Waveguiding in photonic crystals
Embedded cavities and waveguides in three-dimensional silicon photonic crystals

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To fulfil the promise that complete-photonic-bandgap (cPBG) materials hold for optoelectronics applications, the incorporation of three-dimensionally engineered defects must be realized. Previous attempts to create and characterize such defects were limited because of fabrication challenges. Here we report the optical and structural characterization of complex submicrometre features of unprecedented quality within silicon inverse opals. High-resolution three-dimensional features are first formed within a silica colloidal crystal by means of two-photon polymerization, followed by a high-index replication step and removal of the opal template to yield embedded defects in three-dimensional silicon photonic crystals. We demonstrate the coupling of bandgap frequencies to resonant modes in planar optical cavities and the first waveguiding of near-infrared light around sharp bends in a complete-photonic-bandgap material.

Photonic crystals (PhCs), or materials possessing a periodic modulation in their dielectric constant, have been proposed as media in which photons can be manipulated in a similar fashion to electrons in semiconductors. Specifically, PhCs can exhibit a complete photonic bandgap, or a range of frequencies over which light cannot propagate in any direction within the medium\(^1\)\(^-\)\(^3\). The unique optical properties of complete-photonic-bandgap (cPBG) materials provide the basis for numerous applications, such as low-threshold lasers\(^4\), low-loss waveguides\(^4\)\(^-\)\(^6\), on-chip optical circuitry\(^7\) and fibre optics\(^8\)\(^-\)\(^9\). For the majority of cPBG-based applications, functionality is provided through the precise, controlled incorporation of three-dimensionally engineered defects.

Since the advent of PhCs, much work has been devoted to harness their ability to three-dimensionally manipulate photons\(^10\)\(^-\)\(^11\). Of paramount importance has been the need to develop a means to incorporate high-quality, pre-engineered defects within a cPBG material. Defects disrupt the PhC lattice periodicity, creating states for otherwise forbidden bandgap frequencies. The defect geometry, composition and position can be engineered to design PhCs with tailored functionalities. For example, embedded resonant cavities and waveguides can enable the creation of low-threshold lasers and optical circuits\(^12\)\(^-\)\(^7\). Although exquisitely controlled defects have been designed and fabricated in two-dimensional PhCs, the complete confinement of light can only be achieved by extending the PBG into the third dimension. However, the realization of three-dimensional (3D) systems containing defined defect structures has presented a difficult set of fabrication and materials challenges. A review of the recent efforts to introduce defects in 3D PhCs operating at optical frequencies is presented in ref. 12.

Previous attempts to define defects in 3D PhCs have been limited in terms of the dimensionality and placement of the defects\(^12\). Also, defects have not been optically characterized in a material that has a cPBG (ref. 13). In this article, we introduce defects of arbitrary shape and position within a cPBG material and optically characterize these features. Light is guided around sharp bends in a 3D PhC, demonstrating the first waveguiding of near-infrared (NIR) light within a cPBG material. This represents a major milestone in the use of 3D PhCs for cPBG applications\(^10\).

RESULTS

Silicon inverse opals\(^16\)\(^-\)\(^17\) containing a variety of complex, high-resolution, multidimensional embedded features, including waveguides and optical cavities, can be created from two-photon polymerization (TPP) in colloidal crystals\(^14\)\(^-\)\(^15\). Figure 1 presents vertical cross-sections of submicrometre-resolution air defects defined within silicon–air inverse opals exhibiting a variety of defect geometries, including multibend and vertical waveguides (Fig. 1a,b), Y-shaped splitters (Fig. 1c) and embedded planar cavities (Fig. 1d). The complexity and resolution of these features demonstrates the unprecedented potential and flexibility of this approach to add functionality to PhCs that can exhibit a cPBG in the NIR.

The alignment of defects with respect to the PhC lattice is also a matter of paramount importance. This is made evident in two-dimensional (2D) PhCs by the strong dependence of the quality factor of resonant cavities on small lattice variations\(^18\). The use of in situ fluorescence confocal microscopy during TPP affords good registration accuracy\(^19\). In Fig. 2, features written at the surface of a colloidal crystal were imaged using in situ fluorescence confocal microscopy (Fig. 2a,c) directly after TPP, and again using scanning electron microscopy (SEM) after silicon inversion (Fig. 2b,d–f). Figure 2e,f best demonstrates the registration to the PhC lattice, the high edge resolution and that the silicon inversion produces a high-fidelity replication of the initial TPP features.
FABRICATION

The realization of a 3D PhC that has both a cPBG and embedded pre-defined defect structures has posed a number of materials and fabrication challenges. Here, we present an overview of the fabrication procedure (Fig. 3) that was used (see Methods and Supplementary Information for a more detailed description).

First, a modified vertical deposition technique using a temperature gradient was used to grow an artificial opal on a double-side-polished silicon substrate from pre-calcined silica spheres (~725 nm or 925 nm in diameter). A thin conformal film of amorphous alumina was then grown around the spheres by means of atomic layer deposition (Fig. 3a), enhancing the mechanical stability of the sample and providing control over the degree of interpenetration between the spheres. The interpenetration is important, as it directly influences the size of the cPBG of the final structure and facilitates the etching of the silica colloids during the final inversion step (Fig. 3d).

In the second major fabrication step, polymer features were embedded within the colloidal crystal by means of TPP (Fig. 3b), using a modulated beam rastering approach. To facilitate in situ observation during the TPP process and the registration of the TPP features with the underlying PhC lattice, a fluorescent dye was added to the monomer solution. Features with 500-nm linewidths and less than 100-nm edge resolutions were easily achieved with TPP. Under optimal alignment conditions, even narrower features (~100 nm) have been reported. Consequently, the creation of features smaller than the colloid-sphere diameter, a resolution sufficient for most features of interest, is straightforward.

Next, the colloidal PhC containing embedded TPP features was infiltrated with a conformal amorphous silicon (a-Si) layer by means of chemical vapour deposition (CVD) (Fig. 3c) at 325°C. Features with 500-nm linewidths and less than 100-nm edge resolutions were easily achieved with CVD. Under optimal alignment conditions, even narrower features (~100 nm) have been reported. Consequently, the creation of features smaller than the colloid-sphere diameter, a resolution sufficient for most features of interest, is straightforward.

In the final step, the a-Si overlayer was removed using reactive ion etching (RIE), exposing the top half of the alumina-coated silica colloids. The termination geometry obtained after RIE prevents the deleterious effects resulting from surface resonances, as reported in ref. 27. The alumina and silica were then chemically etched with an ethanolic solution of hydrofluoric acid (Fig. 3d). The polymer features are transparent in the NIR, and thus were only removed for imaging purposes (Figs 1 and 2) by means of calcination at 500°C in air or through the use of oxygen plasma etching.

COUPLING OF PHOTONS TO PLANAR CAVITIES

Designed defects are necessary to support optical modes that cannot exist in the surrounding cPBG material. Here we investigate the effect of an embedded planar cavity on the optical response of an opal-based structure before and after infilling with...
air planar cavities in silicon inverse opals can also be fabricated. Of the defect mode. As the vertical cross-section in Fig. 1d shows, methodical studies to improve the bandwidth and transmittance of the PhC lattice, as well as the a-Si filling fraction. All these flexibility in the cavity shape, size and registration with respect to the opal, which is subsequently inverted in a-Si, there is complete truncation differences, which have been shown to greatly affect the coupling efficiency in PhC waveguides. Likewise, in Fig. 5, the coupling efficiency in PhC waveguides (each with a $5 \times 5 \mu m^2$ square cross-section) extending through the thickness of a silica opal: horizontal cross-section (a), vertical cross-section (b). Scale bars: 50 $\mu$m and 25 $\mu$m for a and b, respectively. c, d, Corresponding SEM (c) and IR transmission (d) micrographs collected after inverting the sample in silicon and removing the colloidal template. Scale bars: 50 $\mu$m. The IR micrograph was collected with a bandpass filter centred at 1.48 $\mu$m. The sphere diameter is 925 nm. 

**Figure 4** Optical spectra from a planar cavity embedded in a PhC. a, b, Reflection (top) and transmission (bottom) spectroscopy collected from the same PhC in regions with (red line) and without (blue line) a planar cavity after growing a thin alumina coating (13 nm thick) on the opal (a) and after filling 40% of the remaining pore volume with a-Si by means of CVD (b). The planar defect (dimensions $\sim 1 \times 150 \times 150 \mu m^3$) is embedded halfway through the thickness of the PhC. The sphere diameter is 725 nm. 

a-Si (Fig. 4). Spectroscopy was carried out and compared between regions in the PhC with and without the planar cavity (dimensions $\sim 1 \times 150 \times 150 \mu m^3$), which was embedded halfway through the thickness of the PhC. Notches in the reflectance and transmittance of the first stop-band revealed that some frequencies coupled to optical modes supported by the embedded planar cavity. Because the embedded cavities are written within an existing opal, which is subsequently inverted in a-Si, there is complete flexibility in the cavity shape, size and registration with respect to the PhC lattice, as well as the a-Si filling fraction. All these parameters can be independently varied, enabling future methodological studies to improve the bandwidth and transmittance of the defect mode. As the vertical cross-section in Fig. 1d shows, air planar cavities in silicon inverse opals can also be fabricated. 

**DISCUSSION**

In this article we have demonstrated a flexible route for the introduction of complex, multidimensional features within 3D PhCs, and have provided evidence of waveguiding of NIR light through such features. This represents the first use of a cPBG to manipulate NIR light within a 3D PhC. To further facilitate the creation of optically active devices, the techniques described in this paper could be used to incorporate optical emitters or nonlinear materials within embedded features. This fabrication route is also broadly compatible with PhCs formed by other means, including holographic, conventional and two-photon lithographies, extending the range of crystal structures in which complex defects can be formed. As we have demonstrated, it is now possible to fabricate and characterize complex features with unprecedented quality in cPBG materials. This represents a major step forward in adding functionality to PhCs (ref. 10), extending their viability for the 3D manipulation of photons in all-optical devices. It is our opinion that this work may lead to a paradigm shift in the PhC community, prompting theorists to design defects with sophisticated functionalities for 3D-photonic-bandgap materials. 

**METHODS**

The fabrication procedure used in this work involved the following steps: (1) artificial opal assembly; (2) first atomic layer deposition of alumina; (3) TPP; (4) second atomic layer deposition of alumina (optional); (5) CVD of a-Si; (6) reactive ion etching of top a-Si layer; (7a) wet etch of silica and alumina or...
confirmed by spectroscopy (see Supplementary Information, Fig. S5). Automatically repeated for 80–130 cycles, depending on the requisite layer thickness. T o obtain air defects, TPP features were removed using a static CVD system was used with disilane (Si2H6, 98%, Gelest). For samples assembled from 725-nm-diameter spheres, two cycles were used (∼50 mbar, 15 h, 325 °C); opals made from 925-nm-diameter spheres required a third cycle (see Supplementary Information, Fig. S7). Structures were filled until the trigonal interstitial sites closed off. The ratio of the radius to the centre-to-centre separation of the silicon-defined air spheres was 0.408.

ETCHING
Reactive ion etching was used to expose the silica colloids (see Supplementary Information, Fig. S8). A Uniaxis 790 series RIE was used with a power of 70 W, chamber pressure of 100 mtorr, and O2 and SF6 gas flow rates of 20 s.c.c.m. A typical etch lasted 1 min, and the d.c. voltage readout was 35–40 V. Samples were masked by placing a ∼50-μm-thick Kapton film containing a 1 × 2 mm² opening over the sample. This enabled the selective exposure of a small region during RIE, reducing the probability of sample lift-off during subsequent processing. The silica microspheres and ALD alumina were then removed by means of wet etching using freshly prepared, ethanol hydrofluoric acid (5% in 50% ethanol, 45% water) for ∼40 min for thick samples. (See Supplementary Information, Fig. S9, for more information on oxide removal.)

MICROSCOPY AND SPECTROSCOPY
Electron micrographs were taken with an SEM (Hitachi S-4700) or dual-beam focused-ion-beam (FIB) microscope (FEI Strata DB-235). For FIB milling, the ‘slm’ control file was used with a 5,000–7,000 pA ion aperture for roughing cross-sections, followed by a 100–300 pA aperture for cleaning cross-sections. Milling times were ∼1–2 h per location. Spectroscopy was collected using a 4, 0.1 numerical aperture objective on an optical microscope (Hyperion 2000) coupled to a Fourier transform infrared spectrometer (Vertex 70) and outfitted with a spatial aperture to reduce the collection spot diameter to ∼75–150 μm. Reflection spectra were normalized to a protected silver mirror with >95% reflectivity over the wavelengths used (Melles Griot). Transmission spectra in the manuscript were normalized to air (in the Supplementary Information, spectra were normalized to transmission through the same silicon substrate from a region without a colloidal crystal). Infrared micrographs were collected with an InGaAs 320 × 256 pixel camera with three-stage cooling (Xeva-FPA-320, Xenic). Bandpass filters (Thorlabs and Edmund Optics) had centre wavelengths of 75–150 μm.
wavelengths of 1,300 nm, 1,350 nm, 1,400 nm, 1,480 nm and 1,550 nm, and full-width at half-maximums of 10.5 nm or 12 nm.

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Author contributions S.A.R. carried out the TPP; FIB, SEM, confocal and IR microscopy. F.G.S. carried out the ALD, CVD and bandgap calculations. S.A.R. and F.G.S. both performed HF etching, spectroscopy, RIE, and grew colloidal crystals. All authors conceived and designed the project, participated in discussions about the research and wrote the manuscript.

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