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Posted Date: 24 August 2024

doi: 10.20944/preprints202408.1686.v1

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Article

Enhancing Performance of Nanocrystalline SnO₂ by Photonic Curing Using Impedance Spectroscopy Analysis

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Abstract: Wide-bandgap tin oxide (SnO₂) thin-films are frequently used as an electron-transporting layers in perovskite solar cells due to superior thermal and environmental stabilities. However, its crystallization by conventional thermal methods requires typical temperatures between 300 and 900 °C [1] with up-to 60 min crystallization times. These post-processing conditions severely limit the choice of substrates and reduce the large-scale manufacturing capabilities. This work describes the intense pulsed light-induced crystallization of SnO₂ thin films using only 500 µs of exposure time. Thin-film properties are investigated using both impedance spectroscopy and photoconductivity characteristic measurements. Nyquist plot analysis establishes that the process parameters have a significant impact on the electronic and ionic behaviors of the SnO₂ films. Most importantly, we demonstrate that light-induced crystallization yields improved topography and excellent electrical properties through enhanced charge transfer, improved interfacial morphology and better ohmic contacts compared to thermally-annealed SnO₂ films.

Keywords: Impedance spectroscopy; Photonic curing; SnO₂; Dark injection current transient, Photo-Celiv

1. Introduction

Electron-transporting layers (ETLs) are critical components in most optoelectronic device architectures, including perovskite solar cells (PSCs). These PSC devices rely on organic-inorganic perovskite materials to efficiently absorb light and generate charge carriers [2–4]. ETL layers are essential to promote efficient electron transport, block holes, align energy levels, and ultimately enhance the efficiency and stability of the perovskite solar cells. Choosing appropriate ETL materials is essential for the performances of PSCs. Typical ETL materials require processing between 150 and 500 °C, resulting in higher processing times and energy costs. Most importantly, this prevents their integration on most low-cost substrates requiring processing temperatures below 150 °C [5,6]. In this context, intense pulsed light annealing also sometimes referred-to as photonic curing (PC) [7] is an emerging technique ideally suited for large-scale manufacturing as it relies on short, high-intensity light pulses to anneal materials selectively and rapidly [8,9]. In this process, the optical energy absorbed by the active material can sustain carefully-controlled light-induced annealing with minimal substrate damage. As a result, even metals with relatively high melting-points can be successfully sintered on low-cost plastic- or paper-based substrates [10–12]. As such, this technique is also especially well-suited for roll-to-roll (R2R) manufacturing [13]. SnO₂ metal-oxide thin films were first utilized as ETL for perovskite-based solar cells nearly a decade ago [14,15]. It has since emerged as the preferred material for PSC over TiO₂ and ZnO due to its large band gap, higher charge mobility, and better stability under ambient conditions [16–18]. A few years later SnO₂ films were photonic annealed in just 20 ms, enabling the fabrication of PSCs with reduced hysteresis and a 15 % power conversion efficiency [10]. However, these previous studies did not address the effect of photonic curing on the electronic properties of SnO₂ films. To investigate this, we used impedance spectroscopy (IS), a rapid technique for evaluating these properties. IS is a powerful tool to shed light on the kinetic processes taking place within electrochemical systems [19,20]. During measurement, a small alternative current (AC) signal is coupled with a direct (DC) voltage applied to the device. The phase difference between the DC voltage and

AC current is measured over a wide frequency range to identify the various physical effects in the device. As a result, IS measurements can access the physical and chemical processes of various types of devices, including optoelectronic devices, fuel cells, and solid state batteries [21]. It is a non-destructive [15,22,23] tool that can effectively be used to optimize the stability and performance of these devices by characterizing their charge transport properties [19,24]. Typically, the IS measurements exhibit two arcs corresponding to low frequency (LF) and high frequency (HF) responses. Series resistance (R_s), charge-transfer resistance (R_{CT}) and parallel capacitance can be determined from the HF and LF responses.

This work explores the impact of the photonic curing parameters on the thin-film SnO_2 properties using IS and photocurrent characteristic analysis to unveil and control the ionic and electronic kinetics within the treated SnO_2 layer. As we demonstrate, this improved understanding and control leads to enhanced electronic properties with great potential for improved perovskite solar cell manufacturability.

2. Experimental Section

The commercial patterned FTO substrates (SHENZHEN HUAYU UNION TECHNOLOGY, resistance 7 Ohm/sq) doped with fluorine are cleaned using a sequential process of DI water, acetone, and IPA in an ultrasonic bath for 10 minutes each. After drying with a nitrogen spray gun, residual organic contaminants are removed by performing a 15 min O_2 plasma treatment (Plasma Etch, PE-100LF). To prepare the SnO_2 solution, a colloidal precursor of SnO_2 obtained from Alfa Aesar (15 % in H_2O colloidal dispersion CN: 044592.A3), were diluted with DI water to a concentration of 3 % by volume. The SnO_2 solution is spin-coated onto the clean FTO substrate in one step in air (3000 rpm for 30 s). The edges of the FTO electrodes are then cleaned with a dry cotton swab to enable electrical and IS measurements (Figure 1). For TA, SnO_2 films were annealed using a hot plate at 150 °C for 30 minutes under ambient air. For photonic curing, each sample is treated using Novacentrix PulseForge system (500 V/3 A power supply with 3 capacitors provides a radiant energy greater than 20 $\text{J}\cdot\text{cm}^{-2}$ using lamp system (7.6 cm x 60.8 cm) with an illumination area of 300 mm x 75 mm. The Paois (Fluxim AG, SN:20121) tool was used for all electrical and IS measurements. SEM (SU8230 Hitachi) and AFM (Bruker, MultiMode8) were used for topography inspection.

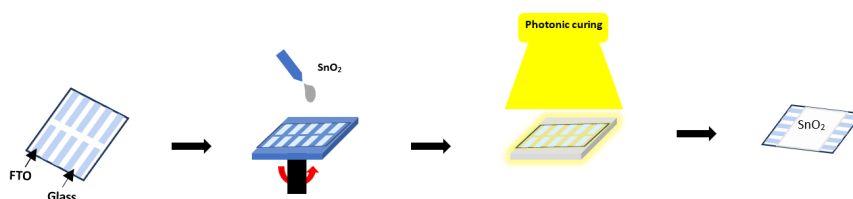


Figure 1. Illustration of the SnO_2 sample fabrication process.

3. Results and Discussion

After deposition of colloidal SnO_2 films using the protocol, samples are post-processed using varying pulse durations and energy densities using the methodology described in the Experimental section. To investigate the impact of PC on the electrical properties of SnO_2 films, we conduct flash annealing for pulse durations of 500, 1500, 2500, and 3500 μs , followed by photocurrent measurements. This allowed us to optimize our photonic annealing parameters and define the high photoconductivity range for SnO_2 films. Photocurrent analysis is used to map the different zones photoconductivity. Pulses ranging from 500 to 3500 μs are utilized to complete the photo-responsivity characterization. Figure 2a shows the I-V responses in the dark and under illumination for two samples photonic treated using pulse duration of 2500 μs and respectively 2 $\text{J}\cdot\text{cm}^{-2}$ and 4 $\text{J}\cdot\text{cm}^{-2}$. A low photo-responsivity indicates that the illumination and dark curves approach the overlap limit, while a high photo-responsivity indicates a clear offset (more than 0.5 order of magnitude) between the I-V characteristics in the dark and under illumination. Based on such measurements, Figure 2b displays photo-responsivity map

for samples photonicallly-treated using different pulse duration vs energy density. To shed light on these results, IS and SEM characterization are conducted. SnO_2 is highly transparent, which makes it difficult to using photonic curing [25]. To mitigate this problem, we used substrates with FTO patterns that act as a structural support and a stable base for the growth of SnO_2 nanoparticles. This helps promote the transmission of heat generated when light is absorbed by the nanoparticles [26], which can increase the local temperature around the nanoparticles and promote the recrystallization process. FTO substrates exhibit rougher surfaces than glass [27], promoting superior adhesion and growth of SnO_2 nanoparticles [28]. Their conductivity enhances the electrical properties of resulting SnO_2 films. The FTO substrate's roughness directly influences both the diameter and alignment of SnO_2 nanoparticles [29]. Areas with FTO patterns acting as a blanket allow for changes in nanoparticle recrystallization depending on the energy density used.

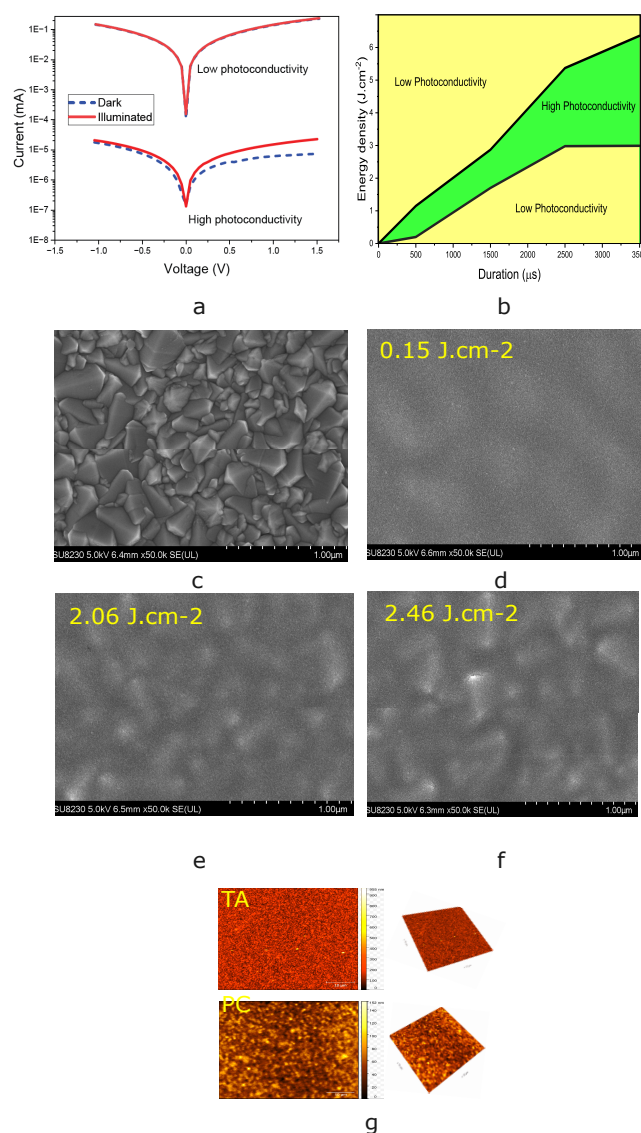


Figure 2. a) I-V responses in the dark and under illumination for two samples photonicallly treated using pulse duration of 2500 μs and respectively 2 and 4 J.cm⁻², b) Photo-response map for samples photonicallly treated using different pulse duration vs. energy density based on the criterion of Figure 2a, c) SEM images of FTO/glass, d-f) SEM images of PC of SnO_2 samples on FTO/glass. g) Atomic Force Microscopy (AFM) images 2D and 3D of thermally and photonicallly-annealed samples.

Figure 2c displays SEM images of the bare FTO substrate and SnO₂ films deposited on FTO and photonically-treated using energy densities of 0.15, 2.06 and 2.46 J.cm⁻² (Figure 2(d-f)). As the energy density is increased from 0.15 to 2.06 and 2.46 J.cm⁻² using 1500 μs pulse durations, the recrystallization of SnO₂ film helps in binding the films to the substrate and reveals the FTO grain profile underneath. This process indicates that higher energy densities lead to improved film-substrate adhesion and more pronounced exposure of the underlying grain structure. The photonic curing of SnO₂ wet films enables water evaporation and subsequent crystallization of SnO₂ nanoparticles [30]. The degree of crystallization will greatly affect the photoconductivity of SnO₂ films and their ability to carry charge carriers [31,32]. Its properties will largely depend on two independent parameters: the energy density and pulse duration of the pulsed light.

To obtain quantitative information and better understand surface morphology and roughness, we also conduct AFM analysis on samples subjected to different types of annealing treatments. Figure 2g shows the surface roughness of the films samples with a scan area of 5x5 μm². It highlights the improvement in surface uniformity after optimal photonic treatment of SnO₂, with a root-mean-square roughness of 14.01 nm, compared to 45.57 nm for the thermally-annealed sample. This significant improvement underscores another important advantage of photonic treatment in enhancing the quality of SnO₂ as an electron transport layer (ETL) in perovskite solar cells.

This section focuses on the variation of IS results for SnO₂ films treated with different energy densities and pulse durations of 500, 1500, 2500, and 3500 μs. For these measurements, the SnO₂ film is deposited onto FTO glass, and its electrochemical behavior can be represented by an equivalent circuit that produces a semicircle on the Nyquist diagram. Figure 3(a-d) displays IS results for SnO₂ samples treated using these different pulse durations and energy densities. When the pulse duration is fixed and the energy density is increased, the semicircle decreases until it reaches its minimum, and then the arc widens. The frequency response exhibits two distinct behaviors. At high frequencies (HF), it is dominated by the resistance attributed to electronic transport (R_{CT}). At low frequencies (L_F), it is dominated by the recombination resistance (R_{rec}) related to ionic diffusion and charge accumulation at contacts [33,34]. In Figure 3, it corresponds to the second semicircle inclined at 45° to the real axis in the Nyquist graph [35]. The semi-circle in the high-frequency region is generally related to the counter-electrode and its interface [36]. A smaller half-circle suggests lower R_{CT} and better photoconductivity of the device. These Nyquist plots suggest that our devices' equivalent circuit can be accurately modeled by a resistor-capacitor (RC) pair in the dark AC regime [37]. As such, the interface contribution can be derived from the equivalent circuit's parameters [38]. The series resistance (R_s) can be obtained by measuring the shift of the semi-circle from the origin along the horizontal axis [39]. However, the time constant related to the physical phenomenon dominating at both low and high frequencies is described by ($\tau_{HF}.\omega_{HF} = 1$, $\tau_{LF}.\omega_{LF} = 1$, with $\omega_{HF,LF} = 2\pi f_{max,HF,LF}$) [37]. The time constants can be deduced from the IS results identifying the peak of the semicircle, which corresponds to the maximum frequency, or by calculating $\tau = Req.Ceq$, as shown in the Table 1.

Table 1. IS parameters extracted from the Nyquist plot for thermally-annealed and photonically-treated samples at 0 V in dark condition at 0.07 V perturbation. Photonic treatment is performed using 3500 μs pulse duration at 3.55 J.cm⁻² energy density.

Device	R_s (kΩ)	R_{CT} (MΩ)	Ceq (pF)	τ_{HF} (μs)
Thermally-annealed	1.96	0.99	0.88	0.87
Photonically-treated	3.06	0.49	0.78	0.38

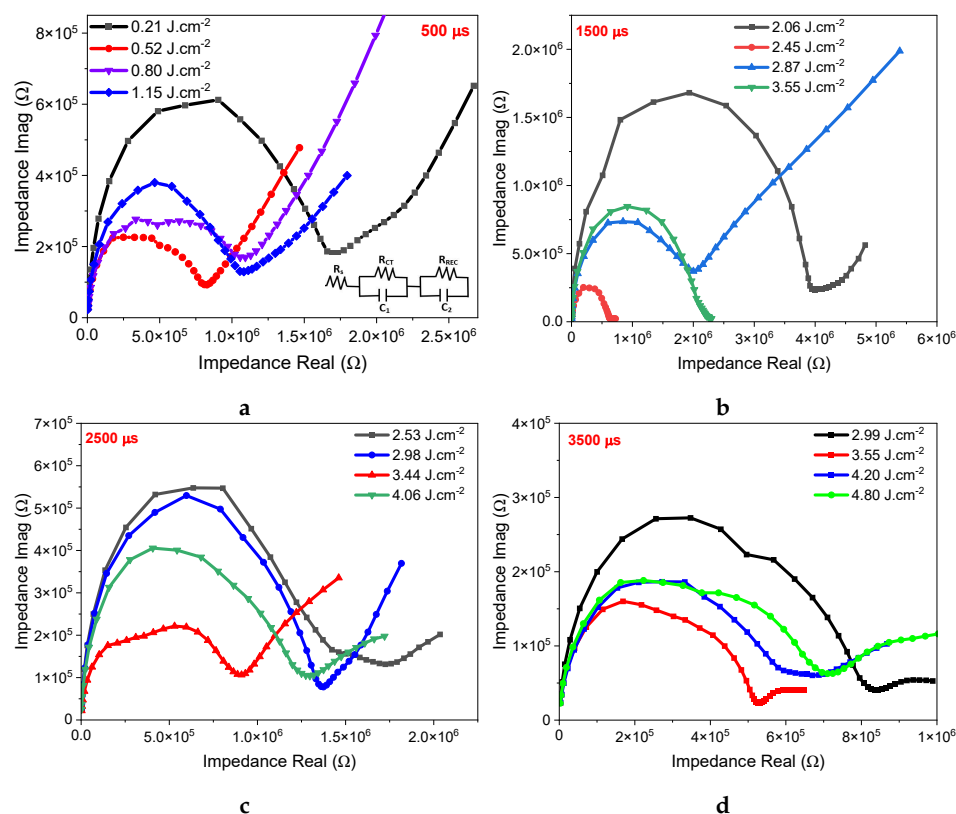


Figure 3. IS for photonic annealing films, respectively with pulse duration of 500, 1500, 2500 and 3500 μs .

Figure 4a compares the Cole-Cole plot for films photonic-treated using 500, 1500, 2500, and 3500 μs with energy densities of 0.52, 2.45, 3.44, and 3.55 $\text{J}\cdot\text{cm}^{-2}$ respectively, with a typically film sample crystallized using standard thermal annealing. Clearly, the physical and chemical properties of the resulting SnO_2 films appear greatly affected by pulse duration and energy density. When the pulse duration is 3500 μs and the energy density is 3.55 $\text{J}\cdot\text{cm}^{-2}$, the high-frequency arc is becoming smallest, suggesting that the film is less resistive and facilitating charge transfer. In comparison, the thermally-annealed sample exhibits the largest semicircle compared to all photonic-treated samples. This suggests an increased imaginary impedance associated with a decrease in charge transfer. Figures 4(b-d) compares the imaginary impedance, capacitance, and conductance versus frequency for the best thermally-annealed and the best photonic-treated films at conditions (3.55 $\text{J}\cdot\text{cm}^{-2}$, 3500 μs). In Figure 4b, the high-frequency (HF) peaks appear between 10^5 to 10^6 Hz for both samples. The response time can be obtained by taking the inverse of the peak frequency from the imaginary impedance graph. Table 1 presents the IS parameters extracted from the spectra. There, the R_{CT} value for the thermally-annealed sample is roughly twice the value achieved using optimal photonic curing conditions. This suggests that the SnO_2 /FTO interface provides a low R_{CT} under the effect of photonic annealing, which facilitates charge carrier transport. The resulting time constant is 0.8 μs for the thermally-annealed film compared to 0.38 μs for optimal photonic curing conditions. This suggest that photonic-induced crystallization promotes a faster response time, resulting in low recombination and more dominant ionic diffusion behavior [40,41]. At low frequencies, the thermally-annealed device doesn't exhibit any measurable peak, which is consistent with the presence of a single semicircle in Figure 4b. In contrast, the impedance plot of the photonic-treated device is curved at low frequencies, explaining the start of the second semicircle in this region. Frequency, time constant, and conductivity values are good indicators of process kinetics [42,43]. Indeed, dark IS can be directly related to carrier density, mobility and conductivity [35].

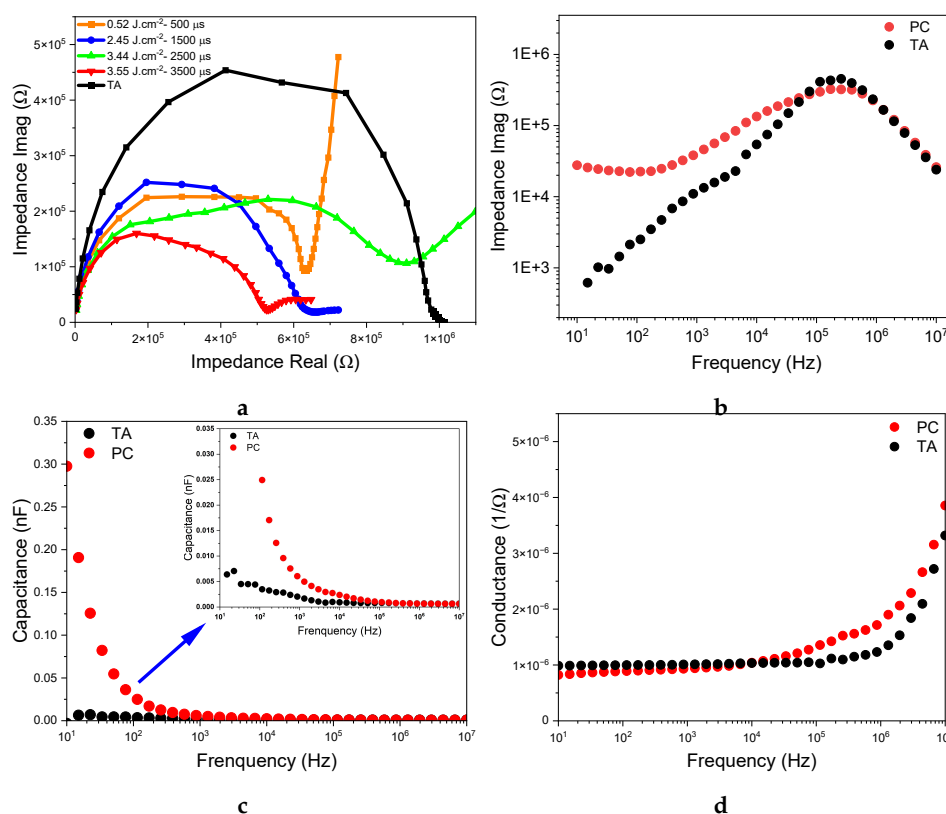


Figure 4. a) Cole-Cole plot for films thermally and photonically-treated using 500, 1500, 2500, and 3500 μs with energy densities of 0.52, 2.45, 3.44, and 3.55 J.cm⁻²; b,c and d) Comparison of imaginary impedance, capacitance, and conductance vs. frequency for typical thermally annealed and photonically treated samples.

Figure 4(c-d) shows capacitance and conductivity evolutions as a function of the operation frequency. Figure 4(c) illustrates two distinct capacitance behaviors, each corresponding to a specific polarization process. This distinction makes it possible to identify specific capacitive processes directly from the plot [44,45]. The high-frequency capacitance C_{HF} (above 100 kHz) exhibits a plateau in the order of 1 pF for both thermally- and photonically-treated devices and is rather similar for both annealing processes. This region represents the geometric capacitance and is due to the intrinsic dielectric polarization of the SnO₂ layer [44]. However, the photonic treatment achieves higher capacitance values at low frequencies (below 1 KHz) compared to the thermally-annealed device. This is primarily due to the accumulation of charges or ions [46,47], resulting from the polarization of the interfaces between the SnO₂ layer and the electrodes. At low frequencies, the increase in capacitance is dominated by ionic movement in the dark and electronic movement in the light [48,49]. In circuits that exhibit capacitive behavior, the capacitor offers less resistance to the flow of alternating current as the frequency increases. Accordingly, Figure 4(d) shows an increase in conductance for both devices in the high-frequency region. This behavior is consistent with that of semiconductors, where capacitance and conductance vary inversely [50–52].

Measurements in Figure 5(a,b) compare the dark injection transients for the photocurrent rise and decay for the thermally- and photonically-treated (3.55 J.cm⁻², 3500 μs) samples. This time-of-flight technique is useful for determining majority carrier mobility and trapping, especially in thin films [53]. Figure 5a illustrates that the currents for the photonically-treated film rises to 2.7 mA, compared to 2.3 mA for the thermally-annealed film. The current also increases more rapidly in the photonically-treated sample, reflecting the interrelationship between charge carrier generation and recombination. Therefore, the rapid increase in current for the PC sample can be attributed to the fast accumulation of photogenerated carriers [54]. Figure 5b compares the decay of the transient

current. After reaching its maximum, the current decay depends on the charge capture coefficient [55]. The decay graph illustrates the speed of charge recombination after being excited by a 1.2 V pulse voltage. A shorter carrier lifetime suggest faster recombination and a high carrier capture rate, which implies more rapid current decay for the thermally-annealed sample. In contrast, photonic curing yields a lower recombination rate, resulting in slower decay and longer current holding times. The photogeneration and recombination processes have a significant impact on the density and mobility of charge carriers. Figure 5c compares the charge mobility using the photo-CELIV technique, using the following expression.

$$\mu = \frac{2d^2}{3A \cdot t_{max}^2 (1 + 0.36 \frac{\Delta J}{J_{max}})} \quad (1)$$

Where d is the SnO_2 film thickness, A is the slope of the extraction voltage ramp, t_{max} is the time related to the current peak, and ΔJ is the difference between the maximum current and the displacement current plateau. Photo-CELIV is a technique used to extract charge mobility by illuminating the device. The measurement displays the current overshoot and the time at which the current reaches its maximum, which is an essential parameter for quantifying mobility. However, it should be noted that Photo-CELIV only measures fast carriers and cannot distinguish between the mobility of electrons or holes. The Photo-CELIV measurements for the film after optimized photonic treatment yield $4.56 \times 10^{-2} \text{V cm}^2 \text{s}^{-1}$, compared with $3.66 \times 10^{-2} \text{V cm}^2 \text{s}^{-1}$ for thermally-annealed film. This measurement does not precisely reflect the mobility of the SnO_2 material. However, it serves as a characterization for comparing the fastest or maximum carrier mobility values. This higher maximum mobility compared to thermal annealing is consistent with previous results.

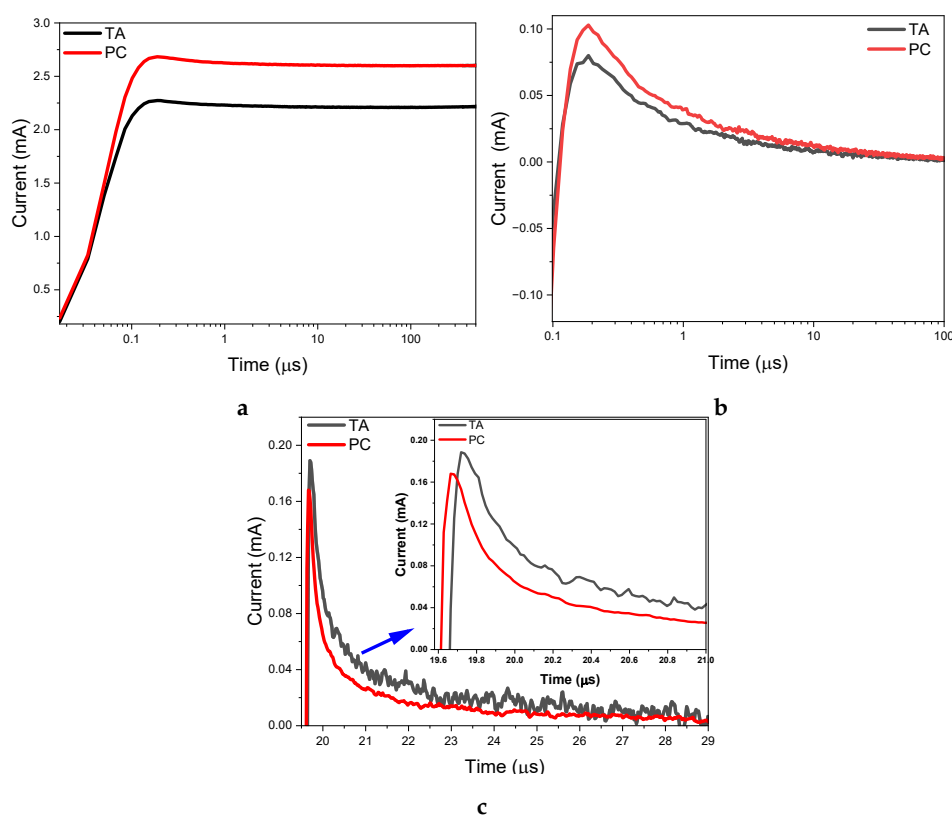


Figure 5. a,b) Dark injection transients for the photocurrent rise and decay for the thermally and photonic-treated samples, c) Charge mobility using the photo-CELIV technique for the thermally- and photonic-treated samples.

4. Conclusion

In summary, we propose an optimized photonic annealing approach to improve electrical properties of SnO₂ thin-films compared to standard annealing. SnO₂ thin-films play an essential role in emerging device architectures, especially as the electron-transporting layer (ETL) for perovskite-based solar cells. We use impedance spectroscopy to analyze the electrical behavior of SnO₂ films in the dark. The results indicate that the impedance spectroscopy response depends significantly on both energy density and pulse duration, shedding light on the resulting ionic and electronic transfer. Additionally, we demonstrate that the photonic treatment yields SnO₂ layers with enhanced electrical performance and a significantly-reduced manufacturing time compared to standard thermal annealing. This would be a great advantage for large-scale manufacturing of better and cheaper perovskite-based solar cells.

Author Contributions: Writing - original draft, Methodology, Resources, Conceptualization, Formal analysis, M.A.S; Supervision, visualization, Review and editing, S.G.C and R.I. All authors have read and agreed to the published version of the manuscript.

Funding: The authors would like to acknowledge the financial support received from NSERC with the following Discovery grants (RGPIN-2023-05211, RGPIN 2022-03083) as well as the Canada Research Chairs (CRC-2021-00490)

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

ETL	Electron-transporting layer
PSC	perovskite solar cell
TA	Thermal annealing
PC	Potonic curing

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