Transparent Conductors



All-Solution-Processed, Scalable, Self-Cracking Ag Network Transparent Conductor

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A scalable and a fully solution-based method is developed to produce environmentally stable Ag micro/nanowire networks for transparent conductors. By applying a self-cracking, water-soluble acrylate copolymer film as a photoresist mask, the need for formal photomask fabrication and costly vacuum and lithographic facilities is obviated. Also, an increase in adhesion and a decrease in roughness of the metal networks is demonstrated by depositing metal into the regions created by the glass etch step. As a result, the networks can potentially be inexpensively scaled to large areas, as well as be flexible after removal from the substrate. They also exhibit record values of figures of merit that have been employed in the literature, offering a possibly excellent replacement for ITO.

1. Introduction

Materials with simultaneous high electrical conductivity and high optical transmittance are essential for numerous optoelectronic devices and applications, such as flat panel displays (TVs and computer monitors), touch screen displays (smart phones and tablets), thin film (e.g., perovskite) solar cells, organic lightemitting diode (OLED), electrochromic displays, and electromagnetic shielding.^[1–6] The predominant material used for such a transparent conductor or transparent conducting electrode (TCE) is tin-doped indium oxide (ITO). ITO has optical transmittance greater than 80% in the visible range, and sheet resistance of about $10 \Omega \square^{-1}$. There are two major reasons researchers are working to develop a replacement for ITO as a

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TCE: i) ITO contains indium, a rare earth metal that is relatively rare and costly to refine, as well as relatively brittle, limiting its use in flexible applications; and ii) fabrication of ITO and related TCE's requires vacuum-based deposition processes, such as physical vapor deposition (e.g., sputter deposition or thermal evaporation) or chemical vapor deposition. The cost of equipment for large scale implementation of these processes adds to the cost of the TCE, and thus to that of the end product.

Previously existing indium-free, allsolution processed TCEs include metal nanowires,^[7,8] chemically derived graphene oxide,^[9,10] and lithography-

processed metallic networks.^[11,12] Among these, the latter exhibits the best performance, but its production is costly and not generally scalable. Although the lithographic process makes highly controllable periodic patterns, such an aspect is not necessary for most applications of TCEs. With random patterns, a self-cracking method^[13,14] avoids lithographic processes but, to date, the metal must be deposited under vacuum because selfcracking templates (e.g., TiO₂,^[13] CA600,^[13] albumin,^[14] acrylics, etc.) that survive conventional wet processes have not been available. Vacuum-based deposition has higher facility costs and makes less efficient use of metal than solution-based deposition. Xian et al.^[15] and Gupta et al.^[16] have introduced electroless plating with self-cracking templates using Si as reducer or catalyst, but such a substrate is nontransparent.

Here, we present a method to produce scalable Ag metal networks on glass substrates which avoids both lithographic processes and vacuum metal deposition, and the network is continuous, such that its performance as a TCE is not impeded by thermally annealed junctions/contacts between individual nanowires. Silver is chosen because of its good electrical conductivity and chemical stability. Our process starts with photoresist-coated glass, soft-baked but not yet UV exposed. A water-based, self-cracking solution (e.g., nail polish) is then spincoated. This solution self-cracks upon drying, yielding a cracked film that functions as a photomask for the underlying resist. After UV exposure and development of the resist, a nonwater-soluble random pattern of gaps/crackles in the resist under the cracked nail polish film remains. We then use buffered oxide etch (BOE) to etch the exposed glass under the gaps, thus creating channels in the substrate and increasing the adhesion^[17] of metal in the subsequent step, electroless SCIENCE NEWS __ www.advancedsciencenews.com

deposition of Ag. The photoresist is then removed by lift-off in acetone, yielding a connected network of Ag wires on glass. The thickness of the Ag wires can be increased by electroplating, which also both increases conductivity and reduces sample roughness by filling in the etched glass channels.

2. Results and Discussion

Figure 1 is a schematic of our fabrication process for an allsolution-processed TCE. First, bis(trimethylsilyl)amine (HMDS) and then Shipley S1805 photoresist are spin coated onto a glass substrate, and soft-baked to dry. A water-based nail polish (e.g., Qian Zhi Xiu Co., Shenzhen, China) solution is then spin coated over the photoresist, and allowed to dry. This nail polish selfcracks as it dries in air, but does not adhere to the underlying photoresist to such an extent as to crack that as well. Figure 2a shows an optical microscope image of cracked nail polish on photoresist on glass. Another important property of this nail polish is that it blocks UV light, which makes it an appropriate photoresist mask. After 405 nm UV illumination for 2 s, only the photoresist not covered by the nail polish (i.e., in the gaps created by the cracking) is exposed and decross-linked, and thus becomes soluble in resist developer (MF319). As the nail polish mask also dissolves in that water-based developer, it is also removed in this step. After developing for 1 min and drying with nitrogen gas, one is left with a random network of cracks in the photoresist over the glass, the crack pattern having been transferred from the self-cracking nail polish. An example photoresist pattern is shown in Figure 2b.

The intermediate photoresist pattern is stable in both acidic and alkaline solutions, and so can act as a mask for subsequent BOE etch and Ag electroless deposition processes. To protect the back and sides of the glass substrate from the BOE and Ag electroless deposition processes, they are covered with tape. Notably, though we only present Ag networks in this work, the chemical stability of our cracked photoresist patterns suggests they can be used as masks for electroless deposition of other metals, and so this technique should be applicable to producing networks composed of a broad set of materials. In our implementation of the process, the substrates are placed in a



Figure 1. All solution-processed TCE fabrication process. a) Spin coat photoresist and soft bake; (b) spin coat self-cracking material; (c) material self-cracks upon drying in air; (d) UV expose photoresist (PR); (e) develop photoresist; (f) etch glass; (g) electrolessly deposit silver; (h) lift off photoresist; (i) electroplate silver.





Figure 2. Optical microscope images of three stages of preparation of cracked silver networks. a) Self-cracked material (nail polish) on photoresist on glass. b) Cracked photoresist (after removal of cracked nail polish) on glass. c) Silver network on glass, made by nail polish process. d) Silver network on glass, made by egg white process. Scale bar in (a), pertaining to all four images, is $500 \,\mu\text{m}$.

BOE solution for 2 min, which etches channels into the substrate $\approx 1 \, \mu m$ deep. The resulting etched channels' surfaces also facilitate increased adhesion of the subsequent electrolessly deposited Ag, as compared to non-etched surfaces, because of increased roughness and silanol groups created on the glass.^[17] The electroless Ag deposition process utilizes Tollens' reagent.^[18] This deposition method involves three reactions. First, the aqueous salt AgNO₃ is converted to silver oxide (Ag₂O) by OH⁻ ions and a brown precipitate forms. Second, aqueous ammonia is added which dissolves the brown silver oxide and forms aqueous $[Ag(NH_3)_2]^+$. Third, the $[Ag(NH_3)_2]^+$ complex is reduced by adding a solution which contains aldehyde groups, usually a sugar like glucose or dextrose. We used a dextrose solution which was added to the beaker containing the sample and Tollens' reagent, under continuous stirring. After about 2 min, about 100 nm-thick silver deposits on all surfaces in contact with the solution, including both photoresist and exposed glass on the top side of the substrate, as well as the tape on the protected back and sides of the substrate. The stripping of the photoresist to lift off the unwanted silver is performed by placing the substrate in acetone for 5 min, leaving behind a silver network at the bottom of the etched channels on a glass substrate, as shown in Figure 2c. The tape is also removed from the substrate back at this time. The fabrication process thus described is all-solution based.

The thickness of the silver in the etch channel bottoms can be increased by electroplating, with the existing silver network as the cathode facing a silver plating anode in silver electroplating solution. The thickness of the electroplated silver can be controlled by plating time, and can be built up to fill the entire depth of the etched channels as depicted in Figure 1i, without broadening the line widths. Also, using different spin speeds for the diluted nail polish ("crack material" in Figure 1b) allows one to control the thickness of the polish and, as a result, the average domain size of the nail polish crack pattern. The higher the spin



speed, the thinner the nail polish and smaller the domain size. In **Figure 3**, we show optical microscope images of completed TCEs made using various spin speeds. By increasing the speed from 1000 to 6000 rpm in 1000 rpm increments, we observe the average domain size decreases from ≈ 200 to $\approx 50 \,\mu\text{m}$ and metal coverage ratio increases from ≈ 20 to $\approx 25\%$. In **Figure 4**, we also show how optical transmittance changes with spin speed. Higher speed yields smaller domain sizes and higher metal coverage ratio, thus lowering transmittance. This observation is consistent with our previous cracking work.^[13]

In an alternative process, the diluted nail polish can be replaced by 0.6 g mL^{-1} egg white/water solution. However, the smallest domain size we have been able to obtain from an egg white process is larger than 500 µm (Figure 2d). This large domain size is not suitable for applications that need fine features (e.g., displays). All samples presented in this paper are made by the nail polish process except that in Figure 2d.

Electroplating processes have been used previously^[14] to increase conductivity of already completed metallic networks, but there were two main drawbacks. First, although thick electroplating of metal decreases sheet resistance dramatically, it also increases the sample surface roughness. Second, because electroplating deposits metal in all directions, the widths of the metallic wires will increase along with their thickness. For samples having a high ratio of metallic wire width to domain size, increasing the wire width will increase the metal coverage ratio of the sample and thus decrease the optical transmittance. Our method solves both of these problems by electroplating the silver within the etched channels, which largely constrains the wire widths to the channel widths for thicknesses equal to the depth of the channels. In Figure 5, we show atomic force microscope (AFM) images of a representative sample before electroless deposition, after electroless deposition and after two stages of electroplating. We also show a cross-section view indicating the contributions of Ag at the various deposition stages, Figure 5d. One can see that electroless deposition provides a thin, nearly conformal coating of the exposed surfaces





Figure 4. Transmittance of all-solution-processed TCEs as a function of wavelength obtained with samples in Figure 3.

in the glass trench, and that electroplating walls fills the trench, nearly always with some degree of "dog ears" protruding above the top edges. Once the trench is filled at its middle, further plating forms a smoother wire, as shown. The surface roughness of such samples is generally less than those prepared without etched substrates because silver deposits from the negative height relative to the glass surface.

Figure 6 shows optical transmittance and sheet resistance before and after electroplating $\approx 2.5 \,\mu\text{m}$ thick silver. After electroplating, we were able to achieve sheet resistances below $0.01 \,\Omega \,\square^{-1}$ with only a slight ($\approx 1\%$) reduction in transmittance. The electroplating time for the sample shown in Figure 6 is 380 s. Our best value of $0.008 \,\Omega \,\square^{-1}$ is the lowest sheet resistance for a TCE we have found in the literature. **Figure 7**a is a photograph of a 100 mm-diameter glass substrate coated by our $2.2 \,\Omega \,\square^{-1}$ TCE, placed in front of a building, illustrating both the scalability of our process and the high transparency. In



Figure 3. Optical microscope images of completed TCEs made by the nail polish process. The differences in domain size result from various nail polish solution spin speeds. Spin speed, nominal domain size, and metal coverage ratio, respectively, are: (a) 1000 rpm, 197 μm, 20.5%; (b) 2000 rpm, 117 μm, 21.5%; (c) 3000 rpm, 95 μm, 22.0%; (d) 4000 rpm, 73 μm, 22.4%; (e) 5000 rpm, 68 μm, 24.5%; (f) 6000 rpm, 49 μm, 25.6%. Scale bar in (a), pertaining to all four images, is 500 μm.







Figure 5. AFM images of a representative nail polish-based sample. a) Pre-electroless deposition, (b) post-electroless deposition, (c) post-electroplated, all for the same area; (d) profiles along the cut lines indicated in the AFMs, showing the contributions to Ag thickness by electroless deposition (red), partial electrodeposition (yellow), and overfilled electrodeposition (gray).

addition, the resistance and transmittance of samples with low $R_{\rm s}$ changed little (\approx 1 and 2%, respectively) after 8 months in ambient air.

Some applications (e.g., wearable devices) of TCEs desire mechanical flexibility. As an optional step, by placing our postelectroplating sample in BOE for 5 min, we can etch the glass undercutting the silver network and release the silver network



Figure 6. Transmittance and sheet resistance of a typical low-resistance sample before and after electroplating. Sheet resistance was measured by the 4-point van der Pauw method.

from the substrate, making it free-standing and/or transferrable to a flexible substrate. Figure 7b is a photograph of a piece of a free-standing silver TCE $(0.01 \,\Omega \,\square^{-1})$ about 1 cm² in area, with resistance-measurement electrodes attached, placed above the digital readout of a resistance meter, showing high conductance (i.e., low resistance of 0.3 Ω) and high optical transparency. Side view and top view SEM images at various magnifications of a free-standing silver network on a stainless steel support are also shown in Figure 7.

One figure of merit (FoM) widely used to evaluate the performance of TCEs is the ratio of electrical conductance to optical conductance ($F_{\rm TCE} = \sigma_{\rm dc}/\sigma_{\rm opt}$), which can be written as $F_{\rm TCE} = Z_{\rm o}/\{2R_{\rm s}(T^{-1/2} - 1)\}$, where $Z_{\rm o} = \sqrt{\mu_{\rm o}/\varepsilon_{\rm o}} \sim 377 \,\Omega$ is the impedance of free space, R_s is the measured sheet resistance, and T the measured transmittance at $550 \text{ nm.}^{[2,19-22]}$ Another FoM that may be more physical is that proposed by Haacke,^[23] significantly more heavily weighing transmittance, $\phi = T^{-10}/T^{-10}$ $R_{\rm s}$, which has units of Ω^{-1} . There are vet others in the literature.^[24–27] basically refinements of these two. Our results. shown in Figure 8, compare favorably, in fact superior, in all FoM schemes with those of various silver TCEs from the literature; we use F_{TCE} and ϕ here to illustrate this. Our pre-electroplated results shown as red circles ($F_{\rm TCE}$ up to 1000; $\phi \sim 0.09$) are slightly better than almost all previous preparations, including those that use vacuum silver deposition (self-cracking template: right triangles, $F_{\text{TCE}} = 504$, $\phi = 0.03^{[13]}$; nanotrough: crosses, $F_{\text{TCE}} = 327$, $\phi = 0.03^{[28]}$) and solution silver deposition (silver nanowires: down triangles, $F_{\text{TCE}} = 212$, $\phi = 0.02^{[8]}$; lithography template: diamonds, $F_{\text{TCE}} = 376$, $\phi = 0.01^{[11]}$). The F_{TCE} of our post-electroplated samples (red squares, F_{TCE} up to 208 000, $\phi \sim 14$) is also higher than that of an electroless-based process that requires Si as a substrate (up triangles, $F_{TCE} = 1341$, $\phi = 0.13$)^[15] and an electroplating-based process that needs vacuum silver as a seed layer (left triangles),^[14] which appeared to have the previous record FoM values ($F_{\text{TCE}} \sim 20\,000, \phi = 1.3$). For reference, commercial ITO (blue star) has an $F_{\rm TCE} \sim 350$, $\phi \sim 0.03$.^[29] It should be noted, however, that this and all FOMs are most useful when comparing TCEs at the same or comparable transmittance or sheet resistance levels. In addition to the physical performance and solution-based fabrication advantages of the Ag network presented herein, the conductor material cost compares favorably to that of extant emerging and legacy TCE technologies,^[30] with a calculated consumed Ag cost of under 1.00 m^{-2} .

3. Conclusion

We have demonstrated an all-solution processed transparent conductor that can be scalable and inexpensively produced, via two innovations. First, we used a self-cracking material as a UV mask to avoid high cost lithography and vacuum processes, and second, we etched the glass substrate to create channels which are filled by subsequent electroplating. Moreover, using Ag as the metal material, the network films are chemically and environmentally stable, and compatible with other soft processes, offering them as a possible cost-effective, smart transparent conductor for e.g., OLEDs, thin film solar cells, and electromagnetic shielding.







Figure 7. Photography and SEM images of samples. a) Photograph of a 100 mm-diameter glass substrate coated by a TCE $(2.2 \Omega \square^{-1})$ placed in front of a building (red dash outline added for guidance). b) Photograph of a $\approx 1 \text{ cm}^2$ piece of a free-standing Ag network TCE $(0.01 \Omega \square^{-1})$, with electrodes attached, placed above the readout of a resistance meter (blue outline added for guidance). Side view (c) and top view (d), (e) SEM images at various magnifications of the free-standing silver network in (b). Scale bars: (c) $10 \mu m$; (d) $50 \mu m$; (e) $10 \mu m$.

4. Experimental Section

Preparation of Substrates: We demonstrated our process with two types of glass substrates. The first were $\approx 25 \times 25 \text{ mm}^2$, 1.1 mm thick glass substrates cut from $\approx 25 \times 75 \text{ mm}^2$ microscope slides. The second were 100 mm diameter soda lime glass wafers (University Wafer no. 1631). Both substrates were first sonicated in acetone (J.T. Baker Chemicals) for 10 min, followed by another 10 min sonication in isopropyl alcohol (IPA) (J.T. Baker), and then dried by blowing nitrogen gas.

Preparation of the Cracked Photoresist on Glass: HDMS (Ultra Pure Solutions, Inc) and positive photoresist (MicropositTM S1805, Dow Chemical Co.) were both spin-coated (Laurell Technologies Corp.) at 1000 rpm for 15 s and then 4000 rpm for 45 s. The coated substrates were baked on a hot plate (Barnstead Thermolyne) at 110 C for 1 min. Nail polish (Qian Zhi Xiu Co., Shenzhen, China) and deionized (DI) water were mixed with volume ratio 5:1. The diluted nail polish was spin coated for 15 s at 500 rpm and for 45 s at speeds between 1000 and 6000 rpm,



Figure 8. Transmittance versus sheet resistance of completed nail polishbased TCEs before (red circles) and after (red squares) electroplating Ag. Results from other approaches using silver, including commercial ITO (blue star), are included for comparison. Sheet resistance in this work was measured by the 4-point van der Pauw method.

depending of the desired domain size. The samples were then allowed to air dry for 5 min after the nail polish. They were then UV flood exposed (USHIO USH-350DS) and developed (in MicropositTM MF-319, Dow Chemical Co.), and rinsed in water for 5 min and dried by blowing nitrogen gas.

Silver Electroless Deposition: A buffered oxide etch (BOE) solution (7:1 volume ratio of 40% NH₄F in water to 49% HF in water) from J.T. Baker Chemicals was employed. Samples were placed in the BOE for 2 min. After BOE etch, the samples were immersed in DI water for 5 min. The Tollens' reagent process used a solution made by mixing a 60 mL of 0.1 M L^{-1} silver nitrate (Sigma–Aldrich Corp.) solution with a 30 mL, 0.8 M L^{-1} potassium hydroxide (J.T. Baker) solution. A brown silver oxide precipitate forms, which is then dissolved by adding ammonia hydroxide (J.T. Baker) dropwise, with stirring. A 6 mL, 0.25 M L^{-1} dextrose solution (Fisher Scientific) is added to this solution. The etched sample is then dropped into the mixed solution for 2 min with stirring. The volume of solution above is suitable for electroless deposition of a $25 \times 25 \text{ mm}^2$ sample. One can increase the volumes of all solution proportionally for larger samples.

Silver Electroplating: Constant 5 mA current (Gamry Instruments, Inc., Interface 1000 system) was applied across silver plating anode (Esslinger & Co.) and Ag network, both placing in ready-to-use silver plating solution (Krohn Industries, Inc.). The silver anode was sonicated in acetone and IPA for 10 min before use.

Characterization: A scanning electron microscope (JEOL JCM-6000), optical microscope (Olympus BX61) and AFM (Vecco Dimension 3100) were used to characterize the samples. Sheet resistances were measured by the van der Pauw method. A current source (Keithley 224) and voltage meter (Keithley 175A) were connected to samples using silver paste and gold wires. Optical transmittance was measured by using an integrating sphere system (Ocean Optics Spectroclip-TR) with a halogen light source (Ocean Optics HL-2000-FHSA) and spectrometer (Ocean Optics Maya 2000 Pro). All transmittance data presented are normalized to that of a clean, glass substrate. The performance of aluminosilicate glass coated by 175 nm ITO^[29] was compared with all other TCEs.

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Conflict of Interest

The authors declare no conflict of interest.

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