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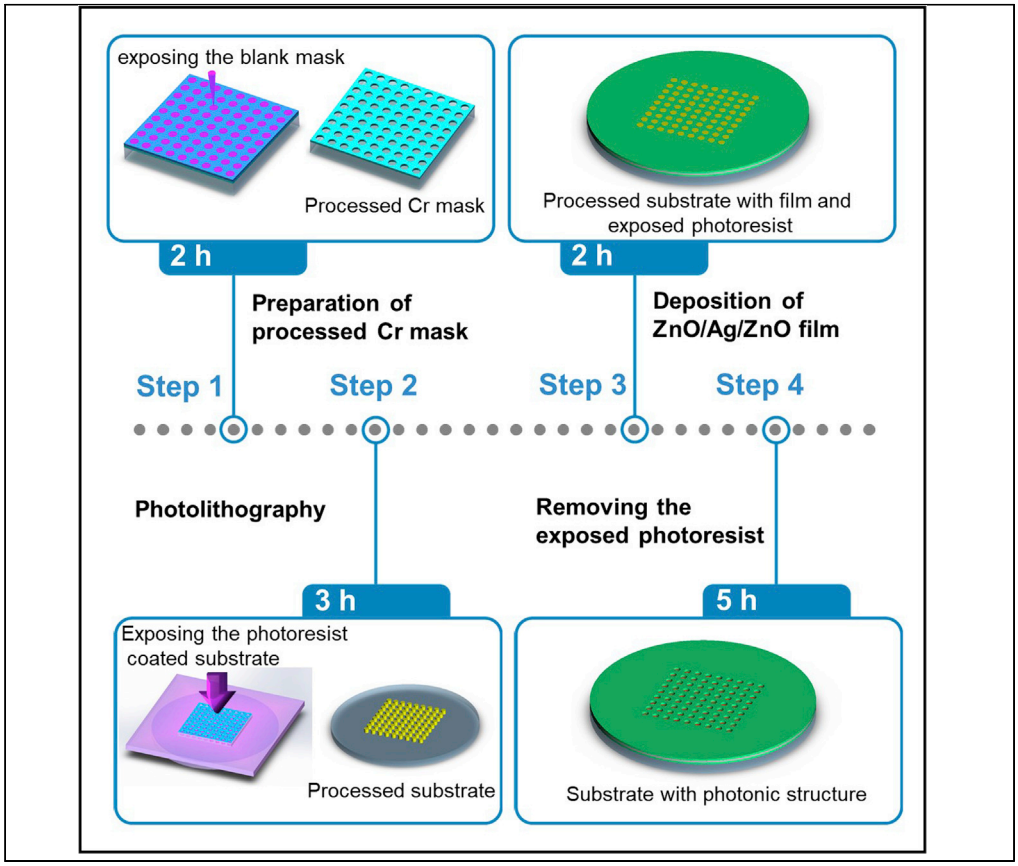
Protocol

Protocol for fabricating a photonic structure consisting of ZnO/Ag/ZnO film perforated with 2D periodic apertures based on photolithography technology



A ZnO/Ag/ZnO film with 2D periodic apertures can have a high visible transmittance and low emittances in atmospheric windows for visible-infrared compatible camouflage and a high transmittance in 5–8 μm for heat dissipation by radiation. This following protocol details the fabrication process of such a photonic structure based on photolithography technology. Also described are the related challenges and troubleshooting steps.

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Highlights

A protocol for ZnO/Ag/ZnO film perforated with a 2D periodic array of apertures

High visible transmittance, infrared emittance low in 3–5 and 8–14 and high in 5–8 μm

Comparison of negative photoresist, bi-layer liftoff, and dry-etching techniques

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Protocol for fabricating a photonic structure consisting of ZnO/Ag/ZnO film perforated with 2D periodic apertures based on photolithography technology

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SUMMARY

A ZnO/Ag/ZnO film with 2D periodic apertures can have a high visible transmittance and low emittances in atmospheric windows for visible-infrared compatible camouflage and a high transmittance in 5–8 μm for heat dissipation by radiation. This following protocol details the fabrication process of such a photonic structure based on photolithography technology. Also described are the related challenges and troubleshooting steps.

For complete details on the use and execution of this protocol, please refer to Dang and Ye (2021).

BEFORE YOU BEGIN

The protocol below describes the specific steps to prepare a photonic structure consisting of a ZnO/Ag/ZnO film perforated with a 2D periodic array of apertures based on negative photoresist photolithography technology. This protocol can also be used for other multi-layer films with designed apertures.

Refer to ''key resources table'' for a list of equipment needed for this protocol.

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Acetone	Sinopharm Chemical ReagentCo., Ltd,	Cat#10000418
Isopropyl alcohol	Sinopharm Chemical ReagentCo., Ltd	Cat#80109218
Concentrated sulphuric acid (98 wt. %)	Sinopharm Chemical ReagentCo., Ltd	Cat#10021660
H_2O_2 (30 wt. %)	Sinopharm Chemical ReagentCo., Ltd	Cat#10011228
N-methylpyrrolidone (NMP)	Sinopharm Chemical ReagentCo., Ltd	Cat#40082581
Deionized water	USTC Center for Micro and Nanoscale Research and Fabrication	N/A
$Ce(NH_4)_2(NO_3)_6$	Suzhou Research Materials Microtech Co., Ltd	N/A

(*Continued on next page*)

Continued		
REAGENT or RESOURCE	SOURCE	IDENTIFIER
Negative photoresist	Futurrex ,Inc.	NR9-3000PY
Developer	AZ Electronic Material USA Corp	AZ300MIF
Cr mask Blank	Changsha Shaoguang Chrome Blank Co., Ltd	SG2506 (2.5 inch)
Silica substrate	Suzhou Research Materials Microtech Co., Ltd	JGS2 (4 inch in diameter, 500 μm in thickness)
ZnO target	Kurt J. Lesker	3 inch in diameter
Ag target	Zhongnuo New Material technology Co. Ltd	3 inch in diameter
Software and algorithms		
Klayout	KLayout EDA Tool	https://github.com/klayoutmatthias/klayout
Other		
Maskless lithograph system	Advantools (China) Co.,Ltd.	ATD 1500
Optical aligner	German SUSS	MA6
Spin coater	German SUSS	Lab Spin6
Hot plate	German SUSS	Delta HP8
Magnetron sputtering deposition system	Kurt J. Lesker.	LAB 18
Optical microscope	Leica Microsystems	DM8000M
Ultrasonic Cleaner	Kunshan Shumei Ultrasonic Instrument Co., LTD	KQ-250DE
UV-visible-NIR spectrophotometers	SolidSpec	3700 DUV
Fourier transform infrared (FTIR) spectrometer	Bruker	VERTEX 80
External golden integrating sphere	Bruker	A562

MATERIALS AND EQUIPMENT

The photoresist (NR9-3000PY) should be stored in shade at a temperature of 5°C, while other chemicals in the table should be stored in shade at a temperature of 22°C. The nano/micro fabrication is operated in a cleaning room (ISO Class 5).

⚠ CRITICAL: The acetone and isopropyl alcohol are hazardous chemicals because of their inflammability. The $Ce(NH_4)_2(NO_3)_6$, H_2O_2 and concentrated sulphuric acid are extremely hazardous because they are corrosive and oxidative. One should wear face shield, anti-erode glove and protective apron and operate in a fume cupboard for safety. Due to the fragility of the mask and substrate, all steps have to be operated with extra care.

Alternatives: The general chemicals (e.g., acetone, isopropyl alcohol and concentrated sulphuric acid, etc.) applied in this protocol can be replaced by those from other sources. The instruments applied in this protocol can be replaced by those which own similar functions. For example, to measure the visible transmittance of the sample, another UV-visible-NIR spectrophotometers (Agilent Cary 7000 UMS) is also applicable.

STEP-BY-STEP METHOD DETAILS

Preparation of the processed Cr mask

Timing: 2 h

The following steps describe the process of fabricating the processed mask based on a Cr mask blank (with resist), as the schematic shown in Figure 1.

⚠ CRITICAL: The following steps to fabricate the processed mask are based on a maskless writing tool (a laser writer). Other similar equipment may require a different set of steps.

Note: The mask blank is a standard commercial soda lime square substrate (2.5 inch) with an AZ photoresist coating (positive tone).

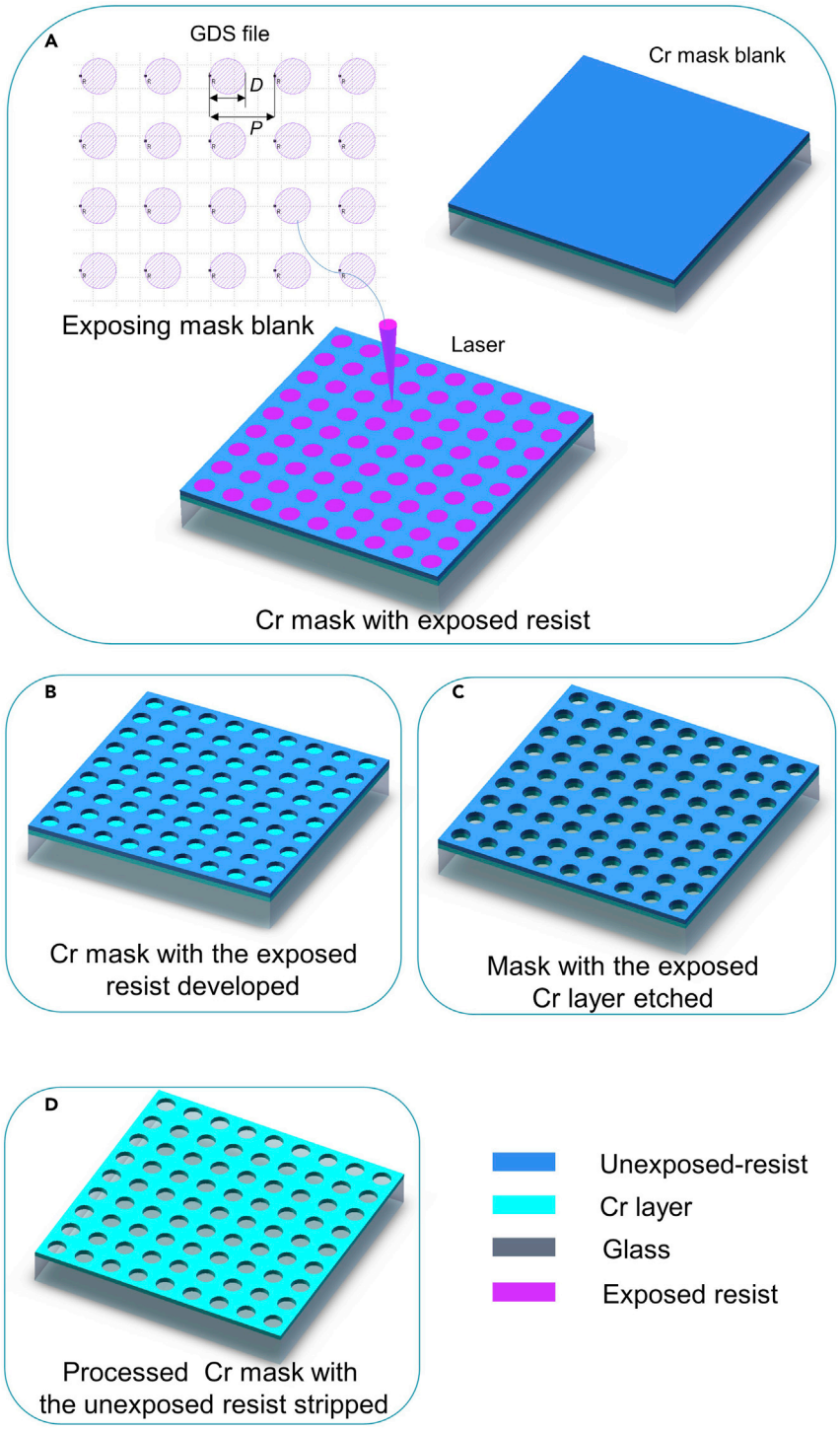

Figure 1. The steps of preparation processed Cr mask

(A) Expose the Cr mask blank with a maskless lithograph system.

(B) Develop the exposed resist.

(C) Etch the exposed Cr.

(D) Strip the unexposed resist layer and the processed Cr mask is prepared.

1. Expose the Cr mask blank with a maskless lithograph system, as shown in Figure 1A.
   a. Plot the layout of the 2D periodic array of circles using the free software Klayout. In a layout of 3 cm × 3 cm, the diameter (*D*) and the period (*P*) of the circles are 3 μm and 5.5 μm, respectively. Save the layout in a format of GDS.

   Note: This layout for the maskless lithograph system is a 2D structure (i.e., no depth). In Klayout, a circle is approximated as an equilateral polygon. The size of the GDS file depends on the side number of the polygon, the number of circles and the size of the layout (3 cm × 3 cm in this work). If the GDS file is too large, it cannot be imported into the maskless lithograph system. In this protocol, the number for the sides of the equilateral polygon is 16 and the size of the layout is 3 cm × 3 cm. (Problem 3).

   b. Import the GDS into the maskless lithograph system (ATD 1500) which can write the mask blank directly with a laser whose operating wavelength is 405 nm.
   c. Place a mask blank on the table of ATD 1500. The mask is kept on the table by vacuum suction. According to the scale of the mask blank (i.e., 2.5 inch), the machine can automatically find the 4 edges of the mask by scanning. Through the coordinates of the edges, the center point of the mask can be determined. The coordinate of each point to be exposed will be determined as well according to the imported GDS file. During the exposing process, the laser is fixed and the table moves under the control of stepping motors. Driven by the table, the mask reaches the exposing position according to the coordinate and the corresponding region be exposed by the laser.
      i. The operating power of ATD 1,500 is 66 mW and the offset is −0.0006 mm.
      ii. The photo-chemical reaction occurs at the required region due to the laser exposure on the resist of the mask blank.
      iii. The exposing process takes ~30 min.

2. Develop the exposed mask in AZ300MIF for 50 s to strip the exposed resist, which exposes the underneath Cr layer, as shown in Figure 1B.
3. Fix the developed mask in Deionized water for 30 s.
4. Put the fixed mask in $Ce(NH_4)_2(NO_3)_6$ for 70 s to remove the exposed Cr by a wet-etching process.
5. Use deionized water to rinse the mask.
6. Blow-dry the mask by N_2 and the mask with patterned Cr and resist is prepared, as shown in Figure 1C.
7. Strip the unexposed resist layer with a Piranha solution. The processed Cr mask for photolithography is prepared, as shown in Figure 1D.

   ⚠ CRITICAL: The piranha solution is very dangerous. A detailed description can be found in the "critical" note of the "materials and equipment" section. An alternative to the piranha solution for stripping unexposed resist is AZ300T, which is much less hazardous (Rackus et al., 2015). A detailed preparation of the piranha solution is introduced here. With a volume ratio of 3:1, measure H_2SO_4 (98 wt. %) and H_2O_2 (30 wt. %), respectively. Add H_2O_2 into H_2SO_4 slowly, but not vice versa. Because this is an exothermic process, let the mixture cool down before next step. Diluted and exhausted piranha solution is stored in a proper container with a lid and a "Hazardous Waste, Acid Contaminated Objects" label and in a cabinet compatible with strong acid solution, kept separated from strong basic solution and flammable solvents. Waste collection and proper disposal is periodically arranged by vendors which are legally licensed and certified for these procedures. A detailed piranha solution standard operating procedure can also be found in Stevic (2018).

Photolithography on silica substrate

Timing: ~3 h

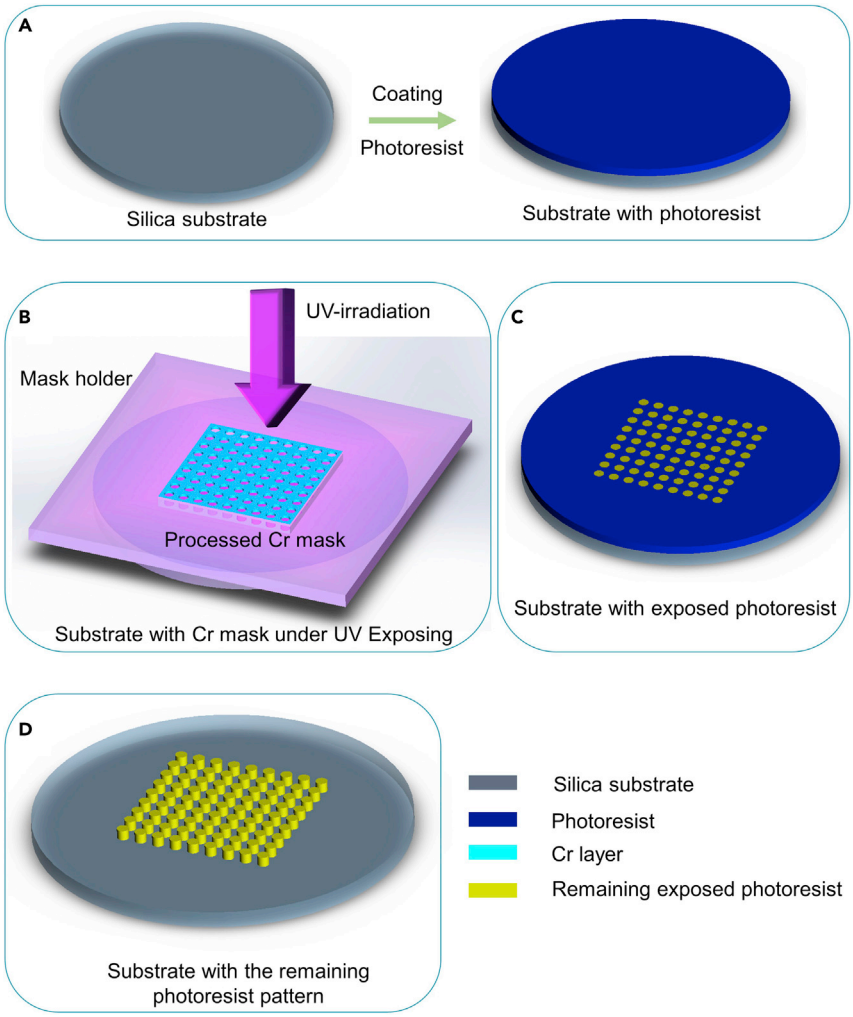

Figure 2. The process of preparing the substrate with exposed photoresist pattern for depositing film

(A) Coat the silica substrate with photoresist.

(B) With the processed Cr mask, expose the photoresist-coated substrate by an optical aligner.

(C) The silica substrate with exposed photoresist.

(D) The substrate with the remaining exposed photoresist after stripping the unexposed photoresist.

The following steps describe the photolithography process on a silica substrate with the processed mask, as shown in Figure 2.

8. Clean the silica substrate (4 inch in diameter, 500 μm in thickness) with isopropyl alcohol, acetone and deionized water.
   a. In a crystallizing dish, clean the silica substrate with isopropyl alcohol. Use deionized water to wash the remaining isopropyl alcohol.
   b. In another crystallizing dish, clean the silica substrate with acetone. Use deionized water to wash the remaining acetone.
   c. Blow-dry the silica substrate by N_2.

⚠ CRITICAL: It is important to clean the silica substrate to prepare the required photonic structure on the substrate because a cleaner substrate has a better adhesion for the deposited film.

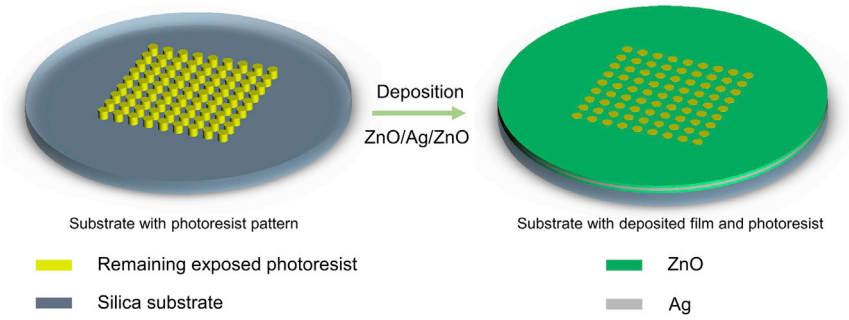

Figure 3. Deposition of ZnO/Ag/ZnO film based on magnetron sputtering

9. Pre-bake the silica substrate on the hot plate (SUSS Delta HP8) at 120°C for 120 s for a good adhesion of the photoresist on silica.
10. Spin the photoresist (NR9-3000PY) on the silica by a spin coater (SUSS Lab spin6) with a rate of 4,000 rpm for 40 s. The substrate with photoresist is prepared, as shown in Figure 2A.

Note: The thickness of the NR9-3000PY photoresist is approximately 2 μm. For a liftoff process, a thicker photoresist (negative tone) is easier to be stripped due to the formed undercut (Shields and Allsopp, 2011). It usually suggested that the thickness of the deposited film (i.e., 102 nm in this work) should not exceed approximately 30% of the thickness of the photoresist, and the lower the better.

11. Bake the photoresist-coated substrate on the hot plate at 120°C for 60 s to dry the photoresist.
12. With the processed mask on center of the substrate, as shown in Figure 2B, the photoresist-coated substrate is exposed by the optical aligner (SUSS MA6) with a power density of 21.5 mW/cm^2 for 12 s. The substrate with exposed photoresist is prepared, as shown in Figure 2C.
13. Post-bake the photoresist-coated substrate on the hot plate at 120°C for 60 s to let the photochemical reaction induced by the exposure to UV proceed.
14. Develop the exposed substrate in AZ300MIF for 30 s to strip the unexposed resist. The substrate with the remaining photoresist pattern is prepared.
15. Fix the developed substrate in deionized water for 30 s.
16. Blow-dry the substrate by N_2.
17. Check the prepared pattern with an optical microscope. If the pattern is intact, this substrate with photoresist pattern is completed for deposition, as shown in Figure 2D.

Deposition of ZnO/Ag/ZnO film

Timing: 2 h

The following steps describe the deposition process to fabricate a ZnO/Ag/ZnO film on the silica substrate after lithography based on magnetron sputtering (LAB 18), as shown in Figure 3.

Note: The sputtering atmosphere is generated by Ar gas. In this magnetron sputtering instrument, the power supply for a specific target position is explicit, either a RF power or a DC power. The RF power is provided by Kurt J. Lesker R601 with the corresponding automatic matching network controller (Kurt J. Lesker MC2). The DC power is provided by Kurt J. Lesker DC02 BP. The RF or DC mode can be handled by the machine via software control. To complete a specific deposition, the target should be mounted on the corresponding target position.

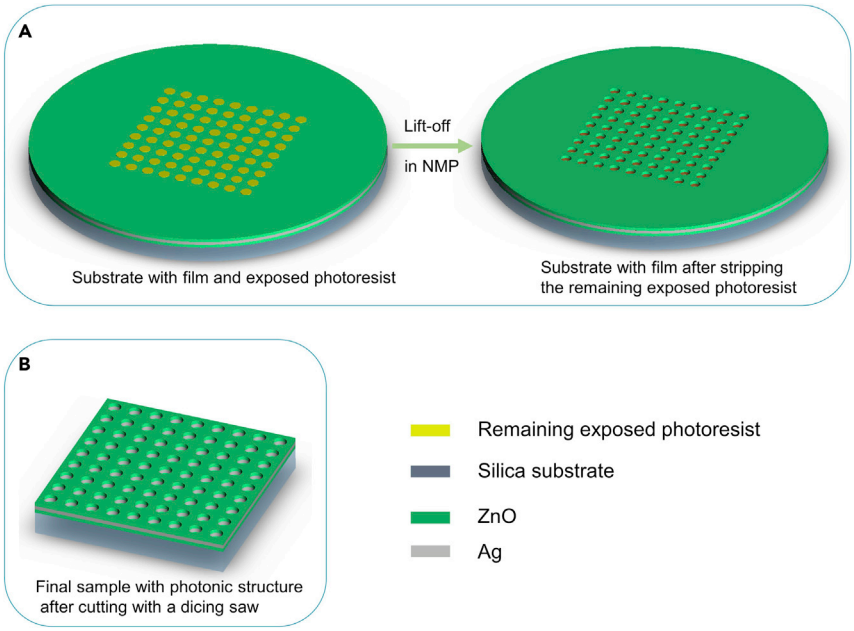

Figure 4. Stripping the exposed photoresist with the nearby film remaining well to prepare the final sample

(A) The lift-off process in NMP to strip the exposed photoresist.

(B) The final successful sample.

18. The chamber is pumped to a pressure of 4.8×10^{-6} Torr after the silica substrate is loaded.

19. Put the ZnO target at RF position for sputtering.
    a. A pressure of 1.5×10^{-2} Torr is set to induce plasma.
    b. The operating power is 150 W.
    c. The operating pressure is 3×10^{-3} Torr.
    d. The pre-sputtering time is 60 s.

    Note: The pre-sputtering step here is to clean the surface of the target with the sample protected by a baffle.

    e. The depositing time is 1,000 s to deposit 50 nm-thick ZnO layer, as its growing speed is 0.5 Å/s.

20. Put the Ag target at DC position for sputtering.
    a. A pressure of 1.5×10^{-2} Torr is set to induce plasma.
    b. The operating power is 150 W.
    c. The operating pressure is 5×10^{-3} Torr.
    d. The pre-sputtering time is 120 s.
    e. The depositing time is 28 s to deposit 14 nm-thick Ag layer, as its growing speed is 0.5 Å/s.
21. Repeat step 19 and the depositing time is 760 s to fabricate a 38 nm-thick ZnO layer. The substrate with deposited film and exposed photoresist is prepared as shown in Figure 3.

Liftoff process to strip the exposed photoresist

Timing: 5 h

The following steps describe the process of stripping the remaining photoresist, as shown in Figure 4.

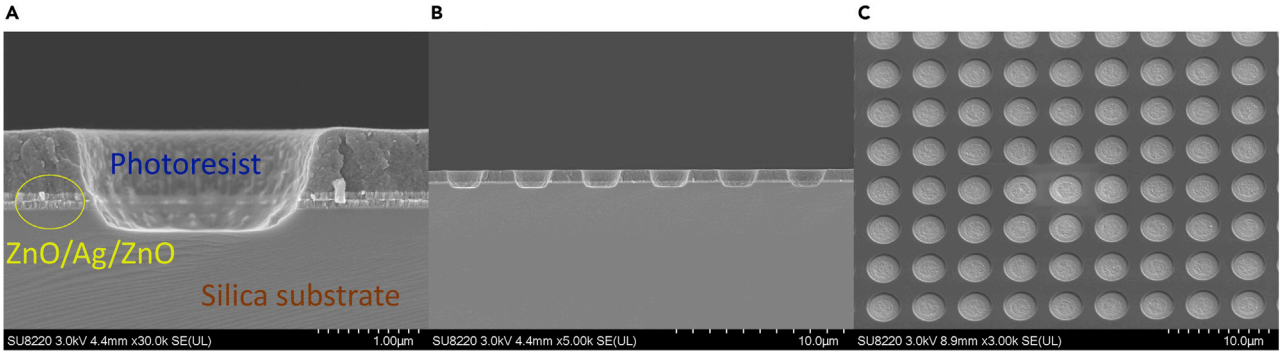

Figure 5. The SEM result of the photonic structure with a 2D array of apertures prepared by dry-etching technology

(A) Side-view SEM result of the ZnO/Ag/ZnO film with an etched aperture (with photoresist).

(B) Side-view SEM result of the film with an array of apertures (with photoresist).

(C) Top-View SEM result of the film with 2D array of apertures (without photoresist). The silica substrate is also etched, showing a rough surface.

22. Let the NMP solution permeate into the photoresist by keeping the sample immersed for 4 h in NMP kept at 80°C with a thermostatic bath (Rosamond et al., 2011).
23. Strip the photoresist by ultrasonic shaking, as shown in Figure 4A.
    a. The power of the ultrasonic machine is 250 W.
    b. The operating frequency of the ultrasonic machine is 40 kHz.
    c. The bath time in the ultrasonic tank is 5 s firstly.
    d. The advancement of the lift-off can be checked by visual inspection on an optical microscope to observe whether the remaining photoresist is cleanly stripped and the deposited film is delaminating. If there is remaining photoresist, keep the sample immersed in NMP with a thermostatic bath for 10 min again. Repeat sub-steps a, b and c in step 23. In step 23.c, the bath time in the ultrasonic tank is 3 s.
    e. Repeat sub-step 23.d, until the remaining photoresist is completely stripped and the nearby deposited film remains well, as shown in the right image of Figure 4A.
24. Cut the excessive part of the substrate with a dicing saw and the final successful sample is prepared, as shown in Figure 4B.

Note: In this protocol, the negative photoresist liftoff process (Kim et al., 2020; Xia et al., 2020) is introduced to prepare a multi-layer structure with a 2D array of apertures. To prepare such a photonic structure, we also tried bi-layer liftoff process (Hwang et al., 2009; Mao et al., 2013) and dry-etching technology (Lehmann and Widmer, 1980; Nojiri, 2015). However, the bi-layer liftoff process failed as it is difficult to strip the exposed-photoresist with the nearby film remaining well. The resolution and density of the pattern produced in this work makes it challenging to control the over-development of the bottom layer to achieve full opening and maintaining good adhesion of the resist mask. The dry-etching process is operated on an inductively coupled plasma etch system (Oxford, PlasmaSystem100 ICP 180). The detailed chemistry of the plasma used and etching recipe are introduced as follows. The mixture of gases is BCl_3(14sccm) /Ar(6sccm). The power of ICP is 1,000 W. The power of RF is 150 W. The pressure of the chamber is 2 mTorr. The temperature of the bottom electrode table is 30°C. The pressure of the He backing is 10 Torr. The time for etching is 2 min 30 s. In the process of dry-etching, the silica substrate will also get etched as shown in Figures 5A and 5B. The rough surface (Figure 5C) of the silica substrate will strongly scatter light, significantly reducing the visible transmittance. Therefore, we do not recommend this dry-etching technology. Perhaps improved results can be obtained but optimization of this dry-etching technology is out of the scope for this work.

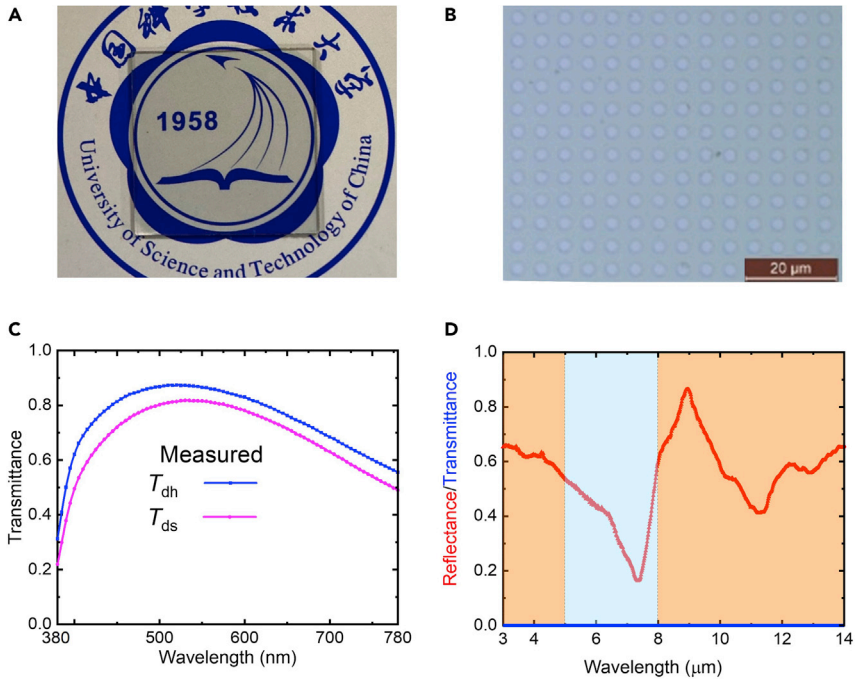

Figure 6. The prepared successful photonic structure on silica substrate and its visible and infrared radiative properties

(A) The photo of the prepared sample.

(B) The microscope photo of the photonic structure with a 2D array of apertures.

(C) The measured T_{dh} and T_{ds} of the prepared sample under normal incidence.

(D) The measured infrared reflectance (13° incidence) and transmittance of the sample (normal incidence). Figures are adopted with changes from Ref. (Dang and Ye, 2021).

Measuring the visible transmittance and infrared emittance

Timing: ~1 h

The following steps describe the process of measurements of visible transmittance and infrared emittance.

25. Apply a UV-visible-NIR spectrophotometers with a $BaSO_4$ integrating sphere to measure the directional-hemispherical transmittance (T_{dh}) and the directional-specular transmittance (T_{ds}) under normal incidence. To measure T_{dh}, the sample is put at the front window of the $BaSO_4$ integrating sphere. To measure T_{ds}, the sample is put between the integrating sphere and the grating.
26. Apply a Fourier transform infrared (FTIR) spectrometer with an external golden integrating sphere to measure the infrared reflectance of the sample.
27. Apply the FTIR spectrometer to measure the infrared transmittance of the sample.
28. The emittance of the sample can be calculated by one minus the sum of reflectance and transmittance.

Note: For the measurement of infrared reflectance, the incident angle is 13°, which should be noticed to analyze the surface plasmon resonance occurred in this photonic structure.

EXPECTED OUTCOMES

The prepared sample is transparent for visible light as shown in Figure 6A and it has a 2D array of apertures, as shown in Figure 6B. Under normal incidence, the measured T_{dh} reaches 0.9 and the

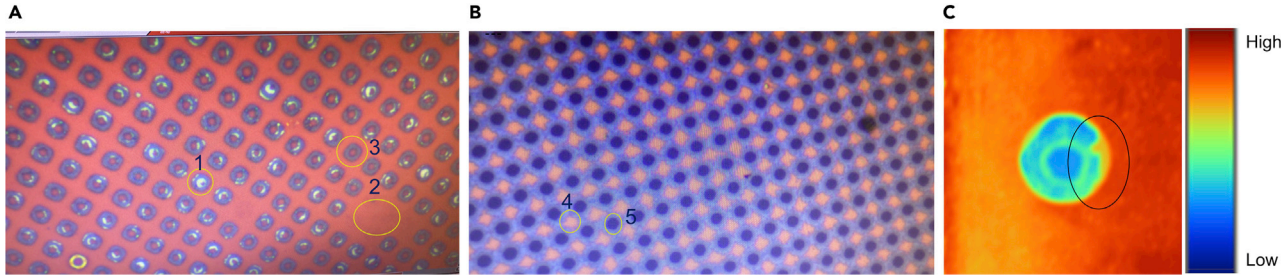

Figure 7. The previous unsuccessful samples

(A) The exposed photoresist is not stripped cleanly. Region 1 shows the remaining photoresist. Region 2 shows the unperfect pattern. Region 3 shows the out-of-round aperture.

(B) The film delaminated from the substrate. Region 4 shows the remaining film. Region 5 is the aperture without exposed photoresist.

(C)The sample with edge damaged under infrared camera. The enclosed region by black line shows the damaged part.

measured T_{ds} reaches 0.8, as shown in Figure 6C. The measured infrared reflectance and transmittance are shown in Figure 6D. The measured transmittance in 3–14 μm is nearly 0, indicating the sum of emittance and reflectance is ~1. In 3–5 μm, the reflectance reaches 0.65 (i.e., emittance=0.35). In 8–14 μm, the reflectance reaches 0.86 (i.e., emittance=0.14). With these low emittances, the sample can be applied for infrared camouflage. In 5–8 μm, the reflectance can reach 0.18 (i.e., emittance=0.82), indicating a performance of heat dissipation by radiation.

LIMITATIONS

This protocol should be applied carefully as there are limitations. Based on the negative photoresist liftoff process in this protocol, a multi-layer film with designed apertures can be prepared. However, when the scale of the aperture changes, this protocol may be ineffective. For example, when the aperture is smaller, the processed Cr mask may not get prepared through the maskless lithograph system (step 1) and the exposed photoresist may not get stripped from the substrate with the nearby film existing well (step 23). The operating parameters (e.g., power and time, etc.) for the equipment (e.g., ATD 1500 and SUSS MA6, etc.) in this protocol may be not applicable for other equipment with similar function. Due to the limitation of the equipment, the optical properties of the photonic sample at a higher temperature (>25°C) cannot be measured to investigate the influence of temperature on the performance of this photonic sample.

TROUBLESHOOTING

Problem 1

An important step is to strip the exposed photoresist on the substrate (step 23) with the nearby film existing well using ultrasonic bath. However, the exposed photoresist is not easy to be stripped from the substrate and the ZnO/Ag/ZnO film will delaminate from the substrate in an ultrasonic bath as well. If the ultrasonic bath time is not enough, the photoresist may be not cleanly stripped, as shown in Figure 7A. The region 1 in Figure 7A shows the remaining photoresist. On the other hand, if the ultrasonic time is too long, the film will delaminate from the substrate, causing the fabrication failed as well, as shown in Figure 7B. The region 4 shows the remaining film and the region 5 is the aperture with no exposed photoresist. The region between the remaining film (i.e., 4) and the aperture (i.e., 5) should be the deposited film. (step 23)

Potential solution

The substrate should be well cleaned to enhance the adhesion between the film and the substrate. The ultrasonic bath in step 23 is operated at 80°C to make the exposed photoresist easily be stripped. The bath time should be controlled carefully (e.g., 3 s one time) to make sure the ZnO/Ag/ZnO film is still adhered on the substrate with the exposed photoresist stripped gradually.

Problem 2

As the Cr mask blank may be not perfect, the observed damages (e.g., bubbles in the photoresist layer on the mask) may cause the prepared mask inapplicable for next step, as shown in Figure 7A (region 2). (steps 1–5)

Potential solution

Expose a new Cr mask with the maskless lithograph system again.

Problem 3

In Klayout, a circle will be approximated as an equilateral polygon. If the polygon has not enough sides, it will be out-of-round, as shown in Figure 7A (region 3). If the polygon has too many sides, the GDS file will be too large to be imported into the maskless lithograph system. (step 1)

Potential solution

Select an appropriate number for the sides of the equilateral polygon in Klayout (e.g., 16 sides in this protocol).

Problem 4

When using the magnetron sputtering to prepare ZnO and Ag, the deposing rate may change as the pressure in the chamber and the operating condition of the system are not completely stable. (steps 19–21)

Potential solution

Calibrate the depositing rate of each material before conducting the deposition process.

Problem 5

After deposition of Ag layer, the silver will get oxidized gradually which will influence the performances of the sample. After storage for a long time, the film will delaminate, as shown in Figure 7C. According to the observation of an infrared camera, the edge of one sample is damaged. (steps 24 and 25)

Potential solution

The photonic sample should not be stored for a long time before conducting the other experiments. The temperature of the photonic sample is controlled below 180°C which is safe for silver temporarily (Reddy et al., 2017).

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources should be directed to and will be fulfilled by the lead contact, Hong Ye (hye@ustc.edu.cn).

Materials availability

This study did not generate new unique reagents.

Data and code availability

The published article includes all datasets/code generated or analyzed during this study.

ACKNOWLEDGMENTS

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AUTHOR CONTRIBUTIONS

H.Y. conceived the idea and supervised the study; S.D., J.Z., F.P., Y.W., W.L., X.W., and Y.H. conducted the experiments. S.D. drafted the manuscript. All authors contributed to manuscript editing.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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