Application of standard and modified Judd–Ofelt theories to thulium doped fluoroindate glass

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Abstract

Fluoroindate glasses of the following compositions: (40–x)InF₃–20ZnF₂–16BaF₂–20SrF₂–2GdF₃–2NaF–xTmF₃ with x = 1,3 mol% were prepared in a dry box under an argon atmosphere. The absorption spectra at room temperature in the spectral range 350–2200 nm were obtained. The spectra obtained for each sample had similar absorption and only the amplitude of the different bands changed as the concentration of Tm³⁺. The experimental oscillator strengths were determined from the areas under the absorption bands. Using the standard and modified Judd–Ofelt theory, intensity parameters Ω₂ (λ = 2.4,6) and (λ = 2,3,4,5,6), respectively for f-f transitions of Tm³⁺ ions as well as transition probabilities, branching ratios and radiative lifetimes for each band were determined. The results are compared with those of other glasses described in the literature.

1. Introduction

Fluoroindate glasses seem to be particularly useful hosts for rare earth ions due to their optical quality [1–3], chemical stability [4] and phonon cut-off frequency (<500 cm⁻¹) [5]. We have recently described several optical properties of Er³⁺ and Pr³⁺ ions in such glasses [1–3].

The Judd–Ofelt 4f–4f intensity model [6,7] describes the absorption and emission properties of rare earth ions in a number of compounds. However, it has been observed in many cases that this standard theory leads to a negative phenomenological Ω₂ intensity parameter for the Pr³⁺ ion [8], including fluoroindate glasses [2]. These results are in contradiction with the definition of the Ω₂ parameter. Several authors assume that higher order contributions to the forced electric dipole matrix elements might be of considerable importance to solve the problem [9,10]. These introduce additional effective operators of even and odd ranks. Recently, other authors have discussed the problem of the Pr³⁺ ion in fluoride glasses, including fluorescence measurements of branching ratios [11], and using normalized oscillator strengths [12] to calculate the parameters within the standard Judd–Ofelt theory. In recent papers [13,14] results have been published showing the effect of including higher order contributions to the forced electric dipole matrix elements in Pr³⁺ and Tm³⁺ ions in fluoroindate glasses. Such results show that: Ω₂ for Pr³⁺ is positive and, for Pr³⁺ as well as for Tm³⁺ a better fit can be obtained.

In this work we report the results of the application of the standard model [6,7] and modified
oscillator strengths [14] for the Tm$^{3+}$ ion in fluoroindate glasses to analyze the effect on the optical properties of Tm$^{3+}$ in this host.

2. Theory background

We use the standard 4f–4f intensity model described elsewhere [6,7], and a modified oscillator strength discussed in Ref. [13]. Thus, only a short summary and the most essential formulas will be given.

From the standard 4f–4f intensity model, the oscillator strength of a transition between two multiplets is given by

$$f = \frac{8\pi^2 m c \sigma}{3h(2J + 1)} \chi \sum_{i=2,3,4,5,6} \Omega_i \left( f^{N} \Psi_i \Psi_i' || U^i || f^{N} \Psi' \Psi J \right)^2,$$

(1)

where $m$ is the mass of the electron, $c$ is the velocity of light, $h$ is the Planck's constant, $\sigma$ is the mean energy (in cm$^{-1}$) for the transition, $\chi = (n^2 + 2)^2/9n$ is the Lorentz local field correction ($n$ being the refractive index of the medium) and $U^i$ is a unit operator of rank $i$.

The intensity parameters, $\Omega_i$, are determined from a least-squares fit to the measured oscillator strengths, using Eq. (1). It was demonstrated [13] the form of total oscillator strength (modified oscillator strength). It, in its even component, takes into account both contributions of forced electric dipole and of dynamic coupling [15], and the odd component takes into account the contribution due to the odd rank parameters, and is given by

$$f = \frac{8\pi^2 m c \sigma}{3h(2J + 1)} \chi \left( \sum_{i=2,3,4,6} \Omega_i \left( f^{N} \Psi_i \Psi_i' || U^i || f^{N} \Psi' \Psi J \right)^2 \right)$$

$$+ \xi^2 \sum_{i=1,3,5} \Omega_i \left( f^{N} \Psi_i \Psi_i' || U^i || f^{N} \Psi' \Psi J \right)^2,$$

(2)

where $\Psi = \alpha SL$ and $\xi = \sigma/\Delta E$. For Tm$^{3+}$ ion $\Delta E \approx 60000$ cm$^{-1}$ [16,17], which to a first approximation is equal to the energy difference between the baricenters of the ground configuration ($f^N$) and the first opposite parity excited configuration ($f^{N-1} 5d$).

The matrix reduced elements in Eq. (2) have been tabulated by Nielson and Koster (1963) [18].

The reduced matrix elements for the Tm$^{3+}$ ion with $\lambda = 1, 3$ and 5, were taken from Ref. [14] and those with $\lambda = 2, 4, 6$ were taken from Carnall (1977) [19].

The total spontaneous emission probability between the $J$ and $J'$ levels is given by

$$A_{J'J} = \frac{64\pi^4 \sigma^3}{3h(2J + 1)} \chi S_{cd},$$

(3)

where $\chi = n(n^2 + 2)^2/9$ is the effective field corrections at a well-localized center in a medium of isotropic refractive index $n$, $S_{cd}$ is the electric dipole line strengths defined by

$$S_{cd} = e^2 \sum_{i=2,3,4,5,6} \Omega_i \left( f^{N} (\alpha SL) \Psi_i || U^i || f^{N} (\alpha SL) \Psi J \right)^2.$$

(4)

$A_{J'J}$ is related to the radiative lifetime $\tau_R$ of an excited state by

$$\tau_R = \frac{1}{\sum_{J'} A_{J'J}}.$$

(5)

The branching ratio, $\beta_{J'J}$, corresponding to the emission from an excited $J$ level to $J'$ is

$$\beta_{J'J} = \frac{A_{J'J}}{\sum_{J'} A_{J'J}}.$$

(6)

The multiphonon relaxation rate between two $J$ levels in glasses may be adequately described by a single phonon frequency model [16] by the formula

$$W_{nr} = B e^{-\Delta E/(n(\omega, T) + 1)},$$

(7)

where $B$ and $\alpha$ are constants characteristic of the host material, $\Delta E$ is the energy gap between the emitting and the closest lower electronic level, $\omega$ is the stretching frequency of the glass-forming groups, and $p = \Delta E/\hbar \omega$ is the number of phonons which must be emitted in order to conserve energy during the transition. The explicit temperature dependence of $W_{nr}$ through the Bose–Einstein occupation number, $n = 1/\exp(\omega/kT) - 1$, provides a way to determine the number and energy of the phonons involved in the decay processes. These expressions are based on the assumption that phonons of a single energy, $\hbar \omega$, are active in the transition.
3. Experimental procedure

The fluoroindate glasses with batch compositions (mol%) \((40-x)\text{InF}_3-20\text{SrF}_2-20\text{ZnF}_2-16\text{BaF}_2-2\text{NaF}-2\text{GdF}_3-x\text{TmF}_3\) \((x=1\) and 3 mol\%) were prepared in a dry box under Ar atmosphere by melting ultrapure powders (Merck, Fluorotran) in a platinum crucible at 800°C for 1 h and then at 850°C for fining. After the fining process the liquid was poured into a brass mold at 260°C and then cooled to 20°C. The obtained samples were transparent and were non-hygrosopic. The samples were cut and polished into the shape of parallelepipeds. The refractive indices were measured using a Abbé Refractometer and a value of 1.48 ± 0.05 was obtained for two samples \((x=2, 3\) mol\%). The density was measured by Archimedean’s principle using xylene as an immersion liquid and a density of 4.84 g/cm³ was obtained for the sample of 3.0 mol%.

The absorption spectra were recorded in the visible spectral range using a 0.75 m monochromator (Spex) with an AsGa photomultiplier cooled to −30°C, and in the infrared spectral range using a PbS detector at room temperature (Fig. 1). The absorption path length of the sample was 1.30 mm for the sample with 3.0 mol% concentration.

The oscillator strength is obtained from the area under the absorption band with the wavelength \((\lambda)\) corresponding to the band baricenter, using

\[
f = \frac{4.318 \times 10^{-9}}{C\lambda^2} \int k(\lambda) \, d\lambda,
\]

where \(k(\lambda)\) is the spectral absorption coefficient, \(\lambda\) in nm, and \(C\) and \(l\) are the concentration of Tm³⁺ ions in mol/1000 cm³ and the absorption path length, respectively.

4. Results

4.1. Absorption spectra

Fig. 1 shows the absorption spectrum of Tm³⁺ in fluorindate glass at room temperature in the spectral range from 350 to 850 nm. The spectrum obtained for each sample have similar bands. There was little difference in the amplitudes of the different bands as the concentration of Tm³⁺ changed. The energy level diagram obtained from the absorption spectra of Tm³⁺ in fluorindate glass at room temperature is shown in Fig. 2(a).

4.2. Oscillator strengths and spectroscopical parameters

Table 1 gives the oscillator strengths calculated with the standard model Eq. (1), \(\lambda=2,4,6\), and with the modified theory Eq. (3), \(\lambda=2,3,4,5\) and 6. Also, it is included the deviation \(d(d_\text{cal} - d_\text{expt})\).
Table 1
Experimental oscillator strengths in units of $10^{-6}$, $f_{\text{Exp}}$, obtained from the absorption spectra at room temperature, and oscillator strengths, $f_{\text{Cal}}$, calculated from the standard model (Eq. (1)), $\lambda = 2,4,6$, and from (Eq. (3)) modified oscillator strength, $\lambda = 2,3,4,5,6$ for Tm$^{3+}$ ion in fluorindate glass $x = 3$ mol%, deviation $d$ and RMS (in units of $10^{-4}$)

<table>
<thead>
<tr>
<th>$S^L$</th>
<th>Energy (cm$^{-1}$)</th>
<th>$f_{\text{Exp}}$</th>
<th>$f_{\text{Cal}}, \lambda = 2,4,6$</th>
<th>$d$</th>
<th>$f_{\text{Cal}}, \lambda = 2,3,4,5,6$</th>
<th>$\Delta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1F$_3$</td>
<td>5945</td>
<td>1.65</td>
<td>2.00</td>
<td>0.35</td>
<td>1.69</td>
<td>0.04</td>
</tr>
<tr>
<td>1H$_3$</td>
<td>8333</td>
<td>1.44</td>
<td>1.67</td>
<td>0.23</td>
<td>1.40</td>
<td>-0.04</td>
</tr>
<tr>
<td>1H$_4$</td>
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<td>2.61</td>
<td>2.36</td>
<td>-0.25</td>
<td>2.55</td>
<td>-0.06</td>
</tr>
<tr>
<td>1F$_3$</td>
<td>14 777</td>
<td>3.59</td>
<td>3.52</td>
<td>-0.07</td>
<td>3.55</td>
<td>-0.04</td>
</tr>
<tr>
<td>1F$_2$</td>
<td>15 198</td>
<td>0.41</td>
<td>0.72</td>
<td>0.31</td>
<td>0.64</td>
<td>0.23</td>
</tr>
<tr>
<td>1G$_4$</td>
<td>21 367</td>
<td>1.23</td>
<td>0.66</td>
<td>-0.57</td>
<td>1.28</td>
<td>0.05</td>
</tr>
<tr>
<td>1D$_2$</td>
<td>27 933</td>
<td>2.78</td>
<td>2.73</td>
<td>-0.05</td>
<td>2.78</td>
<td>0.00</td>
</tr>
<tr>
<td>RMS</td>
<td></td>
<td></td>
<td></td>
<td>0.16</td>
<td></td>
<td>0.03</td>
</tr>
</tbody>
</table>

and the root-mean-square (RMS) deviation of the observed ($f_{\text{Exp}}$) and calculated ($f_{\text{Cal}}$) oscillator strength [14]. The excited J levels are given in the column 1, and in the other columns are given $f_{\text{Exp}}$, $f_{\text{Cal}}$ and $\Delta$. The average wavelengths were taken to be the baricenters of the absorption bands.

Table 2
Values of the gap energy $\Delta E$, transition probabilities $A_{\alpha\beta}$ and branching ratios $B_{\alpha\beta}$, between multiplets $I$ and $J$, and radiative life time $\tau_R$, and emitting level multiphonon rate $W$ of Tm$^{3+}$ in fluorindate glass ($x = 3$ mol%), calculated from the standard theory ($\lambda = 2,4,6$) and modified theory using the better set of phenomenological parameters ($\lambda = 2,3,4,5,6$), obtained in Ref. [12].

<table>
<thead>
<tr>
<th>Transitions</th>
<th>$\Delta E$ (cm$^{-1}$)</th>
<th>$\lambda = 2,4,6$</th>
<th>$\lambda = 2,3,4,5,6$</th>
<th>Emitting level multiphonon rate $W$ (s$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^3F_2 \rightarrow ^3H_4$</td>
<td>5945</td>
<td>150.89</td>
<td>1.0000</td>
<td>6.63</td>
</tr>
<tr>
<td>$^1H_3 \rightarrow ^3F_3$</td>
<td>8333</td>
<td>201.84</td>
<td>0.9770</td>
<td>1.02</td>
</tr>
<tr>
<td>$^3H_4 \rightarrow ^3F_3$</td>
<td>4438</td>
<td>34.46</td>
<td>0.0351</td>
<td>0.36</td>
</tr>
<tr>
<td>$^1H_4 \rightarrow ^3H_4$</td>
<td>6826</td>
<td>125.39</td>
<td>0.1278</td>
<td>3.10</td>
</tr>
<tr>
<td>$^1G_4 \rightarrow ^3F_3$</td>
<td>12 771</td>
<td>281.16</td>
<td>0.8370</td>
<td>1.50</td>
</tr>
<tr>
<td>$^3G_3 \rightarrow ^3G_3$</td>
<td>6179</td>
<td>15 019</td>
<td>0.7698</td>
<td>1.17</td>
</tr>
</tbody>
</table>
The phenomenological intensity parameters, $\Omega_i$, (in unit of $10^{-20}$ cm$^2$), used in this work [14] are $\Omega_2 = 1.77$, $\Omega_4 = 2.30$, $\Omega_6 = 1.69$, $\Omega_2 = 1.15$, $\Omega_3 = 25.24$, $\Omega_4 = 1.79$, $\Omega_5 = 2.49$, $\Omega_6 = 1.48$.

Table 2 gives the energies of the possible $J \leftrightarrow J'$ transitions involving the seven $J$ levels obtained from the absorption spectrum of the sample with a concentration of 3 mol%. The quantities $A_{l'}, \beta_{l',l}$, and $\tau_R$ and $W$, were calculated on the basis of Eqs. (3)-(7), respectively, and the results are included in Table 2.

4.3. Thermalization

In Tm$^{3+}$-doped fluoroindate glass the $^3F_3$ and $^3F_2$ level separation, $\Delta E$, as obtained from the absorption spectra (Fig. 1), is 421 cm$^{-1}$. Thus, at room temperature thermalization of the two levels occurs and the effective radiation transition probability of the two levels has to be calculated using the formula

$$A_{\text{eff}} = \frac{12 \exp(-\Delta E/kT) \sum A(^3F_2) + 4 \sum A(^3F_3)}{12 \exp(-\Delta E/kT) + 4},$$

(9)

where $kT = 209$ cm$^{-1}$ (at room temperature), for $\Omega_{2,4,6,2} \sum A(^3F_2) = 851.91$ s$^{-1}$, $\sum A(^3F_3) = 2741$ s$^{-1}$, the $A_{\text{eff}} = 2199$ s$^{-1}$ and for $\Omega_{2,4,5,6,2} \sum A(^3F_2) = 754.45$ s$^{-1}$, $\sum A(^3F_3) = 2761.17$ s$^{-1}$ the $A_{\text{eff}} = 2184.98$ s$^{-1}$.

From Eq. (8), assuming that the stretching frequencies for the fluoroindate glasses are similar to that of fluoride glasses, $h\nu = 500$ cm$^{-1}$ and $B = 1.59 \times 10^{10}$ s$^{-1}$ and $\alpha = 5.19 \times 10^{-3}$ cm$^{-1}$ [5], the multiphonon relaxation rate, $W_{\text{MP}}$, from the $^3F_2$ level calculated in this work was $1.9 \times 10^{-9}$ s$^{-1}$.

From these results, owing to the multiphonon relaxation rate from the $^3F_2$ level, one eventual laser transition can be expected mainly from the $^3F_3$ level. The parameters, $\alpha$ and $B$, are dependent on the host material but approximately independent of the specific $J$ level of a given lanthanide. To illustrate this situation, Fig. 2 shows the energy levels obtained from the absorption spectra of Tm$^{3+}$ in fluoroindate glass.

5. Discussion

From Fig. 1 the intensity of the hypersensitive transitions $^3H_6 \rightarrow ^3F_4$, $^3H_6 \rightarrow ^3H_4$ and $^3H_6 \rightarrow ^1D_2$ of the Tm$^{3+}$ ion [8] are approximately equal to the intensities of the other transitions ($^3H_6 \rightarrow ^3H_5$, $^3H_6 \rightarrow ^3F_3$, $^3H_6 \rightarrow ^1D_2$). This similarity is opposite to the absorption observed for this type of transition for the Er$^{3+}$ and Pr$^{3+}$ ions in glasses of the same composition.

From Table 1 the oscillator strengths for Tm$^{3+}$ in fluoroindate glasses are similar to those in other fluoride glasses [20], but are smaller than in borate, phosphate, germanite and tellurite glasses [21,22]. This fact is due to the lower phonon frequency in the fluoroindate glass. Consequently, for concentrations <5.0 mol% rare earth ions, these results confirm the suitability of fluoride glasses, with respect to multiphonon losses, as hosts for laser operation in the infrared.

From Table 2, the transitions that have greater contribution from the odd rank parameters to the oscillator strengths $^3H_6 \rightarrow ^3F_4$, $^3H_6 \rightarrow ^3H_5$, $^3H_6 \rightarrow ^3H_4$ and $^3H_6 \rightarrow ^1G_4$, show the greater differences between the transition probabilities, $A_{l'}$, calculated with the 4f–4f standard and the modified models, the greatest differences being in the transitions from the $^1G_4$ level. The magnetic-dipole contribution to the transition $^3H_6 \rightarrow ^3H_5$ is not taken into account. These results show that there are various potential laser transitions that may be predicted based on the transition probabilities, branching ratio, radiative lifetimes, and multiphonon relaxation rates for Tm$^{3+}$ in fluoroindate glasses. These transitions are included in Table 3. Diagrams 2(b) and (c) in Fig. 2 show possible processes to obtain a four-level laser system and a three-level laser system, respectively.

Also, there are transitions at 1200, 1500 and 1800 nm that may be of interest as sources for optical waveguides.

From Fig. 2, due to the spread of the energy level structure of Tm$^{3+}$, and the multiphonon transition losses, virtually all the transitions,
Table 3
Selected possible laser transitions of Tm\(^{3+}\) in fluoroindate glasses

<table>
<thead>
<tr>
<th>Transitions</th>
<th>(\Delta E (\text{cm}^{-1}))</th>
<th>(\lambda = 2,4,6)</th>
<th>(\lambda = 2,3,4,5,6)</th>
<th>Emitting level multi-phonon rate (W (\text{s}^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>( ^{3}\text{F}_4 \rightarrow ^{3}\text{H}_6 )</td>
<td>5945</td>
<td>150.89</td>
<td>0.0000</td>
<td>127.53</td>
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<tr>
<td>( ^{3}\text{H}_5 \rightarrow ^{3}\text{H}_6 )</td>
<td>8333</td>
<td>201.84</td>
<td>0.9770</td>
<td>169.91</td>
</tr>
<tr>
<td>( ^{3}\text{H}_4 \rightarrow ^{3}\text{F}_4 )</td>
<td>6826</td>
<td>125.39</td>
<td>0.1278</td>
<td>135.09</td>
</tr>
<tr>
<td>( ^{3}\text{H}_4 \rightarrow ^{3}\text{H}_6 )</td>
<td>12 771</td>
<td>821.16</td>
<td>0.8370</td>
<td>884.71</td>
</tr>
<tr>
<td>( ^{3}\text{F}_3 \rightarrow ^{3}\text{H}_4 )</td>
<td>6444</td>
<td>175.04</td>
<td>0.0638</td>
<td>176.28</td>
</tr>
<tr>
<td>( ^{3}\text{F}_3 \rightarrow ^{3}\text{F}_6 )</td>
<td>8832</td>
<td>450.65</td>
<td>0.1644</td>
<td>453.86</td>
</tr>
<tr>
<td>( ^{3}\text{F}_3 \rightarrow ^{3}\text{H}_6 )</td>
<td>14 777</td>
<td>2110.69</td>
<td>0.7698</td>
<td>2125.71</td>
</tr>
<tr>
<td>( ^{3}\text{F}_2 \rightarrow ^{3}\text{F}_4 )</td>
<td>9253</td>
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<td>0.1707</td>
<td>128.80</td>
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<tr>
<td>( ^{3}\text{F}_2 \rightarrow ^{3}\text{H}_6 )</td>
<td>15 198</td>
<td>644.45</td>
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<td>570.72</td>
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<tr>
<td>( ^{1}\text{G}_4 \rightarrow ^{1}\text{F}_4 )</td>
<td>13 034</td>
<td>146.01</td>
<td>0.1318</td>
<td>284.64</td>
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<tr>
<td>( ^{1}\text{F}_4 \rightarrow ^{3}\text{F}_4 )</td>
<td>15 429</td>
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<tr>
<td>( ^{1}\text{D}_2 \rightarrow ^{3}\text{F}_4 )</td>
<td>21 367</td>
<td>643.27</td>
<td>0.5807</td>
<td>1253.99</td>
</tr>
<tr>
<td>( ^{1}\text{F}_3 \rightarrow ^{3}\text{F}_4 )</td>
<td>12 735</td>
<td>776.11</td>
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<tr>
<td>( ^{1}\text{F}_3 \rightarrow ^{3}\text{H}_4 )</td>
<td>13 156</td>
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<tr>
<td>( ^{1}\text{H}_4 \rightarrow ^{3}\text{H}_4 )</td>
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<td>1330.25</td>
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<td>( ^{1}\text{H}_4 \rightarrow ^{3}\text{H}_6 )</td>
<td>19 600</td>
<td>2829.39</td>
<td>0.1566</td>
<td>2873.34</td>
</tr>
<tr>
<td>( ^{1}\text{F}_3 \rightarrow ^{3}\text{F}_6 )</td>
<td>21 988</td>
<td>3994.68</td>
<td>0.2211</td>
<td>4056.73</td>
</tr>
<tr>
<td>( ^{1}\text{H}_6 \rightarrow ^{3}\text{H}_6 )</td>
<td>27 933</td>
<td>8189.88</td>
<td>0.4534</td>
<td>8317.09</td>
</tr>
</tbody>
</table>

except the \( ^{3}\text{F}_2 \rightarrow ^{3}\text{F}_3 \) decay, are radiative, and any as, \( ^{3}\text{F}_4 \rightarrow ^{3}\text{H}_6, ^{3}\text{H}_6 \) and \( ^{3}\text{F}_4 \rightarrow ^{3}\text{H}_4 \) have been used in fluorozirconate fiber laser [23]. Transitions from the \( ^{3}\text{H}_4 \) level at 800, 1500 and 2300 nm, and \( ^{3}\text{F}_4 \) level at 1800 nm are particularly interesting for practical applications; since they can be brought about by excitation of the \( ^{3}\text{H}_4 \) level by a semiconductor laser diode at 790 nm.

6. Conclusions

In this work we present the calculated spectroscopic properties of various Tm\(^{3+}\) transitions in multicomponent fluoroindate glass using the standard 4f–4f intensity model and with a better fit obtained with modified Judd–Ofelt theory [11]. The more important effects of odd rank parameters on the spectroscopic parameters appear in the transitions in which there are a greater contribution to the oscillator strengths. Based on spectroscopic parameters, \( A_{4f}, B_{4f}, r_{2} \) and \( W \) of Tm\(^{3+}\), various transitions suitable for laser action can be suggested. Also in this paper with Tm\(^{3+}\) as with Er\(^{3+}\) and Pr\(^{3+}\) [1–3], the \( \Omega_{2} \) intensity parameters may be associated with the micro-structural homogeneity (high local symmetry) around the Tm\(^{3+}\) ions.

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