

RESEARCH ARTICLE

Solution-Based Ultraviolet Photodiode with ZnO Nanoparticle/Poly(3,4-Ethylenedioxythiophene) Polystyrene Sulfonate Heterojunction and Negative Capacitance

Jin Li^{1,2,3†}, Xin Chang^{1†}, Shunpu Li⁴, Yi Zhang³, and Daping Chu^{1,5*}

¹Centre for Photonic Devices and Sensors, University of Cambridge, Cambridge CB3 0FA, UK. ²School of Instrumentation and Optoelectronic Engineering, Beihang University, Beijing 100191, China. ³National Institute of Extremely Weak Magnetic Field Infrastructure, Hangzhou, China. ⁴College of New Materials and New Energies, Shenzhen Technology University, Shenzhen 518118, China. ⁵Cambridge University Nanjing Centre of Technology and Innovation, Jiangbei New Area, Nanjing, China.

*Address correspondence to: dpc31@cam.ac.uk

†These authors contributed equally to this work.

Ultraviolet (UV) detectors have been widely applied to extensive fields, such as photoelectric microsensors, UV imaging, optical communications, and biological detection. The demand for high-performance UV detectors with low cost and easy fabrication drives the development of novel materials and structures such as wide-bandgap semiconductors, polymers, photodiodes, and phototransistors. Here, we demonstrate a new UV photodiode implemented using solution-processing-based zinc oxide (ZnO) nanoparticles (NPs) and a poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) heterojunction. Moreover, the fabrication process is simple and suitable for large-scale production. First, solution-based films of ZnO NPs and PEDOT:PSS were prepared, and their optical and electrical properties were individually characterized. Strong optical absorption by ZnO NPs for wavelengths below 400 nm was experimentally observed, while absorption by PEDOT:PSS in the same wavelength range is trivial. The ZnO-NPs-PEDOT:PSS heterojunction was then characterized for its electrical rectification behavior and photoresponse, from which a potential barrier height of 0.64 eV and UV current gain of 91.2 were determined. The resistor–capacitor time constant was also calculated to be 946.46 ms at zero bias, and its dependence on reverse bias voltage was determined. Finally, negative capacitance was first observed in this kind of heterojunctions under forwarding bias, which was further investigated regarding a threshold effect and transition frequency. This new type of UV photodiodes based on ZnO-NPs-PEDOT:PSS heterojunctions is highly promising for high-resolution applications, such as ZnO NPs-based spatial light modulators (SLMs), where the ascendant properties of the heterojunction can be fully utilized to achieve the high-resolution capability of optically addressed SLMs.

Introduction

The development of ultraviolet (UV) detectors has been extended to various fields from academia and industry because of their advantages exhibited in wide fields including astronomy, biology, and defense. Over the past decade, various types of materials and structures have been proposed by researchers, such as thin-film photodetectors using wide-bandgap semiconductors (GaN and SiC) [1,2], Schottky metal–insulator–semiconductor-type photodiodes using an InGaN thin film with a low leakage current of 10^{-7} to 10^{-8} A [3], and p-i-n photodiodes yielding a high spectral responsivity of 0.24 A/W under the bias of -3.0 V [4]. Additionally, one-dimensional ZnO nanowires with a high internal gain of $\sim 10^8$ [5] and a solution-processed ZnO nanoparticle (NP) photoconductor with a large dark resistance of more than 10^{12} Ω and a slow fall time exceeding 2 min have been explored [6].

Nonetheless, achieving high-performance UV detectors invariably involves intricate and costly fabrication techniques, including epitaxy and chemical vapor deposition [7,8]. Therefore, the development of low-cost UV detectors with a simple fabrication process is significant. In this paper, a UV-sensitive heterojunction was demonstrated using solution-based ZnO NPs and poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS).

Nakano et al. [9,10] initially demonstrated the Schottky behavior using the experimentally prepared contact film using ZnO NPs and PEDOT:PSS in 2007 and 2008, employing hydrothermally grown single-crystal ZnO and attaining a remarkable on/off current ratio of 10^7 . Alternative fabrication techniques have been documented, including the ultrasonically assisted chemical vapor deposition method [11] and the radio frequency sputtering method [12]. These methods are intrinsically expensive. More importantly, the photodiode formed using

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PEDOT:PSS and single-crystal ZnO or amorphous ZnO suffer from limited resolution due to lateral charge diffusion [13], rendering them unsuitable for high-resolution photoelectric device applications, such as spatial light modulators (SLMs) with optically addressed function based on liquid crystal materials [14,15]. The optically addressed SLMs' resolution limit can be improved by using the advantages of photoelectric properties (i.e., high photocurrent on-off ratio) of ZnO NPs [16].

In this paper, a new ZnO-NPs-PEDOT:PSS heterojunction based on a solution approach was successfully fabricated by spin coating, characterized by its low material cost and simplicity in the fabrication process. The ZnO has a wide bandgap of 3.35 eV and an eco-friendly nature; thus, it is suitable for UV sensing. Additionally, PEDOT:PSS, a widely adopted organic p-type conductor, has found extensive applications in solar cells, light-emitting diodes, and flexible electronics [17,18]. Notably, its weak optical absorption in the visible range enhances its suitability for UV detectors. Negative capacitance behavior was analyzed and discussed, which can enhance the detection capability of heterojunctions, increase the photocurrent of devices, and enhance the optoelectronic response characteristics of devices. Methods and Experiments provides details on the fabrication process and experimental methods. The subsequent sections introduce the material characterization of ZnO NPs and PEDOT:PSS in section "Material characterization", followed by the optical and electrical characterization of the heterojunction in section "Heterojunction characterization". Subsequently, the observation and investigation of negative capacitance (inductive behavior) are discussed.

Materials and Methods

The fabrication process of solution-based heterojunctions adopts a simple and economical approach. The fabrication of the solution-based ZnO-NPs-PEDOT:PSS heterojunctions has 4 steps. The first step is that an ITO glass substrate was cleaned beforehand in an ultrasonic bath with deionized water, acetone, and isopropyl alcohol consecutively for 10 min each. The second step is the preparation process of PEDOT:PSS thin films utilized in the experiments, which includes 2 substeps. The first substep is that the PEDOT:PSS-based diluted water suspension (Clevios PH-1000, 0.38 wt%) was spin-coated (4,000 RPM, 30 s) on an ITO glass substrate to form a thin film. The second substep is that the PEDOT:PSS-based thin film is processed by thermal annealing at 200 °C in the air for 1 h. The third step is the preparation of the ZnO NPs film, which has 3 substeps. The first substep is to dilute the ZnO NPs purchased from Sigma-Aldrich with ethanol solution to form a 75 mg/ml ZnO-NPs-based solution. The second substep is that the ZnO-NPs-based solution was spin-coated (4,000 RPM, 30 s) on top of the PEDOT:PSS thin film. The third substep is that after the ZnO-NPs-PEDOT:PSS thin film was cooled down, the ZnO-NPs-PEDOT:PSS thin film sample was baked in the air at 200 °C for 1 h. The last step is that aluminum electrodes were deposited on the top of the ZnO-NPs-PEDOT:PSS thin film through a thermal evaporation process, where the electrode thickness is 100 nm.

Results

Material characterization

To begin with, ZnO NP thin films underwent scanning electron microscopy (Leo Variable pressure SEM), as shown in Fig. 1A below. These ZnO-NPs films were implemented using the spin

coating approach, where the ZnO-NP solution is first dropped on a precleaned SiO₂ substrate and then spin-coated at the conditions of 3,000 RPM and 30 s. To experimentally measure the size of the ZnO NPs in solution, a dynamic light scattering approach was employed, revealing a size distribution ranging from 20 to 110 nm, with a predominant range of 30 to 60 nm [19]. Subsequently, the optical absorption measurement of the ZnO-NPs film prepared by the spin coating (3,000 RPM and 30s) process on a glass substrate was conducted using UV-visible spectroscopy, as shown in Fig. 1B (reference absorption by glass has been subtracted). The absorption spectrum exhibited a pronounced rise in the UV region below 400 nm, with negligible absorption in the visible range. The energy bandgap of ZnO NPs was determined using the well-known Tauc plot (inset of Fig. 1B), and it was found to be 3.29 eV by extrapolating the intersection point of the linear part with the x-axis.

Additionally, the resistor-capacitor (RC) time constant (denoted by τ_{RC}) is a very important parameter of ZnO NPs used for UV detectors. Here, the τ_{RC} was investigated as it represents the time consumption from unbalanced charge carriers to relax to an equilibrium state. In the context of photodiodes, the response time mainly relies on τ_{RC} , in addition to other factors such as the charge collection time in the depleted (drift) and undepleted regions (diffusion) [20]. Here, the ZnO-NPs' τ_{RC} was measured by impedance spectroscopy using Auto lab PGSTAT302, where the dc bias voltage is 0.1 V and the frequency is set to 0.1 Hz to 1 MHz. The experimental samples are a sandwiched structure that is composed of ITO, ZnO NPs, and aluminum electrodes. The prepared sandwiched sample processing is as follows. First, a ZnO NP dispersion (1.15 g/ml, 3,000 RPM, 30 s) is spin-coated on precleaned ITO glass. Then, the sample is processed by thermal treatment at 200 °C in air for 1 h. After that, a thermal evaporation process was utilized to deposit an aluminum electrode onto the top of the ZnO-NPs-ITO glass. The thickness of the aluminum electrode was 100 nm. Based on the measurement of a surface profiler, the ZnO-NP thin film of the samples has a thickness of 2 μ m, and the size of the sample was 1 mm $\times$ 1.5 mm. The real impedance against the imaginary impedance (Nyquist plot) of the sample measured in the dark is shown in Fig. 1C (red). From Fig. 1C, the profile of the Nyquist curve is a semicircle, indicating one relaxation mechanism, and it can be represented by one RC parallel circuit. After the values of equivalent R and C were extracted by curve fitting, we found that the R and C are 4.16 k Ω and 13.4 nF, respectively. Consequently, the RC time constant was determined to be 55.87 μ s. A similar measurement was conducted under UV illumination (1.58 mW/cm²), resulting in an RC time constant determined to be 8.39 μ s, which is lower than that obtained in the absence of light. Furthermore, the measurements under a range of UV power densities were performed, and the resistance and τ_{RC} results are shown in Fig. 1D. Based on Fig. 1D, we found that the equivalent resistance decreases as the UV power density increases (left), and the τ_{RC} also drops with a higher UV power density (right).

The optical absorption analysis of the PEDOT:PSS-based thin film was also carried out. The PEDOT:PSS thin film was formed by spin-coating on glass (3,000 RPM, 30 s). In addition, the absorbance increases as the wavelength approaches the near-infrared region because of the intraband transitions of free charge carriers, while the enhanced absorption below 400 nm is attributed to the energy transition at the PEDOT backbone [21,22]. The results (Fig. 1E) revealed significantly weaker absorption than the ZnO NPs thin film in the visible range. This

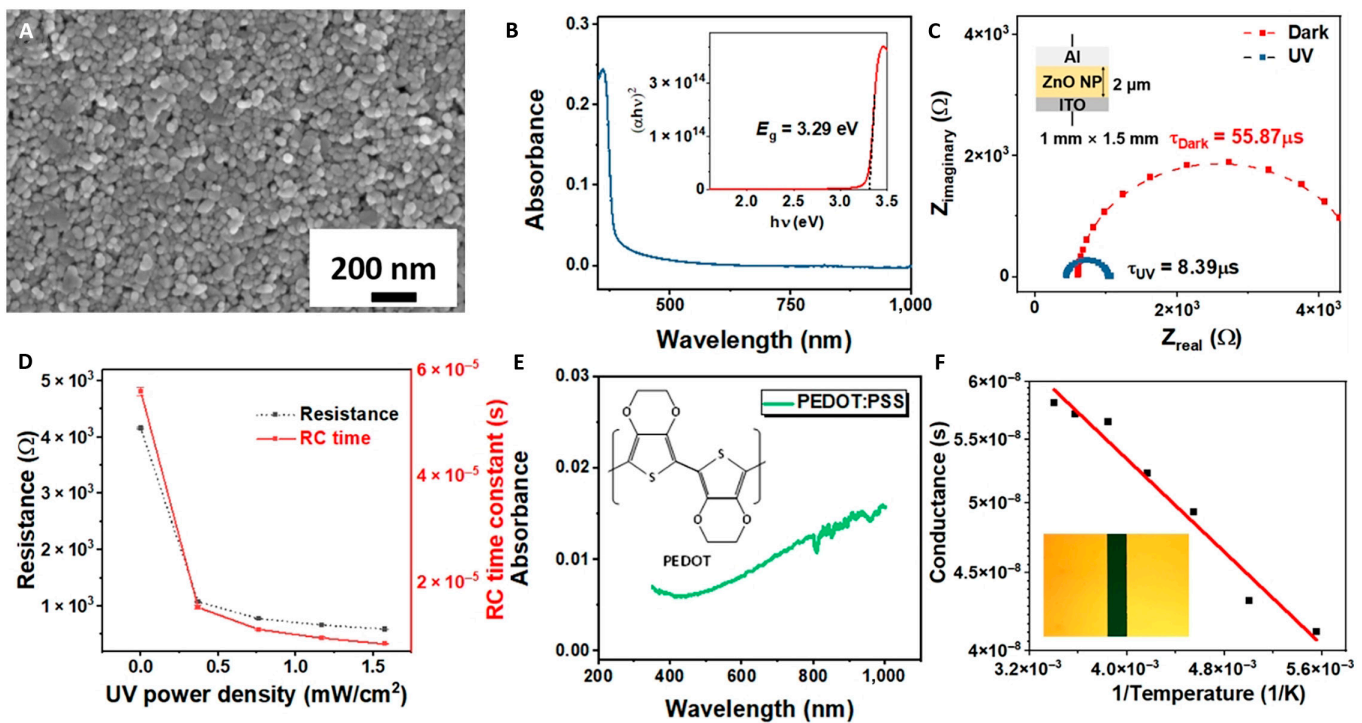


Fig. 1. Characterization of ZnO NPs and PEDOT:PSS thin films. (A) A SEM image of ZnO NPs. (B) The absorption spectrum of the ZnO NPs thin film using UV–Vis spectroscopy and Tauc plot (inset). (C) The Nyquist plot of the ZnO NPs sample with UV (in the dark background [red curve]) and without UV (black curve). (D) Resistance and τ_{RC} of the ZnO-NPs sample under different UV power densities. (E) The absorption spectrum of the PEDOT:PSS thin film using UV–Vis spectroscopy. (F) Low-temperature measurement of the PEDOT:PSS sample.

implies that the PEDOT:PSS has a trivial impact in the UV sensitivity of heterojunction-based photodiodes.

Besides, to obtain the electrical performance of the PEDOT:PSS thin film, low-temperature conductivity measurements were performed on PEDOT:PSS thin-film samples using a Lake Shore cryogenic probe station, and the conductance results are shown in Fig. 1F. The PEDOT:PSS thin-film sample was fabricated by the same spin-coating process, followed by thermal deposition of aluminum electrodes (100 nm). Charge transport within PEDOT:PSS is of considerable importance, and it is widely acknowledged that the electrical conduction mechanism adheres to a variable-range hopping (VRH) model [18,23]. The VRH model could describe the charge transport as hopping between localized states, and the conductivity is a function of temperature (T). According to the VRH model, electrical conductivity (σ) can be quantitatively determined by the following equation:

$$\sigma(T) = \sigma_0 \exp \left[- \left(\frac{T_0}{T} \right)^\alpha \right] \quad (1)$$

where σ_0 is the conductivity prefactor. T_0 represents the potential barrier height, and α is a measure of the transport process. The conductance (in log scale) versus $1/T$ was plotted and shows a linear relationship ($\alpha = 1$). The Arrhenius-like relationship indicates that this is a thermally activated process and is a nearest-neighbor hopping type [24].

Heterojunction characterization

The composition diagram of the ZnO-NPs-PEDOT:PSS heterojunction to measure optoelectronic properties is shown in Fig. 2A. First, the IV characteristics of the heterojunction were measured using an Agilent 5146 semiconductor analyzer. The

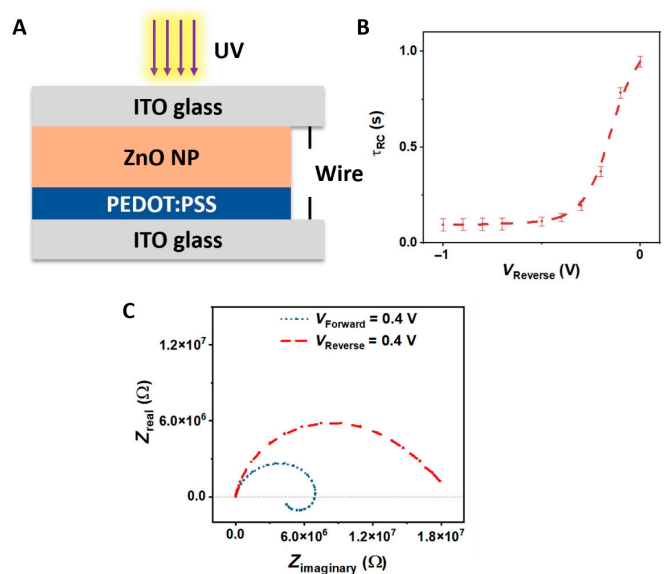


Fig. 2. Characterization of optoelectronic properties of heterojunctions. (A) Schematic composition diagram of the heterojunction for the optoelectronic measurement. (B) The τ_{RC} of the heterojunction at different reverse-bias voltages. (C) The pseudoinductive behavior at forward bias.

measured result indicated that the on/off current ratio reaches 6.7×10^3 at 2 V. Then, the barrier height was also determined using capacitance–voltage measurement. The test result showed that the barrier height was 0.64 eV by extracting the intersection point of the $1/C^2 \sim$ reverse-bias voltage ($V_{Reverse}$) plot with the x-axis. The current gain generated by UV illumination reached 91.2, and the fall time (τ_{fall}) from 90% to 10% of its step

height was measured to be 11.4 s. Moreover, photogenerated conductance under different UV powers was measured, and the exponential relationship with an exponent index of 0.321. The fall time is associated with the trap states that widely exist in ZnO NP thin films, which prolongs the fall time because photogenerated charge carriers experience 2 essential processes (i.e., trapped and re-excited states) before they are recombined. Typically, the trapping and re-excitation process consumes more time than direct recombination. Achieving rapid switching can be accomplished through impedance matching and exploiting the fast-switching part of the fall time.

Figure 2B presents the measurement of τ_{RC} for the prepared heterojunction. The τ_{RC} without applying a dc bias voltage is 946.46 ms (as shown in the inset). Based on Fig. 2B, when the reverse-bias voltage was applied to -1 V, the τ_{RC} rapidly drops to 94.36 ms. Therefore, a faster response of the photodiode can be achieved at a larger reverse-bias voltage. Notably, a pseudoinductive behavior, characterized by negative capacitance, emerged at the lower frequencies when the heterojunction was subjected to forward bias Fig. 2C (blue). A more systematic set of measurements was carried out at various forward and reverse biases, and Fig. 3 below shows the testing results.

Figure 3A and B clearly illustrates the presence of negative capacitance, which is observed only under forward bias at low frequency and disappears when the bias is reversed. Furthermore, we also found that the parallel equivalent resistance becomes decreased when the applied forward-bias voltage was increased, while it exhibited an increase with higher reverse-bias voltage. Negative capacitance can enhance the detection capability of junctions, increase the photocurrent of devices, and enhance the optoelectronic response characteristics of devices. When the incident light shines on the PD, the excited photogenerated electrons are transitioned to the conductive band. Meanwhile, the photogenerated holes were accordingly captured by the

defect centers inside the material, improving the photoelectric gain of the device.

Different theories have been proposed to illustrate the occurrence of the negative capacitance effects in various systems. In the short-based PN junction, it was interpreted as the depopulation of minority carriers, which results in a variation of the positive and negative (i.e., sign) of the capacitance [25]. For Schottky diodes, the explanation involves the loss of interfacial charge lower than the Fermi level, which governs the current of ionic species between the oxidizing and reducing processes [26,27]. In bipolar organic diodes, the mechanism accounting for negative capacitance is primarily trap-assisted recombination [28]. In polymers, it was reported that the injected current lagging behind the AC voltage due to the finite transit time [29–31] and increased charges trapped at the interface [32] are responsible for the negative capacitance. In the context of the heterojunction photodiode, 2 key observations stand out.

Additionally, it was observed that a threshold in the forward-bias voltage, approximately at 0.2 V, exists below which negative capacitance is not present. This observation aligns with the results reported by Mora-Seró et al. in the negative capacitance experiments [27]. The frequencies of the intersection points between the Nyquist plot and the x -axis (imaginary impedance is zero) are plotted against the forward-bias voltage in Fig. 3c. The frequency at the intersection represents the transition from capacitive to inductive behavior. Moreover, the minimum frequency of 0.08 Hz was obtained at 0.2 V, while it was 3.45 Hz at 1 V. Evidently, when the applied bias voltage was increased, a higher transition frequency was provided, suggesting the presence of a “lagging” mechanism linked to negative capacitance, akin to the mechanism observed in OLEDs [29,30]. Nevertheless, the precise origins of negative capacitance warrant further investigation in future research endeavors.

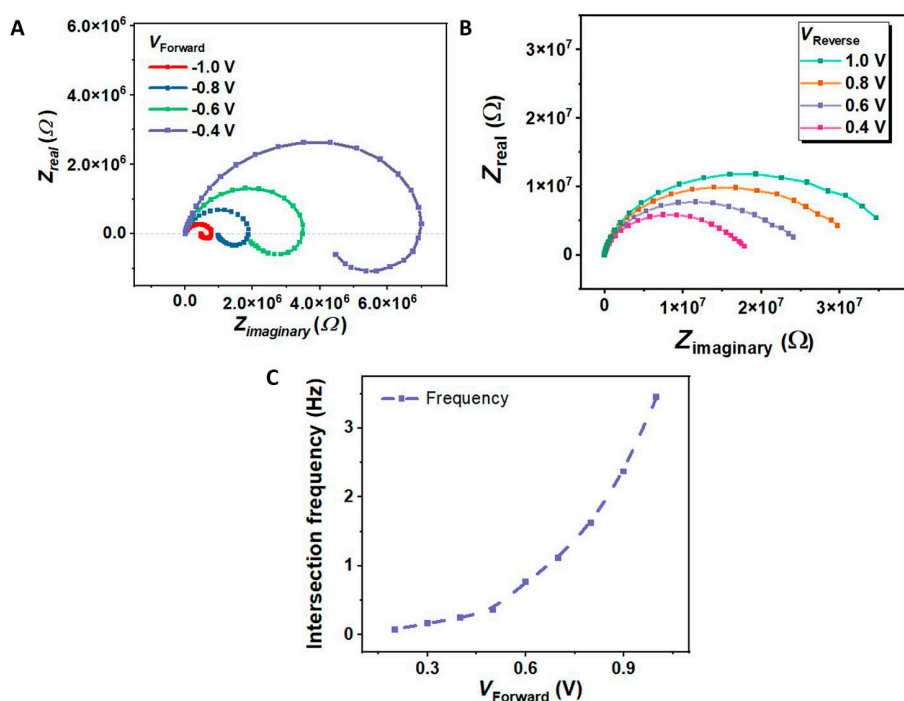


Fig. 3. Nyquist curve of the ZnO-NPs-PEDOT:PSS heterojunction at the different biases. (A) forward bias and (B) reverse cases. (C) The extracted transition frequency curve when different bias voltages were applied in the forward case.

Table. A comparison list of the response times of different photodetectors based on ZnO

No.	Device structure	Rising time(s)	Reference
1	Ag/ZnO nanorods	Thousands	[33]
2	ZnO/SnO ₂ nanofibers	Hundreds	[34]
3	ZnO ultraporous nanoparticles networks	~250	[35]
4	ZnO nanofibers	~100	[36]
5	ZnO films/BiOCl	25.83	[37]
6	ZnO NWs/Pbs quantum dots	9	[38]
7	ZnO nanorods/graphdiyne nanoparticles	6.1	[39]
8	ZnO quantum dots/graphene	5	[40]
9	ZnO homojunction	3.9	[41]
10	ZnO NWs	1.1	[42]
11	ZnO NRs/Graphene	0.3	[43]
12	ZnO NRs/PEDOT:PSS	0.11	[44]
13	Solution-based ZnO-NP- PEDOT:PSS	0.0745	The proposed device

Finally, we also conduct a comparative analysis of the rise time exhibited by the proposed device in contrast to other ZnO-based photodetectors. A comparison list of the response times of the various approaches for photodetectors using ZnO is shown in Table. Most of the existing works of ZnO-based photodetectors demonstrated that the response times have achieved an excellent performance from hundreds of milliseconds to hours. The proposed method yields a rising time of 0.0745 s. Notably, the rise time achieved in this study is significantly shorter than most of the existing photodetectors based on ZnO. The key factor is that the PD in this work adopts the ZnO-NPs/PEDOT:PSS heterojunction structure, ZnO is a wide bandgap semiconductor with high electron mobility, while PEDOT:PSS is a common conductive polymer with favorable hole conduction performance. The heterojunction interface formed between the two can efficiently separate the photo-generated electron-hole pairs. Through optimizing the quality and thickness of the film during the preparation process, the charge transport efficiency can be further enhanced, reducing the time for charge accumulation and shortening the rise time. These results indicated that the proposed device exhibits the best photosensitivity and that the good photoswitch property is suitable for fast optically addressed spatial light modulators (OASLM) applications.

Conclusion

This paper experimentally demonstrated a new UV photodiode through a ZnO-NPs-PEDOT:PSS heterojunction, which was fabricated utilizing an entirely solution-based process for low cost, simple fabrication, and potential for large-scale fabrication. First, the properties of ZnO NPs and PEDOT:PSS were individually characterized. Then, the electrical and optical properties of the ZnO-NPs-PEDOT:PSS heterojunction were investigated. A negative capacitance effect was also

experimentally observed in this kind of heterojunction for the first time. Moreover, its threshold voltage and transition frequency were also further studied. The experimental results indicated that the rise time achieved by the proposed ZnO NP photodetector stands at a mere 0.0745 s, significantly outperforming other ZnO-based photodiodes. The trap states in ZnO NPs are highly beneficial to high-resolution applications such as OASLM, so the heterojunction formed by ZnO NPs and PEDOT:PSS is highly promising for high-resolution, high-responsivity, and high-speed OASLM devices.

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Author contributions: X.C. and J.L. contributed equally to this work. X.C. observed the phenomenon and carried out experiments. X.C. and J.L. discussed experiments, analyzed data, and wrote the manuscript. S.L. provided guidance and revision of the manuscript. Y.Z. helped review and revise the manuscript. D.C. supervised the project and revision of the manuscript.

Competing interests: The authors in this manuscript declare that all authors have no competing interests.

Data Availability

The datasets used in this manuscript are available through this work's corresponding author based on a reasonable request.

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