

Flash-Lamp Annealing for Enhanced Electrical and Optical Properties of AZO/AgNW/ZnO Transparent Conductive Films

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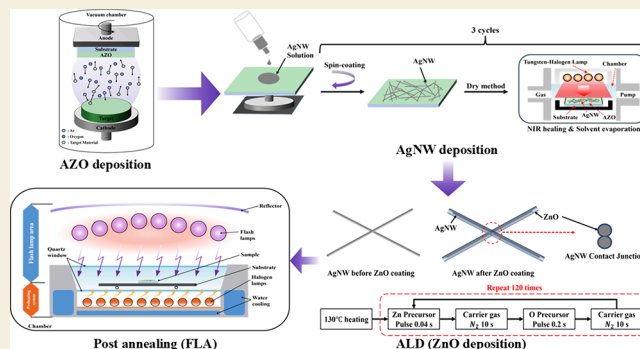
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ABSTRACT: Transparent conductive oxides act as transparent electrodes in touch screens, flat panel displays, and solar cells. Their unique optical properties, such as visible light transmission and infrared reflection, enable applications such as low-emissivity windows, infrared reflectors, gas sensors, photocatalysts, and even plasmonic devices. Current market is dominated by indium tin oxide with indium belonging to the critical raw material. Therefore, there is a strong need for alternatives such as aluminum-doped zinc oxide (AZO). In this work, we present the influence of the millisecond (ms)-range flash lamp annealing (FLA) on structural, optical, and electrical properties of AZO films decorated with silver nanowires (AgNWs). The FLA for 23 ms improves the layer conductivity by five times, the transmittance in the visible spectral range improves by about 5% for the sample with 1.0 mg/mL AgNWs and the figure of merit increases by an order of magnitude. The scanning electron microscopy images show the formation of nanojunctions at the crossing points of Ag NWs that is mainly responsible for the improvement of electrical conductivity, while improved crystallinity of the entire coating increases the transmittance.

KEYWORDS: transparent conductive films (TCF), aluminum-doped zinc oxide (AZO), silver nanowires (AgNWs), oxide–metal–oxide (OMO), flash lamp annealing (FLA), optoelectronic properties



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1. INTRODUCTION

Transparent conductive films (TCFs) are widely applied in OLEDs, touch panels, and smart windows, where achieving high optical transmittance along with low sheet resistance remains a central research challenge.¹ Indium tin oxide (ITO), the most commonly used transparent conductive material, exhibits over 90% transmittance at 550 nm and a low sheet resistance of 10–15 Ω/sq , establishing it as the industrial standard. However, issues such as brittleness, high expense, and indium scarcity have motivated a continuous search for alternative materials. Among these, ZnO has gained significant attention due to its wide bandgap, high transparency, and compatibility with low-temperature solution processing, enabling its broad use in flat-panel displays, photosensors, and touchscreen electronics.² Unfortunately, its inherently lower electrical conductivity compared with ITO limits its capability as a standalone replacement.

To overcome this limitation, Al-doped ZnO (AZO) has emerged as a promising candidate for next-generation of transparent conductive oxides.³ Al³⁺ substituting Zn in the ZnO lattice increases the free-electron concentration and significantly enhances electrical conductivity close to that

achievable for ITO layers while relying on abundant only elements such as Al, Zn, and O.⁴

In addition to metal oxides, carbon nanotubes, graphene, and metallic nanowires have also gained traction as potential ITO alternatives.⁵ Metallic nanowires, in particular AgNWs with high conductivity, offer tunable aspect ratios (lengths vs nanowire diameter) that enable application across a broad range of devices.⁶ Ag- and Cu-based nanowires form efficient percolation pathways due to their high aspect ratios, enabling sheet resistances competitive with ITO.^{7,8} Despite these advantages, issues such as electrical connections between individual NWs, thermal instability, vulnerability to oxidation, and surface roughness hinder their widespread use.^{9,10} The possible solution relies on the coating of metallic nanowires with a protective layer. OMO (oxide/metal/oxide) structures incorporating protective top layers such as TiO₂, AZO, and

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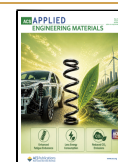


Table 1. Comparison of Optoelectrical Performance and Annealing Conditions in Multilayer Transparent Electrodes

structure	FoM ($\times 10^{-3} \Omega^{-1}$)	sheet resistance (Ω/sq)	transmittance	annealing method	annealing time (sec.)	ref
AZO/AgNW/ZnO	6.20	10.48	76.08% (550 nm)	Xe-Flash lamp	0.023	this work
AZO/AgNW/AZO	6.0	8.6	74.40% (550 nm)	thermal (250 °C)	3600	9
AZO/AgNW/AZO	4.1	27.6	80.5	as-deposited	0	21
AZO/pd/AZO	3.53	45	83.2%	thermal (400 °C)	5400	22
AZO/AgNW		5–30	75–93	as-deposited	0	23
AZO/AgMgAl	5.5	45	87%	laser (0.5 μJ)		24
AgNW/GZO	~ 8.0	11.9 ± 1.3	$\sim 80\%$	APPJ (plasma jet)	during process	25

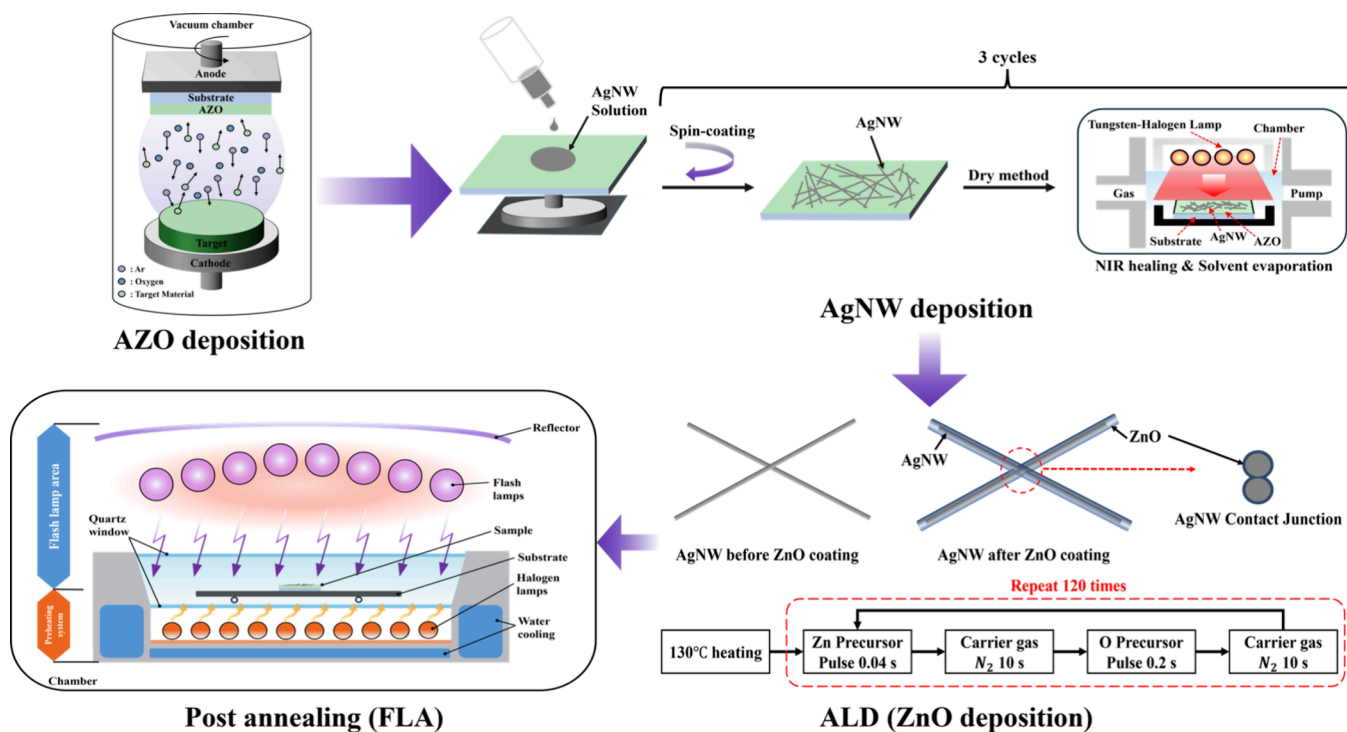


Figure 1. Schematic representation of sample fabrication. From the top to the right: (1) Deposition of AZO film, (2) spin-coating of AgNWs followed by drying using halogen lamps, (3) deposition of ZnO by ALD, and (4) welding of Ag NWs by FLA.

ZnO have been extensively investigated.¹⁰ These three-layer electrodes frequently surpass ITO in electrical conductivity and optical performance at equal total thickness. Various semiconductors including ITO, ZnO, AZO, MoO₃, and SnO₂ have been utilized as top/bottom dielectric layers surrounding the Ag-, Cu-, Al-, and Pt-metallic core.^{11–14} Previous studies further indicate that ZnO is particularly advantageous as a protective layer for AgNWs because Ag⁺ has a larger ionic radius than Zn²⁺ and does not readily diffuse through the ZnO lattice, contributing to improved chemical and thermal stability.¹⁰ In addition, ZnO encapsulation has been experimentally demonstrated to significantly enhance the mechanical durability and adhesion of AgNW networks, maintaining stable electrical performance under repeated bending and preventing structural degradation such as nanowire fragmentation and detachment.¹⁵ These effects are attributed to the conformal coating of ZnO, which embeds the nanowires, improves interfacial adhesion, and suppresses thermally and mechanically induced degradation, as reported in the ZnO/AgNW/ZnO multilayer systems. Consequently, OMO structures are considered to be one of the most promising candidates for next-generation TCFs due to their enhanced adhesion, oxidation resistance, and thermal durability.^{5,16}

The electrical and optical performance of the OMO electrodes strongly depends on the junction quality between individual NWs, oxide film density, and interfacial properties, making post-treatments indispensable for controlling junction welding and improving interface characteristics. Previous work has shown that AgNWs naturally interconnect and form thermally welded junctions upon thermal annealing.⁹ Conventional furnace annealing and RTA rely on heating the entire substrate, resulting in long ramp-up and cooling times and extensive heat transfer through the substrate. These factors often lead to nonuniform heating, interfacial deformation, morphological instability, and issues such as Plateau–Rayleigh breakup, causing nanowire spheroidization, fracture, and increased electrical resistance.^{6,10}

In contrast, flash-lamp annealing (FLA) offers an ultrafast and substrate-friendly alternative. The underlying principle of FLA involves a millisecond-range light pulse generated by xenon lamps. The Xe-lamp emission covers the UV and visible spectral range from 300 to 800 nm. The energy delivered to the sample surface is absorbed by the top layer within the pulse duration causing ultrafast heating while the substrate, unaffected by the thermal treatment, acts as a heat reservoir, ensuring rapid cooling.¹⁷ Because the thermal response and annealing occur on nearly identical time scales, FLA enables

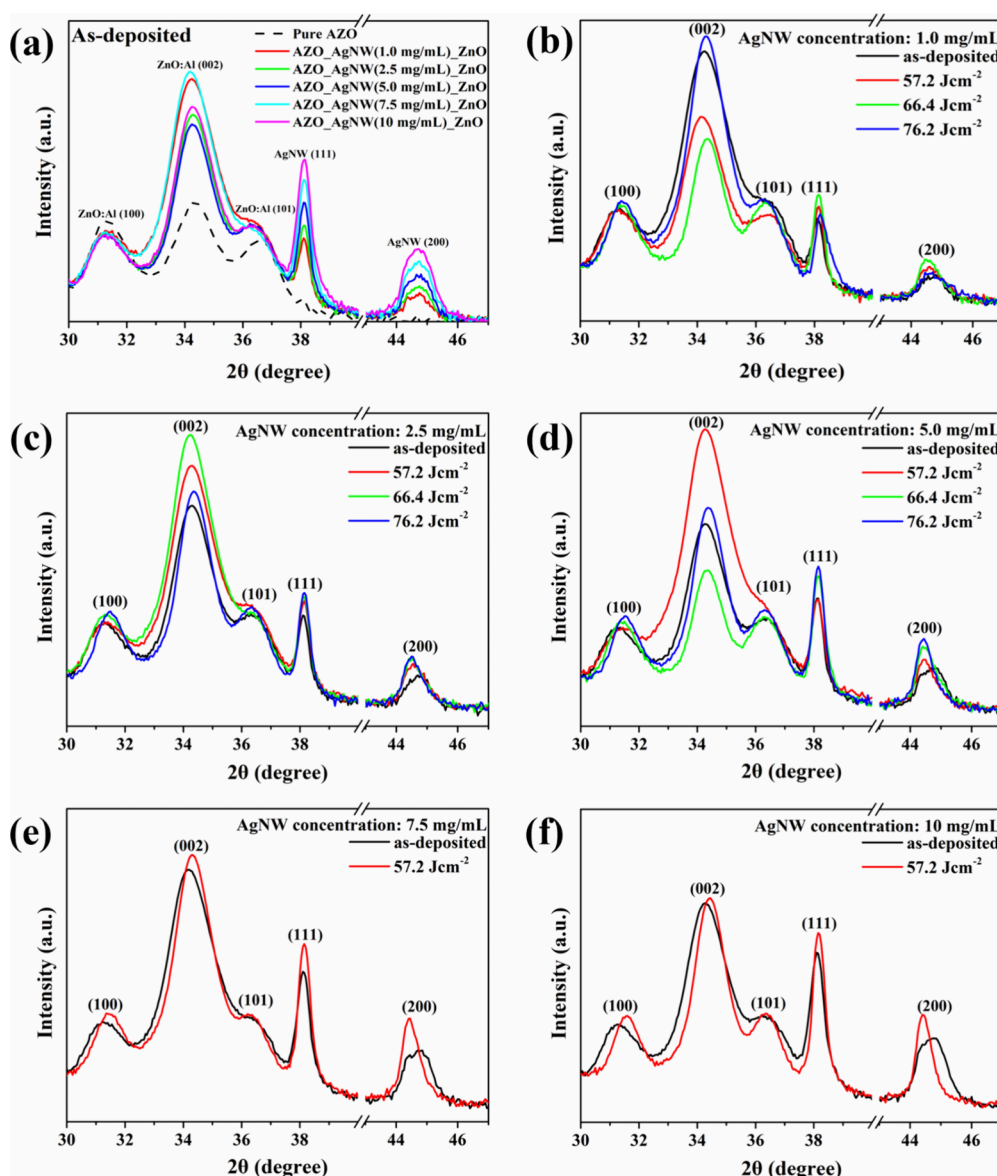


Figure 2. (a) XRD pattern of as-deposited films and (b–f) samples containing different concentrations of AgNWs before and after FLA.

high-temperature effects without damaging the substrate and diffusion of the material between the layers or the NW core and the shell.⁵

However, only a limited number of studies have investigated FLA-based welding process of AgNWs,^{18–20} while, to the best of our knowledge, the influence of FLA process on the OMO structure like AZO/AgNW/ZnO has not been comprehensively clarified.

To systematically compare the performance and processing conditions of previously reported multilayer transparent electrodes, a comparative summary is provided in Table 1.

As shown in Table 1, various annealing approaches, including conventional thermal annealing, laser irradiation, and plasma-based processing, have been applied to OMO and double-layer transparent electrodes.^{9,21–25} Although these methods can achieve comparable figures of merit (FoM), they generally require long processing times. In contrast, flash-lamp annealing (FLA) enables similar performance within an ultrafast millisecond time scale, highlighting a significant advantage in processing efficiency.

In this study, AZO/AgNW/ZnO transparent conductive films are employed as a representative TCF electrode to explore the potential of millisecond range flash-lamp annealing as a very fast postdeposition treatment. Particular attention is paid to how FLA influences AgNW junction welding and the optoelectronic properties of the AZO layer, with the focus on the electrical conductivity and optical transmittance of entire coatings. The structural and optoelectronic investigations demonstrate that the FLA pulse for 23 ms significantly improves the AZO and AgNWs crystallinity and reduces the coating resistivity while the coating transmittance stays above 80%. It is shown that the FLA-treated AZO films containing moderate concentration of Ag-NWs are promising candidates for next-generation transparent conductive films.

2. EXPERIMENTAL SECTION

2.1. Materials and Methods

2.1.1. Film Fabrication. Schematic representation of the sample fabrication is shown in Figure 1. Prior to film deposition, glass substrates ($15 \times 15 \times 0.5 \text{ mm}^3$) were cleaned using an ultrasonic

cleaner sequentially in ethanol, isopropyl alcohol, and deionized water for 10 min each, resulting in a total cleaning time of 30 min. An aluminum-doped zinc oxide (AZO) bottom electrode layer was deposited by DC magnetron sputtering using a ZnO/Al₂O₃ (98/2 wt %) ceramic target. The sputtering process was carried out at a power of 200 W, a substrate temperature of 200 °C, and a working pressure of 15 mTorr, with argon (Ar, 25 sccm) and oxygen (O₂, 3 sccm) as the sputtering gases. The deposition time was 30 min, and presputtering was performed for 5–7 min prior to deposition to stabilize the plasma and remove surface contaminants from the target. The thickness of the deposited AZO layer was approximately 1.2 μm (see Figure S1).

AgNWs with diameters of about 25–35 nm and lengths of 30–60 μm, dispersed in isopropyl alcohol (IPA), were used to form the conductive network. The AgNWs were deposited onto the AZO layer by spin coating, with the coating process repeated three times.²¹ Each spin-coating step was performed at 2500 rpm for 45 s, during which 35 μL of the AgNW dispersion was dropcasted per coating cycle. To remove residual solvent, a near-infrared (NIR) drying process was applied in a top-down configuration at 200 °C for 6 s while maintaining a fixed lamp-to-sample distance of 10 cm. The density of the Ag-NWs on the top of the AZO film was controlled by changing the concentration of Ag NWs in the dispersion. To serve as a capping layer on the AgNW network and protect the nanowires from surface degradation (e.g., sulfidation and oxidation), a zinc oxide (ZnO) layer with a nominal thickness of approximately 10 nm was deposited by atomic layer deposition (ALD). During ALD, the substrate temperature was maintained at 130 °C. One ALD cycle consisted of a Zn precursor pulse of 0.04 s, followed by a nitrogen (N₂) purge for 10 s, an O precursor pulse of 0.2 s, and a subsequent N₂ purge for 10 s. This cycle was repeated 120 times, and N₂ was used as both the carrier and purge gas. ALD was employed to ensure a conformal and uniform ZnO coating over the AgNW network. Using this process, transparent conductive films (TCFs) with sandwich structures containing different AgNW densities were fabricated.

2.1.2. Flash-Lamp Annealing. As a post-treatment process for the TCFs, flash-lamp annealing (FLA) was applied using short pulses of xenon lamps. The samples were irradiated with millisecond-range light pulses, and the energy density was varied between 57.2, 66.4, and 76.2 J cm⁻². All FLA treatments were conducted under a nitrogen (N₂) atmosphere. More details on FLA can be found elsewhere.¹⁹

2.2. Characterization

The crystalline structure of the TCFs was analyzed by X-ray diffraction (XRD) using a D8 Advance X-ray diffractometer (Bruker, Karlsruhe, Germany) with Cu Kα radiation ($\lambda = 1.5406 \text{ \AA}$) and a scintillation counter. The Bragg–Brentano geometry was applied for diffraction angles ranging from 20° to 80°, with a scan rate of 2 °/min. The surface morphology and microstructural features were examined by scanning electron microscopy (SEM), with SEM imaging carried out in SEM mode using a RAITH150 TWO electron-beam lithography system (Raith GmbH) at an acceleration voltage of 2.6–3.0 kV. The thickness of the AZO layer was measured using a surface profilometer (KLA Alpha Step AS-500). A step edge was formed by masking part of the substrate during deposition, and the step height was measured after removal of the mask. The thickness and layer structure were further confirmed by SEM and energy-dispersive spectroscopy (EDS) analysis. The optical properties were characterized by measuring the optical transmittance using a UV–visible–near-infrared (UV–Vis–NIR) spectrophotometer (Solid-Spec-3700 DUV, Shimadzu) in a wavelength range of 300–800 nm, and optical defect characteristics were investigated by photoluminescence (PL) spectroscopy. Samples were excited with a diode-pumped solid state laser operated at 320 nm at room temperature. The PL signal was recorded by a Hamamatsu photomultiplier H7732-10 and a 550 iHR Horiba spectrometer. To increase the signal-to-noise ratio, the laser light was chopped with a mechanical chopper at 25 Hz and the lock-in amplifier was used. The change of the sheet resistance upon postdeposition annealing was

measured at room temperature using a Hall effect measurement system (HMS-9709A, Lake Shore)

3. RESULTS AND DISCUSSION

3.1. Structural Properties

The crystallinity of the samples was analyzed by using X-ray diffraction. Figure 2a shows the X-ray diffraction patterns of as-deposited AZO and AZO/AgNW/ZnO films. The as-deposited AZO film (without AgNWs) shows three peaks at about 31.28°, 34.40°, and 36.72° due to (100), (002), and (101) reflections (according to JCPDS card no. 36-1451), respectively. After Ag NW deposition, two new peaks appeared at 38.10° and 44.78° due to reflections from (111) and (200) of silver (JCPDS card no. 04-0783). Interestingly, after AgNW deposition, the intensity of the (100) reflection from AZO decreases as expected but the intensity of the (002) and (101) reflections increase significantly. The decrease of the signal is expected since the scattering and absorption of the X-ray should increase with adding NWs on the top but the increase of the other reflections cannot be explained solely by this effect. All samples were measured using the same X-ray beam parameters. One explanation can be improved crystallinity caused by thermal treatment during Ag-NWs processing and ZnO capping layer deposition. Moreover, the (002) and (101) reflections shift to smaller angle suggesting some changes in the crystallinity due to deposition and annealing of Ag-NWs, where the annealing corresponds to the FLA process performed after the ZnO capping layer deposition. However, XRD studies conducted on an AZO sample without Ag NWs but annealed at 130 °C for 2 h (ALD process) show no changes in spectrum intensity (see Figure S2), indicating that thermal effects alone are insufficient to explain the observed behavior.

Therefore, additional mechanisms must be considered to account for the observed changes in the peak intensities. The change in peak intensities after AgNW deposition (Figure 2a) is attributed to a redistribution of crystallographic texture driven by modifications in surface energy and interfacial conditions. The deposition of the AgNW solution may alter the surface chemistry and surface energy of the AZO layer due to residual organics or interface modification. As a result, growth along the *c*-axis directions, corresponding to the (002) and (101) planes, becomes more favorable compared to that along the (100) plane. In ZnO-based systems, the (002) plane has the lowest surface free energy and thus preferentially promotes *c*-axis growth, whereas the (100) plane corresponds to a relatively higher surface energy. Therefore, even changes in surface conditions alone can induce a preferential reorientation of the crystallites.

In addition, the introduction of the AgNW network and subsequent processing steps can induce interfacial residual stress within the AZO layer. Such stress is known to strongly influence crystallographic texture evolution in oxide thin films, leading to a redistribution of the preferred orientations. This interpretation is also consistent with the peak shifts observed in the XRD patterns, indicating the development of strain within the film.

Furthermore, it is also possible that, during the AgNW solvent evaporation process under ambient conditions, oxygen diffuses into the AZO layer and promotes the *c*-axis-oriented growth of ZnO. Such an oxygen-rich environment can enhance

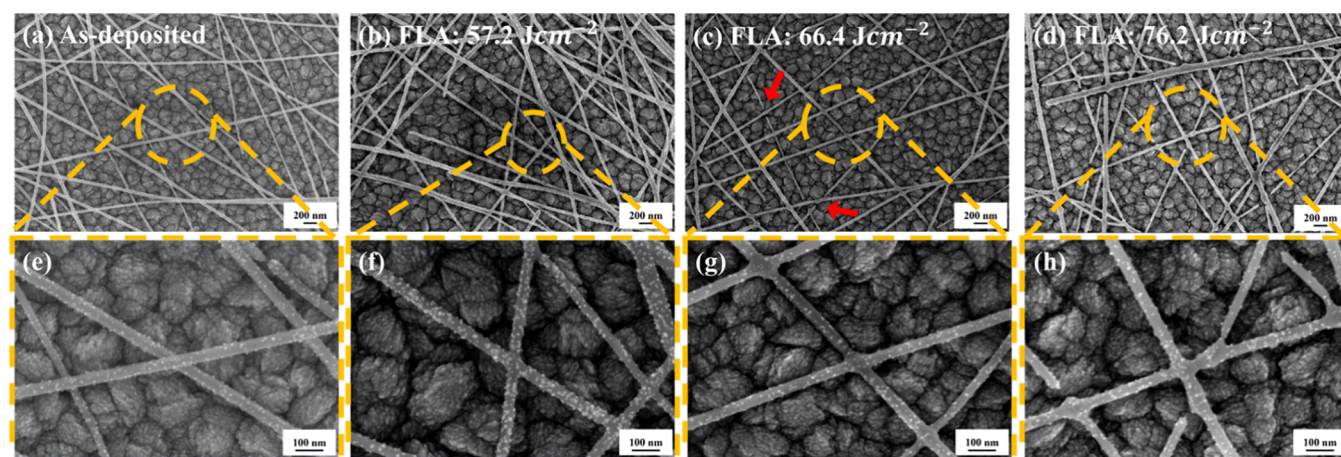


Figure 3. SEM images of AgNW networks with a fixed concentration of 2.5 mg/mL under different flash-lamp annealing (FLA) conditions: (a) as-deposited, (b) 57.2 J cm⁻², (c) 66.4 J cm⁻², and (d) 76.2 J cm⁻². (e–h) Corresponding magnified SEM images of panels (a–d), respectively, showing the morphology of AgNW contact junctions.

the (002) and (101) orientations while suppressing the (100) orientation.

Therefore, the observed changes in peak intensities can be consistently explained as the combined result of surface-energy-driven texture evolution, oxygen-mediated crystallization, and residual stress effects induced by the introduction of the AgNW network and subsequent processing steps.

Figure 2b–f shows the data obtained from samples after annealing. The diffraction patterns from as-deposited samples are added for comparison. Again, the signal intensity obtained from AZO films deviates from expectations. In particular, the (100) and (002) peaks exhibit a slight shift toward higher 2θ angles with increasing FLA power density, suggesting the development of compressive residual strain within the AZO layer, likely associated with interfacial interaction.

The scattering of the signal intensity remains difficult to interpret and is likely associated with residual stress and structural disorder in the multilayer system. However, the full width at half-maximum (fwhm) decreases for all samples after FLA treatment, indicating improved crystallinity and an increase in crystallite size of the AZO layer. This improvement is expected to reduce carrier scattering, thereby contributing to a decrease in the resistivity.

Using Scherrer eq 1, we have calculated that the average crystal size of AZO increases from approximately 11 ± 1 nm in the as-deposited sample to about 14 ± 1 nm after annealing (the fitting results are summarized in Table 1 and Figure S3).

The Scherrer equation used is given below:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

D refers to the average crystallite size, and K is the shape factor determined by the crystallite shape, for which a value of 0.9 was used. λ represents the wavelength of the X-ray source (Cu $K\alpha$), β is the full width at half-maximum (fwhm, in radians) of the diffraction peak corrected for instrumental broadening, and θ is the Bragg diffraction angle.

With increasing FLA power density, the diffraction intensities of the Ag (111) and Ag (200) peaks increase, as well. In addition, the Ag (200) diffraction peak exhibits a noticeable shift toward lower 2θ angles. According to Bragg's law ($n\lambda = 2d \sin \theta$), a decrease in the diffraction angle corresponds to an increase in the interplanar spacing (d -

spacing). Therefore, the observed low-angle shift of the Ag (200) reflection indicates an expansion of the average d -spacing, which can be attributed to the development of residual tensile strain in the AgNW network during the FLA process.²⁶ Using Scherrer eq 1, we calculate the average diameter of Ag NWs to be about 26 ± 1 nm, which is according to the information provided by supplier. The fitting results related to Ag NWs are summarized in Supporting Information Table 2 and Figure S4.

It should be noted that samples with AgNW concentrations of 7.5 and 10 mg/mL could not be annealed at an energy density higher than 57.2 J cm⁻², due to the melting of AgNWs. As the AgNW concentration increases, the metallic nanowire network becomes denser, resulting in significantly enhanced optical absorption during FLA irradiation. Under these FLA conditions, excessive localized heating occurs, resulting in junction disconnection or structural damage of the AgNW network. Based on these findings, it can be concluded that both the AgNW concentration and the FLA power density are critical factors. In this study, the variation in fwhm was used to compare and analyze the changes in crystallite size under different annealing conditions.

3.2. Morphological Properties

In the as-deposited film, AgNWs form an interconnected random network on the AZO layer, namely, a percolated network. The intrinsic characteristics of AgNWs, such as their diameter and length, can contribute to a reduction in contact resistance, and this contact resistance constitutes a major component of the overall sheet resistance of the film.^{8,27} In addition, the ZnO capping layer can decrease the sheet resistance by filling the voids between AgNWs and tightening the contact at the AgNW junctions. Moreover, the ZnO layer can act as an encapsulation layer, protecting the nanowires from oxidation and thereby improving their mechanical and thermal stabilities.¹⁵ These considerations indicate that the high resistance at nanowire junctions rather than the resistance of the nanowire themselves is the primary limiting factor. Accordingly, SEM analysis is employed to focus on the random AgNW network and AgNW junctions.

Figure 3 presents SEM images of AgNW films with a fixed concentration of 2.5 mg/mL as a function of the FLA power density. After FLA treatment, numerous nanowire junctions

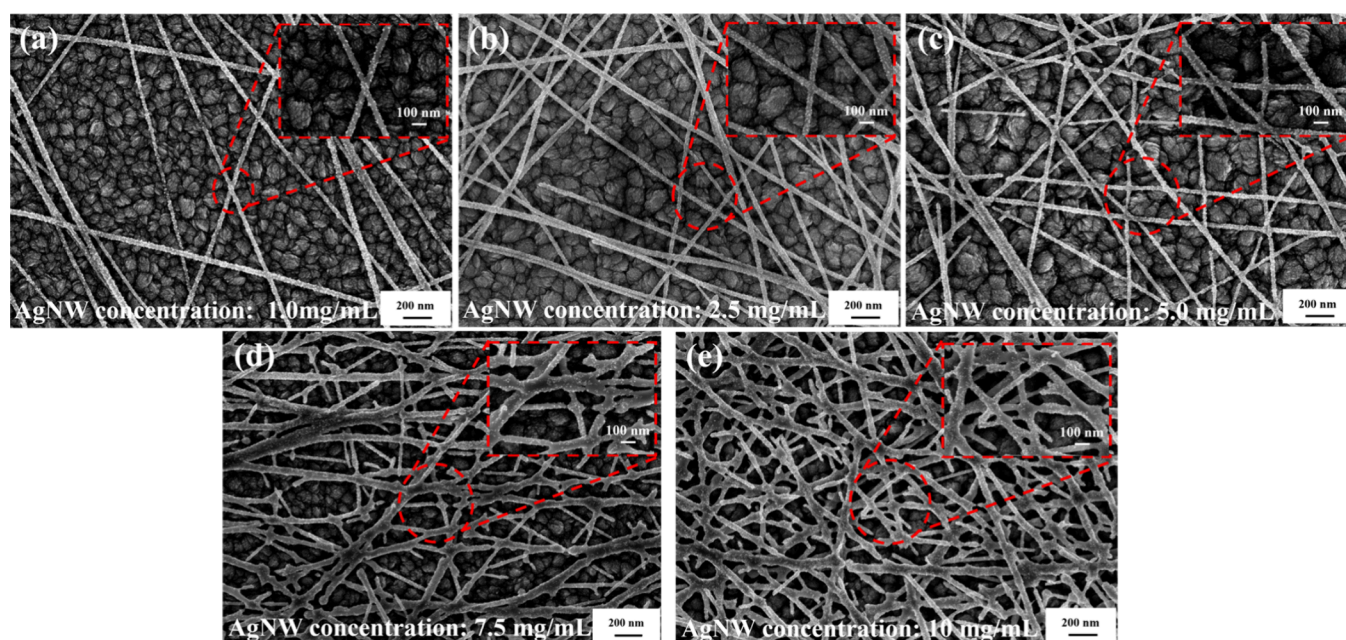


Figure 4. SEM images of AgNW networks with different AgNW concentrations of (a) 1.0, (b) 2.5, (c) 5.0, (d) 7.5, and (e) 10 mg/mL after FLA treatment at a fixed energy density of 57.2 J cm^{-2} . The dashed boxes indicate the regions shown in the magnified insets.

are thermally welded, forming improved internanowire contacts. From a morphological perspective, such junction welding suggests a reduction in internanowire contact resistance and indicates enhanced network connectivity.

At a AgNW concentration of 2.5 mg/mL, SEM images obtained at an FLA power density of 66.4 J cm^{-2} further reveal improved welding at nanowire junctions and overlapping wire regions. This observation indicates that localized melting occurred, thereby strengthening the nanowire bonding. However, even at 66.4 J cm^{-2} , partial fracture of some nanowires is shown (Figure 3 c, arrow), suggesting that this energy density is close to the damage threshold for the AgNW network at this concentration.

At the highest FLA power density of 76.2 J cm^{-2} , pronounced morphological changes in the nanowires are clearly evident. The Ag network exhibits features indicative of melting followed by coalescence, and several nanowires become partially sintered and discontinuous. These features are consistent with the occurrence of Plateau–Rayleigh instability.^{6,10} Such morphological degradation results in the loss of percolation pathways, which explains the degradation of electrical performance under high FLA conditions, as discussed later.

In addition to the fixed-concentration study, when the FLA power density was fixed at 57.2 J cm^{-2} and applied to AgNW films with concentrations of 1.0, 2.5, 5.0, 7.5, and 10 mg/mL, progressive damage to the AgNW network was observed for concentrations above 5.0 mg/mL. These morphological changes are shown in Figure 4.

To summarize, the morphological analyses demonstrate that FLA can serve as an effective means to induce nanowire welding within the AZO/AgNW/ZnO films. However, these improvements are achievable only within an optimized processing window; beyond a certain AgNW density or FLA energy input, morphological instabilities arise that negate the beneficial effects. We inferred that junction resistance could be reduced when the FLA power was 57.2 J cm^{-2} and the AgNW concentration was below 2.5 mg/mL.

3.3. Optical Properties

3.3.1. Transmittance Measurements. The optical properties of TCF made of AZO/AgNW/ZnO were analyzed by comparing the changes in optical transmittance and band structure before and after FLA treatment. In the as-deposited state, the transmittance in the visible region decreases with increasing AgNW concentration, and a very low transmittance is observed at AgNW concentrations higher than 7.5 mg/mL (see Figure 5). Figure 5b–f shows the optical transmittance spectra of AZO/AgNW/ZnO TCFs with AgNW concentrations of 1.0, 2.5, 5.0, 7.5, and 10 mg/mL, respectively. Each panel compares the as-deposited sample to FLA-treated samples at different energy densities (57.2 , 66.4 , and 76.2 J cm^{-2}). The insets in each panel represent the corresponding Tauc plots used to extract the optical band gap. After FLA treatment, the transmittance shows a slight increase with increasing FLA power density to the threshold value corresponding to the melting or degradation of AgNWs. The threshold flash energy density is different for samples with different AgNW concentration due to enhanced light absorption with increasing the AgNW concentration. At an energy density of 57.2 J cm^{-2} , the average transmittance in a wavelength range of 550–700 nm varies from 74.17% to 42.04% depending on the AgNW concentration. In general, when AgNW networks possess a discontinuous structure, light scattering and absorption by nanowires and metallic clusters are enhanced, whereas as the Ag network becomes closer to a continuous structure (continuous film or continuous percolation network) through FLA treatment, optical absorption tends to decrease.⁵ Therefore, it can be inferred that FLA improves the morphological continuity of the AgNW network, thereby mitigating scattering losses and consequently increasing the overall transmittance. To further verify the influence of postgrown annealing on the optical band gap (E_g) of AZO, we applied the Tauc plots (see insets in Figure 5). The Tauc plot is a widely used approach to interpret shifts in the absorption edge and changes in band structure through the

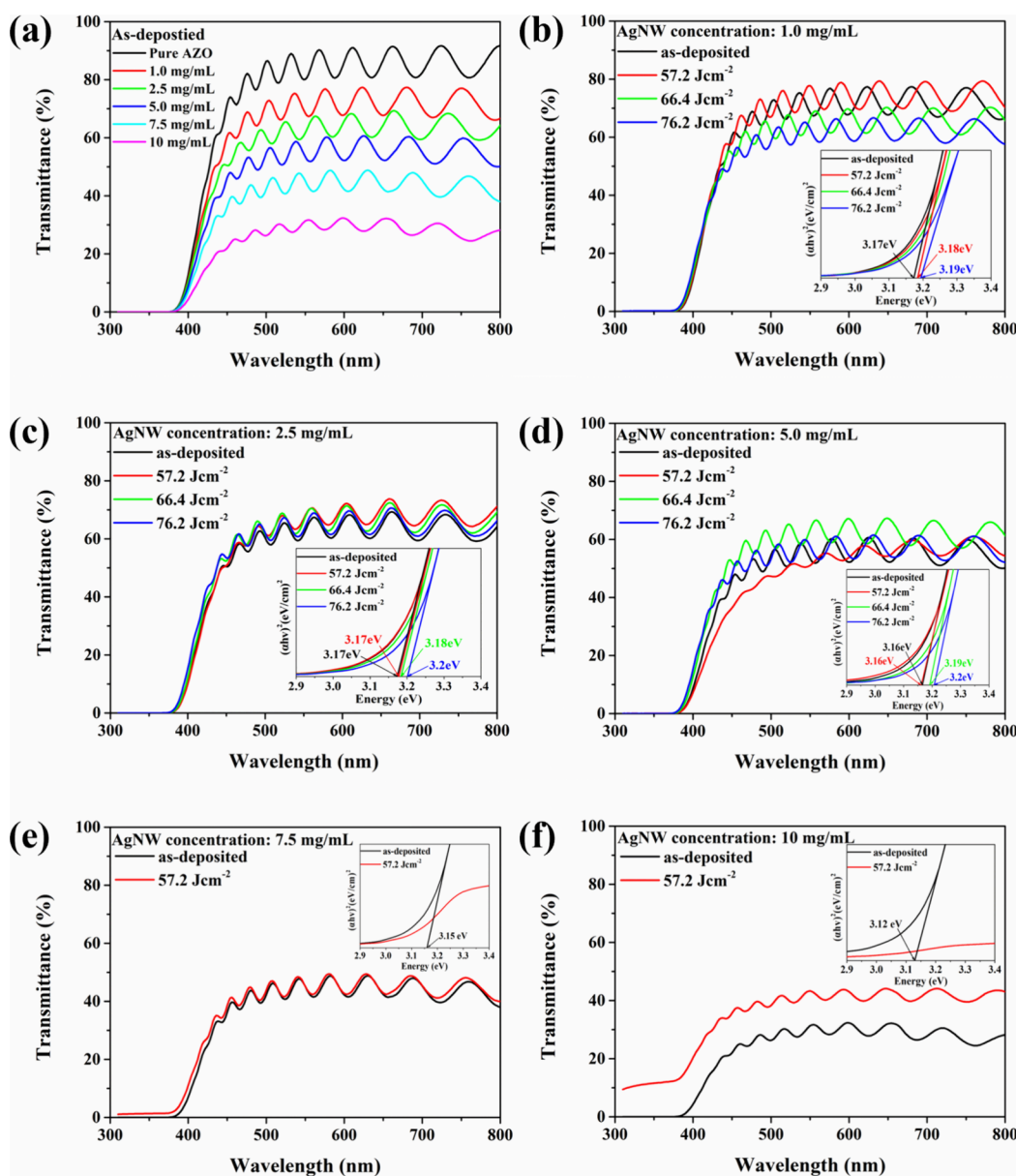


Figure 5. Transmittance as a function wavelength for (a) as-deposited samples with different AgNWs concentration and (b–f) samples with AgNW concentrations of 1.0, 2.5, 5.0, 7.5, and 10 mg/mL, respectively, under different FLA energy densities (as-deposited, 57.2, 66.4, and 76.2 J cm^{−2}). The insets show the estimated band gap change after annealing estimated from the Tauc Plot.

relationship between the absorption coefficient and photon energy. This relationship can be expressed as follows:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (2)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, E_g is the optical band gap, and A is a material-dependent constant. This analysis has been widely applied, particularly for AZO-based thin films, to effectively explain the blue shift of the absorption edge and the widening of the optical band gap induced by an increase in carrier concentration.²⁸ In this study, E_g was extracted by plotting the relationship between $(\alpha h\nu)^2$ and $h\nu$ and extrapolating the linear region to its intersection with the x -axis. Through this approach, we aimed to confirm that the increase in transmittance observed after FLA treatment is closely related not only to reduced scattering losses but also to band structure modulation associated with

improved crystallinity of AZO and changes in carrier concentration.

Before FLA treatment, the E_g of AZO is about 3.17 eV. After FLA treatment, up to an AgNW concentration of 5.0 mg/mL, the optical band gap increases from approximately 3.17 to 3.2 eV with increasing FLA power density. The increase in the optical band gap can be explained by two main factors. First, FLA treatment reduces the density of deep-level defect states such as oxygen vacancies, thereby decreasing sub-bandgap absorption and sharpening the absorption edge, which leads to an increase in E_g of AZO. These oxygen vacancies and deep-level defect states will be discussed in more detail through photoluminescence analysis presented later. Second, the observed blue shift can be attributed to the Burstein–Moss (BM) effect.²⁹ In heavily doped semiconductors, an increase in the free electron concentration leads to the occupation of low-energy states at the bottom of the conduction band. According

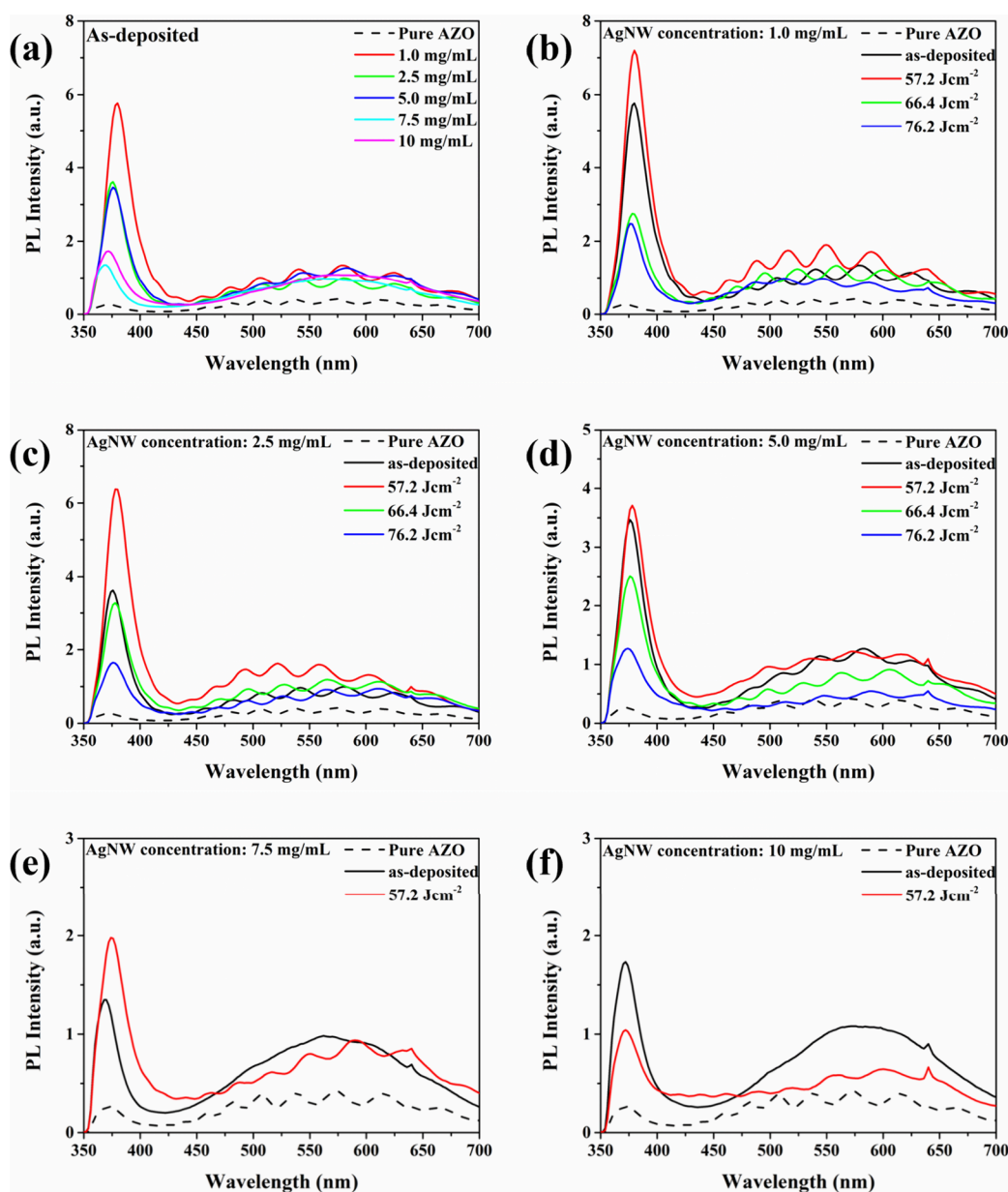


Figure 6. Photoluminescence spectra of the as-deposited samples and samples annealed by FLA. (a) The PL from as-deposited samples with different AgNW concentration. (b–f) The change of the PL spectra caused by flash lamp annealing for samples coated with different AgNW concentrations.

to the Pauli exclusion principle, optical transitions are allowed only to higher-energy states in the conduction band, resulting in an apparent increase in the optical band gap.³⁰ This band gap widening induced by FLA is consistent with enhanced charge transport and can be attributed to conduction band filling associated with an increased carrier concentration. These results indicate that the modification of the optical band structure is closely correlated with the electrical state of the composite films rather than being purely an optical effect.

3.3.2. Photoluminescence (PL) Spectroscopy. Figure 6 presents the room-temperature photoluminescence (PL) spectra of AZO/AgNW/ZnO films as a function of AgNW concentration and FLA conditions, providing detailed information about defect states and optical quality. All samples exhibit a sharp near-band-edge (NBE) ultraviolet emission centered at approximately 376 nm (~ 3.3 eV), which is characteristic of the radiative recombination of free excitons in

ZnO.³¹ Additionally, a relatively weak and broad green-red emission band in the range of ~ 2.4 – 2.5 eV is observed in most samples. This green-red emission is generally attributed to deep-level emission (DLE) associated with oxygen-related point defects in ZnO, such as oxygen vacancies (V_o) and oxygen interstitials (O_i).^{29, 31} The PL characteristics prior to FLA treatment were first examined as a function of AgNW concentration. At an AgNW concentration of 1.0 mg/mL, the intensity of the NBE emission is significantly enhanced, whereas for higher AgNW concentrations, the NBE intensity gradually decreases. A noticeable blue shift of the NBE peak is also observed. This behavior can be attributed to the Burstein–Moss (band-filling) effect, in which electron injection from Ag nanostructures into ZnO increases the free carrier concentration and raises the Fermi level, effectively widening the optical band gap.³²

Table 2. Summary of the Electrical and Optical Properties of AZO/AgNWs/ZnO Coatings Annealed by FLA for 23 ms at Different Energy Densities

AgNW concentration	annealing parameter	resistivity ($\times 10^{-3} \Omega \text{cm}$)	sheet resistance (Ω/sq)	average transmittance (550 nm–700 nm)	transmittance (550 nm)	FoM ($\times 10^{-3} \Omega^{-1}$)
1.0 mg/mL	as-deposited	6.72	55.45	73.39%	72.12%	0.69
	FLA 57.2 J cm^{-2}	1.27	10.48	74.17%	76.08%	6.20
	FLA 66.4 J cm^{-2}	1.63	13.45	65.56%	62.39%	0.66
	FLA 76.2 J cm^{-2}	3.03	25.00	62.89%	65.15%	0.55
2.5 mg/mL	as-deposited	6.07	50.08	63.49%	61.68%	0.16
	FLA 57.2 J cm^{-2}	1.11	9.16	68.49%	66.99%	1.99
	FLA 66.4 J cm^{-2}	3.96	32.67	66.26%	66.21%	0.49
	FLA 76.2 J cm^{-2}	1.81	14.94	64.86%	63.21%	0.68
5.0 mg/mL	as-deposited	0.72	5.94	56.24%	54.58%	0.40
	FLA 57.2 J cm^{-2}	0.75	6.19	55.94%	50.72%	0.18
	FLA 66.4 J cm^{-2}	1.15	9.49	62.76%	61.96%	0.88
	FLA 76.2 J cm^{-2}	1.09	8.99	57.73%	58.76%	0.54
7.5 mg/mL	as-deposited	0.33	2.72	45.04%	46.18%	0.16
	FLA 57.2 J cm^{-2}	0.44	3.63	45.83%	46.30%	0.13
	FLA 66.4 J cm^{-2}					
	FLA 76.2 J cm^{-2}					
10 mg/mL	as-deposited	0.21	1.73	30.31%	30.86%	0.004
	FLA 57.2 J cm^{-2}	0.91	7.51	42.04%	43.23%	0.03
	FLA 66.4 J cm^{-2}					
	FLA 76.2 J cm^{-2}					

Simultaneously, the presence of AgNWs influences defect-related emission pathways. As the AgNW concentration increases, the visible deep-level emission in the green region remains at a comparable overall level. However, interference-induced oscillations become progressively broader and smoother. In this study, such spectral evolution is interpreted as being related to defect states associated with oxygen interstitials rather than as a simple change in defect density. Accordingly, before FLA treatment, variations in the PL spectra with increasing AgNW concentration are more prominently reflected in the relative spectral shape of the NBE emission than in the green-red deep-level emission. Therefore, the PL analysis primarily focuses on changes in the NBE characteristics, including peak position and spectral evolution, rather than on the absolute intensity variation of the DLE.

The blueshift of the NBE emission can be explained by the increase in free electron concentration induced by the introduction of metallic nanostructures through the Burstein–Moss effect, while the green-red emission has been widely reported to originate from defect levels in ZnO, particularly those associated with oxygen vacancies.^{29,33} The increase in AgNW concentration is more likely to modify the distribution of charge recombination pathways involving defect states rather than directly eliminating the defects themselves, thereby affecting the overall PL spectral shape.^{31,33}

Previous studies have reported that, in ZnO incorporating metallic nanostructures, the NBE emission intensity initially increases with increasing dopant concentration, reaches a maximum at an optimal concentration, and subsequently decreases at higher concentrations, whereas defect-related emission tends to continuously decrease with increasing nanostructure content.³⁴ This behavior indicates that plasmon–exciton coupling between ZnO and AgNWs can be optimized at a specific concentration, leading to the most effective enhancement of the NBE emission.³⁴ We have to consider the decrease of the transmittance of the layer with

increasing AgNW concentration that may also affect the excitation and emission process.

The effect of FLA treatment on the PL intensity was then analyzed. For FLA-treated samples, the NBE peak intensity increases relative to the as-deposited state up to a AgNW concentration of 7.5 mg/mL when processed at an FLA energy density of 57.2 J cm^{-2} . This observation suggests that millisecond-scale rapid thermal processing stabilizes the junctions between AgNWs while minimizing damage to the substrate, which is consistent with the SEM observations discussed earlier. The influence of this junction stabilization on the electrical properties will be discussed in detail in a subsequent section.

In contrast, at FLA power densities exceeding 57.2 J cm^{-2} , the NBE emission intensity becomes lower than that of the as-deposited samples. This behavior can be attributed to lattice distortion induced by local overheating and inhomogeneous thermal expansion within the lattice. Such structural instability is likely to enhance nonradiative recombination pathways, resulting in the observed quenching of the NBE emission.

Regarding defect-related emission after FLA treatment, the green emission band around ~530 nm exhibits change in spectral shape and relative contribution depending on the FLA condition while remaining at an overall level comparable to that of the as-deposited samples. Unlike previous reports in which postannealing under oxygen-rich conditions led to a pronounced reduction in green PL intensity in AZO films,³⁵ the present FLA conditions suggest that the green emission is not uniformly suppressed. Instead, the observed behavior is more appropriately interpreted in terms of a redistribution of recombination pathways.³⁴

In other words, rather than assuming complete elimination of oxygen-vacancy-related defect states during the FLA process, the relative contributions of radiative recombination via defect levels and band-edge recombination appear to vary with the FLA power density. From this perspective, the persistence of the green DLE band after FLA indicates that

changes in the NBE distribution provide a more meaningful indicator than the absolute intensity of the DLE.

It is well-known that variations in the density of oxygen-vacancy-related defects can influence both the electrical carrier concentration and the structural lattice parameters. According to Vanheusden et al., a strong correlation exists between the V_O density and the electron carrier concentration in ZnO, and suppression of oxygen vacancies under oxidizing conditions leads to a reduction in n-type carrier concentration.³⁵ In the present study, samples were annealed in a nitrogen atmosphere; therefore, the reduction of oxygen vacancies concentration is not expected.

Meanwhile, the NBE peak, which exhibited a blue shift prior to FLA due to the band-filling effect at high doping levels, shows a slight red shift after FLA treatment. This shift can be increased resistivity, i.e., reduced carrier concentration.

For example, one study reported that green photoluminescence can arise from donor–acceptor pair recombination between donor-like V_O and acceptor-like V_{Zn} defect centers.³² Such findings indicate that green emission in ZnO can originate from various combinations of intrinsic defects. However, considering the AZO/AgNW/ZnO structure and the FLA processing conditions employed in this study, the persistence of green emission after FLA treatment is not fully consistent with a simple defect-suppression model based solely on the removal of oxygen vacancies.

Overall, the PL characteristics as a function of AgNW concentration and FLA treatment reflect not a simple creation or annihilation of specific defects but rather a redistribution of recombination pathways governed by changes in the thermal and stress states of the AgNW network and the defect landscape within ZnO. These effects collectively influence both the optical and electrical properties of the transparent conductive films. The PL results are in good agreement with the structural and electrical analyses, indicating that under appropriate FLA conditions (e.g., 57.2 J cm^{-2}), improvements in electrical performance can be achieved without inducing structural degradation relative to the as-deposited samples, as will be demonstrated in the subsequent electrical property analysis.

3.4. Electrical Properties

The electrical properties of the AZO/AgNW/ZnO transparent conductive films strongly depend on both the AgNW concentration and the flash-lamp annealing (FLA) energy density, as summarized in Table 2 and Figure 7. In the as-deposited state, the resistivity (ρ) decreases sharply with increasing AgNW concentration, dropping from $6.72 \times 10^{-3} \text{ } \Omega\text{cm}$ at 1.0 mg/mL to $0.21 \times 10^{-3} \text{ } \Omega\text{cm}$ at 10 mg/mL , corresponding to an approximately 32-fold enhancement in conductivity. This concentration-dependent improvement in conductivity is in good agreement with the percolation dominated transport mechanism commonly observed in AgNW-based transparent electrodes and OMO composite structures.

FLA treatment further modifies the electrical response, exhibiting a clear optimum at an intermediate energy density. At 57.2 J cm^{-2} , low-density AgNW films show the most pronounced improvement compared with the as-deposited state. For instance, at 1.0 mg/mL the resistivity decreases from 6.72 to $1.27 \times 10^{-3} \text{ } \Omega\text{cm}$, corresponding to an approximately 5-fold reduction, and a similar level of improvement is observed at 2.5 mg/mL (6.07 to $1.11 \times 10^{-3} \text{ } \Omega\text{cm}$). This

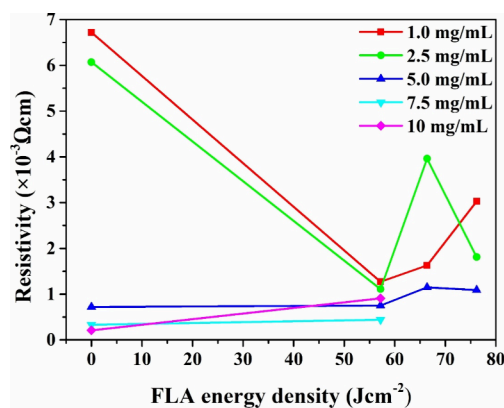


Figure 7. Resistivity as a function of FLA energy density for samples containing different AgNW concentrations.

behavior can be attributed to the millisecond pulsed process, which improves the contacts between nanowires and reduces the contact resistance without imposing long-term thermal exposure on the substrate. Indeed, significant conductivity enhancement under optimized flash conditions has also been reported in similar TCF systems processed by xenon FLA.^{5,36}

When the FLA energy density exceeds 57.2 J cm^{-2} and increases to 66.4 and 76.2 J cm^{-2} , the resistivity shows a reversal and increases again, particularly at higher AgNW concentrations. This can be explained by the fact that dense AgNW networks absorb more incident energy during the flash, leading to intensified localized heating and the formation of nonuniform electrical pathways. Under such high-energy conditions, the electrical continuity can be degraded due to thermally induced morphological instability of the metallic nanowires. In this study, the samples with AgNW concentrations of 2.5 and 5.0 mg/mL processed at 76.2 J cm^{-2} exhibit higher resistivity values, which indicate partial disruption of the conductive pathways. At even higher AgNW concentrations (7.5 and 10 mg/mL), the conductive network fails to maintain stable electrical continuity under high-energy FLA conditions, as confirmed by morphological analysis using SEM (see Figure 4), and therefore, resistivity measurements could not be obtained.

The sheet resistance (R_s) follows the same overall trend as the resistivity. After FLA treatment at 57.2 J cm^{-2} , the R_s of the 1.0 – 2.5 mg/mL AgNW films decreases sharply, reaching $10.48 \text{ } \Omega/\text{sq}$ for 1.0 mg mL^{-1} and $9.16 \text{ } \Omega/\text{sq}$ for 2.5 mg/mL . In contrast, when the FLA energy exceeds the critical condition, R_s increases again. For example, at 1.0 mg/mL , R_s increases from 10.48 to $25.00 \text{ } \Omega/\text{sq}$ as the FLA power density increases, indicating that excessive flash energy deteriorates the AgNW network and electrical uniformity.

Because transparent conductive films require a balance between electrical and optical performance, the figure of merit (FoM) was evaluated using Haacke's definition, $\Phi_{\text{TC}} = T^{10}/R_s$ (at 550 nm). In this data set, the highest FoM is achieved for the 1.0 mg/mL sample processed at 57.2 J cm^{-2} , yielding an FoM of $6.2 \times 10^{-3} \text{ } \Omega^{-1}$ based on a transmittance of 76.08% at 550 nm and $R_s = 10.48 \text{ } \Omega/\text{sq}$. As the AgNW concentration increases further, the FoM decreases because the penalty from reduced transmittance becomes dominant even when the resistivity remains low (e.g., at 10 mg/mL). This trend is consistent with previous OMO-related studies, where the maximum FoM generally appears at an intermediate or

optimized metal (or nanowire) loading that minimizes R_s while maintaining high visible transmittance.

For reference, optimized OMO trilayer structures such as $\text{SnO}_2/\text{Ag}/\text{SnO}_2$ have reported maximum FoM values on the order of $6.0 \times 10^{-2} \Omega^{-1}$ through precise control of metal thickness and oxide design, demonstrating that the FoM is highly sensitive to optical losses in the metal containing layer.¹² This represents another comparable approach for maximizing the electrical and optical performance in a TCF architecture.

Overall, the electrical results clearly indicate that, in the as-deposited state, the AgNW concentration primarily governs the baseline conductivity that an FLA condition of 57.2 J cm^{-2} provides an effective processing window that significantly improves ρ and R_s in the low AgNW concentration regime and that excessive flash energy induces electrical instability particularly in high-density AgNW networks, leading to increased resistivity, elevated sheet resistance, and reduced FoM.

4. CONCLUSIONS

In summary, the study demonstrates that AZO/AgNW/ZnO composite transparent electrodes processed by flash-lamp annealing can simultaneously achieve improved electrical and optical properties through process optimization. In particular, it clearly shows that process design considering the interplay between the AgNW concentration and FLA energy density is a key factor in realizing high-performance transparent electrodes. The lowest resistivity is obtained for samples annealed at 57.2 J cm^{-2} and decreases with increasing the AgNW concentration. The best value of FoM is achieved for a sample containing 1.0 mg/mL with an average transmittance of 76% at 550 nm. These findings highlight that the combined optimization of flash-lamp annealing condition and AgNW concentration is essential for achieving high-performance AZO/AgNW/ZnO transparent conductive films.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsanm.6c00278>.

Additional XRD analysis and supporting results, including cross-sectional SEM images and EDS analysis for AZO thickness verification (Figure S1), reference XRD comparison of AZO before and after thermal treatment (Figure S2), fwhm variation and Scherrer crystallite-size estimation for AZO (002) (Figure S3 and Table S1), and Ag (111)-based fwhm analysis for AgNW diameter estimation (Figure S4 and Table S2) (PDF)

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Notes

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