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TITLE:**The Effect of Anodization Parameters on the Aluminum Oxide Dielectric Layer of Thin-Film Transistors****AUTHORS AND AFFILIATIONS:**

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SUMMARY:

Anodization parameters for growth of the aluminum-oxide dielectric layer of zinc-oxide thin-film transistors (TFTs) are varied to determine the effects on the electrical parameter responses. Analysis of variance (ANOVA) is applied to a Plackett-Burman design of experiments (DOE) to determine the manufacturing conditions that result in optimized device performance.

ABSTRACT:

Aluminum-oxide (Al_2O_3) is a low cost, easily processable and high dielectric constant insulating material that is particularly appropriate for use as the dielectric layer of thin-film transistors (TFTs). Growth of aluminum-oxide layers from anodization of metallic aluminum films is greatly advantageous when compared to sophisticated processes such as atomic layer deposition (ALD) or deposition methods that demand relatively high temperatures (above 300 °C) such as aqueous combustion or spray-pyrolysis. However, the electrical properties of the transistors are highly dependent on the presence of defects and localized states at the semiconductor/dielectric interface, which are strongly affected by the manufacturing parameters of the anodized dielectric layer. To determine how several fabrication parameters influence the device performance without performing all possible combination of factors, we used a reduced factorial analysis

based on a Plackett-Burman design of experiments (DOE). The choice of this DOE permits the use of only 12 experimental runs of combinations of factors (instead of all 256 possibilities) to obtain the optimized device performance. The ranking of the factors by the effect on device responses such as the TFT mobility is possible by applying analysis of variance (ANOVA) to the obtained results.

INTRODUCTION:

Flexible, printed and large area electronics represent an emerging market that is expected to attract billions of dollars in investments in upcoming years. To achieve the hardware requirements for the new generation of smartphones, flat panel displays and internet-of-things (IoT) devices, there is a huge demand for materials that are lightweight, flexible and with optical transmittance in the visible spectrum without sacrificing speed and high performance. A key point is to find alternatives to amorphous silicon (a-Si) as the active material of the thin-film transistors (TFTs) used in the drive circuits of most of the current active-matrix displays (AMDs). a-Si has low compatibility to flexible and transparent substrates, presents limitations to large-area processing, and has a carrier mobility of about $1 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$, which cannot meet the needs of resolution and refresh rate for next generation displays. Semiconducting metal oxides (SMOs) such as zinc oxide (ZnO)¹⁻³, indium zinc oxide (IZO)^{4,5} and indium gallium zinc oxide (IGZO)^{6,7} are good candidates to replace a-Si as the active layer of TFTs because they are highly transparent in the visible spectrum, are compatible to flexible substrates and large area deposition and can achieve mobilities as high as $80 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$. Moreover, SMOs can be processed in a variety of methods: RF sputtering⁶, pulsed laser deposition (PLD)⁸, chemical vapor deposition (CVD)⁹, atomic layer deposition (ALD)¹⁰, spin-coating¹¹, ink-jet printing¹² and spray-pyrolysis¹³.

However, few challenges such as the control of intrinsic defects, air/UV stimulated instabilities and formation of semiconductor/dielectric interface localized states still need to be overcome to enable the large-scale manufacturing of circuits comprising SMO-based TFTs. Among the desired characteristics of high performance TFTs, one can mention the low power consumption, low operation voltage, low gate leakage current, threshold voltage stability and wideband frequency operation, which are extremely dependent on the gate dielectrics (and the semiconductor/insulator interface as well). In this sense, high- κ dielectric materials¹⁴⁻¹⁶ are particularly interesting since they provide large values of capacitance per unit area and low leakage currents using relatively thin films. Aluminum oxide (Al_2O_3) is a promising material for the TFT dielectric layer since it presents a high dielectric constant (from 8 up to 12), high dielectric strength, high electrical resistivity, high thermal stability and can be processed as extremely thin and uniform films by several different deposition/growth techniques^{15,17-21}. Additionally, aluminum is the third most abundant element in the Earth's crust, what means that it is easily available and relatively cheap compared to other elements used to produce high- κ dielectrics.

Although deposition/growth of Al_2O_3 thin (below 100 nm) films can be successfully attained by techniques such as RF magnetron sputtering, chemical vapor deposition (CVD), atomic layer deposition (ALD), the growth by anodization of a thin metallic Al layer^{17,18,21-26} is particularly interesting for flexible electronics owing to its simplicity, low cost, low temperature, and film thickness control in nanometric scale. Besides, anodization has a great potential for roll-to-roll

(R2R) processing, which can be easily adapted from processing techniques already being used at industrial level, permitting quick manufacturing upscaling.

Al₂O₃ growth by anodization of metallic Al can be described by the following equations



where the oxygen is provided by the dissolved oxygen in the electrolyte solution or by the adsorbed molecules at the film surface, whereas the water molecules are promptly available from the electrolyte solution. The anodized film roughness (which affects the TFT mobility due to carrier scattering at the semiconductor/dielectric interface) and the density of localized states at the semiconductor/dielectric interface (which affects the TFT threshold voltage and electrical hysteresis) are strongly dependent on anodization process parameters, to name a few: the water content, the temperature and the pH of the electrolyte^{24,27}. Other factors related to the Al layer deposition (like evaporation rate and metal thickness) or to post-anodization processes (like annealing) can also influence the electrical performance of fabricated TFTs. The effect of these multiple factors on response parameters can be studied by varying each factor individually while keeping all other factors constant, which is an extremely time-consuming and inefficient task. Design of experiments (DOE), on the other hand, is a statistical method based on the simultaneous variation of multiple parameters, which permits the identification of the most significant factors on a system/device performance response by using a relatively reduced number of experiments²⁸.

Recently, we have used multivariate analysis based on a Plackett-Burman²⁹ DOE to analyze the effects of Al₂O₃ anodization parameters on the performance of sputtered ZnO TFTs¹⁸. The results were used to find the most significant factors for several different response parameters and applied to the optimization of the device performance changing only parameters related to the anodization process of the dielectric layer.

The current work presents the whole protocol for manufacturing TFTs using anodized Al₂O₃ films as gate dielectrics, as well a detailed description for the study of the influence of the multiple anodization parameters on the device electrical performance by using a Plackett-Burman DOE. The significance of the effects on TFT response parameters such as the carrier mobility is determined by performing analysis of variance (ANOVA) to the results obtained from the experiments.

PROTOCOL:

The protocol described in the present work is separated into: i) preparation of the electrolytic solution for anodization; ii) substrate cleaning and preparation; iii) anodization process; iv) deposition of the TFT active layer and drain/source electrodes; v) TFT electrical characterization and analysis and vi) application of ANOVA to determine the significance of the manufacturing

factors in the TFT mobility.

1. Preparation of the electrolytic solution for anodization

1.1. Perform all the procedures of the protocol inside a cleanroom or a laminar flow cabinet, to avoid dust or contaminants during the sample preparation.

1.2. Prepare two solutions of tartaric acid (0.1 M) in different water/ethylene glycol volume ratios (16% and 30%), which will be used as the anodization electrolytic solution. Use the water content in the electrolytic solution as fabrication parameter of the anodized layer.

1.3. In a 150 mL beaker, dissolve 1.5 g of tartaric acid into 16 mL of deionized water and 84 mL of ethylene glycol to obtain a 16% water electrolyte stock solution. For a 30% water electrolyte stock solution, use 1.5 g of tartaric acid, 30 mL of deionized water and 70 mL of ethylene glycol. Stir both solutions using a magnetic bar for 30 min.

1.4. Separate about 10-20 mL of ammonium hydroxide (NH_4OH) solution (as purchased, 28 – 30% NH_3 in volume) in a 20 mL beaker to make the rough adjustment of the pH of the electrolytic solution.

1.5. Prepare 80 mL of a diluted solution (about 2% in volume) from the original NH_4OH solution to make the fine control of the pH of the electrolytic solution.

1.6. Separate the electrolyte solution into a 150 mL beaker to adjust the pH of the solution.

1.7. Measure the pH of the electrolytic solution using a bench pH meter. Start pipetting the more concentrated NH_4OH until the pH is close to the desired pH (5 or 6).

1.8. Pipette the more diluted NH_4OH solution into the electrolytic solution until the pH is set in the desired value. Prepare the electrolyte solutions at pH values of 5 and 6 to study the effect on the anodization process.

2. Substrate cleaning and preparation

2.1. Use 20 mm x 25 mm glass slides (1.1 mm thick) as substrates.

2.2. Sonicate the glass slides in a heated (60 °C) alkaline detergent solution (5% in deionized water) for 15 min. Rinse abundantly in deionized water and dry in clean dry air (CDA) or nitrogen.

2.3. Sonicate the glass slides in acetone (ACS reagent grade or superior) for 5 min. Dry the substrates in CDA or nitrogen.

2.4. Sonicate the glass slides in isopropanol (ACS reagent grade or superior) for 5 min. Dry the substrates in CDA or nitrogen.

2.5. Insert the substrates into the chamber of a plasma cleaner, close the lid and evacuate the chamber using a vacuum pump.

2.6. When the vacuum is achieved, switch on the RF generator at medium power (10.5 W) for 5 min. After plasma cleaning, the substrates are ready for aluminum gate deposition.

3. Aluminum gate electrode evaporation

3.1. Insert the glass slides into mechanical shadow masks to deposit an aluminum stripe of 25 x 3 mm. This aluminum stripe will be used as the TFT gate electrode and the aluminum oxide layer formed by anodization will be the TFT dielectric layer. Example of shadow mask design for the gate electrode is presented in the supplementary files.

3.2. Place the substrates with the shadow mask inside the chamber of the thermal evaporating chamber for the aluminum layer deposition. Shut the chamber. Start the chamber evacuation procedure. Wait until the chamber pressure is below 2.0×10^{-6} mbar to start the thermal evaporation.

3.3. Deposit the aluminum layer. Use two different thicknesses (60 nm and 200 nm) to evaluate the effect on the dielectric layer. Use two different evaporation rates 5 Å/s and 15 Å/s to study the influence of the Al evaporation rate.

3.4. Remove the samples from the evaporation chamber after aluminum evaporation.

3.5. Remove the glass slides with the aluminum stripe from the masks and check if the aluminum layer was properly deposited. The electrode is ready for the anodization process.

4. Anodization process of the aluminum layer

4.1. Attach two alligator clip connectors in a plastic lid that fits on top of the beaker. This lid can be 3-D printed.

4.2. Connect one of the clip connectors to the aluminum strip of a glass slide and the other to a gold-plated stainless-steel sheet (0.8 mm thick, 20 x 25 mm). Face both electrodes towards each other with a separating distance of about 2 cm.

4.3. Use approximately 150 mL of the electrolytic solution (after pH adjustment) in a 150 mL beaker. Use a small magnetic bar to stir the solution during the anodization procedure.

4.4. Place the beaker on top of a magnetic stirrer with heating. Adjust the temperature to the desired value (40 °C and 60 °C were used in the current paper).

4.5. Immerse the electrodes in the electrolytic solution by covering the beaker with the plastic

lid attached to the clip connectors.

4.6. Connect the aluminum electrode to the positive output and the golden-plated stainless-steel electrode to the negative output of a current/voltage source and measuring unit (SMU).

4.7. Calculate the submerged area of the aluminum electrode and apply a constant current equivalent to the desired current density (we used two values 0.45 mA/cm^2 and 0.65 mA/cm^2) and monitor the linear increase of the voltage until the pre-set final value (we used $V_F = 30 \text{ V}$ and $V_F = 40 \text{ V}$).

4.8. After the final voltage is achieved, switch the SMU from the current source to the voltage source and apply a constant voltage (equal to the final voltage) during a time long enough to the current decrease next to zero (about 5 min). Use a script in Python 2.7 to automatically control the SMU during anodization process. A copy of this script is available in the supplementary files section.

4.9. Remove the electrodes from the electrolytic solution, rinse abundantly with deionized water, dry with CDA or nitrogen and store the Al/Al₂O₃ glass substrates until use.

4.10. To observe the effect of annealing on the dielectric layer, anneal the substrates in an oven at 150°C for 1 h.

5. Deposition of the ZnO Active layer

5.1. Insert the substrates with the anodized aluminum oxide layer in appropriate mechanical shadow masks for active layer deposition.

5.2. Place the substrates with the masks inside the chamber of the sputtering system. Use a ZnO (99.9%) sputtering target. Close the chamber and start the evacuating procedure.

5.3. Adjust the Ar pressure to 1.2×10^{-2} Torr and the RF power to 75 W and start the ZnO deposition. Control the deposition rate at 0.5 \AA/s . Stop the ZnO deposition when the active layer thickness achieves 40 nm.

5.4. Open the chamber and remove the samples.

6. Drain and source electrodes deposition

6.1. Insert the samples with the sputtered ZnO layer in appropriate mechanical shadow masks for TFT source/drain electrodes deposition. An appropriate drain and source electrode spacing is $100 \text{ }\mu\text{m}$, with a lateral overlapping of 5 mm. A template of the drain/source mask design is supplied with the supplementary files. In such a configuration, notice that both drain and source electrodes are identical and can be interchangeable without change on the device operation.

6.2. Place the samples attached to the shadow masks inside the chamber of the thermal evaporating system and start the procedure for aluminum evaporation.

6.3. Deposit a 100 nm Al layer at a deposition rate of 5 Å/s to obtain the drain/source electrodes on top of the active layer, finishing the TFT manufacture procedure.

6.4. Remove the TFTs from the evaporation chamber, check the quality of the deposited electrodes and store them protected from light until use.

7. TFT electrical characterization

7.1. Place the TFTs on a semiconductor probe station or custom sample holder. Connect the gate, drain and source electrodes using spring-probe connectors for electrical contacts.

7.2. Connect the probes to a two-channel source-measuring unit (recommended Keithley 2612B or similar). Connect the gate electrode to the “high” output/input of channel 1 and the drain (or source) electrode to the “high” output/input of channel 2. Short the “low” output/input terminals of both channels and the source (or drain) electrode, which remained disconnected.

7.3. Obtain characteristic TFT curves. Obtain the *output curve* by applying constant voltage bias at the gate (V_g) and sweeping the drain-source voltage (V_{DS}) and recording the drain-source current (I_{DS}). Obtain the *transfer curve* by recording the drain-source current (I_{DS}) while sweeping the gate voltage (V_g) and maintaining the drain-source voltage (V_{DS}) constant.

7.4. Plot the square root of the drain current versus the gate voltage ($(I_{DS})^{1/2}$ vs. V_g) and obtain the carrier mobility in the saturation regime (μ_s) from the curve slope and the threshold voltage from the x-axis intercept of the linear portion of the curve.

7.5. If wanted, determine other performance parameters from the transistors curves as described elsewhere¹⁸.

8. ANOVA and influence of design factors on device performance

8.1. Use a software to set a *design of experiment* (DOE) based on a Placket-Burman matrix considering 8 fabrication factors. We used Chemoface, which is a free, user-friendly software developed by Federal University of Lavras (UFLA), Brazil³⁰.

8.2. Use as factors the anodization parameters: i) the thickness of the Al layer; ii) the Al evaporation rate; iii) the water content in the electrolytic solution; iv) the temperature of the electrolyte; v) the pH of the electrolytic solution; vi) the current density during anodization; vii) the annealing temperature and viii) the final voltage of anodization.

8.3. For each factor, consider two levels, as given by **Table 1**.

8.4. Assemble the Plackett-Burman design table aided by the DOE software as given by **Table 2**.

8.5. Prepare the TFTs varying the fabrication parameter according to the 12 generated “runs” from **Table 2**. Each run provides a representative variation of the fabrication factors without the need to perform all 256 (2^8) possible combinations for a two-level, eight-parameters experiment.

8.6. Feed the DOE table from the software with the performance data from TFT characterization (e.g., TFT mobility in saturation) following the manufacturing directions of each run.

8.7. Add as many replicates from different devices using the same fabrication factors to increase the number of degrees of freedom for the analysis.

8.8. Perform ANOVA from the data and analyze the output to determine which anodizing parameters influence most the TFT performance.

REPRESENTATIVE RESULTS:

Eight different aluminum oxide layer manufacture parameters were used as the fabrication factors which we used to analyze the influence on the TFT performance. These factors are enumerated in **Table 1**, where the corresponding “low” (-1) and “high” (+1) values for the two-level factorial DOE are presented.

For simplicity, each manufacturing factor was named by a capital letter (A, B, C, etc.) and the corresponding “low” or “high” level represented by -1 and +1, respectively. The Plackett-Burman DOE matrix considering eight factors varying in two levels results 12 experimental runs, with the combination of levels given by **Table 2**.

Each experimental run from **Table 2** defines the fabrication conditions used to produce the Al_2O_3 layer used as the dielectric layer of a set of transistors with similar expected characteristics. Each set of transistors was electrically characterized by the TFT output and transfer curves. To obtain the mobility in the TFT saturation regime, we use the relationship between the channel current (I_D) and the gate voltage:

$$I_D = \frac{wC_i}{L} \mu \frac{(V_G - V_{th})^2}{2} \quad (4)$$

where w is the channel width, L , the channel length, and C_i , the dielectric layer capacitance per unit area. The transfer curve for a TFT built according to manufacturing parameters given by run #3 from **Table 2** is shown in **Figure 1**. The $I_D^{1/2}$ vs. V_G curve is also depicted in **Figure 1**, allowing the evaluation of the TFT mobility (μ) from the slope of the curve and the threshold voltage (V_{th}) from the extrapolation of the linear region to the horizontal axis.

The values for the mobility for all built transistors according to the 12 runs parameters were

computed in a table and used to feed input of the PB DOE assembled using the DOE/ANOVA analysis software (Chemoface). For each set of fabrication parameters, 6 replicated TFTs were built, resulting in 72 devices. By performing ANOVA, it is possible to rank the most significant factors, which can be graphically expressed using a Pareto chart of effects as shown in **Figure 2a**. **Figure 2** presents the results from the analysis considering the TFT mobility as the response parameter. Similar analysis can be done for different device response parameters (on/off ratio, V_{th} , etc.). **Figure 2b** shows the table of effects and corresponding factor significance. The results demonstrate that the most significant factor for the TFT mobility is the final voltage (H) used during the anodization process. The final voltage is directly proportional to the dielectric layer thickness. The growth ratio is about 1.2 nm/V, which results, for example, in a 48 nm thick layer when using a final voltage of 40 V. Other significant factors were (in the following order): the Al evaporation rate (factor B), the thickness of the Al layer (factor A), the water content in the electrolyte (factor C) and the pH of the electrolyte (factor E). Moreover, all significant factors were found to be “negative”, which means that the TFT mobility decreases as the factor is changed from the “low” (-1) level to the “high” (+1) level given by **Table 1**. The significance of the manufacturing factors can be used as a direction to obtain optimized TFT performance for a particular response parameter (TFT mobility, in the current case).

Figure 1: Transfer curve obtained from a TFT manufactured according to Run #3. The slope of the $(I_{DS})^{1/2}$ vs. V_G allows the determination of the TFT mobility and the intercept with the x-axis, the threshold voltage (V_{th}).

Figure 2: (a) Pareto chart of effects on the TFT mobility. **(b)** Table of effects and corresponding factor significance.

Table 1: Manufacturing parameters of the aluminum oxide TFT dielectric layer. Each factor has a corresponding “low” (-1) or “high” (+1) value.

Table 2: Plackett-Burman (PB) design of experiment matrix

DISCUSSION:

The anodization process used to obtain the dielectric has a strong influence on the performance of the TFTs fabricated, keeping constant all geometrical parameters and the fabrication parameters of the active. For the TFT mobility, which is one of the most important performance parameters for TFTs, it can vary more than 2 orders of magnitude by changing the manufacturing factors in the range given by Table I. Therefore, the careful control of the anodization parameters is of great importance when fabricating devices comprising anodized Al_2O_3 gate dielectrics. The presence of localized states due to charges/dipoles at the semiconductor/dielectric layer is one of the most significant causes of change in the device performance, especially for TFT mobility. Substrate cleaning is very important to avoid spurious variation of electrical parameters from device characterization. Use of alkaline residue-free detergent, use of deionized water for abundantly rinsing the substrates, use of analytical pure acetone and isopropanol for substrate cleaning and plasma cleaning are of extreme importance to assure the cleaning of the substrates and the reproducibility of the process. Rinsing and drying the substrates after the growth of the

anodized layer have also be undertaken with extreme care. Control of the pH of the electrolyte, of the temperature of the electrolyte and stirring the electrolyte solution during anodization are also sources of random variation of the results. Contamination by dust also needs to be avoided by performing all steps inside a clean-room or a laminar flow cabinet. The type of acid used in the electrolyte also affects strongly the anodization process, however, because the effect of such factor cannot be properly quantified in a DOE, we used only tartaric acid, which results in good results for anodization.

The use of ANOVA to determine the significance of each manufacturing factor is an extremely powerful tool for device performance optimization. However, to obtain reliable results, it is essential to guarantee that the variance in the analyzed response parameter is due to factor variation and not by miscarried experimental procedure. A key point is to make as many replicates of each experimental run as possible. Although this increases the number of experiments that need to be performed, it increases the analysis reliability by increasing the number of degrees of freedom of the experimental design. A good strategy which was adopted in the current procedure was to produce 2 samples with 3 TFTs each. Therefore, the experimental run was repeated just once, but we had 6 replicated results from different devices. This also allowed to evaluate the variance for TFTs from the same substrate (same dielectric and semiconducting layers) and for TFTs from different substrates (different dielectric and semiconducting layers but fabricated according to the same procedure). If the variance for devices fabricated according to the similar manufacturing factors is low compared to the variance due to substantial changes in the manufacturing factors, the reproducibility of the process is acceptable.

As stressed before, Plackett-Burman design of experiments is very convenient for experiments with a high number of factors, since it permits a considerable reduction in the number of experiments. For 8 experimental factors, the number of experiments compared to a full-factorial design is reduced from 256 (2^8) to only 12. However, this reduction has the cost that the interaction between the factors cannot be evaluated. Therefore, for systems which the influence of the cross-factors is expected to be relevant, PBD is not the best option. A possibility is to use a PBD to screen the most significant factors and, in a second moment, to use a full-factorial design for the most significant factors from the PBD to determine the influence of the factor interactions.

The use of the experimental design software Chemoface³⁰ in the analysis is optional and the results should not be dependent of it. All the calculations needed to determine the effects of the factors on the system response can be performed manually (extremely time-consuming), by a custom computer-aid script, or by other professional software such as Minitab or Design-Expert. However, Chemoface is a user-friendly and cost-free interface which is available for download without any restriction.

The current work demonstrates feasibility of manufacturing thin-film transistors comprising Al_2O_3 dielectric layer grown by anodization of metallic aluminum. This process can be easily extended to flexible substrates, allowing mass production of flexible electronic circuits. The use

of Plackett-Burman design of experiments combined to ANOVA is a quick and powerful method to screen the influence of manufacturing factors in the device response, permitting the TFT performance optimization.

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DISCLOSURES:

The authors have nothing to disclose.

REFERENCES:

1. Fortunato, E. M. C. et al. Fully Transparent ZnO Thin-Film Transistor Produced at Room Temperature. *Advanced Materials*. **17** (5), 590–594 (2005).
2. Fortunato, E. M. C. et al. Wide-bandgap high-mobility ZnO thin-film transistors produced at room temperature. *Applied Physics Letters*. **85** (13), 2541–2543 (2004).
3. Nomura, K. et al. Thin-film transistor fabricated in single-crystalline transparent oxide semiconductor. *Science*. **300** (5623), 1269–1272 (2003).
4. Noviyana, I. et al. High Mobility Thin Film Transistors Based on Amorphous Indium Zinc Tin Oxide. *Materials* **10** (7) (2017).
5. Nomura, K. et al. Amorphous Oxide Semiconductors for High-Performance Flexible Thin-Film Transistors. *Japanese Journal of Applied Physics*. **45** (5B), 4303–4308 (2006).
6. Kamiya, T., Nomura, K., Hosono, H. Present status of amorphous In–Ga–Zn–O thin-film transistors. *Science and Technology of Advanced Materials*. **11** (4), 044305 (2010).
7. Lin, C. I., Fang, Y. K., Chang, W. C. The IGZO fully transparent oxide thin film transistor on glass substrate. *International Journal of Nanotechnology*. **12** (1/2), 3 (2015).
8. Craciun, V. et al. Optical properties of amorphous indium zinc oxide thin films synthesized by pulsed laser deposition. *Applied Surface Science*. **306**, 52–55 (2014).
9. Suh, S., Hoffman, D. M. A new metal-organic precursor for the low-temperature atmospheric pressure chemical vapor deposition of zinc oxide. *Journal of Materials Science Letters*. **8**, 789–791 (1999).
10. Lin, Y.-Y., Hsu, C.-C., Tseng, M.-H., Shyue, J.-J., Tsai, F.-Y. Stable and High-Performance Flexible ZnO Thin-Film Transistors by Atomic Layer Deposition. *ACS Applied Materials & Interfaces*. **7** (40), 22610–22617 (2015).
11. Walker, D. E. et al. High mobility indium zinc oxide thin film field-effect transistors by semiconductor layer engineering. *ACS Applied Materials & Interfaces*. **4** (12), 6835–41 (2012).
12. Meyers, S. T. et al. Aqueous Inorganic Inks for Low-Temperature Fabrication of ZnO TFTs. *Journal of the American Chemical Society*. **130** (51), 17603–17609 (2008).
13. Krunks, M., Mellikov, E. Zinc oxide thin films by the spray pyrolysis method. *Thin Solid Films*. **270** (1–2), 33–36 (1995).
14. Adamopoulos, G., Thomas, S., Bradley, D. D. C., McLachlan, M. A., Anthopoulos, T. D. Low-

voltage ZnO thin-film transistors based on Y_2O_3 and Al_2O_3 high-k dielectrics deposited by spray pyrolysis in air. *Applied Physics Letters*. **98** (12), 123503 (2011).

15. Branquinho, R. et al. Aqueous combustion synthesis of aluminum oxide thin films and application as gate dielectric in GZTO solution-based TFTs. *ACS Applied Materials and Interfaces*. **6** (22), 19592–19599 (2014).

16. Shan, F. et al. Low-Voltage High-Stability InZnO Thin-Film Transistor Using Ultra-Thin Solution-Processed ZrOx Dielectric. *Journal of Display Technology*. **11** (6), 541–546 (2015).

17. Lin, Y. et al. A Highly Controllable Electrochemical Anodization Process to Fabricate Porous Anodic Aluminum Oxide Membranes. *Nanoscale Research Letters*. **10** (1), 495 (2015).

18. Gomes, T. C., Kumar, D., Fugikawa-Santos, L., Alves, N., Kettle, J. Optimization of the Anodization Processing for Aluminum Oxide Gate Dielectrics in ZnO Thin Film Transistors by Multivariate Analysis. *ACS Combinatorial Science*. acscombsci.8b00195 (2019).

19. Min Li. et al. Dual Gate Indium–Zinc Oxide Thin-Film Transistors Based on Anodic Aluminum Oxide Gate Dielectrics. *IEEE Transactions on Electron Devices*. **61** (7), 2448–2453 (2014).

20. Liu, A. et al. Eco-friendly water-induced aluminum oxide dielectrics and their application in a hybrid metal oxide/polymer TFT. *RSC Advances*. **5** (105), 86606–86613 (2015).

21. Berndt, L. Anodization of Aluminum in Highly Viscous Phosphoric Acid. PART 2: Investigation of Anodic Oxide Formation and Dissolution Rates. *International Journal of Electrochemical Science*. 9531–9550 (2018).

22. Huang, S.Z., Hwu, J.G. Electrical characterization and process control of cost-effective high-k aluminum oxide gate dielectrics prepared by anodization followed by furnace annealing. *IEEE Transactions on Electron Devices*. **50** (7), 1658–1664 (2003).

23. Iino, Y. et al. Organic Thin-Film Transistors on a Plastic Substrate with Anodically Oxidized High-Dielectric-Constant Insulators. *Japanese Journal of Applied Physics*. **42** (Part 1, No. 1), 299–304 (2003).

24. Hickmott, T. W. Electrolyte effects on charge, polarization, and conduction in thin anodic Al_2O_3 films. I. Initial charge and temperature-dependent polarization. *Journal of Applied Physics*. **102** (9), 093706 (2007).

25. Majewski, L. A., Schroeder, R., Grell, M. One Volt Organic Transistor. *Advanced Materials*. **17** (2), 192–196 (2005).

26. Hickmott, T. W. Temperature dependence of the dielectric response of anodized Al– Al_2O_3 –metal capacitors. *Journal of Applied Physics*. **93** (6), 3461–3469 (2003).

27. Hickmott, T. W. Interface states at the anodized Al_2O_3 –metal interface. *Journal of Applied Physics*. **89** (10), 5502–5508 (2001).

28. Anderson, M. J., Whitcomb, P. J. *DOE Simplified: Practical Tools for Effective Experimentation*. (CRC Press: Boca Raton, 2015).

29. Ferreira, S. L. C. et al. Robustness evaluation in analytical methods optimized using experimental designs. *Microchemical Journal*. **131**, 163–169 (2017).

30. Nunes, C. A., Freitas, M. P., Pinheiro, A. C. M., Bastos, S. C. Chemoface: a novel free user-friendly interface for chemometrics. *Journal of the Brazilian Chemical Society*. **23** (11), 2003–2010 (2012).

Figure 1

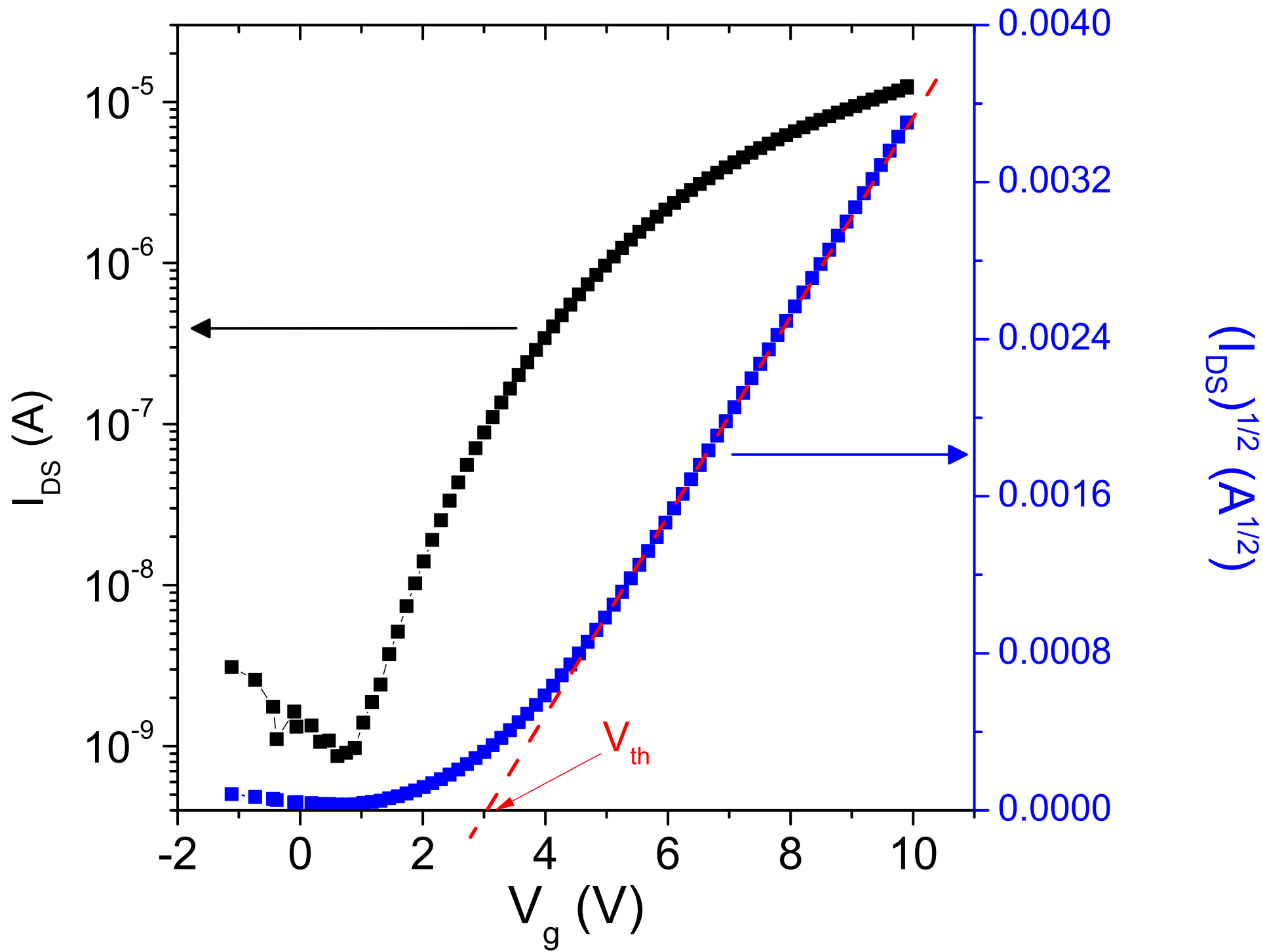
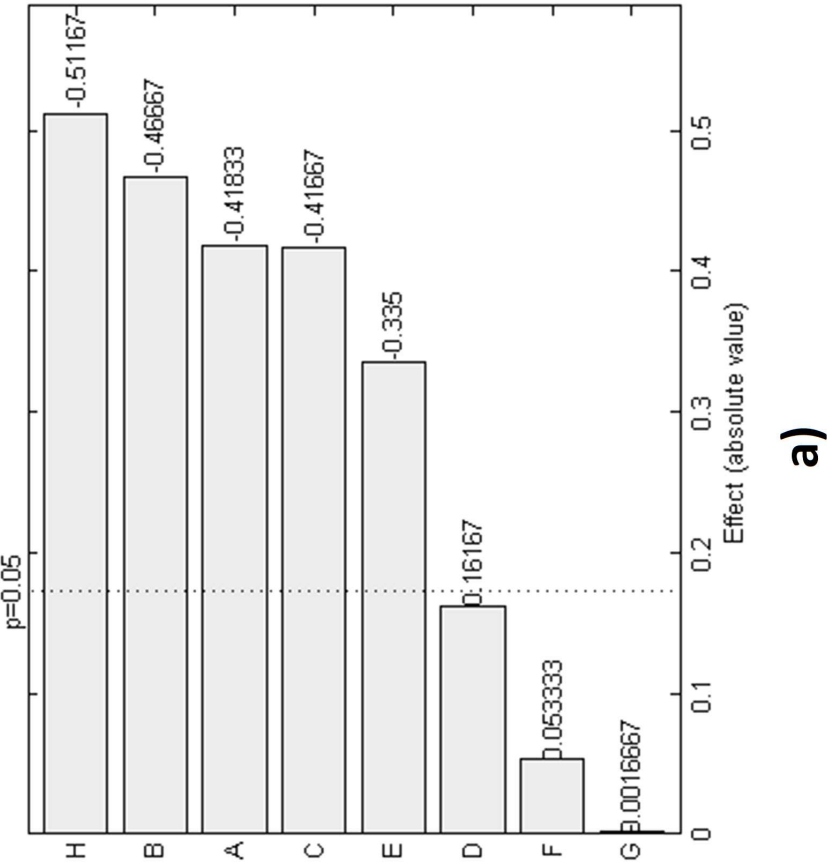


Figure 2



	Effect	Error	t	p	significant
H	-0.5117	0.0542	-9.4458	0.0025	yes
G	0.0017	0.0542	0.0308	0.9774	no
F	0.0533	0.0542	0.9846	0.3974	no
E	-0.3350	0.0542	-6.1844	0.0085	yes
D	0.1617	0.0542	2.9845	0.0584	no
C	-0.4167	0.0542	-7.6920	0.0046	yes
B	-0.4667	0.0542	-8.6150	0.0033	yes
A	-0.4183	0.0542	-7.7228	0.0045	yes

A: thickness of the Al layer; **B:** Al evaporation rate;
C: Water content; **D:** temperature of the electrolyte;
E: pH of the electrolyte; **F:** current density;
G: Annealing; **H:** final voltage.

b)

Table 1: Manufacturing parameters of the aluminum oxide TFT dielectric layer.
Each factor has a corresponding “low” (-1) or “high” (+1) value.

	Factors	Unit	“Low” value (-1)	“High” value (+1)
A	Thickness of Al-layer	nm	60	200
B	Al evaporation rate	Å /s	5	15
C	H ₂ O content	%	16	30
D	Temperature of electrolyte	C	40	60
E	pH of the electrolytic solution	-	6	5
F	Current density	mA/cm ²	0.45	0.65
G	Annealing	C	No thermal treatment	Annealed at 150 °C
H	Final voltage	V	30	40

Table 2: Plackett-Burman (PB) design of experiment matrix.

Run	A	B	C	D	E	F	G
1	-1	-1	-1	-1	-1	-1	-1
2	1	-1	1	1	-1	1	1
3	-1	-1	-1	1	-1	1	1
4	1	-1	-1	-1	1	-1	1
5	1	1	-1	1	1	1	-1
6	-1	1	-1	1	1	-1	1
7	1	1	1	-1	-1	-1	1
8	-1	-1	1	-1	1	1	-1
9	1	-1	1	1	1	-1	-1
10	1	1	-1	-1	-1	1	-1
11	-1	1	1	-1	1	1	1
12	-1	1	1	1	-1	-1	-1

[illegible]

Name of Material/Equipment	Company	Catalog Number
Acetone	LabSynth	A1017
Aluminum (Al) Wire Evaporation	Kurt J. Lesker Company	EVMAL40060
Ammonium hydroxide solution	Sigma Aldrich	338818
Chemoface - Software to set a design of experiment (DOE)	Federal University of Lavras (UFLA), Brazil	
Cleaning detergent	Sigma Aldrich	Alconox
Ethylene glycol	Sigma Aldrich	102466
Isopropanol	LabSynth	A1078
Glass substrates	Sigma Aldrich	CLS294775X50
L-(+)-Tartaric acid	Sigma Aldrich	T109
Mechanical shadow mask for deposition of the sputtered ZnO active layer	Lasertools, Brazil	custom mask
Mechanical shadow mask for TFT gate electrode	Lasertools, Brazil	custom mask
Mechanical shadow mask for TFT source/drain electrodes	Lasertools, Brazil	custom mask
Plasma cleaner	MTI	PDC-32G
Sputter coating system	HHV	Auto 500
Stiring plate	Sun Valley	MS300
Thermal evaporator	HHV	Auto 306
Two-channel source-measuring unit	Keithley	2410
Two-channel source-measuring unit	Keithley	2612B
Ultrasonic bath	Soni-tech	Soni-top 402A
Zinc Oxide (ZnO) Sputtering Targets	Kurt J. Lesker Company	EJTZNOX304A3

Comments/Description

ACS reagent grade

1.5 mm (0.060") Dia.; 1lb; 99.99%

ACS reagent, 28.0-30.0% NH₃ basis

Free software developed by Federal University of Lavras (UFLA), Brazil - <http://www.ufla.br/chemoface/>

Alkaline detergent for substrate cleaning

ReagentPlus, ≥99%

ACS reagent grade

Corning microscope slides, plain

≥99.5%

10 mm x 10 mm square.

25 mm long stripe, 3 mm wide.

100 μm stripes, separated by 100 μm gap, overlapping of 5 mm

Compact plasma cleaner with vacuum pump

RF sputtering system with thickness and deposition rate control

Stiring plate with heating control

it has a high precision sensor for measure the thickness and rate of deposition of thin films

Keithley model 2410 or similar/for anodization process

Dual channel source-measure unit (SMU) for TFT measurements

Ultrasonic bath with heating control

3.0" Dia. x 0.250" Thick; 99.9%

Rio Claro, March 30th 2020

Nam Nguyen, Ph.D.

Manager of Review/JOVE

Dear Dr. Nguyen,

Please consider the reviewed versions of the manuscript and the video entitled “The Effect of Anodization Parameters on the Aluminum Oxide Dielectric Layer of Thin Film Transistors”, authored by Tiago C. Carneiro, Dinesh Kumar, Neri Alves, Jeff Kettle and Lucas Fugikawa-Santos, for publication in Journal of Visualized Experiments.

We believe that we attended all the required changes, as commented bellow.

We hope that the manuscript will accomplish the necessary requirements for publication in your journal.

Sincerely,

Lucas Fugikawa-Santos, PhD

Sao Paulo State University - UNESP

Campus of Rio Claro

Av. 24-A, 1515,

Physics Department

13506-900

Rio Claro, SP, Brazil

Comments on the reviews:

Editorial comments:

1. Audio Track Quality:

a. @11:26-11:51 Previous note: Dr. Jeff Kettle is out of sync- meaning his audio does not match his video (movement of his lips.) The video is early / the audio is late. This will need to be fixed.

Video and audio were synchronized.

b. @11:51 The second clip of Jeff Kettle is okay

2. On-Screen Text:

a. @04:14 Lower text has an extra space after "Remove the"

Extra space deleted.

b. @04:20 Lower text has an extra space after "abundantly the"

Extra space deleted.

c. @04:26 Lower text has an extra space after "Dry the"

Extra space deleted.

d. @04:44 Lower text has an extra space after "Dry the"

Extra space deleted.

e. @04:53 Lower text has an extra space after "Remove the"

Extra space deleted.

f. @05:02 Lower text has an extra space after "Dry the"

Extra space deleted.

g. @05:08 Lower text has an extra space after "Load the"

Extra space deleted.

h. 08:39 Please hold on this chapter title for at least another second. It disappears before we can fully read it

Exposure time of the title was increased 1.25 s.

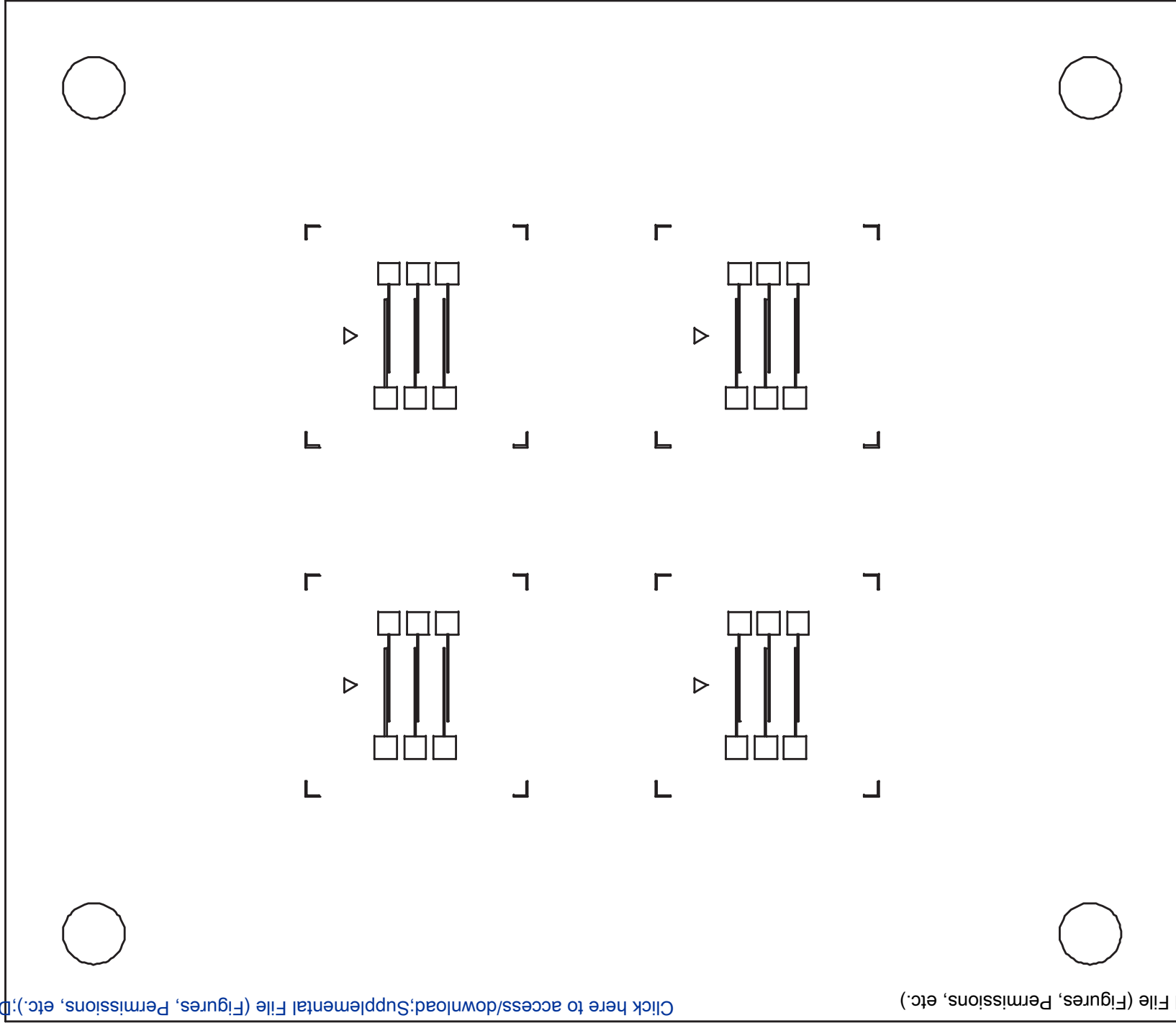
3. One minor inconsistency-the first sonication (in the detergent, step 2.2.2) is for 10 minutes in the manuscript but 15 minutes in the video.

We changed in the manuscript for 15 min.

80 mm

70 mm

80 mm



80 mm



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Supplemental Coding Files

[Keithley_2400_Al2O3_growth.py](#)





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Effect of anodization parameters on the properties of aluminum oxide used as dielectric layer of thin-film transistors

Author(s):

Tiago C. Gomes, Dinesh Kumar, Neri Alves, Jeff Kettle and Lucas Fugikawa-Santos

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