Photoluminescence and spectral parameters of Eu\(^{3+}\) in sodium–aluminum–tellurite ceramics

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**Abstract**

Eu\(^{3+}\) ion doped sodium–aluminum–tellurite (Eu\(^{3+}\):NAT) opaque ceramics have been synthesized by solid-state reaction. The amorphous structure of Eu\(^{3+}\):NAT was confirmed by X-ray diffraction pattern. Under the excitation of violet light, 11 obvious emission bands including infrequent blue and green peaks have been recorded and they almost cover whole visible spectral region. By using Eu\(^{3+}\) as a probe, the local structure of rare earth ion in NAT ceramics has been investigated. Based on emission spectrum, the Judd–Ofelt intensity parameters \(\Omega_2\) and \(\Omega_4\) have been calculated to be \(2.95 \times 10^{-20}\) and \(0.39 \times 10^{-20}\) cm\(^2\), respectively. Low phonon energy and bright visible fluorescence indicate that rare earth doped NAT ceramics are promising candidates for illumination, display and detection devices.

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1. Introduction

Rare earth (RE) doped solid-state materials have tremendous applications for sensor, color display, white lighting and optical communication [1–8]. Currently, a great deal of research has been focused on RE doped glasses owing to their extensive applications in laser and optical communication devices [9,10]. Conversely, little investigation was carried out on the luminescence properties of RE doped opaque ceramics, because the opacity limited their applications in optical transmission devices. However, such opaque characteristic can improve the absorption efficiency, which increases the luminescent efficiency of the RE ions. Based on this point, opaque ceramics are more competitive than glasses in the applications of RE doped opaque ceramics, because the opacity limited their applications in optical transmission devices. However, such opaque characteristic can improve the absorption efficiency, which increases the luminescent efficiency of the RE ions. Based on this point, opaque ceramics are more competitive than glasses in the development of white lighting, sensor and planar medical display devices. For instance, Tanabe and co-workers have reported a Ce:YAG (Y\(_2\)Al\(_5\)O\(_{12}\)) opaque glass-ceramic phosphor for white LED, and predicted that it is a promising candidate material for realization of resin-free and long-life white LED devices [11]. Hence, further attempts at developing RE doped opaque ceramics have important significance in the development of luminescent materials.

Aluminum–tellurite opaque ceramics are derived from tellurite system and possess lower phonon energy, high optical nonlinearity and large solubility of RE ions [12,13]. In addition, Al\(_2\)O\(_3\) introducing will enhance the dispersion of RE ions, helping to limit the concentration quenching effect on photoluminescence [14]. Therefore, they have seductive potential as luminescence hosts for optoelectronic devices. On the other hand, the optical characteristics of RE ions are mainly due to electric-dipole transitions that can be affected by the RE ion surroundings [15]. Thus, it is important to understand the local environments of RE ions in NAT opaque ceramics. Owing to the relative simplicity of energy level structure [16], Eu\(^{3+}\) can reflect the local structure around the RE ions in various hosts. Therefore, it can be used as a probe to investigate the local environments of RE ions in NAT opaque ceramics.

In this work, Eu\(^{3+}\) doped sodium–aluminum–tellurite opaque ceramics with low phonon energies were prepared. X-ray diffraction (XRD) pattern and fluorescence spectra have been used to characterize the structure and luminescence properties of Eu\(^{3+}\):NAT opaque ceramics, respectively. Judd–Ofelt parameters \(\Omega_2\) and \(\Omega_4\) have been derived from the emission spectrum and radiative properties were predicted by the Judd–Ofelt theory. RE doped NAT ceramics with various visible emissions will bring a promising foreground in developing illumination, display and detection devices.

2. Experimental

Eu\(^{3+}\) doped NAT ceramics were prepared from high-purity sodium carbonate (Na\(_2\)CO\(_3\)), aluminum oxide (Al\(_2\)O\(_3\)), tellurium oxide (TeO\(_2\)) and europium oxide (EuO\(_x\)) powders. The molar host compositions are 15% Na\(_2\)O, 15% Al\(_2\)O\(_3\) and 70% TeO\(_2\). Based on the host weight, additional 1 wt% EuO\(_x\) was added to dope the host. The pulverized and fully mixed raw materials were preheated at 200 °C for 2 h and melted in a platinum crucible at 950 °C for 20 min using an electric furnace, and then quenched to room temperature in aluminum mold. The ceramic sample was subsequently annealed at 300 °C for 1 h, after that slowly cooled down to room temperature. The volatility losses of TeO\(_2\) were estimated by accurate weighing of raw materials, ceramics and platinum crucible before and after melting, and the actual
The molar host composition of the NAT ceramic sample after melting is evaluated to be 15.006% Na2O, 15.006% Al2O3, and 69.988% TeO2 [17].

For experimental measurements, the annealed ceramic sample was sliced and polished to be pieces with two parallel sides. Excitation and emission spectra of the sample were recorded at room temperature using a Perkin-Elmer LS 55 luminescence spectrometer with an 8.3-W xenon discharge lamp source and a R928 photomultiplier sample detector. Powder XRD pattern was obtained on Rigaku D/max-3B diffractometer with a copper tube.

3. Results and discussion

3.1. XRD pattern and luminescence of Eu3+:NAT opaque ceramics

Eu3+:NAT opaque ceramic sample appears opalescent, opaque and glazed under sunlight and emits intense red lights under the excitation of 365 nm ultraviolet light-emitting diode (UV-LED), as illustrated in Fig. 1(a) and (b), respectively. Fig. 2 shows the XRD pattern of Eu3+:NAT ceramic powders. The powders exhibit typical broad diffraction maxima of an amorphous structure and no obvious crystalline peaks can be found within the XRD resolution limits.

Emission spectrum of 1 wt% Eu2O3 doped NAT ceramics under 382 nm excitation is given in Fig. 3. Eleven emission bands locating at 418.5, 427.5, 445.0, 465.0, 487.0, 511.5, 535.5, 554.5, 586.0, 614.0 and 702.0 nm almost cover the whole visible spectrum region and they are attributed to the 5D3 → 7F1, 5D3 → 7F2, 5D3 → 7F3, 5D3 → 7F4, 5D2 → 7F2, 5D2 → 7F3, 5D1 → 7F1, 5D1 → 7F2, 5D1 → 7F3, 5D0 → 7F2 and 5D0 → 7F4 transitions, respectively. The blue and green emissions are rarely observed in other Eu3+ doped oxide glasses and ceramics, but here, these two bands are clearly recorded. Compared with other Eu3+ doped solid materials, the multichannel emission behavior of Eu3+:NAT ceramics is similar to the case in 5Li2O–5K2O–5BaO–10Bi2O3–75TeO2 glass system, which has low phonon energy in oxide glass systems [18].

The excitation spectrum for 614 nm red emission of 1 wt% Eu2O3 in NAT ceramics is shown in Fig. 4. There are six obvious excitation peaks parking at 362.0, 382.0, 394.5, 415.5, 465.5 and 534.0 nm, respectively, which covers 350–550 nm spectral range well, indicating that UV, violet, blue and green laser diodes (LDs)
and light-emitting diodes (LEDs) are efficient pumping sources in obtaining Eu\textsuperscript{3+} emissions.

The energy level diagram is depicted in Fig. 5. The energy level diagram shows the energy gaps among the levels of 5D\textsubscript{3}, 5D\textsubscript{2}, 5D\textsubscript{1} and 5D\textsubscript{0}. The emission efficiencies of 5D\textsubscript{3}, 5D\textsubscript{2} and 5D\textsubscript{1} levels are related with the non-radiative rates (W\textsubscript{NR}) tightly. W\textsubscript{NR} can be expressed by

$$W_{NR} = W_{F} + W_{ET},$$

(1)

where $W_{F}$ is the multiphonon decay rate and $W_{ET}$ is the relation rate by energy transfer. For the low concentration of Eu\textsuperscript{3+} doping NAT ceramics, the contribution of $W_{ET}$ can be ignored temporarily, and the factor dominating the quantum efficiency is $W_{F}$, which is associated with the phonon energy. $W_{F}$ is expressed by [8]:

$$W_{F} = W_{0} \exp \left( \frac{-\Delta E}{\hbar \omega} \right).$$

(2)

where $W_{0}$ is the transition probability extrapolated to zero energy gap, which is independent of the electronic nature of the RE ion. $\alpha = \ln[\Delta E/(\hbar \omega_0)] - 1$, where $g$ is the electron phonon coupling strength [8]. $\Delta E$ is the energy gap to the next lower level and $\hbar \omega_0$ is the phonon energy of the host matrix. According to Eq. (2), $W_{NR}$ should be large in silicate, phosphate and borate hosts with higher phonon energies ($\hbar \omega_0 = \sim 1100, ~ 1300$ and $\sim 1400 \text{ cm}^{-1}$, respectively) and it is the main reason why the emissions of Eu\textsuperscript{3+} from 5D\textsubscript{3}, 5D\textsubscript{2} and 5D\textsubscript{1} levels are difficult to observe in silicate, phosphate and borate hosts but can be recorded in NAT ceramics clearly. The maximum stretching vibration of Te–O and Al–O vibration are proved to be $\sim 738$ and $\sim 760 \text{ cm}^{-1}$ [19,20], respectively. Thus, the maximum phonon energy in the NAT ceramics is estimated to be $\sim 760 \text{ cm}^{-1}$. The low maximum phonon energy provides the opportunity for obtaining effective multichannel emissions in RE doped NAT ceramics.

In Eu\textsuperscript{3+}:NAT ceramic system, the multiphonon relaxation processes from 5D\textsubscript{3} to 5D\textsubscript{2} levels are accomplished by four phonon bridging and those from 5D\textsubscript{2} to 5D\textsubscript{1} levels are completed by three phonon bridging. All of them belong to high order processes ($\geq 3$ phonons) and the probability is much lower than those of low order processes such as in the cases of Eu\textsuperscript{3+} doped traditional oxide materials [18]. When Eu\textsuperscript{3+}:NAT ceramics are pumped by violet light, the relaxations from 5G\textsubscript{2} to 5L\textsubscript{6} and then from 5L\textsubscript{6} to 5D\textsubscript{0} are very fast because the energy gaps between them are only $\sim 800$ and $\sim 1000 \text{ cm}^{-1}$, respectively. The rapid relaxation to 5D\textsubscript{2} and three slower relaxations from 5D\textsubscript{3}, 5D\textsubscript{2} and 5D\textsubscript{1} levels maintain the considerable population of 5D\textsubscript{0}, 5D\textsubscript{2} and 5D\textsubscript{1} levels, which is beneficial to obtaining effective blue and green emissions of Eu\textsuperscript{3+}.

### 3.2. Eu\textsuperscript{3+} local environment and Judd–Ofelt analysis

The emission spectrum of Eu\textsuperscript{3+}:NAT ceramics under 465 nm excitation is given in Fig. 6 and it is separated into three main emission peaks (right inset in Fig. 6) along the abscissa of wavenumber by Gaussian multi-peaks fitting. Ignoring the potential 5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{3} transition, the integrating emission intensities and the intensity ratios were derived and listed in Table 1.

Proverbially, the intensity of the 5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{3} transition is extremely sensitive to chemical bonds in the vicinity of Eu\textsuperscript{3+}, which increases with the decreasing of the site symmetry of Eu\textsuperscript{3+} center. On the other hand, the intensity of the 5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{1} transition is independent of the surroundings of Eu\textsuperscript{3+}. Therefore, the 5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{2} transition intensity ratio which is called as the asymmetry ratio ($R$) is widely used as a criterion of the coordination state and the site symmetry for the RE ions [21]. In Eu\textsuperscript{3+}:NAT ceramics, the asymmetry ratio $R = 1.893$ is lower than those of phosphate and alakalitellurite glasses [22,23], suggesting that the symmetry of the site occupied by Eu\textsuperscript{3+} in NAT ceramics is higher than those of phosphate and alakalitellurite glasses.

Judd–Ofelt intensity parameters are essential indicators in judging radiative potential of RE ions in different hosts, which are usually derived from absorption spectrum. However, owing to the special energy level structure of Eu\textsuperscript{3+}, they can be calculated from the emission spectrum. The transitions of Eu\textsuperscript{3+} from 5D\textsubscript{0} to 7F\textsubscript{J} ($J = 2, 4, 6$) are electronic-dipole allowed and the spontaneous emission probability $A_{ed}$ from initial manifold $\psi_{i}$ to terminal manifold $\psi_{f}$ is given using the following expression [24,25]:

$$A_{ed} = \frac{64\pi^2 e^4 \lambda^3}{3h(2\lambda + 1)} \frac{n(n^2 + 2)^2}{9} \times \sum_{t=2,4,6} \Omega_t (\psi_{f} | U(t) | \psi_{i})^2,$$

(3)

where $h$ is the Planck constant, $e$ is the electron charge, $n$ is the refractive index, and $\lambda$ is the wavenumber of transition. $\Omega_t$ are the Judd–Ofelt parameters. The term $\langle \psi_f | U(t) | \psi_i \rangle^2$ is the square of the matrix elements of the tensorial operator, which connects $| \psi_f \rangle$ to $| \psi_i \rangle$.

<table>
<thead>
<tr>
<th>Item</th>
<th>Wavenumber (cm\textsuperscript{-1})</th>
<th>Integrating intensity (a.u.)</th>
<th>Intensity ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{3} ($l_1$)</td>
<td>17064</td>
<td>8.745</td>
<td>–</td>
</tr>
<tr>
<td>5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{2} ($l_2$)</td>
<td>16258</td>
<td>16.555</td>
<td>$I_2/I_1 = 1.893$</td>
</tr>
<tr>
<td>5D\textsubscript{0} $\rightarrow$ 7F\textsubscript{4} ($l_4$)</td>
<td>14255</td>
<td>1.070</td>
<td>$I_4/I_1 = 0.122$</td>
</tr>
</tbody>
</table>
the final state \(|\psi_f\rangle\) and is considered to be independent of host matrix.

The \(5D_0 \rightarrow 7F_1\) of Eu³⁺ ions is a magnetic-dipole transition and the spontaneous emission probability of magnetic-dipole transition \(A_{md}\) is

\[
A_{md} = \frac{64\pi^4n^3}{3h(2J+1)}n^3S_{md},
\]

where \(h\) is the Planck constant, \(v\) is the wavenumber of transition, \(J\) is the total angular momentum of the excited state, \(n\) is the refractive index, \(S_{md}\) is the magnetic-dipole line strength, which is a constant and independent of the medium.

The value of \(A_{md}\) can be estimated using the value of LBLB (Li₂O–BaO–La₂O₃–B₂O₃) glasses \([26]\). The relationship is

\[
A_{md} = \left(\frac{n}{n'}\right)^3 A'_{md},
\]

where \(n = 1.854\) and \(n' = 1.64\) are the refractive indices of NAT ceramics and Li₂O–BaO–La₂O₃–B₂O₃ glasses, respectively, \(A'_{md}\) is the magnetic-dipole transition probability of Eu³⁺ doped Li₂O–BaO–La₂O₃–B₂O₃ glasses \([26]\).

Due to selection rules and the unique nature of transition intensities of Eu³⁺ ions, each of the \(||\psi_f||U(|\psi_i|)|\psi_f\rangle^2\) values decided the intensities of the transitions since the remaining two are zero. Thus, the \(\Omega_t\) can be determined from the ratio of intensities of \(5D_0 \rightarrow 7F_2, 4, 6\) transitions to the intensity of \(5D_0 \rightarrow 7F_1\) transitions as follows:

\[
\frac{\int I_f(v)dv}{\int I_{md}(v)dv} = \frac{A_f}{A_{md}} = \frac{64\pi^4n^3}{3h(2J+1)}\frac{n(n^2+2)^2}{8A_{md}}\Omega_t||U(|\psi_i|)|\psi_f\rangle^2,
\]

(6)

The derived \(\Omega_2\) and \(\Omega_4\) parameters are listed in Table 2. The \(\Omega_6\) intensity parameter was not determined because the \(5D_0 \rightarrow 7F_2\) emission in this system cannot be detected and recorded. Judd–Ofelt intensity parameters reflect local structure and bonding in the vicinity of RE ions. For example, \(\Omega_2\) exhibits the dependence on the covalence between RE ions and ligand anions and gives information about the asymmetry of the local environment of Eu³⁺ site \([27]\). Table 2 compares the intensity parameters of Eu³⁺ doped NAT ceramics as well as other crystals, glasses and glass–ceramics. Here, \(\Omega_2\) in NAT ceramics is larger than those of fluoride glasses \([28]\), LaF₃ crystals \([29]\), together with fluorosilicate and oxyfluoroborate glass ceramics \([30,31]\). It is close to the values of oxyfluoroborate and fluorophosphate glasses \([31,32]\), while it is smaller than the rest \([22,23,33,34]\). This indicates a lower asymmetry and covalence environment in NAT ceramics. Moreover, it should be mentioned here that \(\Omega_2\) in NAT ceramics is much smaller than that of alkali-lutetite glasses. This indicates that the introducing of Al₂O₃ can enhance the symmetry of tellurite ceramic system.

In Eu³⁺:NAT ceramics, Al resides two different environments corresponding to tetrahedrally and octahedrally coordinated sites. The tetrahedral coordination of Al in NAT ceramics necessitates the formation of a coordinate type of bond between Al and one of its neighboring Te atoms, with the Al atom taking an electron from the Te. The Te donates one electron and becomes a Te⁺⁺ center (the subscript and superscript indicate the coordination and charge state, respectively). The other three Te atoms are bonded covalently with same Al atom. Similarly, for the octahedral coordination of Al, three such Te⁻⁻ centers are created, with the remaining three Te atoms covalently bonded. Thus, the tetrahedral and octahedral coordination of Al in NAT ceramics result in an increase in the concentration of Te⁺⁺ centers, while the number of Te⁻⁻ defects remains unchanged \([35]\). Hence, the environments around Eu³⁺ in NAT ceramics take on higher symmetry and lower covalence, which is in conformity with the lower \(\Omega_2\) and \(R\)-values.

### 3.3. Radiative properties of Eu³⁺:NAT opaque ceramics

Judd–Ofelt parameters obtained from Eq. (6) have been used to predict some important radiative properties such as transition probabilities, branching ratios and lifetimes for the excited states of Eu³⁺ ions. The radiative transition probability \(A\) for a transition \(|\psi_f|\rightarrow|\psi_i\rangle\) can be calculated from the following equation:

\[
A(|\psi_f|, |\psi_i\rangle) = A_{ed} + A_{md},
\]

(7)

where \(A_{ed}\) and \(A_{md}\) are the electric and magnetic-dipole radiative transition probabilities, which have been given by Eqs. (3) and (5), respectively.

The predicted radiative lifetime \(\tau_{rad}\) of an excited state in terms of \(A_{t}\), the total radiative transition probability of an excited state, is given by

\[
\tau_{rad}(|\psi_i\rangle) = \frac{1}{A_{t}(|\psi_i\rangle)}.
\]

(8)

The branching ratio \(\beta\) corresponding to the emission from an excited level to its lower levels is given by

\[
\beta(|\psi_f|) = \frac{A(|\psi_f|, |\psi_i\rangle)}{A_{t}(|\psi_i\rangle)}.
\]

(9)

With \(\Omega_t\) parameters, the related radiative properties of Eu³⁺:NAT ceramics were calculated. The spontaneous transition probability of electric-dipole transition \(A_{ed}\) and magnetic-dipole transition \(A_{md}\), the total transition probability \(A_{t}\), the fluorescence branching ratio \(\beta\) and the radiative lifetime \(\tau_{rad}\) are presented in Table 3. In the \(5D_0 \rightarrow 7F_2\) emission, \(\beta\)-value is as high as 62.8%, which is higher than the other transitions, indicating that the \(5D_0 \rightarrow 7F_2\) transition is the main emission of Eu³⁺. The calculated radiative lifetime for \(5D_0\) level is about 3.51 ms, and this value is longer than those of Eu³⁺ doped alkali-lutetite glasses \([26]\), La₂O₃–3B₂O₃ crystals \([36]\) and Lu₂O₃ ceramics \([37]\). All these results indicate that Eu³⁺:NAT ceramics can be an effective luminescence material.

#### Table 2

<table>
<thead>
<tr>
<th>Host matrix</th>
<th>(\Omega_2 (10^{-20} \text{ cm}^2))</th>
<th>(\Omega_4 (10^{-20} \text{ cm}^2))</th>
<th>(\Omega_6 (10^{-20} \text{ cm}^2))</th>
<th>Refs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluoride glasses</td>
<td>0.51</td>
<td>4.19</td>
<td>2.79</td>
<td>([28])</td>
</tr>
<tr>
<td>LaF₃:Eu³⁺ crystals</td>
<td>1.19</td>
<td>1.16</td>
<td>0.39</td>
<td>([29])</td>
</tr>
<tr>
<td>Fluorosilicate glass ceramics</td>
<td>1.38</td>
<td>0.84</td>
<td>–</td>
<td>([30])</td>
</tr>
<tr>
<td>Oxyfluoroborate glass ceramics</td>
<td>2.1</td>
<td>4.05</td>
<td>1.92</td>
<td>([31])</td>
</tr>
<tr>
<td>Fluorophosphate glasses</td>
<td>3.24</td>
<td>5.11</td>
<td>2.89</td>
<td>([32])</td>
</tr>
<tr>
<td>Oxyfluoroborate glasses</td>
<td>3.45</td>
<td>2.93</td>
<td>0.97</td>
<td>([33])</td>
</tr>
<tr>
<td>Fluoroaluminate glasses</td>
<td>5.35</td>
<td>1.18</td>
<td>1.30</td>
<td>([34])</td>
</tr>
<tr>
<td>Phosphate glasses</td>
<td>6.91</td>
<td>5.01</td>
<td>5.76</td>
<td>([35])</td>
</tr>
<tr>
<td>Silicate glasses</td>
<td>9.65</td>
<td>6.68</td>
<td>1.15</td>
<td>([36])</td>
</tr>
<tr>
<td>Alkali-tellurite glasses</td>
<td>11.06</td>
<td>4.38</td>
<td>0.96</td>
<td>([37])</td>
</tr>
<tr>
<td>NAT ceramics</td>
<td>2.95</td>
<td>0.39</td>
<td>–</td>
<td>Present work</td>
</tr>
</tbody>
</table>
Table 3
Predicted spontaneous transition probabilities, radiative lifetime and branching ratios of Eu\textsuperscript{3+}:NAT ceramics.

<table>
<thead>
<tr>
<th>Transition</th>
<th>( A_{\text{ed}} ) (s(^{-1}))</th>
<th>( A_{\text{rad}} ) (s(^{-1}))</th>
<th>( \lambda_{\text{rad}} ) (ms)</th>
<th>( \beta ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5D(^0) ( \rightarrow ) 7F(_1)</td>
<td>0</td>
<td>94.63</td>
<td>285.07</td>
<td>3.51</td>
</tr>
<tr>
<td>5D(^0) ( \rightarrow ) 7F(_2)</td>
<td>178.98</td>
<td>0</td>
<td>62.8</td>
<td></td>
</tr>
<tr>
<td>5D(^0) ( \rightarrow ) 7F(_4)</td>
<td>11.46</td>
<td>0</td>
<td>4.0</td>
<td></td>
</tr>
</tbody>
</table>

4. Conclusions

Eu\textsuperscript{3+} doped sodium–aluminum–tellurite ceramics have been designed and fabricated as potential luminescent materials. Eleven emission bands including blue and green peaks have been clearly recorded, and they almost cover the whole visible spectral region under ultraviolet radiation. The maximum phonon energy of Eu\textsuperscript{3+}:NAT ceramics has been estimated to be \( \sim 760 \) cm\(^{-1}\), which is beneficial to achieving high fluorescence efficiency of RE ions. Judd–Ofelt intensity parameters \( \Omega_2 \) and \( \Omega_4 \) have been derived from the emission spectrum, and the values are \( 2.95 \times 10^{-20} \) and \( 0.39 \times 10^{-20} \) cm\(^2\), respectively. Low phonon energy and bright visible fluorescence indicate that RE doped NAT ceramics can promote the development of illumination, display and detection devices.

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