

Full paper

Achieving high-resolution pressure mapping via flexible GaN/ ZnO nanowire LEDs array by piezo-phototronic effect

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ABSTRACT

Simulating human tactile sensing through high-resolution electronics is a significant and challenging topic in artificial intelligence. A flexible p-GaN film/n-ZnO nanowire light-emitting diode (LED)-based pressure sensor array, with the merits of flexibility, high resolution, good transparency, fast response, stability, and lightweight, is fabricated through laser lift-off (LLO) process and hydrothermal growth of ZnO nanowires directly on a flexible GaN film, which is used for acquiring the two-dimensional (2D) pressure distribution mapping by reading the illumination intensities from all LED pixels parallelly. The intensity of each pixel composed by a GaN/ZnO nanowire heterostructure LED can be enhanced by the local compressive strain based on piezo-phototronic effect. A high spatial resolution of $2.6 \mu\text{m}$ and a fast response time of 180 ms were obtained, and also the sensor arrays can still function well after 4000 bending circles. The promising flexible LEDs-based pressure sensor array gains some technological innovations in the field of tactile sensing, with potential applications in smart skin, biomedicine, optical MEMS, and touchpad technology.

1. Introduction

Strain sensor emulating human tactile sensing is crucial for health and environment monitoring, human-machine interfaces and next-generation robotics, which needs to meet some significant characteristics including high spatial resolution, fast response, high sensitivity, and large scale [1–6]. The rapidly developing flexible LED arrays could be employed as strain sensors to indicate the 2D pressure distribution through different light emission intensities, with plentiful advantages such as flexibility, high efficiency, energy conservation, visualization, and portability, which could bring new technological innovations in the applications of electronic skins, wearable technologies, flexible screens, personal signatures, and biomedical diagnostics [7–14]. At present, many tactile sensors based on changes in resistance or capacitance corresponding to various applied pressure have been reported, which

are commonly fabricated by conductive elastic composites (such as mixed rubber with single-wall carbon nanotubes) or microstructured elastic dielectric layers (normally combined with field-effect transistors), possessing a millimeter order resolution [15–18]. To meet demands of electronic skin with a spatial resolution higher than $50 \mu\text{m}$ (human skin), our group has reported a ZnO nanowire/p-GaN LED-based pressure sensor array which can map the 2D pressure distribution with a superior spatial resolution of $2.7 \mu\text{m}$ on account of piezo-phototronic effect [1]. Subsequently, considering a rigid substrate cannot meet demands of the artificial skin due to the lack of flexibility, a series of controllable and flexible pressure sensors composed by inorganic (etc. ZnO, CdS nanowires)/organic (PEDOT: PSS) hybridized LED arrays also based on piezo-phototronic effect have been exploited [9,19–22]. However, compared with GaN-based sensors, these flexible pressure sensors exhibited some drawbacks including lower resolution,

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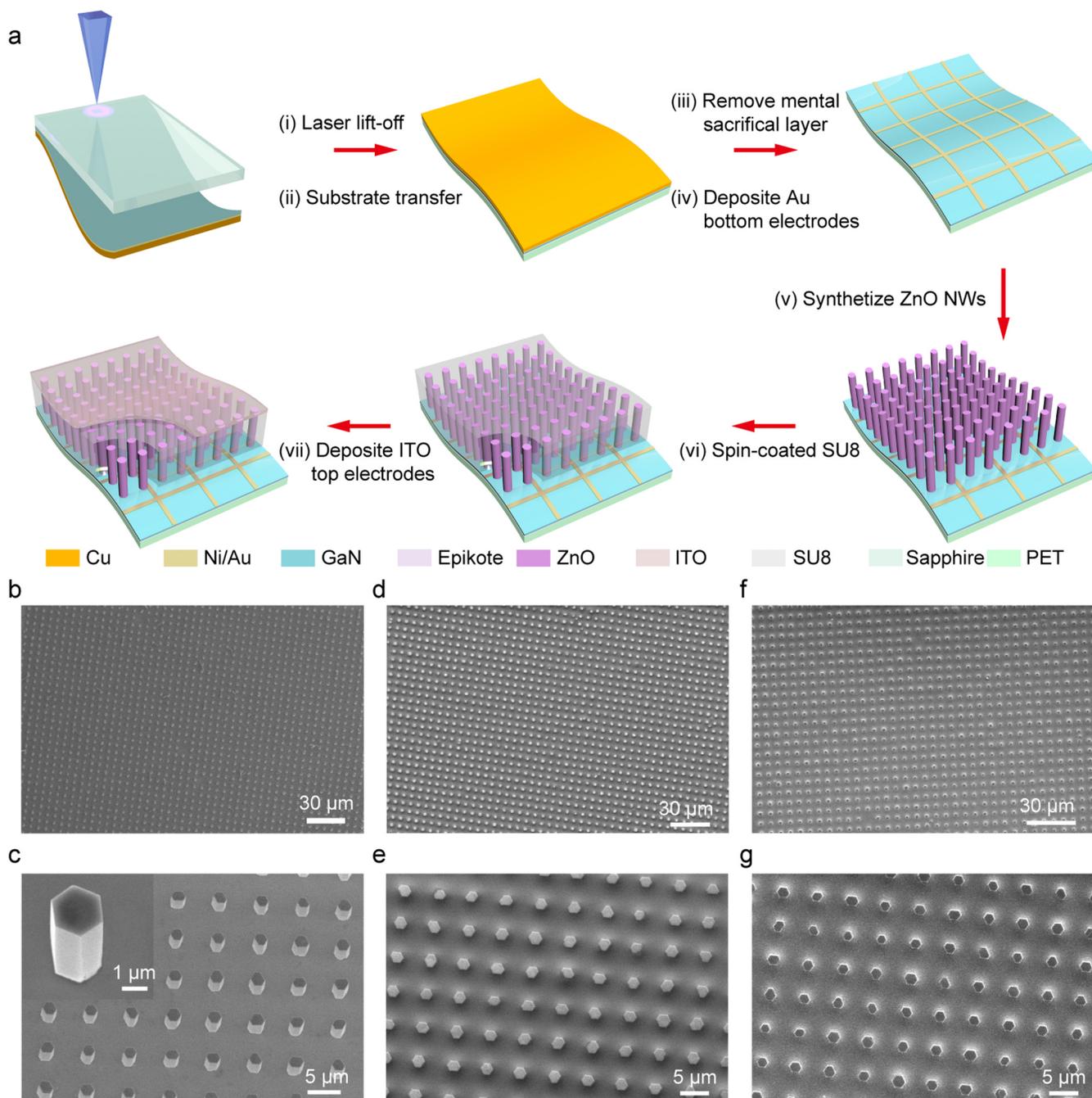


Fig. 1. Schematic fabrication process and the structure of the flexible p-GaN/n-ZnO nanowire LEDs array. (a) Schematic diagram of the device fabrication consisting of GaN LLO process and the synthesis of ZnO nanowires array. (b, c) SEM images of the as-grown ZnO nanowire arrays on the flexible p-GaN film, with an average diameter of $2\ \mu\text{m}$ and length of $4\ \mu\text{m}$. (d, e) The wrapped and exposed tips of ZnO nanowires after spin-coated SU 8 and oxygen plasma etching to improve the robustness of the structure, showing a clean surface. (f, g) SEM images corresponding to ITO layer deposited onto the wrapped and etched ZnO nanowires as a top common electrode.

poorer stability, shorter lifespan, and nonepitaxial nanowires growth. Particularly, it is a critical step to combine flexibility property with the advantages of GaN-based pressure sensors. Notably, the rigidity of GaN/Sapphire structures comes from a $500\text{-}\mu\text{m}$ -thick sapphire substrate, while $5\ \mu\text{m}$ GaN layer is flexible. Therefore, we hope to separate GaN film from sapphire and fabricate it with ZnO nanowires array together as a flexible strain sensor, achieving high-resolution pressure mapping via flexible GaN/ZnO LEDs array and potentially promoting the development of prospective human-machine interface system.

Here, we report a flexible pressure sensor array based on p-GaN

film/n-ZnO nanowire heterostructure LED to map spatial pressure distribution with an ultrahigh resolution of $2.6\ \mu\text{m}$. Each p-GaN/n-ZnO nanowire LED plays the role as a pixel to sense the local pressure or force. It is firstly realized to directly grow ZnO nanowire arrays on flexible GaN films. As is well-known, GaN is an ideal direct wide band gap semiconductor material in light-emitting devices due to its good chemical stability, environmental compatibility, and excellent optoelectronic properties [23–26]. Generally, the epitaxial growth of GaN crystal relies on a rigid substrate, which has limited its application in flexible optoelectronics for a long time. That is, if GaN crystal can be separated from rigid substrate, it may potentially enable more

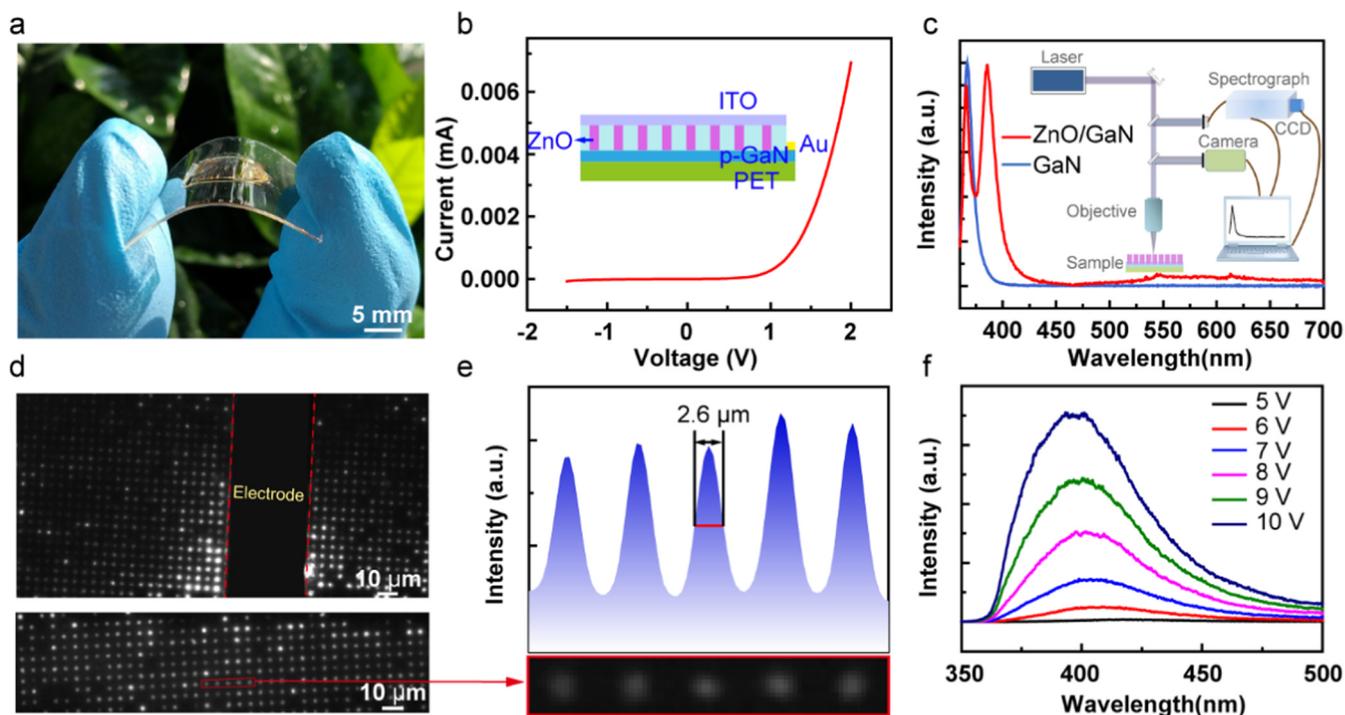


Fig. 2. Electrical and optical characterizations of the flexible fabricated LEDs array devices. (a) Optical image of bending the prepared LEDs array device. (b) I-V curve of the flexible device measured at the natural condition. (c) PL spectra from flexible GaN substrate before and after ZnO nanowires growth at room-temperature. The inset is corresponding optical measurement system. (d) Optical images of the lighted p-GaN/n-ZnO LED arrays at an applied bias of 10 V. Each nanowire is a single light emitter representing a pixel unit of the pressure sensor. (e) Five typical nanowire LEDs (marked with a red rectangle in d) and a corresponding line profile of their emission intensity with an estimated spatial resolution of 2.6 μm . (f) Room temperature EL spectra of lit-LED arrays at different applied bias of 5, 6, 7, 8, 9 and 10 V.

development space in flexible optoelectronic devices [27–31]. Accordingly, we fabricated flexible GaN substrate through LLO and transfer process, and then ZnO nanowire arrays were epitaxially grown on flexible GaN film directly to form a LED-based pressure sensor. A pressure distribution mapping can be acquired by reading the illumination intensities from all LED pixels in parallel, obtaining a high spatial resolution of 2.6 μm and a fast response time of 180 ms. Upon compressive strain, the positive piezoelectric polarization charges are generated at the local interface of the p-n junction on account of piezophotronic effect, which further alters the energy band structure and thus promotes the recombination between electrons and holes to enhance light-emitting intensity. Therefore, the device may be utilized in prospective applications as smart skins in virtue of their preponderance including flexibility and ultrahigh spatial resolution.

2. Experimental section

2.1. Fabrication process of flexible GaN thin film

About 2.5- μm -thick undoped GaN buffer layer and 2.5- μm -thick Mg-doped p-type GaN layer were epitaxially grown on c-plane sapphire substrate through metal organic chemical vapor deposition (MOCVD) to be used for stripping. For fabricating flexible GaN thin film, Ni/Au sacrificial layers were deposited by DC magnetron sputtering (Kurt J. Lesker, PVD75), with the thickness of about 20 nm and 100 nm, respectively. Subsequently, a 50- μm -thick Cu layer was electroplated on the Ni/Au conducting layers, followed by the LLO process to separate the whole structures from sapphire substrates using a pulsed KrF laser (COHERENT LSX 200 K), which possesses a 248 nm wavelength and a 25 ns typical pulse duration to irradiate the back side of the sapphire substrate at a power density of 254 mJ/cm^2 [30]. To ensure the flatness of the film, the structures need to experience the process of hot pressing firstly after LLO. Considering the defect surface and intrinsic (undoped)

properties exhibited on the back side of GaN, a 200- μm -thick PET film was attached at the back side of the GaN film by epoxy resin to expose the front side for preparing the device. After the structures were placed in an oven at 373 K for 2 h to solidify, Cu/Au/Ni sacrificial layers were removed by wet chemistry to obtain a clear and damage-free flexible GaN substrate.

2.2. Fabrication of flexible GaN/ZnO nanowires LEDs array

A lattice with line width of 300 μm and spacing of 30 μm was obtained by photolithography with positive photoresist. Subsequently, layers of Ni/Au (10 nm/25 nm) were deposited using DC magnetron sputtering to form the bottom grid electrodes. After removing the photoresist, patterned cylinder (SUN-9i photoresist) was formed by photolithography with a diameter of 2 μm and spacing of 5 μm . Then, a mask layer of 35-nm-thick SiO_2 was deposited by RF magnetron sputtering, and the photoresist was removed subsequently [32]. The flexible GaN substrate with SiO_2 pore mask was put into a nutrient solution containing 40 mM hexamethylenetetramine (HMTA) (Alfa Aesar) and 40 mM zinc nitride (Alfa Aesar) to synthesize ZnO nanowires array at 85 $^\circ\text{C}$ for 4 h in an oven [1,33]. The c-axis oriented GaN locations exposed to the nutrient solution will be grown epitaxially ZnO nanowires, yielding uniformly patterned single-ZnO-nanowires arrays on a scale of centimeters accordingly. And then, a SU-8 (Microchem) photoresist layer was spin-coated to wrap around as-grown ZnO nanowires. The tips of the ZnO nanowires need to be exposed according to the device structure, so the top part of the SU-8 was etched away by the process of oxygen plasma. Finally, an about 300-nm-thick ITO film was deposited forming the top common electrodes by magnetron sputtering to accomplish the flexible heterostructure devices.

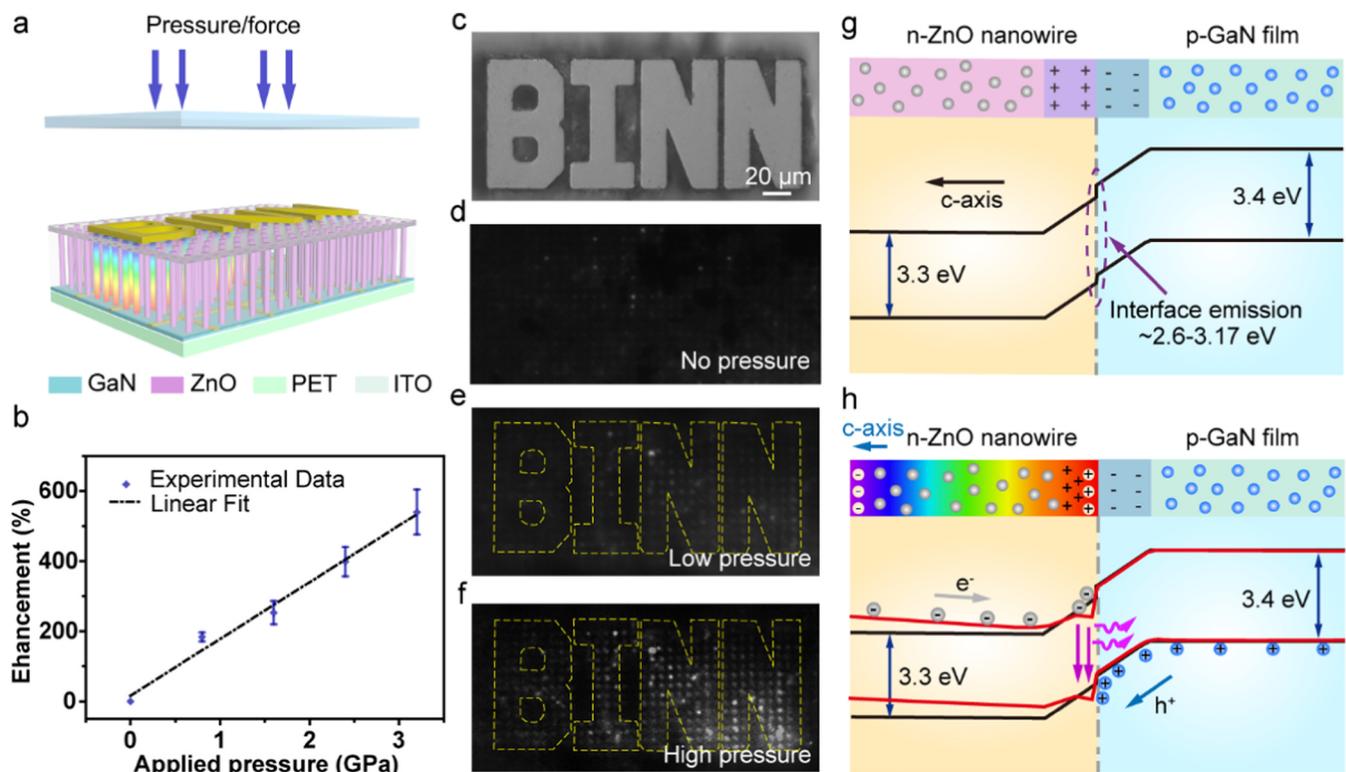


Fig. 3. Working mechanism of pressure sensor and enhancing light emission by piezo-phototronic effect. (a) Schematic illustration of pressure distribution mapping performance. (b) An approximately linear relationship between enhancement factor E and applied pressure value of the nanowires LED-based strain sensor. (c) Optical image of the convex-character sapphire seal of “BINN” used in the pressure measurement. (d–f) Electroluminescence images of the device at zero, low and high pressure, respectively. These images clearly demonstrate emission intensity would be enhanced only at compressed LED pixels by convex character. (g, h) Energy band of p-GaN/n-ZnO heterostructure before and after applied compressive strain, respectively.

2.3. Characterization and measurement of the flexible devices

The morphologies of as-grown and photoresist-coated ZnO nanowire arrays were characterized by a cold-field-emission scanning electron microscope (SEM, Hitachi, SU8020) and a hot-field-emission SEM (Quanta 450), respectively. X-ray diffraction (XRD) was accustomed to record the sample phase through an X-ray diffractometer (PANalytical, X'Pert 3 Powder) with a Cu K α radiation source. The flexible device was powered by Maynuo DC Source Meter M8812. I-V characteristic was acquired by Keithley 4200-SCS. Photoluminescence spectra of ZnO nanowires stimulated by a nanosecond pulsed laser at wavelength of 355 nm were detected through an optical multichannel analyzer (Andor, SR-500i-D1-R) coupled with a confocal μ -PL system (Zeiss M1). Electroluminescence spectra were collected by a spectrometer Edinburgh FLS 980 with scan step of 0.05 nm. The optical images were taken by an inverted microscope (Zeiss Observer Z1) furnished with an HQ2 camera, which also constructed the piezo-phototronic effect measurement system with a 3D micromanipulation stage and a dynamometer, monitoring magnitude of force corresponding to the variation in light-emitting intensity through collecting and processing images. A sapphire substrate with a convex character pattern of “BINN” was utilized to apply a normal force on the flexible device.

3. Results and discussion

The fabrication process of the flexible p-GaN film/n-ZnO nanowire heterostructure arrays is schematically illustrated in Fig. 1a. To combine the property of flexibility and high resolution in a single strain sensor, the GaN substrate needs to be separated from sapphire through LLO process and then attached upon the PET substrate. The transferred flexible GaN film can be found in Fig. S2a. Ni/Au grid bottom electrodes are deposited to acquire Ohmic contact with p-GaN film (Fig.

S2b). The patterned ZnO nanowire arrays pointing c-axis upwards from flexible GaN substrate are synthesized by low-temperature hydrothermal method assisted by photolithography, thus the position, diameter and length of the nanowires can be superbly and effectively controlled [33,34]. Vertical n-type ZnO nanowires are observed to be aligned uniformly and orderly through epitaxial growth on a flexible p-GaN film, the SEM images of which have been illustrated in Fig. 1b, c and Fig. S2d–f. Each nanowire with an average diameter of 2 μ m and length of 4 μ m acts as a basic independent pixel of the sensor. After the six-sided cylindrical ZnO nanowires are infiltrated by SU-8, oxygen plasma etching needs to be used to expose the heads of the nanowires (Fig. 2d, e). Subsequently, a transparent indium tin oxide (ITO) film acting as a common top electrode is deposited directly on the top of the nanowires etched by oxygen plasma for electron injection (Fig. 2f, g). Specifically, the tiny fluctuation in the diameters and lengths of nanowires will not influence the sensing of devices. And more detailed procedures are explained and illustrated in Fig. S1, Supporting Information, and Experimental section.

Optical performances and electrical characteristics of the flexible LEDs array are presented in Fig. 2. The corresponding optical image of a transparent and flexible LEDs array has been given in Fig. 2a. Current-voltage (I-V) characteristics of the LED device show a good rectification effect, demonstrating a reasonable p-n junction characteristic and the possibility of luminescence (Fig. 2b). The inset schematically illustrates the section structure of the device. To further indicate the crystalline quality of the ZnO nanowires, photoluminescence (PL) spectrum of individual ZnO nanowires at room-temperature have been acquired, which indicated a near-band-edge (NBE) emission peak of ZnO at 385 nm dominated with only an extremely broad and weak defective luminescence (Fig. 2c). PL spectra of the structures consisting of ZnO nanowires and GaN film are pumped by a nanosecond pulsed laser (355 nm) with pumping power at 1.75 kW/cm², the inset of which

illustrates the optical measurement system. Optical images of lighted nanowire LED arrays at a bias of 10 V have been measured and displayed in Fig. 2d, in which each nanowire stands for a single light emitter and a pixel unit to sense local pressure. Light emission images corresponding to different applied bias voltages are displayed in Fig. S3a–f, indicating ZnO nanowire pixels will be gradually and uniformly lit with the increase of applied bias voltage and almost all nanowire pixels are lighted at bias of 10 V. Fig. 2e presents an enlarged image of five representative nanowire LEDs marked by a red rectangle in Fig. 2d and their corresponding line profiles of emitting intensity, clearly indicating no crosstalk between adjacent emission pixels. According to definition of the full-width at half-maximum (FWHM) of the LED emission pixels, the resolution of the flexible device reaches up to 2.6 μm and it represents an extremely high value, which outdistances the resolution of human skin. Electroluminescence (EL) spectra of the flexible heterostructure LED arrays are obtained under different bias at room temperature, as indicated in Fig. 2f. Moreover, if we optimize the size and space distance of nanowires, the spatial resolution can still be improved and reaches a much higher value. The EL intensity will enhance with the increase of applied bias voltage, corresponding to the band-bending theory of a p-n heterojunction [35]. A strong violet-blue emission centered around 410 nm, as well as no defect-related emission, can be observed, which further demonstrates relative perfect crystallinity quality. The full EL emission spectrum of the LED device at applied voltage of 7 V is also presented in Fig. S3g.

To inquire how to modulate the LED emission intensities through piezo-phototronic effect, a pressure mapping performance of flexible p-GaN/n-ZnO nanowire heterostructure array is tested through pressing a convex-character stamp “BINN” made of sapphire on the top of devices, resulting in an induced piezopotential in the compressed nanowires, while no piezopotential in uncompressed nanowires, as shown in Fig. 3a. Specifically, the wurtzite crystals possessing hexagonal structure, such as ZnO, GaN, and ZnS, have an obvious anisotropy in the c-axis and perpendicular to the direction, which naturally present piezoelectric effect owing to their non-centrosymmetry property [36]. As for ZnO crystal structure, the Zn^{2+} and O^{2-} are tetrahedrally coordinated, the centers of which overlap each other at the strain free state. Under compressive stress, a relative displacement proceeds between the center of Zn^{2+} and that of O^{2-} leading to a dipole moment, which is known as the induced piezopotential [37]. Detailed measurement setup has been displayed in Supporting Information (Fig. S4). It needs to be noted that the emission light of pressure mapping was captured from the side of sapphire convex character rather than the side of flexible PET substrate, thus avoiding the scattering and absorption of PET. The dependence of LED light emission intensities on applied pressures has also been investigated, which indicates the illumination intensities get stronger with the increase of applied pressure. Enhancement factor E of the LED emission intensity is defined as $E = (I_p - I_0/I_0)$, where I_0 is defined as an image taken by CCD without strain applied (background signal) and I_p represents whole illumination intensity of the LED under applied pressure. Fig. 3b indicates an approximately linear relationship between enhancement factor E and pressure. In particular, the force mentioned in Fig. 3b referring to the applied value displayed on dynamometer is larger than the actual force subjected to the nanowires array, due to the flexibility property of device may give rise to the surrounding PET substrate without ZnO nanowires shares a part of stress, as explained in Fig. S4. Thus, Fig. 3b mainly reveals an increasing tendency of enhancement factor E with increasing applied pressure and E factor reaches the maximum value of $\sim 530\%$ at a high applied pressure. Furthermore, the electroluminescence mapping images are acquired by pressing a convex sapphire character of “BINN” onto the LED devices as seal and Fig. 3c displays the optical images of the convex sapphire seal. The pressure distribution mappings corresponding to different light intensities of the flexible sensor under zero, low and high pressures are presented in Fig. 3d–f, respectively, which clearly and directly indicates a gradually

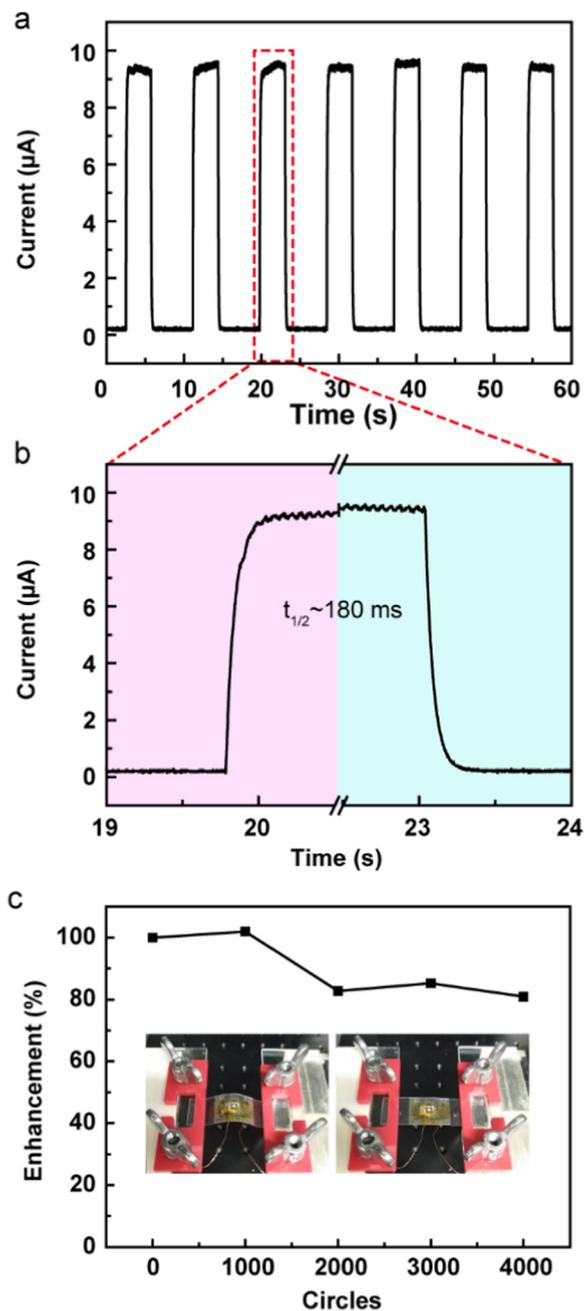


Fig. 4. Characterizations of response and stability for the flexible GaN/ZnO nanowires LED-based pressure sensor. (a) Fast response and recovery of the device when applying a periodic pressure. (b) An enlarged image (marked with a red rectangle) demonstrating a response and recovery time of 180 ms. (c) Mechanical stability of the flexible LED-based pressure sensor for 4000 bend-release cycles.

stronger illumination intensity under the stepped-up pressure. It is observed that LED emitting intensities towards the pixels compressed by the convex sapphire character are enhanced, while non-compressed pixels present virtually no change in emission intensity. Therefore, it is proven that piezo-phototronic effect can effectively enhance the light emission of flexible p-GaN/n-ZnO nanowire LED arrays.

To better explain the inner physical mechanism of piezo-phototronic effect worked on LED device, energy band diagrams of n-ZnO/p-GaN heterostructure before and after applied a pressure are illustrated in Fig. 3g, h. Upon compressive strain, the strain in GaN film is much smaller than that inside ZnO nanowires owing to the low coverage of ZnO nanowires on the GaN film, thus piezopotential comes into being

in the side of ZnO nanowire. It has been demonstrated that the c-axis direction of ZnO nanowire should be hydrothermally grown pointing away from GaN film [38], thus positive piezoelectric polarization charges are generated in the charge-depletion zone under compressive strain, which indicates the created piezoelectric field keeps the same direction with the built-in electric field. As a result, the ZnO conduction band edge are decreased at the interface and the transport of electrons are accelerated to the interface [39,40]. A dip is hence induced at the local band structure and electrons are temporarily trapped near the interface at ZnO side, which increases the number of photons created through radiative transition and the carrier recombination rate at the contact interface [41,42].

To further detect more comprehensive characterizations of the flexible LED-based pressure sensor, the response rate, stability and repeatability of the flexible sensors have also been researched. The flexible LED-based pressure sensor possesses a fast response and recovery. Fig. 4a indicates the relationship between current and time under seven cycles of applied pressure, with a time resolution of about 180 ms realized from the enlarged plots in the Fig. 4b. It is noted that the process of applied pressure is relatively slow through linear motor, leading to the slowness of measured response time to some extent, which demonstrates response time greatly depends on the period of time required to apply/retract the mechanical stress. Furthermore, the flexibility and stability of the pressure mapping sensor have also been researched by comparing the light emission performance before and after the thousands of bend-release cycles with a tensile strain of 0.79%. Fig. 4c reveals the light-emitting intensity does not present significant decrease, the inset of which shows the bending process. Moreover, the device is fairly stable and it can be lit up after placed in a normal temperature and atmosphere over half of a year (Fig. S5).

4. Conclusion

In summary, we have designed and prepared a flexible and stable LED-based pressure sensor array composed of p-GaN/n-ZnO nanowire heterostructure possessing a high spatial resolution of 2.6 μm (much better than human skin resolution of 50 μm) and a fast response time of 180 ms to map pressure distributions, which meets the demand of the artificial skin performance. Strain-induced polarization charges could effectively decrease the barrier height of local surface at the ZnO side, and thus enhance light emission through promoting recombination rate between electrons and holes due to piezo-phototronic effect. A pressure distribution could be obtained by parallelly detecting different intensities of EL emission from LED arrays. The approach is scientific and credible to combine the merits of high resolution and fast response of rigid GaN substrate LED with flexibility of organic LED, which may be more ideally applied in highly intelligent and flourishing human-machine interfaces and make an important step in flexible photonic integration technologies, with potential application prospects in smart skin, touch panel technology, biomedicine science, and optical MEMS.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.nanoen.2019.01.076.

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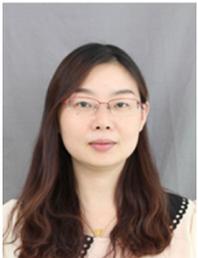
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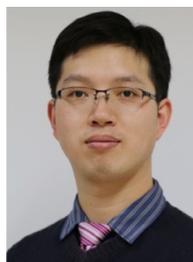
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