Photonic Curing of Solution-Processed Oxide Semiconductors with Efficient Gate Absorbers and Minimal Substrate Heating for High-Performance Thin-Film Transistors

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ABSTRACT: In this study, photonic curing is used to rapidly and effectively convert metal-oxide sol–gels to realize high-quality thin-film transistors (TFTs). Photonic curing offers advantages over conventional thermal processing methods such as ultrashort processing time and compatibility with low-temperature substrates. However, previous work on photonically cured TFTs often results in significant heating of the entire substrate rather than just the thin film at the surface. Here, sol–gel indium zinc oxide (IZO)-based TFTs are photonically cured with efficient gate absorbers requiring as few as five pulses using intense white light delivering radiant energy up to 6 J cm−2. Simulations indicate that the IZO film reaches a peak temperature of ∼590 °C while the back of the substrate stays below 30 °C. The requirements and design guidelines for photonic curing metal-oxide semiconductors for high-performance TFT applications are discussed, focusing on the importance of effective gate absorbers and optimized pulse designs to efficiently and effectively cure sol–gel films. This process yields TFTs with a field-effect mobility of 21.8 cm2 V−1 s−1 and an IOn/Ioff ratio approaching 108, which exceeds the performance of samples annealed at 500 °C for 1 h. This is the best performance and highest metal-oxide conversion for photonically cured oxide TFTs achieved to date that does not significantly heat the entire thickness of the substrate. Importantly, the conversion from sol–gel precursors to the semiconducting metal-oxide phase during photonic curing is on par with thermal annealing, which is a significant improvement over previous pulsed-light processing work. The use of efficient gate absorbers also allows for the reduction in the number of pulses and efficient sol–gel conversion.

1. INTRODUCTION

Metal oxide semiconductors such as indium gallium zinc oxide (IGZO) and indium zinc oxide (IZO) have received significant attention over the past few decades due to their desirable electrical and optical properties. Oxide semiconductors are a unique group of materials that demonstrate high field-effect mobility even in the amorphous phase. This is due to their unique electron orbital structure, in which the conduction band is made up of large, spherical s-orbitals.1–3 These materials are particularly interesting as a replacement for amorphous silicon in thin-film transistors (TFTs) for display applications because oxide semiconductors often have mobilities 10–100× higher than amorphous silicon.1,4–6 Transistor performance can be controlled by incorporating materials with a similar orbital configuration such as tin, zinc, and gallium. In particular, the metal cation composition of the semiconductor can be used to adjust the mobility, on/off current ratio, and turn-on voltage in the TFT.5,6

In addition to their superior electrical performance, there are well-developed wet-chemical synthesis techniques for amorphous oxide thin films, making these materials easy and cheap to produce. Sol–gel methods are the most commonly employed technique because of the simplicity of the synthesis

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and the ability to easily tune the film composition. The solution-based nature of sol–gels also allows for a wide range of deposition techniques such as spin coating, dip coating, ink-jet printing, and spray coating. Despite these benefits, one limiting factor in the synthesis and fabrication of sol–gel metal-oxide TFTs is that a relatively high-temperature postdeposition annealing step is required to convert the sol–gel into the semiconducting oxide. An intermediate metal hydroxide phase \((\text{M}−\text{OH})\) is often formed at low processing temperatures instead of the desired metal-oxide phase \((\text{M}−\text{O})\). Films with significant hydroxide content exhibit poor electrical performance and do not yield high-quality TFTs. Temperatures in excess of 300 °C are required to decompose metal hydroxide bonds and form a dense metal-oxide network.

The desire to create high-quality flexible transistors on low-temperature plastic substrates has driven an investigation into alternative thermal processing methods for oxide semiconductors. Some of the most popular are microwave annealing, photochemical activation, and pulsed-light processes based on flashlamps. The latter, commonly known as photonic curing, is particularly advantageous as compared to conventional thermal annealing because photonic curing offers the ability to rapidly process films at high temperatures, greatly reducing processing time. Photonic curing on both silicon and glass substrates has been reported for various oxide semiconductor compositions. These reports have studied the effects of the number of pulses, pulse energy, and fire rate on TFT performance and demonstrated the ability to cure and produce TFTs with photonic curing. However, these processes often result in substantial substrate heating.

With a properly designed device stack, photonic curing can produce transient heating of thin films to several hundred degrees on substrates with a low thermal budget (e.g., plastic). This can be accomplished because the very short heating intervals (typically 1–2 ms) result in nonequilibrium heating and thus create a thermal gradient across the material stack. This ensures that only the thin film and the top few microns of the substrate are heated above the working temperature of the substrate, while the majority of the substrate remains significantly cooler (Figure S1). This allows the thin film to be processed at temperatures above the typical glass transition point of the substrate. While photonic curing has been focused primarily on sintering metal patterns on low-temperature substrates, it has also been used for a wide range of applications such as photonic soldering on poly(ethylene terephthalate) (PET) and polyimide (PI) foils, processing CuInSe\(_2\) solar cells, nanostructured TiO\(_2\) films, recrystallization of amorphous silicon, and conductivity enhancement of graphene films. Recently, the feasibility of photonic curing metal-oxide dielectric and semiconductor sol–gel films on polymer substrates has been demonstrated as well.

The continued optimization and development of an efficient photonic curing process for solution-processed oxide semiconductor films could enable truly high-performance, high throughput transistors.

Here, we report the use of photonic curing using a xenon flashlamp to rapidly convert IZO sol–gels yielding TFTs with higher mobility than a conventional 500 °C anneal, excellent on/off ratios, and negligible hysteresis. By carefully designing the device structure and optimized photonic pulse conditions, we have overcome the limitations of previous photonic curing studies such as insufficient absorption of the incident curing pulse and excessive substrate heating. With these advancements, we have demonstrated that photonic curing can rapidly and efficiently convert oxide sol–gels to high-performance semiconductor thin films for TFTs. The effects of different gate metals, thermal conductivity of the substrate, and pulse shaping were investigated, and curing conditions were simulated using SimPulse, a photonic curing simulation software. We show that photonic curing heats the oxide thin film to ~590 °C while the bottom of the substrate remains at or near room temperature. We demonstrate efficient conversion of sol–gel precursors to semiconducting metal-oxide films with >80% metal-oxide \((\text{M}−\text{O})\) fraction using extremely short (≤30 ms) photonic curing processes. This conversion represents a significant improvement over previous pulsed-light processing for TFTs and is on par with our samples that were thermally annealed at 500 °C for 1 h. The TFTs made using these photonically cured films had a field-effect mobility of 21.8 cm\(^2\) V\(^{-1}\) s\(^{-1}\), negligible hysteresis, and an \(I_{on}/I_{off}\) ratio approaching 10\(^8\). This mobility exceeds the performance of devices that were thermally annealed for 1 h at 500 °C. Thus, we show that photonically cured TFTs using efficient gate absorbers outperform conventional thermally processed devices while enabling a drastic reduction in processing time, as well as reducing substrate heating. We also demonstrate how accurate simulations can be achieved by characterizing the absorbance of the material stack being processed.
2. EXPERIMENTAL SECTION

2.1. IZO Sol−Gel Synthesis. In this study, IZO sol−gel is chosen for the thin-film semiconducting layer because, compared to other semiconducting oxides, IZO tends to provide reasonably high mobility while still maintaining good turn-off characteristics. The molar ratio of In/Zn is 60:40, which was determined to be the optimal ratio in previous reports.\(^4,5\) Indium zinc oxide (IZO) sol−gels were synthesized using a process adapted from Street et al.\(^4\) To prepare a 0.1 M solution, indium nitrate hydrate In(NO\(_3\))\(_2\)·H\(_2\)O (CAS 207398-97-8) and zinc nitrate hexahydrate Zn(NO\(_3\))\(_2\)·6H\(_2\)O (CAS 10196-18-6) were dissolved in 15 mL of 2-methoxyethanol. The In/Zn ratio was fixed at 60:40 which required 271 mg of indium nitrate, and 178 mg of zinc nitrate. Once all precursors were added to the flask, the flask was placed in an oil bath at 80 °C and magnetically stirred for 1 h. The flask was then removed from the oil bath and allowed to cool. The solution was then transferred to a scintillation vial for storage.

2.2. Thin-Film Transistor Fabrication. Bottom-gate staggered TFTs (Figure 1) were fabricated on Corning Eagle XG display glass (25.4 mm × 25.4 mm × 0.7 mm, MTI Corporation). Prior to fabrication, the glass was cleaned by sonication in solvents. A 150 nm molybdenum gate electrode was deposited by DC magnetron sputtering and subsequently patterned using liftoff. The dielectric layer was 42 nm of HfO\(_2\) deposited using plasma-enhanced atomic layer deposition (PEALD) at 100 °C. Immediately prior to depositing in the IZO, the dielectric was cleaned in a piranha solution consisting

Figure 2. Absorbance (a) and reflectance (b) spectra for 150 nm molybdenum and 150 nm aluminum gate films. The dashed line represents the PulseForge 1300 emission intensity (au). The shaded region (400−700 nm) indicates the highest intensity region for the flashlamp. (c) Simulated temperature profiles using Al or Mo gate using the absorbance shown in panels (a) and (b). The black and red lines indicate the top surface temperature using Mo or Al, respectively, while the green line represents the temperature at the back of the substrate, which is the same in both cases. The same photonic curing pulse was used for both simulations. High absorption in the Mo gate results in a simulated peak temperature of ~590 °C, while the high reflectance of Al limits the simulated peak temperature to only ~60 °C. (d) Simulated temperature for Mo on glass and silicon substrates demonstrates that, for the same absorption and pulse conditions, the substrate thermal conductivity plays a large role in the thermal response. The solid lines (black, blue) show the top surface temperature, while the dashed lines represent the temperature at the back of the substrate.
of one part hydrogen peroxide to four parts sulfuric acid. The IZO sol–gel was then spin-coated onto the dielectric using a static dispense and then ramping to 3000 rpm and spinning for 30 s. The sample was transferred to a hotplate and baked at 150 °C for 10 min to remove excess solvent. This spin-coating process was repeated to yield a two-layer film (12–15 nm). After spin coating, the sol–gel film was thermally processed. This was accomplished either by conventional thermal annealing or photonic curing. Conventional thermal anneals were carried out in a custom-designed three-zone tube furnace with 5 in. diameter and 24 in. heating length (Sentry Tech Corp.). The sample temperature was ramped from room temperature to 200, 300, 400, or 500 °C at a rate of approximately 30 °C min⁻¹, and the dwell time was 1 h. The photonic curing process is described below. After thermal processing, the semiconductor island was patterned using photoresist and then etched in the ion mill. Source/drain electrodes were then formed by liftoff of 100 nm of aluminum deposited by e-beam evaporation. This formed a channel with a 100 nm of aluminum deposited by e-beam evaporation. This formed a channel with 500/60 μm. Finally, a gate contact hole was patterned and opened by etching the dielectric in the ion mill.

2.3. Photonic Curing System. Prior to curing, absorption data for the material stack was collected on a UV–vis spectrometer. The absorption properties and the material stack were modeled in SimPulse photonic curing software from NovaCentrix. This model estimates the temperature profile through the provided material stack during photonic curing. Samples were photoinitiated cured on a NovaCentrix PulseForge 1300 equipped with four lamp drivers, a 5 kW power supply, and a 24 mm diameter xenon flashlamp (beam size 150 mm × 75 mm). Figure 1a shows an illustration of the photonic curing process with a uniform beam illuminating the sample. Samples are placed on a vacuum graphite chuck (NovaCentrix EX-1), a PulseForge accessory, which is set to 7 mm below the lamp housing. The graphite chuck greatly increases the thermal conductivity at the back side of the sample, allowing for more rapid cooling. A National Institute of Standards and Technology (NIST)-traceable bolometer was used to measure the radiant exposure of each pulse used to cure the samples and calibrate the simulated temperature. All photoinitiated cured samples were processed in an ambient environment.

2.4. Material Characterization. The absorbance and reflectance of several potential gate metals were measured, including 150 nm of sputtered Mo and 150 nm of e-beam evaporated Al on 700 μm Eagle XG display glass (Figure 2). A Cary 7000 UV–vis–near-infrared (NIR) spectrophotometer was used to determine the transmittance, reflectance, and absorbance of the metal films. The reflectance measurement was taken using an integrating sphere. Using the measured values for transmittance and reflectance, the value of absorbance was calculated at each wavelength (A = 1 − T − R). This absorbance value, taken from the most intense region of the lamp spectrum, is the input value to the SimPulse model and is a crucial parameter to estimate accurate temperature profiles.

X-ray photoelectron spectroscopy (XPS) was used to study the chemical composition of processed IZO films. XPS measurements were taken using a Ulvac PHI VersaProbe III system. Depending on the sampling areas, two spot sizes were used: 100 μm with a power of 25 W under 15 kV or 20 μm of 4.5 W and 15 kV. The survey scans were taken using 280 eV pass energy and 1 eV per step. The high-resolution scans were obtained with the pass energy of 55 eV and 0.1 eV per step. Prior to all measurements, the sample was sputtered with 3 kV monatomic argon to remove surface contamination. Multipak software was used to perform peak-fitting and compositional analysis. All peaks were referenced to the C 1s peak at 284.5 eV, which was still present at a level of a few percentage points even after the sputter cleaning.

The surface morphology was studied using atomic force microscopy (AFM). AFM measurements were taken using a Digital Instruments NanoScope III scanning probe microscope. Surface morphology was imaged with the instrument in tapping mode. Surface measurements of 1 μm × 1 μm sections were imaged and analyzed using Gwyddion software to determine the surface roughness.

2.5. Electrical Characterization. TFT device characterization was accomplished using a Keysight B1500 semiconductor device parameter analyzer. Transfer curves and output curves were measured using a forward sweep followed by a reverse sweep of the gate or drain voltage, respectively. Field-effect mobility in the linear (low-field) region was calculated using the transconductance method. The device is operated in the linear region (V_D = 0.1 V), and the linear drain current is differentiated with respect to the gate voltage to obtain the transconductance, g_{m}. The oxide capacitance, C_{ox}, was measured using Mo/HfO_2/Al capacitors in which the HfO_2 was deposited using conditions identical to those used in TFT fabrication. The capacitance was measured with a gate voltage of 5 V and a frequency of 100 Hz, yielding a capacitance of C_{ox} = 337 nF cm⁻². The linear mobility is then extracted using the linear (where V_D is small) transconductance. Equation 1 shows the partial derivative of the square-law model yielding g_{m} which can be used to find the field-effect mobility.

$$\frac{\partial I_D}{\partial V_{GS}} = \frac{1}{W L C_{ox}} \mu_{FE} \left( V_{GS} - V_T \right) V_D$$

The subthreshold slope was calculated by reciprocating the slope of the I_D–V_G transfer curve in the turn-on (V_{ON} < V_G < V_T) region. This was plotted against log(I_D) and the minimum subthreshold slope was taken from each curve. The TFT turn-on point (V_{ON}) is identified as the point on the transfer curve where the current abruptly increases, indicating charge accumulation in the channel. Hysteresis is defined as the difference in V_{ON} between the forward and reverse sweep of the transfer curve measured in saturation.

3. RESULTS AND DISCUSSION

3.1. Effect of Gate Metal Absorption and Substrate Thermal Conductivity on Sample Heating. The material stack that was subjected to photonic curing in this work (Figure 1c) includes the glass substrate, Mo gate, HfO_2 gate dielectric, and IZO sol–gel (glass/Mo/HfO_2/IZO). In oxide semiconductors devices, there is essentially zero light absorption in the dielectric and semiconductor layers because they are typically thin (<40 nm) layers of wide band gap materials and do not absorb in the region where the lamp has highest intensity (Figure 2a,b). The glass substrate alone absorbs only ~8% of the incident optical energy (Table 1). Thus, the gate metal should be the primary absorbing layer and essentially acts as a heating element for the IZO sol–gel film. After initial experiments using aluminum gates, we found that the absorbance of the aluminum was only 5% due to high
between pulses and prevent cumulative substrate heating.

Table 1. Film Parameters for Common Gate Metals and a Glass Substrate

| Candidate     | CTE (ppm °C⁻¹) | Absorbance % |
|---------------|----------------|--------------|
| Molybdenum    | 5.1            | 58           |
| Aluminum      | 24             | 5            |
| Eagle XG glass| 3.2            | 8            |

reflectance (Figure 2a,b). Molybdenum, on the other hand, is an excellent choice for the gate material because it has a high melting point, a low coefficient of thermal expansion (CTE), and a high absorbance of ~58%. Once the absorbance for the material stack is characterized that value can be utilized to simulate the temperature throughout the material stack using SimPulse software. The effect of gate metal absorption can clearly be seen in Figure 2c. Because the Al gate reflects ~95% of the light, the peak-simulated temperature on Al is only ~60 °C, while the Mo gate reaches ~590 °C for an identical photonic curing pulse. This underscores the importance of designing devices with an efficient absorber layer, and it also shows why the proper characterization of the absorption is critical to obtaining accurate temperature simulations. The Mo gate layer was robust during photonic curing until the simulated temperature in the film exceeded 600 °C, which occurred at 6.3 J cm⁻² of radiant exposure. Cracks and ablation were observed in Mo gates with more intense pulses (Figure S2). Aluminum, on the other hand, remained intact, showing no signs of cracking or ablation even when radiant exposures as high as 18 J cm⁻² were used. Even with shaped curing pulses with energies as high as 16 J cm⁻², the temperature in the aluminum did not exceed 160 °C (Figure S3). Aluminum has a high CTE of 24, so if there was substantial heating in the aluminum film, cracking and film ablation would be expected at energies much lower than that of molybdenum. A summary of absorbance and CTE for common materials is shown in Table 1.

The thermal conductivity of the substrate plays a substantial role in the thermal profile achieved on the thin film during photonic curing. Substrates with high thermal conductivity behave as an efficient heat sink, making it difficult to achieve a high temperature in the thin film. This is demonstrated in Figure 2d using SimPulse, a proprietary photonic curing thermal modeling package that is resident on all PulseForge tools. Here, the thermal profile generated from the same pulse used for curing is shown with both a glass substrate and silicon substrate (thermal conductivity, κ = 1.2 and 148 W m⁻¹ K⁻¹, respectively), upon which the same thickness of Mo is coated as the metal gate absorber. On the glass, where the thermal conductivity is low, the surface temperature on the Mo reaches 590 °C and then the film rapidly cools. This is because the transient curing conditions are met for this material stack and pulse design. The metal gate absorber heats rapidly during the pulse, but the bottom of the substrate remains unheated. After the pulse ends, the heat is conducted away from the absorber into the substrate, which allows for this rapid cooling (Figure S1). On the other hand, with silicon as the substrate, its high thermal conductivity allows the incident energy to be conducted throughout the substrate. We can directly observe this from the simulation results in Figure 2d, which show that on the silicon the surface temperature of the Mo only reaches a maximum temperature around 80 °C and does not experience rapid cooling like the glass substrate. This is due to the fact that most of the energy is not maintained in the top few microns of the film/substrate, and instead, it is thermally conducted into the entirety of the substrate. This can be seen in Figure 2d where the surface and back of the silicon substrate reach thermal equilibrium after only 3 ms, whereas the glass substrate reached thermal equilibrium after roughly ~800 ms (Figure 3c).

3.2. Photonic Curing Pulse Design. The PulseForge photonic curing system allows for extremely precise control over the pulse parameters such as pulse voltage, pulse length, duty cycle, etc., enabling a high degree of control over the thermal profile along with excellent uniformity over a large-area substrate. Because of the unique ability to deliver very high-energy density pulses over a short period of time (ms), the PulseForge can heat a thin film to a high temperature (>500 °C) without heating the majority of the substrate beyond its maximum working temperature. Design rules for achieving these transient curing conditions have been previously described in detail by Schroder et al.32 Figure 3 shows the simulated temperature profile through the material stack for different photonic curing pulse designs using SimPulse. The photonic curing processing window was limited

Figure 3. Simulated temperature profile of the glass/Mo stack using a variety of photonic curing pulse designs, showing the temperature profile at the surface of Mo (blue), 10 μm into the substrate (orange), and at the back surface of the glass substrate (green). (a) Example of a simple pulse cure. (b) Pulse shaping using 11 micropulses to create a shaped pulse. The micropulses increase the duration of the cure by keeping the film temperature elevated. The lamp on/off times that were used to create this shaped pulse are listed in Table 2c. A multiple-shaped pulse cure thermal profile consisting of three-shaped pulses, each identical to (b), that are separated by enough time for the substrate to cool to room temperature between pulses and prevent cumulative substrate heating.

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by the conditions that caused gate cracking and ablation (see the Supporting Information). This resulted in keeping the maximum simulated surface temperature just below 600 °C. A single pulse cure (Figure 3a) very rapidly heats the thin film to a peak temperature of ~590 °C, but the estimated curing time near that temperature is only a few microseconds. For photonically cured sol–gels, we have found that it is advantageous to increase the curing time using multiple-shaped pulses. The shaped pulse was designed around maintaining the maximum simulated surface temperature, which was achieved by utilizing micropulses. The micropulses allow for a custom tailoring of the thermal profile by controlling the on/off time of the lamp. Once a pulse is designed, the temperature can be scaled up by increasing the lamp voltage. The micropulses in this work were designed such that the sample would heat and maintain a given temperature, which is analogous to a “ramp-and-hold” profile during a conventional thermal annealing. While a single pulse can result in similar temperatures, the time spent at the maximum temperature is much shorter than a shaped pulse. The shaped pulse allows for the time at elevated temperature to be increased and therefore requires fewer overall pulses than if a single pulse was used to obtain sol–gel conversion.

Figure 3b shows the profile for a cure consisting of 11 micropulses to increase the effective curing time at an elevated temperature. This shaped pulse had a radiant energy of 6.2 J cm⁻² per pulse (which includes 11 micropulses). The on/off timings of individual micropulses (Table 2) were customized such that the maximum temperature in the film did not exceed 590 °C. The tool and simulation parameters can be viewed in Figure S4. The shaped pulse increased the estimated curing time over a simple pulse from a few microseconds to approximately 2 ms. If longer cures are desired, the shaped pulse can be repeated multiple times. A three-shaped pulse cure is shown in Figure 3c, where each shaped pulse is the micropulse design shown in Figure 3b. Multiple pulses in this work were accomplished using a fire rate of 0.3 Hz, which is slow enough to allow the system to reach thermal equilibrium and return to room temperature before the next pulse occurs. This ensures that a gradual, continuous heating of the substrate can be avoided. The temperature of the back surface of the substrate is briefly increased to 30 °C, but returns to room temperature before the next pulse, as shown in Figure 3c.

### 3.3. Limitations of Previous Photonically Cured Sol–Gels

Despite the promising capabilities of photonic curing, there are two key factors that have limited the ability to efficiently cure oxide semiconductor TFTs. First, many studies processed oxide semiconductor thin films on silicon substrates. However, when substrates with high thermal conductivity are used, sustaining an elevated temperature in the thin film becomes challenging because the high thermal conductivity substrates act as a heat sink. In fact, the power required to reach a given temperature is proportional to the square root of the substrate thermal conductivity. Therefore, a silicon substrate requires roughly 11 times as much power as a glass substrate to reach the same temperature because its thermal conductivity is roughly 120 times higher. To overcome this, higher radiant exposure and rapid-fire rates are required (Table S1), thus requiring a larger electrical input to generate a pulse capable of delivering such a high radiant exposure. When high-energy pulses with rapid flash rates are used, it is likely that the entire substrate and film stack are at the same elevated temperature because the transient heating criteria are not met. In fact, some reports using pulsed light on silicon have confirmed this by measuring elevated temperatures in the Si substrate with a thermocouple. Thus, photonic curing on high thermal conductivity substrates such as silicon requires careful consideration of pulse design and high radiant exposure to achieve sufficiently high curing temperatures for oxides.

The second factor that has limited the progress of previous photonic curing studies is using a material stack that does not efficiently absorb the lamp spectrum and heat the thin film. Thin oxide semiconductor films have negligible absorption by themselves and require an absorption layer to heat the film. In a bottom-gate TFT structure, it is the gate that acts as this absorption layer. Some previous studies have used an aluminum gate as the absorbing layer, but, as previously discussed, aluminum is such an excellent reflector that it only absorbs ~5% of the incident light (Figure 2a,b). In this case, the absorbance in the metal gate is similar to the absorbance in the glass substrate, so it is difficult to distinguish whether the heating is caused by absorption in the gate or in the substrate. If the material stack has insufficient absorption, minimal heating will occur during each pulse. This can be overcome by rapidly delivering multiple pulses of light, which results in a gradual heating of the entire film stack and substrate rather than heating only the top few microns of the structure, or using extremely intense pulses (Table S2). This can also be observed in previous photonic curing studies where In₃O₅ was processed on glass and the back of the substrate reached simulated temperatures in excess of 300 °C over a period of 10–24 s. Interestingly, temperatures as high as 1000 °C were reported in those studies without experiencing any film ablation or delamination from the CTE mismatch. Despite the rapid nature of photonic curing, we have observed that the thermal effects of high-temperature processes (such as cracking and ablation) still occur with molybdenum gates (Figure S2). Molybdenum also offers a relatively good CTE match to the glass substrate, which minimizes thermal stress that occurs during curing. Using efficient gate absorbers, the heating is localized in the metal gate, and the heat is efficiently transferred into the sol–gel film. This also allows the number of pulses to be reduced and the fire rate to be greatly reduced, mitigating some of the thermal stress and gradual substrate heating.

### 3.4. Chemical Composition of IZO Films

The chemical composition and key bonding information for IZO films were characterized using X-ray photoelectron spectroscopy (XPS).
bonding. The metal oxygen (M−OH) peak has been widely used to assess the relative proportions of metal oxygen bonding vs metal hydroxide bonding.11,43,44 In this work, the M−OH to M−O conversion of IZO sol−gels during photonic curing was compared to the conversion that occurs during traditional thermal processing. The photoelectron peak of interest is the O 1s peak because it reveals the relative proportion of metal oxygen bonding vs metal hydroxide bonding. The metal oxygen (M−O) binding energy is located at 529.9 eV, and a second peak at slightly higher binding energy is attributed to metal hydroxide (M−OH) bonding. To determine the proportion of metal oxygen and metal hydroxide bonds, a series of Gaussian and Lorentzian peaks were fit, and the relative areas were extracted. The spectra were then normalized to compare different samples, as shown in Figure 4.

As expected, thermally annealing the IZO at 200 °C for 1 h resulted in only a slight reduction in the metal hydroxide peak, decreasing the M−OH fraction from 47 to 38%. This level of processing correlates with a device with large hysteresis, poor mobility, and a large positive turn-on voltage (Figure 5a). In contrast, samples annealed at 300, 400, and 500 °C demonstrated a large reduction in metal hydroxide bonding, yielding M−OH fractions as low as 16%. The samples at 400 and 500 °C had the same ratio of metal oxygen and metal hydroxide bonds, indicating that no further conversion was taking place at temperatures above 400 °C for 1 h anneals. The relative percentages of metal oxide and metal hydroxide for various annealing conditions are summarized in Table 3. These data underscore the importance of achieving a high annealing temperature in the IZO thin film layer for proper conversion of the semiconductor.

### Table 3. Relative Fraction of Metal-Oxide (M−O) vs Metal Hydroxide (M−OH) Bonding in IZO Sol−Gel Films for Various Annealing Conditions

| Annealing Condition | M−O % | M−OH % |
|---------------------|-------|--------|
| unannealed          | 53    | 47     |
| 200 °C              | 62    | 38     |
| 300 °C              | 79    | 21     |
| 400 °C              | 84    | 16     |
| 500 °C              | 84    | 16     |
| 1 pulse             | 65    | 35     |
| 5 pulses            | 75    | 25     |
| 10 pulses           | 74    | 24     |
| 15 pulses           | 82    | 18     |

The XPS spectra for photonically cured samples also showed a substantial degree of conversion from M−OH to M−O bonding. A single-shaped pulse cure as shown in Figure 3b (the micropulse design in Table 2) and a lamp voltage of 520 V reduced the M−OH content in the IZO film from 47 to 35%, which is slightly better than a 1 h 200 °C thermal anneal. Despite being at elevated temperatures for only 2 ms, this photonic curing process begins to convert the sol−gel into the metal oxide. Furthermore, the amount of metal hydroxide in the film decreased as the number of pulses increased, indicating some time dependence in converting these films. The hydroxide peak reduced from 47% in the unannealed (as-deposited) sample to only 18% in the sample photonicly cured with 15 pulses. The entire duration of the processing was 50 s, with the estimated curing time being approximately 30 ms. This conversion is essentially the same as what was achieved at 500 °C for 1 h. Previous TFT studies using either silicon or aluminum gates reported M−O percentages no greater than ~60%,15,18,23 (comparable to the 200 °C sample in this work), indicating that the semiconductor film was only partially cured. In contrast, by designing an effective absorber layer, we can achieve 65% M−O conversion with one pulse and reach 82% M−O with 15 pulses, which is approximately 20−30% higher conversion than previously reported in literature.15,23

### 3.5. IZO Film Morphology.

The morphology and roughness of photonicaily cured films after processing was investigated using atomic force microscopy (AFM). Scans taken over 1 μm² of the films show smooth, continuous films with a surface roughness of ~1 nmRMS for all of the photonicilly cured samples (Figure 6). A negligible change in surface roughness was observed in the samples that were photonicilly cured with 1, 5, 10, or 15 pulses. Despite rapid heating during the photonic curing process, the adhesion of the sol−gel to the dielectric is sufficient to prevent delamination or
wrinkling, and no voids were created from gasification. It is a strong possibility that reaching an elevated temperature up to 590 °C may have caused some phase separation or partial crystallization. Previous studies have shown that ternary compositions such as IZO and IGZO are less likely to crystalize than phase-pure compositions such as ZnO or In₂O₃ when annealed up to 550 °C. However, nanocrystallites have been observed at similar temperatures and may explain why given the same M–O percentage for the 400 and 500 °C devices, the 500 °C device performance is significantly improved.47

3.6. Thermally Annealed TFTs. The electrical performance and transport in the IZO semiconductor layer were evaluated using bottom-gate, top-contact TFTs. The I–V characteristics and performance metrics of photonically cured TFTs were compared against control devices that underwent traditional thermal annealing in a tube furnace. For all devices, the field-effect mobility was extracted from the transfer curve in the linear (low-field) region of operation. Control TFTs were thermally annealed for 1 h in air at 300 °C (a, d), 400 °C (b, e), or 500 °C (c, f). The forward trace is represented by a solid line and the reverse trace by a dashed line. In the transfer characteristics (a–c), the dashed gray line represents gate leakage current (I₇₆).

Figure 5. Surface morphology obtained using AFM for samples photonically cured with (a) 1 pulse, (b) 5 pulses, (c) 10 pulses, and (d) 15 pulses. Each image inset specifies the surface roughness measured over 1 μm² of the film.

Figure 6. Transfer characteristics (a–c) and output characteristics (d–f) for thermally annealed IZO TFTs. Devices were annealed for 1 h in air at 300 °C (a, d), 400 °C (b, e), or 500 °C (c, f). The forward trace is represented by a solid line and the reverse trace by a dashed line. In the transfer characteristics (a–c), the dashed gray line represents gate leakage current (I₇₆).
3.7. Photonically Cured TFTs. The transfer curves and output curves of photonically cured IZO TFTs are shown in Figure 7. As expected based on the XPS data, increasing the number of pulses improved the device performance. The device processed with only one pulse showed poor TFT action (Figure S5), having a field-effect mobility value ranging from 1 to 1.5 cm² V⁻¹ s⁻¹. As the number of pulses increased, the turn-on voltage shifted more negative. This is consistent with the film becoming more conductive and thus requiring a larger negative voltage to deplete the channel and turn the device off. Only a small amount of hysteresis is observed in the five-pulse device, and after 10 pulses, the hysteresis becomes negligible. The mobility also slightly increases with more pulses, going from 15.7 cm² V⁻¹ s⁻¹ with 5 pulses to 21.8 cm² V⁻¹ s⁻¹ with 15 pulses. The quoted mobility values are the average of 10 random devices for each group. The respective standard deviations of mobility for the 5, 10, and 15 pulses were 0.47, 0.5, and 0.79 cm² V⁻¹ s⁻¹. These mobility values are more than 8 times greater than previous results using photonic curing on silicon substrates, which had field-effect mobilities around 2.5 cm² V⁻¹ s⁻¹.¹⁵,¹⁶ These photonically cured devices exhibited slightly higher gate leakage (as seen in Figure 7) than thermally annealed devices. At 520 V, even one pulse produced a peak temperature near the cracking threshold for molybdenum gates. Small cracks in the gate metal were observed in a very small number of devices on the perimeter of the sample because we were operating near the cracking threshold, which may have led to defects in the gate dielectric and increased gate leakage current. Near the edges where there is less thermal mass, the cooling is less efficient as the heat near the edges has

Table 4. Summary of Metal-Oxide Conversion and TFT Performance Metrics for Thermally Annealed and Photonically Cured IZO Devices

| Annealing Condition | M–O (%) | M–OH (%) | Mobility (cm² V⁻¹ s⁻¹) | STD (cm² V⁻¹ s⁻¹) | V_ON (V) | Hysteresis (V) | SS (mV dec⁻¹) |
|---------------------|---------|----------|-------------------------|-------------------|----------|---------------|--------------|
| unannealed          | 53      | 47       | N/A                     | N/A               | N/A      | N/A           | N/A          |
| 200 °C              | 62      | 38       | 0.005                   | N/A               | 8.3      | 8             | N/A          |
| 300 °C              | 79      | 21       | 1.3                     | 0.08              | 2.5      | 3.5           | 125          |
| 400 °C              | 84      | 16       | 7.3                     | 0.1               | 2        | 1.5           | 125          |
| 500 °C              | 84      | 16       | 17.1                    | 0.15              | 5.2      | ~0            | 150          |
| 1 pulse             | 65      | 35       | 1-1.5                   | N/A               | 1.5      | 2.6           | N/A          |
| 5 pulses            | 75      | 25       | 15                      | 0.47              | 3        | 0.2           | 190          |
| 10 pulses           | 74      | 24       | 17.3                    | 0.5               | 8        | ~0            | 205          |
| 15 pulses           | 82      | 18       | 21.8                    | 0.78              | 7.3      | ~0            | 280          |

The value of V_ON for the 200 °C and one-pulse device are less certain due to the large amount of hysteresis present in the devices. The values of subthreshold slope for 200 °C and one-pulse devices are omitted because lower drain current and higher gate leakage can cause errors in the calculation of the subthreshold slope.

Figure 7. Transfer characteristics (a–c) and output characteristics (d–f) for photonically cured IZO TFTs. Devices were cured with 5 pulses (a, d), 10 pulses (b, e), or 15 pulses (c, f). The forward trace is represented by a solid line and the reverse trace by a dashed line. In the transfer characteristics (a–c), the dashed gray line represents gate leakage current (I_G).

3.7. Photonically Cured TFTs. The transfer curves and output curves of photonically cured TFTs are shown in Figure 7. As expected based on the XPS data, increasing the number of pulses improved the device performance. The device processed with only one pulse showed poor TFT action (Figure S5), having a field-effect mobility value ranging from 1 to 1.5 cm² V⁻¹ s⁻¹. As the number of pulses increased, the turn-on voltage shifted more negative. This is consistent with the film becoming more conductive and thus requiring a larger negative voltage to deplete the channel and turn the device off. Only a small amount of hysteresis is observed in the five-pulse device, and after 10 pulses, the hysteresis becomes negligible. The mobility also slightly increases with more pulses, going from 15.7 cm² V⁻¹ s⁻¹ with 5 pulses to 21.8 cm² V⁻¹ s⁻¹ with 15 pulses. The quoted mobility values are the average of 10 random devices for each group. The respective standard deviations of mobility for the 5, 10, and 15 pulses were 0.47, 0.5, and 0.79 cm² V⁻¹ s⁻¹. These mobility values are more than 8 times greater than previous results using photonic curing on silicon substrates, which had field-effect mobilities around 2.5 cm² V⁻¹ s⁻¹.¹⁵,¹⁶ These photonically cured devices exhibited slightly higher gate leakage (as seen in Figure 7) than thermally annealed devices. At 520 V, even one pulse produced a peak temperature near the cracking threshold for molybdenum gates. Small cracks in the gate metal were observed in a very small number of devices on the perimeter of the sample because we were operating near the cracking threshold, which may have led to defects in the gate dielectric and increased gate leakage current. Near the edges where there is less thermal mass, the cooling is less efficient as the heat near the edges has
nowhere to go, resulting in more cracking. In the center of the substrate, the entirety of the substrate acts as a heat sink allowing for efficient cooling. These perimeter devices were not representative of the majority of the samples and were not included in the data analysis. While devices in the center did not demonstrate any visible curing for the given processing conditions, a higher gate leakage was observed when compared to thermally annealed devices. Despite this slight increase in gate leakage that is observed at larger biases, the gate current was still several orders of magnitude lower than the drain current, and the devices exhibited clean turn-off characteristics. Overall, these results demonstrate that photonic cured TFTs that spend only $\sim 30$ ms at an elevated temperature ($50$ s total processing time, accounting for the time the lamp is off) outperform devices that were thermally annealed at $500^\circ$C for $1$ h ($\mu = 21.8$ and $17.1$ cm$^2$ V$^{-1}$ s$^{-1}$, respectively). The TFT characteristics of thermally annealed and photonic cured devices are summarized in Table 4.

4. CONCLUSIONS

In summary, we have demonstrated that photonic curing can efficiently and rapidly convert an IZO sol–gel into a high-quality metal-oxide semiconductor thin film. In this study, we examined the chemical composition, surface morphology, and electrical transport in photonically cured IZO films. The importance of characterizing and understanding material parameters such as absorption and thermal conductivity were addressed and their impacts on the thermal response were presented. We designed photonic curing pulses to transiently heat the thin transparent IZO sol–gel film to $\sim 590^\circ$C, while the bottom of the glass substrate remained below $\sim 30^\circ$C. This photonic curing process produced smooth, uniform semiconductor films with $\sim 1$ nmRMS surface roughness. In as few as five pulses, photonically cured devices exhibited a M–O conversion fraction of 75% and a mobility of 15.7 cm$^2$ V$^{-1}$ s$^{-1}$. We observed that increasing the number of pulses from the lamp could further improve film conversion and TFT performance. By increasing the estimated curing time from $2$ ms (1 pulse) to $30$ ms (15 pulses), we achieved 82% M–O conversion and increased the TFT mobility to 21.8 cm$^2$ V$^{-1}$ s$^{-1}$. These results demonstrate that photonically cured oxide TFTs can achieve similar chemical conversion and significantly improved mobility compared to identical devices annealed at $500^\circ$C for $1$ h. Along with superior electrical performance, the photonic curing process also drastically reduced the processing time to convert the sol–gel film from over $1$ h to $\sim 50$ s.

**ASSOCIATED CONTENT**

 Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c01421.

Mechanism of photonic curing; effect of gate metal on curing; thermal profiles for a less absorbent metal; summary of selected previous photonic curing work on silicon and glass; simulation of temperature profiles using SimPulse; and single pulse and low-temperature thermal processing results (PDF)

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**Notes**

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