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A Study of Solid-State Lighting of Nano Phosphors with Photoluminescence Transition Metal

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Abstract - As discussed in detail, lanthanide-based luminescent materials are known to be better candidates for w-LED applications. They are prepared either by complexing Ln3+ ions with suitable organic ligands or by doping Ln3+ in an appropriate inorganic lattice having different dimensions. Luminescent properties of such materials depend on their synthesis methods, composition and more importantly on the structure as well as chemical bonding. Therefore, basic information on the structure is necessary beforehand to provide a better overview of the property and features. Perovskiteoxide materials have deep structural flexibility regarding the components, covering the majority of the periodic table, and have predictable relationships between structure and property. Like phenotypes, most perovskite oxide matrices in the blue region of a visible spectrum have their characteristic emission peaks. Peruvian oxide phosphoruses also have operation compatibility at far higher temperatures and atmospheres of oxidation. The maximum number of existing transitions from deep ultraviolet to midinfrarot has shown that the luminescent properties of phosphorus with trivalent rare earth ions make those appropriate candidates for lamp phosphorous and device display applications. It is difficult to synthesize these compounds bulkily or nanosely with the desired features. A number of soft-chemical methods have therefore been used in this work in addition to the conventional solid-state synthesis.

Keywords - Solid-State Lighting, Nano Phosphors, Photoluminescence Transition Metal, w-LED applications, synthesis methods

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INTRODUCTION

As a general remark, fundamental and applicative research efforts to improve performances of WL LED sources are currently mainly devoted to nanophosphors (i.e., Nano-sized phosphors) due to changes of several properties related to scaled-down size and opportunities of engineered functionalities for the desired application. For instance, an increasing trend to miniaturization favored by the progress in nanotechnology and the development of Nano-medicine have prompted interest in Nano phosphors as luminescent markers for imaging in medical diagnosis and therapy as well as multiplexed biological labeling. Such applications take advantage from up-conversion luminescence, large anti-Stokes shifts (up to 500 nm), excellent photo-stability, high luminescence quantum efficiency, long luminescence lifetime, narrow emission lines, high color purity, and removal of UV excitation-induced photo-damage to biological samples, quantum cutting, and absence of photo-bleaching and photo-blinking in Nano phosphors. It is worth stressing that the peculiar spectroscopy of the Nano phosphors does not result from quantum size effects which dominate the emission spectra of semiconductor quantum dots. Instead, the active role is played by size-scale, doping, site symmetry, phase of the crystalline matrix, dopant-ligand distance and strength of the coordination, as well as influence of the surfaces. In this review paper, we overview the spectroscopy of the Nano phosphors with particular attention to the effects of spatial confinement and surface-to-volume ratio on their emission performances in terms of electron-phonon dynamics, luminescence quenching, confinement of dopants, and tuning of multicolor emission for the generation of WL emission and dynamical characteristics (emission decay and rise time, line broadening and line shift). Moreover, we present and discuss the observed occurrence of unconventional WL emission, where “unconventional” refers to a new class of efficient broadband white-light-emitting Nano phosphors consisting of nominally un-doped rare earth (RE)-free oxide Nano powders and transition-metal (TM)-doped nanoscale hosts excited by monochromatic infrared-excitation. By comparison with the literature, the actually available strategies to obtain WL emission mainly consider RE dopants and single-phase compounds containing REs as stoichiometric components.

Since a huge amount of literature is already available dealing with the general properties of phosphors and their spectroscopy, the main intent of this review paper is not to provide a didactic overview of the field again. Instead, we overview the physics of Nano phosphors, which is a fascinating and still not completely understood research field, and introduce the reader to a very recent unconventional phenomenon in the field of WL emission. In this spirit, the presented discussion and examples aim at summarizing the existing knowledge in such a way to point out the main differences, achievements, and challenges in the field of Nano phosphors as compared to bulk phosphors. While keeping in mind this purpose and in order to make the readership of this review contribution open to non-specialist readers, we clearly indicate nomenclature and provide self-consistent knowledge of basic concepts, definitions, and spectroscopic mechanisms mentioned throughout the paper. For more informative fundamental knowledge, we suggest a preliminary reading of Reference to the reader new to the field of the spectroscopy of phosphors.

SPECTROSCOPY OF NANOPHOSPHORS

Spectroscopy of Nano phosphors is currently of worldwide interest, in the fundamental and applicative fields, favored by both the progress in the nanofabrication field and experimental observation pointing out changes of the general properties of lanthanide-activated phosphors introduced by nanoscale resizing. Chemical and physical techniques, such as, sol−gel, combustion, micro-emulsion, co-precipitation, laser ablation, ultrasonic spray pyrolysis, and template-based routes, are currently used for the synthesis of Nano phosphors with control on size and shape, chemical composition, phase, doping amount, surface passivation, and functionalization steps. Early experimental evidences of size-dependent spectroscopy of nanophosphors were remarkably increased photoluminescence quantum efficiency with high radiative efficiency (~20%) and luminescence lifetime enhanced of more than 5 orders of magnitude (ns versus ms) with respect to the bulk counterpart crystals in the case of ZnS nanocrystals doped with Mn (dopant content ranging from 1% for crystal size of 70 ´Å to nearly 18% for crystal size of 3.5 ´Å). Nanoscale-related emission enhancement and tunability were also reported for 20 nm size yttrium oxide (Y2O3) nanoparticles co-doped with Yb and Er ions and for decreasing particle size (from 55 to 13 nm) due to the small Bohr radius of the exciton. Enhanced emission efficiency can result from the combined effect between the energy levels of lanthanides and dopant-induced symmetry breaking of the host lattice favored by surface lowered coordination too. Moreover, improved efficiency of up-conversion transitions was obtained in hexagonal and cubic NaREF4 (RE = Y, Pr to Lu) nanocrystals and NaYF4 nanoparticles co-doped with Yb, Er, and Tm by changing the shapes (polyhedra-, rod-, plate-, and dot-like). As previously outlined, the characteristic emission lines of RE elements are strictly related to their peculiar electronic configuration with the valence electrons 4f shielded from interactions with the host crystal lattice and ligands by means of outer shells. Unlike RE elements, the unfilled outermost more extended and delocalized d-orbitals of TMs, hybridize with ligand orbitals leading to splitting of the atomic (unperturbed) levels in the host crystal field. Hence, while the energy levels of the TM dopants are automatically influenced by the host crystals at any size-scale, spatial confinement has poor effects on RE-characteristic emissions stemming from intra-configurational f-f transitions. On the other hand, as the emission features of REs can be due to d-f transitions too, host-dependent energy-shifts of the RE emissions can be observed. Since the excited states of RE elements can have L–S coupling and J mixing, a single 2S+1LJ multiplet can describe the excited states of REs sensitive to the host crystal and yield host-dependent emission of RE centers. Therefore, in Nano phosphors, dopant-ligand distance and site symmetry may influence the emission from the excited states of RE dopants due to modified J–L coupling and J mixing effects with respect to the lower energy levels. Moreover, changes in the emission efficiency can be induced by dopant-related symmetry breaking in the host material, that can be more effective because of site distortion favored by both the choice of dopant and surface lowered coordination.

1. Tuning of the Color Output

For lighting applications of Nano phosphors aiming at a simplified device design, broad range tunability of the color output and multicolor emission by single wavelength excitation are the main applicative purposes. In order to be able to implement this simple scheme, RE elements are key components due to their rich energy level diagram. As Figure 1shows, multicolor output can be obtained by exciting different f-f transitions of the same RE element as well as by combining the emission from different RE elements.

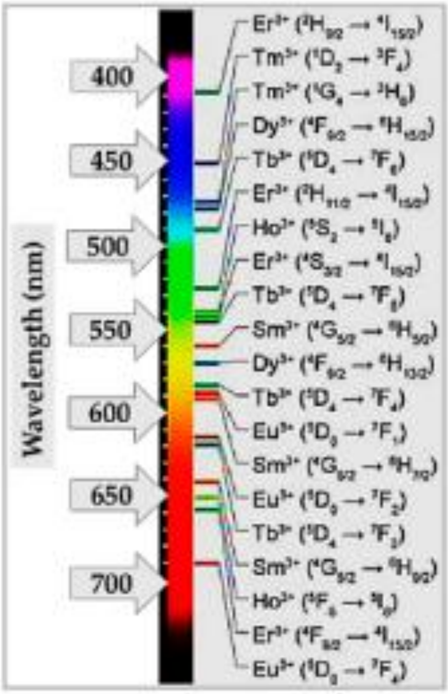


Figure 1. Diagram of the color output resulting from the main f-f transitions of RE elements

The achievement of multi-peak tunable emission from Nano phosphors requires proper choice and balance of dopant species and dopant−dopant interaction as well as suitable host material and site symmetry due to the influence of lattice vibrations and crystal field on the radiative transitions of the dopants. A single dopant emitter may allow the production of WL emission based on tuning of the intensity ratio between two different emission colors. Alternatively, different dopant(s)–host combinations can yield multiple color emissions by means of balanced spectral overlap and relative ratio of different emission colors. Also, luminescence may be phase-dependent through the site symmetry of dopants in different host lattices and dopant–dopant interaction in multiple lattice sites.

2. The Role of the Dopant Content

The dependence of the output light on the dopant amount is a way to tune multicolor emission and the relative emission intensity. For instance, in the case of NaYF4 nanocrystals doped with Yb, Tm, and Er, for a concentration ratio Yb3+/Tm3+ set to 20/0.2 mol %, the color output can be changed from blue to white by means of Er3+ content increased from 0.2 to 1.5 mol %. Under excitation with a 980 nm wavelength, LiYF4 nanocrystals co-doped with Yb3+, Tm3+ (0.5%), and Er 3+ (0.1%, 0.2%, 0.3%, 0.5%) exhibit blue and green emission with green-to-blue ratio of the photoluminescence intensity dependent on the Er3+ concentrations. Yttrium oxide nanoparticles co-doped with Er3+, Tm3+, and Ho3+ ions, show red-to-green emission ratio changing versus particle size and up-conversion output color tunable from blue to red upon changes of the dopant amount. In cubic phase α-NaYF4 nanocrystals doped with high concentrations of Tm3+ and Yb3+ ions, energy transfer processes from Er3+ to Yb3+ impact on the relative intensities of the blue, green, and red emissions from Er3+, leading to color output tunable from yellow to red for Yb3+ amounts increased from 25 to 60 mol %.

3. The Role of Surfaces

Other effects accounting for the differences in the emission issues between bulk and nanoscale phosphors are surface-to-volume ratio, surface-related defects, and disordering. In general, the surface-to-volume ratio influences the fraction of the weakly coordinated surface dopant ions versus the strongly coordinated inner dopant ions. Also the contribution of defects, contaminants, and solvent/surfactants in wet chemical synthesis approaches has to be taken into account for reducing the size. Energy transfer being dependent on the ion–ion spacing and the critical distance for observing energy transfer being of the order of several nanometers, confinement and dopant amounts are expected to impact on the control of energy transfer in nanophosphors. For instance, in NaYF4 nanocrystals co-doped with Yb3+ (20 mol %) and Er3+ (2 mol %), 980 nm excitation energy is effectively absorbed by the Yb3+ ions and transferred to the nearby Er3+ ions that exhibit three emission bands (525, 542, and 655 nm) due to multiphonon relaxation processes bridging different excited states. Both total emission intensity and the green-to-red intensity ratio increase for nanocrystal size increasing above 50 nm because of a decreased quenching of the up-conversion emission from surface defects and ligands. In turn, red and green color emissions from weakly coordinated surface dopants play a dominant role for nanocrystal size scaled down to a few nanometers (ultra-small nanophosphors). On this basis and for biomolecule labeling purposes, synthesis approaches of nanophosphors need to control structural order, phase, phase purity, dopant content, structure (core-shell), and size for decreasing size down to tens of nanometers. In order to circumvent luminescence quenching due to surface quenching for decreasing size, confinement of dopants by a core-inert shell architecture is a valid strategy that limits the contribution of surface quenching species, enables better stability in water media and reduced fluorescence quenching due to adsorbed hydroxyl species, suppresses cross-relaxation and energy-transfer dopant–dopant interactions, as well as enhances the up-conversion efficiency. For example, sub-10 nm-sized LiYF4 nanocrystals co-doped with Yb3+-Tm3+ pairs or Yb3+-Er3+ pairs showed enhanced photoluminescence and color emission (bright blue, sky blue, aqua, aquamarine, and green) tunable by dopant amount when encapsulated by an outer LiGdF4 shell. Luminescence enhanced up to 30 times was found in the case of 8 nm NaYF4 nanocrystals, co-doped with Yb3+ and Tm3+ ions, after their embedding by a 1.5 nm thick NaYF4 shell. For a NaGdF4 matrix having the core doped with

Yb3+-Tm3+ pairs and the shell doped with Eu3+, excitation by 980 nm wavelength induces multi-step energy transfer from core to shell (through the transitions Yb3+ (core) → Tm3+ (core) → Gd3+ (core and shell) → Eu3+ (shell)) mediated by Gd3+ and strong red emission from Eu3+ results. Notably, in the absence of the core-shell architecture, the coupling among the different ions quenches the emission and up-conversion of Eu3+ ions. Modulation of the up-conversion emission was also demonstrated in core-shell designs under pulsed laser pumping that controls the energy transfer between dopants through pulse duration.

4. Phonon Modes and Excited State Dynamics

At the nanoscale regime, the density of phonon states changes from a continuous (Debye approximation) to a discrete distribution, exhibits a size-dependent cutoff frequency for phonon modes and is unchanged for high frequencies phonon modes. Whenever the low-energy phonon modes are absent, complete direct phonon relaxation between the levels with an energy gap less than the cut-off phonon frequency is completely suppressed. Hence, whereas spatial confinement has poor impact on the non-Radiative decays yielding the emission of high-frequency phonons, less non-Radiative relaxation channels result from a lowered number of allowed phonon modes: This means that only non-radiative relaxation involving electronic states with energy gaps close to or higher than the Debye energy of the lattice are size-independent. Therefore, in general, the electron–phonon interaction is size-dependent. Thermal relaxation is size-dependent too. Indeed, as a consequence of the low energy phonon modes being discrete, following excitation thermal equilibrium of nanoparticles may be inhibited because of no low-energy phonon state available to non-radiatively depopulate an excited level of an emitting center. Hence, a level that in bulk material would fast relax, at the nanometer-scale retains its population long enough to decay radiatively rather than by the one-phonon decay. The size-dependent electron–phonon interaction also influences energy transfer dynamics because energy transfer occurs by migration of electronic excitation between sensitizer-activator by resonant processes and/or by phonon-assisted transfer. On the basis of the energy transfer mechanism, as the doping content increases, the reduced average ion–ion spacing implies increased probability per unit time of energy transfer. Turning to the nanoscale-regime, since the probability that the donor can find nearby acceptors decreases, the numbers of acceptors and donors get smaller and defect sites act as traps, negatively impacting on the efficiency of energy migration across dopant sites. Indeed, in a nanoparticle, core and surface atoms exhibit different coordination and symmetry of the surroundings, meaning that distorted surface bonds introduce defects and energy levels shifting as compared to the ones of the same core counterpart. This condition of out of resonance between energy levels of core and surface ions implies it requires emission or absorption of at least one phonon to bridge the energy difference. Another important consequence of the size confinement of lanthanide dopants is the change of the excited state dynamics (emission decay and rise time, line broadening and line shift). Electron–phonon coupling plays a role in affecting the width of emission lines in nanophosphors.

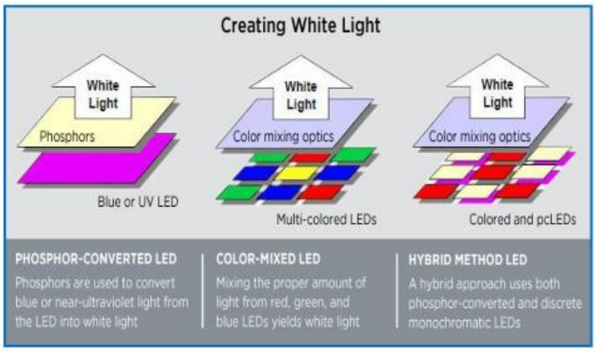
WHITE-LIGHT EMITTING DIODES (W-LEDs)

White LEDs, in contrast to traditional LEDs, provide light that is polychromatic rather than monochromatic. White LEDs' ultimate goal is to replace incandescent and fluorescent bulbs for general illumination. White LEDs, on the other hand, are seen as a promising alternative to regular LEDs. White LEDs having better features to those of traditional light sources, such as high luminous efficiency, tunable colour temperatures, good colour rendering properties, and long lifetime, are always being sought for.. General illumination in homes and workplaces, street lighting, vehicle lighting, and backlighting technologies are all provided by this manufacturer. This yellow-emitting Yttrium Aluminum Garnet (YAG: Ce)-doped Cerium-doped LED chip was used to create the first white LED.

Different Approaches of Generating Solid State White Led Devices

Using three diodes that emit red, green, and blue light, or a near-UV LED that excite several phosphors that emit over the entire spectral range, solid-state white light can be generated. The third, most widely used alternative involves down-conversion of some of the blue LED light to longer wavelengths in such a way that white light is generated. Figure 2 illustrates two major approaches for creating white light with LEDs.

- Multi-LED chip approach, in which light coming from three monochromatic red, green and blue (RGB) LEDs are mixed, was resulting in white light to human eyes.
- Phosphor conversion, in which a GaN-based blue or UV LED chip is coated with a yellow or metachromatic phosphor.



PHOSPHOR-CONVERTED LED
Phosphors are used to convert blue or near-ultraviolet light from the LED into white light

COLOR-MIXED LED
Mixing the proper amount of light from red, green, and blue LEDs yields white light

HYBRID METHOD LED
A hybrid approach uses both phosphor-converted and discrete monochromatic LEDs

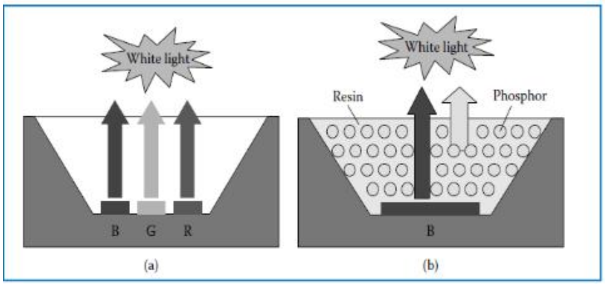
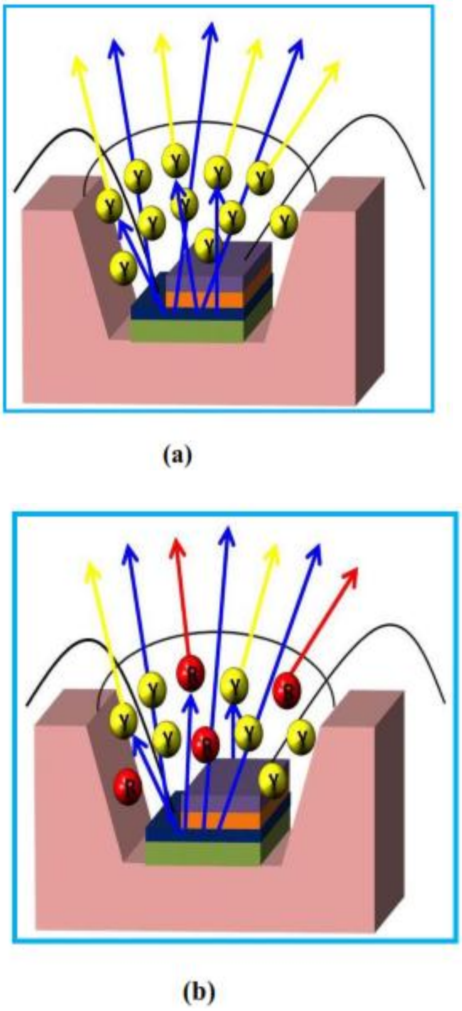
- 

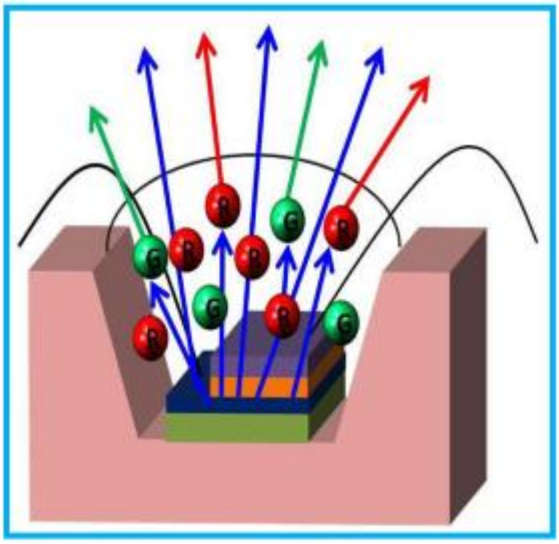
Figure 2 White light produced by (a) multi-LED chip and (b) phosphor conversion approaches (where RGB stands for red, green, and blue primary LEDs)

Human eyes see the combination of phosphor and LED chip light as white. The most common and simplest phosphor-converted white LED is a one-phosphor-converted (1-pc) white LED, which consists of a yellow-emitting phosphor and a blue LED chip. Color rendering index (Ra) of the 1-pc white LED is so low (Ra 80) because of the phosphor's comparatively narrow emission spectrum. As a result, it cannot be used as a general light source. In addition, the lack of red colour in the phosphor results in high colour temperatures (cold/blue light) in the 1-pc white LED, which often does not allow colour temperature tunability. Several phosphors (such as red and green phosphors) are coupled to a blue LED to "fill in" the gap between blue and yellow light while strengthening the red half of the spectrum, resulting in an LED with great colour rendition and variable temperature. Pumping an RGB phosphor blend with a UV or near-UV LED chip can also produce white LEDs with high colour rendering. Down converting the wavelength of the LED chip into a suitable visible light and managing light quality of white LEDs are fundamental functions of phosphor-converted white LEDs. Because of this, down conversion phosphors are an essential component of white LED technology.

Principle of White Light Generation in LEDs

For the first commercially accessible white LED, a blue LED chip was combined with a yellow phosphor made from $Y_3Al_5O_{12}:Ce^{3+}$ (YAG:Ce^{3+}). YAG-based w-LEDs have a poor colour rendering and heat quenching of luminosity as its main shortcomings. There are other approaches, such as using InGaN diodes with nUV/UV phosphors and red, green, and blue phosphors to produce white light (recommended). Most commercially available white LEDs are made by pre-coating phosphors onto blue diode chips, because the luminous efficiency of blue chip-based white LEDs is significantly superior to that of nUV/UV type. Figure 3 illustrates the three various ways in which LEDs can produce white light, based on the phosphors they use. Figure 3 (a) shows a combination of blue LED diodes and yellow phosphor particles, which is highly efficient but has a poor colour rendering index (CRI) (Ra).





(c)

Figure 3 (a) A blue LED + Yellow phosphors (b) A blue LED + Yellow phosphors + Red phosphors (c) A blue LED + Green phosphors + Red phosphors

Figure 3 (b) illustrates the second method, which is to add the right red phosphor to the first type, so that the Ra may be improved and the luminous efficiency is appropriate. It is the third type from figure.3 (c) that is made by mixing a blue LED diode chip with red and green phosphors, which have a high Ra but a poor efficiency. Due to the constant production of heat, the stability of LED luminescence is largely dependent on the phosphors that are used. w-LEDs have been recommended as a replacement for conventional light sources because of their promise of large energy savings and a reduction in carbon emissions.. This means that in terms of general illumination, the most essential qualities of white LEDs are luminous efficacy and colour rendering, which are identical to those of bulb and tube lamps. Color temperature and chromaticity coordinates are also significant for white LEDs.

NANOSCALE-RELATED UNCONVENTIONAL PRODUCTION OF WHITE LIGHT

Among designs of WL-emitting LEDs, the most common ones exploit a single-phase host material: (1) singly doped with a lanthanide ion having a complex energy level structure able to emit multiple colors (for instance, Sm3+, Eu3+, or Dy3+), (2) co-doped with red, green, and blue emitting or yellow and blue emitting RE ions, (3) including ion–ion pairs (for instance, Ce3+/Mn2+, Ce3+/Tb3+, Ce3+/Ho3+, Tb3+/Sm3+, etc.) participating in energy transfer processes, (4) triply-doped with ions emitting different colors (Ce3+/Li+/Mn2+, Eu3+/Tb3+/Tm3+, Eu2+/Tb3+/Eu3+). A survey of the literature enabled the observation that WL emission results from materials at least doubly-doped with different RE ions or including RE ions as stoichiometric components. For instance:

- Nano-sized compounds incorporating Nd as a dopant or a full stoichiometric component emit anti-Stokes wide band emission,
- Yb-doped: Y3Al5O12 (YAG) nano-crystalline ceramics emit bright anti-Stokes WL,
- Anti-Stokes WL emission is yielded by YAG (Yb3Al5O12) and (Yb,Y)2O3 nano-powders, LiYbF4 nanocrystals, (Yb3+,Ln3+, Tm3+, or Ho3+)-doped YVO4 powders, un-doped and Er-doped LiYbP4O12 nanocrystals,
- Under near-infrared excitation excitation in vacuum, single compound lanthanide oxides (Yb2O3, Sm2O3, CeO2) and Yb3Al5O12, (Yb,Y)2O3 crystals emit WL by up-conversion mechanisms,
- Er2O3 emits up-conversion luminescence by band-to-band multiphoton excitation under vacuum and near-IR excitation,
- bright up-conversion WL is emitted by Ln-doped LaF3 nanoparticles and LaF3 nanoparticles co-doped with Tm3+, Tb3+, and Eu 3+,
- White LEDs were designed based on oxynitride or nitride phosphors doped with RE elements,
- Luminescence with high quantum efficiency values, comparable to conventional crystalline phosphors, was observed under excitation with deep-UV light of RE-free transparent phosphate glasses,
- a red phosphor to be used as a high-power warm WL-emitting LED was obtained by a RE-free approach based on Mn4+ and Mg2+ doped BaMgAl10O17.

Alternatively, under monochromatic IR-excitation, first WL broadband emission was observed from a nominally un-doped oxide nanomaterial (i.e., in the absence of luminescent RE centers as main components) and in the presence of a TM-dopant independently on the temperature. To detail, WL emission was demonstrated in the following nanoscale-sized materials:

- Cr-doped Gd3Ga5O12 (GGG) nano-powders excited by a continuous wave (CW) laser diode emitting at 803.5 nm,
- Nominally un-doped yttrium oxide (Y2O3) and Nd-doped (up to 20%) Y2O3 nano-powders excited by a CW laser diode emitting at 803.5 and 975nm,
- yttrium silicate (γ-Y2Si2O7) and Yb3+/Er3+/Tm3+ triply-doped Y2Si2O7 nano-powders excited by a CW laser diode emitting at 975 nm,
- Erbium oxide (Er2O3) nano-powders excited by a laser diode emitting at 800 and 975 nm.

CONCLUSION

The materials that have a direct or indirect impact on the advancement of current technology or the development of new technology are considered to be technologically relevant. Improved performance, increased activity, or changes to the overall behaviour of a gadget can all be used to attain this goal. Transition metal ions and trivalent rare earth ions (lanthanides) have been used as luminous centres (dopants) in various host matrices to make phosphors for solid state lighting applications, which have resulted in significant progress in the field of phosphors in recent years. For the most part, phosphors are comprised of a host material such as a sulphide, phosphate, aluminate, oxide, molybdate, tungstate, or silicate with or without an appropriate dopant (transition metal ions and lanthanides). The dopant ions are housed in an insulator or wide bandgap semiconductor in the luminous host lattice. Some of the luminous phosphors' physical and chemical behaviours, such as emission intensity and lifetime qualities, are also controlled by the host substance. A few ppm to a few moles of the host lattice are typical concentrations for the dopant or activator ions in most situations. We've addressed in depth the advantages of using lanthanide-based luminescent materials in W-LED applications. Inorganic lattices with varied dimensions can either be doped with Ln3+ ions or complexed with organic ligands. It is the synthesis technique, composition, and most crucially, the structure and chemical bonding of these materials that determines their luminescent capabilities. Phosphors with trivalent rare-earth ions show the largest number of transitions extending from deep ultraviolet to the mid-infrared, making them suitable for lamp phosphors and device displays. With the requisite qualities, the synthesis of such compounds in bulk or Nano form is a difficult undertaking. The development of effective blue light producing luminescent nanophosphors for electroluminescence and field emission display device applications has received substantial interest recently. Engineers studied a variety of new blue phosphor systems, including CaWO4, ZnGa2O4, and ZnGa2O4 containing Europium ions, in an effort to produce new, more durable materials for use in electronic displays. Double perovskite-structured phosphors like Ca3WO6 emit in the visible range of the spectrum. Consequently, it is envisaged that the synthesis technique can be modified appropriately to yield efficient luminous materials that can emit in three colours by doping them with Dy3+ and Eu3+ ions. The luminous properties of Ca3WO6 phosphor materials were investigated with this in mind throughout the duration of the current work."

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