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Doping of 1,1-dicyano-2,2-bis(methylthio)ethylene in ZnO to improve photoresponse of organic solar cells and photodetectors under UV-cut light irradiation

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ZnO has been utilized as the electron transport layer in organic optoelectronic devices, however, the shortcoming of ZnO is that activation by UV light is essential and ZnO-based optoelectronic devices give poor photoresponse under UV-cut light irradiation. We developed 1,1-dicyano-2,2-bis(methylthio)ethylene (DBE)-doped ZnO which can be activated by visible light irradiation. The DBE-doped organic solar cells showed better photovoltaic performances under UV-cut light irradiation than those of undoped ZnO-based devices and DBE-doped photodetectors also showed better frequency response without applied voltage under monochronic laser irradiation (450 nm) than that of undoped ZnO-based ones.

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1. Introduction

Organic optoelectronic devices, such as organic solar cells (OSCs) and organic photodetectors (OPDs), have recently attracted much attention as next-generation optoelectronic devices owing to properties that can overcome the limitations of their inorganic counterparts, lightweight, mechanical flexibility, and amenability to printed devices.^{1–3} Some organic optoelectronic devices contain zinc oxide (ZnO) for the electron transport layer (ETL) because of its features such as high electron mobility and hole-blocking nature, fine transparency in visible and NIR light, and ease of thin-film preparation by the sol-gel method using readily available and cheap precursor materials.^{4–6} Moreover, ZnO exhibits good thermal and chemical stability under ambient conditions compared with traditional ETL materials for OSCs and OPDs, such as LiF and Ca,^{7,8} which gives improved device durability and easier handling under ambient conditions to those organic optoelectronic devices.^{9–11} This should accelerate the social implementation of OSCs and OPDs.

ZnO has several shortcomings in the utilization of organic optoelectronic devices: (i) preparation temperature (~200 °C) which is higher than the mp of commercially available plastic substrates, and (ii) photocatalytic activity that can decompose organic active materials. Many researchers have studied ways to overcome both shortcomings and reported effective methods for organic optoelectronic devices.^{12–16} However, ZnO has another issue to overcome; ZnO requires activation by UV light for smooth carrier transfer and ZnO-based optoelectronic devices showed poor photoresponse under UV-cut light irradiation.^{17–20} This is an important issue to be solved because it is reported that photo-irradiation without UV light gives a distorted current–voltage trace for OSC and OPD characteristics. The unsmooth carrier transport in optoelectronic devices with unactivated ZnO by UV light should be originated by the impossibility of hole transport from ZnO to the electron acceptor or donor materials after

charge separation [Fig. 1(a)].^{17,18} In the usual case of small-scale experiments, ZnO is activated by UV light, and a hole carrier is generated in ZnO, which can give smooth carrier transportation from the ZnO to organic active materials in organic optoelectronic devices [Fig. 1(b)]. However, there are different conditions for the actual use of the organic optoelectronic devices; OSCs should be covered by UV-cut films to avoid degradation of active layer materials and OPDs usually be irradiated by monochromatic optical laser without UV light.^{21–24} To overcome this issue, chemical doping for ZnO could be effective [Fig. 1(c)].

In this paper, we described the preparation of doped ZnO with visible light absorption and demonstrated its use for OSCs and OPDs. 1,1-Dicyano-2,2-bis(methylthio)ethylene (DBE)²⁵ was used as a dopant for the preparation of doped ZnO films [Fig. 1(d)]. DBE-doped ZnO exhibits weak absorption at ca. 400–500 nm and the DBE-doped ZnO-based OSCs and OPDs showed better photoresponse under UV-cut light irradiation than that of ZnO-based OSCs.

2. Experimental methods

2.1. Materials

1,1-Dicyano-2,2-bis(methylthio)ethylene (DBE) was synthesized by a previously reported procedure.²⁵ Zinc acetylacetonate hydrate, regioregular P3HT (MW 42,000), and 1,2-dichlorobenzene (DCB) were purchased from Sigma-Aldrich, Japan. A PEDOT:PSS dispersion in water (Clevios P) (1.3 wt%) was purchased from H. C. Starck. The indium tin oxide (ITO) substrates (sheet resistance = 10 Ω sq.) and Au (99.995%) were purchased from Furuuchi Chemical Corporation. PC₆₁BM was purchased from Frontier Carbon Corporation, Japan.

2.2. Fabrication of ZnO films with and without DBE

An ITO-coated glass substrate was cleaned by ultrasonication in isopropanol and following UV/ozone treatment for 30 min. The precursor solution for ZnO was prepared by dissolving Zn(AcAc)₂ (125 mg) in 2-methoxyethanol (1.10 ml) with

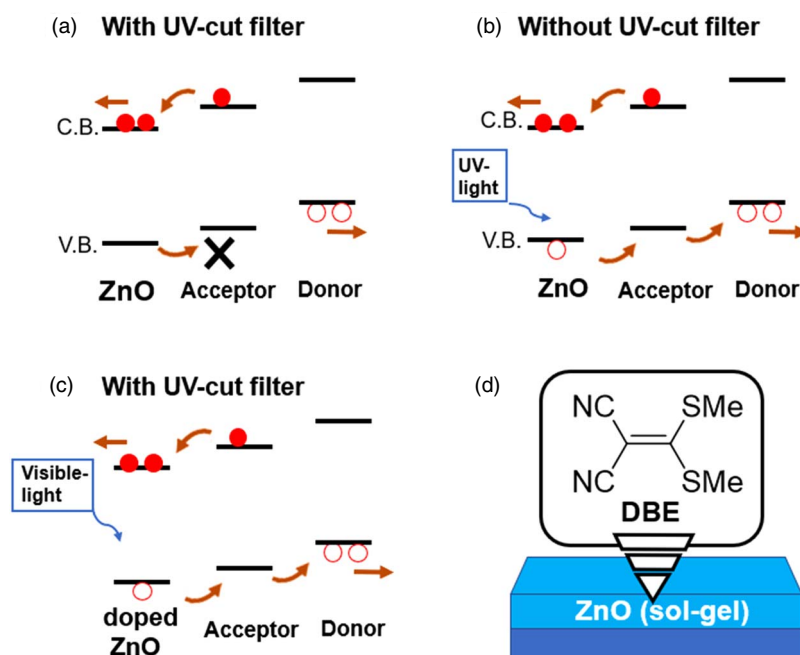


Fig. 1. Illustration of carrier transfer under irradiation of solar light without (a) and with UV-cut filter (b) between donor:acceptor and ZnO in organic optoelectronic devices. Carrier transfer between donor:acceptor and doped ZnO (c) under irradiation of solar light with UV-cut filter. The chemical structure of DBE (d).

140 ml of acetyl acetone, and the precursor solution for ZnO with DBE was prepared by dissolving $\text{Zn}(\text{AcAc})_2$ (115 mg) in 2-methoxyethanol (1.25 ml) with 74 mg of DBE. The precursor solutions were stirred vigorously at 60 °C for 2 days under Ar atmosphere. ZnO films were prepared by spin coating of the precursor solution (1000 rpm, 60 s) and subsequent thermal annealing for 60 min at 250 °C.

2.3. Fabrication of optoelectronic devices

An *o*-dichlorobenzene (ODB) solution of the active materials (regioregular P3HT and PC_{61}BM , 1.25:1, 30 g l⁻¹) was spin-coated onto the prepared ITO/ZnO substrate at 700 rpm for 40 s. After the spin coating, the resulting film (thickness; ~200 nm) was solvent-annealed for 40 min in a closed container with ODB. A PEDOT:PSS (Clevios® P) hole transporting layer was fabricated by spin coating at 2000 rpm for 60 s with a patterned surface protective film (Nitto Denko Corporation, E-MASK TP200). An Au electrode was deposited by thermal evaporation under vacuum (approximately 10⁻² Pa) through a metal mask (active area: 10 mm²). Finally, the device was covered with sealing film (Cellel, Kureha Extech). All fabrication steps except the Au deposition step were performed in air (the relative humidity: ~30%).

The photovoltaic properties of the device were measured under simulated solar illumination (AM1.5, 100 mW cm⁻²) from a solar simulator based on a 150 W Xe lamp. The light source was a SAN-EI Electric XES-40S1 solar simulator, which was calibrated with a standard silicon photovoltaic detector. The *J*-*V* measurements were performed under ambient conditions with and without a UV-cut filter ($\lambda > 390$ nm). The active area of the device was defined by using a 10.0 mm² photo mask.

The properties of the photodetection of the devices were characterized by using a NIR laser with a wavelength of 450 nm. The light from a laser (Thorlabs LPS-4224-830-FC) was collimated by using a fiber collimator (Thorlabs,

F240FC-B) and was illuminated from the top of the devices. The illuminated light was a Gaussian profile with a beam diameter of 2 mm. The current-voltage characteristics of the devices were measured by a digital electrometer (ADCMT, 8252), and the frequency response of the devices was measured by the network analyzer function of a USB-based multi-function instrument (Digilent, Analog Discovery 2). The laser was intensity-modulated in the frequency range from 10 Hz to 100 kHz, and the current was converted to a voltage by a 50 Ω resistor followed by a voltage amplifier with a voltage gain of 20 and a bandwidth of 20 MHz (Turtle Industry, T-WBA01Z).

2.4. Analytical measurements

XPS analysis was performed by using the Shimadzu AXIS-ULTRA DLDXPS apparatus. Absorption spectra of the ZnO films were measured using Agilent Technologies Cary 8454 UV-vis. The photoelectron spectra in air (PESA) were measured by using AC-2 (RIKEN Keiki Co.).

3. Results and discussion

ZnO films were prepared basically by following a previously reported procedure, that is, the sol-gel method using zinc acetylacetonate hydrate [$\text{Zn}(\text{AcAc})_2$].^{26–28} Although $\text{Zn}(\text{AcAc})_2$ was poorly soluble in 2-methoxyethanol, the solvent used in the sol-gel method, its solubility was improved by the addition of a small amount of acetylacetone. We speculated that $\text{Zn}(\text{AcAc})_2$ and acetylacetone would react in 2-methoxyethanol to generate a tris(acetylacetonato) complex that is soluble in the 2-methoxyethanol solution [Fig. 2(a)]. Then, spin coating on the substrates and annealing would give uniform ZnO films. In the case of DBE-doped ZnO, DBE was added in place of acetylacetone in the 2-methoxyethanol solution of $\text{Zn}(\text{AcAc})_2$. We selected DBE as the dopant for obtaining ZnO that can be activated by visible light because DBE is a small π -conjugated molecule with a good electron-accepting nature and sufficient

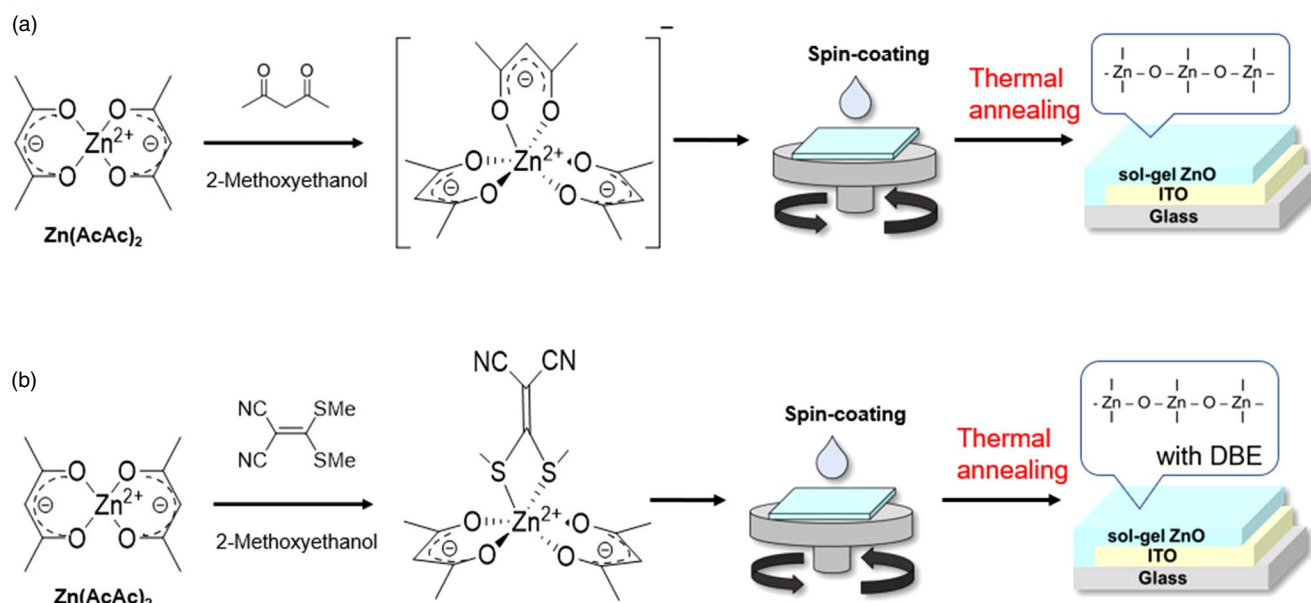


Fig. 2. Fabrication of ZnO (a) and DBE-doped ZnO (b) films from $\text{Zn}(\text{AcAc})_2$ precursor.

solubility in 2-methoxyethanol. After stirring at 60 °C for 2 days, a yellow precursor solution was obtained. We speculated that DBE coordinated to $\text{Zn}(\text{AcAc})_2$ and thermal annealing gave the DBE-doped ZnO film [Fig. 2(b)]. The DBE-doped ZnO film was further studied by X-ray photoelectron spectroscopy (XPS) measurements (Fig. 3). The DBE-doped ZnO film showed a spectral component of Zn $2p_{3/2}$ at 1022.2 eV and two spectral components of O 1s at 531.8 and 530.3 eV [Figs. 3(a), 3(b)], which were fitted by a Gaussian/Lorentzian deconvolution. The similarity of these XPS component peaks to those of reported sol-gel ZnO component peaks^{26,27,29} meant that ZnO was generated by using DBE instead of acetylacetone. The slight shift of the component peak of Zn $2p_{3/2}$ (1022.2 eV) from the reported value (1021.7 eV) implies the coordination of S atoms of DBE to the Zn atom because it is reported that the XPS peak of S-bound Zn is slightly larger than that of O-bound Zn.³⁰ Furthermore, the XPS component peak of S $2p_{3/2}$ of the DBE-doped ZnO film (167.5 eV) was shifted from that of drop-cast DBE film (165.7 eV), which also suggests the coordination of the S atoms of DBE to the Zn atom [Fig. 3(c)]. In addition, the XPS component peak of N 1s for the DBE-doped ZnO film was detected at 399.9 eV in the cyanide region, which means that the cyano moieties of DBE remained in the ZnO film [Fig. 3(d)]. However, the peak shift from that of drop-cast DBE film (398.1 eV) implies that the cyano moieties of DBE also coordinated to the Zn atom.^{31,32}

We measured the absorption and the PESA spectra of DBE-doped and undoped ZnO films (Fig. 4). The DBE-doped ZnO film exhibits a weak absorption in the visible-light region (ca. 400–500 nm), whereas the undoped ZnO shows no absorption in the visible-light region. As regards the ETL of inverted-type OSCs, weak absorption is favorable because the ETL should have good transparency for visible light.^{29,33} In contrast to the undoped ZnO film, the DBE-doped ZnO film showed photoelectron emission in PESA (5.75 eV). This indicates that the DBE-doped ZnO film has an energy state that can realize hole transfer from ZnO to active layer materials.

To confirm the carrier transport of DBE-doped ZnO-based optoelectronic devices without UV activation, OSCs and OPDs were fabricated according to reported procedures (ITO/ZnO/P3HT:PCBM/PEDOT:PSS/Au/barrier film).^{26–28} P3HT and PCBM are utilized as active layer materials because their properties for optoelectronic devices are guaranteed and they are affordable and readily available.^{34,35} The DBE-doped ZnO film is not appropriate for use in optoelectronic devices because the film has low uniformity (Fig. 5). Thus, DBE-doped ZnO films for the optoelectronic devices were prepared from a mixed precursor solution of undoped ZnO and DBE-doped ZnO (9:1). Figure 6 and Table I show the current–voltage characteristics and photovoltaic properties of DBE-doped and undoped ZnO-based OSCs under simulated solar light irradiation with and without a UV-cut filter ($\lambda > 390$ nm). The DBE-doped ZnO-based OSC showed similar typical current–voltage characteristics to the undoped ZnO-based device under simulated solar light irradiation with UV light. This implies that the electron transport properties and the hole-blocking nature of the DBE-doped ZnO film as ETL are not different from those of the undoped ZnO film. Despite the visible-light absorbing nature of the DBE-doped ZnO film, the short circuit current (J_{SC}) value of the DBE-doped ZnO-based OSC was similar to that of the undoped ZnO-based device. This should be due to the weak absorption nature of the DBE-doped ZnO film in the visible light region and the small thickness of the ETL film (ca. 30 nm). Under UV-cut light irradiation, the current–voltage curve of the undoped ZnO-based OSC showed distortion in the first quadrant, whereas that of the DBE-doped ZnO-based OSC did not. The fill factor (FF) of the DBE-doped ZnO-based OSC (ca. 0.61) is higher than that of the undoped ZnO-based device (ca. 0.52) under UV-cut light irradiation. This means that the DBE-doped ZnO film can be activated by visible light irradiation, which should be beneficial for OSCs with UV protection coating to improve photodurability.

The photodetection properties of undoped and DBE-doped ZnO-based OPDs were also measured under monochromatic laser irradiation at 450 nm (2.0 mW) (Fig. 7). The current–

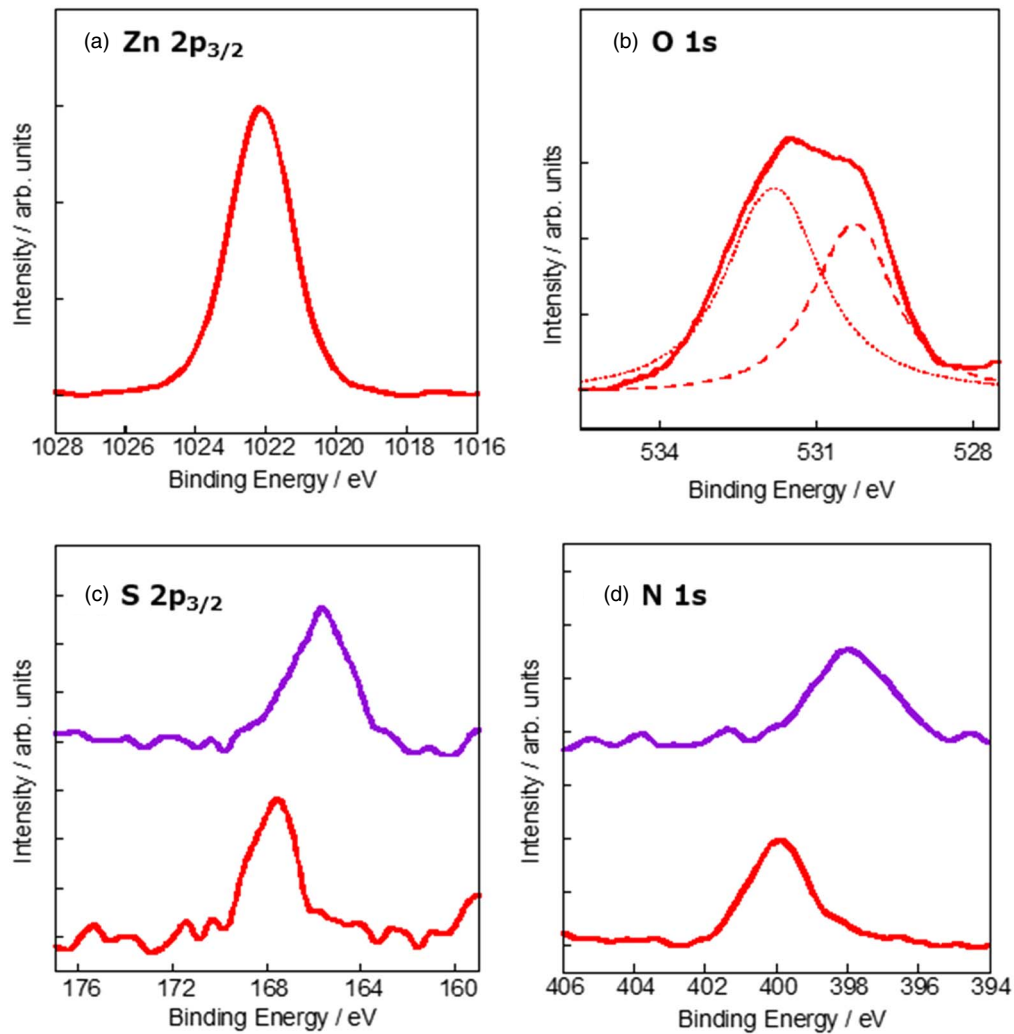


Fig. 3. XPS spectra of Zn 2p_{3/2} (a), O 1s (b), S 2p_{3/2} (c), and N 1s (d) for DBE-doped ZnO film (red trace) and drop-cast DBE film (purple trace).

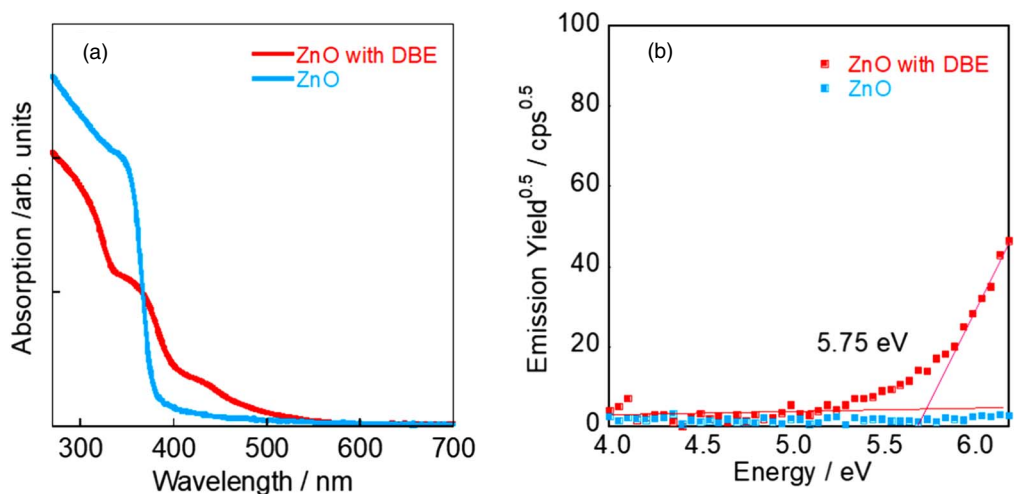


Fig. 4. Absorption spectra (a) and photoelectron spectra in air (b) of undoped ZnO and DBE-doped ZnO films.

voltage curves of undoped and DBE-doped ZnO-based OPDs are shown in Figs. 6(a) and 6(b). The current–voltage curve of the undoped ZnO-based device clearly showed distortion, unlike that of the DBE-doped ZnO-based OPD, suggesting that the DBE-doped ZnO film was activated by the monochromatic laser irradiation. The distorted current–voltage curve indicates poor photoresponse of the undoped ZnO-

based device. In fact, the frequency response without applied voltage of the undoped ZnO-based device was lower than that of the DBE-doped ZnO-based OPD [Figs. 7(c) and 7(d)]; the cut-off frequency ($f_{-3\text{dB}}$), which is defined as the frequency in which the output signal is decreased by 3 dB, of the undoped ZnO-based device is 13.8 kHz, whereas that of the DBE-doped ZnO-based OPD is 21.1 kHz. Although

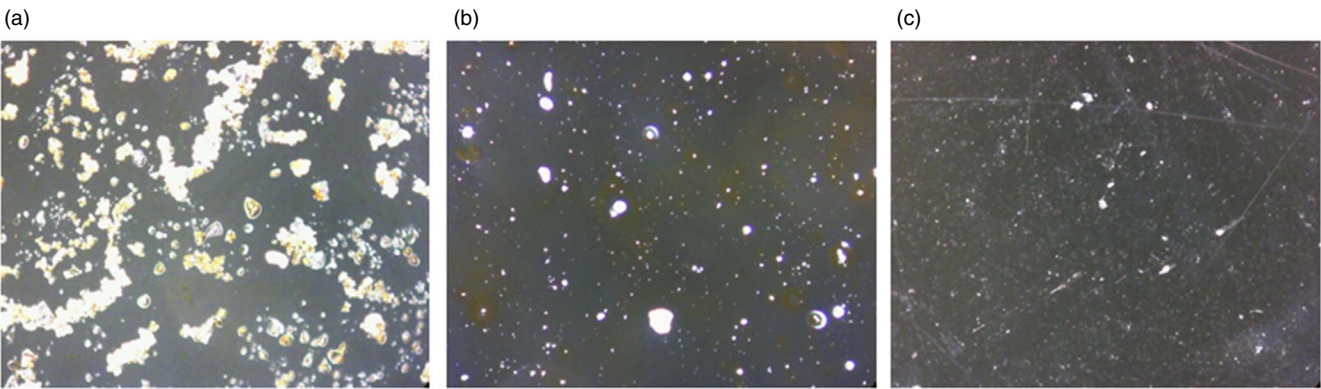


Fig. 5. Optical microscopic images (x100) of ZnO films with DBE (a) and DBE-doped ZnO films prepared from a mixed precursor solution of undoped ZnO and DBE-doped ZnO (b: 3:1 and c: 9:1).

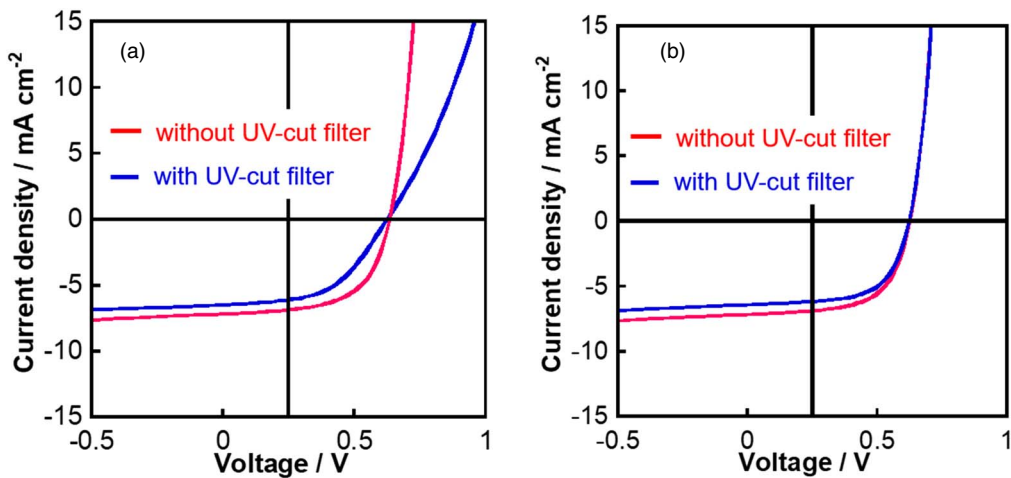


Fig. 6. Current–voltage characteristics of OSCs composed of undoped ZnO (a) and DBE-doped ZnO (b) under simulated solar light irradiation with and without UV-cut filter.

Table I. Photovoltaic properties of OSCs composed of undoped ZnO and DBE-doped ZnO under simulated solar light irradiation with and without UV-cut filter.

ETL material	UV-cut filter	$J_{SC}/\text{mA cm}^{-2}$	V_{OC}/V	FF	PCE/%
ZnO	—	7.1 (± 0.1)	0.61 (± 0.01)	0.60 (± 0.02)	2.7 (± 0.1)
	○	6.2 (± 0.1)	0.61 (± 0.01)	0.52 (± 0.04)	2.1 (± 0.3)
DBE-doped ZnO	—	7.0 (± 0.3)	0.61 (± 0.02)	0.61 (± 0.01)	2.7 (± 0.2)
	○	6.3 (± 0.2)	0.61 (± 0.01)	0.61 (± 0.02)	2.4 (± 0.1)

the cut-off frequency values of the two devices at various applied voltages (≥ 0.4 V) are similar (ca. 21 kHz), photo-detection without voltage application is more valuable than that with voltage application. Together, the results indicate that DBE doping is effective in overcoming one of the shortcomings of ZnO as an ETL material for optoelectronic devices.

4. Conclusions

We have prepared DBE-doped ZnO films and used them for the photoresponse improvement of OSCs and OPDs composed of ZnO-based ETLs under UV-cut light irradiation. The DBE-doped ZnO film exhibited visible light absorption at ca. 400–500 nm, and its ionization potential was 5.75 eV. These data suggest that the DBE-doped ZnO film can realize smooth hole transfer from ZnO to active layer materials by

visible light activation. The DBE-doped ZnO-based OPV and OPD did not show distorted current–voltage curves under UV-cut light and monochromatic laser (450 nm) irradiation. In addition, the DBE-doped ZnO-based OPD showed better frequency response without applied voltage than the undoped ZnO-based device. These results should be beneficial for the practical application of ZnO-based optoelectronic devices because the shortcoming of ZnO film, that is, the need to be activated by UV light, has been little studied compared with other shortcomings of ZnO films, i.e. their high preparation temperature and high photocatalytic activity. The DBE doping might possibly be effective in other ETL materials-based OSCs that show distorted J – V characteristics.³⁶⁾ Moreover, DBE doping may confer a visible light activation nature to other metal oxides, thereby leading to the development of interesting materials for optoelectronic devices.

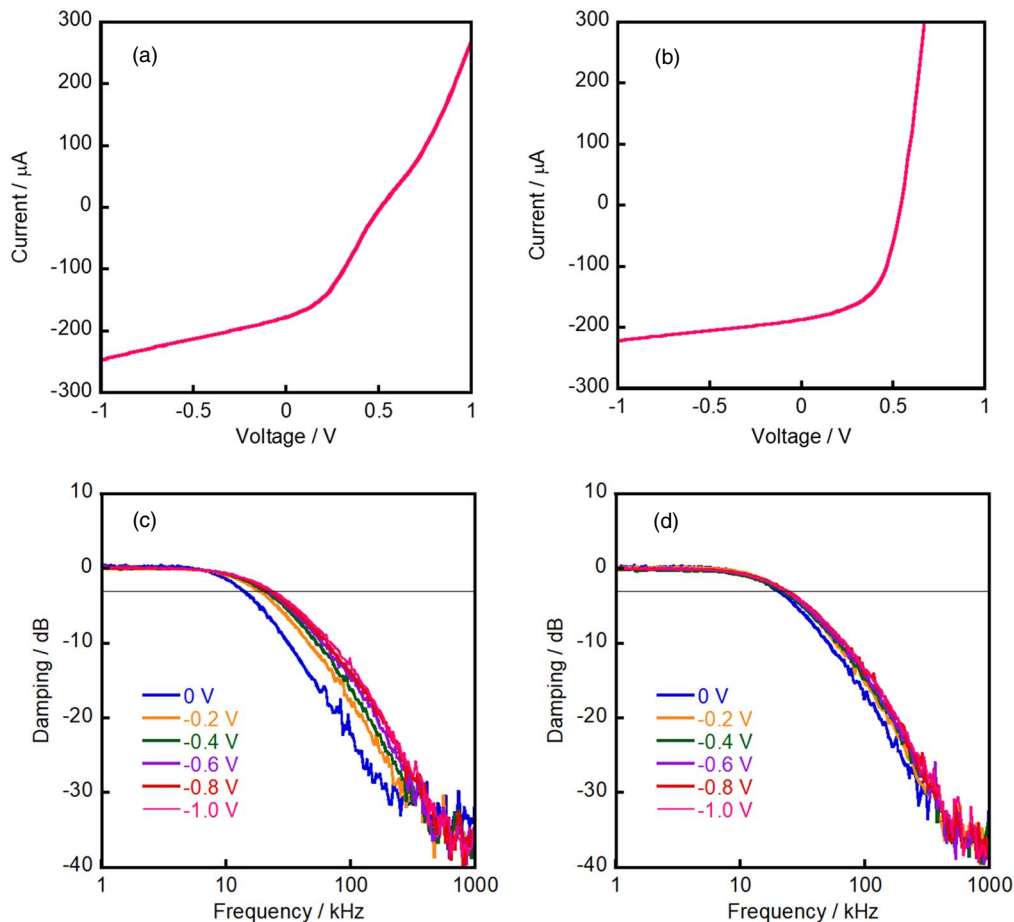


Fig. 7. Current–voltage characteristics of OPDs composed of undoped ZnO (a) and DBE-doped ZnO (b) (illuminated optical power: 2.0 mW). Frequency response dependence on the applied voltage of OPDs composed of undoped ZnO (c) and DBE-doped ZnO (d).

Further study and development of DBE doping for ZnO and other metal oxides for optoelectronic devices are underway in our laboratory.

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