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Appearance of new photoluminescence peak and spectral evolution of Eu^{3+} in $La_2Zr_2O_7$ nanoparticles at high pressure



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ABSTRACT

Lots of efforts have been vested recently on tuning the structure and optical properties, particularly for the luminescent nanophosphors. High pressure in that direction has shown great potential to tune the performance of nanophosphors. However, very little attention has been paid on tuning the optical properties of technologically important La₂Zr₂O₇ (LZO) pyrochlore nanoparticles (NPs) and its doped counterpart La₂Zr₂O₇:Eu³⁺ (LZOE) NPs under elevated pressure. Equally little effort has been given on local structure evolution of Eu^{3+} ion as a function of pressure in the doped LZOE NPs. Here in this work, LZO depicted strong oxygen vacancy blue emission under ambient and up to 5.54 GPa pressure, and beyond that, there is an evolution of pressure induced green and red photoluminescence (PL) band. This is projected in color tuning from blue \rightarrow green \rightarrow white in low-intermediate-high pressure regime. Emergence of unusual visible PL band at elevated pressure is ascribed to structural phase transition from ordered pyrochlore to cotuunite structure which endows large disordering in the LZO structure as well as induces lattice distortion of ZrO₆ octahedra. High-pressure PL excitation spectra of the LZOE NPs throw interesting results wherein the charge transfer band is retained in ordered pyrochlore and disappears in high pressure cotuunite phase. As the pressure is raised, the asymmetry ratio of the LZOE NPs exhibits monotonic decrease up to ~19.77 GPa and saturation beyond that, implying a high local symmetry around Eu³⁺ ions from high pressure compression. This work is a way forward in the area of understanding structure-property correlation, pressure induced color tunability and modulating local structure of lanthanide dopant in the area of luminescent NPs.

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1. Introduction

High pressure spectroscopy is one of the most fascinating areas of recent research owing to the observation of several new phenomena, properties and applications at elevated pressure.[1–4] High pressure effects have led to development of new materials [1], improved phase stability [5], phase transitions [6,7], band gap narrowing [6], superconductivity [8], materials of ultrahigh hardness [9], linker rotation in photoluminescent metal organic framework [10], etc.

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https://doi.org/10.1016/j.jallcom.2021.159438 0925-8388/© 2021 Elsevier B.V. All rights reserved. Pressure is one of extremely critical physical parameters. High pressure applied on materials leads to restricted intermolecular attraction giving changes in bond length, crystal field, etc. Under high pressure, atomic volume of bulk materials is expected to decrease by 10% or more [11]. The associated bond length contraction leads to important changes in material properties, including their electronic structure [11]. These physico-chemical changes in turn significantly influence the optical properties of materials.

In fact, recently high-pressure effects have led to very interesting optical phenomena. Liu et al. recently observed high pressure induced blue shift and enhanced photoluminescence (PL) in 9-(3-(1,2,2-triphenylvinyl)-phenyl)anthracene crystal.[12,13] PL of metal nanoclusters was found to increase by up to 2 orders of magnitude at pressures up to 7 GPa [14]. High-pressure effects on the properties of carrier traps in energy storage phosphors were well explained by Majewska et al.[15] There have been reports wherein local structure of dopant ions in luminescent materials modulated

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Fig. 1. (a) XRD patterns and (b) Raman spectra of the LZO and LZOE NPs. The standard pattern corresponding to ICDD 17-0450 is also included in Fig. 1a.

from low to high symmetry at elevated pressure [16–18]. High pressure has found to display profound influence not only on conventional PL but also on upconversion and persistent luminescence [19–21]. A paper published by Guo et al. has claimed an increase in persistent luminescent duration whereas Zhang et al. reported enhanced upconversion luminescence in KAIF₄ layered structure at high pressure [20,21]. High pressure luminescence measurement has not only yielded exciting and novel optical phenomena but also rendered new kinds of pressure sensors [22,23].

Recently, $A_2B_2O_7$ type pyrochlores have been catching a lot of attention among materials science community owing to their interesting features such as structural flexibility, ability to accommodate large number of dopants, high thermal/mechanical/chemical/radiation stability, high oxygen conductivity, high dielectric constant, etc. [24–27]. It is considered an extremely important class of functional materials for applications in clean energy, catalysis, nuclear waste hosts, scintillators, phosphors, thermal sensors, etc [24–30].

High pressure spectroscopy in general and luminescence spectroscopy in particular has fetched a lot of interest in pyrochlores because of their intrinsic structural flexibility to exist in defect fluorite, ideal pyrochlore, cotunite, etc., which display structure sensitive emission [16,24,28,31]. As a result, high pressure spectroscopy on pyrochlores has been a subject of intense discussion. There has been a lot of reported research work pertaining to phase and structural evolution on similar aspects. [6,7,31–33].

In literature, we could find only one report on high pressure luminescence of pyrochlores on undoped La₂Sn₂O₇ [6,7,31–33]. Despite this report offers interesting results, there is a complete lack of research on this direction in general. Among pyrochlores, La₂Zr₂O₇ (LZO) is regarded as one of the most explored pyrochlores for thermal barrier coating, transparent ceramic, nuclear waste host, luminescent host, catalysis, etc [34–39]. Based on our previous work, it was found that LZO displayed intense violet-blue (400 nm) and weaker near infrared (707 nm) emission due to the presence of oxygen vacancies under ultraviolet irradiation [38,40]. This was concluded based on density functional theory calculations. Any role of La³⁺ (f⁰) and Zr⁴⁺ (d⁰) in PL process was completely ruled out as no unpaired f- or p-electrons are available for electronic transition.

Hence, in this work, we synthesized LZO and $La_2Zr_2O_7:Eu^{3+}$ (LZOE) nanoparticles (NPs) and explored their high-pressure PL. Being the most sensitive lanthanide dopant, Eu^{3+} ion was selected for the doping studies owing to its pure magnetic dipole transition (PMDT) and forced electric dipole transition (FEDT). Using Eu³⁺ ions as local structural probe, we would deepen our understanding on the pressure-dependent PL behaviors of the LZO NPs. A diamond anvil cell was used for the high-pressure measurement and ruby peak was used as pressure reference.

2. Experimental

Both the LZO and LZOE NPs (5.0 mol % of Eu^{3+}) were synthesized using a molten salt synthesis [41]. There were two steps involved: co-precipitation and molten salt addition followed by thermal treatment. Aqueous solution mixing of La(NO₃)₃·6H₂O, ZrOCl₂ and Eu (NO₃)₃·6H₂O followed with NH₄OH(aq) addition led to the co-precipitation of a single source precursor La(OH)₃·Eu(OH)₃·ZrO (OH)₂·nH₂O. This single source precursor was mixed with a KNO₃-NaNO₃ salt mixture and heated at 650 °C to get pure phased LZOE NPs. For the undoped LZO NPs, the same procedure was employed without adding Eu(NO₃)₃·6H₂O. The instrumentation used in this work pertaining to XRD, FESEM, Raman, and PL measurements has been well documented in our earlier work [42,43].

3. Results and discussion

3.1. Phase purity and structure: Powder XRD and Raman spectroscopy

The XRD patterns of the LZO and LZOE NPs (Fig. 1a) match completely with the cubic lattice of $La_2Zr_2O_7$ pyrochlore [38,40]. These patterns completely match with the standard XRD pattern of LZO corresponding to ICDD No. 17–0450. The exactly identical XRD patterns of the LZO and LZOE NPs confirmed that Eu^{3+} ion entered the lattice site without disturbing the actual structure and did not lead to any unwanted phase. Due to close similarity in structure of ordered pyrochlore (OP) and defect fluorite (DF) structure, it is extremely difficult to make any distinction between powder XRD patterns of the OP and DF phases of $La_2Zr_2O_7$. To establish correct information on structure of the LZO and LZOE NPs, as a highly sensitive technique for metal-oxygen vibration, Raman spectroscopy was carried out.

It is known that the DF structure belongs to Fm-3 m space group (SG) whereas the OP structure belongs to Fd-3 m SG. Fm-3 m SG depict only one Raman peak (F2g mode) due to oxygen ion vibration in a tetrahedral environment formed by four A/B-cations of $(A/B)_4O_7$. On the other hand, the OP structure with $A_2B_2O_6O'$ and Fd-3 m SG



Fig. 2. (a & b) SEM images of the LZO and LZOE NPs, respectively. (c) EDX spectrum and (d) elemental mapping of the LZOE NPs.

displays 6 Raman peaks owing to cation-anion vibration whereas the highest wave number Raman mode is ascribed to the vibration of oxygen sublattice (AO₈/BO₆) [24,27]. Raman spectra of the LZO and LZOE NPs (Fig. 1b) shows a typical group of six peaks located at 307, 330, 404, 501, 527, and 600 cm^{-1} owing to A_g, E_g and 4 F_{2g} modes. The band located around 750 cm⁻¹ is attributed to the distortion of ZrO₆ octahedra. The fact that we are getting six peaks in the Raman spectra at desired peak position confirms that the LZO and LZOE NPs both stabilize in the OP structure wherein lanthanum exists as LaO₈ in the form of distorted scalenohedra and Zr exists as ZrO₆ in perfect octahedra.

3.2. Morphology, average size and purity: SEM and EDX

SEM images of the LZO and LZOE NPs (Figs. 2a and 2b, respectively) clearly show that most of the individual particle assumes spherical shape. The spherical LZO NPs have an average particle size of around 40–45 nm while the LZOE NPs have a reduced average particle size of 30–35 nm. This reduction in the particle size upon doping is ascribed to lattice strain and distortion caused by ionic radius difference of Eu³⁺ and La³⁺/Zr⁴⁺ ion. We could not rule out the fact that both the LZOE NPs are clustered and aggregated, but the individual NPs look highly monodispersed in both of them. EDX spectrum of the LZOE NPs (Fig. 2c) clearly shows the presence of La, Zr, O, and Eu elements. The presence of Eu³⁺ ion from the EDX spectrum of the LZOE NPs also confirms its incorporation in the LZO lattice. The peaks of C and Al come from carbon tape and aluminum sample holder used in performing the SEM and EDX experiments. Elemental mapping image of the LZOE NPs (Fig. 2d) shows highly uniform and homogenous distribution of all the constituent elements. It was further established using specific individual element mapping images of La, Zr, O and Eu of the LZOE NPs as shown in Fig. S1.

3.3. Pressure dependence PL of the LZO NPs

3.3.1. *Emission spectroscopy*

Emission spectra of the LZO NPs at different pressure up to ~31.8 GPa (Fig. 3a) gave interesting behaviors in terms of peak profile evolution. The excitation spectra of the LZO NPs at different pressures (Fig. 3b) were quite featureless with just one broad band peaking around 350 nm, which predominately has origin to different combinations of defects. The PL emission spectra displayed only one blue band in the region around 450 nm up to 8.29 GPa. We observed similar violet blue emission from the LZO NPs at ambient temperature and pressure which was ascribed to the presence of oxygen vacancies [38,40]. Under NUV irradiation, the photoexcited holes combine with electrons tapped in the oxygen vacancies giving bright blue emission. Based on the DFT calculations, it was proposed that neutral, singly ionized, and doubly ionized vacancies all contributed to blue emission in the LZO NPs [38,40]. Beyond 5.54 GPa, there was an evolution of a new emission peak at around 520 nm (G-band) and 635 nm (R-band) which persisted and even intensified as we went to higher pressure. Moreover, at higher pressure, the blue band at



Fig. 3. Pressure dependent (a) emission spectra, (b) excitation spectra, (c) Raman spectra, and (d) color coordinate diagram of the LZO NPs. The arrow in Fig. 3d serves as a guide to eyes by showing increasing pressure from left to right.

450 nm got feeble in intensity with an increase in extent of broadening. There have been a few previous reports on high pressure studies of pyrochlores. Li et al. carried out XRD and Raman measurements of Dy₂Ti₂O₇ under high pressure and found structural phase transition and band gap lowering at high pressure of ~35 GPa, which was attributed to band structure change [6]. On the other hand, Zhao et al. carried out PL measurements on another frustrated pyrochlore $Eu_2Sn_2O_7$ up to ~34 GPa [7]. It was observed that the symmetry ratio (A_{12}) increased initially up to 17.9 GPa and then there was a reduction. Symmetry ratio is defined as the ratio of 590 nm orange band (${}^{5}D_{0} \rightarrow {}^{7}F_{1}$, PMDT) and 615 nm red band (${}^{5}D_{0} \rightarrow {}^{7}F_{2}$, FEDT). These authors ascribed the observed phenomenon to the suppression of trigonal distortion in SnO₆ at 17.9 GPa owing to an isostructural transition wherein a kink is exhibited by Sn-O-Sn and Eu–O–Eu bonding angles and lengths. In another work by the same group on Lu₂Sn₂O₇ [31], a new high-pressure PL peak was observed at around 2.0 eV above 2 GPa, which was ascribed again to the suppression of trigonal distortion in SnO₆. Pressure induced structural phase transition from OP to DF and to cotuunite structure of pyrochlore compounds was also observed in rare earth hafnate pyrochlores by Turner et al. [33]. We believe that the evolution of the G-band and R-band beyond 5.54 GPa was also associated to some kind of structural changes in our LZO NPs.

To see if any kind structural phase transition has happened or new structure has formed, we carried out pressure dependence

Raman spectroscopy (Fig. 3c). From these spectra, it is clearly inferred that there was a new Raman peak evolved around 825 cm⁻¹ beyond 5.54 GPa. It also intensified as we moved to higher pressure with our measurement range. The particular peak was ascribed to structural phase transition from OP structure to highly disordered cotuunite structure, which has orthorhombic unit cell instead of cubic unit cell with Pnma SG [24,33]. Rittman et al. found the stabilization of cotuunite phase in rare earth zirconate pyrochlore at high pressure with no site preferences for different types of cations [32]. The onset of cotunite phase from our LZO NPs started at a relatively low pressure of ~5.54 GPa compared to titanates, which can be ascribed to larger defect formation energy in the latter compared to the former [44]. The cotuunite phase led to much higher level of structural distortion in ZrO₆, which changed Zr–O–Zr bond angle and bond distance and may lead to the evolution of new G- and R-bands at pressure beyond 5.54 GPa.

High pressure PL relies on the wavelength evolution of the R1 sharp fluorescence line of Cr^{3+} :Al₂O₃ as a function of applied pressure, which is called the ruby scale for pressure measurements. Utilization of the linear red shift of the ruby fluorescence R1 line ($E_{2g} \rightarrow {}^{4}A_{2g}$ emission following ${}^{4}A_{2g} \rightarrow T_{2g}$ or T_{1g} excitation) as a function of applied pressure is the most standard pressure scale. The emission spectra of ruby used as the pressure scale for our LZO NPs study are also shown in Fig. S2.

3.3.2. Pressure driven Color tunability

Color tunable phosphors are considered excellent discovery in luminescent materials as they can be applied in a wide variety of applications [22,45]. Moreover, achieving white color emission is considered a boon for solid-state lighting and optoelectronic industry. In fact, it is the most enlightening invention in the area of solid-state lighting owing to its usefulness to mankind with low cost, high energy output, low power consumption, high brightness, and environment benignness [29,30]. In most phosphors, white light emission is achieved via the culmination of red, green and blue emitting phosphors as the RGB combination to give white emission. This requires different kinds of dopants in most of reported phosphors such as Eu³⁺, Tb³⁺, Tm³⁺, Dy³⁺, Sm³⁺, etc. [46]. The combined doping unnecessary exposes the lattice to high strain which may affect the radiative transition probability and the quantum yield of the synthesized phosphors. Color coordinates of the LZO NPs under the applied pressures were calculated and the CIE index diagram has been shown in Fig. 3d. Herein in ambient condition, the LZO NPs emitted blue light at 450 nm owing to oxygen vacancies wherein green and red emission appeared beyond 5.54 GPa owing to the distortion of ZrO₆ octahedra and the phase transformation from OP to cotuunite structure. The emission color evolved from blue \rightarrow green \rightarrow white in the low \rightarrow intermediate \rightarrow high pressure regimes. This demonstrates the high potential of pressure induced LZO NPs for wide color gamut spectroscopic applications. Furthermore, the distinct blue \rightarrow green \rightarrow white color change highlighted its usability as optical pressure sensors with visualization advantages.

3.4. Pressure dependence PL of the LZOE NPs

3.4.1. PL excitation

Fig. 4a shows the excitation spectra of the LZOE NPs at different pressures up to 50.76 GPa at emission wavelength of 612 nm. At ambient condition, the excitation spectra consisted of dual features: a broad band around 275 nm owing to $O^{2-} \rightarrow Eu^{3+}$ charge transfer known as charge transfer band (CTB) and several fine peaks between 370 and 500 nm owing to Eu^{3+} intra-configurational f-f bands (IFBs). Within these IFB transitions, the peaks in the range of 380–400, 425–450, 460–475, and 490–500 nm are ascribed to $^7F_0 \rightarrow ^5L_6$, $^7F_0 \rightarrow ^5D_3$, $^7F_0 \rightarrow ^5D_2$, and $^7F_0 \rightarrow ^5D_1$ transitions, respectively. There is significant Stark splitting in these peaks endowed by host crystalline field. Interestingly, it was seen from the spectra that the intensity of the CTB kept decreasing gradually, reduced significantly beyond 5.51 GPa, and then almost disappeared. Meanwhile, not much change could be seen for the IFBs in the spectral



Fig. 4. Pressure dependent (a) excitation spectra, (b) emission spectra, (c) color coordinate diagram, and (d) asymmetry ratio of the LZOE NPs. The arrow in Fig. 4c serves as a guide to eyes by showing increasing applied pressure from left to right.

profile. It needed to be understood here that the CTB mainly came from the LZO host contribution whereas the IFBs from Eu³⁺ dopant contributions. As seen from Fig. 3c, there was a structural phase transition in the LZO NPs from OP to highly disordered cotuunite structure at around 5.54 GPa. The disappearance of the CTB that seemed to be coming from OP phase disappeared in the orthorhombic cotunite high pressure phase owing to the high level of structural distortion in ZrO₆. Since the identity of Eu³⁺ ion remained the same in both OP and high pressure cotunite phase, the excitation band coming from the IFBs of Eu³⁺ ion remained unaltered.

3.4.2. PL emission

Fig. 4b shows the emission spectra of LZOE NPs at different pressure upto 50.76 GPa under excitation wavelength of 390 nm. Under all the conditions of applied pressures, the emission spectra prominently displayed dual features: a peak around 592 nm owing to ${}^{5}D_{0} \rightarrow {}^{7}F_{1}$ transition known as PMDT ($\Delta J = \pm 1$) and a peak around 612 nm consisting of two Stark components (~610 and 630 nm) owing to ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ known as FEDT ($\Delta J = \pm 2$). Intriguingly, the FEDT was more intense than the PMDT at almost entire pressure domain, which confirmed a lowering of the local symmetry of the LZO NPs. It is a well-known fact that PMDT is not affected much by external factors such as temperature, pressure, ligand field, crystal field, etc., whereas FEDT is strongly influenced by all these factors. Accordingly, it can be seen from Fig. 4b that PMDT was not affected much by the change of pressure in terms of either profile evolution or intensity. On the other hand, it can be seen that the number of stark components and the profile pattern of FEDT remained the same while there was intensity change of this peak as the pressure increased. It is quite visible from Fig. 4b that there was a gradual reduction of the FEDT intensity as the pressure increased. The reduced red emission (~610-640 nm) at elevated pressure was ascribed to enhanced probability of non-radiative transition owing to large defect density and increased phonon energy of the host lattice.[18] Here it needs to be stressed that indeed Ω_2 intensity parameter of the ${}^5D_0 \rightarrow {}^7F_2$ transition is influenced more by small angular changes in the first coordination sphere and not due to distortions around the chemical environment of Eu³⁺ ion.

We also deciphered the color coordinates of the LZOE NPs under all the designated applied pressure and showed the CIE index diagram in Fig. 4c. The red to orange color ratio was tuned with red intensity decreasing and moving towards orange-yellow hue.

To understand the evolution of the local symmetry around Eu³⁺ ions in the LZOE NPs, we determined the integral emission intensity ratio of the FEDT to MDT transition, I_{612}/I_{592} , which is normally used as an important parameter to give information about local structure around Eu³⁺ ion in doped phosphors. Fig. 4d shows the variation of I_{612}/I_{592} during the pressure variation from ambient condition to 50.76 GPa. Since the PMDT intensity remained almost constant whereas there was a gradual reduction of the FEDT intensity over the pressure ranges of 0–50.76 GPa, I_{612}/I_{592} decreased monotonically up to 19.77 GPa and then followed with a saturation beyond that pressure regime, which may be due to the intensity equalization of FEDT to PMDT. The reduced value of I_{612}/I_{592} with increasing applied pressure suggested an increased local symmetry around Eu³⁺ ions.

Also, the emission peaks broadened at high pressure for both PMDT and FEDT. The same is ascribed to closing of energy difference between ground and excited states and expansion of sub-energy levels within them under high pressure [47]. The emission spectra of ruby used as the pressure scale for the high-pressure studies of the LZOE NPs were shown in Fig. S3.

4. Conclusion

Here in this work high pressure PL measurement were carried out on both the LZO and LZOE NPs. The nanomaterials were synthesized

using a molten salt method and characterized using XRD. Raman spectroscopy, FESEM, and EDX. These characterizations suggested that both the LZO and LZOE NPs assumed spherical shape having tens of nanometer in size and were stabilized in ordered pyrochlore structure. High pressure PL measurements on the LZO NPs showed the evolution of new visible PL band at pressure beyond ~5.5 GPa. The same was ascribed to pressure induced structural phase transition from ordered pyrochlore structure to disordered cotuunite phase having high degree of disordering. This unusual PL evolution at high pressure endowed in tuning the color emitted by the LZO NPs from blue to green to white color which demonstrated the potential of high-pressure induced design of color tunable phosphors. Eu³⁺ doping in LZO lattice displayed quite interesting result in both PL excitation and emission spectroscopy. Structural phase transition from ordered pyrochlore to disordered cotuunite phase was also reflected in the disappearance of $O^{2-} \rightarrow Eu^{3+}$ CTB in the latter and its presence in the former while applied pressure had no influence on intra-configuration f-f bands of Eu³⁺ ion. The PL emission spectra depicted change in the local structure of Eu³⁺ ion from asymmetric to symmetric environment at elevated pressure, which reflected in tuning red/orange ratio emitted from the LZOE NPs. This study highlighted the influence of elevated pressure tuning the luminescence of nanocrystals and understanding the local structure of dopant ions in nanocrystalline host lattices, which would further affect the luminescence properties of lanthanide doped nanophosphors. Our finding also emphasizes the role of pressure in phase transition and its impact on optical properties of nanophosphors.

CRediT authorship contribution statement

Santosh K. Gupta: Conceptualization, Data curation, Formal analysis, Visualization, Writing - review & editing. **H. Abdou:** Data curation, Methodology, Investigation. **Y. Mao:** Conceptualization, Supervision, Visualization, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jallcom.2021.159438.

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