Colorimetric Ethanol Indicator Based on Instantaneous, Localized Wetting of a Photonic Crystal

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Accessibility
Supporting Information for

A Colorimetric Ethanol Indicator based on Instantaneous, Localized Wetting of a Photonic Crystal

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Methods

Fabrication of inverse opal films (IOF)

The inverse opal films were synthesized using an evaporative co-assembly method based on a procedure by Hatton et al.1 Aqueous solutions of templating polystyrene (PS) colloids with a diameter of 360 nm were either synthesized by surfactant-free emulsion polymerization2 or purchased commercially (IDC/Invitrogen). The solutions of colloids were diluted to 0.1 wt% and combined with 0.5 vol% prehydrolyzed tetraethyl orthosilicate (TEOS) solution. The TEOS solution was prepared by combining ethanol, TEOS, and 0.1M HCl with a 3:2:2 volume ratio (1.5 ml EtOH, 1 ml TEOS, 1 ml HCl) in a glass vial and stirring at room temperature for 1 hour to prehydrolyze the TEOS. A Si wafer was cut into 1×5 cm slides and immersed in piranha solution (H₂SO₄:H₂O₂=3:1) for 30 minutes at 120 °C. After thoroughly rinsing with DI water, the substrate was suspended in the solution of colloids and left at 65 °C for 3 days. After the co-assembly process was completed, the template was removed by calcination at 500 °C over 12 h (5 h heating time, 2 h at 500°C, 5 h cooling time) to carefully control temperature gradients and prevent crack formation. For the IOF deposited on glass rods and vials, PS colloids were replaced with 360 nm diameter poly(methylmethacrylate) (PMMA) spheres; all other procedures remained the same as for Si substrates.

Deposition of silane molecules

IOF and glass substrates were functionalized with silanes using a solution-based method. Silane solutions were prepared by dissolving 1 vol% of 2-[methoxy(polyethylenoxy)9-12propyl]trimethoxysilane (PEG) and/or acetoxyethyl(dimethylchlorosilane (ACE) in toluene. Toluene was selected as the solvent, because it easily dissolves both PEG and ACE, which ensures that the selected mixture is accurately transferred to the surface (Figure 2c). Before
functionalization, the surfaces were activated by exposure to oxygen plasma for 10 min or by submersion in a 3:1 piranha solution for 30 min at 120 °C. For the contact angle measurement, glass substrates were placed in the silane solution for 10 minutes to allow the covalent bonding of silanes onto the oxide surface. For the dip-coating process, IOF substrates were suspended in the silane solutions containing ACE for only 40 s to avoid the unwanted vapor-phase attachment of ACE molecules on the upper untreated IOF portion. The highly active Si-Cl bond ensured covalent binding of ACE to IOF surface. The final functionalization with a solution containing only PEG was set at 10 min. The size of the coated segment was controlled by the immersion depth of the wafer in the solution.

**Characterizations**

Scanning electron microscopy (SEM) images were taken using the SE2 detector on a Zeiss Ultra field-emission microscope with a gun voltage of 5 kV and a working distance of ~3 mm. The cross-sectional SEM images were obtained by physically breaking the IOF substrate and then imaging the cleaved edge of the wafer. Contact angle measurements were conducted on a Kruss contact angle analyzer under ambient conditions.
Supporting Figure

**Figure S1.** Optical images illustrating the contact angle of water on PEG-treated glass, 40% EtOH on ACE-treated glass, water on ACE-treated glass.

Supporting Table

**Table S1.** The silane ratios for different ethanol concentrations

<table>
<thead>
<tr>
<th>Ethanol concentration</th>
<th>ACE: PEG</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-5%</td>
<td>PEG</td>
</tr>
<tr>
<td>5-10%</td>
<td>1:1</td>
</tr>
<tr>
<td>10-20%</td>
<td>5:1</td>
</tr>
<tr>
<td>20-30%</td>
<td>10:1</td>
</tr>
<tr>
<td>30-40%</td>
<td>25:1</td>
</tr>
<tr>
<td>&gt;40%</td>
<td>ACE</td>
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</table>
Reference
