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Application of Rare Earth Upconversion Luminescent Material in Anti-counterfeiting Technologies

Hongsheng Lu[1,a,*]

[1]School of Chemistry and Molecular Engineering, Nanjing Tech University, Nanjing, 211800, China

a. peter@njtech.edu.cn

**corresponding author*

Abstract: Rare earth upconversion luminescent nanomaterials exhibits several unique advantages including large anti-stokes shifts, narrow emission bands, adjustable fluorescence lifetimes, and multi-mode emission, making them ideal for anti-counterfeiting applications. This paper explores the application of these materials in various printing technologies, including inkjet printing, screen printing, nano-imprint lithography, and aerosol spray, for the creation of secure anti-counterfeiting patterns. Rare earth doped upconversion luminescent nanomaterials can be printed into a variety of anti-counterfeiting patterns, which has significant application potential in the fields of optical anti-counterfeiting, information storage and labeling. The upconversion luminescence mechanism is discussed in detail, followed by an analysis of recent advances in the field. The existing anti-counterfeiting printing technology and excitation mode of rare earth upconversion luminescence nanomaterials in the anti-counterfeiting and security field in recent years. Finally, rare earth upconversion luminescent materials have great potential for application in the field of secure anti-counterfeiting, and the unique luminescent mode ensures the uncopyability of anti-counterfeiting products. are addressed, and the prospect and expectation of the future development direction are presented. UCNPs, anti-counterfeiting printing technology, excitation mode together, is expected to contribute to the field of security technology.

Keywords: upconversion luminescence, rare earth elements, security, exciting forms, printing technology.

1. Introduction

In this era of rapid technological development, how to judge the authenticity of products purchased has become one of the hot topics. At the same time, the significance of secure anti-counterfeiting has become increasingly important. By accurately identifying information, the social security coefficient is also constantly improving, and it is expected to establish a unique identification code database for important documents such as ID cards and passports, promoting the safe development of the world. However, despite the increasing investment in the field of secure anti-counterfeiting in recent years, the counterfeiting problem has not been well resolved. Physical, chemical, digital and other anti-counterfeiting technologies have been broken through by counterfeit manufacturers through clever means, while optical anti-counterfeiting has gradually become a hot spot in the field of secure anti-counterfeiting due to its higher anti-counterfeiting dimension and difficulty of duplication. Compared with traditional luminescent materials such as organic fluorescent dyes and quantum dots, rare earth upconversion luminescent materials have attracted the attention of researchers due to their excellent tunable performance in the fields of secure anti-counterfeiting and biomedicine[1–5]. Compared with other nanomaterials, rare earth upconversion luminescent materials have attracted the attention of researchers in security, anti-counterfeiting, biomedicine, among other fields, due to their outstanding tunable performance. Additionally, the increasing prevalence of counterfeit products in the market, coupled with rising government and institutional investments in anti-counterfeiting security, has further driven the development of these materials in the field of security and anti-counterfeiting 6-7 In today's era of heightened information security, the importance of secure identification and verification has grown significantly. By accurately identifying and recognizing critical information, social security is constantly being enhanced[8]. One potential future development is the creation of a global database for important documents, such as identity cards and passports, which would further promote global security and stability. Therefore, improving anti-counterfeiting system has become a key focus for researchers.

Among various efforts to enhance security, optical anti-counterfeiting material emerged as a major area of interest. Up-conversion luminescence is a nonlinear luminescence process that can absorb two or more low-energy photons and generate high-energy photons [9]. Due to characteristics like a large anti-Stokes shift, low biological toxicity, resistance to photobleaching, upconversion nanoparticles (UCNPs) has become a hot topic in security and anti-counterfeiting sectors.

Typically, UCNPs consists of three main components, the host matrix or stroma materials, sensitizer ions, and activator ions. Sensitizers ions, such as Nd3+ and Yb3+, are responsible for absorbing the energy of 808 and 980 nm lasers, respectively, due to their large absorption cross-sections. The activator ions, which serve as the core luminescent component in the upconversion system, emit characteristic spectral bands after being excited by multiple photons. After receiving the energy of multiple photons from the sensitizer or absorbed directly from the excitation light, the activator ions transition to higher energy levels and then return to the ground state by radiative transitions and release the light energy. Common activators include $Er3^{+}$, $Ho3^{+}$, and $Tm3^{+}$. $Er3^{+}$ and $Ho3^{+}$ are notable for their red/green emitting properties, while $Tm3^{+}$ is well-known for its blue/ultraviolet emission. By adjusting the structural composition and process parameters of UCNPs, their emission color, intensity, and fluorescence lifetime can be fine-tuned in multiple dimensions [10-13]. Rare earth up-conversion luminescent material, which combine rare earth ions ($Ln3^{+}$) with inorganic matrix material, boast high stability, multi-mode emission, and adjustable fluorescence lifetimes, making them ideal for applications in information imaging, encryption and other security-related fields [14-16]. When UCNPs are combined with graphics encoding technology and anti-counterfeiting printing technology, it is possible to create intricate anti-counterfeiting patterns on various substrates. These patterns are difficult to replicate, ensuring the security of important information. UCNPs’ stable and adjustable luminescence properties make them particularly suitable for high-value items of high-level precision security [17]. For example, multi-color UCNPs have been utilized to develop multidimensional fluorescent QR codes, which can be printed directly onto pharmaceutical capsules and read using a specific smartphone application [18].

In the past 5 years, the application of UCNPs in security and anti-counterfeiting has expanded significantly, showing great potential for protecting of consumer rights and enhancing information security. This paper first outlines the luminescence mechanism of UCNPs, followed by a summary of the recent advancements in excitation modes and anti-counterfeiting printing technologies — such as inkjet printing, screen printing, nano-imprint lithography and aerosol spray printing. Finally, the paper discusses the challenges and future prospects of UNCPs in security and anti-counterfeiting.

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2. Up-conversion luminescence mechanism

Up-conversion luminescence, that is, Anti-Stokes luminescence, means that the material is excited by low-energy light and emits high-energy light, that is, when excited by light with long wavelength and low frequency, the material emits light with short wavelength and high frequency. Near-infrared, NIR) is an excitation light source that cannot be recognized by human eyes. When applied to UC anti-counterfeiting ink, it can produce personalized patterns or colors and carry rich spectral information. And its composition and structure are complex and diverse, and it is difficult to obtain and imitate. In addition, these materials usually have excellent stability, so they are effective in anti-counterfeiting applications. The luminescence mechanism of rare earth upconversion materials mainly includes ESA, ETU, PA, EMU.

2.1. Excited state absorption (ESA)

Excited State Absorption (ESA) occurs when rare earth ions or transition metal ions in a material absorb multiple photons and transition to a high excited state. Eventually, the ions return to the ground state via radiation transitions, releasing energy. In this process, the ions gradually absorb low-energy photons, transitioning to increasingly higher excited states. However, the absorption cross section of rare earth ions is small ($<< 10^{-21}$ cm^2), and the up-conversion luminescence efficiency of ESA is low.

2.2. Energy transfer upconversion (ETU)

The luminescence process of ETU involves the energy transfer between two or more ions. After one kind of sensitizer absorbs the excitation light and reaches the excited state, the energy is transferred to another activator ion through resonance energy transfer (FRET) or electron exchange non-radiative transfer (Dexter ET), and the latter transits back to the ground state through the excited state and emits photons. In the process of ETU, the energy level of sensitizer ion should match the energy level of activator ion, and the distance between them should be close. Efficient ETU luminescence can be achieved by reasonably selecting the types of luminescent ions and regulating the concentration of doped ions[19]. In practice, cross relaxation refers to the energy transfer process involving partial excitation energy that occurs between ions of the same excited state. Cross relaxation is generally considered as detrimental, as it can contribute to fluorescence quenching. It is only observed when the doping concentration is high.

2.3. Photon Avalanche (PA)

In the Photon Avalanche (PA) process of optical upconversion, the key factors are involved: weak ground-state absorption, strong excited-state absorption, and effective cross-relaxation. Under pump light excitation, some electrons are excited to an intermediate energy level. These electrons then absorb additional energy to transition to a higher energy level. Energy transfer occurs between excited ions and ions in the ground state, creating more ions in the intermediate state. Through repetition, a large number of photons and electrons are generated, creating a strong gain effect. When the excitation intensity exceeds a specific threshold, the cycle is triggered by excited state absorption (ESA), resulting in the activator transitioning from the ground state to excited state. The activator undergoes efficient cross-relaxation with the sensitizer, and the photons in the sensitizer's excited state produce an avalanche effect, resulting in intense up-conversion emission [20]. By adjusting the pump power and the size structure of nanoparticles, the nanoparticles are expected to be used in nano-imaging and sensing fields.

2.4. Energy migration-mediated up conversion (EMU)

Energy Migration-Mediated Upconversion (EMU), first proposed by Wang in 2011, involves the excitation of sensitizers—such as ions or molecules—by low-energy photons [21]. In the energy migration-mediated up-conversion (EMU) process, first, the low-energy photons excite the sensitizer (such as sensitized ion or molecule) to the excited state. These excited state sensitizers then transfer energy through dipole-dipole interactions or resonant energy transfer. As energy accumulates, the activated ions eventually transition to higher energy level and finally emit up-converted light as photons with higher energy. This mechanism effectively integrates the energy from multiple low-energy photons, resulting in strong upconversion emission. Also, the EMU process is largely limited by ion concentration.

Upconversion luminescence mode provides high sensitivity and difficult-to-copy anti-counterfeiting features by emitting short-wavelength fluorescence at a specific wavelength, enhancing the security of identification. Moreover, it allows multiple layers of information encoding, combined with intelligent detection technology, to achieve real-time verification, effectively countering counterfeiting and widely used in fields such as tickets, credentials, and product packaging.

3. UNCPs excitation mode

3.1. NIR Single-Wavelength Excitation

In the near-infrared (NIR) single-wavelength excitation process, a near infrared light source excites a particular fluorescent dye or nanomaterial. The excited state molecules or ions subsequently return to the ground state by a radiative or nonradiative process, releasing a fluorescent signal that can be detected. One of the most common strategies for anti-counterfeiting applications involves using NIR single wavelength excitation (808/980/1550 nm) to create fluorescent patterns. For instance, printing QR code labels on the packaging of goods is the most common anti-counterfeiting measure. By combining UCNPs and QR code technology, optical QR code with enhanced security can be generated, offering a potential anti-counterfeiting strategy [22]. Sangeetha and other researchers injected positive and negative charges into the PMMA film, then soaked the film in the surface-modified β-$NaYF_4$：$Gd3^+$/$Er3^+$/$Yb3^+$@$NaYF_4$ solution, and used AFM nano electrostatic printing technology to make the two-dimensional code pattern with uneven thickness. It is found that the profile height distribution of the pattern is highly consistent with the emission intensity distribution at 545 nm under the excitation of 980 nm, which indicates that nano electrostatic printing technology improves the security of the QR code in terms of size and complexity [23].

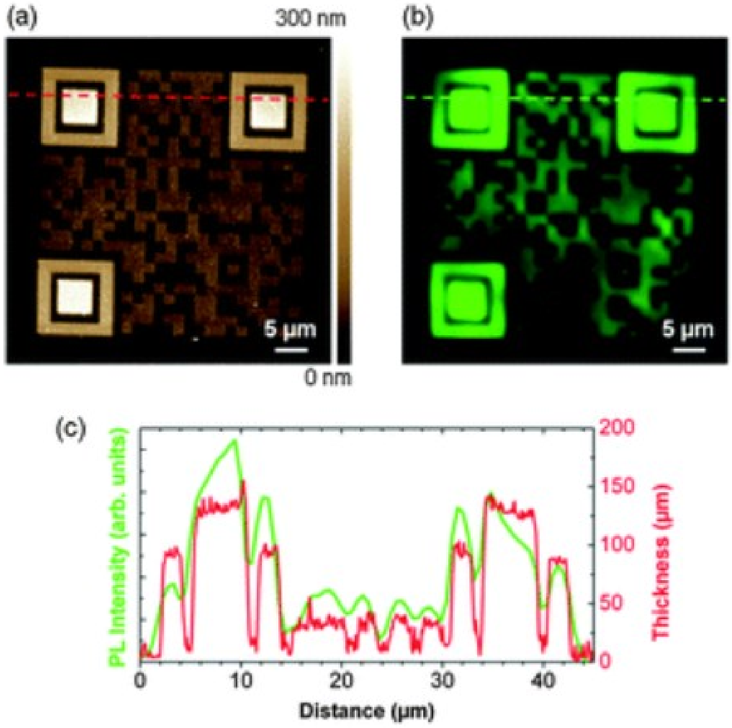

Figure 1: (a) AFM topographical image of a 40 µm × 40 µm QR code constructed from green-upconverting β-$NaYF_4$:Gd^{3+},Er^{3+},Yb^{3+}/$NaYF_4$ core–shell NCs, presenting features of three different thicknesses; (b) upconversion PL mapping (at 545 nm) of the QR code shown in (a) under 980 nm cw laser irradiation; (c) plot showing a good correlation between the thickness corresponding to the red cursor line in (a) and the PL intensity variation along the green cursor line in (b)[23].

3.2. NIR/UV Dual-Wavelength Excitation

The security level of the anti-counterfeiting pattern excited by NIR single wavelength is relatively low, and it is easy to be imitated and replaced by other fluorescent materials. In order to improve the security level of anti-counterfeiting, researchers have invested a lot of energy in designing dual-wavelength NIR/UV excited UCNPs for full-color display, anti-counterfeiting security and information encryption.

The NIR/UV dual wavelength excitation process combines up-conversion luminescence and down-conversion luminescence, using two light sources with different wavelengths to achieve more efficient signal output. In this process, up-converted luminescence generates high-energy photons by absorbing multiple low-energy photons (such as near-infrared light), while down-converted luminescence emits low-energy photons by absorbing high-energy photons (such as ultraviolet light). This combination produces unique fluorescence characteristic under the excitation of different wavelengths, so that authenticity is effectively distinguished. As a result, this dual-wavelength excitation method has broad applications in security labels, biological identification, intelligent packaging and the like, and improves the safety and reliability of the anti-counterfeiting technologies.

An example of this application is the development of a lead-free perovskite structure (BMSI) designed by Xu [25], which exhibits excellent photoelectric properties. Its inorganic framework consists of angle-linked SnI_6^{4-} octahedra anchored to two layers of organic cations through strong N-H.. I hydrogen bonds. Protonated cations of MA reside in the interstices of the perovskite framework through moderate hydrogen bond interactions, while two adjacent organic layers are connected together through weak van der Waals forces. These properties allow bulk crystals to be cleaved into microplates easily, maintaining long-term stability. The peak positions of the Raman spectrum and the photoluminescence (PL) spectra taken before and after etching showed no significant changes, which verifies the structural integrity and durability of BMSI. To further test the robustness of BMSI, the Raman and PL spectra of the microchips (D = 1.5 μm) before and after the etching treatment (50 ccm Ar, 100 W power) were compared. In the experiment, the characteristic peaks of the Raman spectrum and the PL spectrum are almost unchanged, which proves that the microstructure of BMSI is not damaged after the etching process. The stability and the service life of the anti-counterfeiting material are greatly improved.

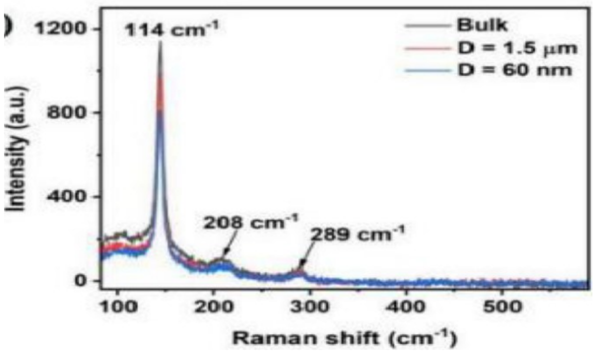

Figure 2: Raman and PL spectra of BMSI microchips (D = 1.5 μm) before and after etching (50 ccm Ar, 100 W power) [25]

3.3. TTA-UC (Triple-Triplet Annihilation Up-Conversion)

Compared with the anti-counterfeiting patterns excited by single wavelength and double wavelength, TTA-UC's ability to convert low-energy photons into high-energy emission can be customized according to the different material systems used to meet specific application requirements. TTA-UC materials have significant advantages in anti-counterfeiting because of their high-sensitivity fluorescence signal which are generated by converting low-energy photons into high-energy photons. These materials have unique luminescent properties, enabling them to emit different colors of light at specific wavelengths, thus effectively distinguishing authenticity. In addition, TTA-UC materials generally exhibit good thermal and chemical stability to accommodate a variety of environmental conditions, ensuring long-term effectiveness of the security label. By designing different TTA-UC material combinations, multiple signal outputs can be achieved, which further increases the difficulty of counterfeiting.

These properties make TTA-UC materials widely applicable to fields such as currency, identity documents, luxury goods, drugs and food. A multi-mode light-emitting system, developed by Chen [Citation needed], combines TTA-UC organic fluorescent molecules (sensitizer and annihilator) with lanthanide-doped phosphors of different afterglow colors. This novel system emits fluorescence (FL), up-conversion (UC) and afterglow luminescence through a double excitation mode (365/532 nm). The resulting luminescent material is patterned using direct writing technology, enabling multi-dimensional anti-counterfeiting applications.

3.4. Ink Preparation for TTA-UC Applications

In the ink preparation of inks for TTA-UC anti-counterfeiting applications, two types of polymers, namely poly-alpha-pinene (PαP) and polyisobutylene (PIB), are used to construct a resin network. The solid matrix allows the dye molecules and fluorescent powder to be well dispersed (figure 2c). PαP chemically reacts with singlet oxygen, while PIB physically prevents external oxygen from penetrating into the system. These two types of polymers help maintain the stability of the TTA-UC emission in air, preventing oxygen from quenching the triplet excited state.

The method is advantageous because of simple manufacture, low cost, flexible coding and difficulty in imitation. It opens a new way for multi-dimensional anti-counterfeiting [26].

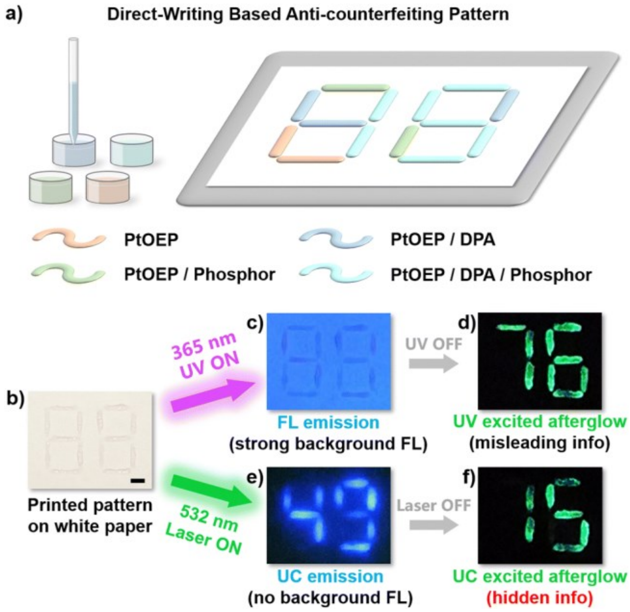

Figure 3: patterning of multi-mode luminescent materials for multi-dimensional security applications. (a) a schematic diagram of multi-dimensional luminescent pattern preparation based on the direct writing method. The luminescent properties of each part of the pattern are carefully designed and programmed by controlling the composition of the ink. (b) Printed pattern on white paper (left, scale: 2mm). (c) Printed pattern excited by ultraviolet light at 365 nm (FL mode). The FL emission mode exhibits strong background fluorescence from the paper substrate. (d) The printed pattern shows afterglow emission after ultraviolet irradiation, displaying the obviously misleading information "76" (ultraviolet excited afterglow mode). (e) Printed pattern excited by a 532 nm CW laser area light source (UC mode). UC emission image showing clear information "49" was recorded through a 520 nm short pass filter. (f) The printed pattern shows afterglow emission after laser irradiation at 532 nm, showing the true hidden information "15" (UC excited afterglow mode) [26].

4. Anti-counterfeiting printing technology

4.1. Inkjet printing

Inkjet printing is a printing technique that ejects liquid ink through nozzles onto paper to form images and text. The combination of rare earth upconversion luminescent material (UCNPs) and the printing ink offers great application potential in safety anti-counterfeiting.

With the development of science and technology, information security is becoming more and more complex, such as two-dimensional code, but inkjet printing has great application potential in the production of complex patterns. Its advantages include high quality image output, low initial acquisition cost, and support for multiple print media. In a study by Lin et al [27], the protocol for performing color printing ink containing UCNPs was developed. It ultimately prints multicolor patterns of the popular Tuzki character. Softwares such as ImageJ, as shown in the figure 3e-3g, was used to separate these overlapping patterns into distinct RGB layers, demonstrating the feasibility of 3D QR codes for anti-counterfeiting purposes.

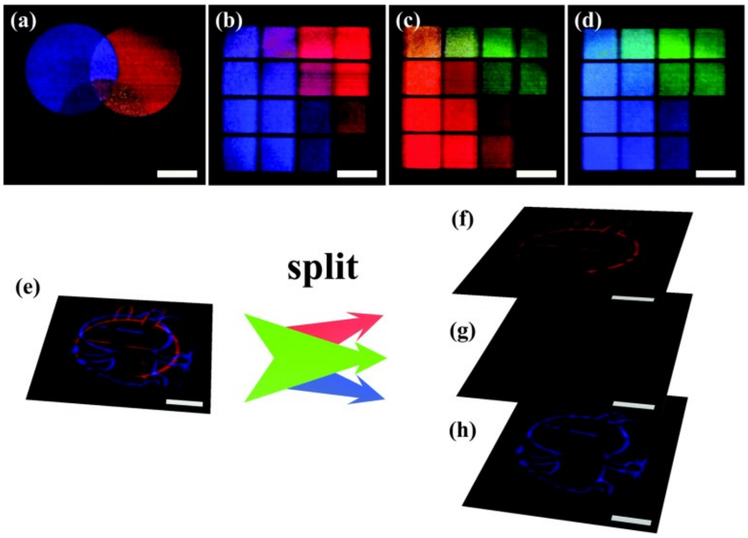

Figure 4: multicolor printing based on RGB. (a) three primary colors based on RGB. The color is adjusted by printing (b) blue and red luminescent inks, (c) red and green luminescent inks, and (d) blue and green luminescent inks. Photographs of 48 luminescent samples excited by near infrared light printed on A4 paper were printed by superimposing two luminescent inks for printing at 0, 25, 75 and 100% surface coverage. (e) Red, green and blue inks printed on the same overlapping surface. The printed pattern can be divided into three layers according to different color channels, (F) red, (G) green and (H) blue. The scale represents 5mm [27].

4.2. Screen printing

Screen printing involves using stencils on a screen to push ink through a mesh onto various materials like fabric, paper, or metal. This technique offers vivid colors, durable patterns and suitability for mass production. However, the cost of plate making is high, and the performance of details is limited with each color requiring a separate plate.

In a study conducted by Wang et al [28]., $NaYF_4^{3+}/Eu^{3+}$ micro-nano particles were used to analyze luminescence properties under 980 nm laser excitation. The researchers prepared a screen-printing agent by mixing these particles with alcohol and printing anti-counterfeiting patterns on paper using a custom screen template. After air-drying the printed typefaces, they were exposed to 980 nm laser irradiation, and the resulting images were captured using a camera. The diffraction peaks of the printed $NaYF_4^{3+}/Eu^{3+}$ particles matched the standard $NaYF_4$ card, with no impurities detected. The imaging results show that the particles exhibited stability and reliability in anti-counterfeiting applications, although they were somewhat influenced by the natural environmental factors. Screen printing is more suitable for mass production of low-quality images. Nonetheless, screen printing with UCNPs shows great potential in anti-counterfeiting identification.

4.3. Nano imprint lithography (NIL)

Nanoimprint lithography (NIL) is an advanced nanofabrication technique that uses high-resolution molds to imprint micron and nano-scale patterns onto a variety of material surfaces, such as silicon wafers and glass. Despite the challenges of mold wear and low imprint speed, its simple process flow and strong adaptability make it have an important position in the development of modern nanotechnology.

In a study by Zeinab Chehadi, the photoluminescence of Eu^{3+} was enhanced by approximately 200 times through coupling between resonances and nano-scale structures. This enhancement was observed within a relatively small angle (≈±16° around the vertical direction) [29], making NIL a promising approach for improving the emission efficiency.

4.4. Aerosol spray printing technology

Aerosol jet printing (AJP) is a high-resolution 3D printing technology that enables the manufacture of complex patterns and structures by atomizing liquid materials into tiny particles and spraying them onto substrates. AJP has advantages including high resolution and multi-material compatibility, making it particularly useful in the fabrication of high-precision electronic devices. However, few studies have explored the use of AJP for patterning UCNPs, though it is expected that AJP and rare earth upconversion luminescent materials will have good potential in the future [30-31]. Future research is expected to focus on improving its precision, expanding material compatibility, and refining techniques for large-scale production.

The combination of rare earth upconversion luminescent materials and the above printing technologies provides a development direction of new anti-counterfeiting materials in the later period. Ink jet printing technology and aerosol jet printing technology greatly meet the requirements of anti-counterfeiting for high precision and complex patterns, and improve the difficulty of counterfeiting at the same time, providing users with a good experience. Screen printing can be used for mass production of products with the same design, and the technical difficulty is low, which well meets the anti-counterfeiting requirements of daily necessities. As a hot research topic, nano-imprint lithography technology can generate high-resolution and low-toxicity patterns on various substrates through template imprint hardening, which is suitable for biomedical anti-counterfeiting field.

5. Limitations and Prospect of Anti-counterfeiting Technologies

However, the development of UCNPs is still restricted by many factors. Although the core-shell structure, MOFs and other means can improve the quantum yield and luminous intensity of UCNPs, it is still not as efficient as the following conversion. It is also challenging to keep the low toxicity and stability of the rare earth materials prepared in the laboratory after synthesizing ink remains to be solved. The pattern definition made by screen printing technology is not high, and the cost of aerogel jet printing technology is too high, which hinders the application of rare earth materials in the field of anti-counterfeiting security. Moreover, it is difficult to recognize and read patterns made of rare earth upconversion luminescent materials, and a single device cannot read multi-layer patterns. Therefore, it is urgent for more researchers to devote themselves to the research in this field and jointly promote the wider application of rare earth upconversion materials in the engineering field. Through continuous innovation and efforts, it is expected to solve the above problems and contribute more to the future engineering practice and scientific and technological development.

6. Conclusion

In such important fields as medicine and food, fake and inferior products have caused great harm to human beings. Therefore, it is necessary to apply rare earth upconversion luminescent materials to the field of security and anti-counterfeiting. Due to its unique non-toxicity and bleaching resistance, it can become one of the hot spots in the current research. The application of rare earth upconversion luminescent materials in anti-counterfeiting technologies holds significant promise due to their unique properties. UCNPs can better exert its effects by using core-shell structure, MOFs and other means. At the same time, by NIR single wavelength excitation and other excitation modes combined with optical material inkjet printing, screen printing, nano-imprint lithography and aerosol spray printing technology, the UCNPs high-resolution anti-counterfeiting pattern can be printed, and the multi-mode dynamic response function in a wide spectral range can be realized, which are of great significance for the development of new optical anti-counterfeiting materials.

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