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Stepwise anodic electrodeposition of nanoporous NiOOH/Ni(OH)$_2$ with controllable wettability and its applications
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A nanostructured surface with diverse wettability which has the properties of superhydrophilicity, superhydrophobicity and distinguished wetting contrast is important to several key technologies. To meet these requirements, a facile and cost-effective approach via the electrochemical fabrication of a nano-sponge-like nickel oxyhydroxide/nickel hydroxide film on conductive glass followed by the application of external stimuli or chemical treatments is presented. The external stimuli of an environmental chamber and UV/ozone can dynamically and selectively control the aspect of wettability between hydrophobic and superhydrophilic in a short period. In addition, the aid of a chemical treatment with self-assembled non-fluorinated silane on annealed nickel hydroxide film can result in superhydrophobicity without degradation of optical transparency. The effectiveness of these achieved functions is demonstrated using transparent 2D microfluidic channels and direct selective electroplating.

Keywords: nickel oxyhydroxide; nickel hydroxide; electrochemical fabrication; superhydrophilic; superhydrophobic; microfluidic channels; selective plating

1. Introduction

Habitats’ survival adaptations in nature with wide-ranging conditions lead to diversity in the property of wettability, examples of which can be found in the superhydrophobic properties of the lotus leaf, the superhydrophilic properties of the moss leaf and the hydrophobic/hydrophilic pattern on the back of the desert beetle.[1–3] Inspired by these natural occurrences, researchers developed various applications of diverse wettability, including self-cleaning, anti-icing/anti-fogging, microfluidic channels, cell encapsulations and micropatterning.[4–8] The artificial control of surface topography and surface energy via external stimuli or chemical treatments using a one-step or two-step method can achieve a great range of wettability.[9–14] For example, by using dark storage and ultraviolet (UV) light irradiation, photosensitive inorganic materials such as hierarchical TiO$_2$ or ZnO nanorod reversibly convert the water contact angles (WCAs) between superhydrophilic ($<10^\circ$) and superhydrophobic ($>150^\circ$), owing to the recovery and generation of oxygen vacancies from bridging sites.[15,16] A superhydrophobic surface is usually fabricated by roughening the surface structures, whose processes include layer-by-layer deposition, sol-gel coating and electrochemical deposition, followed by a chemical treatment with a low-surface-energy material (fluorinated or hydrocarbon silanes, phosphates and thiols).[12,17]

One of the great challenges in achieving an extreme wetting contrast greater than 140$^\circ$ (difference of the WCA between hydrophobicity and hydrophilicity) is the extraordinarily long response time (several days or weeks) due to the slow recovery of hydrophobicity in the dark, which is infeasible for technological applications. Furthermore, when fabricating a superhydrophobic surface, it is usually difficult to maintain the optical transparency because the increase of the surface roughness enhances the effect of Mie scattering, which hereby hinders the optical applications.

Several groups of researchers achieved highly transparent superhydrophobic coatings, typically using SiO$_2$-based materials, but the generation of extreme wetting contrast was seldom dealt with in these experiments.[18–20] Park et al. [21] successfully created a superhydrophilic/superhydrophobic surface on transparent SiO$_2$ substrates, but the fabrication process was complicated and expensive. Alternatively, photosensitive azobenzene, thermal-responsive poly(N-isopropylacrylamide) (PNIAAm) and solvent-responsive block copolymer composed of polystyrene and poly(acrylic acid), offer rapid response times (several minutes) but exhibit a small wetting contrast ($<40^\circ$) due to their inherently smooth surfaces.[22–25]

Up to now, limited research has been conducted on creating multifunctional coatings with a facile switch
between two extreme wetting states with non-degraded optical transparency in the superhydrophobic state in a simple and cost-effective way. This paper therefore aims to electrochemically fabricate a nanostructured nickel oxyhydroxide (NiOOH) film on a fluorine-doped tin oxide (FTO) and then expose it to external stimuli or a chemical treatment in a process which is inexpensive, relatively fast and results in ease of controlling and reproducing surface morphologies. Upon exposure of the as-prepared NiOOH film to an environmental chamber (EC, at 85°C and with 70% relative humidity [RH]) and UV/ozone, a rapid, reversible and vast change occurs, moving from a hydrophobic (142.4° ± 2°) to a superhydrophilic (1.1° ± 1°) state within 60 minutes. In addition, with the further aid of a chemical treatment of non-fluorinated octadecyltrimethoxysilane (ODS) self-assembled monolayers (SAMs) on nanoporous NiOOH/Ni(OH)₂ film, a superhydrophobic Ni(OH)₂ surface (151° ± 1°) was achieved without degradation of optical transparency. To demonstrate the repertoire of these achieved properties, the disposable and rewritable two-dimensional (2D) microfluidic channels with high transparency and wetting-contrast enhanced selective electroplating were investigated.

2. Experimental method
2.1. Fabrication of the nanoporous NiOOH film
The experiments were carried out using an anodically electroplated NiOOH film on flat FTO glass (Roughness root-mean-square: 30.4 nm) followed by the external triggers of exposure to an EC and UV/ozone or the chemical modification of the ODS-SAMs. A CHI 660E potentiostat/galvanostat was used in the electrochemical process in a standard three-electrode configuration with a platinum mesh as the counter electrode, saturated Ag/AgCl as the reference electrode and FTO as a working electrode. The FTO glass (1 cm × 1.5 cm), with a sheet resistance of 7.6 Ω□ and an optical transmittance of 80%, was first rinsed with 4% PK-LCG545 (Parker Corp.) and deionised (DI) water at 50°C for 15 minutes with sonication, respectively. The clean FTO glass was then immersed into a neutral plating solution composed of 0.13 molar concentration (M) sodium acetate, 0.13 M nickel sulfate and 0.1 M sodium sulphate at room temperature. The step-wise constant current densities were employed for the anodic electrodeposition of nanoporous NiOOH film on FTO. The three-step procedure started from a very low current density of 0.005 mA/cm² for 30 minutes and then jumped to 0.05 mA/cm² for the next 30 minutes and continued to 0.5 mA/cm² for the last 30 minutes, as shown in Figure 1(a).

2.2. External stimuli and chemical treatment
The as-deposited superhydrophilic NiOOH film was exposed to an EC at 85°C with 70% RH (HongHui, THH80L) or a hot plate at 100°C for 60 minutes to form the hydrophobic Ni(OH)₂ film. Then, the hydrophobic Ni(OH)₂ film was put into a UV/ozone atmosphere (Kingo, TK-110) for 10 minutes to restore the WCAs back to their original state of superhydrophilicity. Furthermore, the samples annealed using the hot plate were immersed into a solution of 3% (v/v) ODS and 0.5% (v/v) butylamine in toluene for 20 minutes. Subsequently, the

Figure 1. (a) Anodic plating profile of nanostructured γ-NiOOH; (b) and (c) top and cross-sectional FE-SEM images of as-prepared nanostructured γ-NiOOH/FTO, respectively; (d) and (e) HR-TEM image of as-prepared nanostructured NiOOH film and its corresponding SAED pattern, respectively; (f) SAED pattern of nanostructured α-Ni(OH)₂ film after exposure to a hot plate for 60 minutes.
ODS-SAMs treated samples were annealed at 100°C for 60 minutes to form the tight hydrogen bonds between the hydroxyl groups of Ni(OH)\(_2\) films and the hydrolysed trimethoxy groups of ODS. All the samples showed a similar sheet resistance (7.6 Ω/□ to 7.8 Ω/□) to the bare FTO, even after different treatments.

2.3. Effects of the electrochemical conditions

The same plating parameters were applied to single-crystalline TiW to study the influence of native difference in substrates. Furthermore, combined cathodic and anodic plating was introduced to the FTO where the plating parameters of cathodic plating were under −8 V for 3 minutes, followed by the same process of anodic plating.

2.4. Applications

The wetting contrast between the hydrophobic Ni(OH)\(_2\) film and superhydrophilic NiOOH film was applied using rewritable 2D microfluidic channels and direct selective electroplating. The masks (UV/ozone-resist stickers) with a Y-shaped, smile pattern and four vertical stripes were fabricated in the microscale via CO\(_2\) laser ablation. Subsequently, the pre-designed masks were attached to the whole of the hydrophobic Ni(OH)\(_2\) surfaces treated with the EC in advance, whereas the unmasked areas were exposed to the UV/ozone atmosphere to generate superhydrophilicity. Finally, the superhydrophilic-hydrophobic pattern was created after peeling the masks away. The water droplets with a micro volume were injected into the Y-shaped and smile pattern. For the direct selective electroplating, the sample with the superhydrophilic-hydrophobic striped pattern was immersed into commercial nickel (Caswell) electrolyte for 270 seconds under −0.95 V. Similarly, the wetting contrast between the superhydrophobic ODS/Ni(OH)\(_2\)/FTO and the hydrophilic FTO was demonstrated using transparent 2D microfluidic channels. Here, the bottle-shaped mask made from the electroplating Ni layer was applied to protect the underside of the FTO from chemical contamination with regard to the anodic plating of Ni(OH)\(_2\) film and the chemical treatment of the ODS-SAMs. In contrast, the unmasked areas were subjected to successive Ni(OH)\(_2\) deposition and ODS-SAMs treatment to create superhydrophobicity. Lastly, the nickel mask could easily be removed with an ordinary tape so that the bottle-shaped 2D microfluidic channels were obtained.

2.5. Characterisation

The WCAs of each sample were measured with DI water by a contact angle goniometer (Sindatek Model 100SB) at five different points. To calculate the surface free energy of each state and analyse its components, DI water and methylene iodide (MI) were employed as probing liquids. The morphology, atomic structure and surface state of the NiOOH/Ni(OH)\(_2\) film were determined using a field-emission scanning electron microscope (FE-SEM, Hitachi S-4800), a high-resolution transmission electron microscope (HR-TEM, Tecnai, G220S-Twin) and X-ray photoelectron spectroscopy (XPS, Physical Electronics 5600).

3. Results and discussion

The three-step constant current densities were employed to anodically electrodeposit the nanoporous NiOOH films on the polycrystalline FTO glass, as shortened as NiOOH/FTO. Figure 1(b) and (c) show the top view and cross-sectional FE-SEM images of the three-dimensional (3D) nano-sponge-like NiOOH/FTO with average pore sizes of 150 nm and a thickness of 600 nm. The corresponding selected-area electron-diffraction (SAED) pattern from the HR-TEM image (Figure 1(d)) identifies that a polycrystalline γ-NiOOH film with interplanar spacing of 6.9 Å was formed (Figure 1(e)). After being subjected to the hot plate, the morphology remained the same; however, the corresponding SAED pattern in Figure 1(f) shows that the interplanar spacing expanded to 7.6 Å, which corresponds to the α-Ni(OH)\(_2\) film. Furthermore, the relatively poor crystallinity of the annealed Ni(OH)\(_2\) film was observed compared to the as-prepared NiOOH film. Therefore, the change in chemical composition and local crystallinity induced from thermal energy with the combination of special nano-sponge-like geometry is expected to have a profound effect on the surface wettability.[27,28]

The wettability of the as-prepared and stimuli-treated films was evaluated using contact angle measurements (Figure 2(a)). The nanostructured γ-NiOOH film is a superhydrophilic material with WCAs of 4.9° ± 1°, resulting from the combined effect of an intrinsically high water affinity on the edge plane and a 3D nano-sponge-like morphology. Accordingly, enhanced 2D and 3D capillary effects both lead to water spreading and penetration.[29,30] After being heated with the EC (85°C with 70% RH) and the hot plate (100°C) for 60 minutes, the WCAs increase to 142.4° ± 2° and 112.5° ± 5°, respectively. The main difference between these two heat sources is that the EC provides sufficient hot water vapours, which plays an important role in mediating wettability. The de-wetting evolution from superhydrophilic to hydrophobic is owing to the change in chemical composition and local crystallinity. The α-Ni(OH)\(_2\) film comprises a stacked Ni(OH)\(_{2-x}\) layer with an intercalation of anions and water molecules in-between, where the basal planes of the Ni(OH)\(_{2-x}\) layer possess inherent hydrophobic properties.[31] Furthermore, the symmetry
of the water molecules (C2v) does not match that with the Ni(OH)2−x layer (D3h or O h), meaning that the consistent supply of water vapours from the EC causes a local rearrangement of crystallographic orientation, leading to local disorder in the Ni(OH)2 film.[32] The disorder feature results in the formation of a nanoscale discontinuous three-phase contact line (TCL).[33] All these results make the Ni(OH)2 film prefers air traps rather than water absorption. The disorder transition in local crystallinity as a result of thermal treatments of the Ni(OH)2 film is illustrated in Figure 2(c).

After the hydrophobic Ni(OH)2 film was placed into a UV/ozone atmosphere (265 nm) for 10 minutes, its superhydrophilic state (1.1° ± 1°) was restored again. The wetting contrast of nanostructured films between the alternative exposure to the EC and UV/ozone is over 140°. This process was repeated for over 10 cycles and good reversibility of the surface wettability was obtained (Figure 2(b)).

An XPS of O 1 s-orbital (s) spectra was applied to verify the surface chemical state and the surface water adsorption (Figure 3). In the O 1 s region, as-prepared and UV/ozone treated samples were mainly deconvoluted into three peaks, which correspond to a lattice of O2− (529.5 eV), OH− (531.1 eV) and hydroscopic H2O (532.2 eV).[34] On the other hand, the O 1 s spectrum of the EC-treated sample was only comprised of an OH− characteristic peak. The table inset in Figure 3 indicates that the normalised O2−/OH− ratios for the as-prepared, EC-treated and UV/ozone-treated samples were 0.97, 0.00 and 0.32, respectively. It is apparent that as-prepared stoichiometric γ-NiOOH was reduced to pure Ni(OH)2 via the EC treatment and then converted back to OH−-enriched γ-NiOOH during the UV/ozone treatment. Compared to the as-prepared sample, the OH− enrichment of the UV/ozone treated sample comes from the bridging of hydroxyl radicals to its surface during hydrolysis caused by UV/ozone process.[35] It is also noteworthy that the amount of surface hydroscopic H2O is in good agreement with the wetting and de-wetting behaviour of samples under different conditions.

To advance the understanding of wetting behaviours of NiOOH and Ni(OH)2, respectively, surface free energy...
Figure 3. XPS of nanostructured as-prepared film (black curve) and its exposure to EC (red curve) for 60 minutes and UV/ozone (green curve) for 10 minutes. Note: The table in the inset is the summary of O 1 s peak fitting results. Please view the online version for the colour reference (doi:10.1080/1023697X.2015.1102657).

was calculated according to Owens-Wendt method (Table 1).[36] By applying the DI water and MI as probing liquids, it can be derived that the surface free energy of the NiOOH film is 80.96 mN/m with a large portion of polar interaction (30.28 mN/m), while the surface free energy of Ni(OH)₂ film is 25.07 mN/m with little polar interaction (3.08 mN/m).

The long-term stability of the as-prepared NiOOH film was monitored in an ambient atmosphere (Figure 4(a), black curve). The WCAs gradually increased due to surface contamination from the environment and finally reached a saturated state. The Ni(OH)₂ film annealed using the hot plate was modified with a 3% solution of ODS in toluene for 20 minutes and marked as ODS/Ni(OH)₂/FTO. The red curve of Figure 4(a) shows that a superhydrophobic surface (151° ± 1°) with a sliding angle of 5° was achieved and maintained over one week due to a significant reduction in surface energy via the permanent chemical adsorption of long hydrocarbon chains of ODS-SAMs. The exposed long hydrocarbon chains exhibit non-polar properties, which help trap air and form air pockets. To form compact SAMs on FTO, the degrees of surface hydroxylation and free sites to form silanol (Si-OH) groups are critical.[37] Thanks to the terminated hydroxyl groups and nano-sponge-like structure of Ni(OH)₂ film, the surface coverage of ODS-SAMs could be remarkably enhanced. When water droplets were in contact with ODS/Ni(OH)₂/FTO, they were suspended on the top of the surface asperities and the air pockets, which fit the wetting behaviour described in Cassie-Baxter Theory.[38]

Furthermore, the optical transparency and superhydrophobicity are exhibited in Figure 4(b), which shows the clearly readable characters of “HKU” underneath the ODS/Ni(OH)₂/FTO, suspended with the spherical shape of the water droplets. According to UV-visible spectra, the transmittance of ODS/Ni(OH)₂/FTO was slightly enhanced in the ultraviolet range and maintained in the visible range compared to the bare-FTO glass. The non-degraded optical transmittance may probably be attributed to inherent transparent features and the subwavelength pore size of nickel hydroxide films so that the effect of Mie scattering is mitigated.[39]

The effects of electrochemical conditions on the morphologies are also investigated. Figure 5(a) and (b) shows that curved nanoporous microspheres are obtained on single-crystalline TiW, which is significantly different with the morphology on polycrystalline FTO (flat porous nano-sponges) through anodic plating. The native differences of these two substrates are the amounts of defects. There are low amounts of defects in near single-crystalline TiW, offering fewer active sites for nucleation than that of FTO. As a result, the growth of NiOOH prefers on the existing islands of NiOOH nuclei, which finally turns to a spherical structure on TiW in order to reduce surface energy. More interestingly, applying combined cathodic and anodic plating leads to the coexistence of rod-like and nanoporous structures (Figure 5(c)
Table 1. The surface free energy of as-prepared NiOOH film and EC-treated Ni(OH)\(_2\) film.

<table>
<thead>
<tr>
<th>Solid</th>
<th>WCAs of DI water (°)</th>
<th>WCAs of MI (°)</th>
<th>(\gamma_s) (mN/m)</th>
<th>(\gamma_d) (mN/m)</th>
<th>(\gamma_p) (mN/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-prepared</td>
<td>6.9</td>
<td>4</td>
<td>80.96</td>
<td>50.68</td>
<td>30.28</td>
</tr>
<tr>
<td>EC 60 minutes</td>
<td>138</td>
<td>71.6</td>
<td>25.07</td>
<td>21.98</td>
<td>3.08</td>
</tr>
</tbody>
</table>

Note: \(\gamma_s\) = surface free energy of a solid; \(d\) = dispersion component; \(p\) = polar component.

Figure 4. (a) Long-term stability of the as-prepared NiOOH film and ODS/Ni(OH)\(_2\)/FTO film stored in an ambient environment for eight days; (b) optical transmittance of the bare FTO and ODS/Ni(OH)\(_2\)/FTO film in the UV-visible range.

Note: The insets in Figure 4(a) show the corresponding photographs of water droplets after eight days and the sliding angle of 5° in the superhydrophobic state.

Figure 5. (a) and (b) FE-SEM images of curved porous microspheres on single-crystalline TiW through anodic plating under different magnifications; (c) and (d) FE-SEM images of a mixture of rod-like and porous nanostructures through combined cathodic and anodic plating on FTO under different magnifications.

and (d)). It is because this cathodic plating generates electrically insulated nickel oxide and nickel hydroxide which block some active sites during anodic plating. As a result, two distinctive nanostructures coexist, which are believed to have a unique optical response compared to a single nanostructure. Their corresponding optical responses will further be investigated in future study.
Open-air 2D microfluidic channels are of great interest compared to conventional 3D microfluidic channels for screening tests, pharmaceutical control, food tests and environmental analysis due to facile inlet access and easily direct observation. The surface-tension-confined micro-fluids resulting from a wetting contrast were first proposed by Lam et al. [40] by creating a hydrophobic-hydrophilic pattern. The microfluidic drops were automatically propagated along the superhydrophilic Y-shaped paths, with an average width of 260 μm observed without any externally propelled forces, as though a virtual wall was surrounded (Figures 6(a) to (e)). In the case, the hydrophobic Ni(OH)₂ region with EC treatment serves as a virtual wall which precisely guides and confines the flow direction of microfluidic drops. Ideally, a small height and large wetting contrast favour the motion of microfluidic drops due to the large capillary force. A flow speed of 0.82 mm/second using DI water as microfluidic drops was measured. Furthermore, good reversibility in wettability upon the alternative treatment of EC and UV/ozone makes it possible to erase the Y-shaped pattern on the Ni(OH)₂/FTO and change it into a smile pattern. Figure 6(f) shows wet eyes and mouth on a dry face, demonstrating that an arbitrary pattern with high wetting contrast can simply be reproduced using the same patterning technique.

To create a transparent superhydrophobic-hydrophilic pattern on FTO, another mask material fabricated by electroplating a bottle-shaped Ni layer (width = 500 μm, diameter = 2 mm, length = 1.2 cm) was employed to preserve the intrinsically hydrophilic property of FTO. Then, the Ni(OH)₂ film was anodically electroplated on FTO covered with a patterned nickel layer and followed by permanent ODS-SAMs modification. Lastly,
Figure 7. (a) Cathodic waves of cyclic voltammetry for nickel electroplating on the surface of the as-prepared NiOOH and EC-treated Ni(OH)2 films; (b) FE-SEM image of nickel nanoparticles embedded into superhydrophilic domains of nanoporous NiOOH film at \(-0.95\) V for 270 seconds.

Note: Inset in Figure 7(b) is a photograph of the nickel stripes.

the patterned nickel layer can simply be removed by ordinary tape due to its weak adhesion to the smooth FTO surface, meaning that the predetermined design of a bottle-shaped microchannel can be obtained. Figure 6(g) shows the injection process of the microfluidic drops into a transparent 2D microfluidic channel. It is clear that the wetting contrast between the superhydrophobic (ODS/Ni(OH)2/FTO) and hydrophilic (FTO) domains prevents the green water inks from laterally overflowing, even though their height overwhelm the physical thickness (600 nm) of the nano-sponge-like Ni(OH)2 film (Figure 6(h)). The flow speed of the green water inks was quick (\(~5\) mm/second).[41] Such a microfluidic channel with irreversible wettability and rapid flow speed is well-suited to disposable devices. The influence of design, wetting contrast and geometry of microfluidic channels as well as the viscosity and surface tension of the test liquid on the flow speed of the microfluidic drops will be investigated in detail in the future.

Conventional photolithographic techniques need specific masks, while direct selective plating is mask-free and thus saving on materials and simplifying the process.[42–44] The NiOOH/Ni(OH)2 films were found to be as electrically conductive as bare FTO, even after EC or UV/ozone treatment. Possessing the demonstrated features of easy patternisation and distinguished wetting contrast, the application of NiOOH/Ni(OH)2 films in direct selective plating was also evaluated. A difference in the onset voltage of the nickel plating on the superhydrophobic NiOOH film (V\(_{\text{onset}}\): \(-0.91\) V) and the hydrophobic Ni(OH)2 film (V\(_{\text{onset}}\): \(-1.05\) V) was observed (Figure 7(a)). The superhydrophilic domains are more reactive to the metallic electroplating due to their high surface free energy and good wettability, leading to a relatively low barrier of excess free energy for heterogeneous nucleation.[45] The native difference in surface activity allows the nickel electroplating to occur only on the superhydrophilic stripes if the employed potential is less than \(-1.05\) V. Here, a superhydrophilic-hydrophobic pattern could be regarded as a virtual mask when immersing the entire surface into a nickel-plating electrolyte. As observed in the inset of Figure 7(b), the direct selective plating of nickel stripes was achieved. Furthermore, the sizes of nickel nanoparticles correspond well with the pore size of nano-sponge-like NiOOH film (Figure 7(b)). This proves that the electrolyte is first absorbed and penetrated into porous structures due to a strong capillary force, and then nickel nanoparticles nucleate and agglomerate each other in the confined space.

4. Conclusion

A facile and cost-effective method of fabricating a nano-sponge-like NiOOH/Ni(OH)2 film is developed, the surface wettability of which can be manipulated with external triggers or chemical treatment. The chemical composition and local crystallinity of the nanostructured film dynamically respond to exposure to an EC and UV/ozone. The extreme wetting contrast above 140° can be accomplished within 60 minutes. The surface wettability can further be boosted up to a superhydrophobic state (151°) with the aid of ODS-SAM modification. The diverse status of the surface wettability achieved in a facile way bridges the gap between theory and practical application. Transparent 2D microfluidic channels with disposable or rewritable features, as well as direct selective plating of metallic stripes, have successfully been demonstrated. The findings open up new opportunities in developing metal oxyhydroxide and hydroxide materials which are sensitive to external triggers.

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