Active electrochemical high-contrast gratings as on/off switchable and color tunable pixels

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To be viable for display applications, active structural colors must be electrically tunable, on/off switchable, and reversible. Independently controlling the first two functions, however, is difficult because of causality that ties the real and imaginary parts of the optical constants or changing overlap of fields during structural variations. Here, we demonstrate an active reflective color pixel that encompasses separate mechanisms to achieve both functions reversibly by electrochemically depositing and dissolving Cu inside the dielectric grating slits on a Pt electrode with ΔV < 3 V. Varying the modal interference via Cu occupancy in the slits changes the CIE space coverage by up to ~72% under cross-polarized imaging. In the same pixel, depolarization and absorption by the dissolving porous Cu switches the color off with a maximum contrast of ~97%. Exploiting these results, we demonstrate an active color-switching display and individually addressable on/off pixel matrix that highlights their potential in reflective display applications.
ideally, electrically tunable reflective pixels for display applications1,2 or optically variable devices (OVDs)3 should change colors over a wide range upon applied bias, switch off when needed, exhibit high contrast against the background, and remain reversible. Controlling the first two criteria with a single structural color design is particularly challenging because they require seemingly distinct mechanisms. Active color tuning requires a change in the real part of the optical constants or an ordered structural variation, whereas on/off switching demands a change in the imaginary part or the introduction of structural disorder to increase and broaden the absorption. Because the real and imaginary parts of the optical constant are tied by causality (i.e., Kramers–Kronig relation) and structural variations affect the coupling between fields, color tuning is inevitably accompanied by intensity modulations, challenging independent control of color and intensity. This suggests that color tuning and on/off switching should rely on separate mechanisms, which are not straightforward to encompass in one system. Studies focused on one or the other have relied on electromechanical Mis structures4 and plasmonic or photonic designs paired with electrochromic polymers5–8, liquid crystals9,10, metal electrodeposition11–13, metal-to-dielectric electrochemical conversion14,15, metal hydrogenation16, and ion intercalation17–19.

Prolonged reversibility is another important requirement. While plasmonic colors offer attractive merit20,21, the workhorse metals, namely Ag and Al, are easily degraded, compromising the color stability with repeated use. Other metals with superior chemical stability such as Pt are generally avoided because of large optical damping factors associated with interband transitions22. Nonetheless, vibrant structural colors, largely insensitive to the optical loss of the metal, are achievable using a 1-dimensional (1D) dielectric grating on the respective metal, rotated 45° against the input polarization and observed through a cross (orthogonal) polarizer23. Vibrant structural colors from such a cross-polarized scheme have also been reported for plasmonic nanowires24 and dielectric nanoparticles on Ag25. For the dielectric 1D grating, the color is defined by the reflected field rotated 90° across the s-polarized (s-pol) or p-polarized (p-pol) resonance wavelengths. The p-pol resonance, described by the rotated 90° across the s-pol or p-pol resonance field over the HCG period. For the bare HCG with a height of 100 nm, a ~n phase shift occurs with the Rayleigh–Wood anomaly (Fig. 1d, top). When the empty slits are filled with Cu, the resonance redshifts by ~135 to ~200 nm over the range of considered periods (Fig. 1d, bottom) since the number of resonance field anti-nodes in the HCG unit cell is reduced (Supplementary Fig. 1). Unlike most structural color designs paired with electrochromic polymers or electrodeposition schemes, this shift is not determined by refractive index variations nor changes to the field overlap, but rather the ‘muting’ of resonance fields inside the HCG slit and the subsequent change in modal interference. We note that as a consequence of this mechanism, the colors are dependent on the incident angle since off-normal incidence introduces odd modes (i.e., β1, β3, etc) that modify the interference conditions.

For an HCG rotated ~45° against the incident polarization and observed through a cross-polarizer (Fig. 1a), the s-pol resonances for both bare and Cu-filled HCGs are directly associated with large reflection intensities as the tilt angle of the polarization ellipse is close to 45° at the resonances and ~45° otherwise (Fig. 1e). This results because the p-pol resonance is attenuated by the lossy metals, leaving the s-pol resonance alone to determine the tilt angle. The scheme offers a useful benefit for color generation. For both bare and Cu-filled HCGs, the s-pol resonance is not as strongly affected by the metallic loss as the p-pol response since its field maxima are distant from the metal surface. As a result, the cross-polarized reflection intensities remain strong for both HCGs (Fig. 1f). We note that, unlike the
bare and Cu-filled HCGs, partially Cu-filled HCGs rely on both s and p-polar responses to generate the cross-polarized reflection (see Supplementary Note 2 and Supplementary Fig. 2), leading to a cross-polarized response much broader than the s-polar response alone. This is attributed to the interference of the fundamental p-polar mode within the bare part of the HCG, also yielding a \( \sim \pi \) phase shift across the wavelengths.

Operation of electrochemical structural color pixel. To experimentally observe the spectral shifts, we configured our imaging setup according to the scheme of Fig. 1a, with details shown in Supplementary Fig. 3. Polarized white light was directed and focused onto the active color pixel through a 50/50 beam splitter and low NA objective lens, respectively, while the reflected light was filtered by a linear polarizer arranged orthogonally to the incident polarization. This ensures a dark background and hence high contrast by blocking unrotated or 180°-rotated fields from areas outside the HCG. To electrically control the colors, we integrated the HCG into an electrochemical cell comprising a Pt working electrode (WE), ITO counter electrode (CE), and electrolyte composed of copper (II) nitrate trihydrate (1 M; \( \text{Cu(NO}_3\text{)}_2 \cdot 3\text{H}_2\text{O} \)) dissolved in dimethyl sulfoxide (DMSO) (see Methods). Although use of a reference electrode was prohibited by the miniature cell size, the CV characteristics were largely consistent over repeated cycles. Using e-beam lithography, \( 50 \times 50 \mu\text{m}^2 \)-sized HCGs with periods from 290 to 510 nm were created from an e-beam-evaporated TiO\(_2\) film atop of the Pt electrode (see Methods). The pixels were designed with the same slit width used in Fig. 1 and height of 100 nm, predicted to provide a large s-polar spectral shift of \( \sim 170 \text{nm} \) between bare and Cu-filled HCG responses (see Supplementary Note 3 and Supplementary Fig. 4).

By applying a voltage across the two electrodes via a potentiostat (Fig. 2a), Cu occupancy in the slits was controlled. Cu deposits inside the slits when a cathodic bias (i.e., potential needed to reduce Cu\(^{2+}\) ions to Cu (s)) is applied to the WE. In an open circuit, the Cu dissolves over a few minutes due to the nitric acid in the electrolyte solution\(^{33}\). This can be halted with a small negative bias that cancels the excess oxidative current. Dissolution is accelerated with an anodic bias via oxidation. Distinct HCG morphologies throughout these processes are observable from top-view scanning electron microscopy (SEM) images and cross-sectional high-resolution transmission electron microscopy (HR-TEM) (Fig. 2b). On a clean Pt surface

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**Fig. 1 Shift in optical response between bare and Cu-filled HCG under s-pol light.** a Schematic of a bare and Cu-filled HCG normally excited by the superposition of s- and p-pol light. The reflected electric field vector is rotated via phase shifts to the s- and/or p-pol field. b Dispersion curves of waveguide-array modes (left) and their modal profiles at the wavelength of 550 nm (right) for the bare (top) and Cu-filled HCG (bottom). c Calculated 0th order reflection of bare (top) and Cu-filled HCG (bottom) as a function of grating bar height and wavelength. d-f Phase of the 0th order s-pol reflected field (d), tilt angle of polarization ellipse (e), and cross-polarized reflection (f) for the bare (top) and Cu-filled HCG (bottom) as a function of period and wavelength.
within the HCG slits (stage I), Cu accumulates displaying smooth and uniform coverage (stage II). In the event the deposited Cu exceeds the HCG height, it is no longer laterally restricted, and receives an increased ion flux that promotes nucleation of larger (>~100 nm) crystals (stage III). Scanning transmission electron microscopy (STEM) and energy dispersive spectroscopy (EDS) confirm that the HCG and deposited metal are composed of TiO\(_x\) and Cu, respectively (Fig. 2c). Additional characterization of the Cu is provided in Supplementary Note 4 and Supplementary Fig. 5. Unlike Cu deposition, dissolution occurs spatially sporadically, leaving the metal strip initially disordered (stage IV), the effect of which we describe later.

The use of Pt as the WE electrode, whose chemical stability is well-established, ensures robust and prolonged reversibility over repeated cycles. A step potential alternating between −1.7 and 0.7 V for 3.9 and 24 s, respectively, was repeatedly applied to a 350 nm-period pixel. Peak-to-peak wavelengths in the cross-polarized reflection spectra and associated images of the pixel remain unchanged up to 540 cycles (Fig. 2d), which represent a >2-fold improvement over results from electrodeposited Ag on Au nanodomes, the reversibility of which is limited by the growth of Ag/Au alloys\(^{11,34}\).

**Electrochemical control of Cu morphology and corresponding optical response.** The temporal profile of the measured current in response to an applied voltage (Fig. 3a) provides mechanistic insight into the unique set of operational modes offered by the pixel. We label these modes as 1–6 in the current profile and describe their corresponding cross-polarized reflection images.
Across pixels from $p = 290$ to $510$ nm (Fig. 3b, Supplementary Fig. 6, and Supplementary Video 1). All pixels start as bare HCGs covering a wide range of colors defined by the period-dependent s-pol resonance (mode 1). Upon application of a cathodic potential, Cu ions near the exposed Pt surface are immediately reduced, creating a negative spike in the current. Obeying 1D diffusion-dominated reduction in response to a step potential, this current then decays as $t^{-0.5}$ (Supplementary Fig. 7) from a decreased ion concentration gradient at the deposited surface that lowers the incoming flux of reducible Cu ions. The resultant rising Cu surface redshifts and broadens the response via s- and p-pol modal contributions within the unfilled parts of the HCG as described earlier (mode 2). As the accumulated Cu reaches the HCG height (mode 3), the colors are determined by the period-dependent s-pol resonance of the Cu-filled HCG, shrinking the CIE color space coverage by $\sim 72\%$ (Fig. 3c). We note that if the Cu thickness exceeds the HCG height, eventually forming a rough connected film, the HCG birefringence is annulled, eliminating the pixel intensity. This, however, is not our preferred ‘off’ mode because once formed, the Cu on the HCG bars cannot be easily removed due to poor charge transfer.

Applying a step potential of 0.7 V at 4.3 s reverses the current, described by a spike in positive current followed by an exponential decay (Fig. 3a). This decay represents the progressive removal of Cu that leaves the Cu increasingly porous, transitioning the pixels to a black mode (mode 4). For highly disordered systems, it has been shown that energy can be equally partitioned over the wavelengths with similar coupling efficiencies rendering the absorption broadband. Optical images of dissolving Cu-filled pixels without the polarizers confirm this effect through a marked darkening. We note, however, that the images do not show a fully black state (Supplementary Fig. 8a) because the disorder occurs only within the HCG slits (i.e., $\sim 45\%$ of the pixel area). The black state is achieved with the aid of crossed polarizers due to polarization scrambling by the porous Cu (Supplementary Fig. 8b) which further reduces the cross-polarized reflection and yields maximum on/off switching contrasts ranging from $\sim 78$ to $\sim 97\%$ (Supplementary Fig. 9). As the oxidative current approaches zero (mode 5), a potential difference across the area of each pixel due to the nontrivial resistivity of the porous Cu (Supplementary Note 5, Supplementary Figs. 10 and 11) causes faster removal of Cu near the edges than the center. Eventually, the pixels return to mode 1 (mode 6). The measured cross-polarized reflection spectra of selected pixels with $p = 290$, 370 and 450 nm displayed over deposition and dissolution time (Fig. 3d) provide a clearer description of the spectral changes, whose qualitative trends from mode 1–3 agree well with our earlier analytical calculations. Peak wavelength shifts of 199, 70 and 53 nm were observed for the three pixels with the corresponding chromaticities shown in Supplementary Fig. 12. These shifts are comparable to recent achievements from metal electrodeposition and ion intercalation designs while the...
measured on/off contrasts are among the highest when compared to those based on electrochromic polymers (Supplementary Note 6 and Supplementary Fig. 13).

Finite-difference time-domain (FDTD) simulations of the cross-polarized 0th order reflection spectra (Fig. 3e) using the models schematically illustrated in Fig. 3f confirm the bottom-up filling and random removal of Cu during the reductive and oxidative steps. A plane wave source and laterally periodic boundary conditions were used to capture all processes while avoiding the large computational demand associated with finite simulation volumes.

During deposition, nanosized Cu grains were assumed to fill the slits by accumulating from the bottom. Excess Cu deposited above the HCG height was modeled to grow in the form of a semi-elliptical cylinder to reflect the cross-sectioned profile in Fig. 2b (III) (See Supplementary Fig. 14). To mimic the disordered morphology of Fig. 2b (IV) during dissolution, randomly distributed ~30–140 nm-sized pores were continuously introduced until the Cu was completely removed. For comparison to the time-resolved spectra of Fig. 3d, the temporal evolution of the simulated spectra during deposition and dissolution is shown in Fig. 3e, described in detail in Supplementary Note 7 and Supplementary Fig. 15. The simulated trends qualitatively agree with the measured results (Fig. 3d), supporting the modeled mechanisms. Discrepancies are also observable, one of which is the sharper 1st order diffraction onset for the simulation. This is because the model uses periodic boundary conditions and a planewave source whereas our measurements probe a waveplanar source.

Conversely, the initial disorder accompanying Cu dissolution is actively tunable structural colors as dynamic display elements.

Demonstration as display elements. To highlight the practical applicability of our pixels, we present two types of demonstrations. The first describes a color-switching display that exploits the active tunability of the pixel colors and the second showcases a 3 × 5 pixel matrix featuring on/off switching. We fabricated fruit-shaped pixels with sizes between 30 and 90 µm, displaying green and blue colors passively encoded using HCGs with \( p = 290–450 \) nm (Fig. 4a). By applying a step voltage of \(-1.5 \) V for 0.5 s, the fruits change colors to orange and yellow, and rapidly return to their original state by reversing the voltage to 1.45 V for 0.8 s. This sub-second color tuning can be repeated with excellent regularity (see Supplementary Video 2) and without switching off due to the limited porous Cu volume. To switch off the tuned color, a larger potential of \(-1.9 \) V was applied for 3 s to increase the initial Cu volume, followed by an oxidative potential (See Supplementary Fig. 18 and Supplementary Video 3).

For our second demonstration, an individually addressable 3 × 5 pixel matrix comprising 50 × 50 µm² sized HCGs with \( p = 400 \) nm was prepared. Details of the fabrication procedure and the multichannel current control are provided in Supplementary Note 8, Supplementary Figs. 19 and 20. To demonstrate on/off switching of the untuned color from each pixel, a short step potential of \(-1.7 \) V was applied for 0.3 s to quickly deposit Cu within the HCG slits, followed by a reverse potential of \( 0.7 \) V for 2.2 s to dissolve it and yield the porous morphology. To sustain the off state, a negative potential of \(-0.37 \) V that cancels the excess cell potential driving the acidic dissolution of Cu must be applied (Supplementary Fig. 21). Fig. 4b and Supplementary Video 4 demonstrate selected pixels of the matrix sequentially turn off. By biasing multiple pixels, the same matrix displays the words 'SURE' and 'NANO' (Fig. 4c and Supplementary Videos 5–6), proving its feasibility as an alphabetic display. We note that the current performance is limited by cross-talk driven by potential gradients across pixels, which may be addressed by adding diffusion barriers that act as isolation banks. Our designs achieve switching rates up to 2.17 and 0.48 s⁻¹ for the on and off state, respectively, and color tuning rates up to 2 and 1.25 s⁻¹ for the change and return, respectively. These speeds are comparable or even faster than those of previous metal electrodeposition designs1, and can be further enhanced by increasing the electrolyte conductivity or decreasing the pixel area to improve the switching kinetics. We also note that the current design relies on the use of separate polarizers for incident and reflected light. Although this is simple to implement in a microscope, it is not so straightforward in an actual display where space is major constraint. To this end, ‘Janus’ metasurfaces are a promising solution, where different optical functions are encoded for light of opposite propagation directions via meta-atoms with broken out-of-plane symmetry in a thin sheet38. Such a metasurface supporting asymmetric transmission (e.g., forward-propagating linearly polarized light and backward-propagating orthogonally polarized light)39 offers a possible alternative to our cross-polarized scheme.

In summary, we have demonstrated a dynamic structural color pixel that achieves actively tunable colors and on/off switching through distinct optical mechanisms entailing the electrochemical redox of Cu ions on the exposed parts of a Pt electrode below an HCG. Modal interference of waveguide-array modes in the HCG and the status of the second s-pol mode which can be ‘muted’ or ‘unmutted’ by the Cu occupancy determine the bare and Cu-filled HCG colors under our cross-polarized imaging setup. This mechanism contrasts with those based on surface plasmon polaritons and yields vibrant colors free of plasmonic loss. Conversely, the initial disorder accompanying Cu dissolution yields an off state via broadband plasmonic absorption and depolarized scattering. In addition to the superior chemical stability of Pt, high contrast, and low operational potential (\( AV < 3 \) V), our pixels offer the key functions needed to promote actively tunable structural colors as dynamic display elements.
Methods

Pixel fabrication. A sub-1nm thick Ti wetting layer and ~170 nm-thick Pt layer were sequentially DC sputtered onto a wet oxidized Si wafer (8000 Å) in an Ar atmosphere. Negative (ma-N 2403) or positive resist (PMMA A4) with thicknesses between 300 and 350 nm were spin-coated on top of the Pt substrate. Using e-beam lithography, repeating rows of 10 μm-long grating patterns were written to create 50 × 50 μm²-sized pixels. Depending on type of resist, substrates were either developed with AZ300MIF for 60 s or a 3:1 mixture of IPA and water for 45 s. 100 nm-thick TiOx was e-beam evaporated onto the developed substrates at a 0.04 nm/s deposition rate, followed by lift-off.

Electrochemical cell fabrication. A Pt WE supporting the pixels and ITO CE with two drilled holes (0.75-mm-diameter) for electrolyte injection were separated by a 60-μm-thick thermal film (Meltonix 1170-60) whose central volume was cut out to host the electrolyte. Adhesion between the components was ensured by heating the film up to 135 °C. Electrolyte composed of copper (II) nitrate trihydrate (1 M; Cu(NO3)2∙3H2O) dissolved in dimethyl sulfoxide (DMSO) was injected into the cell via one of the holes in the CE using a micropipette. Air was simultaneously drawn from the other hole with a separate micropipette to avoid trapped bubbles in the cell. Adhesive Cu tape was attached to each end of the WE and CE to provide electrical connection to the potentiostat (CompactStat.h, IVIUM) terminals.

Optical measurement. Cross-polarized reflection was measured with a home-built confocal microscope. White light from a halogen lamp was delivered through a linear polarizer and normally illuminated onto the pixels inside the electrochemical cell. The reflected signal was filtered by a separate polarizer oriented orthogonal to the original one. To obtain an image, a 0.3 NA objective lens was used to collect and deliver light into a CCD (STC-TC202USB-AS, SENTECH). For the spectrum, a 0.15 NA objective lens and spectrometer (Acton SP2300, Princeton Instruments) fiber-coupled to the pinhole were used.

Characterization. Top-view spatial analysis of the pixels was performed with a field-emission scanning electron microscope (JSM-6700F, JEOL) at an operating voltage of 15.0 kV. Side-view analysis was performed by cross-sectioning the HCGs via focused ion beam milling (crossbeam 540, ZEISS). Lattice fringes were acquired with a field-emission transmission electron microscope (JEM-2100F, JEOL) while EDS information was acquired in scanning transmission electron microscopy (STEM) mode, at an operating voltage of 200 kV.

Calculations. FDTD simulations were performed to compute the cross-polarized reflection and electric field distributions, while rigorous coupled-wave analysis (RCWA) was used to obtain the 0th order s and p-pol reflection efficiencies. For the FDTD simulations, a plane-wave light source was normally injected onto a grating rotated 45° with respect to the incident electric field vector. Periodic and perfectly matched layer boundary conditions were used for the x-y and z dimensions, respectively. Rectangular side-profiles were used to model the grating and fully deposited Cu for the modal analysis of Fig. 1 while trapezoidal gratings and granular Cu nanoparticles were used to simulate the experimental conditions of Fig. 3. To obtain the s and p-pol electric field distributions, plane-wave sources with light perpendicular and parallel to the grating vector were injected. A mesh volume of 2 × 2 × 2 nm³ was used to model the grating and Cu.

For the bare HCG analysis, a modal expansion method was used, with details provided in Supplementary Note 1. The modal profile in Fig. 1b was calculated at a 550 nm wavelength. Surface impedance boundary conditions (SIBC) were used for analyzing the Cu-filled HCG.

Data availability
Data presented in this publication is available on Figshare with the following identifier (https://doi.org/10.6084/m9.figshare.19771957.v1).

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Fig. 4 Demonstration of active color display. a Cross-polarized OM images of an apple, blueberries, and lemon, switching color in response to an applied potential alternating between −1.5 V and 1.45 V for 0.5 and 0.8 s, respectively, acquired at odd cycle numbers. b, c Cross-polarized OM images of a 3 × 5 pixel matrix showing (b) each pixel sequentially turned off and on and (c) letters by turning selected pixels off, proving its feasibility as an alphabetic display (all scale bars: 50 μm).
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