

具有pH、力、光刺激响应型发光材料的研究综述

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摘要

刺激响应型发光材料作为一种能够将外部环境变化转化为可观察到的光学信号的智能化材料, 是连接物理世界与信息感知的重要桥梁。本文聚焦于pH刺激响应、力刺激响应与光刺激响应三大体系, 阐述了其核心响应机制、材料设计策略及部分应用研究进展。

关键词

pH刺激响应, 力刺激响应, 光刺激响应, 发光材料

A Review of Research on Luminescent Materials Responsive to pH, Mechanical Stimulation, and Light

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Abstract

Stimulus-responsive luminescent materials, as intelligent materials capable of converting changes in the external environment into observable optical signals, serve as a vital bridge connecting the physical world with information sensing. This paper focuses on three major systems—pH-responsive, force-responsive, and light-responsive materials—and discusses their core response mechanisms, material design strategies, and recent advances in applied research.

Keywords

pH-Responsive, Force-Responsive, Light-Responsive, Luminescent Materials

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1. 引言

随着科技向智能化、信息化时代的发展，智能化材料成为关键驱动力之一。智能化材料，也称刺激响应型材料，是一种能够感知外界特定环境刺激，并使光学信号产生变化的材料。这类材料可以将接收到的外部“信号”直接转化为易于观测的光学信号，实现了从“感知”到“可视化”的衔接，并且因其在实际应用中的巨大潜力而受到越来越多的关注[1]-[6]。根据不同的外界刺激，这类材料可以分为光刺激响应型发光材料、力刺激响应型发光材料、热刺激响应型发光材料、化学刺激响应型发光材料等[7][8]。本综述将主要聚焦于化学(pH)、力和光三类刺激响应型材料，旨在通过对比，分析出不同刺激源下发光行为的异同，为构建新型多功能智能化材料提供理论依据。

2. pH 刺激响应型材料

pH 刺激响应型材料在新一代智能化发光体系中起着至关重要的作用。在过去的研究中，比较常见的 pH 刺激响应型材料有四苯乙烯(TPE)[9]、香豆素[10]、萘酰亚胺[11]、二吡咯甲烷硼(BODIPY)[12]等，这些材料被广泛应用于药物输送、癌症诊断和治疗等生物医学领域[13]-[18]。除此之外，pH 刺激响应型材料也被应用于数据存储、信息防伪与加密系统[19]。所谓 pH 刺激响应型材料，就是材料随着环境 pH 变化而使其光学性质发生改变[20]-[22]。目前，大多数 pH 刺激响应型材料的分子结构中都存在对质子敏感的基团，如羧基、氨基、磺酸基、酚羟基等，这些基团都易于质子化或是水解，在环境 pH 变化时极易发生质子化、去质子化过程，从而使分子内电荷转移态、能级结构发生改变，引起其光学性质也发生改变[23]-[25]。了解分子结构与 pH 刺激响应行为之间的关系，并探索 pH 刺激响应机制，对于 pH 刺激响应型材料的开发和应用具有重要意义。

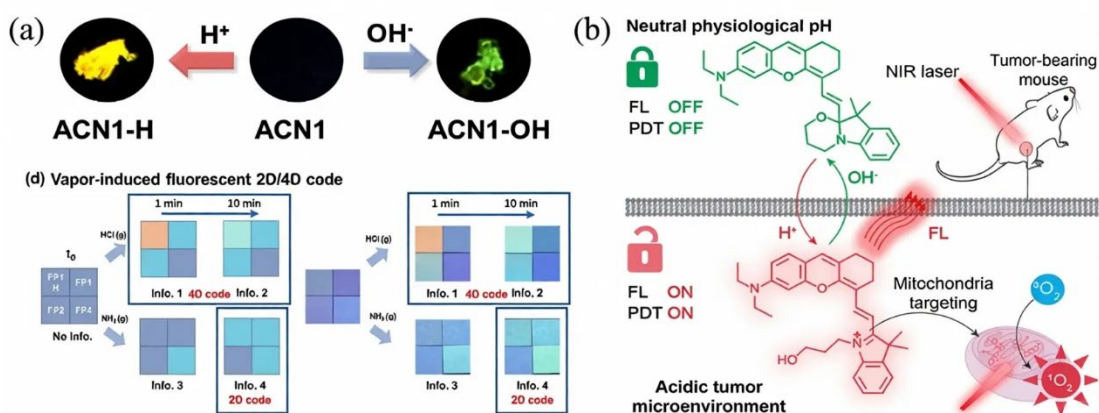


Figure 1. (a) Solid state fluorescence “on” behavior of ACN 1 and four-dimensional (4D) fluorescence encoding; (b) The response mechanism of LET-15 in acidic tumor microenvironment

图 1. (a) ACN 1 固态荧光“开启”行为，以及四维(4D)荧光编码；(b) LET-15 在酸性肿瘤微环境下的响应机制

尹梅贞教授课题组[26]设计合成了一系列具有 pH 刺激响应的氰基二苯乙烯衍生物(ACNs), 其中, 化合物 ACN 1 同时含有吡啶和酚羟基两种官能团, 具有酸、碱双重刺激响应性。如图 1(a)所示, ACN 1 固体状态下不发光, 但在酸或碱的外界刺激下, 可以通过电离增强的分子内电荷转移(ICT)机制, 实现“开启”的荧光行为。酸会使 ACN 1 吡啶部分质子化发出蓝绿色荧光, 碱会使 ACN 1 酚羟基部分去质子化发出绿色荧光。基于此, 他们开发了隐形墨水、显色剂、多重信息隐藏的高级防伪与信息加密系统, 同时, 还开发了四维(4D)荧光编码, 大大增强了信息加密过程的复杂性和安全性。

林静教授课题组[27]设计合成了一种近红外半菁光染料 LET-15, 该染料有羟基, 具有 pH 刺激响应性。如图 1(b)所示, 在 pH 为中性的正常组织中, LET-15 分子呈闭环的构象, 分子不发光。但是在酸性肿瘤微环境下, LET-15 分子被环境中质子特异性激活, 使醚键断裂, LET-15 分子转变为开环的构象, 从而表现出强烈的近红外吸收和荧光发射, 并靶向线粒体。在 660 nm 激光照射下, 被激活的 LET-15 分子能高效地产生活性氧来杀伤肿瘤细胞, 同时, 还可以最大限度地减少对正常组织的损伤。这项工作为设计开发用于肿瘤精准光动力治疗的、可被肿瘤特异性激活的近红外光敏剂提供了有价值的指导。

张红雨教授课题组[28]设计合成了一种希夫碱化合物 PIHN, PIHN 可以在溶液中生长出三种不同晶型的晶体, 分别为 PIHN-B (块状)、PIHN-N (针状)和 PIHN-P (片状), 其中, 不发光的 PIHN-B 晶体暴露在主链含 2~3 个碳原子的短链脂肪酸(如乙酸、丙酸)蒸气中时, 能发出绿色荧光, 而本身就发光的 PIHN-N 晶体和 PIHN-P 晶体在相同条件下发光几乎没有变化(如图 2(a)所示)。该工作首次报道了依赖于晶型的酸蒸气响应行为, 并提出了不同于传统质子化响应的机制: PIHN-B 分子与酸之间形成了氢键, 使原本扭曲的 PIHN-B 分子构型变得更加平面化, 进一步限制了分子的振动, 从而产生了荧光。当酸分子离开晶体后, 荧光又会消失。基于此, 他们还开发了一种新颖的文件信息安全保护方法。

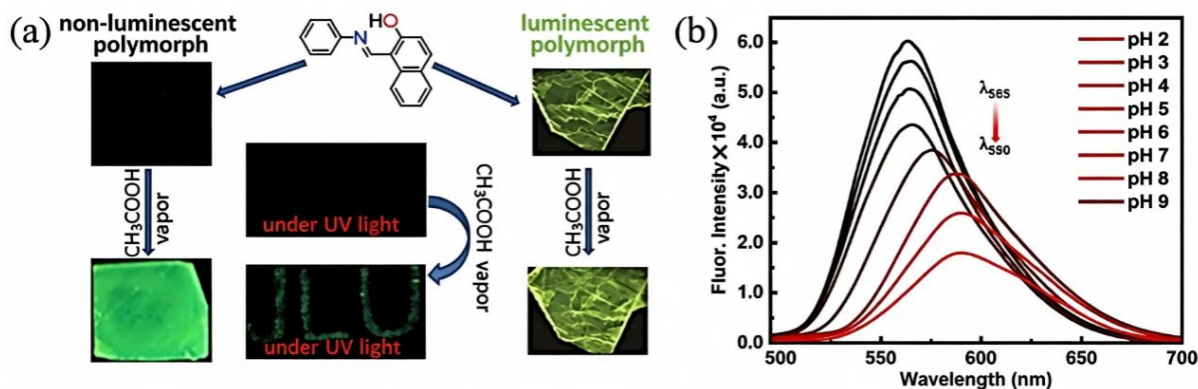


Figure 2. (a) Molecular structure of PIHN and fluorescence photos before and after acid stimulation in different crystal phases; (b) Fluorescence emission spectra of compound 6c at different pH values (pH range from 2 to 9)

图 2. (a) PIHN 的分子结构及其不同晶相酸刺激前后荧光照片; (b) 化合物 6c 在不同 pH 值(pH 范围从 2 到 9)下的荧光发射光谱

Mandal 等人[29]成功开发了一类新型 pH 刺激响应的双吡咯乙烯基席夫碱荧光团(6a~6c), 它们在溶液和固体状态下都发出强烈的荧光, 并且具有大于 120 nm 的斯托克斯位移和良好的荧光量子产率(PLQY)。化合物 6a~6c 对环境 pH 变化表现出可逆的颜色变化和荧光“开关”行为。例如, 化合物 6c 在 pH > 5 时, 日光下溶液为橙色, 在 UV 下发出黄色荧光; 在 pH < 5 时, 溶液变为红色, 荧光光谱发生红移(如图 2(b)所示)。研究表明, 酸性条件下亚胺键被选择性质子化, 分子通过与抗衡阴离子(如 TFA)形成主-客体复合物(如 6c·2H⁺), 构建了稳定的三维超分子结构, 导致光谱红移。这个过程是可逆的, 并伴随晶体形貌变化, 在 pH 传感和智能材料领域有应用潜力。

3. 力刺激响应型材料

力刺激响应型材料也是智能化材料的一种,是指材料在外界机械力(如压力、拉伸力、剪切力、研磨等)的刺激下,光学性质发生显著性变化。这类材料能够根据所接受的外界机械力大小和方向的变化来动态调整其发光颜色,体现出独特的应变性能和应用潜力[30]。力刺激响应型材料的响应机制通常涉及到了材料内部的分子结构、聚集态的微观排列或者是电子能态的改变,并且这个变化过程可能是可逆的,也可能是不可逆的[31]-[33]。力刺激响应型材料广泛应用于信息加密、生物传感、工程检测、数据储存等领域[34]-[39]。

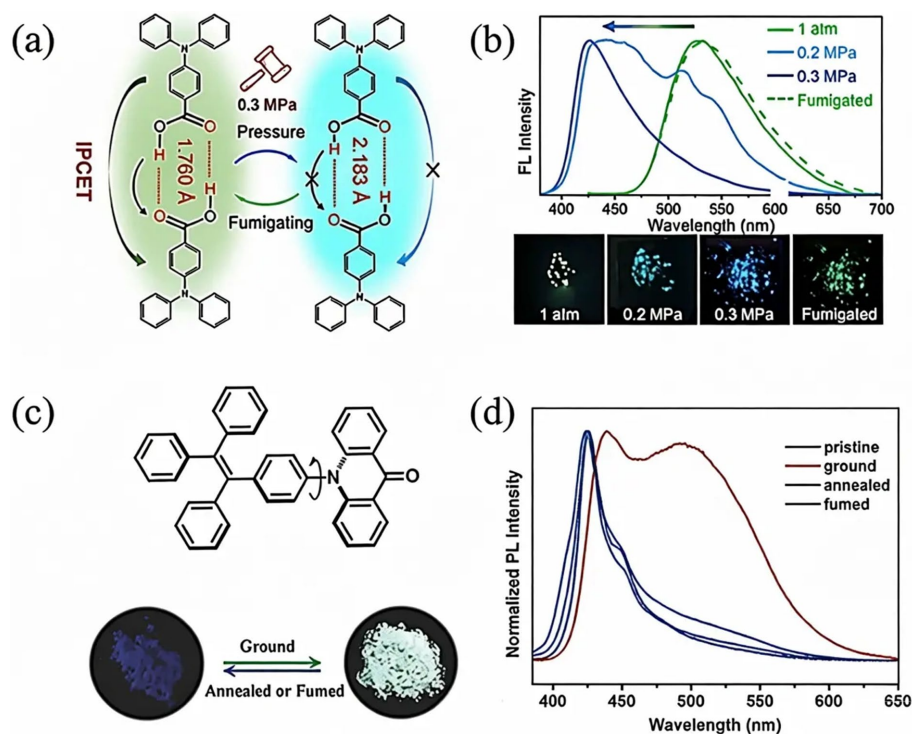


Figure 3. (a) TPAC molecule; (b) The force stimulus responsiveness of AD-TPE molecules
图 3. (a) TPAC 分子; (b) AD-TPE 分子的力刺激响应性

付红兵教授课题组[40]报道了一种力刺激响应变色材料 TPAC, 如图 3(a)、图 3(b)所示, 该晶体在极低的 0.3 GPa 的压力下, 荧光颜色从绿色转变为蓝色, 蓝移了大约 105 nm, PLQY 从 19.6% 提升到了 43.7%。产生这种现象的原因是羧酸分子间通过二聚形成了键长为 1.760 Å 的强分子间氢键, 为分子间质子耦合电子转移(IPCET)过程提供了质子的转移通道, 导致 TPAC 分子处于电荷转移态, 从而发出长波长、低效率的绿光, 而当施加微小压力时, 氢键距离被拉长到了 2.183 Å, 关闭了 IPCET 过程, TPAC 分子回到局域激发态, 从而发出短波长、高效率的蓝光。该工作为力刺激响应型材料提供了理想模型, 也为设计新一代智能响应材料开辟了新道路。此外, 王凯教授课题组[41]也研究了低压下 AIE 分子三苯乙烯(Tripe)的力刺激响应行为。在 0.8 GPa 的压力下, Tripe 分子的荧光强度增加了 1.8%, 荧光发射从 432 nm 红移到 440 nm, 这是由于 C-H... π /C-H...C 氢键增强了, 从而抑制了非辐射跃迁速率。

田文晶教授课题组[42]通过四苯基乙烯(TPE)与吡啶酮(AD)相连接, 设计合成了一种 D-A 型的共轭分子 AD-TPE。在 AD-TPE 晶体的原始状态下, AD-TPE 分子间通过偶极-偶极间的相互作用及弱超分子作用形成 H-聚集体, 由于 H 聚集体的跃迁偶极子是平行排列的, 会导致其 PLQY 较低, 大约为 1%。经过

研磨后,如图 3(c)、图 3(d)所示,AD-TPE 粉末发出明亮的青色荧光,PLQY 提升到了 43%。这是因为研磨导致 AD-TPE 分子从定形态转变为无定形态,使分子堆积结构被破坏,TPE 与 AD 单元之间的扭转角减小到了 50° 以下,从而使分子内电荷转移(ICT)态占主导,增加辐射跃迁的可能性,实现 AD-TPE 分子从暗到亮的增强荧光变化。

王传增教授课题组[43]设计合成了四种具有 D-A-D 结构的二苯甲酮衍生物 1a、1b、1c、1d。研究表明,四种化合物不仅具有良好的热稳定性,力刺激响应性能也可以通过分子结构进行有效的调控。除了化合物 1c,其余物质在研磨条件下都会表现出从无荧光发射到明显的荧光发射的转变。以化合物 1d 为例,研磨后它的荧光发射峰位红移了大约 29 nm,PLQY 从 0.50%提升到了 49%。他们认为,研磨过程破坏了材料的结晶度,形成非晶态结构,有效地削弱了分子间的 π - π 相互作用,从而增强了机械力诱导的荧光发射行为。

韩天宇教授课题组[44]制备了一种力刺激响应发光材料 AHM,该材料对压力具有高敏感性。在对 AHM 施加机械力后,它的 PLQY 从 2.8%提高到了 6.2%,荧光发射波长蓝移了大约 67 nm。通过研究分析,他们证实了荧光增强的原因是 AHM 分子内部因研磨形成了晶格缺陷。粉末 X 射线衍射(PXRD)结果显示,研磨后只有部分晶体结构被破坏,材料仍保持着一定的半结晶性。

4. 光刺激响应型材料

在众多的外界刺激源中,光是一类极具应用潜力的清洁能源,并且来源充足、使用安全性高。相较于 pH、机械力等其他类型的外界能量输入方式,光照刺激不需要与目标材料发生物理接触,这一特性可有效避免接触式刺激对材料表面结构造成的损伤。

光刺激响应型材料是一种能在特定波长和强度的光照条件下可逆或不可逆地改变材料光学性质的智能功能化材料[45]-[48]。这类材料的刺激响应机制主要源于光激发下发生的可逆的光异构化反应,或产生光致电子转移、能量传递等过程。光照前后,光刺激响应型材料的分子结构发生显著性变化,导致吸收光谱和荧光发射光谱也发生变化,继而展现出光刺激响应行为。这类材料在超高密度光存储、多重防伪、光学逻辑器件等领域展现出了巨大的应用潜力[49]-[51]。

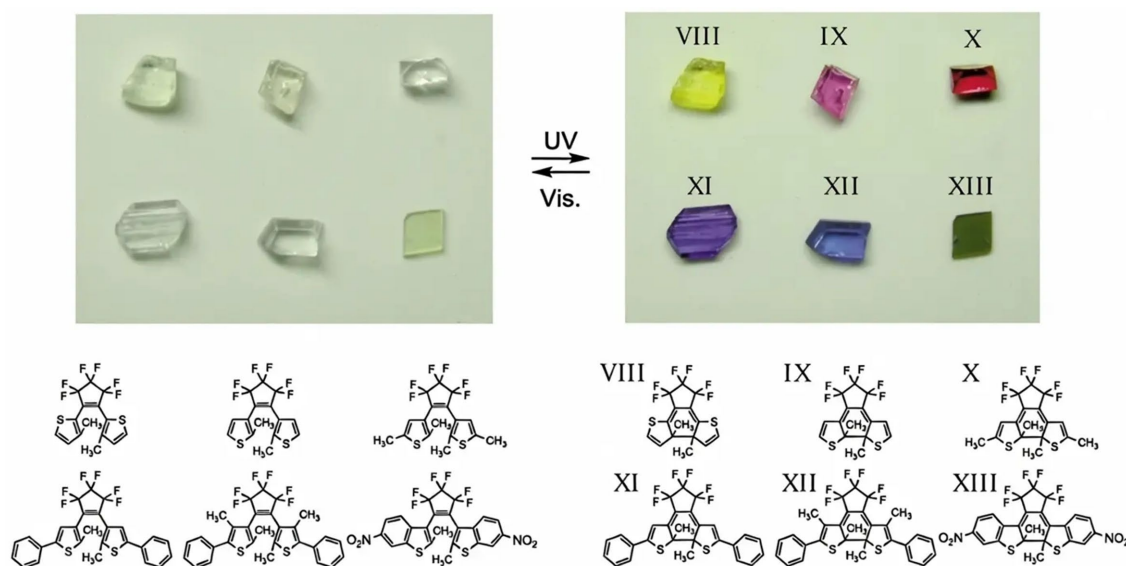


Figure 4. Ring opening and closing isomers of diarylethylene derivatives under illumination conditions, and their crystal photos
图 4. 二芳基乙烯类衍生物光照条件下开环闭环异构体,及其晶体照片

光刺激响应行为在智能材料领域受到了广泛的关注，其中，光环化反应是实现光学性能动态调控的重要途径之一。Irie 等人[52]总结了一系列性能优异的二芳基乙烯类衍生物，该类分子具有两种开环和闭环的异构体，在室温下都是热力学稳定的，并可在 365 nm 紫外光照射下，分子由开环转变为闭环，使共轭长度得到扩展，进而引发了吸收光谱与发射光谱的明显红移，在宏观上表现为材料颜色从无色(或浅色)向黄色、红色、蓝色或绿色等高对比度色彩转变(如图 4 所示)；反之，在大于 500 nm 的可见光激发下，分子又可恢复到开环状态，实现褪色过程。此类光刺激响应材料在高密度光存储、可擦写光记录及动态防伪等领域展现出潜在的应用潜力。

唐本忠院士团队[53]研发了一种四苯基乙烯类衍生物 TPE-4N，该材料在固态下表现出了一种有趣的光刺激响应发光行为：如图 5(a)所示，初始状态下几乎不发射荧光的 TPE-4N 晶体，在经过一定时间的紫外光照后，可以发出明亮的绿色荧光。并且可以通过 150°C 或丙酮熏蒸处理后恢复到不发光的状态。经过机理探究，他们认为产生这种变化的原因不是永久性的光化学变化，而是因为紫外光照射导致了 TPE-4N 分子表面发生了微小的分子构象变化，使晶体变成无定形态。该研究为分子光化学领域拓展了新的视角，为设计新型固态智能发光材料开辟了新思路。

王键吉教授课题组[54]设计合成了一种苯并噻唑类衍生物 1，并从溶剂中成功制备了细长的 1 晶体。如图 5(b)所示，当从晶体侧面进行紫外光照射时，可以观察到晶体向背光一侧发生弯曲，并且可以通过改变光照方向来调控晶体的弯曲方向，PLQY 也有一定程度的提升。他们通过核磁证明这一结果并不是因为发生了光化学反应，通过对不同弯曲角度的 1 单晶进行结构解析，表明光照下产生弯曲的原因是光照诱导分子在固态下的协同运动，具体表现为分子层间发生滑移。该研究揭示了固态下光驱动分子运动的新机制，为理解光-机械响应行为提供了新的视角。

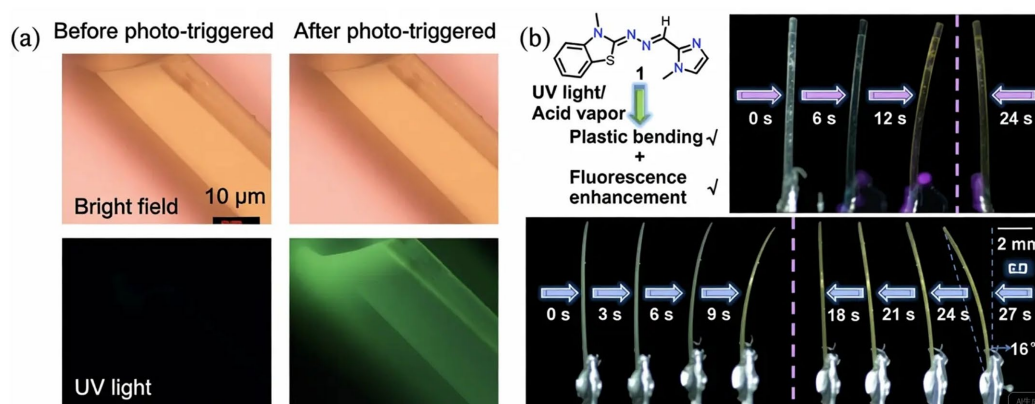


Figure 5. (a) Fluorescence photos of TPE-4N before and after illumination; (b) Compound 1 crystal light stimulus response behavior

图 5. (a) TPE-4N 光照前后荧光照片；(b) 化合物 1 晶体光刺激响应行为

特别地，朱亮亮教授课题组[55]设计并合成了一系列羟基修饰的多硫芳烃，其中固态化合物 1 在 365 nm 紫外光照射下，荧光颜色瞬间从白色变成黄色，仅需 7 s 就可以达到光稳态，实现超快速响应行为，他们证明光诱导自由基的生成是产生这种颜色变化的原因。并且，由于分子在晶体状态下的反平行分子排列和由氢键主导的强非共价网络，稳定了自由基，需要长达 3 个月的时间才能被淬灭。该研究为固态光致变色材料提供了新思路。

5. 结论与展望

刺激响应型材料，特别是 pH、力与光刺激响应体系，通过将外界刺激直接转化为直观的光学信号，

已经成为构筑智能传感与显示平台的核心材料。当前的研究已经在刺激响应机制的阐释、新型材料的开发及多功能集成应用方面取得了显著进展，从单分子设计到聚集态调控的协同策略是未来高性能刺激响应材料的关键，然而，该领域仍然面临着若干挑战：材料的响应灵敏度、可逆性及稳定性有待提升；多刺激协同响应的精准设计与解耦机制尚不明确；面向生物学及柔性电子等实际应用时，材料的生物相容性、可加工性与器件集成工艺仍是瓶颈。总之，刺激响应型材料的世界广阔而深邃，从基础的分子设计、机理揭示到最终的应用落地，仍有大量富有挑战性的工作等待探索。

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