## Pressure-enhanced light emission and its structural origin in Er:GdVO<sub>4</sub>

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## Pressure-enhanced light emission and its structural origin in Er:GdVO<sub>4</sub>

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Rare earth phosphors have been widely studied because of their sharp emission lines and excellent optical performance. However, photoluminescence (PL) tuning by crystal field in  $\text{Er}^{3+}$  embedded phosphors has always been a challenge. Here, we demonstrate that pressure can help to enhance the red and green light emission simultaneously in Er:GdVO<sub>4</sub>. Synchrotron X-ray diffraction investigations revealed that a structural phase transition was responsible for the enhancement. Our work brightens the future prospects for applications of  $\text{Er}^{3+}$ -based PL materials in various fields, such as high power lasers and (bio) medical imaging. *Published by AIP Publishing*. [http://dx.doi.org/10.1063/1.4973993]

The photoluminescence (PL) effect has led to broad applications in various fields, such as road safety markers, trace element analysis in food, medical imaging for diagnosis, and high-performance laser systems, etc.<sup>1–5</sup> Photoluminescence can be observed in various material systems. Some typical PL materials are semiconducting GaAs, GaP, and CdTe in the form of quantum dots and epitaxial films, insulating rare-earthion-doped borates, phosphates, aluminates, vanadates, and tungstates (for example, Nd:YAlO<sub>3</sub>) in the form of single crystals, and some special nanostructures as well.<sup>6–12</sup> The PL effect also provides characteristic fingerprints of certain materials, for example, the determination of the chirality of single-wall carbon nanotubes.<sup>13–15</sup> Some rare earth ions, such as  $Er^{3+}$ ,  $Eu^{3+}$ , and  $Nd^{3+}$  also have the characteristic PL spectra due to the special electronic energy levels of their 4f orbital.<sup>16–18</sup> As a result, they have been used as light emission centers to produce high power lasers, which can cover the spectrum from ultraviolet (UV) to infrared (IR) light.<sup>16,18–20</sup> To achieve higher efficiency and intensity of the PL effect, there are two main routes. The most common is the chemical modification of the materials themselves. Energy transfer is the most effective way to achieve higher PL intensity.<sup>17,21–25</sup> Choosing a proper parent matrix structure for emission centers is also important, especially for those sensitive to the crystal field. Eu<sup>3+</sup> is one of these cases.<sup>26–29</sup> Another method is to use external fields, such as an electrical or magnetic field. In As/GaAs quantum dots, the magnetic field confines the exciton wave function and changes the radiative recombination.<sup>30</sup> Similar PL enhancement effects from using a magnetic field were also observed in

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a strong spin-orbit-coupling organic-inorganic system.<sup>31</sup> Even though an electrical field usually suppresses the PL effect,<sup>32–34</sup> there are also some cases in which the PL is enhanced.<sup>35,36</sup>

Beyond these methods, pressure represents an alternative way of modifying the PL behavior, as the lattice structure of most materials can be easily changed by external pressure. A Nd<sup>3+</sup>-doped YVO<sub>4</sub> crystal was studied under high pressure<sup>37</sup> and its PL spectrum showed a clear red shift with pressure, while the PL intensity of all modes was somehow suppressed under high pressure. Another positive pressure effect on PL behavior was observed in Eu:YF<sub>3</sub> bulk material, in which the PL intensity was enhanced before the phase transition up to 7.5 GPa, and the intensity decreased after the phase transition.<sup>38</sup> Er<sup>3+</sup> containing optical materials are of broad interest due to their stable optical behavior (weak dependence on crystal field) and wide-spectrum light emission (from green light to red light to IR light).<sup>16,39</sup> However, tuning their PL behavior by changing their matrix crystal structure is difficult. Here, we have tentatively enhanced the PL effect of both the red and green light emission bands in an Er<sup>3+</sup> embedded phosphor using a highpressure technique. This work provides a possible way to produce highly efficient phosphors and high power lasers using high-pressure synthesis or thin film techniques, which are important for small-scale PL devices.

Under ultraviolet laser excitation with a wavelength of 325 nm, the crystal emits bright green light, as shown in Figure 1(a). The spectrum is provided in Figure S1 in the supplementary material. Figure 1(b) is a photograph of the sample in the diamond anvil cell (DAC) at 30 GPa, taken at a higher magnification. Figure 1(c) displays a schematic diagram of the  $Er^{3+}$  ion electron energy level structure.

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The UV-light-excited PL spectrum is used to help determine the reality of the PL signal in the range of 540 to 750 nm and exclude any possible Raman signal from the crystal itself that might be present when collecting the PL spectra excited by the 532 nm green laser.

The pressure-dependent red light emission spectra are presented in Figures 2(a) and 2(b). Some representative line plotted spectra are displayed in Figure S2(a) in the supplementary material. The highly accurate and sensitive Raman system allowed us to collect a series of fine PL spectra. In the low-pressure range below 9.9 GPa, the PL peaks are very sharp, but their intensities decrease with rising pressure. The sharp and narrow red light emission is a typical characteristic of the rare earth ion  $\mathrm{Er}^{3+}$ . The emission band from 650 to 675 nm is due to the light emission between the  ${}^{4}F_{9/2}$  (excitation state) and  ${}^{4}I_{15/2}$  (ground state), as indicated by the red arrow in Figure 1(c). The suppression effect on the PL from high pressure is very prevalent,<sup>37,40</sup> and this could be explained by the reduced absorption efficiency of the laser under high pressure.<sup>37,41,42</sup> In addition, the disorder caused by pressure could be another factor and may also reduce both the absorption efficiency and the emission efficiency. This trend change starts from 9.9 GPa, when the intensity of the first emission band increases before reaching a maximum at 11.7 GPa. As the pressure increases to 25 GPa, the PL intensity is strongly suppressed, and it is hard to identify any peak when the pressure reaches 30 GPa. The pressuredependent intensity of a representative PL mode is plotted in Figure 2(b) and clearly shows the fourfold enhancement of the PL intensity for this mode, while the whole PL intensity enhancement is also doubled on average. The significant changes of the PL intensity near 10 GPa and 25 GPa could



FIG. 1. Direct optical observation of the Er:GdVO<sub>4</sub> sample in a diamond anvil cell and electron energy level scheme. (a) Magnified photograph (5×) of the green light emission from Er:GdVO<sub>4</sub> excited by a 325 nm laser, (b) magnified photograph (20×) of the Er:GdVO<sub>4</sub> sample inside the diamond anvil cell under 30 GPa, and (c) an electron energy level scheme of  $Er^{3+}$  ions.

correspond to possible structural phase transitions. Most peaks show a red shift under high pressure, but the peak positions do not shift much, which differs from the PL effect found in many  $Eu^{3+}$ -based optical materials. This is consistent with the previous research on  $Er^{3+}$ -based optical materials, however, where the PL effect of  $Er^{3+}$  always shows weak crystal field dependence.

The green light emission is much stronger than the red light emission in the Er:GdVO<sub>4</sub> single crystal. To obtain a reliable PL data within our system limitations, the excitation power for green light emission was reduced to 1% of the laser output power (200 mW). The results are presented in Figures 2(c) and 2(d). Some representative line plotted spectra are displayed in Figure S2(b) in the supplementary material. The green light emission is attributed to the  ${}^{4}S_{3/2} {}^{-4}I_{15/2}$  and  ${}^{2}\text{H}_{11/2}$  -  ${}^{4}\text{I}_{15/2}$  emissions, as illustrated by the green arrows in Figure 1(c). Similar to the red light emission trend, the green light emission is first suppressed in the low-pressure range below 9.9 GPa. Thereafter, the PL effect is strongly enhanced from 9.9 GPa and reaches a maximum at 11.70 GPa. Figure 2(d) demonstrates the pressure effect on the intensity and peak position of the main PL mode. Here, we also managed to achieve a fourfold enhancement of the green light emission, which raises the possibility of producing a high power green light laser using a Er:GdVO<sub>4</sub> single crystal in the form of a high-pressure phase. The green light emission is also strongly suppressed above 30 GPa, which is consistent with the red light emission. All the peaks show a red shift trend with pressure except those near 9.9 GPa and 23 GPa.

To understand the different PL behavior at various pressures, we investigated the crystal structures by synchrotron X-ray micro-diffraction (wavelength: 0.6199 Å). The pressure-dependent XRD patterns are presented in Figure 3(a) from 1.5 to 30 GPa. Above 30 GPa, the diffraction signal is very weak, and the sample seems to become amorphous. As indicated by the dashed line near  $2\theta = 10.5^{\circ}$ , a new diffraction peak appears from 7.4 to 11.4 GPa, which signals a phase transition. This new peak is almost invisible above 11.4 GPa, and the main peak near  $2\theta = 10^{\circ}$  also disappears, which suggests the end of the phase transition process. Similar to other  $AVO_4$  materials,<sup>46,47</sup> such as  $EuVO_4$  and ScVO<sub>4</sub>, Er:GdVO<sub>4</sub> undergoes a phase transition from a zircon-type structure to a scheelite-type structure, starting from 7.4 GPa in our experiment, which is very similar to the phase transition pressure in Eu:  $GdVO_4$  (7.4 GPa)<sup>48</sup> and EuVO<sub>4</sub> (6.8 GPa).<sup>46</sup> Previous Raman results of Eu: GdVO<sub>4</sub><sup>48</sup> show that the scheelite phase coexists with the zircon phase even at  $\sim 16$  GPa. In our case, the XRD data clearly shows that the phase transition finishes above 11.4 GPa, which is close to 10.1 GPa in EuVO<sub>4</sub><sup>46</sup> (EuVO<sub>4</sub> has a lower starting transition pressure). A typical characteristic of the zirconscheelite phase transition is the relative intensity change in the first two diffraction peaks. The low-pressure phase T1 is assigned to a tetragonal zircon-type structure (space group:  $I4_1$ /amd), and the high-pressure phase T2 is a tetragonal scheelite-type structure (space group:  $I4_1/a$ ). Starting from 23.1 GPa, it is expected that the crystal has a monoclinic structure with space group C2/c (equal to I2/a).<sup>49</sup> The pressure-dependent phase diagram and lattice parameters are presented in Figure 3(b), while the atomic structures of the



FIG. 2. The red and green light emission under high pressure, excited by a 532 nm laser. (a) Pressure-dependent red light emission; (b) the pressuredependent intensity of the main red light emission mode; (c) pressuredependent green light emission; and (d) the pressure-dependent PL intensity of the main green light emission mode.

three different phases are presented in Figures 3(c)-3(e). Note that there is a small amount of the T2 phase coexisting with the T1 phase in the low-pressure range. Details of the T1 and T2 phase volume fractions are provided in Figure S3 in the supplementary material. Two representative refinement patterns are displayed in Figure S4. We repeated the XRD experiment at ambient conditions using two samples with different grinding times and found that the T2 phase



FIG. 3. The structure evolution under high pressure. (a) X-ray diffraction patterns of an Er:GdVO<sub>4</sub> powder sample at various pressures. The low-pressure phase T1 (space group: I41/amd) transforms to a high-pressure phase T2 (space group: I41/a), starting from 7.4 GPa and finishing above 11.4 GPa. A second phase transition is found near 23.1 GPa and the new highpressure phase can be assigned to a monoclinic phase with space group C2/c. (b) Pressure-dependent phase diagram and lattice parameters (T1-d: T1 dominating; T2-d: T2 dominating). (c)-(e) Atomic structures of the three phases (purple atoms: Gd/Er (large size), brown atoms: V (medium size), and green atoms: O (small size)).

content increased when the grinding time was longer, as shown in Figure S5 in the supplementary material. It is reasonable that T2 phase ratio increases with a longer grinding time due to the shear stress, which could allow the phase transition at a much lower pressure.<sup>50-52</sup> A previous nonhydrostatic experiment shows that the phase transition of pure GdVO<sub>4</sub> occurs near 5 GPa.<sup>53</sup> Sometimes, the shear stress can reduce the phase transition pressure by a factor of 3-4.52 Here, we do not exclude the possibility that the sample itself may have a small amount of T2 phase but this does not affect the discussion of our results. Second order Birch-Murnaghan equation of state (EOS) analysis was also performed for both the T1 and T2 phases, as shown in Figure S6 in the supplementary material. The bulk modulus of the T1 phase is  $102.4 \pm 14.0$  GPa, which is slightly smaller than EuVO<sub>4</sub>.<sup>46</sup> The bulk modulus of the T2 phase is  $136.5 \pm 15.4$  GPa, which is comparable with the scheelite YVO<sub>4</sub>.<sup>49</sup>

The correlation between the PL behavior and the structural change can be easily established based upon the detailed high-pressure structural analysis. When the pressure is lower than 9.9 GPa, the first tetragonal phase T1 dominates, and it is responsible for the PL effect, while the pressure causes lattice distortion and disorder, which weakens the absorption efficiency and suppresses the PL.37,41,42 Above 9.9 GPa, the second phase T2 dominates, and the PL starts to become stronger. When the phase transition finishes above 11.7 GPa, the PL intensity reaches a maximum. After that, the PL is also suppressed by pressure. The enhancement behavior of the light emission in the T2 phase should relate to the change of crystal field or the local atomic environment. As shown in Figure 4, the O coordination number of the Er light emission centers is four in both the T1 and T2 phases. However, the V coordination number of the Er light emission centers increases to eight in the T2 phase, rather than six in the T1 phase. The strong suppression of PL around 30 GPa is probably related to weak absorption efficiency and the low symmetry of the high-pressure monoclinic phase. Meanwhile, the lattice distortion and atomic disorder further suppress the PL efficiency. This is contrary to what occurs in Eu:YF<sub>3</sub>, where the enhancement behavior occurs before the phase transition, and the PL effect is



FIG. 4. The atomic environment of  $Er^{3+}$  emission centers. (a) Er-V (upper) and Er-O (lower) bonds for T1 phase; (b) Er-V (upper) and Er-O (lower) bonds for T2 phase.

suppressed after the phase transition.<sup>38</sup> Here, PL enhancement in Er:GdVO<sub>4</sub> is found when the phase transition finishes. This means that the mechanisms are different and that increasing the density of the light emission centers cannot benefit PL efficiency in all systems. More importantly, the scheelite-type structure is quenchable, can be synthesized by high-pressure methods and can even be stabilized simply by chemical doping, which suggests the possibility of using a scheelite-type material as a matrix optical material for high power lasers. The collective enhancement of green and red light emission suggests that the infrared light emission of  $\mathrm{Er}^{3+}$  would also be enhanced. Thereafter, the upconversion efficiency in this kind of material would be promoted as well, which would help to stimulate their application in medical imaging. Depositing a thin film with a scheelite-type phase is also feasible, which would help to scale up production and have benefits for the miniaturization of highly efficient optical devices.

In summary, we report the simultaneous enhancement of red and green light emission in Er:GdVO<sub>4</sub>. By compressing the sample with a diamond anvil cell, the intensity of the main red and green light emission modes was found to increase fourfold under a pressure of ~11.7 GPa, while the overall PL intensity doubled under compression. Synchrotron-based X-ray diffraction measurements indicated that a pressure-induced structural transition accounts for the PL enhancement. Therefore, high-pressure compression could be an effective approach to achieving highly efficient photoluminescence and hence, brighten future prospects for applications of PL materials in various fields.

See supplementary material for experimental details, selected PL spectra, and additional structural analysis.

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