

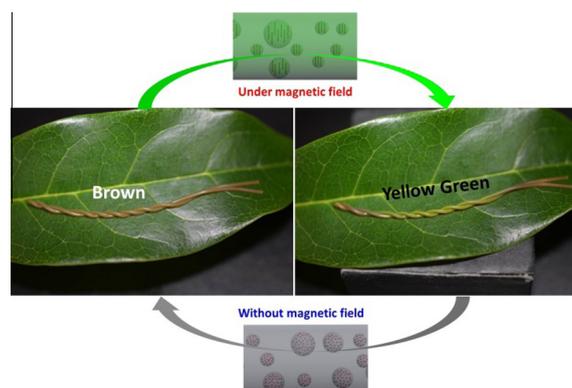
## Regular Article

## Facile fabrication of magnetically responsive PDMS fiber for camouflage

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## GRAPHICAL ABSTRACT

A new type of photonic crystal PDMS fiber exhibits tunable structural color upon exposure to external magnetic field.



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## ABSTRACT

A new type of photonic crystal PDMS fiber which exhibits tunable structural color upon exposure to external magnetic field is described in this article. The novel magnetic field responsive fiber was prepared from embedding ethylene glycol droplets (containing Fe<sub>3</sub>O<sub>4</sub>@C nanoparticles) into PDMS. In the presence of an external magnetic field, Fe<sub>3</sub>O<sub>4</sub>@C nanoparticles which dispersed in ethylene glycol droplets formed one dimensional chain-like structures along the magnetic field. As a result, the color of the fiber changes to yellow green. By contrast, when the magnetic field was removed, the color of the fiber will disappear and display its original color. Moreover, this novel PDMS fiber has good mechanical properties and could keep its color under a fixed magnetic field no matter it was stretched or squeezed. This study is expected to have some important applications such as none-powered and functionalized fibers for camouflage.

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## 1. Introduction

Camouflage has drawn more and more interests due to their special properties such as making animals or objects

invisible around the surrounding environment. Many researchers have done some excellent works in this field, one of the most inconceivable works is the fabrication of invisibility cloaking [1–7], in which a device is used to render an object invisible to incident radiation. The device achieves invisibility often by bending the ray trajectory around the object. However, the problem is that the perfect invisibility is unachievable and the device cannot hide itself. Therefore, there also

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need some new strategies to achieve the purpose of camouflage.

Many creatures in nature, such as chameleon, octopus and cuttlefish, are considered to be the great disguiser of the nature because of their capability to rapidly change their body's color in response to the surrounding environment. The reason that these creatures achieve camouflage arises from the translocation of pigments or a rearrangement of reflective units within a large number of chromatophores [8]. Recent studies shown that chameleons shift their color through active tuning of a lattice of guanine nanocrystals within a superficial thick layer of dermal iridophores. By combining two superimposed populations of iridophores, it achieves efficient camouflage and dramatic display [9].

Inspired by the nature creatures, researchers have paid their attention to responsive photonic crystals in order to obtain chromatic transitions. Responsive photonic crystals (RPCs), which tuned their structural colors by external stimuli, have been widely researched in the past two decades. The RPCs display different colors under the stimulation of temperature [10–12], ions [13–15], pH [16], mechanical force [17,18], electrical field [19] and so on, which makes them hold great application in camouflage. However, few works have been done in this field and many challenges exist in developing responsive photonic crystals, including limited tunability of the band gap, a slow response to external stimuli, incomplete reversibility, and difficulty of integration into existing photonic devices [20]. In order to solve these problems, magnetically RPCs have been widely studied in the past decades [21–23]. Compared to the other RPCs, magnetically RPCs can rapidly responsive to magnetic field through its magnetic components incorporated into one dimensional chain-like photonic crystal structures. Because of its fast responsive to the external magnetic field, many applications have been developed, such as printing [24,25], humidity sensor [26,27], anti-counterfeiting materials [28]. However, the conventional film shape of RPCs makes them not suitable for the application of wearable electronics. To this end, some researchers have put their attention to the photonic-crystal-based structurally colored fibers (SCFs) [29–31]. These SCFs are fabricated through assembling colloidal crystals on the surface of the fibers. But the combination between colloidal crystals and the surface of the fiber is unstable, leading to the photonic structures which assembly on the surface of the fibers are easily broken.

Herein, an efficient strategy for the preparation of magnetic field responsive (MFR) fiber is described. The MFR fiber was made of embedding EG droplets which contain  $\text{Fe}_3\text{O}_4\text{@C}$  nanoparticles into PDMS fiber in a microtubule. Then another PDMS was coated on the surface of the fiber after it was taken out from the microtubule. The color of the MFR fiber which arises from the assembly of  $\text{Fe}_3\text{O}_4\text{@C}$  nanoparticles under magnetic field can quickly switched between brown and yellow green. Moreover, the novel PDMS fiber also has good mechanical properties, the fiber will keep its color regardless of it was stretched or squeezed under a fixed magnetic field. In addition, the MFR fiber can be woven into fabrics for potential applications such as detection and camouflage.

## 2. Experimental

### 2.1. Chemicals and materials

Ferrocene ( $\text{Fe}(\text{C}_5\text{H}_5)_2$ ,  $\geq 98\%$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ ,  $\geq 30\%$ ), acetone ( $\text{C}_3\text{H}_6\text{O}$ ,  $\geq 99\%$ ), ethylene glycol ( $\text{C}_2\text{H}_6\text{O}_2$ ,  $\geq 99\%$ ) were purchased from Shanghai Chemical Factory, China. Silicone liquid (10 cSt) and PDMS (Sylgard 184) were purchased from Dow Corning, PDMS (Sylgard 184) was supplied as a kit containing two separate components: the base material (part A) and the curing agent (part B). All the chemicals were of analytical reagent grade and used directly without any further treatment.

### 2.2. Synthesis of $\text{Fe}_3\text{O}_4\text{@C}$ nanoparticles

$\text{Fe}_3\text{O}_4\text{@C}$  colloidal nanoparticles were synthesized using a high-temperature hydrolysis reaction procedure that had been reported previously [22]. In a typical experiment, ferrocene (0.50 g) was dissolved in acetone (60 ml). After intense sonication for 15 min, hydrogen peroxide (2.0 ml) was slowly added to the above mixture solution and was vigorously stirred for 30 min by magnetic stirring. Then the precursor solution was transferred to a Teflon-lined stainless autoclave with a total volume of 80 ml and was heated to and maintained at 180 °C. After 72 h, the autoclave was cooled naturally to room temperature. The products were washed by acetone three times and re-dispersed in ethylene glycol (EG) solution as a concentration of 10 mg/ml for the further use.

### 2.3. Fabrication of magnetic field responsive PDMS fibers

The preparation of magnetic field responsive PDMS fiber is illustrated in the Supporting Information (Scheme S1). First, 1.0 ml above homogeneous sample solution was mixed with 4.0 g PDMS precursor (the base material (part A) diluted by silicone liquids with the ratio of 4:1) and 0.4 g PDMS curing agent Part-B respectively. After complete mixing, the mixture suspension was left at room temperature for 1 h in order to minimize bubble formation. Then the mixture suspension was injected into a Teflon tube by using a syringe and curing it at 60 °C for 1 h. Afterwards, the PDMS fiber was taken out and immersed into PDMS precursor (part A and part B with the ratio of 10:1), followed by curing it for another 2 h at 60 °C.

### 2.4. Characterization

Digital photos were obtained by a digital camera (Nikon D7000, Japan). TEM images were obtained using a transmission electron microscope (JEOL-2100F, Japan). Scanning electron microscopy was recorded using desktop scanning electron microscopy (Phenom G2 pro, America). X-ray diffraction pattern was characterized by X-ray diffractometer (XRD, Rigaku D/max2550 V X-ray diffractometer using  $\text{Cu K}\alpha$  irradiation ( $k = 1.5406 \text{ \AA}$ ). Reflection spectra of the magnetic responsive PDMS fibers were obtained using a fiber-optic spectrometer (G2000-Pro-Ex, China). The incident light was aligned perpendicular to the film for all the optical measurements, and optical micrograph were obtained by an optical microscopy (XPF-550, China) mounted on a CCD camera (TCC, 3.3 ICE, China).

## 3. Results and discussion

The fabrication of MFR fiber relies on some magnetic responsive colloidal nanocrystal clusters. Herein,  $\text{Fe}_3\text{O}_4\text{@C}$  superparamagnetic colloidal nanocrystal clusters (SCNCs) which were dispersed in EG solution were used to fabricating MFR fiber. The  $\text{Fe}_3\text{O}_4\text{@C}$  SCNCs suspension displayed brown color in the absence of an external magnetic field. However, when an external magnetic field was applied, the color of the suspension changes to yellow green<sup>1</sup>, as shown in Fig. 1a and b. According to previous studies [22], this is because one dimensional chain-like structures formed in the suspension under an external magnetic field. Fig. 1c shows the corresponding TEM image of  $\text{Fe}_3\text{O}_4\text{@C}$  CNCs, it can be observed that the  $\text{Fe}_3\text{O}_4\text{@C}$  CNCs have a core-shell structure and the diameter of these particles is about 155 nm. Fig. 1d shows the X-ray diffraction patterns of  $\text{Fe}_3\text{O}_4\text{@C}$  SCNCs. From Fig. 1d,  $\text{Fe}_3\text{O}_4$  (JCPDS file 19-0629) and C peaks can be observed clearly.

<sup>1</sup> For interpretation of color in Fig. 1, the reader is referred to the web version of this article.

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