Realization of $p$-type ZnAgO:N thin films on flexible polyimide substrates through co-sputtering for wearable thermoelectric applications

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Realization of p-type ZnAgO:N thin films on flexible polyimide substrates through co-sputtering for wearable thermoelectric applications

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ABSTRACT
In this study, we deposited a series of Ag and N co-doped ZnO thin films (ZnAgO:N) with different Ag atomic percentage (at. %) ratios on flexible polyimide (PI) substrates to realize p-type ZnO-based thin films for wearable thermoelectric applications by in situ co-sputtering of ZnO and Ag by RF and DC magnetron sputtering in a mixture of Ar and N\textsubscript{2}O gases. To evaluate the thermoelectric performance of these ZnAgO:N thin films, we measured the Seebeck coefficient \( S \) and electrical conductivity \( \sigma \) of the thin films with various Ag at. % ratios and calculated the power factor \( S^2\sigma \). These measurements confirmed that the co-doping of Ag and N into ZnO is an effective method for fabricating p-type ZnAgO:N thin films with \( \sigma \) in the range of about \( 10^5 - 3.3 \times 10^6 \) S/m, as a function of Ag at. % ratio on the flexible PI substrates. In addition, the presence of an optimal Ag at. % ratio that leads to a maximum \( S^2\sigma \) for the p-type ZnAgO:N thin films was observed. Raman spectroscopy and x-ray photoelectron spectroscopy revealed that the p-type conductivity in ZnAgO:N thin films originates from the acceptors Ag\textsubscript{Zn} and N\textsubscript{O} formed by the co-dopants Ag and N. As a result, the \( S \) and \( \sigma \) of the p-type ZnAgO:N thin films were investigated to be affected significantly by the acceptors and defects formed by the Ag and N co-dopants in the thin films. The influence of Ag\textsubscript{Zn} and N\textsubscript{O} on the appearance of the p-type conductivity in ZnAgO:N thin films and \( S \) and \( \sigma \) of the thin films was intensively studied.

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Zinc oxide (ZnO) is frequently used to fabricate thin-film-based devices \(^1 \) because of its attractive properties such as superior biocompatibility, a direct band gap of \( \sim 3.4 \) eV, and appropriate electrical conductivity.\(^2,3 \) However, fabricating ZnO thin-film-based devices remains challenging because intrinsic donors, such as zinc interstitials (Zn\textsubscript{i}) and oxygen vacancies (V\textsubscript{O}), make it difficult to deposit p-type ZnO thin films.\(^4,5 \) Therefore, major effort has been expended to find a way to deposit p-type ZnO thin films. To resolve this issue, diverse techniques have been used to deposit p-type ZnO thin films using dopants.

Given its atomic size and electrical role in ZnO, N has been considered as a substituting element for O.\(^6-9 \) Nevertheless, p-type conductivity in N-doped ZnO remains uncertain because N-related acceptors are easily compensated by intrinsic donors in ZnO.\(^10,11 \) The group IB elements, such as Cu and Ag, have also been considered as dopants because of their low self-compensation,\(^12,13 \) but p-type ZnO thin films are difficult to be fabricated because Cu tends to form deep acceptors\(^14 \) and Ag is an amphoteric dopant in ZnO.\(^12,14 \) Meanwhile, after Yamamoto et al. suggested that co-doping of donor and acceptor dopants could make shallow acceptors in ZnO,\(^15-17 \) various p-type ZnO thin films have been deposited by co-doping of donor and acceptor dopants, such as Ga–N and Al–N.\(^18,19 \)

In this study, the p-type Ag and N co-doped ZnO thin film ZnAgO:N were deposited on flexible polyimide (PI) substrates for thermoelectric applications by in situ co-sputtering of ZnO and Ag in a mixture of Ar and N\textsubscript{2}O gases for N doping into thin films.
The Seebeck coefficient $S$ and electrical conductivity $\sigma$ for these films with varying Ag atomic percentage (at. %) ratio were measured. The results indicate that the co-doping of Ag and N into ZnO is effective for depositing $p$-type ZnAgO:N thin films with various electrical characteristics depending on the Ag at.% ratios. These thin films were analyzed by using various analytic techniques to investigate their electrical characteristics.

As detailed in Table I, ZnAgO:N thin films of varying Ag at.% ratios were deposited by co-sputtering of ZnO and Ag targets by RF and DC magnetron sputtering in a mixture of Ar and N$_2$O gases. The DC sputtering power for Ag was adjusted to control the Ag at.% ratio. The RF sputtering power for ZnO was held constant, as was the Ar/N$_2$O ratio. The deposition temperature ranged from 300 K to 310 K. After deposition, the thin films were post-annealed for 4 h in N$_2$ at 1 atm, 573 K. Ag thin-films were formed on these films as electrodes by electron-beam evaporation. Table I lists the Ag and N at.% ratios of ZnAgO:N thin films along with the ratio of DC sputtering power for Ag to RF sputtering power for ZnO (power ratio). Although the Ag at.% ratio is proportional to the power ratio, the N at.% ratio varies irregularly regardless of the power ratio.

Figures 1(a)–1(c) plot $S$, $\sigma$, and power factor $S^2\sigma$ for ZnAgO:N thin films as functions of the Ag at.% ratio at the hot-side temperature of these films. The $S$ of the ZnAgO:N thin films was measured under a lateral temperature gradient (Fig. S1, supplementary material) and the corresponding $\sigma$ was measured by using the van der Pauw method. As shown in Fig. 1(a), $S$ was measured only for thin films D3-2–D7-2 because the other thin films were too resistive to measure $S$. Likewise, $\sigma$ for the ZnAgO:N thin films was also measured only for D3-2–D7-2, as shown in Fig. 1(b). Although the positive $S$ for D3-2–D7-2 confirms $p$-type semiconductivity (Fig. S2, supplementary material), $S$ decreases as the Ag at.% ratio increases. Meanwhile, $\sigma$ increases until the Ag at.% ratio reaches that of D5-2. Thus, the maximum $\sigma$ is $\sim 3.0 \times 10^6$ S/m obtained for D5-2. The opposite slope of $S$ and $\sigma$ as a function of Ag at.% ratio in the range of 0.132–0.307 reflects the fact that they are inversely proportional. In this range of Ag at.% ratio, $S^2\sigma$ increases, as shown in Fig. 1(c). However, when the Ag at.% ratio exceeds 0.307, $S^2\sigma$ decreases because $S$ and $\sigma$ decrease in that range of Ag at.% ratio.

| Thin films | Power ratio | Ag at. % ratio | N at. % ratio | Post-annealing |
|------------|-------------|----------------|----------------|----------------|
| D1-1       | 0           | 0              | 0.034          | ×              |
| D1-2       |              |                |                | ○              |
| D2-1       | 0.031       | 0.101          | 0.072          | ×              |
| D2-2       |              |                |                | ○              |
| D3-1       | 0.056       | 0.132          | 0.033          | ×              |
| D3-2       |              |                |                | ○              |
| D4-1       | 0.075       | 0.221          | 0.044          | ×              |
| D4-2       |              |                |                | ○              |
| D5-1       | 0.094       | 0.307          | 0.064          | ×              |
| D5-2       |              |                |                | ○              |
| D6-1       | 0.113       | 0.404          | 0.064          | ×              |
| D6-2       |              |                |                | ○              |
| D7-1       | 0.131       | 0.489          | 0.057          | ×              |
| D7-2       |              |                |                | ○              |

FIG. 1. (a) $S$, (b) $\sigma$, and (c) $S^2\sigma$ for D3-2–D7-2. The legend indicates the hot-side temperature of the thin films. The cold-side temperature of these films is $\sim 293$ K.
The transition to \(p\)-type ZnAgO:N thin films infers that the Ag in the annealed films plays a key role in the formation of acceptors, such as \(\text{Ag}^{+}\) substitution for Zn\(^{2+}\) (\(\text{Ag}_{\text{Zn}}\)) and N-ion substitution for O\(^{2-}\) (\(\text{N}_{\text{O}}\)).

The x-ray diffraction (XRD) spectra of the \(p\)-type thin films contain the peaks related to ZnO and Ag (Fig. S3, supplementary material).

The appearance of the \(p\)-type conductivity in annealed ZnAgO:N thin films confirms the feasibility of fabricating \(p\)-type ZnAgO:N thin films by co-doping with Ag and N. However, the cause of \(p\)-type conductivity in the thin films remains uncertain. Thus, ZnAgO:N thin films were analyzed by Raman spectroscopy to investigate the types of defects in the ZnAgO:N thin films.

Figure 2 shows Raman spectra of ZnAgO:N thin films deposited on SiO\(_2\)-coated Si substrates instead of PI substrates because PI has more Raman peaks. The Raman spectra of non-annealed ZnAgO:N thin films reveal only a single peak near 520 cm\(^{-1}\) caused by the substrates,\(^{27}\) whereas the Raman spectra of annealed ZnAgO:N thin films reveal peaks near 507 cm\(^{-1}\) and 570 cm\(^{-1}\), related to \(\text{N}_{\text{O}}\),\(^{28,29}\) and a peak near 395 cm\(^{-1}\), related to \(\text{Ag}_{\text{Zn}}\)–O bonds.\(^{30}\) Thus, the low intensity of the corresponding peaks indicates lower concentrations of \(\text{Ag}_{\text{Zn}}\) and \(\text{N}_{\text{O}}\) acceptors in D1-2 and D2-2, which seems to be the reason that \(S\) and \(\sigma\) of D1-2 and D2-2 could not be measured.

The Raman peaks from \(\text{N}_{\text{O}}\) in D3-2 are similar to those for D2-2, but the peak near 395 cm\(^{-1}\) is slightly more evident in D3-2. Thus, the \(p\)-type conductivity in D3-2 seems to be caused mainly by the enhanced concentration of \(\text{Ag}_{\text{Zn}}\).

Meanwhile, a peak near 365 cm\(^{-1}\) appears in the Raman spectra of D4-2–D7-2 and is speculated to originate from the red shift of the peak near 395 cm\(^{-1}\) because of the tensile stress induced by Ag, which has a larger atomic radius than Zn.\(^{23,24}\) The Raman peaks near 365 cm\(^{-1}\), 507 cm\(^{-1}\), and 570 cm\(^{-1}\) become more prominent in the Raman spectra of D4-2 and D5-2. Thus, the increase in \(\sigma\) of D4-2 and D5-2 could be attributed to both the enhancement in the concentration of \(\text{Ag}_{\text{Zn}}\) and the presence of \(\text{N}_{\text{O}}\).

However, the Raman peak near 365 cm\(^{-1}\) decreases with an increase in the Ag at.% ratio. In other words, the \(\text{Ag}_{\text{Zn}}\) concentration in D6-2 and D7-2 is less than that in D5-2, which indicates that the decrease in \(\sigma\) of D6-2 and D7-2 is caused by a decrease in the concentration of \(\text{Ag}_{\text{Zn}}\). Furthermore, the noticeable decrease in the Raman peak near 507 cm\(^{-1}\) of D7-2 indicates that the decrease in \(\sigma\) is more significant in D7-2 than in D6-2.

Unlike \(\sigma\), \(S\) as a function of Ag at.% ratio decreases in \(p\)-type ZnAgO:N thin films. The decrease in \(S\) for D3-2–D5-2 is due to the inverse proportionality between \(S\) and \(\sigma\),\(^{25}\) but the further decrease in \(S\) for D6-2 and D7-2 seems to be related to defect-induced distortions. The Raman peak near 570 cm\(^{-1}\) indicates the presence of defects such as \(\text{Zn}_{\text{Zn}}\) \(\text{V}_{\text{O}}\), and \(\text{N}_{\text{O}}\) in ZnO.\(^{27,34,35}\) The concentration of such defects would increase with increasing dopant concentration in these thin films, which means that distortions degrading the electrical characteristics of thin films could be formed by the defects.\(^{36}\) Moreover, the metallic Ag formed by the over-doping of Ag could degrade the doping efficiency of acceptors, thereby lowering \(S\) and \(\sigma\) further.\(^{25,37,38}\) Consequently, the decrease in \(S\) for D6-2 and D7-2 is attributed to defects stemming from the over-doping of Ag.

By x-ray photoelectron spectroscopy (XPS), we investigated in detail how the acceptors affect electrical characteristics of ZnAgO:N thin films. Figures 3(a)–3(c) show the XPS spectra of Ag 3d, O 1s, and N 1s, respectively. The deconvolution of the O 1s spectrum of D1-2 into three spectra reveals three types of O\(^{2-}\) ions. The O\(^{2-}\) ions surrounded by Zn\(^{2+}\) correspond to the O 1s spectrum with a binding energy (BE) of \(\sim 529.6\) eV. The O\(^{2-}\) ions in O-deficient regions (\(\text{V}_{\text{O}}\)) or bonded on the surface correspond to the O 1s spectra with a BE of \(\sim 531.4\) eV or 532.2 eV, respectively.\(^{39,40}\) The N 1s spectrum of D1-2 looks like noise, which means that \(\text{N}_{\text{O}}\) is not present in this film. Therefore, D1-2 is not the \(p\)-type.

The N 1s spectrum of D2-2 is similar to that of D1-2. Although the O 1s spectrum with a BE of \(\sim 528.8\) eV and the Ag 3d\(_{3/2}\) spectra with BEs of \(\sim 367.2\) eV and 367.4 eV indicate the presence of Ag–O bonds in D2-2, this film is not likely to be \(p\)-type because most Ag ions in this film are in the 3+ or 2+ oxidation state.\(^{47,48}\)

The peak near 399.3 eV in the N 1s spectrum of D3-2 implies the absence of \(\text{N}_{\text{O}}\) acceptors.\(^{49,50}\) However, the Ag 3d\(_{3/2}\) spectrum deconvoluted into two spectra with BEs of \(\sim 367.8\) eV and 368.5 eV indicates the presence of \(\text{Ag}_{\text{Zn}}\) and metallic Ag, respectively.\(^{50}\) The presence of \(\text{Ag}_{\text{Zn}}\) acceptors is also confirmed by the O 1s spectra with BEs of \(\sim 530.1\) eV and 531.4 eV.\(^{47,48}\) Thus,

\[\text{\text{FIG. 2. Raman spectra of ZnAgO:N thin films. Various peaks derived from Ag}_{\text{Zn}}, \text{N}_{\text{O}}, \text{etc., are marked by the Raman shifts.}}\]
the appearance of p-type conductivity in D3-2 is caused by Ag2O acceptors.

D4-2 has Ag 3d5/2 spectra with BEs of ~367.6 eV and 368.6 eV derived from Ag2O and Ag, respectively, indicating the greater concentration of Ag2O in D4-2 because the Ag 3d5/2 spectrum derived from Ag2O is farther from that derived from Ag in D4-2 than in D3-2. The O 1s spectra with BEs of ~530.1 eV and 531.4 eV in D4-2 support the presence of Ag2O, as does in D3-2. In addition, the peak derived from N 1s in Ag2O–N0 bonds appears near 398.8 eV in the N 1s spectrum. The BE of N 1s electrons in Ag2O–N0 bonds is greater than that in Zn–N0 bonds because of the greater electronegativity of Ag than of Zn.25 However, the concentration of N0 seems low because no Ag 3d spectrum derived from Ag2O–N0 is observed. Thus, the enhanced p-type conductivity in D4-2 is due to the enhanced Ag2O concentration and a small amount of N0 in Ag2O–N0 bonds.

Only the Ag 3d5/2 spectrum derived from Ag2O appears at ~367.5 eV,25,49-51 farther from the BE of Ag, which means that the concentration of Ag2O increases further in D5-2. Like in D4-2, the O 1s spectrum indicates the presence of Ag2O in D5-2. The peak with low intensity near 398.2 eV in the N 1s spectrum of D5-2 indicates N0 acceptors in Zn–N0 bonds.49 Therefore, the largest σ measured in D5-2 is attributed to the further enhanced Ag2O concentration and N0 in Zn–N0 bonds.

However, the Ag2O concentration in D6-2 and D7-2 is lower than that in D5-2 because the Ag 3d5/2 spectrum derived from Ag2O shifts to slightly higher BE, ~367.6 eV. In addition, the Ag 3d5/2 spectrum with low intensity derived from Ag appears near 368.4 eV in both thin films. Thus, the doping efficiency of Ag2O would be degraded by Ag.49 Moreover, the peak derived from N0 in Zn–N0 bonds is significantly blurred in D6-2 and D7-2, therefore, the concentration of N0 in these films is lower than in D5-2.

Accordingly, the decrease in σ for D6-2 and D7-2 is attributed to the significantly decreased concentrations of Ag2O and N0.

In summary, the p-type ZnAgO:N with different Ag at. % ratios were deposited on flexible PI substrates by co-sputtering of ZnO and Ag in a mixture of Ar and N2O gases. To evaluate their thermoelectric performance, the Seebeck coefficient and electrical conductivity of the p-type ZnAgO:N thin films were measured and their power factors were calculated. The p-type thin film D5-2 has the best thermoelectric performance in terms of S2σ.

The p-type ZnAgO:N thin films were analyzed by Raman spectroscopy and XPS to investigate their p-type characteristics. The Raman spectroscopy and XPS spectra reveal that the p-type conductivity in ZnAgO:N thin films originates from the Ag2O and N0 formed by the co-dopants Ag and N, and S and σ for these films are affected strongly by the Ag2O and N0 concentrations. However, over-doping of Ag may induce distortions and formation of metallic Ag, which degrade the electrical properties of these films.

Finally, the p-type ZnAgO:N thin films having moderate σ on flexible PI substrates are expected to meet the demand for p-type thin films required to fabricate flexible or wearable thermoelectric thin-film devices.

The supplementary material includes a schematic diagram of the apparatus used to measure the Seebeck coefficient S, the graphs of thermoelectric voltages generated by the Al-doped ZnO thin film (n-type thin film) and p-type ZnAgO:N thin film D3-2, the XRD spectra of ZnAgO:N thin films, the XPS spectra of Ag 3d for annealed ZnAgO:N thin films edited to compare the intensities of Ag 3d5/2 spectra derived from Ag2O acceptors, the three-dimensional graphs of S, σ, and S2σ for ZnAgO:N thin films plotted as a function of Ag at. % ratio for the hot-side temperature of the thin films, and...
a table for the Hall effect measurement results for $\sigma$ of the $p$-type ZnAgON thin films.

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DATA AVAILABILITY

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

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