SCIENTIFIC REPORTS

OPEN

SUBJECT AREAS:
PHOTONIC CRYSTALS
METAMATERIALS
MAGNETIC PROPERTIES AND MATERIALS
SELF-ASSEMBLY

Received
7 August 2012

Accepted
1 March 2013

Published
19 March 2013

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Invisible photonic printing: computer designing graphics, UV printing and shown by a magnetic field

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Invisible photonic printing, an emerging printing technique, is particularly useful for steganography and watermarking for anti-counterfeiting purposes. However, many challenges exist in order to realize this technique. Herein, we describe a novel photonic printing strategy targeting to overcome these challenges and realize fast and convenient fabrication of invisible photonic prints with good tenability and reproducibility. With this novel photonic printing technique, a variety of graphics with brilliant colors can be perfectly hidden in a soft and waterproof photonic-paper. The showing and hiding of the latent photonic prints are instantaneous with magnet as the only required instrument. In addition, this strategy has excellent practicality and allows end-user control of the structural design utilizing simple software on a PC.

Recently, photonic printing technique based on fabrication and manipulation of photonic structures has received increasing attention across science and engineering disciplines because of its applications in photonic circuits[1], on-chip photonic devices[2,3], reflective display units[4–7], outdoor signage and photonic sensors[8–14]. Various photonic printing strategies have been extensively investigated in order to realize the fast and convenient preparation of photonic prints with good tenability and reproducibility. Successful strategies include the self-assembly of colloidal crystals locally on a substrate[15–18], and the change of photonic structure by tuning the refractive index (n), lattice spacing (d), and crystal orientation (θ) in a specific region of the pre-made colloidal crystals or related composite materials[19–28]. Invisible photonic printing, an emerging photonic printing technique with tremendous advantages, is especially useful for steganography and watermarking for anti-counterfeiting purposes. However, it is still not easy to realize because of the many great challenges[29]. First, the mostly reported visible photonic printing creates photonic prints with a permanent contrast of reflection wavelength ($\Delta\lambda \neq 0$), while the invisible photonic printing requires the photonic prints with an initial zero contrast ($\Delta\lambda = 0$) and later a non-zero contrast ($\Delta\lambda \neq 0$) when the latent information is revealed. However, a large amount of responsive content on the print is required in order to obtain a large contrast under external stimuli. Consequently, this will lead to a large deviation from invisibility due to the gradually increased mismatch of refractive index or lattice constant[30]. Secondly, the obtained latent prints must have long-lasting stability, excellent reversibility and the ability of being quickly released with a convenient and non-toxic means such as heating or magnetic field.

Recently, we have been engaged in ongoing efforts aimed at the development of novel photonic materials with special functions[28,31,32]. For example, we reported the fabrication of a novel photonic wordpad from fast magnetically induced self-assembly of superparamagnetic colloidals, which is fixed inside a polyacrylamide glycol gel matrix through an instant radical polymerization[28]. Herein, we demonstrate a novel photonic printing strategy targeting to overcome the above challenges and realize fast and convenient fabrication of invisible photonic prints with good tenability and reproducibility. A variety of graphics with brilliant colors can be perfectly hidden in a soft and waterproof photonic-paper (P-paper) utilizing this novel technique. When the functionalized P-paper is put into a vertical magnetic field, only the prints with magnetic-responsive photonic activities are expected to be changed. The pre-hidden patterns will be revealed due to vastly different reflection wavelength contrast between the background and the print. From the perspective of practical printing technique, this strategy is of great interest due to its superior features, including the ability to (1) generate invisible patterns with magnetic showing effect, (2) obtain end-user control of structural design utilizing simple software on a PC, and (3) allow fast, convenient and scalable production at low-costs. Moreover, the adaptation of the P-paper for invisible photonic printing

using common office laser printers would provide a new strategy for pattern-on-demand invisible photonic prints. This will find great applications in both scientific investigations and daily life, ranging from authentic identification systems to information storage medium.

Results

The fabrication strategy of the invisible photonic prints is outlined in Figure 1. Large-area, shape-controllable and low-cost P-paper (Figure 1a) was fabricated according to literature procedure[33]. The P-paper is a three-phase material system consisting of carbon-encapsulated super-paramagnetic colloidal nanoparticles (CNPs), glycol and commercially available polydimethylsiloxane (PDMS) with physical and chemical stability. The pre-made carbon-encapsulated super-paramagnetic colloidal nanoparticles have magnetite/carbon core/shell structure with negatively charged groups (carboxyl) on the surface (Supporting information).

In a typical preparation process (Figure 2a), a certain amount of CNPs are dispersed in glycol solvent to form a homogeneous solution under ultrasonication, and subsequently mixed with PDMS precursor Part-A (Sylgard184 elastomer base) and PDMS precursor Part-B (Sylgard184 curing agent) using mechanical stirring. The viscous mixture was poured into a container with a specific shape (2.0 cm × 2.0 cm × 1 cm) and cured at 60°C for 2 hours to produce a dark brown polymer matrix. It should be noted that the size of the P-paper only depends on the dimensions of the container and the amount of the liquid precursor. Therefore, fabrication of the P-paper on a larger scale is completely feasible with a larger container and more precursors. After the curing reaction, the glycol solution containing the CNPs can form very stable emulsion-like droplets at micron size throughout the solidified PDMS matrix (Figure 2a, 2b and Figure S7a, S7b). Without an applied external magnetic field, the CNPs in the glycol micro-droplets are randomly dispersed and display a brown color, which is the intrinsic color of the CNPs (Figure 1a). However, under an external magnetic field, the CNPs in the glycol micro-droplets are immediately assembled to form one-dimensional photonic crystal structures along the magnetic field lines and strongly diffract visible light[28]. Consequently, brilliant optical diffraction colors are displayed (Figure 1b, 2a, 3 and Figure S5).

As a photomask, the common transparent slides with computer designed graphics (Figure 1c, 3a) were sealed on the surface of the P-paper under the UV-irradiation (Spectroline SB-100P, 365 nm, 4800 μW/cm^{-2}) and peeled off after exposure. According to literature reports[34–37], UV-irradiation will induce PDMS chain scissions, involving both the main chain and the side groups, ultimately leading to the formation of new Si-O-Si bonds. This silica-like structure is denser and more compact, resulting in the rupture of PDMS networks on the surface region. The formation of the CNPs one-dimensional structures in the glycol micro-droplets is the reason leading to the P-paper's brilliant colors under an external magnetic field[33]. The glycol liquid medium is required for the dynamic assembly of the CNPs. However, the rupture of PDMS networks in the UV-irradiated surface region will lead to the leakage of glycol solution. This will ultimately lead to the loss of magnetic-responsive photonic activity of the P-paper. Therefore, the exposed regions will permanently lose the magnetic-responsive feature after UV-irradiation, while the shielded regions will remain the original dynamic nature.

The optical microscopy images (Figure 2e–2h) clearly demonstrate that the cross-section of the surface layer of the UV-irradiated region no longer produced a color change under the induction of a vertical magnetic field. In contrast, the shielded regions can still strongly diffract brilliant optical color and the magnetic-responsive photonic activity is maintained. These images clearly illustrate that UV-irradiation can effectively destroy the magnetic-responsive photonic activity of the exposed region. The SEM images demonstrate an obvious change between the irradiated region (Figure S8a, b) and the shielded region (Figure S8c, d). After UV-irradiation, the PDMS polymer matrix in the exposed region was clearly wrinkled and tended to rupture. However, the PDMS polymer matrix in the shielded region was relatively smooth with no obvious cracks. In addition, the SEM image (Figure 2d) reveals many holes in the UV-irradiated surface regions and the microstructures of the PDMS were dramatically different from those unexposed PDMS (Figure 2c). The P-paper after UV-irradiation was further characterized by Fourier transform infrared spectrum (FTIR) to obtain additional information on the chemical transformation. The FTIR spectra (Figure S9) of the surface layer of the P-paper with different UV-irradiation time clearly indicate a noticeable decrease in the $-CH_3$ signals with increasing exposure time. Simultaneously, the shape of Si–O bond absorption peak was changed, indicating the structure transformation from CH_3–Si–O– fragments to –O–Si–O– network[34,37].

All the above results are fully consistent with our hypothesis that UV-irradiation will induce chain scissions of the PDMS polymer chains and rupture of the PDMS networks. As a result, the glycol solution leaked out from the glycol micro-droplets. As such, the required liquid medium, in which the dynamic assembly of CNPs occurred, no longer existed. This ultimately led to the loss of the magnetic-responsive photonic activity of the exposed region of the P-paper. To further confirm our hypothesis, two control experiments were carried out. In the first control experiment, all the conditions are the same as above except 0.5 g PDMS was used. The liquid

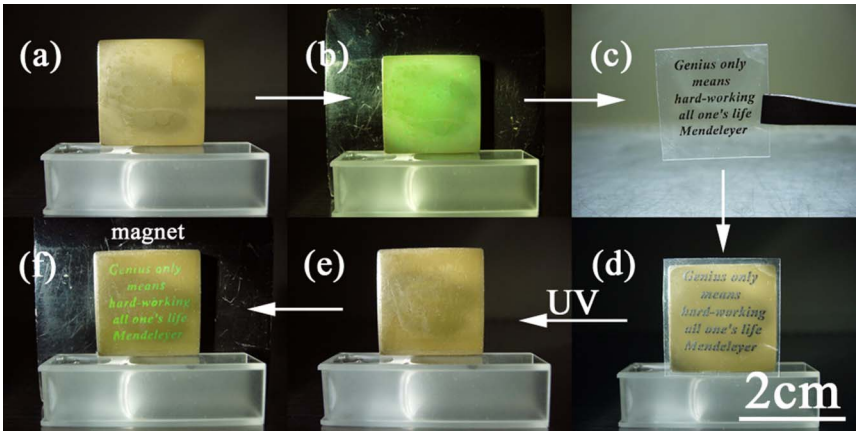

Figure 1 | Schematic illustration of the procedures to prepare invisible photonic prints (a, b) blank P-paper, (c) the common transparent slide with printed-graphics designed on a computer, (d) UV-irradiation of unshielded region, (e) removal of the shielding slide and (e, f) the invisible pattern can be revealed and reversibly hidden through the application and withdraw of a magnetic field.

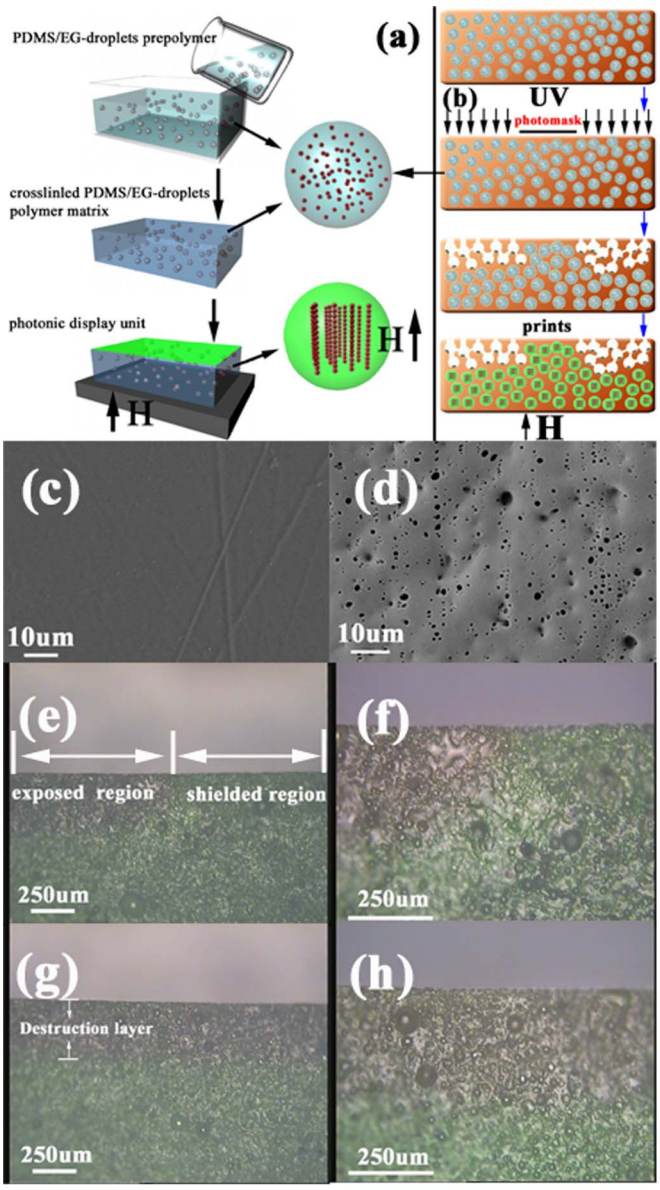

Figure 2 | (a) Schematic illustration the fabrication route of the blank P-paper, (b) Schematic illustrations of the mechanism of preparing the invisible photonic prints, (c, d) Scanning-electron micrographs of P-paper, (c): unirradiated region; (d): irradiated region, (e-h) Obtained optical microscopy images of a cross-section of the printed P-paper under a vertically aligned external magnetic field. The depth of the destruction layer by UV-irradiation is approximately 350 μm deep.

mixture will not be cured after curing at 60°C for 2 hours because it contains less PDMS. After 30 minutes UV-irradiation, the viscous mixture still underwent an instant color change under a vertical magnetic field (Figure S10). This shows that UV irradiation of PDMS will not generate any new chemicals to influence the dynamic assembly of CNPs in the glycol solution. In the second control experiment, a blank P-paper was completely covered by a common transparent slide and was UV-irradiated for 30 minutes. After the UV-irradiation, the P-paper still displayed brilliant color the same as that from the fresh P-paper (Figure S11). This indicates that the UV-irradiation-generated heat can not induce the loss of the magnetic-responsive photonic activity of the P-paper. These results further confirm our hypothesis that the loss of the magnetic-responsive photonic activity of the irradiated region is mainly due to the leakage of the glycol solution in the glycol micro-droplets in the solidified PDMS polymer matrix.

It is worth noting that the depth of the destruction layer by UV-irradiation is only about 350 μm deep and the deeper part still has the magnetic-responsive photonic activity (Figure 2e–2h). This is found to be independent of the UV exposure time in our experiments. The regular light-transmittance levels of the irradiated P-paper with different thickness are compared in Figure S12. There is an obvious decrease in the light transmittance of the P-paper in the visible range with increasing thickness. This is due to the intense light scattering induced by the more and more residual CNPs in the destructed layer of the irradiated region of the P-paper (Figure S7c, S7d). The light transmittance of the irradiated P-paper with thickness of 244 microns can only reach 0.1438% at a wavelength of 550 nm. This can perfectly explain the fact that even if the deeper part of the irradiated region of the P-paper still possesses the magnetic-responsive photonic activities, it can not participate in the reflection of visible range of light wavelength because the visible light cannot penetrate the destructed layer and reach the deeper parts of the P-paper. Therefore, when the treated P-paper was exposed to a vertical magnetic field, the great difference in reflection wavelength (λ) between the shielded region (the pattern) and the exposed region (background) is significantly shown (Figure 4) due to their obviously different magnetic-responsive photonic activity in the surface layer (Figure 2e–2h), and leads to a visible image (Figure 3c). This is an important finding and also the key to the work, because it provides an operable printing strategy that meets the requirements of reflection wavelength in hiding and showing mode, respectively. In addition, no new responsive content is introduced, and the change in surface morphology of the P-paper is negligible from a macro perspective. This results in a close to zero reflection wavelength contrast ($\Delta\lambda$) between the background and latent prints (Figure 4).

Discussion

To test the effectiveness of this novel invisible photonic printing strategy, various patterns were hidden in the P-paper (Figure 3). As shown in Figure 3b, these patterns were invisible at first due to the negligible reflection wavelength contrast ($\Delta\lambda$) between the background and latent prints (Figure 4). However, various brilliant patterns were visualized immediately when these printed P-papers were exposed to a vertical magnetic field (Figure 3c). The optical diffraction color depends on the average particle size of the CNPs included in the P-papers (Figure S1). In addition, the exchange between invisible-form and visible-form are highly reversible (schematic video-Supporting Information Available). This directly demonstrates the

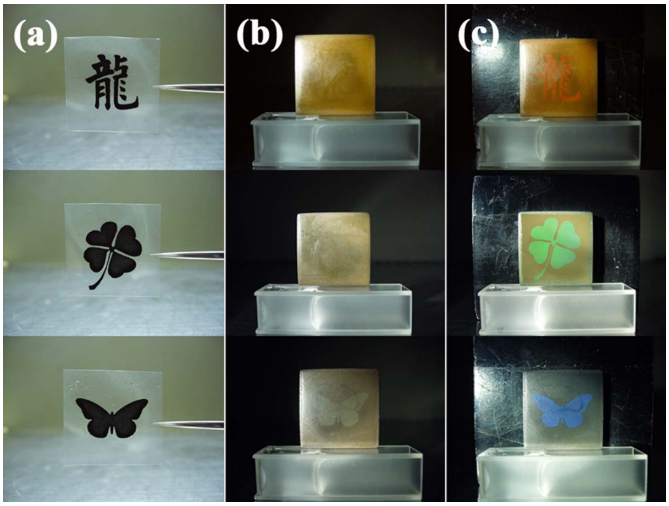

Figure 3 | (a) Photographs of slides with printed-graphics designed on a computer, (b) Photographs of latent graphics on P-papers and (c) Photographs of graphics revealed by a magnetic field.

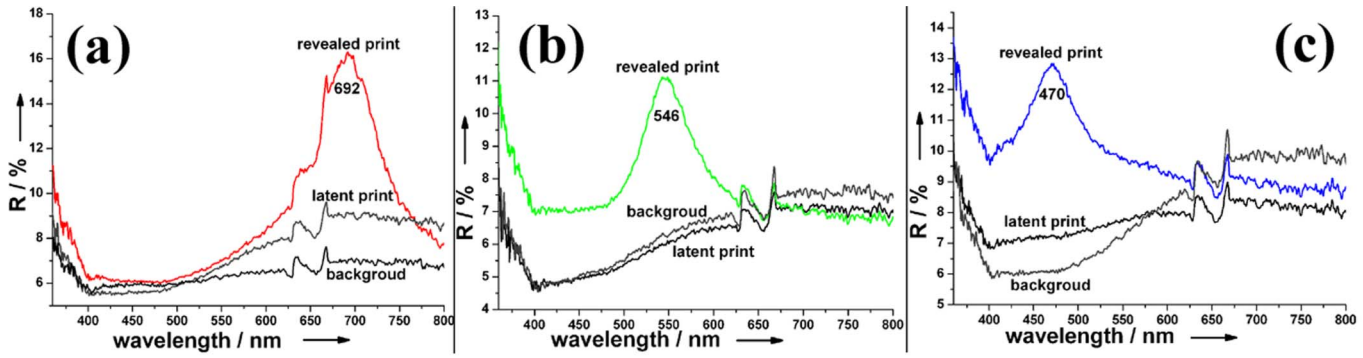

Figure 4 | Representative reflection spectra taken from latent prints, revealed prints and background in each of the printed photonic-paper containing carbon-capped superparamagnetic colloidals nanoparticles with average particle size (a) 200 nm, (b) 140 nm and (c) 125 nm.

good reversibility, stability as well as simplicity of this novel technique. These results indicate that this photonic printing technique is capable of fabricating invisible photonic prints, which can be visualized with brilliant optical diffraction colors under an external magnetic field. In addition, the P-papers with the printed patterns are also very stable: no apparent degradations in optical or mechanical properties were observed after more than three months at room temperature. At the time of submitting this article, these invisible photonic prints can still give high quality images as good as those obtained from freshly prepared prints. This observation is attributed to the long-term stability of the CNPs glycol solution, originating from the low ionic strength of the glycol solution and the non-electrostatic inter-particle repulsion of the one-dimensional photonic structure[33]. Furthermore, the obtained P-paper with invisible photonic prints possess excellent elasticity and can be folded while retaining the image fidelity and reversibility (Figure S6).

In addition, it is noteworthy that this novel invisible printing technique is also applicable to a handwritten-pattern printing method. Accordingly, a gel-ink pen was employed to handwrite "ustc 1958" as shown in Figure S13a. As expected, the letters and numbers were invisible after UV printing (Figure S13c), and the latent image appeared when the sample was put under a perpendicular magnetic field (Figure S13d). The invisible photonic prints also possess good reversibility and durability and the showing and the hiding of patterns can be achieved for many cycles through the application and withdraw of the magnetic field. These observations demonstrate that the invisible photonic printing can be applied to broader fields such as encrypting personal messages and watermarking for anti-counterfeiting purposes.

In summary, we have developed a novel photonic printing technique that enables fast and convenient preparation of invisible photonic prints with good tenability and reproducibility. The showing and hiding of the latent photonic prints is instantaneous, and the only required instrument is a magnet, which is easy to obtain and harmless to human body. The invisible photonic prints were found to be stable for at least three months at room temperature and can be used after prolonged storage. The color of the visualized prints can be controlled by adjusting the size of the superparamagnetic colloidal nanoparticles. In addition, this strategy allows end-user control of the structural design utilizing simple software on a PC and is also applicable to a handwritten-pattern printing method, which reveals the excellent flexibility and practicality of this novel invisible printing technique. With these superior features, it is reasonable to believe that this novel technique will attract more research interests across the science and engineering disciplines.

Methods

Synthesis of monodisperse carbon-encapsulated superparamagnetic colloidal nanoparticles with different average particle size. In a typical synthesis, ferrocene (0.30 g) was dissolved in acetone (30 ml). After intense sonication for 30 min, hydrogen peroxide (1.00, 1.50, and 2.00 ml) was slowly added into the above mixture solution, which then was vigorously stirred for 30 min with a magnetic stirring apparatus. After that, the precursor solution was transferred to the Teflon lined stainless autoclave with a total volume of 50.0 ml and then heated to and maintained at 210°C. After 72 h, the carbon-capped superparamagnetic colloidal nanoparticles with average particle size 200 nm, 140 nm, 125 nm were obtained respectively as shown in the Figure S1. Synthesized superparamagnetic colloidal nanoparticles were initially dispersed in acetone. Then these superparamagnetic colloidal nanoparticles were collected by magnetic separation, and re-dispersed in glycol solution (10 mg/ml) respectively for further use.

Fabrication of P-paper. According to previous report[33], 0.5 ml above homogeneous sample solution containing the carbon-capped superparamagnetic colloidal nanoparticles was mixed with 2.0 g PDMS precursor Part-A (Sylgard184 elastomer base) and 0.4 g PDMS precursor Part-B (Sylgard184 curing agent) respectively. After intense mechanical agitation for 5 min, the viscous mixture (take 1 ml) was transferred to a cubic container (2.0 cm × 2.0 cm × 1 cm), crosslinked 2 hours at 60°C to get an approximately 2000 μm thick P-paper (Figure 2a).

Fabrication of invisible photonic prints. A two-step process was used to prepare the invisible photonic prints. First, graphics designed on a PC were printed on a transparent slide with common office laser printer. Then, as a photomask, the printed slides were sealed to the surface of the prepared P-paper under the UV-irradiation for 30 minutes and peeled off after the irradiation. The entire preparation process is illuminated in Figure 1. An NdFeB permanent magnet with center magnetic field 0.05 T was used to generate a magnetic field, which was attached to the back of the treated P-paper to reveal the latent photonic prints.

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Acknowledgements

Financial support from the National Natural Science Foundation of China (21271163, U1232211 and J1030412) is grateful acknowledged.

Author contributions

H.B.H. and Q.W.C. conceived the research. Q.W.C. supervised the research. H.B.H. designed the experiments, analyzed and interpreted the results, and wrote the manuscript. C.L.C. contributed to its revision. Other authors also contributed extensively to the work presented in this paper.

Additional information

Supplementary information accompanies this paper at http://www.nature.com/scientificreports

Competing financial interests: The authors declare no competing financial interests.

How to cite this article: Hu, H. *et al.* Invisible photonic printing: computer designing graphics, UV printing and shown by a magnetic field. *Sci. Rep.* **3**, 1484; DOI:10.1038/srep01484 (2013).