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Editor-in-Chief
Daniel James



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LUMINESCENCE INVESTIGATION OF PHOSPHOR AND ITS PREPARATION TECHNIQUES

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INTRODUCTION

In modern urban life, luminescence has become an indispensable part of our daily existence, making it difficult to envision life without it. This reliance stems from the widespread use of various lighting devices, including lamps, televisions, mobile screens, LED lights, LED televisions, signals, and displays. Light, as a form of electromagnetic energy, requires another energy source for its creation. The two primary mechanisms for generating light are incandescence and luminescence.

Incandescence refers to light produced by heat energy. When certain materials are heated to sufficiently high temperatures, they begin to emit light. For example, electric heaters produce light through electric current, and metals can appear "red hot" in flames due to incandescence. In traditional incandescent light bulbs, tungsten filaments glow "white hot" by this same principle.

On the other hand, luminescence is the spontaneous emission of light from a substance that does not stem from heat. It represents a form of cold body radiation, which can occur due to chemical reactions, electrical energy, subatomic motions, or stress on a crystal. Unlike incandescence, luminescence, or "cold light," can occur at normal and lower temperatures. The term "luminescence" was officially introduced by the German physicist Eilhardt Wiedemann in 1888, derived from the Latin word "lumen," meaning "light." Substances that exhibit luminescence are referred to as "luminescent materials" or "phosphors," a term meaning "light bearer" in Greek, first coined by the Italian alchemist Vincentinus Casciarolo in the 17th century. Casciarolo discovered a glowing stone, likely barite (BaSO_4), which emitted red light in the dark after being exposed to sunlight, although he was originally seeking to transform it into a noble metal [1].

In 1866, Theodore Sidot synthesized zinc sulfide (ZnS), which became the prototype for phosphors used in contemporary cathode ray tubes. Between the late 19th and early 20th centuries in Germany, Philip E.A. Lenard and his colleagues explored various phosphors, incorporating different rare earth ions along with heavy metal ions into various host materials [2].

The process of luminescence involves an energy source exciting an electron in an atom from its "ground" (lowest energy) state to an "excited" (higher energy) state. The electron then releases the energy as visible light, allowing it to return to its ground state. Luminescence can be observed in natural phenomena such as glowworms, fireflies, and certain marine bacteria and deep-sea creatures. This phenomenon has been utilized across many fields including archaeology, geology, biomedical engineering, chemistry, physics, and various industrial applications for quality control and research and development [3].

PHOTOLUMINESCENCE

The emission of light occurs due to excitation by electromagnetic radiation or photons. This term encompasses both fluorescence and phosphorescence, making it somewhat broad. Photoluminescence has a wide range of applications, from whitening agents in laundry detergents to plasma screens for large displays.

A particular form of luminescence, characterized by a very slow decay, continues to emit light for minutes or even hours. This phenomenon is known as long-lasting or persistent luminescence, and it is often utilized in road safety applications and exit signage.

Luminescence is defined as the emission of photons when a solid absorbs energy, and this phenomenon occurs when certain forms of energy excite the material. When solids are stimulated by short-wavelength light, often ultraviolet (UV) radiation, this process is referred to as photoluminescence. Photoluminescence can be divided into two main types: intrinsic and extrinsic luminescence [4, 5].

INTRINSIC LUMINESCENCE

As the term suggests, intrinsic luminescence originates from within a pure material or crystal and can be categorized into three types:

- **Band-to-Band Luminescence**

This luminescence results from the recombination of an electron from the conduction band with a hole in the valence band, leading to a band-to-band transition. It typically occurs in very pure materials at elevated temperatures, while at lower temperatures, it may convert to exciton luminescence. Examples of materials exhibiting this behavior include silicon (Si), germanium (Ge), and certain III-V compounds like gallium arsenide (GaAs) [2, 5].

- **Cross-Luminescence**

Cross-luminescence occurs when an electron from the valence band recombines with a hole generated in the outermost core band. This type of luminescence is commonly observed in alkali and alkaline-earth halides, as well as in double halides [5].

- **Exciton Luminescence**

Exciton luminescence involves a bound pair of an excited electron and a hole, known as an exciton. As the exciton travels through the crystal, it carries energy, and the recombination of the electron and hole results in luminescence. There are two types of excitons: the Wannier exciton, which consists of an electron in the conduction band and a hole in the valence band held together by Coulomb interaction, typically found in III-V and II-VI inorganic semiconductors; and the Frenkel exciton, which occurs when the spatial extent of the electron and hole wave functions is smaller than the lattice constant, commonly found in organic molecular crystals like anthracene, as well as in inorganic complex salts such as tungstates and vanadates, and in uranyl salts [4, 5].

EXTRINSIC LUMINESCENCE

Extrinsic luminescence arises from the deliberate incorporation of impurities or defects within a phosphor. In ionic crystals and semiconductors, this luminescence can be unlocalized or localized. Unlocalized extrinsic luminescence occurs when free electrons in the conduction band and free holes in the valence band of the host lattice contribute to the luminescent emissions. Conversely, localized extrinsic luminescence happens when the excitation and emission processes are confined to a specific luminescent center.

Photoluminescence is the spontaneous emission of light following electronic excitation, such as excitation by ultraviolet radiation. This luminescence phenomenon is prevalent in both inorganic and organic materials, as well as in semiconductors. However, in some compounds, non-radiative relaxation processes can dominate. When spontaneous light emission does occur, its spectral and temporal characteristics provide critical insights into the metastable emitting state and its connection to the ground state. Therefore, luminescence spectroscopy serves as a valuable method for investigating these properties, allowing for a better understanding of the light emission process and the competing non-radiative photophysical and photochemical processes [6].

Luminescence refers to the emission of optical radiation (infrared, visible, or ultraviolet light) by substances, setting it apart from incandescence, which is the emission of radiation from an object due to its high temperature (black body radiation). Luminescence can occur across a wide range of materials and under various conditions, including atoms, polymers, inorganic and organic molecules, crystals, and amorphous substances, given the right circumstances. Different types of luminescence are named based on the radiation used for excitation and emission.

A key feature of luminescence is that the emitted light is an inherent characteristic of the material itself, with the light emission resulting from some internal or external excitation process. This differs fundamentally from the incandescence of an ordinary light bulb filament, where electrical energy heats the metal atoms of the wire directly, causing them to vibrate and emit heat. The wire then glows white hot, typical of incandescent lighting. This type of light emission produces substantial heat, with electrical energy converting to radiation at approximately 80% efficiency, while the visible light constitutes less than 10% of the total emitted radiation, with the remainder primarily in the form of infrared radiation. The spectrum emitted by a heated object, such as a wire, exhibits characteristics that are largely independent of the specific attributes of the object, as all heated substances emit radiation that closely resembles the behavior of a generic black body [5, 6].

PHOSPHORS

The term "phosphor," derived from the element phosphorus—which emits a glow when exposed to air—refers to materials that essentially consist of an inert, imperfect host crystal lattice infused with intentionally added impurity ions known as dopants. The history of phosphors dates back over a century to 1886, when French chemist Théodore Sidot inadvertently created a prototype of a ZnS-type phosphor. Since that time, research and development in phosphors have seen significant advancements, leading to various inventions and applications.

A crystal lattice is composed of regularly arranged atomic configurations. Crystals are classified into different types based on their symmetries, which determine their invariant properties during translational and rotational operations. These crystals have closely spaced discrete energy levels that combine to form energy bands. Depending on the availability of electrons, these bands create distinct electronic states known as electronic energy bands, which also adhere to the symmetries of the crystals. Within these energy bands, states of lower energy are filled with electrons derived from the bound electrons of the atoms, known as valence bands. Conversely, higher energy bands that are unoccupied by electrons are referred to as conduction bands. Typically, materials with crystal symmetries, such as rock salt or zinc blende, do not possess any electronic states between the top of the valence band and the bottom of the conduction band; this gap is known as the forbidden gap or band gap.

Any deviation from a perfect periodic crystal lattice is considered an imperfection. Common types of imperfections include chemical impurities, vacant lattice sites, and interstitial atoms (atoms located outside of regular lattice positions). Point imperfections are localized at a specific point in the structure, as opposed to line or plane imperfections. Many fundamental physical properties of solids are influenced by imperfections just as much as by the host atoms in the lattice. In some semiconductors, conductivity can be primarily attributed to these imperfections. Even a small concentration of point defects can significantly alter the electrical or optical properties of a solid, making it suitable for various industrial applications, such as solid-state electronic circuits, phosphors for fluorescent lamps and televisions, solid-state lasers, and long-lasting dark vision display devices. The luminescence of crystals is closely linked to the presence of impurities, and the color of many crystals is a result of these imperfections. Overall, the chemical and physical properties of a solid are often governed by its imperfections [7, 8].

PHOSPHOR PREPARATION TECHNIQUES

Phosphor materials are primarily synthesized through high-temperature solid-state reactions or sol-gel processes. In these synthesis methods, a host matrix is created using high-purity starting chemicals, while impurities—known as activators and co-activators—are introduced into the crystal lattice in precise quantities. The activators are mainly responsible for the luminescence, while co-activators can facilitate the effective diffusion of activators into the lattice and may help maintain charge neutrality through compensation mechanisms. The specifics of the synthesis procedure can vary based on the type of phosphor being produced.

SOLID-STATE REACTION

The formation of a phosphor host and the doping process via solid solution is crucial and highly influenced by the reaction temperature and conditions. The purity of the starting chemicals is vital, with typical purities ranging from 99.9% to 99.999%. Minimizing specific contaminants, such as iron (Fe), cobalt (Co), and nickel (Ni), is essential, as these can significantly impair phosphor performance. The required quantities of starting materials are mixed, sometimes with an appropriate flux, and then heated at high temperatures (900–1500°C) in air or in a controlled atmosphere (e.g., nitrogen, carbon, carbon monoxide, or nitrogen with 2–5% hydrogen). The calcination conditions—including firing temperatures, duration, atmosphere, and rates of heating and cooling—are optimized based on empirical results for each specific phosphor. The morphology of the starting chemicals and flux influences the resulting particle size and shape, and low-melting flux materials, such as alkali halides, can help complete the doping at lower temperatures. Detailed methodologies for solid-state reactions are well documented in the literature.

COMBUSTION SYNTHESIS

Combustion synthesis is a method proposed for producing oxide-based phosphors with smaller particle sizes. This technique involves a highly exothermic reaction between organic fuel and metal salts (oxidizers) in an aqueous solution. The reaction starts at low temperatures (around 500°C) and rapidly progresses to completion within minutes, with peak reaction temperatures depending on the molar ratio of fuel to oxidizer.

SOL-GEL TECHNIQUE

The sol-gel process involves the formation of colloidal particles (sols) dispersed in a liquid that can transform into a gel. This gel is characterized by a rigid interconnected network with sub-micrometer pores and polymeric chains averaging microns in length. Sol-gel processes can be classified based on the drying method: standard gels undergo drying above room temperature, aerogels dry at room temperature, and xerogels are processed in a vacuum. The particle size of the final product is influenced by the initial concentration of sols, the gelation and drying process, calcination temperatures, and cooling rates. The sol-gel process offers several advantages over traditional high-temperature solid-state reactions, particularly in the synthesis of fine powders like phosphors. Since all starting materials are mixed at the molecular level within the sol solution, a high degree of homogeneity is achievable. Doping with impurities (activators/co-activators) is straightforward and highly effective. Additionally, the very small pore sizes in properly dried gels facilitate intimate mixing of components, and the high surface area of powders obtained through the sol-gel process allows for lower processing temperatures. Given that phosphor materials are extremely sensitive to impurities—even at parts per billion levels—the low-temperature nature of sol-gel processes helps minimize the risk of cross-contamination. The selection of precursors is critical in this method.

CO-PRECIPIATION METHOD

The co-precipitation method is used to create submicron-sized, spherical-shaped phosphors for various display applications. In this approach, solutions containing the constituents of the phosphor are precipitated by adding appropriate hydroxides, such as ammonia solution or oxalate. After filtering and drying, the resulting precipitates are fired at specified temperatures. Since the starting precipitate consists of very small particles, co-precipitates generally require lower firing temperatures to synthesize the desired phosphors. For instance, small, spherical-shaped (Y, Gd) $\text{BO}_3:\text{Eu}^{3+}$ can be synthesized through this method.

HYDROTHERMAL TECHNIQUE

The hydrothermal method enables the synthesis of oxide- and silicate-based phosphors at low temperatures and high pressures. This technique can be performed in either open or closed systems. In an open system, the solid is directly exposed to reacting gases, which also act as the pressure medium. In the seeded hydrothermal process, the inclusion of a polymeric stabilizer promotes nucleation and crystallization of oxide particles under hydrothermal conditions.

SPRAY PYROLYSIS

Recently, spray pyrolysis has emerged as a method for producing submicron and spherical-shaped phosphors. An ultrasonic spray generator creates fine droplets of the appropriate precursors. Each phosphor particle is of high phase purity since it originates from one droplet in which the constituents are uniformly mixed. The preparative conditions, precursor concentrations, nature of additives, and flow rates dictate the size and morphology of the phosphor particles. As the droplets traverse a reactor at elevated temperatures for a brief duration, they dry, decompose, and crystallize. However, a drawback of this process is the tendency for particles to be hollow, resulting in diminished brightness and lifetime, as hollow phosphors typically exhibit lower thermal and mechanical stability [9].

CONCLUSION

In conclusion, luminescence plays a crucial role in our modern urban life, influencing a wide range of applications from everyday lighting to advanced technological devices. This fascinating phenomenon, encompassing the spontaneous emission of light through mechanisms such as photoluminescence, is integral to various scientific and industrial fields. The distinction between intrinsic and extrinsic luminescence allows for a better understanding of the underlying processes that govern light emission in luminescent materials, or phosphors.

The preparation of phosphors has evolved significantly over the years, with various synthesis techniques such as solid-state reactions, combustion synthesis, sol-gel processes, co-precipitation methods, hydrothermal techniques, and spray pyrolysis. Each method offers unique advantages, allowing for the tailored design of phosphors capable of meeting specific requirements in their applications. From achieving high purity and homogeneity to producing novel particle morphologies and sizes, these techniques pave the way for the development of phosphors with enhanced luminescent properties.

As the exploration of luminescent materials continues, their significance is likely to expand further, leading to innovations in display technology, lighting solutions, and many other fields. Understanding the principles of luminescence and phosphor synthesis not only furthers scientific knowledge but also encourages advancements that can improve everyday life and foster technological progress.

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