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ABSTRACT

Cu-doped ZnSe/ZnS/L-Cys core–shell QDs are prepared by both nucleation doping and growth doping in an aqueous synthesis method. Transport of photogenerated free charge carriers (FCCs) in these Cu-doped QDs is probed via a combination of surface photovoltaic (SPV), photoacoustic (PA), and electric-field-induced SPV techniques, supplemented by the UV–VIS absorption spectrum and Raman spectrum. The results confirm that the two doping mechanisms result in different doping locations and microelectronic structures of the Cu-doped QDs. The distinctive microelectronic structure of the QDs prepared by nucleation doping, as compared with those prepared by growth doping, results in a number of favorable SPV characteristics. For example, the QDs prepared by nucleation doping exhibit a higher SPV response intensity at 600 nm because of a higher concentration of photogenerated FCCs. The ratio of the strongest SPV response and the strongest PA signal of the QDs prepared by nucleation doping is up to 2.41 times greater than those of the QDs prepared by growth doping. This is because the greater numbers of photogenerated FCCs in the QDs prepared by nucleation doping generate the PV effect rather than the PA effect that is caused by a nonradiative de-excitation process. The position of the shoulder peak of the SPV response at a long wavelength of the QDs prepared by nucleation doping is significantly red-shifted compared with that of the QDs prepared by growth doping, leading to a broader SPV response range in the visible region. The QDs prepared by nucleation doping have a more obvious donor feature than those prepared by growth doping.

I. INTRODUCTION

In the past two decades, group II/VI semiconductor nanocrystals (i.e., quantum dots, QDs) have been extensively investigated because of their unique microelectronic structure and optical and photoelectric properties. They have had a variety of applications to date in solar cells, light-emitting diodes, photonic crystals, and biomarkers. However, some problems are encountered in their practical use, such as the high toxicity of Cd-containing QDs and the photoluminescence self-quenching of some pure QDs. Therefore, efforts are being made to avoid the use of cadmium in QDs, with particular attention being paid to transition-element-doped noncadmium QDs because of their broad Stokes shift and tunable energy band structure. For example, Mn-doped ZnS nanocrystals exhibit higher luminescent efficiency than the undoped nanocrystals owing to emission from the dopant centers. The doping ratio of impurity atoms in a semiconductor nanocrystal under conditions of thermal equilibrium can be increased to much higher levels (50% or more) than in the bulk material, but this has often been difficult in practice. However, theoretical models have aided the development of new synthetic methods, and it has been confirmed that manganese can be doped into CdSe nanocrystals with the zincblende structure, although this may be impossible for nanocrystals with the wurtzite structure.
been found that a variety of nanocrystals can be doped via a decoupling technique, and it is interesting that studies revealed that some properties of these nanocrystals vary depending on the doping site.\textsuperscript{25} Researchers suggested that the formation process of doping QDs can be divided into two cases: nucleation and growth.\textsuperscript{26} The former is to mix the precursor of QD reactant and dopant together at the nucleation initial stage. The latter is that the dopant is added after nucleating and then reducing reaction temperature to inhibit crystal growth.

Up to now, studies on doped QDs have focused mainly on synthetic methods, morphology, and electrical and photoelectrical properties. However, little has been reported about microelectronic structure or the behavior of photogenerated free charge carriers (FCCs) in doped QDs. In previous work, we have confirmed that ZnSe QDs prepared by aqueous synthesis are self-assembled core–shell systems, with a graded bandgap.\textsuperscript{26,27} We have also previously obtained Cu-doped ZnSe QDs capped by L-cysteine (L-Cys) using a modified aqueous synthesis method.\textsuperscript{26}

In the present paper, Cu-doped ZnSe/ZnS/L-Cys self-assembled core–shell QDs are prepared by the modified aqueous synthesis method under different experimental conditions, with the aim of studying the effect of various reaction mechanisms and doping sites on the transport of photogenerated FCCs in Cu-doped QDs. The photovoltaic characteristics of Cu-doped QDs are probed via a combination of surface photovoltaic (SPV), photoacoustic (PA), and electric-field-induced surface photovoltage (EFISPV) techniques, supplemented by ultraviolet-visible (UV–VIS) absorption spectrum and laser Raman spectrum.

II. EXPERIMENTAL

The modified aqueous synthesis of Cu-doped ZnSe QDs capped by L-Cys involves a reaction among selenium-, zinc-, and copper-based precursors. First, the selenium-based precursor is prepared by mixing selenium powder with a NaBH\(_4\) solution with stirring until reaction is complete. The selenium precursor thus obtained, with a concentration of sodium hydrogen selenide (NaHSe) equal to 4 M, is dissolved in de-ionized water with stirring for 18 min, after which L-Cys is added. The pH value of the mixed solution of ZnCl\(_2\) is dissolved in de-ionized water with stirring for 18 min, after which L-Cys is added. The pH value of the mixed solution of ZnCl\(_2\) and L-Cys is adjusted to 11 with a 2 mol/l NaOH solution. The as-prepared Se-precursor is added to this mixed solution, which is then heated to 90 °C under reflux for 3 h with vigorous stirring. The reflux temperature is reduced to 60 °C, and then, to produce different samples, the CuCl\(_2\) solution is added at different time points: immediately, at 20 min, at 40 min, at 80 min, and at 160 min after the temperature reduction. These samples are labeled a, b\(_1\), b\(_2\), b\(_3\), and b\(_4\), respectively. The system has a molar ratio of Zn, Se, Cu, and L-Cys equal to 19.5:0.017:1:5. The samples are precipitated by using acetone. The precipitates are washed several times with both water and alcohol. The precipitates are dried at 60 °C for 6 h and then ground to powder. Finally, Cu-doped ZnSe QDs are obtained. All the reactions are performed under a protective nitrogen atmosphere.

X-ray diffraction (XRD; Rigaku D/max-2500/PC, Japan) and high-resolution transmission electron microscopy (HRTEM; JEOL-2010, Tokyo, Japan) are used to study the crystal structure and the average particle size of the QDs, respectively. Raman scattering spectroscopy (Renishaw, UK) is carried out to investigate the vibrational modes of the QDs. UV–VIS optical absorption spectra (Lambda35, PerkinElmer, USA) of the samples are obtained at room temperature.

Details of the SPV measurements have been described elsewhere.\textsuperscript{29,30} The SPV setup includes a lock-in amplifier (SR830-DSP), a 500-W xenon lamp regulated to 23 Hz with a light chopper (SR540) as the light source, and a sample cell with a sandwichlike structure comprising an indium tin oxide (ITO) electrode/sample/ITO electrode. PA spectroscopy is normalized by the PA signals of carbon black powders. PA spectroscopic analysis is first conducted at different modulation frequencies, ranging from 20 to 80 Hz, to confirm that the detected PA signals are only from the surfaces and phase interfaces of the QDs. SPV and PA techniques can be used to obtain information on photoelectron behavior at phase interfaces, because these techniques are by no means sensitive only to surfaces, but also to the entire surface space charge regions (SCRs) by superband or sub-bandgap absorption, and even to buried interfaces located anywhere in the detected sample, as long as these can be reached by photons.\textsuperscript{31} Figures 1(a) and 1(b) illustrate the setups for the SPV and PA measurements, which are carried out at room temperature.

III. RESULTS AND DISCUSSION

A. XRD and HRTEM

Figure S1 (supplementary material) shows the XRD pattern of sample a and the undoped ZnSe QDs. The average grain sizes of the two samples, as estimated from the well-known Scherrer formula,
are approximately 2.16 nm. This result indicates that the effect of the Cu dopant on the average grain size of the QDs is probably negligible. The diffraction peaks (220) and (311) of the two QDs are located between those of a bulk ZnSe crystal and those of a bulk ZnS crystal. Most likely, the shifts of the two peaks of the QDs are caused by the sulfhydryl group in L-Cys. Sulfur atoms in the L-Cys ligand can partially replace selenium atoms located at the surfaces and boundary of a ZnSe QD and react with Zn atoms to form a ZnS shell layer located between the ZnSe nanoparticle and the ligand. The ZnS shell layer grows preferentially on the (220) and (311) faces. The (111) peak of sample a is wider and splits into two diffraction patterns, as shown in Fig. S1. This may be related to the formation of a small quantity of CuS (klockmannite, KM) in the Cu-doped QDs. The doped copper element is probably in the form of Cu$_2^+$ ions located at Zn atom vacancies on the (111) face of the Cu-doped QDs.

Figure S2 (supplementary material) shows HRTEM of sample a. Three diffraction rings are obvious in the selected area electron dispersion pattern in Fig. S2(b), which is consistent with the XRD result in Fig. S1. The elemental content of sample a, as extracted from the energy dispersion spectrum in Fig. S3 (supplementary material), is listed in Table S1 (supplementary material). The result confirms the presence of Zn, Se, S, Cu, and O elements in the Cu-doped QDs, where the S and O may originate from the capped ligand coordinated with the Cu-doped QDs. In particular, the atomic percentages of Cu and Se in the Cu-doped QDs are 0.69% and 22.0%, respectively. This result is consistent with the initial molar ratio of copper to selenium, which is 1:30 in the mixed solution. The presence of Zn, Se, S, Cu, and O elements in the Cu-doped QDs is closely related to the multiphonon vibrational mode xLO of the ZnS ligand. The present results confirm that these samples have a ZnSe/ZnS/L-Cys core–shell structure, which is in good agreement with Refs. 26 and 27. It is interesting that the weak TO vibrational mode of ZnSe, which results in peak 2 located at 194 cm$^{-1}$, appears only in the Raman spectrum of sample a. More importantly, peak 3 has a tendency to become wider and to shift to a larger wavenumber with increasing doping time. This implies that the doping time influences to a certain extent the molecular structure of the Cu-doped ZnSe/ZnS/L-Cys core–shell QDs.

### C. Room-temperature UV–VIS absorption spectrum

Figure 3 shows the UV–VIS absorption spectra of the Cu-doped ZnSe/ZnS/L-Cys core–shell QDs prepared at different doping times. Exciton peaks of samples a, b, b$_2$, b$_3$, and b$_4$ appear at 372 nm, 355 nm, 376 nm, 358 nm, and 353 nm, respectively. The optical bandgap of nanocrystals can be calculated using the Tauc relation: 

$$E_{\text{g}} = A(h\nu - E_g)^n,$$

where $h\nu$ is the photon energy, $A$ is the absorption coefficient, $E_g$ is the optical bandgap, $A$ is a constant, and $n$ is equal to 1/2 for ZnSe nanocrystals with a direct bandgap. The optical bandgaps $E_{\text{g,UV-VIS}}$ of the samples, estimated using the Tauc relation, are shown in the insets of Fig. 3 and listed in Table I. According to Ref. 38, the bandgaps of as-prepared Cu-doped ZnSe QDs should be larger than that of bulk ZnSe (2.69 eV) because the average grain sizes of the QDs are much smaller than the exciton Bohr radius of ZnSe (4.50 nm). However, the bandgaps of samples a, b$_1$, and b$_2$ (but not those of samples b$_3$ and b$_4$) are smaller than that of the bulk material, and the bandgap value depends on Cu-doping time, as can be seen in Table I.

### D. SPV and PA spectroscopy

Figure 4 shows the SPV and the PA spectroscopy of as-prepared Cu-doped ZnSe QDs for different doping times. The first thing to note is that three SPV response peaks (knee1, knee2, and knee3) appear in the SPV spectroscopy of all five samples. According to our earlier research, knee1, knee2, and knee3 are closely related to band-to-band transitions of photogenerated FCCs in the core ZnSe, the shell ZnS, and the outer-layer ligand, respectively. The two valleys labeled QW1 and QW2 are identified as multiple-quantum-well structures in the ZnSe/ZnS/L-Cys core–shell QD system. Second, the magnitude of the PA signal depends mainly on the degradation of optical energy into thermal phonons through single- and multiphonon relaxation; i.e., this signal results from a nonradiative de-excitation process. Therefore, energy complementarity should exist between the PA and the SPV effects. The photoelectric thresholds, $E_{\text{g,SPV}}$, of the core ZnSe (knee1), the shell ZnS (knee2), and the outer-layer L-Cys (knee3) of the five samples are all the same, as seen in Fig. 4. The photoelectric threshold is evaluated as the abscessa of the largest external tangent of the band. The above result implies that the effect of doping time on the
photoelectric threshold of Cu-doped QDs is negligible. All five samples exhibit two strong PA signals at 316 nm and 798 nm, while any PA signals that appear between these two wavelengths are too weak to be seen. The energy complementarity between the PA and the SPV effects is obvious from a comparison of the PA and the SPV spectra. More importantly, the effects of doping time on the SPV and the PA characteristics of the Cu-doped ZnSe QDs are as follows: (1) the intensity of the SPV response at 600 nm of sample a is dramatically higher than those of samples b₁–b₄. (2) The ratio of the strongest SPV response and the strongest PA signal of sample

TABLE I. Relevant parameters extracted from the UV–VIS absorption spectra and the SPV and the PA spectroscopy of Cu-doped ZnSe/ZnS/L-Cys core–shell QDs prepared with different doping times.

| Parameter | a | b₁ | b₂ | b₃ | b₄ |
|-----------|---|----|----|----|----|
| \(E_{\text{g,UV-VIS}}\) (eV) | 2.53 | 2.55 | 2.47 | 2.69 | 2.78 |
| knee₁ | 3.04 | 3.04 | 3.04 | 3.04 | 3.04 |
| knee₂ | 2.76 | 2.76 | 2.76 | 2.76 | 2.76 |
| knee₃ | 2.07 | 2.07 | 2.07 | 2.07 | 2.07 |
| knee₄ | 1.98 | 1.98 | 1.98 | 1.98 | 1.98 |
| knee₅ | 1.89 | 1.89 | 1.89 | 1.89 | 1.89 |
| knee₆ | 1.69 | . . . | . . . | 1.80 | 1.80 |
| \(I_{\text{max,SPV}}\) (µV) | 44.68 | 19.10 | 21.68 | 20.56 | 20.30 |
| \(I_{\text{max,PA}}\) (µV) | 11.51 | 7.55 | 7.42 | 7.50 | 12.58 |
| \(I_{\text{max,SPV}}/I_{\text{max,PA}}\) | 3.88 | 2.53 | 2.92 | 2.74 | 1.61 |

*\(E_{\text{g,UV-VIS}}\) is the optical bandgap obtained from the UV–VIS absorption spectra; \(E_{\text{g,SPV}}\) is the photoelectric threshold of the SPV spectra at a specific wavelength, namely, the wavelength of one of the shoulder peaks (knees 1–6); \(I_{\text{max,SPV}}\) is the maximum SPV response intensity at 600 nm; \(I_{\text{max,PA}}\) is the maximum PA signal intensity at 315 nm.

*Samples a and b₁–b₄ were prepared at different doping times, as detailed in Sec. II.
a is more than twice the ratio of the other four samples, especially sample b4. (3) The five samples differ clearly from each other in the intensity and position of those small shoulder peaks, especially knee6 in the long-wavelength region. These small shoulder peaks may be closely related to some sub-bandgap charge transfer transitions between the impurity level and some energy band because their photoelectric thresholds are smaller than the bandgap of the QDs. These results reveal that choosing a suitable Cu-doping time may greatly improve the photovoltaic (PV) characteristic of the QDs. According to Refs. 21 and 42, the doping mechanisms for II/VI semiconductor nanocrystals can be divided into two types: nucleation doping and growth doping. Sample a is prepared by nucleation doping, whereas samples b1–b4 are prepared by growth doping, according to their respective Cu-doping times. Specifically, the diffusion length and the concentration of photogenerated FCCs in sample a are much greater than those in the other four samples so that the intensity of the SPV response of sample a is much stronger than those of the other samples in Fig. 4. In addition, the greater number of photogenerated FCCs in sample a leads to the generation of an SPV effect rather than a PA effect, in contrast to samples b1–b4. This should be responsible for the relatively high values of the strongest SPV response and the strongest PA signal of sample a in Fig. 4. The specific value of samples b1 to b4 decreases with increasing doping time, as seen in Fig. 4, even though they are all prepared by growth doping. This descending order is consistent with the trend of broadening of peak 3 of these samples in the Raman spectra in Fig. 2. This phenomenon implies that the doping time not only influences the molecular structure of the Cu-doped QDs but also changes the transport channel of photogenerated FCCs. In other words, some of the photogenerated carriers in the QDs prepared by growth doping release their energy by some form of phonon relaxation (i.e., some nonradiative de-excitation process such as the 1LO vibrational mode of ZnSe and ZnS in Fig. 2), rather than by generating a PV effect. Therefore, a long doping time may be disadvantageous with respect to an increase in the PV efficiency of Cu-doped QDs. For clarity, some relevant parameters extracted from the SPV and PA spectra of these samples are listed in Table 1.

E. EFISPV spectroscopy

Figure 5 shows the EFISPV spectroscopy of the Cu-doped ZnSe/ZnS/L-Cys core–shell QDs prepared at different doping times. According to the principle of the field effect transistor, in an external electric field, a hole in the SCR drifts along the direction of the field, while an electron moves in the opposite direction. Both the diffusion distance and the direction of carrier motion are directly affected by the external field. This will result in a redistribution of charges in the SCR and in a change in SPV response. Taking sample b3 as an example, the intensity of the SPV response of the sample increases with increasing external positive electric field from 0.0 V to 1.0 V; conversely, the intensity of the SPV response of the sample decreases with increasing absolute value of the external negative field from 0.0 V to −1.0 V, as can be seen at λ = 600 nm in Fig. 5 and more clearly in Fig. 6. This means that sample b3 has an n-type SPV
FIG. 6. Changes in SPV response intensity of samples a and b_1–b_4 at a wavelength of 600 nm in the presence of external positive and negative electric fields.

characteristic. Specifically, this sample with n-type SPV characteristic has a surface band bending upward and a donor feature. Sample a also has an n-type SPV characteristic, but the intensity of the SPV response of it in the absence of an external electric field is much higher than that of sample b_3 in Fig. 6 because, as already mentioned, both the diffusion length and the concentration of photogenerated FCCs in sample a are much greater than those in sample b_3. That is, the donor feature of sample a is more prominent than that of sample b_3. In fact, the donor feature of the samples gradually decreases from sample a to sample b_4, on the basis of their EFISPV characteristics. In addition, the intensity of the SPV response of sample b_4 increases with increasing absolute value of external negative electric field from 0.0 V to −1.0 V at λ = 600 nm in Fig. 6. This may be related to a transformation of the depletion layer to an accumulation layer structure in the SCR of the n-type material. It is notable that the small shoulder peak in the long-wavelength region of sample a is at 730 nm, while that of sample b_4 is at 700 nm. The corresponding peak positions of the other three samples are also blue-shifted, to different extents, compared with sample a, as shown in Fig. 5. This result confirms that the impurity level in the respective bandgap, which results from the dopant Cu^{2+} ion, rises gradually from sample a to b_4. Therefore, we assume that in the sample prepared by nucleation doping (sample a), the energy level of the dopant Cu^{2+} ion is located near and below the conduction band of the core ZnSe, as shown diagrammatically in Fig. 7(b). In contrast, the samples prepared by growth doping (samples b_1–b_4) show an increasing trend for the energy level of the dopant Cu^{2+} ion to be located near and below the conduction band of the shell ZnS, as illustrated in Fig. 7(c). The difference in the energy band structure of the Cu-doped ZnSe/ZnS/L-Cys QDs should be responsible for their respective SPV and PA characteristics.

IV. CONCLUSIONS

In summary, Cu-doped ZnSe/ZnS/L-Cys self-assembled core–shell QDs have been prepared via both nucleation doping and growth doping in an aqueous synthesis method. SPV, EFISPV, and PA technologies have been used to probe the transport of photogenerated FCCs in the Cu-doped QDs, supplemented by the UV–VIS absorption spectrum and laser Raman spectrum. The results confirm that the impurity levels of the Cu-doped QDs prepared by nucleation doping are preferentially located at shallow levels in the bandgap of the core ZnSe. In contrast, with increasing doping time, there is a trend for the impurity levels of Cu-doped QDs prepared via growth doping to be located at shallow levels in the bandgap of the shell ZnS. The research reveals the difference of the graded energy bands of the Cu: ZnSe QDs prepared by the two mechanisms above. The difference may be the main reason that leads to a significant distinction of the PV characteristics between the two Cu-doped QDs. These microelectronic structural features are assumed to be responsible for a series of changes in the PV characteristics of the Cu-doped QDs. Compared with the QDs produced by growth doping, the SPV characteristics of the QDs prepared by nucleation doping have the following advantageous characteristics: (1) They display a quite high SPV response intensity at 600 nm because of a high concentration of photogenerated FCCs. (2) The ratio of the strongest SPV response and strongest PA signal of the QDs prepared by nucleation doping is about 1.53–2.41 times greater than those of the QDs prepared by growth doping. This indicates that because of those photogenerated FCCs produced by nucleation doping, the PV effect is enhanced, rather than the PA effect that is an adverse factor to the increase in photoelectric transfer efficiency. This also reveals the energy complementary between the SPV and the PA effects of the samples.
(3) The shoulder peak position of the SPV response at a long wavelength of the QDs prepared by nucleation doping is significantly red-shifted compared with those prepared by growth doping, resulting directly in a broader SPV response range in the visible region.

(4) The QDs prepared by nucleation doping have a more obvious donor feature of an n-type PV material.

SUPPLEMENTARY MATERIAL

See the supplementary material for the research results on the crystal structure of as-prepared Cu-doped ZnSe/ZnS/L-Cys core-shell QDs. The result indicates that the effect of the Cu dopant on the average grain size of the QDs may be negligible. The research confirms that the doped copper element is probably in the form of Cu$^{2+}$ ions located at Zn atom vacancies on the (111) face of the Cu-doped QDs and that a ZnS shell layer has been formed between the Cu-doped core ZnSe nanoparticles and the outer-layer ligand.

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