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Mechanochromism of a Perylene Diimide Derivative-Doped Styrene-Butadiene-Styrene Block Copolymer

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Abstract: Mechanochromic materials respond to external stimuli and provide early warnings of material damage. Perylene diimide (PDI)-based materials have attracted attention because of their outstanding fluorescence performance. However, the application of PDI in mechanochromism is limited by the difficulty for mechanical forces to disrupt the aggregation of PDI and its derivatives, as well as the fluorescence quenching caused by continuous π-π stacking between PDI molecules. To eliminate the fluorescence quenching effect caused by the aggregation of PDI and broaden its application fields, polyhedral oligomeric silsesquioxane (POSS)-PDI-POSS (PPP) was screened as PDI doping. The photophysical properties of PPP in both monomeric and aggregated states in different solvents were studied. Then, PPP and styrene-butadiene-styrene block copolymer (SBS) were mixed to prepare the PPP/SBS film. The mechanochromic properties of PPP/SBS film were explored. The fluorescence emission spectra confirmed that when the PPP mass fraction increased to 0.30%, the PPP/SBS film exhibited mechanochromic behavior under uniaxial deformation due to the changes in the molecular packing.

Keywords: mechanochromism; perylene diimide (PDI); styrene-butadiene-styrene block copolymer (SBS); fluorescence

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0 Introduction

Mechanical stresses in polymeric materials may become excessive, resulting in the breakage of macromolecular chains[1-2], which could cause macroscopic failures and material degradation[3]. To prevent such damage, a reliable method is to continuously monitor the strain. In this regard, mechanochromic materials have gained significant importance owing to their response to external stimuli[4-11]. Mechanochromic materials generally use mechanophores in a polymer matrix to alter their absorption and emission properties under mechanical stresses. A few examples of covalent-bonded mechanophores are 1, 2-dioxetane[12-15] (the four-membered ring of 1, 2-dioxetane emits light upon the cleavage of strained bonds), diacetylene[16-18] and spiropyran[19-22].

Two primary methods for incorporating mechanophores into a polymer matrix are covalent bonding and physical blending. The main drawback of the covalently bonded mechanophores is that they exhibit poor responsiveness to low-strain deformation caused by the rupture of covalent bonds, which can lead to slow deactivation and irreversible bond rupture in some cases. In addition, external factors, such as heat and light, can reduce their utility. The physically blended mechanophores consist of non-covalent aggregates, also known as aggregachromic mechanophores. These mechanophores disassemble under the shear stress generated in the polymer matrix when subjected to external deformation. At certain concentrations, the mechanophores aggregate and exhibit optical properties that differ from those of the isolated monomers[23-25]. The color of these molecules is influenced not only by their chemical structure but also by the arrangement of chromophores. Furthermore, the packing arrangement is sensitive to mechanical simulation, which can alter the conformation of the molecular arrangement, cause a material color change, and exhibit mechanochromic properties.

Wang et al.[26] developed a novel reversible elastomer with bending-induced multicolor switching, enhanced

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sensitivity and emphasized bond scission. The mechanism involves anthracene dimer crosslinks within a reformable sacrificial network, which produces reversible mechanochromism in multinetwork elastomers. Traeger et al.[27] developed folded perylene diimide (PDI) loops. Such PDI loops exhibit mechanoresponsive properties owing to their unique structural arrangements that undergo conformational changes in response to external stimuli. Sagara et al. [28] presented a novel approach for cyclophane-based supramolecular mechanophores. The mechanism involves a transformation in the molecular conformation of the mechanophore within the cyclophane-containing polyurethane film under external deformation, which shifts the fluorescence emission from excimer to monomer. When building mechanochromic materials by using the physical blending method, it is crucial to identify a suitable chromophore, as its molecular arrangement structure has an impact on both the macroscopic color and the material's response to external mechanical force. However, it may be challenging to achieve outstanding fluorescence and mechanochromic properties simultaneously in a single material.

PDI is a kind of pigment with various colors which are influenced by the packing arrangement. According to the existing literature, owing to the different arrangements and assembly structures, PDI can theoretically display colors ranging from green to red or even black[29-31]. PDI exhibits excellent fluorescence properties in the monomer state, and its fluorescence quantum yield can reach 100%[29]. However, the strong π-π interaction between PDI molecules results in fluorescence quenching and low sensitivity for mechanical simulation. Among PDI-based dyes, the polyhedral oligomeric silsesquioxane (POSS)-PDI-POSS (PPP) exhibits significant potential as an aggregochromic component in mechanochromic polymer blends, due to its unique structure that undergoes optical and physical property changes in response to environmental stimuli. Compared with the extended strong π-π stacking in most PDI derivatives, the steric hindrance of POSS tends to reduce the strong π-π interaction between PDI cores in PPP. In our previous research[32], we discovered that the dimer motif is responsible for the arrangement of PPP. Therefore, in the single-crystal structure of PPP, the bilayer structure with POSS and PDI disrupts the extended strong π-π stacking, resulting in high photoluminescence properties.

In this study, we select PPP as the chromophore because of the relationship between its arrangement structure and fluorescence color. We explore the fabrication and characterization of PPP dye in the styrene-butadiene-styrene block copolymer (SBS) matrix, with a particular focus on the additional π-π interaction between the dye and the polymer, which enhances the sensitivity of mechanical simulation. The mechanochromic effects are evaluated by applying uniaxial deformation at room temperature, and a mechanism is proposed to explain how the packing structure of PPP contributes to its color-changing behavior.

1 Materials and Methods

1.1 Materials

Aminoproyl-isobutyl-POSS was purchased from Hybrid Plastic (USA). SBS was purchased from Shanghai Songhan Plasticization Technology Co., Ltd., China and had a styrene/rubber mass ratio of 28/72. Chloroform, hexane, toluene, perylene-3, 4, 9, 10-tetracarboxylic dianhydride (PTCDA), imidazole, silica gel and other chemicals were purchased from Sinopharm Group (China). Unless otherwise stated, reagents and solvents were procured as reagent grade and used without further purification. All reactions were conducted using standard glassware under an inert nitrogen atmosphere.

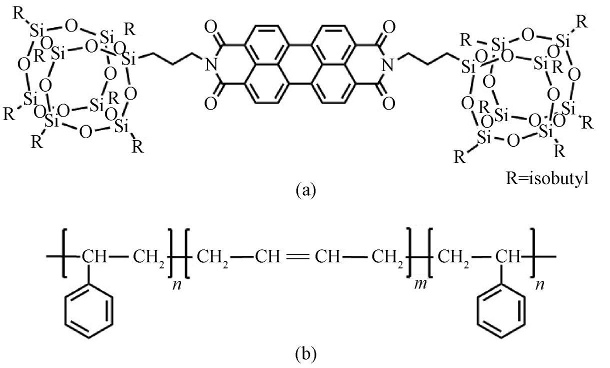
1.2 Sample preparation

PPP and C_{12}-PDI-C_{12} (CPC) conjugates were synthesized in accordance with the standard procedures outlined in Ref. [33].

For PPP, initially, POSS (1.19 g), PTCDA (0.07 g), and imidazole (2.00 g), were placed in an around-bottom flask fitted with a reflux condenser. The mixture was heated in an oil bath at 140 ℃ with vigorous stirring for 6 h under an argon atmosphere. The reaction mixture was then cooled, and it was dispersed in 50 mL ethanol and 50 mL HCl solution (2 mol/L) overnight. Next, the mixture was extracted with 200 mL chloroform twice. The combined organic phase was washed with 5% (mass fraction) $NaHCO_3$ solution until neutral and then washed with brine. Finally, the crude product was purified via silica gel column chromatography (mobile phase: chloroform; retardation factor: 0.3) to obtain a red solid, PPP (yield: 75%).

For CPC, the same reaction procedure was followed, but POSS was replaced with dodecylamine. Each synthesized product was confirmed by ^{1}H NMR and ^{13}C NMR.

SBS (2.7 g) was dissolved in 15 mL chloroform at room temperature. The mixture was stirred until all the SBS pellets were fully dissolved in chloroform. Then, the proper amount of PPP or CPC (mass fraction: 0.05%–1.60%) was added to prepare their respective blends, which were then left to stir for 24 h. The colored solution was then applied to the glass plate under a hood to facilitate the evaporation of the solvent. After drying overnight, the film was removed from the plate and equilibrated to room temperature. The chemical structures of PPP and SBS are shown in Scheme 1.



Scheme 1 Chemical structures: (a) PPP; (b) SBS

1.3 Instrument and characterization

The ultraviolet-visible (UV-vis) spectra of samples in chloroform, hexane and toluene were recorded on a UV-vis spectrophotometer (GENESYS 50, Thermo Fisher Scientific, USA). The fluorescence spectra of the samples were recorded on a fluorescence spectrophotometer (FluoroMax Plus, HORIBA Scientific, USA). Solid-state drawing was carried out at room temperature.

2 Results and Discussion

To investigate the potential of constructing mechanochromic materials by using PPP, the transformation rules of its aggregation state and ground state were first studied. Based on this analysis, films were prepared by blending PPP in a general polymer, and the mechanochromic properties were further studied.

2.1 Photophysical properties of PPP in solution

To investigate the influence of solvents and their concentration on the aggregation state and ground state of PPP, photophysical experiments were conducted in three different solvents: chloroform, hexane and toluene. Figures 1(a) and 1(b) provide a comparison of the UV-vis absorption and fluorescence spectra of PPP in chloroform, hexane and toluene respectively, at a concentration of 1.6×10^{-4} mol/L. PPP shows monomeric absorption characteristics with λ_{max} at 525 nm in chloroform. In hexane, the spectrum shows a noticeable hypochromic shift of 10 nm, attributed to the less polar nature of hexane which leads to less solvation and a greater tendency towards aggregation. In toluene, the spectrum exhibits comparable electronic features, with a redshift of 2 nm compared to the spectra in chloroform, which could result from an additional π-π interaction between the benzene ring in toluene and the PDI core in PPP.

In the corresponding fluorescence spectra, the reduced fluorescence intensity in toluene compared to that in chloroform and hexane can be attributed to the energy dissipation caused by the collision between the benzene ring and PDI. The results suggest that the benzene ring of toluene and the PDI core of PPP undergo π-π interaction, leading to a change in the photophysical behavior of PPP.

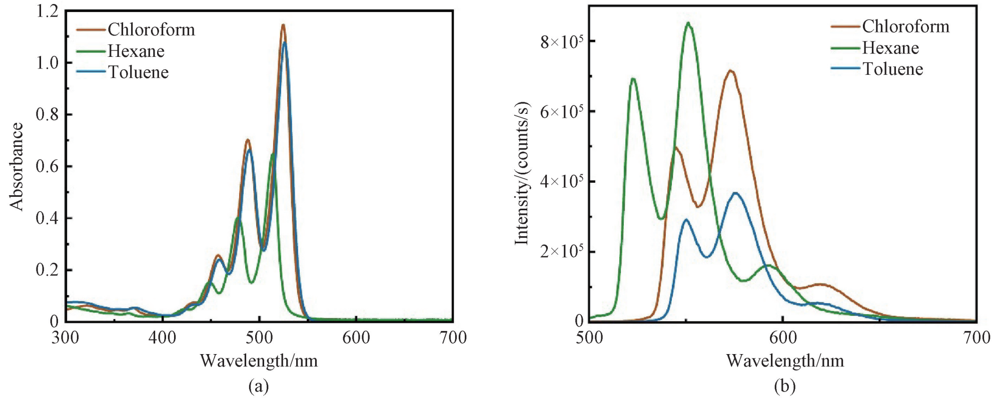


Fig. 1 Photophysical properties of PPP in chloroform, hexane and toluene at a concentration of 1.6×10^{-4} mol/L: (a) UV-vis spectra; (b) fluorescence spectra

2.2 Photophysical properties of PPP in PPP/SBS film

Based on the photophysical properties, the molecular interactions between PPP and toluene were verified. To realize the transformation of the uniaxial deformation applied to the material into the molecular packing, benzene-structured matrices were selected. SBS and styrene were chosen as matrices for mechanochromic materials owing to their benzene structure. It would be verified that PPP had the potential to be a chromophore for mechanochromic materials in these matrices, and supposed that the mechanochromic properties of the material could be enhanced by the interaction between the aromatic matrix and PPP. SBS was chosen as the matrix because of its rubber-like properties and its benzene ring enhancing π-π interaction, which could be further explored upon the application of mechanical stresses. PPP was uniformly dispersed at different mass fractions in the SBS matrix to prepare a film by the solution-scraping-coating method.

To determine the optimal mass fraction of PPP in the matrix, the photophysical properties of PPP/SBS in chloroform were explored.

The spectra in Fig. 2 (a) show a continued bathochromic shift in emission with an increase in the PPP mass fraction. In Fig. 2(b), at lower mass fractions (0.05% – 0.10%), the spectra display oligomeric emission with λ_{max} at 577 nm. As the PPP mass fraction increases to 0.30%, the shoulder peak becomes the maximum peak with λ_{max} at 620 nm, indicative of aggregation formation.

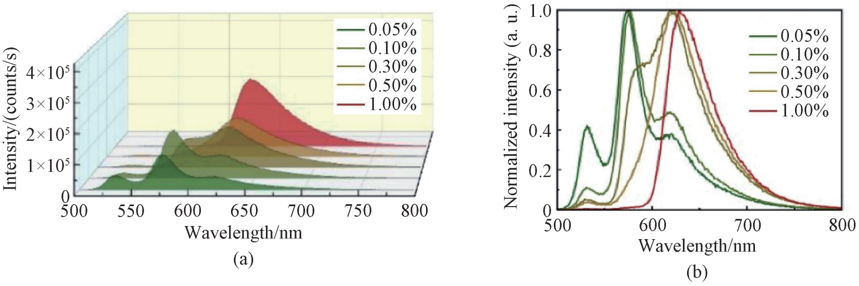


Fig. 2 Fluorescence spectra of PPP/SBS films at different PPP mass fractions: (a) three-dimensional fluorescence spectra; (b) normalized two-dimensional fluorescence spectra

2.3 Effects of uniaxial deformation on PPP/SBS film

This research aims to explore the mechanochromic properties of PPP/SBS films by evaluating their optical properties after subjecting them to uniaxial deformation at room temperature. Figure 3(a) shows the fluorescence spectra of the PPP/SBS film at a PPP mass fraction of 0.30% under different states. The fluorescence spectra of the PPP/SBS film exhibit significant changes after mechanical stimulation. In the unstrained state, the fluorescence spectrum of the PPP/SBS film displays three distinct peaks at 536, 577 and 620 nm. The peak at 620 nm indicates aggregation formation, while the peak at 577 nm represents oligomer formation, as evident in Fig. 2(a). As the strain increases from 0 to 100%, the intensities of both peaks at 577 nm and 620 nm change, which indicates a change in their photoluminescent state. Before the deformation, the ratio of the peak intensity at 577 nm to that at 620 nm (denoted as $I_{577\ nm}/I_{620\ nm}$) is 1.18, and when the strain is 100%, the ratio becomes 2.97 (Fig. 3(b)). These results suggest that external mechanical deformation influences the molecular packing of PPP in the PPP/SBS films. This deformation imparts a change in the balance between different photoluminescent states from aggregation to oligomer or monomer. The change in the fluorescence intensity for different peaks also results from a change in the electronic structure of the chromophore. In its unstrained state, the peaks at 577 nm and 620 nm indicate specific colors of green and orange, respectively. However, when the strain increases from 0 to 100%, the intensities at 577 nm and 620 nm change significantly. The peak at 577 nm now displays a relatively enhanced fluorescence intensity, while the peak at 620 nm is reduced, resulting in the green color becoming dominant. Upon relaxation, the spectrum does not return to its original state, indicating that the induced changes in the molecular packing are not completely reversible.

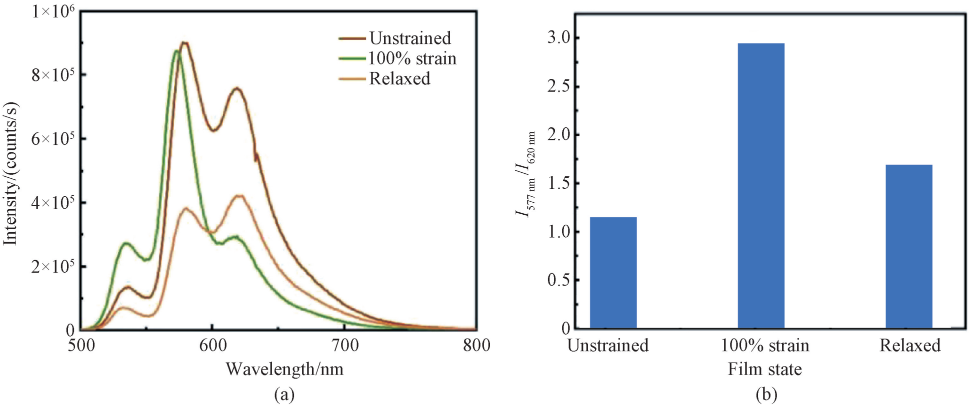
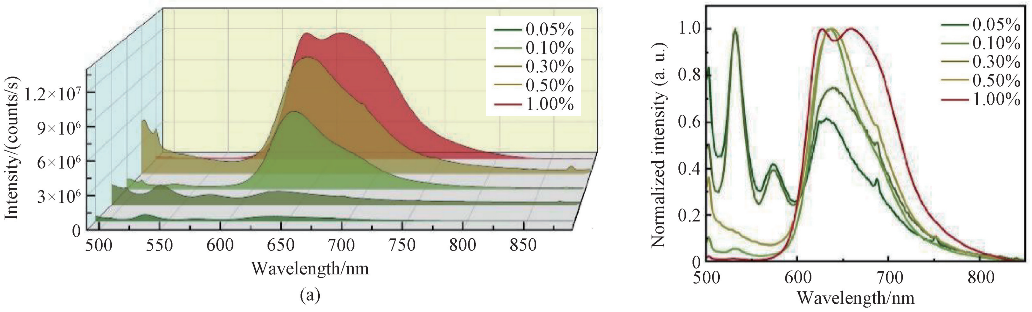


Fig. 3 Effects of uniaxial deformation on photophysical properties of PPP/SBS films at a PPP mass fraction of 0.30%: (a) fluorescence spectra; (b) $I_{577\ nm}/I_{620\ nm}$

2.4 Mechanism and comparison of packing structures

After confirming that mechanical deformation induces changes in the fluorescence intensity of PPP, we investigated the underlying mechanism and contrasted it with the behavior of another dye, CPC, within the same polymer matrix, namely SBS. This comparison enhanced the understanding of how different molecular structures influence these packing structures, as shown in Figs. 2 and 4.

CPC has linear alkyl chains on both sides and exists in an extended π-π stacking in the condensed phase. Figure 4 indicates that CPC in SBS displays two distinct aggregation behaviors. Initially, oligomeric packing is observed at the CPC mass fraction of 0.05% – 0.10%. However, as the CPC mass fraction increases to 0.30%, it shows the continuous π-π stacking.



(a) (b)

Fig. 4 Fluorescence spectra of CPC/SBS films at different CPC mass fractions: (a) three-dimensional fluorescence spectra; (b) normalized two-dimensional fluorescence spectra

Figure 5 illustrates the effects of uniaxial deformation on the photophysical properties of CPC/SBS films at a CPC mass fraction of 0.30%. As the strain increases from 0 to 100%, the excimer emission increases with a slight shift in the peaks from 656 nm to 640 nm. In contrast, at a PPP mass fraction of 0.30%, the PPP/SBS film exhibits two different behaviors, aggregation and oligomerization, which is due to the reduced packing tendency of PPP. CPC exhibits very strong π-π interaction between the perylene planes. A stronger force is required to observe the evident color alternation due to strong π-π interaction between the perylene chromophores. Therefore, the rigidity and steric resistance of POSS in the unique chemical structure of PPP weaken the molecular stacking of PDI, which is an important factor leading to its application in mechanochromic materials.

Figure 6 shows the schematic mechanism of the structural alterations of PPP from the dimer to the monomer in response to external mechanical deformation. In the condensed phase, PPP tends to exist as a dimer motif in the molecular arrangement. However, when external mechanical deformation is applied, it influences the molecular packing of PPP. The external mechanical deformation causes a change in the balance between the photoluminescent states from aggregation to oligomer or monomer. Consequently, a prominent color change from orange to green is observed, which demonstrates the potential of PPP as a mechanochromic dye. PPP exhibits excellent photoluminescence properties with a fluorescence quantum yield of about 0.48, owing to its tendency to form discontinuous dimer packing[32]. Therefore, the addition of PPP to the polymer matrix can simultaneously achieve excellent fluorescence and mechanochromic properties.

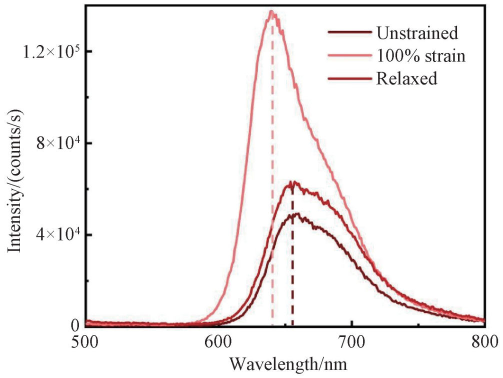


Fig. 5 Effects of uniaxial deformation on photophysical properties of CPC/SBS films at a CPC mass fraction of 0.30%

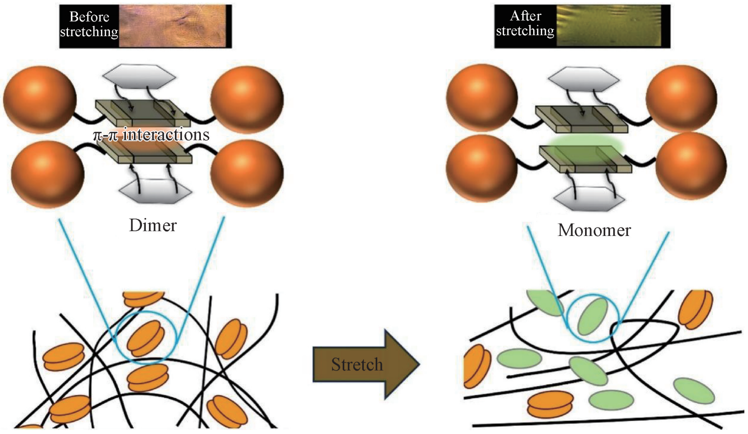


Fig. 6 Schematic illustration of structural alterations of PPP from dimer to monomer in response to external mechanical deformation and respective fluorescence images under UV light (λ =365 nm)

3 Conclusions

In this work, the mechanochromic behavior of the PPP/SBS film was investigated. To study the effects of molecular attraction between PPP and a benzene ring, the photophysical properties were first measured and compared in different solvents (chloroform, hexane and toluene). In toluene, PPP underwent a redshift in both the absorption and emission spectra, confirming that the perylene core of PPP is molecularly attracted to the benzene ring of toluene.

The photophysical properties of the PPP/SBS film revealed different aggregation behaviors. The emission spectra of the PPP/SBS film under uniaxial deformation at a PPP mass fraction of 0.30% showed that the mechanochromic behavior was attributed to the transition from aggregation to oligomer or monomer. The results indicated that PPP was responsible for this mechanochromic behavior owing to its tendency to form a discontinuous packing structure.

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聚苯乙烯-丁二烯-苯乙烯掺杂苝二酰亚胺衍生物力致变色

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摘　要：力致变色材料会对外部刺激做出响应，因此可实现材料损伤预警。苝二酰亚胺（perylene diimide，PDI）衍生物因其出色的荧光性能而受到关注。然而，由于机械力难以破坏 PDI 及其衍生物的聚集，以及 PDI 之间连续 π-π 堆积导致的荧光猝灭，PDI 在力致变色领域的应用受限。为了避免 PDI 聚集引起的荧光猝灭效应，拓宽其应用领域，该文选择多面体低聚倍半硅氧烷（polyhedral oligomeric silsesquioxane，POSS)-PDI-POSS(PPP)分子作为掺杂剂。研究了在不同溶剂中 PPP 单分子态和聚集态的光物理性质。将 PPP 分子与聚苯乙烯-丁二烯-苯乙烯（styrene-butadiene-styrene block copolymer，SBS）混合制备 PPP/SBS 薄膜，探索了此类材料的力致变色效应。荧光发射光谱表明，当 PPP 质量分数增加到 0.30%时，PPP/SBS 薄膜在单轴拉伸变形下因分子堆积行为改变而变色。

关键词：力致变色；苝二酰亚胺；聚苯乙烯-丁二烯-苯乙烯；荧光