Spectroscopic and magnetic properties of neodymium doped in GdPO₄ sub-micron-stars prepared by solvothermal method

G.A.Kumar^{1**†}, Nicolas R. Balli[†], M.Kailasnath[‡], L.Christopher Mimun[†], Chamath Dannangoda[§], Karen S. Martirosyan[§], C.Santhosh^{||}, and Dhiraj K.Sardar[†]

[†] *Department of Physics and Astronomy, University of Texas at San Antonio, San Antonio, Texas 78249, United States*

[‡] *International School of Photonics, Cochin University of Science and Technology, Kochi 682022, India*

[§] *University of Texas at Rio Grande Valley, Brownsville, Texas 78520, United States*

^{||} *Department of Atomic and Molecular Spectroscopy, Manipal University, Manipal 576104, India*

¹Corresponding author e-mail:ajith@gakumar.net

Abstract:

Neodymium-doped gadolinium orthophosphate ($\text{GdPO}_4:\text{Nd}^{3+}$) luminomagnetic sub-micron-stars were prepared by solvothermal method using metal nitrates and phosphoric acid. Monoclinic star shaped in six lobed sub-micron-stars with 600 nm length is obtained with uniform particle size distribution. After heat-treatment at 800°C for 1 hour in air, the stars separate into isolated petal shaped particles and show characteristic emission bands of Nd^{3+} with the strongest emission at 1064 nm. The emission intensities and fluorescence decay times are dependent on the Nd^{3+} concentration with the highest emission intensity and longest fluorescence decay time of 311 μs at 1064 nm with 0.5 mol% Nd^{3+} . Under 808 nm excitation with 12 W/cm^2 power density a quantum yield of 9% was obtained for the 1.0 mol% Nd^{3+} . The presence of paramagnetic Gd^{3+} gives magnetic properties to the phosphor with a calculated magnetic moment of 1,510 and 107,965 Bohr magneton at 300 and 5 K, respectively.

Keywords: Multifunctional materials; Optical properties; Magnetic properties; Sub-micron-stars; Rare earths

1. Introduction

Trivalent neodymium is considered to be the most efficient fluorescent rare earth ion among all rare earths with its well known emission around 1060 nm that finds potential applications in various areas of photonics such as lasers [1], military [2], sensors [3], authentication technologies [4], etc. As an active dopant, Nd^{3+} has been incorporated in many crystalline and amorphous hosts and their spectroscopic properties have been extensively studied over the past several decades [5-6]. In practical applications of rare earth doped materials, the dopant concentration is one of the key parameters that control the luminescence intensity. Though higher concentration is preferable for greater fluorescence output, fluorescence quenching is dominant at higher concentrations. Fluorescence quenching is an undesirable property in all optical materials and is mainly related to the crystal site symmetry and lattice spacing which control the non-radiative energy transfer between the dopants [7]. However, proper choice of a host material can help reduce this problem. The host should have high rare earth solubility property to lessen the clustering of rare earths that takes place as its solubility limit is approached. High rare earth solubility and weak ion-ion interactions have been previously reported in LnPO_4 ($\text{Ln} = \text{Gd}, \text{La}, \text{Y}$) [8,9]. Ceramic powder samples of lanthanum phosphate is normally prepared by solid state reaction above 1000 $^{\circ}\text{C}$. With the introduction of nanotechnology in the past decade, several solution methods have been reported for synthesizing metal phosphates. Rare earth metal phosphates with various sizes and shapes can be prepared by controlling various synthesis parameters such as pH of the reactants, types of precursors, synthesis methods, reaction time, pressure, etc. Following these variables, several shapes such as plates, rods, twin, spheres, cubes, rectangular prism [10-16], with hexagonal/monoclinic phase have been reported in the literature.

Metal orthophosphates (MPO_4 , where M is a metal) have several favorable properties such as high thermal stability of (at about) 2300°C , low water solubility, high refractive index ($\text{YPO}_4 = 1.76$, $\text{LaPO}_4 = 1.85$, $\text{GdPO}_4 = 1.97$), high density ($\text{YPO}_4 = 4.22$, $\text{LaPO}_4 = 5.0$, $\text{GdPO}_4 = 6 \text{ g/cc}$) which make them suitable candidates for variety of applications such as lasers, catalyst, magnets and in medicine. In addition, phosphates like GdPO_4 have interesting mechanical properties and are able to support very high pressure without undergoing phase transformations [17]. Recently, rare earth doped GdPO_4 was used as a multimodal imaging agents in medicine with magnetic [18] and optical imaging features [19]. The strong paramagnetic nature of Gd^{3+} provides magnetic properties to the host enabling the application of such metal phosphates as contrast agents in magnetic resonance imaging (MRI). Multicolor as well as white color emissions were recently reported from rare earth-doped, lance-shaped GdPO_4 nanophosphor [20]. Hydrothermal synthesis method and luminescent properties of various rare earth doped LaPO_4 phosphors were reported by Dai et al. [21]. Recently, we have reported the synthesis and spectroscopic properties of Er^{3+} doped submicron LaPO_4 prepared by homogeneous precipitation method [22]. Metal orthophosphate with well-defined star and petal shaped morphology has not been reported in the literature as yet. In this paper, we report for the first time, Nd^{3+} -doped GdPO_4 with star and petal-shaped morphology having both optical and magnetic properties, thus providing a multifunctional feature for these nanoparticles. Because of the low cytotoxicity, phosphate based materials have a lot of interest in various biomedical applications such as optical/magnetic imaging, drug delivery and other therapeutic applications. We propose that under optimized conditions, the synthesized $\text{GdPO}_4:\text{Nd}^{3+}$ star/petal shaped phosphors could be a potential multimodal contrast in medical imaging applications.

2. Experimental

2.1 Material **synthesis**

GdPO₄:Nd³⁺ nanoparticles are prepared by following solvothermal method[23, 24], using Gd and Nd nitrates (99.9%)(GFS Chemicals, NJ) as the metal precursors ethylene glycol as the solvent (99.8%, GFS Chemicals, U.S.) and phosphoric acid as the phosphate source (99.999+%, Sigma Aldrich). The total molarity was maintained at 0.05M, (Gd+Nd) to PO₄ molar ratio was 2:15 for all reactions and the Nd concentration was varied as 0.5, 1 and 2 mol% (where (Nd/(Nd+Gd))×100 = Nd mol%). First, 4.572×10^{-4} mol of Gd(NO₃)₃ 6H₂O and 9.332×10^{-6} mol of Nd(NO₃)₃ 6H₂O were dissolved in 70 mL ethylene glycol followed by 3.033×10^{-3} mol of H₃PO₄ to obtain a clear solution. The mixture was taken in 100 mL stainless steel autoclaves maintaining the volume of the mixture at 70 mL. The closed autoclave was heated at 130°C for 12 hours in a furnace (30400, Barnstead Thermolyne, U.S.). After completing the reaction, the reactors were taken out and naturally cooled to ambient temperature. The resulting precipitate was centrifugally separated (Z323, Labnet, U.S.) then washed 3 times in ethanol followed by de-ionized water by repeatedly dispersing the centrifuge cake with a sonifier (Cell Disruptor 200, Branson, U.S.) and subsequently centrifuging the suspension at a speed of 10,000 rpm for 10 min. The precipitate was freeze-dried for 24 h (25EL Freezmobile, VirTis, U.S) and collected. In order to remove water and organic residues and increase the crystallinity, the nanoparticles are heat treated for 1 hour at 800°C in a muffle furnace (KSL 1500, MTI, U.S.).

2.2. Characterizations

The obtained nanoparticles were characterized by X-ray diffraction analysis (XRD), Field Emission Scanning Electron microscopy (FESEM) and spectroscopy techniques, and magnetic measurements. The diffraction profiles of nanoparticles were collected with an

automated diffractometer (Ultima IV, Rigaku, Japan) using Ni-filtered Cu K α radiation at 44 keV and 30 mA. The data were collected in the range of 20° to 60° using a step size of 0.05° and a count time of 0.15 second. The morphology and size were examined with a Field Emission Scanning Electron Microscope (STEM-5500, Hitachi, Japan). FTIR spectrum was recorded using infrared spectrometer (Jasco FT/IR6300, U.S.) using KBr pellet method. The optical absorption spectrum was collected using a spectrophotometer (Perkin Elmer Lambda 19, U.S.). In order to record the optical absorption spectrum, the ceramic powder was pressed into a circular pellet of diameter 10 mm and thickness of 0.2 mm using a laboratory hydraulic press (Carver, U.S.). A pressure of 10,000 lb was maintained to make the pellet semi-transparent. Fluorescence measurements were done by exciting the samples using 808 nm laser (Spectra Physics, Model 3900S) and the emission of the sample was recorded by the QuantaMaster 51 spectrofluorimeter (Photon Technology International Inc. NJ, U.S.) with an InGaAs detector (Teledyne Judson Technologies, 062-8451, U.S.). The photoluminescence decay curves of 1060 nm emission were measured on a Quanta Master 40 system (Photon Technology International Inc. NJ, U.S.) using the single shot transient digitizer technique with a nitrogen pumped dye laser (Photon Technology International part GL-3300 + GL-302) as excitation source. The nitrogen laser with 800 ps pulse width is used to pump a high resolution dye chamber to give 523 $\pm$ 0.04 nm light. The collected decay curve was analyzed by Origin 8 software (Origin lab, U.S.). For the quantum yield (QY) measurements, we used 12W/cm² power density at 808 nm and was done using a barium sulfate coated 203 mm diameter integrating sphere (Oriel, Model 70451) mounted on the side of the spectrofluorimeter sample chamber, opposite to the excitation source. More details about quantum yield can be seen in Ref. [25].

Magnetic properties were determined by using Multi-functional instrument Cryogen-free Cryocooler-based Physical Property Measurement System (PPMS) EverCool-II (Quantum Design, U.S.). The hysteresis loops of magnetization were measured at room temperature and 5 K under a maximum applied field of ± 9 T. Zero Field-cooling (ZFC) magnetization curves were measured in the temperature range of 1.9-300 K, using a field of 100 Oe.

3.Results and discussion

Fig. 1 shows the unit cell structure of Nd^{3+} doped GdPO_4 . GdPO_4 crystal structure belongs to the monoclinic system with $P2_1/n$ space group and lattice parameters $a = 0.6621$ nm, $b = 0.6823$ nm, $c = 0.6310$ nm and $\beta = 103.976^\circ$ and $Z=4$ [26]. Since the ionic radii of Nd^{3+} and Gd^{3+} are rather close, Gd^{3+} can be easily substituted by Nd^{3+} .

The phase of the as-prepared gadolinium phosphate was studied with XRD (**Fig. 2**). All peaks were indexed monoclinic gadolinium phosphate (JCPDS No. 01-083-0657) with no evidence of secondary phases within the limit of the instrument sensitivity. Examining the relative intensities of the diffraction peaks relative to the JCPDS standard revealed no discrepancy, suggesting the absence anisotropic particle morphology. ESEM was used to characterize the morphology and size of the products. **Fig. 3** shows a typical FESEM image of prepared gadolinium phosphate. The as-prepared particles (**Fig. 3a**) show 6 lobed star shape with a length of around 600 nm for each lobe and center width 200 nm. A close up observation of the star (**Fig. 3 b**) shows that the lobes have a layered structure. After heat treatment all of them lost their star morphology and turned to isolated petal shaped particles as shown in **Fig. 3c** with the same length as those of the lobes of the original star-shaped particles.

Absorption spectrum (**Fig. 4**) shows almost all well-defined Nd^{3+} transitions with the strongest peaks at 805 and 745 nm. Except for the finestructures in the bands, the overall spectrum is identical to other Nd^{3+} doped materials[5-6]. The as-prepared $\text{GdPO}_4\text{:Nd}^{3+}$ sample shows very weak fluorescence (as shown in **Fig. 5**) which is attributed mainly due to the low crystallinity of the material. In order to increase the crystallinity, the sub-micron-stars were heat treated at 800°C for 1 hour. The X-ray analysis shows that the particles retain their as-precipitated phase with better crystallinity, while SEM image shows only change in the morphology where the particle morphology becomes isolated petals now. Solid state sintering appears to be responsible for this substantial increase in crystallinity and change in morphology. **Fig. 5** shows the fluorescence spectra of the samples heat treated at 800°C as a function of the Nd^{3+} dopant concentration. Comparison of the fluorescence intensity shows that 0.5 mol% Nd^{3+} doped sample shows the brightest emission after heat treatment. It was also found that heat treatment increased the emission intensity up to 50 times while retaining spectral line shape. The emission spectrum shows all four characteristic emission bands of Nd^{3+} originating from the $^4\text{F}_{3/2}$ excited state with the strongest emission peaks centered at 1064 nm and the other three bands at 876, 1342 and 1744 nm.

The significant reduction in the emission intensity of the **as-prepared** material is due the semi-amorphous nature of the as-prepared material. In addition to this, it is also assumed that the presence of high frequency vibrational groups such as organic radicals and hydroxyl groups present may also influence the emission properties. FTIR spectrum of the synthesized sample presented in **Fig. 6** shows the presence of hydroxyl and organic groups. Though there is considerable improvement in the crystallinity and reduction in the water content after heat

treatment the adopted thermal treatment is still insufficient for the optimization of the luminescence performance.

Fluorescence decay characteristics were obtained on the heat treated samples at 1064 nm emission bands and the results are presented in **Fig.7**. The decay curves are fitted with a double exponential function of the form $I(t) = A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2)$, where A_1 and A_2 are the fitting coefficients, τ_1 and τ_2 are the decay times and the average decay time (τ_{av}) was obtained using the expression $\tau_{av} = (A_1\tau_1^2 + A_2\tau_2^2) / (A_1\tau_1 + A_2\tau_2)$ [27]. All fitting procedures were done using the scientific software Origin 9 (Origin Lab Corporation, MA). Results show that 0.5 mol% Nd^{3+} doped sample yields highest decay time of 311 μs and it decreases as Nd^{3+} concentration increases as shown in the decay curves. Quantum yield of the 1064 nm emission shows a highest value of 9 % in 0.5 mol% Nd^{3+} doped sample and thereafter it decreases with Nd^{3+} concentration (**Fig. 8**). The significant reduction in emission intensity and quantum yield is mainly due to the energy transfer between Nd^{3+} ions at higher concentrations.

Room temperature (300 K) and low temperature (5K) magnetization curves obtained using the Vibrating Sample Magnetometer are shown in **Fig.9**. While the room temperature magnetization curve is linear, low temperature curve shows an S shaped curve. Magnetization at 9 T for the low temperature hysteresis curve is close to 143 emu/g which is more than 70 times larger than the room temperature magnetization (2 emu/g). Magnetic moment per nanoparticle was obtained by fitting the magnetization curves with Langevin function [28] and the values obtained are 1,510 and 107,965 Bohr magneton, respectively, at 300 and 5 K. The magnetic properties of the nanoparticle is due to the presence of the paramagnetic Gd^{3+} ion having larger

number of unpaired electrons in the outer orbital. The exponential decay type behavior of the zero field cooling (ZFC) studies (**Fig. 10**) also support the paramagnetic nature of the material.

4. Conclusions

Nd^{3+} doped star shaped sub-micron size GdPO_4 phosphor particles can be successfully synthesized with solvothermal methods with metal nitrate precursors and ethylene glycol as the solvent. Unlike other morphologies the present system maintains the same phase after heat treatment. However, the heat treatment induces change in particle morphology from star shape to petal shape with no change in the length and width of the particle. The presence of near infrared (NIR) emitting Nd^{3+} and paramagnetic Gd^{3+} ions contributes both luminescence and magnetic properties for the phosphor particles making them luminomagnetic phosphors with dual properties. The **as-prepared** star shaped particles are weakly emissive due to the poor crystallinity. The heat treatment results in petal shaped particles that show NIR fluorescence properties with 9% quantum yield at 1064 nm emission at a lower excitation power density of 12 W/cm^2 . The fluorescence properties can be greatly enhanced by optimizing the heat treatment procedure. The NIR luminescence characteristics along with magnetic properties enable them to be potential candidates for biomedical imaging and sensing applications. Furthermore, since both the luminescent and magnetic properties are associated with different dopants within the same crystalline lattice, these properties can be controlled over a wide range by changing the type of dopants as well as their concentrations.

Acknowledgements

Authors GAK, NB and DKS would like to acknowledge the financial support from the National Science Foundation Partnerships for Research and Education in Materials (NSF-PREM)

grant NO-DMR-0934218. LCM would like to acknowledge the partial funding from the NIGMS MBRS-RISE GM060655, the National Center for Research Resources (G12RR013646-12), and the National Institute on Minority Health and Health Disparities (G12MD007591) from the National Institutes of Health. CD and KSM acknowledge the financial support from National Science Foundation (Grants 1126410, 1138205 and HRD-1242090). MK acknowledges the financial support through C.V. Raman post-doctoral fellowship offered by University Grants Commission, Government of India, for the year 2013 -14. CS acknowledges the financial support from DST-FIST (India Grant no.SR/FST/PSI-174/2012).

References

- [1] Jiaqi Hong, Lianhan Zhang, Jing Li, Zhaowei Wang, Jingliang He, Peixiong Zhang, Yaqi Wang, Yin Hang, Spectroscopic, thermal and CW dual-wavelength laser characteristics of Nd:LaF₃ single crystal Opt.Mater.53(2016)10–13.
- [2] In Wikipedia, Retrieved June 16, 2015, from https://en.wikipedia.org/wiki/List_of_laser_applications#Military.
- [3] Xinyi Jiang, Cong Cao, Wei Feng and Fuyou Li, Nd³⁺-doped LiYF₄ nanocrystals for bio-imaging in the second near-infrared window, J. Mater. Chem. B. 4(2016)87-95.
- [4] Pawan Kumar, Jaya Dwivedi and Bipin Kumar Gupta, Highly luminescent dual mode rare-earth nanorod assisted multi-stage excitable security ink for anti-counterfeiting applications J.Mater. Chem. C. 2(2014)10468-10475.
- [5] A. A. Kaminskii, Laser Crystals: Their Physics and Properties, Springer Verlag, New York 2013.
- [6] F. Gan, Laser materials, World Scientific, Singapore 1995.
- [7] W. Chen, Doped Nanomaterials and Nanodevices: Luminescence and applications, American Scientific Publishers, California 2010.
- [8] C.R. Kesavulu, Ch. Basavapoornima, C.S. Dwaraka Viswanath, C.K. Jayasankar, Structural and NIR to visible upconversion properties of Er³⁺-doped LaPO₄ phosphors, J.Lumin.171(2016)51-57.
- [9] Xinyang Huang, Preparation and luminescence characteristics of monazite Eu³⁺:LaPO₄ nanocrystals in NH₄NO₃ molten salt, Opt.Mater.50(2015)81-86.

- [12] A. Mayence, J. R. Navarro, Y. Ma, O. Terasaki, L. Bergstrom and P. Oleynikov, Identification and Structure Solution by Three-Dimensional Electron Diffraction Tomography: Gd-Phosphate Nanorods, *Inorg. Chem.* 53(2014)5067-5072.
- [13] M.T. Colomer, J. Mosa, Thermal evolution, second phases, and sintering behavior of $\text{LaPO}_4 \cdot n\text{H}_2\text{O}$ nanorods prepared by two different chemical synthesis routes, *Ceram. Int.* 41(2015)8080-8092.
- [14] Nuria O. Nuñez, Sonia R. Liviano, Manuel Ocaña, Citrate mediated synthesis of uniform monazite LnPO_4 ($\text{Ln} = \text{La}, \text{Ce}$) and $\text{Ln}:\text{LaPO}_4$ ($\text{Ln} = \text{Eu}, \text{Ce}, \text{Ce} + \text{Tb}$) spheres and their photoluminescence, *J Colloid Interf. Sci.* 349(2010)484-491.
- [15] S. Rodriguez-Liviano, A. I. Becerro, D. Alcántara, V. Grazú, J. M. De la Fuente and M. Ocaña, Synthesis and Properties of Multifunctional Tetragonal $\text{Eu}:\text{GdPO}_4$ Nanocubes for Optical and Magnetic Resonance Imaging Applications, *Inorg. Chem.* 52(2012)647-654.
- [16] Yurong Shi, Yuhua Wang, Dan Wang, Bitao Liu, Yanhui Li, and Lan Wei, Synthesis of Hexagonal Prism ($\text{La}, \text{Ce}, \text{Tb}$) PO_4 Phosphors by Precipitation Method, *Cryst. Growth. Des.* 12(2012)1785-1791.
- [17] R. Lacomba-Perales, D. Errandonea, Y. Meng, and M. Bettinelli, High-pressure stability and compressibility of APO_4 ($\text{A} = \text{La}, \text{Nd}, \text{Eu}, \text{Gd}, \text{Er}, \text{and Y}$) orthophosphates: An x-ray diffraction study using synchrotron radiation, *Phys. Rev. B.* 81(2010)064113(1)-9
- [18] Q. Du, Z. Huang, Z. Wu, X. Meng, G. Yin, F. Gao, L. Wang, Facile preparation and bifunctional imaging of Eu-doped GdPO_4 nanorods with MRI and cellular luminescence, *Dalton T.* 44(2015)3934-40.
- [19] W. Ren, G. Tian, L. Zhou, W. Yin, L. Yan, S. Jin, Y. Zu, S. Li, Z. Gu and Y. Zhao, Lanthanide ion-doped GdPO_4 nanorods with dual-modal bio-optical and magnetic resonance imaging properties, *Nanoscale.* 4(2012)3754-3760.
- [20] A. I. Becerro, S. Rodriguez-Liviano, A. J. Fernandez-Carrion and M. Ocaña, A Novel 3D Architecture of GdPO_4 Nanophosphors: Multicolored and White Light Emission, *Cryst. Growth Des.* 13(2013)526-535.
- [21] J. Dai, G. C. Li, M. Lv, R. S. Zhou, X. Li, Hydrothermal Synthesis and Luminescent Properties of Spherical $\text{LaPO}_4:\text{Ln}^{3+}$ ($\text{Ln}^{3+} = \text{Eu}^{3+}, \text{Dy}^{3+}, \text{Ce}^{3+}, \text{Tb}^{3+}$), *Adv. Mat. Res.* 1120-1121(2015)396-400.
- [22] G.A. Kumar, M. Kailasnath, Vittal Shenoy, C. Santhosh, D.K. Sardar, Spectroscopic characterizations of Er doped LaPO_4 submicron phosphors prepared by homogeneous precipitation method, *N. Saltmarsh, Opt. Mat.* 53(2016)24-29.

- [23] M.A. Hassairi, A. Garrido Hernández, T. Kallel, M. Dammak, D. Zambon, G. Chadeyron, A. Potdevin, D. Boyer, R. Mahiou, Spectroscopic properties and Judd–Ofelt analysis of Eu doped GdPO_4 nanoparticles and nanowires, *J. Lumin.* 170(2016)200-206.
- [24] Ming-Guo Ma, Fu Deng, Ke Yao, Solvothermal synthesis and characterization of LaPO_4 with hierarchical nanostructure in mixed solvents of ethanol and water, *Mat. Lett.* 124(2014)173-176.
- [25] G. Kumar, M. Pokhrel and D. Sardar, Absolute quantum yield measurements in Yb/Ho doped $\text{M}_2\text{O}_2\text{S}$ ($\text{M}=\text{Y}, \text{Gd}, \text{La}$) upconversion phosphor, *Mat. Lett.* 98(2013)63-66.
- [26] Y. Ni, J.M. Hughes, A.N. Mariano, Crystal chemistry of the monazite and xenotime structures, *Am. Mineral.* 80(1995)21-26.
- [27] Mei Chee Tan, G. A. Kumar, Richard E. Riman, M. G. Brik, E. Brown, and U. Hommerich, Synthesis and optical characterizations of infrared emitting $\text{YF}_3\text{:Nd}$ nanoparticles, *J. Appl. Phys.* 106(2009)0063118-1-0063118-12.
- [28] D. Mendoza, F. Morales, R. Escudero and J. Walter, Magnetization studies in quasi two dimensional palladium nanoparticles encapsulated in a graphite host, *J Phys-Condens Mat.* 11(1999)L317-L322.

Fig. captions

Fig. 1. Unit cell structure of GdPO_4 showing the location of Gd (light green), P (orange) and O atoms (red).

Fig. 2. XRD pattern of the as-prepared and heat treated $\text{GdPO}_4:\text{Nd}^{3+}$ nanoparticles in comparison with monoclinic standard JCPDS No. 01-083-0657 shown as vertical lines.

Fig. 3. SEM image of (a) as-prepared, (b) heat treated $\text{GdPO}_4:\text{Nd}^{3+}$ particle. Fig. 3(c) shows a close-up view of the star shaped particle.

Fig. 4. Optical absorption spectrum of 1 mol% Nd^{3+} doped GdPO_4 ceramic. Inset shows the photograph of the semi-transparent ceramic pellet.

Fig. 5. Comparison of the emission spectra of the as-prepared and heat treated $\text{GdPO}_4:\text{Nd}^{3+}$ particles.

Fig. 6. FTIR spectrum of $\text{GdPO}_4:\text{Nd}^{3+}$ indicating the band frequencies of various vibrational groups. AP-As prepare, HT-Heat treated.

Fig. 7. Fluorescence decay curves of 1064 nm emission with Nd^{3+} concentrations.

Fig. 8. Quantum yield of 1064 nm emission as a function of Nd^{3+} concentrations.

Fig. 9. Room temperature and low temperature magnetization curve of $\text{GdPO}_4:\text{Nd}^{3+}$.

Fig. 10. Zero field cooling curve of $\text{GdPO}_4:\text{Nd}^{3+}$ at 100 Oe.

Figures

Fig. 1. Unit cell structure of GdPO₄ showing the location of Gd(light green), P(orange) and O atoms(red).

Fig. 2. XRD pattern of the as prepared and heat treated $\text{GdPO}_4:\text{Nd}^{3+}$ particles in comparison with monoclinic standard JCPDS No. 01-083-0657 shown as vertical lines.

(a)

(b)

(c)

Fig.3. SEM image of (a) as prepared, (b) heat treated $\text{GdPO}_4: \text{Nd}^{3+}$ particle. Figure 3(c) shows a close-up view of the sub-micro-star shaped particle.

Fig.4. Optical absorption spectrum of 1 mol% Nd³⁺ doped GdPO₄ ceramic. Inset shows the photograph of the semi-transparent ceramic pellet.

Fig.5. Comparison of the emission spectra of the as prepared and heat treated GdPO₄:Nd³⁺ particles.

Fig.6.FTIR spectrum of $\text{GdPO}_4:\text{Nd}^{3+}$ indicating the band frequencies of various vibrational groups. AP-as prepared, HT-heat treated.

Fig.7. Fluorescence decay curves of 1064 nm emission with Nd³⁺ concentrations.

Fig.8. Quantum yield of 1064 nm emission as a function of Nd^{3+} concentrations.

Fig.9. Room temperature and low temperature magnetization curve of $\text{GdPO}_4:\text{Nd}^{3+}$.

Fig.10. Zero field cooling curve of GdPO₄:Nd³⁺ at 100 Oe.