

Retraction

Retracted: Application of ZnO Semiconductor Nanomaterial Ink in Packaging and Printing Design

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This article has been retracted by Hindawi following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of one or more of the following indicators of systematic manipulation of the publication process:

- (1) Discrepancies in scope
- (2) Discrepancies in the description of the research reported
- (3) Discrepancies between the availability of data and the research described
- (4) Inappropriate citations
- (5) Incoherent, meaningless and/or irrelevant content included in the article
- (6) Peer-review manipulation

The presence of these indicators undermines our confidence in the integrity of the article's content and we cannot, therefore, vouch for its reliability. Please note that this notice is intended solely to alert readers that the content of this article is unreliable. We have not investigated whether authors were aware of or involved in the systematic manipulation of the publication process.

Wiley and Hindawi regrets that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity.

We wish to credit our own Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

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- [1] C. Guo, "Application of ZnO Semiconductor Nanomaterial Ink in Packaging and Printing Design," *Journal of Chemistry*, vol. 2022, Article ID 6166533, 7 pages, 2022.

Research Article

Application of ZnO Semiconductor Nanomaterial Ink in Packaging and Printing Design

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In order to obtain a flat and clear packaging printing pattern, the author proposes a printing method based on ZnO semiconductor nanomaterial ink. The method uses zinc acetate dihydrate as raw material, ethylenediamine as a complexing agent, absolute ethanol as a solvent, and ethyl cellulose as an auxiliary agent to prepare particle-free ZnO functional ink. The ink was spin-coated on a glass substrate, cured at different temperatures on a heating plate for 30 min, and passed through an X-ray diffractometer, a field emission scanning electron microscope, an infrared spectrometer, a synchronous thermal analyzer, an ultraviolet-visible spectrophotometer, and a transmission electron microscopy were used to characterize the synthesized inks and the resulting films. Experiments show that the decomposition temperature of particle-free ZnO conductive ink is much lower than that of zinc acetate precursor; the film cured at 300°C for 30 min has a smooth surface, uniform particle size, good crystallinity, and transmittance of up to 80%. After inkjet printing on the PI flexible substrate, after curing at 300°C for 30 min, the pattern surface is smooth and clear, and the outline is clear. **Conclusion.** The printing method is based on ZnO semiconductor nanomaterial ink. It has good application prospects in packaging and printing design.

1. Introduction

Nanotechnology is a developing technology that emerged in the 1990s. It is a new technology for studying the laws and characteristics of electrons, atoms, and molecules in the space of 0.10–100 nanometers (i.e., one billionth of a meter). Because nanotechnology will eventually enable human beings to manipulate individual atoms and molecules according to their own wishes, so as to achieve effective control of the microscopic world, it is considered to be a hot subject that has an extremely important impact on the generation and development of a series of high-tech in the 21st century. It is listed as one of the key technologies of the 21st century by countries all over the world, and a lot of manpower and material resources are invested in its research and development [1]. By the second half of the 1970s, there were advocates for the development of nanotechnology, but most mainstream scientists at the time were still skeptical. From the mid-1970s to the late 1980s, many scientists successively prepared nano-sized materials in the laboratory

and found that these materials had many wonderful properties. At present, nanotechnology mainly includes many fields, such as nanobiology, nanomechanics, nanoelectronics, nanomaterials, atomic and molecular manipulation, and nanomanufacturing. As far as nanomaterials are concerned, due to several major characteristics of nanomaterials, such as small size effect, surface effect, quantum size effect, and macroscopic quantum tunneling effect, some new changes have appeared in the characteristics of sound, light, electromagnetism, and thermodynamics. Therefore, nanomaterials are widely used in various fields, such as the application of nanomaterials in the fields of structural parts, electronic devices, the discipline and field of chemistry, biomedicine and health, textiles, aerospace, sports, agriculture, and other fields [2]. With in-depth research on the preparation, characterization, performance testing, and processing of nanomaterials, its application fields have gradually expanded. From the darling of high technology in the past, it has gradually entered the lives of ordinary people and penetrated into people's clothing, food, housing, and

transportation. It can be said that nanometer technology and nanomaterials have greatly changed human production and lifestyle. Semiconductor photocatalytic materials have outstanding advantages such as high catalytic activity, low cost, low requirements for catalytic conditions, and less repollution and have great application value in the fields of environmental purification and new energy [3]. Among the widely studied semiconductor photocatalytic materials, ZnO has the characteristics of high photosensitivity, a strong driving force for redox reactions, and low cost. It has become one of the core photocatalytic materials in contemporary semiconductor photocatalytic technology. ZnO is a common direct wide bandgap II–VI semiconductor material, with nontoxic, low cost, readily available raw materials, wide bandgap, high exciton binding energy (60 meV), strong radiation resistance, good mechanical, electrical coupling, and other advantages. ZnO has good film-forming properties, and common thin-film preparation techniques can be used to prepare ZnO thin films. However, the traditional ZnO functional film preparation method has problems such as a long production cycle, complicated operation, and large waste of raw materials, and it is difficult to meet the development requirements of miniaturization and precision of electronic products [4].

The author used zinc acetate dihydrate as the zinc source of the zinc-based ink, ethylenediamine as the complexing agent, ethanol as the solvent, and ethyl cellulose as the auxiliary agent in the ink and synthesized a particle-free ZnO ink with stable performance. In this way, the superiority of ZnO semiconductor nanomaterial ink in packaging and printing applications is verified.

2. Literature Review

Research on the preparation of ZnO nanomaterials: Onuki et al. proposed the coprecipitation method. A suitable precipitating agent is added to the electrolyte solution in which various components coexist, and a chemical reaction occurs to generate a homogeneous precipitation material, and high-purity nano-powder materials can be obtained by drying or calcining. The coprecipitation method is more convenient to synthesize zirconium oxide, with low requirements for equipment and synthesis conditions, low cost, and short synthesis time [5]. Xu et al. proposed the effects of Zn²⁺ concentration, calcination temperature, and other conditions on the particle size of ZnO nanocrystals during the preparation process and analyzed the mechanism. Experimental results show that the smaller the reaction concentration, the smaller the crystal grain size can be obtained; under the same other reaction conditions, the grain size of the prepared nano-ZnO particles increases gradually with the increase of the calcination temperature [6]. Das et al. proposed metal-organic chemical vapor deposition. The physical vapor deposition method uses high-temperature heat sources such as arc, high frequency, laser, or plasma to heat oxides to vaporize them and then aggregate them into nanoparticles. Among them, the vacuum evaporation method (i.e., heating under vacuum conditions) is the most commonly used. The chemical vapor deposition

method utilizes volatile metal compounds or elemental metal vapors to generate the desired compounds through chemical reactions, mainly including vapor-phase oxidation and vapor-phase pyrolysis [7]. Bairy et al. proposed the hydrothermal method. The main device used in the hydrothermal method is to create a high temperature and high pressure environment in the reactor, which is very different from the outside world. The crystallization products with good properties can be synthesized by using the temperature difference between the inside and outside of the reactor. Crystals with good crystallinity and small particle size can be directly obtained by the hydrothermal method. The preparation process is also relatively simple, and calcination treatment is not required. The crystalline morphology of ZnO grains is very sensitive to the properties of the medium. The grains grown in pure water solution are long and columnar; in a certain alkalinity solution, the grains are tapered; the alkalinity continued to increase, and the grains became biconical. Temperature also has a certain effect on the crystal size [8]. Li et al. proposed fog thermal decomposition. Using the large pressure difference and temperature difference inside and outside the reaction vessel, the metal salt solution can be hydrolyzed to generate a product with uniform components. Compared with other technologies, spray thermal decomposition technology does not require a high vacuum, which greatly simplifies the process and reduces production costs [9]. Ruan et al. proposed the sol-gel method. Zinc oxide quantum dots and thin films are usually synthesized using the sol-gel method. This method is mainly composed of the following three steps: dispersing the precursor in solution, hydrolyzing into the sol, and finally preparing nanostructured materials by drying or even sintering. Using this method, quantum dots were prepared, the size of the product oxide front was successfully regulated, and the size-dependent shift of the absorption and emission spectra of quantum dots was observed [10].

On the basis of the current research, the authors proposed using zinc acetate dihydrate as the zinc source of the zinc-based ink, ethylenediamine as the complexing agent, ethanol as the solvent, and ethyl cellulose as the auxiliary agent in the ink, and they synthesized the properties of a stable particle-free ZnO ink, and the experimental data show that ZnO has a good application prospect in packaging and printing (Figure 1).

3. Research Methods

3.1. Structure of ZnO Semiconductors. ZnO is an amphoteric white oxide (commonly known as zinc white), and the wurtzite structure of ZnO is stable, so it is more common. ZnO with a wurtzite structure has centrosymmetric but no axial symmetry and dangling bonds appear on the crystal surface due to the fracture. The characteristics of the surface section of ZnO crystals have a slight influence on the overall properties of the crystals, which increase with the decrease of their size and even completely change the original properties of the crystals themselves. Therefore, by controlling the structure of zinc oxide, such as size, morphology or surface crystal plane orientation, surface composition, and surface

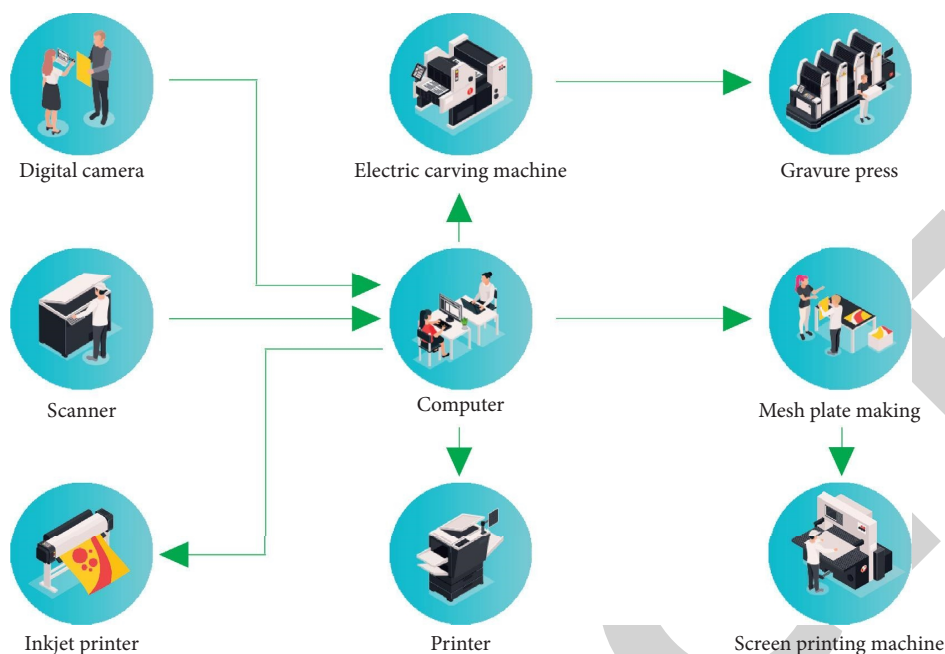


FIGURE 1: Packaging and printing design.

charge, the properties of zinc oxide can be greatly adjusted or changed, thereby obtaining the required luminescence and fluorescence enhancement for applications [11], ferromagnetism, catalytic properties, etc. Among them, micron-sized ZnO has attracted much attention due to its unique properties in the fields of luminescence, photocatalysis, and sensors as shown in Table 1.

3.2. Preparation of Nano-ZnO Arrays. The properties of ZnO strongly depend on its own structure, including grain size, crystal morphology, grain growth orientation, grain aspect ratio, grain density, etc. Meanwhile, the surface properties of ZnO nanoparticles and the packing morphology of the particles also play important roles in many applications. Therefore, it is very meaningful to prepare nano-ZnO arrays with controllable crystal morphology, controllable grain growth orientation, and uniform size. Nano-ZnO arrays have special properties that a single nanomaterial does not possess, so they have broad application prospects, such as sensors, actuators, varistors, catalysts, etc. [12]. There are many ways to prepare nanomaterials, such as the various methods introduced in the previous section; although many nanomaterials with various structures and properties have been synthesized by these methods, there are still some problems, such as the difficulty of particle size and shape, control, disordered arrangement of particles and harsh preparation conditions. Therefore, it is difficult in the field of nanomaterials research to explore a method that can easily prepare particles with controllable size and shape, as well as particle arrangement and order. At present, there are two main methods for preparing well-arranged nanostructure arrays, namely, template synthesis and seeded growth. The template method is more and more mature in research at home

and abroad. The seed crystal growth method is a new process method developed in recent years [13].

3.2.1. Template Method (Template Synthesis). The template method is to introduce monomers, polymer solutions, or melts into the nanopores of the template and obtain structural regularity by chemical or physical methods (electrochemical polymerization, chemical polymerization, polymer solution, and polymer melt methods), resulting in neatly arranged nanomaterials. The template synthesis method can prepare one-dimensional nanomaterials with uniform size, regular arrangement, and adjustable structure, which can effectively prevent the agglomeration of nanomaterials. The particle size and particle size distribution of nanoparticles prepared by the template synthesis method are closely related to the pore size and distribution of the template. At the same time, the formation of nanostructure arrays is also closely related to the ordering of the template. Therefore, template selection and preparation are critical. Templates only provide a mesoscopic environment for the formation of nanomaterial arrays. Nanoparticles also require other synthetic methods to prepare them, such as electrochemical deposition, electroless plating, chemical deposition, sol-gel, etc. [14]. The formation of templates on nanoparticle arrays has certain advantages. However, using certain synthetic methods to prepare nanoparticles, their morphology is not within the control range.

3.2.2. Seed Growth. The seed growth method is a new process method developed in recent years, which can effectively control the grain shape of ZnO nanoparticles and, at the same time, prepare well-arranged ZnO nanoparticle arrays. The basic idea of the seed growth method is divided into three steps: the first step is to select a suitable substrate.

TABLE 1: Properties of zinc oxide.

Chemical formula	Molar mass	Exterior	Odour	Density	Melting point	Solubility	Energy gap	Refraction
ZnO	81.4084 g·mol ⁻¹	White solid	Tasteless	5.606 g·cm ⁻³	1975 °C (break down)	Insoluble: 0.0004 g/100 mL (17.8°C)	3.37 eV	2.0041

In the second step, different preparation methods were used to randomly grow ZnO on the substrate covered with the crystal seed film. At this stage, the growth orientation and morphology of ZnO nanoparticles are not controlled [15]. In the third step, after the random growth of ZnO nanoparticles on the substrate, due to the crystal habit and spatial confinement effect of ZnO nanoparticles, ZnO nanoparticles grow in a specific direction on this basis to form nano-ZnO columnar arrays. The control of the morphology of ZnO nanoparticles is controlled by the choice of external catalysts and raw materials [16].

3.3. Preparations

3.3.1. Ink Preparation. First, 0.006 mol of ethylenediamine complexing agent, 15 mL of ethanol, and 8 μ L of ethyl cellulose auxiliary were mixed evenly, and 0.002 mol of zinc acetate dihydrate was weighed on an electronic balance and then was added to the mixed solution; we continued to stir and dissolved it for 1 h and then filtered with a 0.22 μ m microporous membrane to obtain a particle-free ZnO ink for inkjet printing [17].

3.3.2. ZnO Thin Film Preparation. The glass and PI substrates were ultrasonically cleaned with acetone for 5 min to remove oil stains on the surface of the substrates, then cleaned with deionized water, and dried for hydrophilic treatment before use. The ink was spin-coated on a 2 cm glass sheet, heated to different temperatures, and held for 30 min to form a thin film; the particle-free ZnO ink prepared by a commercial Epson inkjet printer was printed on a PI substrate and heat-treated at 300°C for 30 min on a heating plate [18].

3.3.3. Sample Characterization. The phase of the ZnO thin film was tested by a SmartLab X-ray diffractometer (Cu target, 40 kV/200 mA). The surface morphology of ZnO thin films was measured by JSM-7001F field emission scanning electron microscope [19]. Using NicolettiS5 infrared spectrometer (FTIR) combined with attenuated total reflection iD7ATR (attenuated total reflection) test ZnO thin film surface functional groups [20], the thermal decomposition behavior of ZnO ink and precursor was tested by the STA449F3 synchronous thermal analyzer. The transmittance of the film was measured with a Lambda750S UV-Vis spectrophotometer. The contact angle of the ink was tested with an SL200KS type contact angle tester. Atomic force microscopy (AFM) was used to test the roughness of the films after inkjet printing. A JEM-2100 transmission electron microscope (TEM) was

used to test the morphology, diffraction spots, and high-resolution images of ZnO particles in the films [21]. The viscosity of the ink was measured at a shear rate of 26.4 s⁻¹ using an LVDV-II + procoaxial cylindrical viscometer with a small sample cell. The electrical properties of ZnO films were measured by an HMS-5000 automatic variable temperature Hall effect tester.

4. Analysis of Results

4.1. Thermal Decomposition Behavior of Particle-free ZnO Ink.

Figure 2(a) shows the TG-DSC curves of zinc acetate dihydrate precursor and particle-free ZnO ink (air atmosphere, heating rate of 10°C/min). In order to ensure that the decomposition temperature of zinc amine precursor can be seen during thermal analysis, in the experiment, the amount of solvent was adjusted to 1 mL. As can be seen from Figure 2(a), the thermal decomposition process of zinc acetate dihydrate is divided into three steps: The first step is from room temperature to 110°C, which corresponds to the endothermic peak at 105.7°C on the DSC curve, and the mass loss is 15.3%, which is caused by the loss of crystal water from zinc acetate dihydrate; the second step is 110~260°C, an endothermic peak of 255.5°C appears on the DSC curve, and its mass loss is 0, which corresponds to the melting process of zinc acetate from solid to liquid; the third step is 260~350°C, DSC. The curve appeared to have an endothermic peak at 330.8°C, and the mass loss on the TG curve was 47.1%, which was the process of the decomposition of zinc acetate into ZnO.

Figure 2(b) shows that the behavior of particle-free ZnO ink during heating is much more complex and can be divided into 5 steps: The first step is from room temperature to 80°C, and the mass loss on the corresponding TG curve is 4.6%, which is due to the loss of crystal water by zinc acetate dihydrate; The second step is 80~100°C, with the endothermic peak at 94.4°C of the DSC curve. The corresponding mass loss is 51%, which is caused by a large amount of ethanol volatilization; The third step is 100~120°C, corresponding to the endothermic peak at 100.1°C of the DSC curve, and the mass loss is 6%, corresponding to the volatilization of uncomplexed ethylenediamine; the fourth step is 120~160°C, accompanied by an endothermic peak at 154.5°C, and its mass loss is 0, which is due to the melting of the zinc amine precursor; The fifth step is 160~220°C, and the DSC curve is at 176.7. There is one absorption peak at °C, and the mass loss is 28.3%, which corresponds to the process of the decomposition of the precursor and the formation of ZnO; after the sixth step at 220°C, there is one endothermic peak at 349.8°C, with no mass loss, which corresponds to the crystallization process of ZnO.

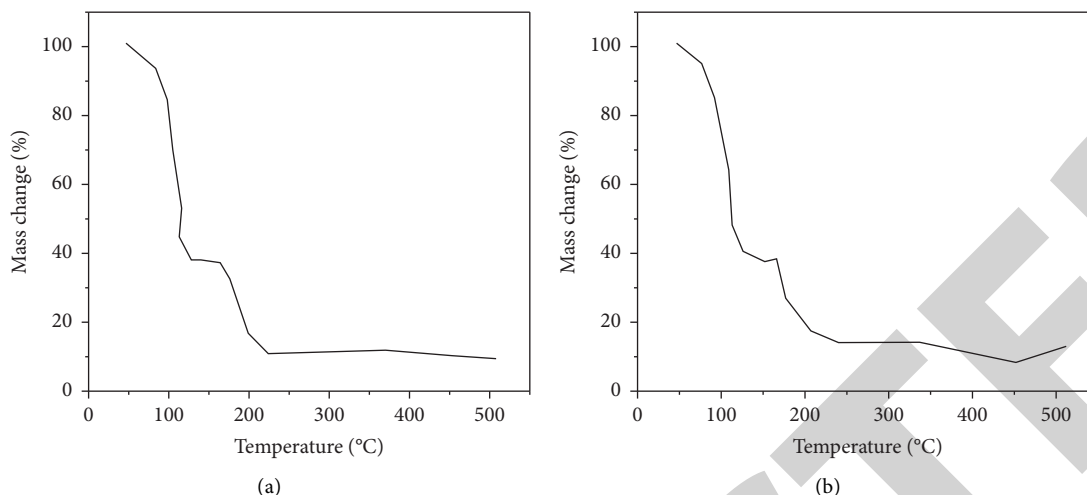


FIGURE 2: TG-DSC curves of zinc acetate dihydrate precursor and particle-free ZnO ink. (a) Zinc acetate dihydrate. (b) Particle-free ZnO ink.

4.2. Film-forming Properties of Particle-free Zinc-Based Conductive Ink.

Figure 3 shows the XRD spectra of the thin films of ZnO inks dried naturally at room temperature and heat-treated at 100, 200, 250, 300, 400, 600, and 800°C for 30 min, respectively. Combined with the thermal analysis results in Figure 2, the thermal decomposition temperature of the particle-free zinc-based ink is 176.7°C, so it can be seen from XRD analysis that the film that is naturally dried at room temperature and 100°C is still a crystalline precursor, and ZnO is not formed. The characteristic diffraction peaks of (100), (002), (101), (102), (110), (103), and (112) crystal planes of ZnO appeared in the thin film samples treated at 200~800°C, which were judged as standard by analysis. ZnO with hexagonal wurtzite polycrystalline structure. The intensity of diffraction peaks of the films increases with the increase of heat treatment temperature, indicating that increasing heat treatment temperature can increase the crystallinity of the films. During the low-temperature period of 200~300°C, the diffraction peak intensity of the (002) crystal plane in the film is the largest, the reason may be that the (002) plane has lower surface energy, and the film grains mainly grow along this plane, the (101) plane in the film gradually becomes the strongest diffraction peak, indicating that the (101) plane in the film may have lower surface energy at high-temperature, and the crystal grains in the film mainly grow along this plane [22].

The double peak at $3500\sim 3000^{-1}$ is the vibrational stretching peak of the $-\text{NH}_2$ functional group. The infrared spectrum 450^{-1} at 200°C and above can be considered as the characteristic absorption peak of the Zn—O bond, indicating that when the heat treatment temperature is 200°C, ZnO has been formed in the film. Among them, the absorption bands at 1588 and 1430^{-1} in the spectral lines at 200 and 250°C are caused by the antisymmetric stretching vibration and stretching vibration of C=O in the carboxyl group. Therefore, it can be judged that the precursor is not completely decomposed, the 3426^{-1} peak can be identified as being caused by the stretching vibration of hydroxyl groups

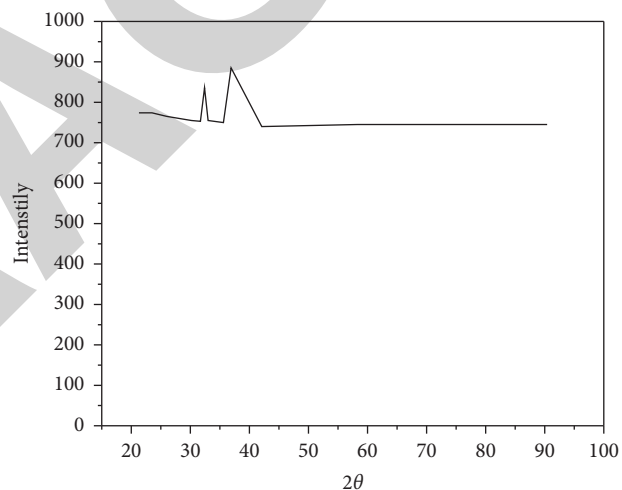


FIGURE 3: XRD spectra of the films obtained after heat treatment of particle-free ZnO inks at different temperatures for 30 min.

of adsorbed water in the sample. When the temperature is higher than 300°C, there is only the (Me-O) peak of Zn-O except for the vibration stretching peak of adsorbed water, indicating that there is no impurity functional group in the film after the temperature is higher than 300°C.

4.3. Printing Performance of Particle-free ZnO Ink on PI Substrate.

Printed on an inkjet printer using particle-free ZnO ink, and SEM images of patterns ($300\ \mu\text{m}$) and lines with different line widths after heat treatment at 300°C for 30 min. The microstructure of the printed pattern is dense, the edge is complete, and the outline is clear. For inkjet printed lines, when the line width of the line is small ($50\ \mu\text{m}$), the edge is slightly broken, mainly because when the line is small, in order to improve the resolution, the distance between the two nozzles of the printer will become larger, and the volume of the droplet ejected by the nozzle will become

TABLE 2: Resistivity, mobility, carrier concentration, and Hall coefficient of the films after heat treatment at 300°C for 30 min.

T/°C	$\rho/(\Omega * cm)$	$\mu/(cm^2 * V^{-1} * S^{-1})$	n/cm^{-3}	$H/(cm^3 * C^{-1})$
300	4.98×10^2	1.78	7.06×10^{15}	-8.8×10^2

smaller, due to the low viscosity of the ink, the wetting angle between the ink and the substrate is small. When the droplet falls on the substrate, it will spread out completely, the entire line is connected by the diffusion between the droplets; When the droplet volume is small, the diffusion is not complete and the edge of the line is more broken, when the line width is larger, the edge of the line is more complete and the outline is clear, mainly because the distance between the nozzles decreases and the volume of the ink droplets becomes larger, and the fusion between the ink droplets is sufficient to make the edge of the line clear and complete. The roughness of the film obtained by inkjet printing and heat treatment at 300°C is 15.2 nm, indicating that the surface is extremely smooth. The resistivity, mobility, carrier concentration, and Hall coefficient of the zinc oxide film after heat treatment at 300°C for 30 min are shown in Table 2.

The above experimental results show that

- (1) A particle-free ZnO functional ink was prepared by using zinc acetate dihydrate, ethylenediamine, anhydrous ethanol, and ethyl cellulose. The thermal decomposition temperature of the ink (176.7°C) can be achieved by a complex reaction between zinc ions and amine groups, much lower than the thermal decomposition temperature of zinc acetate (330.8°C).
- (2) The ink is spin-coated on the glass substrate and cured at different temperatures for 30 minutes. The film can generate ZnO with a hexagonal wurtzite polycrystalline structure when the heat treatment temperature is greater than 200°C, and the transmittance of the film is greater than 80%, among which the thin film prepared at 300°C has the tightest connection between particles and the least pores.
- (3) After inkjet printing on the PI flexible substrate and heat treatment at 300°C for 30 min, the printed pattern has a dense microstructure with complete edges and clear outlines. Inkjet prints lines with different line widths. When the line width is smaller (50 μ m), the edge is slightly broken. When the line width is larger, the edge of the line is relatively complete and the outline is clear. The surface roughness of the pattern after inkjet printing is only 15.2 nm, which has a good application prospect in printed electronics.

5. Conclusion

In summary, the decomposition temperature of the particle-free ZnO conductive ink studied by the author is much lower than that of the zinc acetate precursor; the film cured at 300°C for 30 min has a smooth surface, uniform particle size, good crystallinity, and transmittance of up to 80%. After inkjet printing on the PI flexible substrate, after curing at

300°C for 30 min, the pattern surface is smooth and clear, and the outline is clear. The printing method based on ZnO semiconductor nanomaterial ink has good application prospects in packaging and printing design, can be applied to flexible substrates, can prepare large-area zinc oxide films and complex zinc oxide circuits, and can be applied to solar cells and transparent conductive films, gas sensors, and other devices.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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