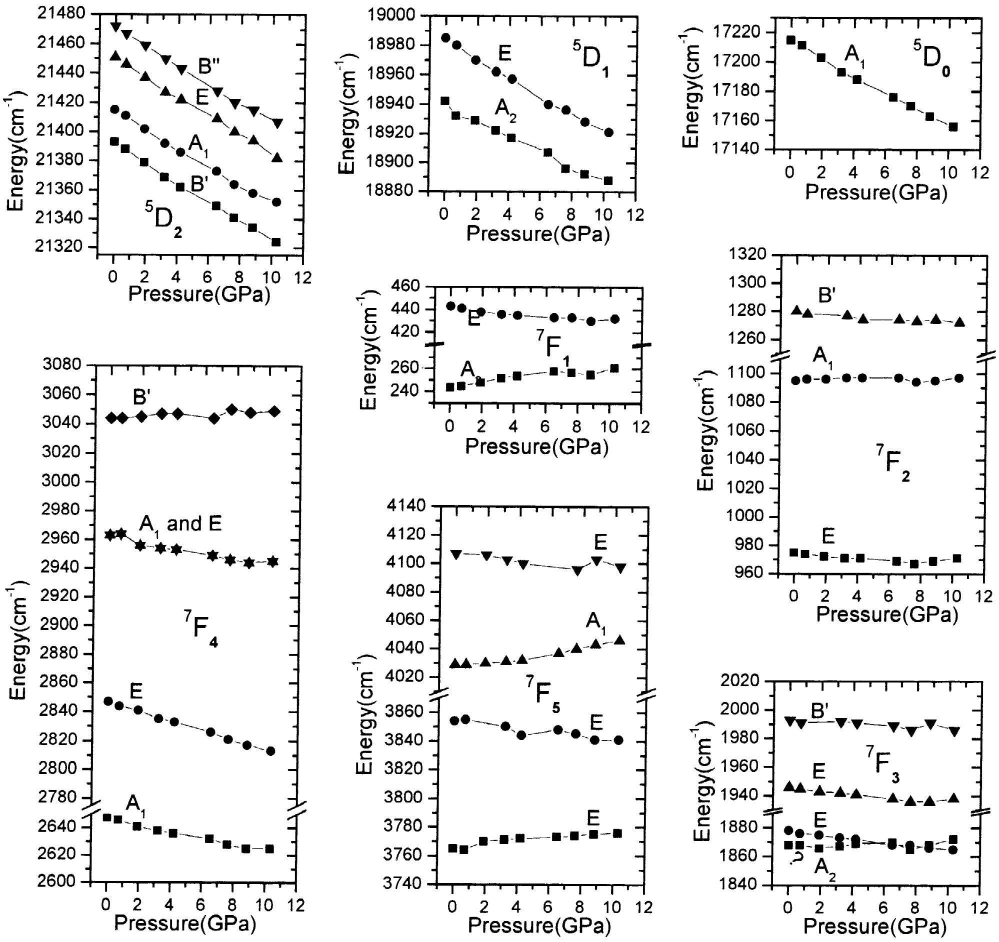

Fig. 3. The pressure dependence of the 25 CF levels of $^7F_{0-5}$ and $^5D_{0-2}$ multiplets for Eu^{3+} in YOCl.

$$H_C = B_0^2 C_0^2 + B_0^4 C_0^4 + B_4^4(C_{-4}^4 + C_4^4) + B_0^6 C_0^6 + B_4^6(C_{-4}^6 + C_4^6) \qquad (3)$$

where the tensor operators C_q^k are related to the spherical harmonics Y_q^k by:

$$C_q^k = [4\pi/2k + 1]^{1/2} \sum_i Y_q^k(\vartheta_i,\varphi_i) \qquad (4)$$

In order to determine these empirical parameters in Eqs. (2) and (3), the simulated calculation of CF levels was performed in two steps.

The first step is a free-ion calculation, in which the matrix of H_F was constructed with an L–S coupling wave-function and the intermediate coupling wave-function used in the next step was determined by diagonalizing the matrix of H_F. The parameters α, β, γ and the ratios of Slater radial integrals, F_4/F_2 and F_6/F_2, were fixed to reasonable values in the pressure range of this work to minimize the number of the free-ion parameters used, they are equal to 20, −550, 1100, 0.1484 and 0.0161, respectively. The observed values of the barycenters of the $^{2S+1}L_J$ multiplets were regarded as the experimental values of their free-ion levels, ignoring the second order effect caused by the CF. In a least-squares refining calculation the best-fit values of F_2 and ζ were gained by fitting to the experimental multiplet barycenters. In the case where the complete CF levels of some multiplets could not be observed experimentally to determine their barycenters, the missing CF levels were replaced with the calculated ones.

In second step a 204×204 matrix of H was constructed with the intermediate coupling wave-function obtained at the last of the first steps, which consists of the elements between the ^7F, ^5D, ^5L and ^5G spectral terms. The ^5L and ^5G terms were involved because their energy levels are very close to, and mixed with the energy levels of the ^5D term. The intermediate coupling wave-function used in this step includes all the septets, quintets, triplets and singlets. In the calculation of this step the free-ion parts of the diagonal elements of H were treated as adjustable parameters, one for each $^{2S+1}L_J$. The J-mixing caused by the CF was also taken into consideration by comprising the off-diagonal matrix elements between the states with different J-values. The eigenvalues of the CF levels in ^7F_J and ^5D_J were obtained by diagonalizing the matrix of H. By

Table 1
Experimental and calculated free-ion energy levels (cm^{-1}) for Eu^{3+} doped in YOCl at different pressures

	Pressure (GPa)											
	0.0		2.0		4.0		6.0		8.0		10.0	
	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.
F_0	0	0	0	0	0	0	0	0	0	0	0	0
$^7F_1$	376	386	375	386	375	386	374	385	374	385	374	385
$^7F_2$	1063	1064	1061	1063	1060	1062	1059	1061	1058	1060	1056	1059
$^7F_3$	1929	1936	1928	1934	1926	1932	1923	1931	1922	1929	1921	1928
$^7F_4$	2912	2932	2908	2930	2904	2927	2901	2925	2898	2923	2895	2921
$^5D_0$	17 215	17 244	17 202	17 232	17 191	17 222	17 179	17 211	17 168	17 200	17 156	17 189
$^5D_1$	18 971	18 953	18 957	18 939	18 945	18 927	18 933	18 915	18 921	18 904	18 909	18 891
$^5D_2$	21 436	21 426	21 422	21 410	21 409	21 396	21 396	21 382	21 384	21 369	21 371	21 355

adjusting the values of the CF parameters to fitting calculated CF levels to the observed ones, the best-fit values of B_q^k were gotten from the refining calculation.

5. Pressure effect on free-ion state

5.1. Free-ion energy levels and free-ion parameters

The experimental values of the eight barycenters of the $^7F_{0-4}$ and $^5D_{0-2}$ multiplets are deduced from the observed CF levels of Eu^{3+} in YOCl in 10 GPa range and are given in Table 1. Within the limits of pressure in present work the multiplet barycenters all descend with increasing pressure, and the descending rates of the $^5D_{0-2}$ multiplets are much larger than those of the $^7F_{0-4}$ multiplets. At 10 GPa, the barycenters of the $^5D_0$, $^5D_1$ and $^5D_2$ multiplets dropped 0.34, 0.33 and 0.30%, respectively. For Eu^{3+} in the YOCl host the best-fit values of F_2 and ζ, the relative changes in F_2 and ζ and the ratios of their relative changes at various pressures are tabulated in Table 2. The calculated values of the free-ion levels by using these parameters are given in Table 1. The rms deviation between the calculated and experimental free-ion levels is not larger than 21 cm^{-1}.

From Table 2 it can be seen that the Slater parameter F_2 and the spin–orbit coupling parameter ζ monotonically decrease with pressurization, which brings about the descents of multiplet barycenters of Eu^{3+} in YOCl under pressure. Within the scope of our experimental pressures the relative reduction in F_2 is always less than that in ζ, and the ratio of their relative reductions are between 0.8 and 0.9.

5.2. Covalency mechanism of reductions in F_2 and ζ under pressure

The pressure forces the nuclear separation between the Eu^{3+} ion and its ligands to decrease in the YOCl:Eu^{3+} crystal, which makes the 4f electrons of Eu^{3+} delocalized, as a result the free-ion parameters decrease. In other words, the reductions of the Slater parameter F_2 and the spin–orbit coupling parameter ζ can be attributed to the 4f electron cloud expansion of the Eu^{3+} ion in YOCl. The expansion is modeled by a combination of two different covalency mechanisms [5]: the central-field covalency mechanism (CFCM) and the symmetry restricted covalency mechanism (SRCM). Agreeably to the model, the 4f orbitals expand when a RE free-ion is incorporated in a crystal or the crystal is compressed. The CFCM relates the reductions of F_2 and ζ of a RE ion in a crystal to an isotropic expansion of the 4f orbitals. This expansion is caused by screening the effective nuclear charge z_{ef} of the RE ion and the following relations nearly hold [5]:

$$\Delta F_2/F_2 = \Delta Z_{ef}/Z_{ef} \tag{5}$$

and

Table 2
Slater parameter F_2, spin–orbit coupling parameter ζ, relative changes of F_2 and ζ, ratio of the relative changes for Eu^{3+} in YOCl:Eu^{3+} at various pressure

	Pressure (GPa)					
	0.0	2.0	4.0	6.0	8.0	10.0
F_2	389.57±0.02	389.28±0.03	389.03±0.02	388.77±0.02	388.54±0.02	388.28±0.03
ζ	1339.75±0.27	1338.68±0.26	1337.52±0.33	1336.46±0.40	1335.63±0.33	1334.74±0.30
$-\Delta F_2/F_2$ (%)		0.07	0.14	0.21	0.26	0.33
$-\Delta\zeta/\zeta$ (%)		0.08	0.17	0.25	0.31	0.37
$(\Delta F_2/F_2)/(\Delta\zeta/\zeta)$		0.88	0.82	0.84	0.84	0.89

$$\Delta\zeta/\zeta = 3\Delta Z_{\mathrm{ef}}/Z_{\mathrm{ef}} \tag{6}$$

This means that the value of $\Delta\zeta/\zeta$ should be about three times as much as that of $\Delta F_2/F_2$.

The SRCM relates the changes of F_2 and ζ to the symmetry-dependent admixture of 4f orbitals and ligand orbitals. The admixture results in the renormalization of 4f orbitals and the relations:

$$\Delta F_2/F_2 = 4\Delta N/N \tag{7}$$

and

$$\Delta\zeta/\zeta = 2\Delta N/N \tag{8}$$

are approximately tenable [5], where N is the renormalization constant. Eqs. (7) and (8) show that the relative reduction in ζ is nearly a half as that in F_2. Practically, the relative reduction in ζ is a little larger than that in F_2 (see data in Table 2).

Combining the CFCM and the SRCM, the relative reductions in F_2 and ζ can be expressed as:

$$\Delta F_2/F_2 = \Delta Z_{\mathrm{ef}}/Z_{\mathrm{ef}} + 4\Delta N/N \tag{9}$$

and

$$\Delta\zeta/\zeta = 3\Delta Z_{\mathrm{ef}}/Z_{\mathrm{ef}} + 2\Delta N/N \tag{10}$$

As seen from Table 2, at pressure of 10 GPa the relative reductions in F_2 and ζ amount to 0.33 and 0.37%, respectively, the relative reduction in F_2 is 0.89 times as large as that in ζ. The direct use of Eqs. (9) and (10) gives $\Delta z_{\mathrm{ef}}/z_{\mathrm{ef}} = -0.082$ and $\Delta N/N = -0.062$ for this case. This result indicates clearly that the contribution from the CFCM is about one third of that from the SRCM for $\Delta F_2/F_2$, however, the contribution from the CFCM is about two times as large as that from the SRCM for $\Delta\zeta/\zeta$.

6. Pressure effect on the crystal-field strength

6.1. Crystal-field parameters

By adjusting the CF parameters to fit the CF observed levels, the best-fit values of B_q^k at various pressures are obtained and shown in Table 3. The rms deviations between the calculated and the observed CF levels is 3–4 cm^{-1}. These parameters B_q^k reproduce the level-scheme of the Eu^{3+} ion in YOCl very well.

6.2. Crystal-field strength

In order to study the dependence of the CF strength on pressure, the CF strength parameter S_{t} at different pressures was obtained using the values of B_q^k in Table 3 according to [14]:

$$S_{\mathrm{t}} = \left\{ \frac{1}{3} \sum_k \frac{1}{2k+1} \left[\left(B_0^k\right)^2 + 2 \sum_{q>0} |B_q^k|^2 \right] \right\}^{1/2} \tag{11}$$

S_{t} is an invariant under arbitrary rotation of the crystal lattice and is also a quantitative measure of the CF strength. The values of S_{t} at different pressures between 0 and 10 GPa are also given in Table 3. The data of S_{t} in Table 3 reveal that the CF acting on the Eu^{3+} ion in $\mathrm{YOCl{:}Eu}^{3+}$ is strengthened slowly with the increase of pressure, at 10 GPa it strengthens by 2.3%. This result is completely contrary to our expectation.

7. Discussion

It is generally agreed that the interatomic distance in a crystal is reduced under pressure and thus the CF acting on an RE ion doped in the crystal strengthens, the Slater parameter F_2 and the spin–orbit coupling parameter ζ decrease, the relative reduction in ζ is less than that in F_2 [8,11]. In many cases, for example, for Eu^{3+} in the host series $\mathrm{R_2O_2S}$ (R=La, Gd, Y) [6,15,16], it is the case. For the isomorphous RE oxyhalides doped with Eu^{3+}, ROX:Eu (R=La, Gd; X=Cl,Br) the CF acting on the Eu^{3+} ion, on the contrary, are weakened with increasing pressure. Though the Slater parameter F_2 and the spin–orbit coupling parameter ζ also decrease with pressure, the relative reduction in ζ is larger than that in F_2.

The result that CF strengthens with pressure for YOCl:Eu is a surprise to us. The crystal structure and chemical composition of YOCl is similar to that of ROX. The CF strength acting on the Eu^{3+} ion in $\mathrm{YOCl{:}Eu}^{3+}$ increases with increasing pressure. The change is similar to the situation in $\mathrm{R_2O_2S{:}Eu}^{3+}$, but in $\mathrm{YOCl{:}Eu}^{3+}$ the rate of CF increase is far less than those in $\mathrm{R_2O_2S{:}Eu}^{3+}$. For $\mathrm{YOCl{:}Eu}^{3+}$ the values of F_2 and ζ decrease and the relative reduction in ζ is also larger than that in F_2 with the increase of pressure, which still is similar to the case in ROX:Eu.

Table 3

CF parameter set B_q^k (cm^{-1}), CF strength parameter S_{t} (cm^{-1}) and rms deviation σ (cm^{-1}) for $\mathrm{YOCl{:}Eu}^{3+}$ at different pressures

Pressure (GPa)	B_0^2	B_0^4	B_4^4	B_0^6	B_4^6	σ	S_{t}
0.0	−827±4	−700±3	866±4	1102±6	417±4	4	399
2.0	−797±4	−720±3	863±4	1141±6	411±4	4	399
4.0	−779±4	−722±3	867±4	1191±6	400±4	3	400
6.0	−758±4	−733±3	869±4	1241±6	388±4	3	402
8.0	−736±4	−736±3	876±4	1290±6	379±4	3	404
10.0	−717±4	−744±3	881±4	1329±6	373±4	4	408

The absolute value of the CF parameter B_0^2 which is mainly responsible for the splittings of $^7F_1$ and $^5D_1$ multiplets showed an decrease with increasing pressure. This is consistent with the decrease of the separations between levels $^7F_1(A_2)$ and $^7F_1(E)$ as well as between $^5D_1(A_2)$ and $^5D_1(E)$. The parameters B_0^4 and B_4^4, which play an important role in the splittings of the $^7F_2$ and $^5D_2$ multiplets, are insensitive to pressure.

Acknowledgements

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