Large-strain and Full-color Change Photonic Crystal Films Used as Mechanochromic Strain Sensors

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Research Article

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Large-strain and Full-color Change Photonic Crystal Films Used as Mechanochromic Strain Sensors

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Abstract

Mechanochromic strain sensors based on photonic crystals, which can change structural color upon mechanical deformation, are promising for many applications including health monitoring and damage detection. Here, a large-strain and full-color change photonic crystal film as a mechanochromic strain sensor is reported. The mechanochromic photonic crystal (MPC) film with grating nanostructure is achieved by a fast and low-cost inverted nanoimprint lithography. The film enables bright and reversible color shift in the full visible range as it is stretched up to a large strain, which is proven in strain direction parallel and perpendicular to the grating. It is demonstrated that the MPC film can be used as a mechanochromic strain sensor able to visualize strain evolution and strain range of low carbon steel in tensile test, which brings a significant step toward visualizing strain monitoring.

Keywords: mechanochromic; photonic crystal; structural color; grating; strain sensor

1. Introduction

Mechanochromic strain sensors based on photonic crystals have recently received a great deal of interest due to their vivid structural color display, easy detection of strain even by the naked eye, and zero energy consumption [1,2]. These sensors find essential applications in health monitoring and damage detection in biomedical devices, smart displays, household products, fingerprint identification, and civil engineering [3,4].

The most commonly used method in this field is based on bottom-up approaches to synthesizing mechanochromic photonic crystals (MPCs) [5]. For example, polystyrene microspheres [6-15], silica nanoparticles [16,17], poly(ethyl acrylate-co-methyl methacrylate) colloidal particles [18], gold nanoparticles [19], and aluminum nanowires [20] have been embedded in elastomers as MPCs. Although the sensors show strain-induced color shift, color shift along with the change of the strain is highly limited because the extension entails significant rearrangement of the nanostructures [21]. Inverse opal photonic crystals with spherical air cavities embedded in elastomers have negligible resistance against deformation of cavities, thereby providing a full-color change [22]. However, delicate procedures of fabrication and low mechanical stability of inverse opal structures restrict practical uses, and only small extensional strain is allowed.

Lithography techniques represent an alternative strategy to prepare MPCs [23]. The main advantages of the lithography methods are the high quality of the products and the reproducibility of the process [24]. With this method, MPCs with periodic cylinder shaped air holes have been prepared. It is reported that the sensor could achieve full-color change over the entire visible light range, but within a small strain of 29% [25]. Moreover, the high costs of the instruments and the complexity of the procedure are hampering the mass diffusion of these techniques. For use in some sensing large strain, e.g., monitoring tensile test of low carbon steel, mechanochromic strain sensors with high-quality structural color and full-color change under large strain are needed. However, mechanochromic strain sensors based on MPCs with all these properties are yet to be demonstrated.

Herein, large-strain and full-color change MPC films used as mechanochromic strain sensors were fabricated through fast and low-cost inverted nanoimprint lithography. The MPC film exhibits a pronounced structural color which shifts in full visible range as it is stretched up to a strain of 100%. Furthermore, the film shows inverse color shift with uniaxial strain in the direction parallel and perpendicular to the photonic crystal. And the MPC film is successfully used as a mechanochromic strain sensor to visualize strain evolution in a stretched low carbon steel.

2. Experimental

2.1 Materials and equipments

Polydimethylesiloxane (PDMS) (Sylgard 184, Dow Corning, Midland, USA) was used as the MPC film material. It consisted of a base polymer (part A) and a curing agent (part B). Polylactic acid (PLA) (MakerPiPLA, Soongon, Shenzhen, China) was used as the mold material. Q235 low carbon steel tensile specimens were purchased from Shandong Zhicheng Metal company.

The PLA mold was manufactured by 3D printer (M3145S, Soongon, Shenzhen, China). Photonic crystal structure on the MPC film were characterized using a field emission scanning electron microscope (SEM) (Sigma 300, Carl Zeiss, Jena, Germany). Structural colors and reflection spectra of the MPC film were measured by a digital single lens reflex camera (EOS 7D Mark II, Canon, Tokyo, Japan) and a miniature fiber optic spectrometer (Flame-S, Ocean Optics, Dunedin, USA). The tensile specimens were stretched by an electronic universal testing machine (WDW-100E, TIME Shijin, Jinan, China).

2.2 Fabrication of MPC film

The experimental procedure for fabricating the MPC film is described in **Fig. 1**. As shown in **Fig. 1a**, the PDMS part A and part B were mixed in a mass ratio of 10:1. The mixture was thoroughly stirred for 5 min at 500 rpm by a mixer. The template with grating nanostructure on top was cleaned in absolute ethanol and deionized water, and placed in the lower mold, then the upper mold was fixed to the lower mold (**Fig. 1b**).

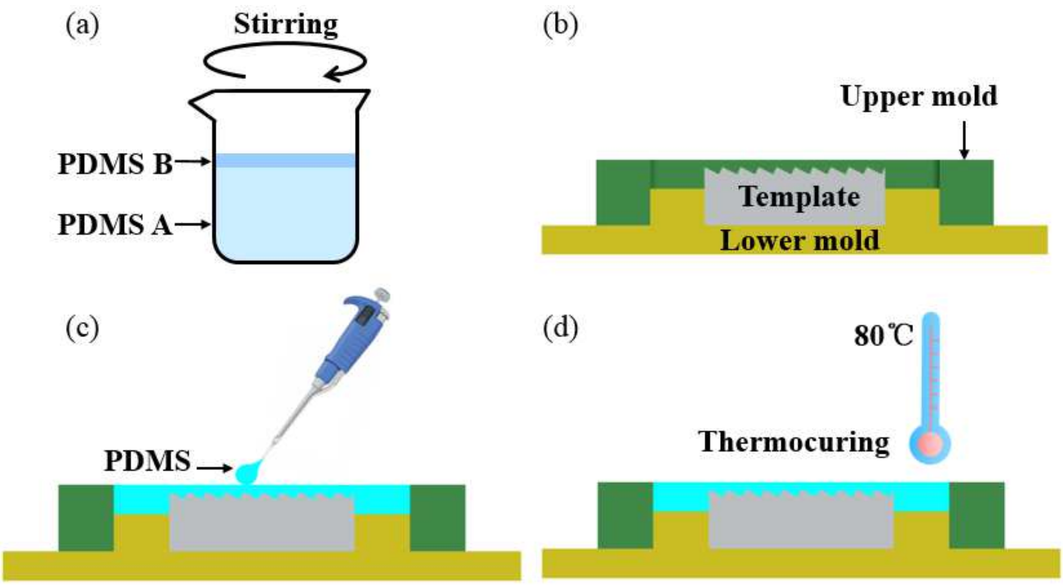

Fig. 1 Schematic fabrication process of MPC film. **a** Preparation of liquid PDMS mixture. **b** Sectional view of a combination of lower mold, template and upper mold. **c** Filling of template and molds with PDMS. **d** Thermocuring of PDMS

As shown in **Fig. 1c**, the mixed PDMS was poured on the template and then left undisturbed for 15 min at room temperature. The PDMS was allowed to flow into the grating of template and the grooves of molds by gravity, and the molds defined the form and size of the MPC film to be created.

The filled molds with liquid PDMS was degassed in vacuum for 15 min and then heated in a vacuum oven at 80° C for 20 min to form a film (**Fig. 1d**). The grating nanostructure was replicated on the surface of the film. After demolding, PDMS MPC film with grating nanostructure on the surface and clamping structures on both ends was obtained. The clamping structures can serve the purpose of keeping the film stressed uniformly and not easy to slip out of clamps during stretching.

2.3 Detection of mechanochromic properties of MPC film

Two kinds of mechanical stretching experiments were performed to explore mechanochromic properties of the MPC film under tensile strain parallel to the grating direction (marked as ε_x, and see **Fig. S1** in Online Resource 1 for the experimental setup) and under perpendicular to the grating direction (marked as ε_y, and see **Fig. S2** in Online Resource 1 for the experimental setup).

3. Results and discussion

3.1. Characterization and mechanochromic mechanism

Fig. 2a, **b** show that the replicated linear grating nanostructure with a period of 800 nm and a line-width of 90 nm exhibits a serrated profile with a height of 130 nm. As shown in **Fig. 2c**, the as-prepared MPC film shows vivid structural colors including blue, green, yellow and red when it is observed from different angles by naked eyes under light. Therefore, the grating nanostructure on the MPC film possesses intrinsic optical characterization of photonic crystals.

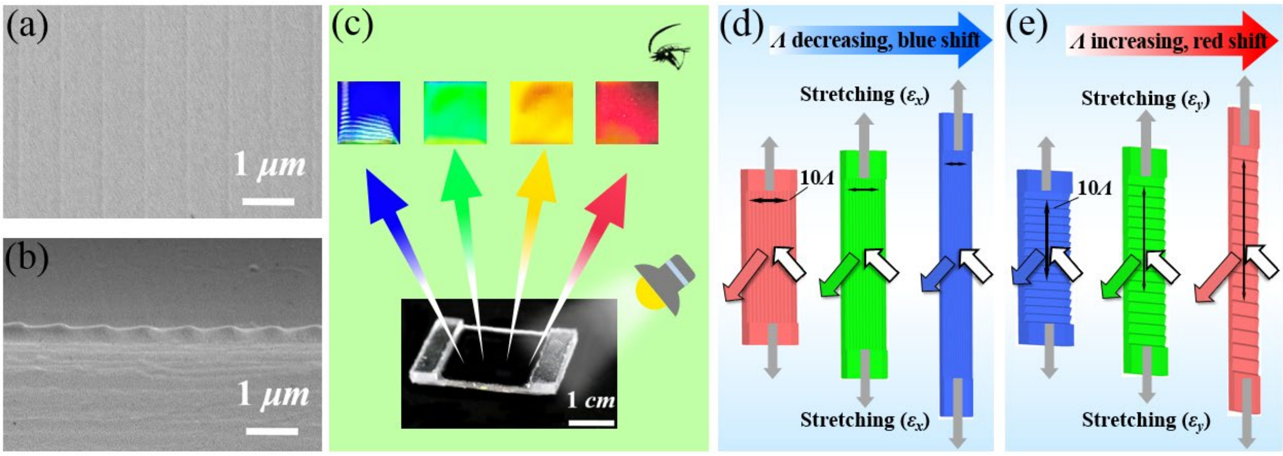

Fig. 2 Characterization of MPC film and mechanochromism mechanism. **a** SEM top view and **b** cross-section view of the MPC film. **c** Photographs of the MPC film when observed from different viewing angles. Schematic of mechanochromic mechanism

of the MPC film for tensile strain **d** parallel to and **e** perpendicular to the grating direction (Λ is structural period of the grating)

The interaction relationship between the grating and the surrounding light can be illustrated by the diffraction optical equation [26]:

$$n_d \sin\theta_{dm} = \frac{m\lambda}{\Lambda} + n_i \sin\theta_i \qquad (1)$$

where n_d and n_i is refractive index of the exit medium and the incident medium, respectively. θ_{dm} and θ_i is the exit angle and the incidence angle, respectively. m is the diffraction order (in general, $m=1$). λ is peak wavelength of the reflected light, which determines structural color of the grating. Λ is structural period of the grating. It can be seen that the structural color can be changed by tuning the structural period. And the structural period of the grating can be tuned by the macroscopic stretching. According to Hooke's law, when the MPC film is stretched along the grating direction (that is, the strain is ε_x) (**Fig. 2d**), the width of cross-section in direction perpendicular to the stretching reduces. Consequently, the period of the grating nanostructure decreases and then the structural color blue shifts. While for the stretching perpendicular to the grating direction (that is, the strain is ε_y) (**Fig. 2e**), conversely, the period of the grating nanostructure increases and the structural color red shifts.

3.2. Mechanochromic properties

To demonstrate that the MPC film is responsive to ε_x, one end of the film is fixed while the other end is stretched constantly and slowly in a direction along the grating. The relationship between stretching deformation and color change of the film is illustrated in **Fig. 3a**. As ε_x increases gradually to 100%, the color continuously shifts from red to blue, covering almost the entire visible range. On the other hand, it is observed that the color shift induced by the strain is highly reversible as a result of elastic deformation (**Fig. 3b**), and mechanochromic response is fully recovered by removing the stretching deformation.

The corresponding coordinates of colors under ε_x ranging from 0 to 100% with interval of 10% are illustrated in the CIE1931 chromaticity diagram (**Fig. 3c**), in which the arrows indicate the change of coordinates during stretching. It can be seen that the movement of the coordinates and the color change shown in **Fig. 3a** are coincident, which indicates the full-color change by stretching. So the tensile strain required for the MPC film to change color over the entire visible range is 100%, which is significantly larger than the tensile strain of 69% required in previous report [21].

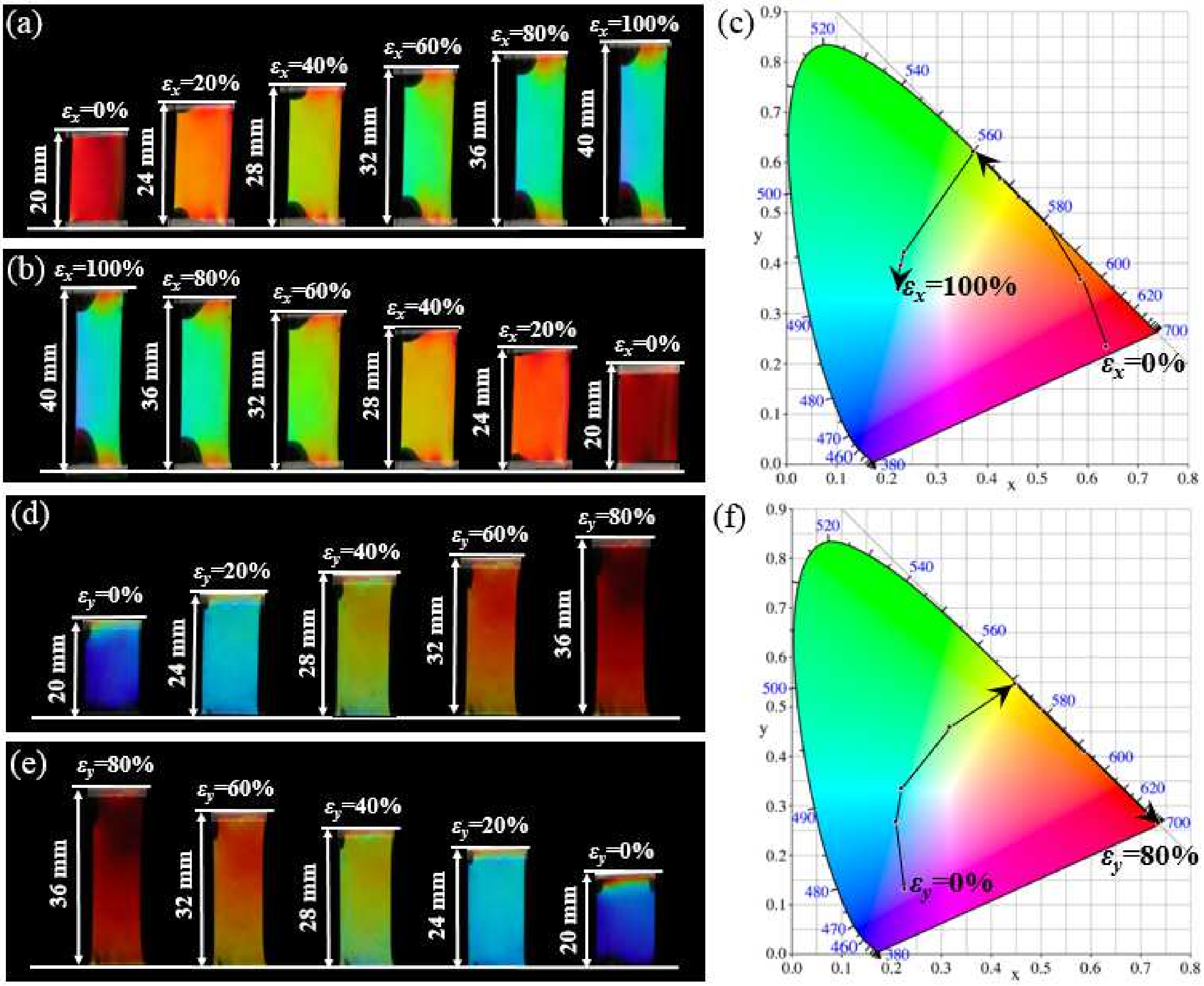

Fig. 3 Mechanochromic properties of the MPC film. Photographs showing a blue-shift and a red-shift of structural color along with increasing **a** ε_x and **d** ε_y, the reversible color change of the film with releasing **b** ε_x and **e** ε_y. Mechanochromic states with increasing **c** ε_x and **f** ε_y in the CIE 1931 chromaticity diagram

To demonstrate that the MPC film is also responsive to ε_y, the color of the film is set to blue at the original length as shown in **Fig. 3d**. When ε_y increases gradually to 80%, the color continuously shifts from blue to red. And when ε_y is recovered to the original state, the blue color is also recovered (**Fig. 3e**). The CIE1931 chromaticity diagram in **Fig. 3f** verifies that the red-shift path covers the entire visible range. Note that the coordinates in **Fig. 3c** are further from the center of the diagram than **Fig. 3f**, which indicates the intensity of the colors in former is higher. The MPC film is thus proven to be responsive to strains both parallel and perpendicular to the grating, which is not demonstrated in previous studies [3].

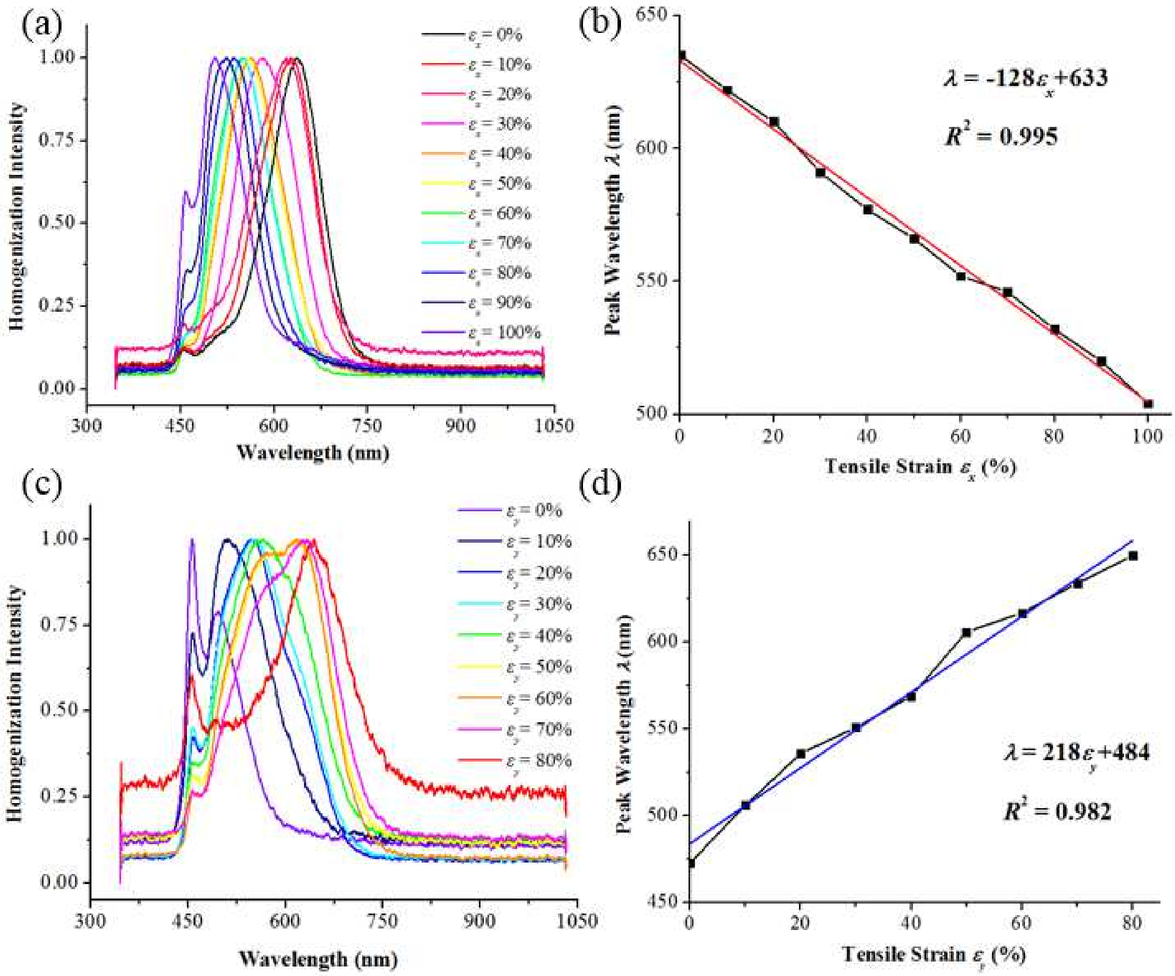

Fig. 4 Relationship between the reflectance spectra and the strain. Reflectance spectra of the MPC film for increasing **a** ε_x and **c** ε_y. λ as a function of **b** ε_x and **d** ε_y

Fig. 4 shows the reflectance spectra of the MPC film and the reflectance peak variation with the applied strain in two directions. The reflectance peak wavelength decreases from 635 nm to 504 nm with ε_x of 100% **(Fig. 4a**, **b)** and increases from 473 nm to 650 nm for 80% with ε_y of 80% (**Fig. 4c**, **d**). It should be stressed that the maximum variation of the peak wavelength is 177 nm (**Fig. 4c**, **d**), which is much larger than that reported previously [2,19,21].

As shown in **Fig. 4b**, the peak wavelength decreases ($\lambda = -128\varepsilon_x + 633$) linearly as a function of tensile strain with high correlation coefficient (R^2) of 0.995. Therefore, the strain of the film with certain color could be calculated using the quantitative relationship, which is necessary for practical use of the MPC film as a mechanochromic strain sensor.

Although color shifts of the MPC film under ε_x and ε_y are exactly opposite, they both can achieve mechanochromism in entire visible light range. However, the film under ε_x has a larger stretching range (100%) than the film under ε_y (80%). Furthermore, mechanochromic properties of the former is superior. The color is brighter (**Fig. 3c**, **f**) and more uniform (**Fig. 3a**, **d**), and the linear correlation between the peak wavelength and the strain is higher (**Fig. 4b**, **d**), which indicating that it is more suitable for application as a mechanochromic strain sensor.

3.3 Application as mechanochromic strain sensor

To demonstrate the utility of the proposed MPC film, the film is used as a mechanochromic strain sensor to visualize strain evolution of a low carbon steel in the tensile test. The sensor was clamped tightly on the surface of a low carbon steel specimen to ensure that the absolute deformation of the sensor is the same as that of the specimen during stretching (see **Fig. S3** in the Online Resource 1 for the experimental setup).

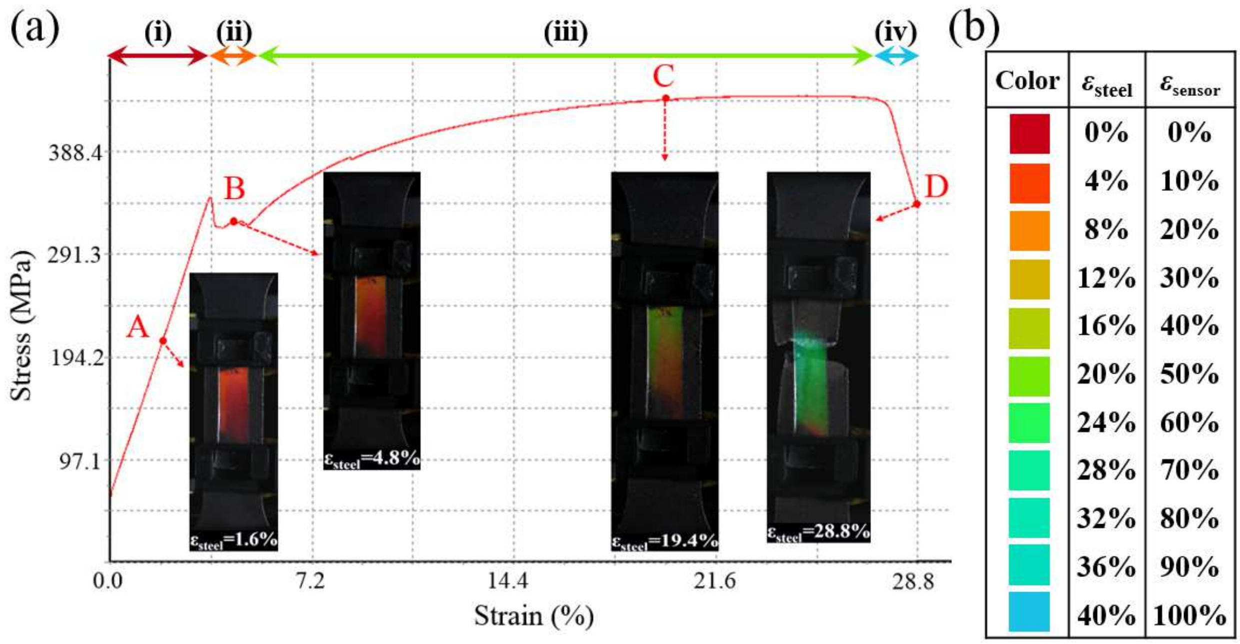

Fig. 5 MPC film as a mechanochromic strain sensor for visualizing strain evolution of low carbon steel in tensile test. **a** Stress-strain relationship and photographs of stretched low carbon steel with sensor on surface. **b** Relationship between sensor color, strain of sensor (ε_{sensor}) and strain of low carbon steel (ε_{steel})

Fig. 5a shows that in the whole process of tensile test, the stress-strain relationship of low carbon steel can be divided into four stages: (i) elastic stage, (ii) yielding stage, (iii) strengthening stage and (iv) necking stage. The inserted photographs in **Fig. 5a** exhibit the color change of the sensor and the deformation of the tensile specimen in the four stages. It can be seen that with the increasing strain, the tensile specimen is stretched in a vertical direction. After the strengthening stage, the deformation concentrates in the necking region, and then the cross-sectional area of this region shrinks sharply until the tensile specimen is broken. Meanwhile, the structural color of the sensor changes continuously from red to blue corresponding to the evolution of strain. And the sensor shows red, orange, green and blue color at point A, B, C and D which is respectively taken from the four stages.

Absolute deformation of the tensile specimen (Δl_{steel}) is equal to that of the sensor (Δl_{sensor}), so the following equations can be established:

$$\varepsilon_{\text{steel}} = \frac{\Delta l_{\text{steel}}}{l_{\text{steel}}} = \frac{\Delta l_{\text{sensor}}}{l_{\text{steel}}} = \varepsilon_{\text{sensor}} \times \frac{l_{\text{sensor}}}{l_{\text{steel}}} = 0.4\varepsilon_{\text{sensor}} \tag{2}$$

Based on equation (2), the relationship between sensor color, $\varepsilon_{\text{sensor}}$ and $\varepsilon_{\text{steel}}$ can be depicted in **Fig. 5b**. It can be seen that the strain of low carbon steel can be judged using the color indicated by the sensor. Note that when the sensor tends to be blue, the low carbon steel is on the verge of breakage and extremely dangerous.

4. Conclusions

A MPC film with grating nanostructure is prepared by inverted nanoimprint lithography. The film enables reversible color shift in the full visible range as it is stretched up to a strain of 100% that is greatly increased compared with previous studies. The MPC film is proven to be responsive to strains in direction both parallel and perpendicular to the grating. Comparison between mechanochromic properties of the film under two strains indicates that the film under strain in a direction along the grating is more suitable for visual sensing applications. The film is used as a mechanochromic strain sensor to visualize strain evolution of a low carbon steel in the tensile test. Four stages in stress-strain relationship and approximate strain of low carbon steel can be judged using the color indicated by the sensor, and accurate strain could be calculated using the quantitative relationship $\lambda = -128\varepsilon_x + 633$. The proposed

MPC film may pave the way for the development of mechanochromic strain sensors for monitoring strain of materials in real time by colors.

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Declarations

Funding

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Conflicts of interest/Competing interests

The authors declare that they have no conflict of interest.

Availability of data and material

The datasets used or analysed during the current study are available from the corresponding author on reasonable request.

Code availability

Not applicable.

Authors' contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Rui Zhang and Zhiyu Yang. Software operation and literature investigation were performed by Xu Zheng and Yanju Zhang. The manuscript was written by Rui Zhang and Qing Wang and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Supplementary Information

Journal of Materials Science: Materials in Electronics

Large-strain and Full-color Photonic Crystal Films as Mechanochromic Strain Sensors

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1. Detection of mechanochromic properties of MPC film under ε_x

In order to detect mechanochromic properties of the MPC film under ε_x, the deformation and structural color detection platform was built, as shown in **Fig. S1**. In the figure, ① is MPC film with vertical grating, ② is digital single lens reflex camera, ③ is incident light source, ④ is 3D-printed angle measurement instrument, ⑤ is digital vernier caliper, ⑥ is notebook computer, ⑦ is fiber optic spectrometer. Firstly, the MPC film with vertical grating was fixed between the two outer measurement claws of digital vernier caliper using clamps, and it was necessary to ensure that the film was in a flat state without deformation. Then vertical height of the film, the camera, and the incident light was adjusted to be in the same horizontal plane. And horizontal position of the camera was adjusted so that the included angle between the normal line of film and the outgoing light was 0°. Imaging area of the film is the largest at this point, which is conducive to the detection effect. Horizontal position of the incident light was also adjusted so that the included angle between the normal line of film and the incident light was 52°. Structural color of the film is red at this point, and red is the initial structural color required by this experiment. Finally, mechanical stretching was applied to the film by the digital vernier caliper with a length increment of 1 mm each time, and real-time structural color was recorded by the camera during stretching. Simultaneously, the fiber optic spectrometer was used to measure reflection spectra of the film.

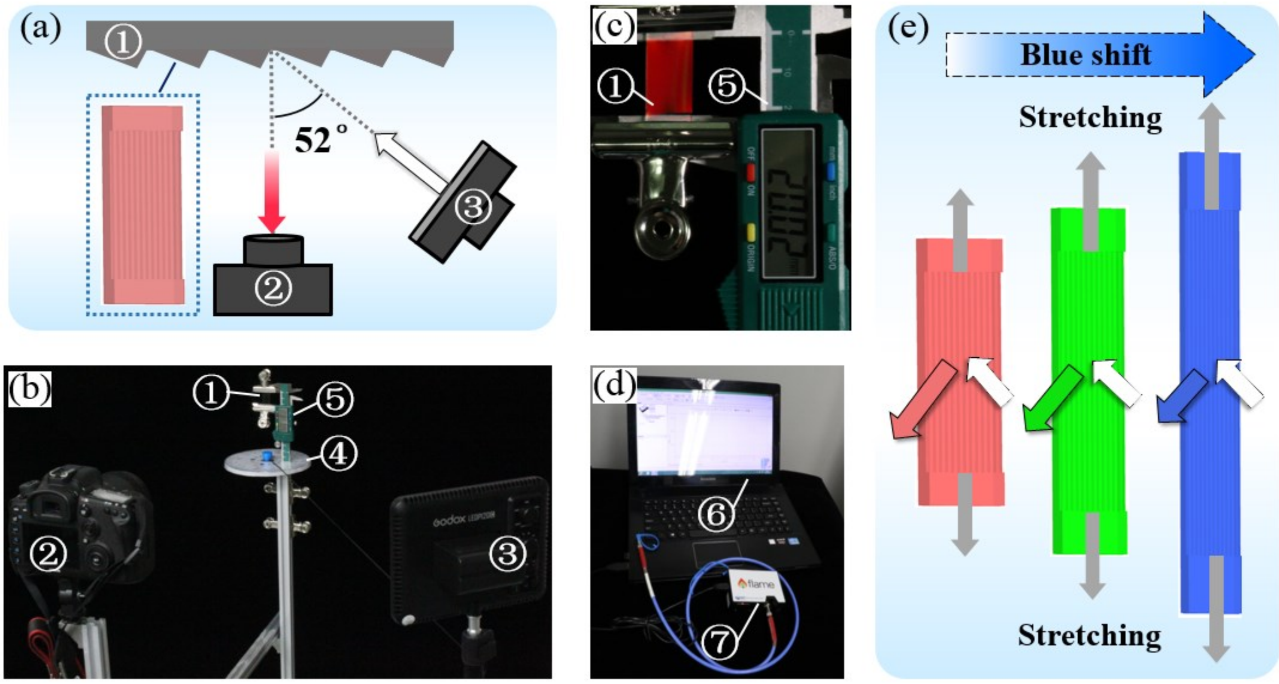

Fig. S1 Deformation and structural color detection platform for studying mechanochromic properties of MPC film under ε_x. **a** Schematic of detection devices (top view). **b** Photograph showing detection devices. **c** Photograph showing the film fixed to the digital vernier caliper (initial state). **d** Spectra measurement device. **e** Schematic of mechanical stretching experiments for mechanochromic properties of the film

2. Detection of mechanochromic properties of MPC film under ε_y

In order to detect mechanochromic properties of the MPC film under ε_y, the other deformation and structural color detection platform was built, as shown in **Fig. S2**. In the figure, ① is MPC film with horizontal grating, and the other devices are the same as in **Fig. S1**. The first step is still that the MPC film with horizontal grating was fixed to the digital vernier caliper without any deformation. And horizontal position of the camera was adjusted so that the included angle between the normal line of film and the outgoing light was 0°. Then the incident light was moved on the horizontal track so that the included angle between the normal line of film and the incident light was 31°. Structural color of the film is blue at this point, and blue is the initial structural color required by this experiment. The mechanical stretching method is the same as that of the MPC film with vertical grating.

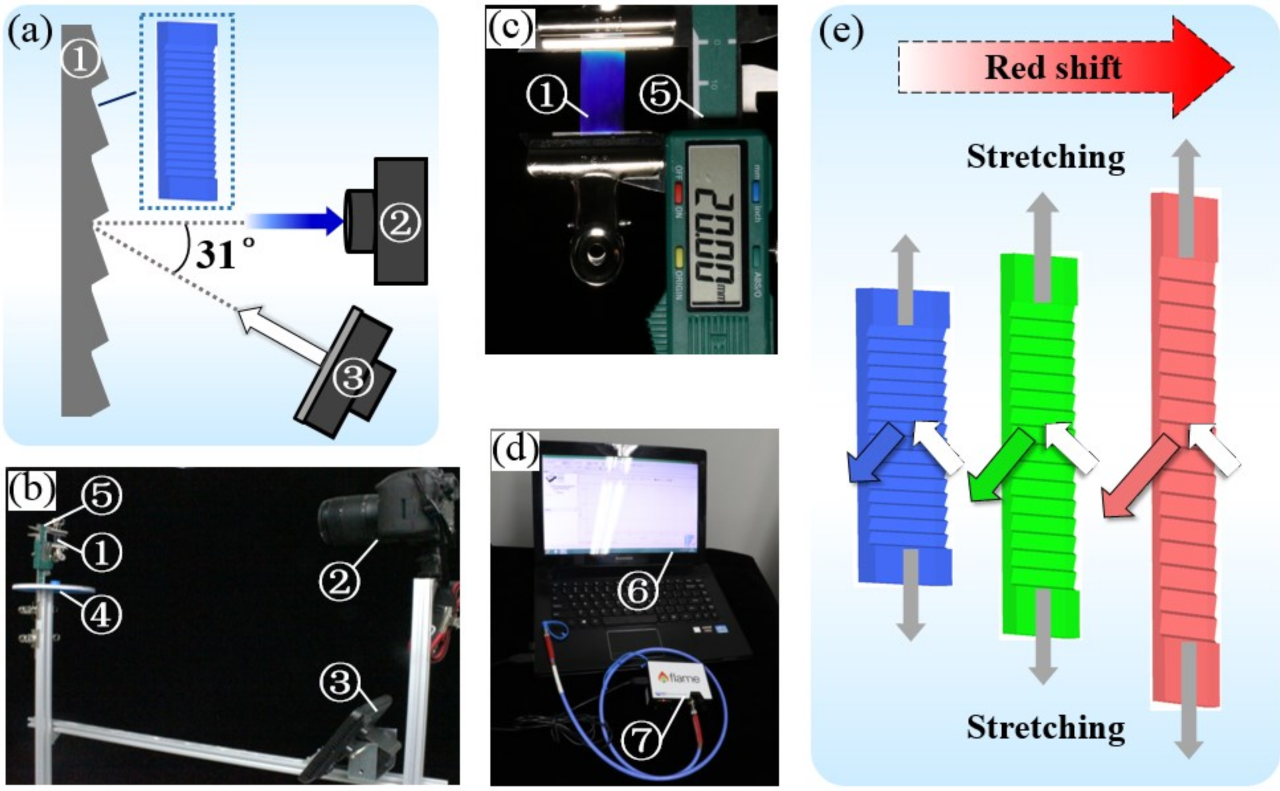

Fig. S2 Deformation and structural color detection platform for studying mechanochromic properties of MPC film under ε_y. **a** Schematic of detection devices (side view). **b** Photograph showing detection devices. **c** Photograph showing the film fixed to the digital vernier caliper (initial state). **d** Spectra measurement device. **e** Schematic of mechanical stretching experiments for mechanochromic properties of the film

3. Application as mechanochromic strain sensor for monitoring tension test on low carbon steel

In order to demonstrate application of the mechanochromic strain sensor based on MPCs, the deformation and structural color demonstration platform was built, as shown in **Fig. S3a**. The mechanochromic strain sensor was clamped on the surface of low carbon steel specimen, as shown in **Fig. S3b**, and the specimen was stretched using a microcomputer controlled electronic universal testing machine. The tensile speed of the universal testing machine was controlled at 2 mm/min. With the increasing tensile strain of the low carbon steel, the changing process of structural color of the sensor was recorded by a digital single lens reflex camera under the incident light. And the stress-strain relationship of low carbon steel during the whole stretching process was plotted by the microcomputer which controlled the universal testing machine.

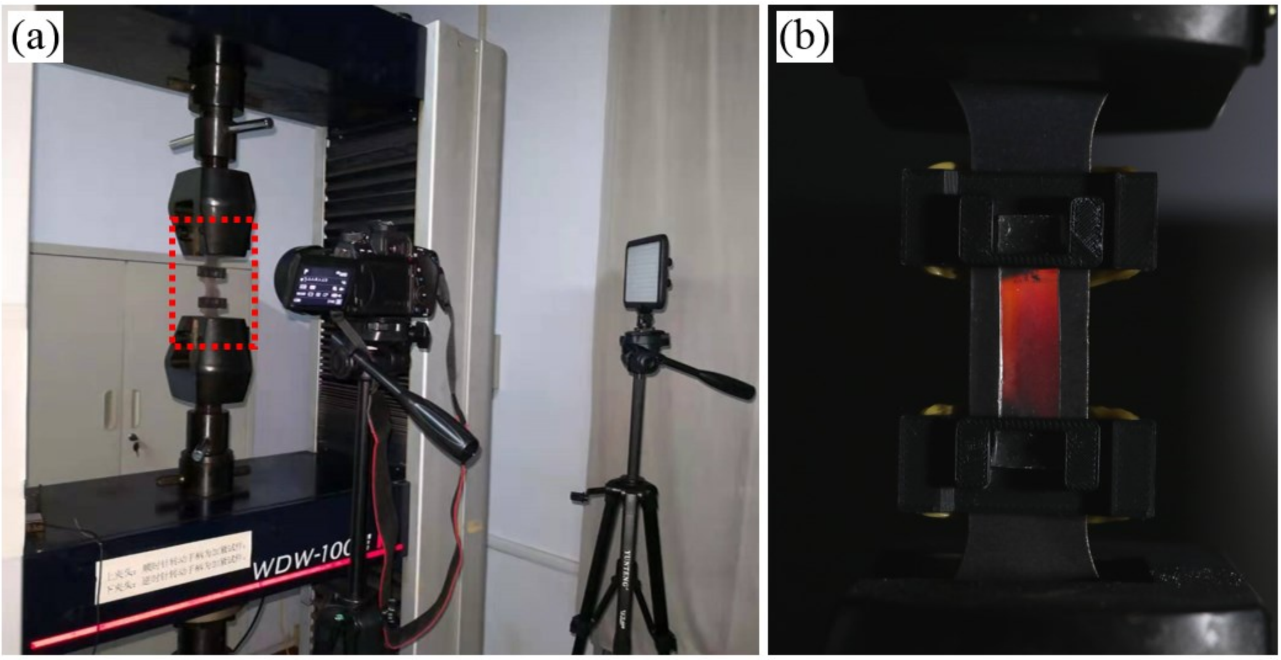

Fig. S3 Deformation and structural color demonstration platform for monitoring tension test on low carbon steel. **a** Photograph showing experimental devices which consist of a microcomputer controlled electronic universal testing machine, a digital single lens reflex camera, and a LED light panel providing the incident light. **b** Photograph showing the low carbon steel specimen with the sensor, which is a larger view of the image in dashed frame in **Fig. S3a**

Figures

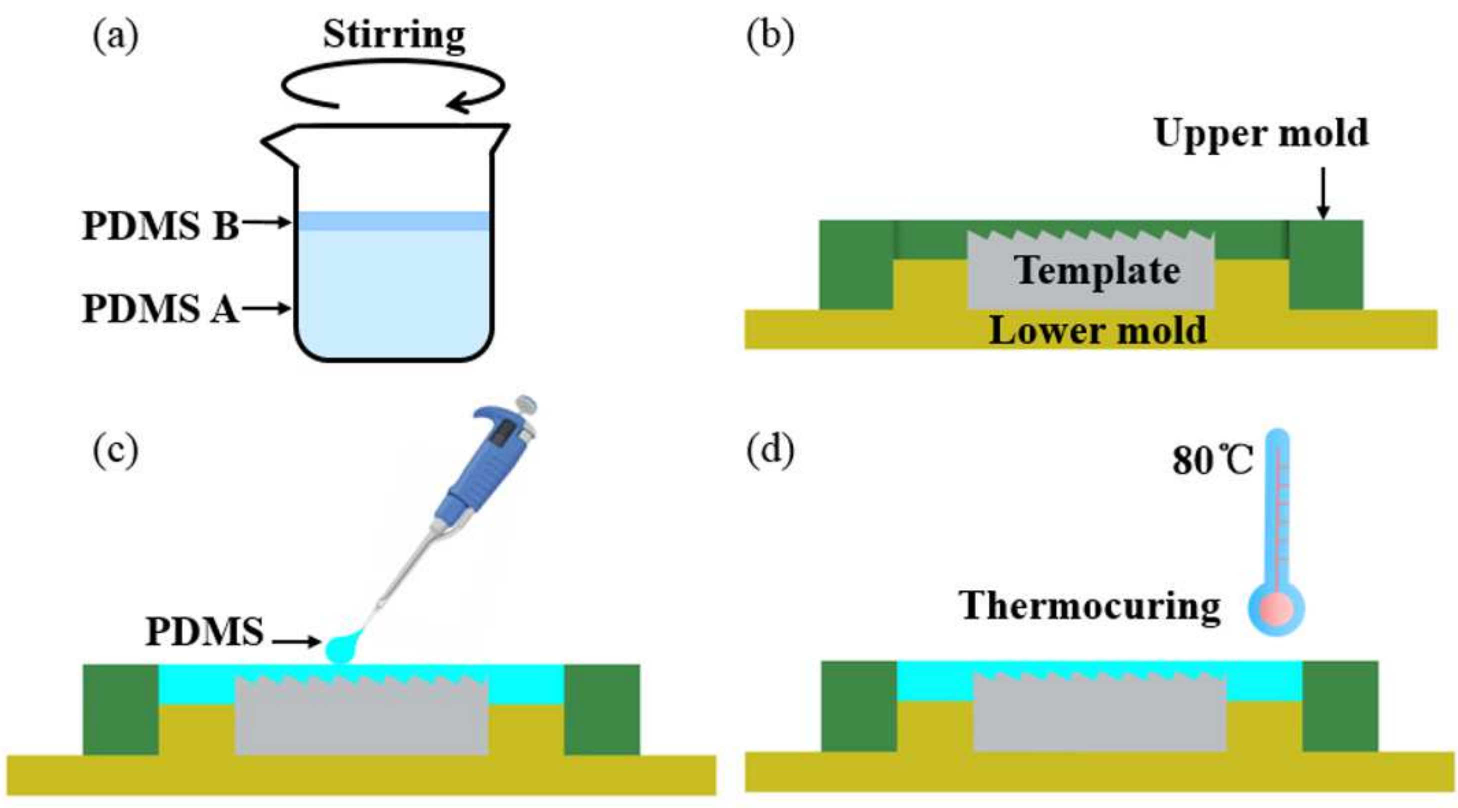

Figure 1

Schematic fabrication process of MPC film. a Preparation of liquid PDMS mixture. b Sectional view of a combination of lower mold, template and upper mold. c Filling of template and molds with PDMS. d Thermocuring of PDMS

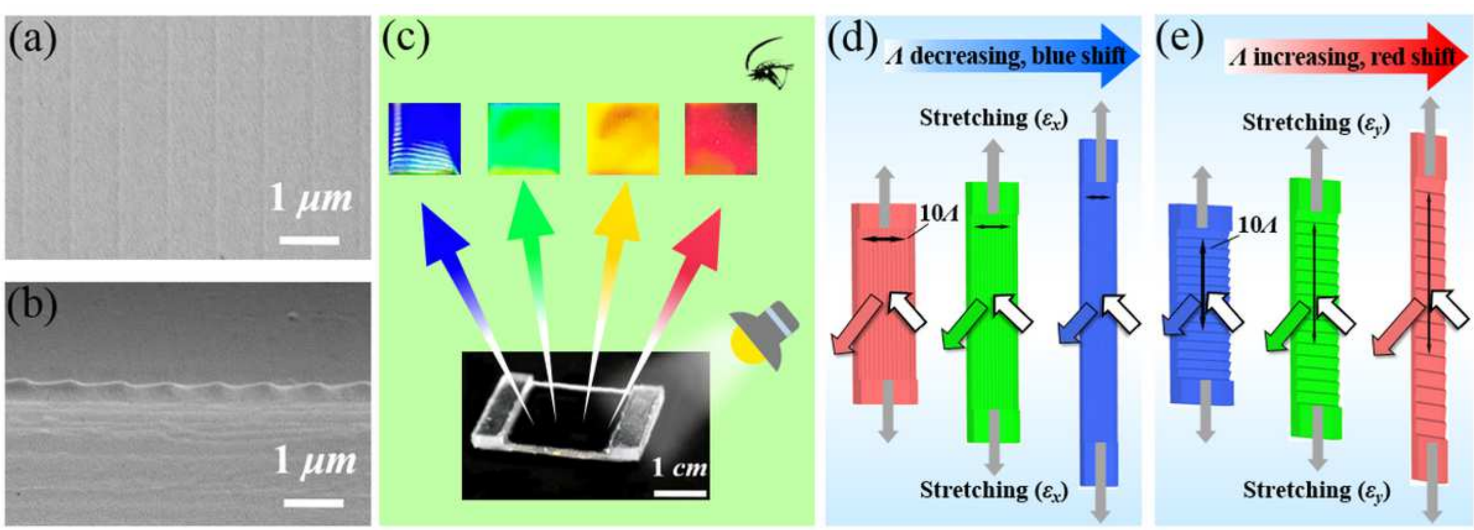

Figure 2

Characterization of MPC film and mechanochromism mechanism. a SEM top view and b cross-section view of the MPC film. c Photographs of the MPC film when observed from different viewing angles. Schematic of mechanochromic mechanism of the MPC film for tensile strain d parallel to and e perpendicular to the grating direction (Λ is structural period of the grating)

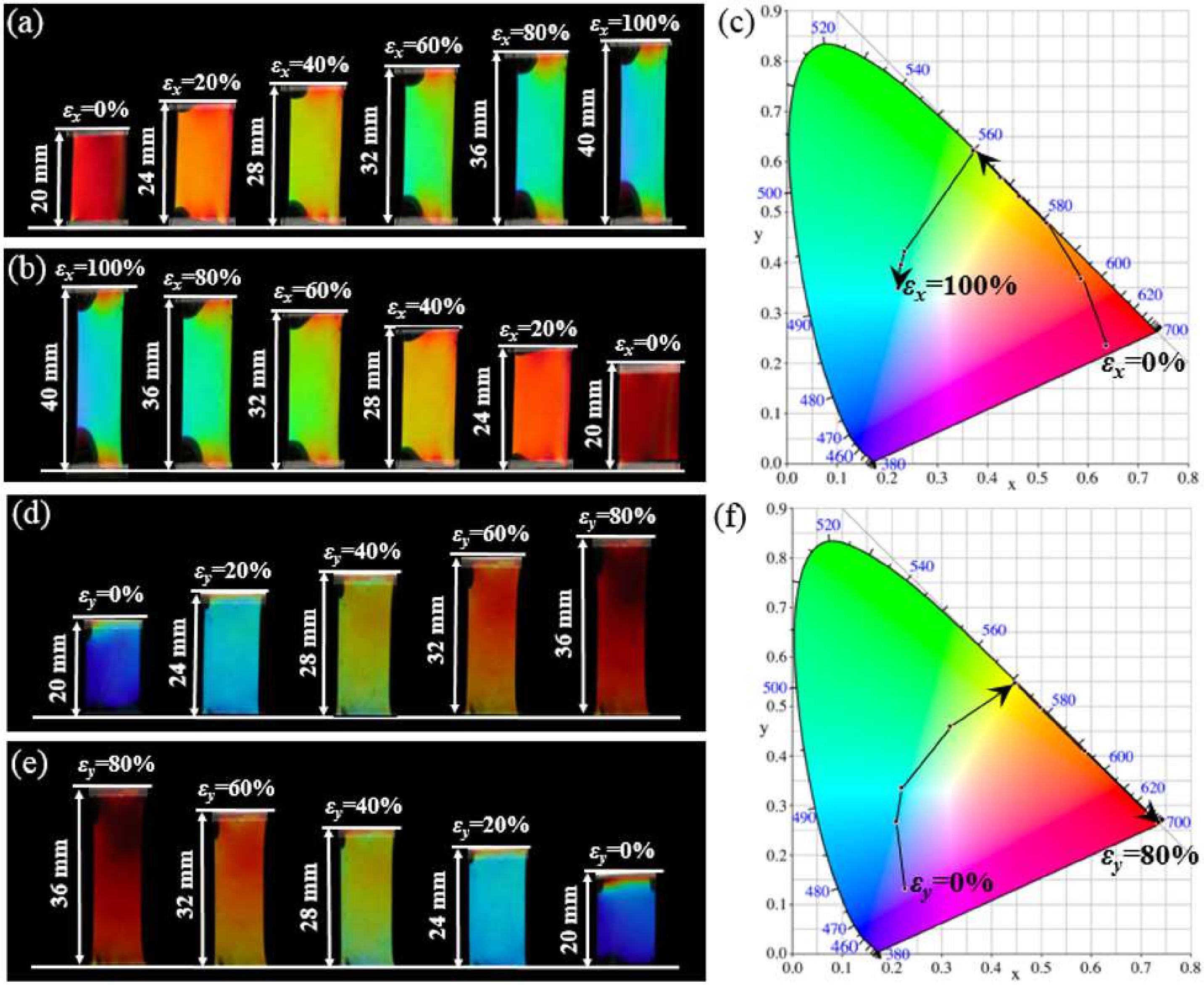

Figure 3

Mechanochromic properties of the MPC film. Photographs showing a blue-shift and a red-shift of structural color along with increasing a εx and d εy, the reversible color change of the film with releasing b εx and e εy. Mechanochromic states with increasing c εx and f εy in the CIE 1931 chromaticity diagram

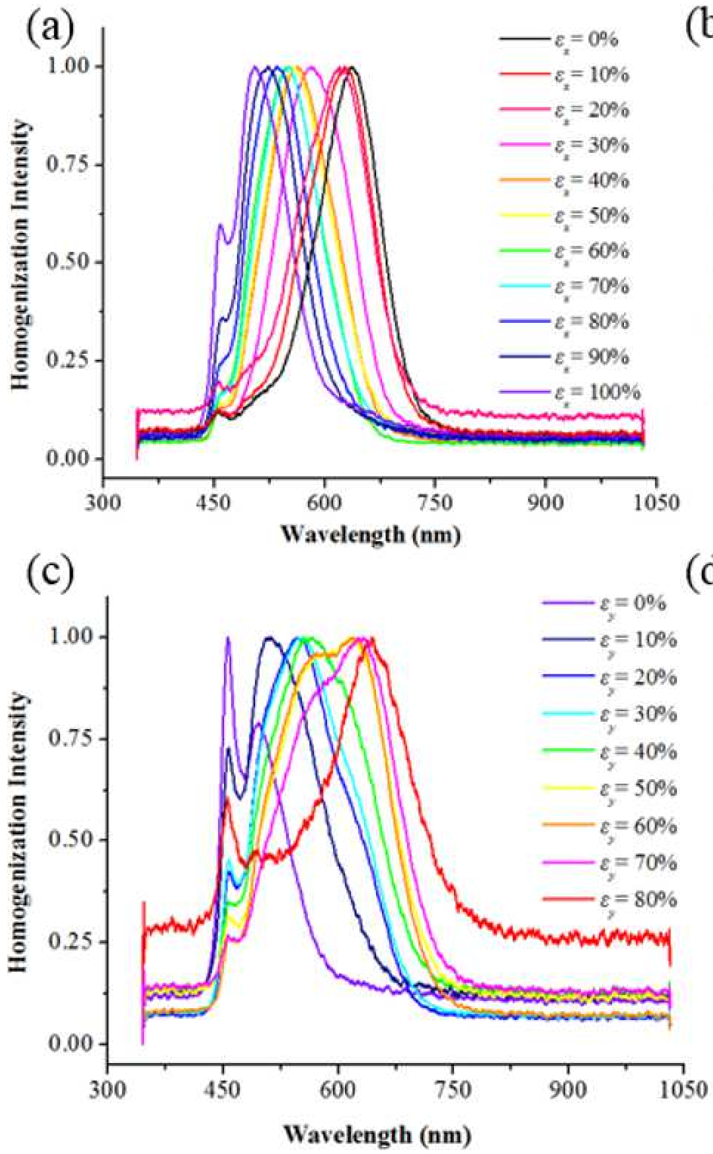

Figure 4

Relationship between the reflectance spectra and the strain. Reflectance spectra of the MPC film for increasing a εx and c εy. λ as a function of b εx and d εy

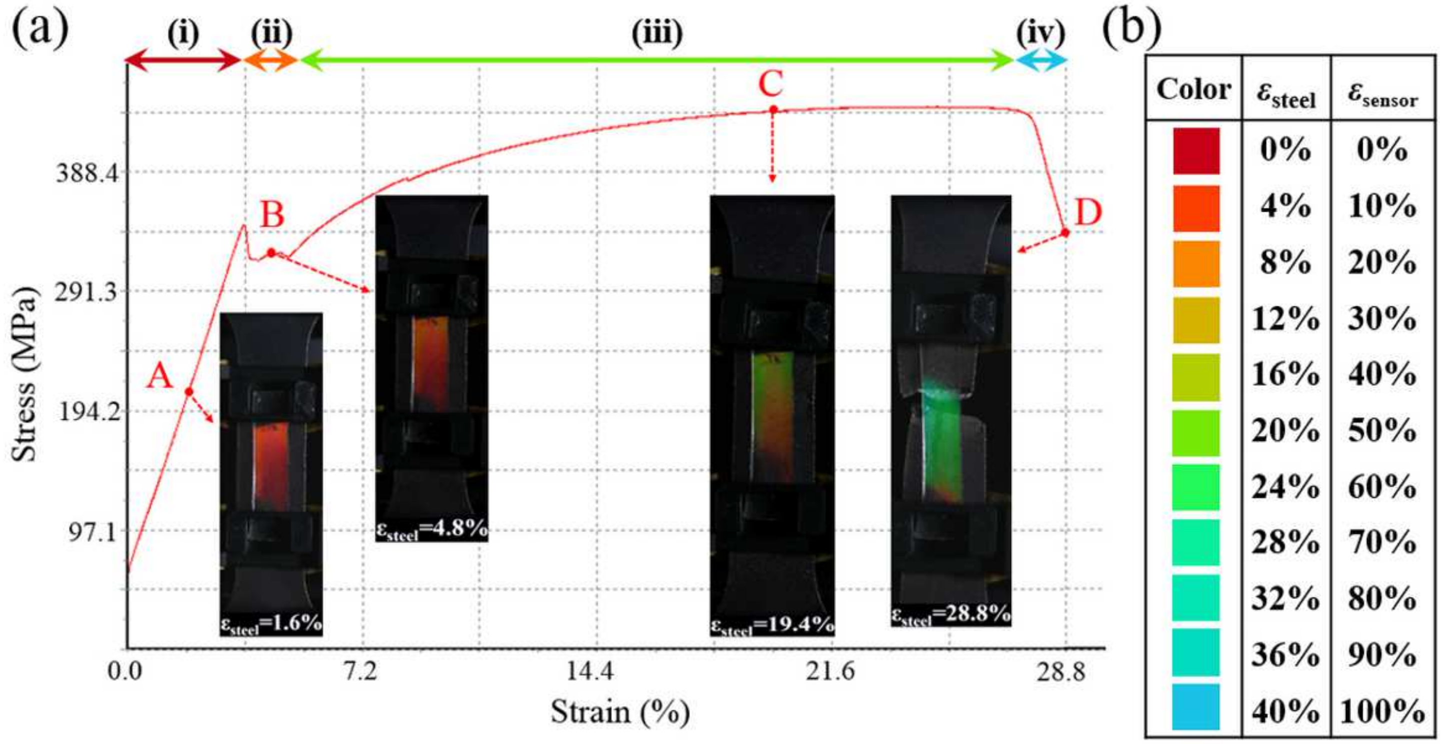

Figure 5

MPC film as a mechanochromic strain sensor for visualizing strain evolution of low carbon steel in tensile test. a Stress-strain relationship and photographs of stretched low carbon steel with sensor on surface. b Relationship between sensor color, strain of sensor (εsensor) and strain of low carbon steel (εsteel)