Flexible transparent heteroepitaxial conducting oxide with mobility exceeding 100 cm$^2$ V$^{-1}$ s$^{-1}$ at room temperature

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Abstract
Flexible and transparent applications have become an emerging technology and have shifted to the forefront of materials science research in recent years. Transparent conductive oxide films have been applied for flat panel displays, solar cells, and transparent glass coatings. However, none of them can fulfill the requirements for advanced transparent flexible devices, such as high-frequency applications. Here, we present a promising technique for transparent flexible conducting oxide heteroepitaxial films: the direct fabrication of epitaxial molybdenum-doped indium oxide (IMO) thin films on a transparent flexible muscovite substrate. An $n$-type epitaxial IMO film is demonstrated with a mobility of 109 cm$^2$ V$^{-1}$ s$^{-1}$, a figure of merit of 0.0976 Ω$^{-1}$, a resistivity of 4.5 × 10$^{-5}$ Ω cm and an average optical transmittance of 81.8% in the visible regime. This heteroepitaxial system not only exhibits excellent electrical and optical performance but also shows excellent mechanical durability. Our results illustrate that this is an outstanding way to fabricate transparent and flexible conducting elements for the evolution and expansion of next-generation smart devices.

Introduction
Transparent conductive oxides (TCOs) exhibit impressive properties, including excellent electrical conductivity and high optical transmittance in the visible light range$^{1,2}$. They have attracted great interest due to their potentially disruptive application in optoelectronics, including flat panel displays, light-emitting diodes, thin-film transistors, solar cells and a variety of other applications that use both their electronic and transmittance features$^{3–6}$. With their dramatically advanced properties for the integration of additional functionalities, flexible electronics- and numerous-related applications have become important research directions for soft technologies and wearable electronics$^{7,8}$. In this research field, transparent flexible conducting components with excellent performance play a key role in almost all optoelectronics. Thus, the development of high-quality transparent flexible conducting elements has attracted a substantial amount of attention and become a promising research direction for next-generation consumer electronics.

Among the well-known TCOs, indium tin oxide (ITO) is the most extensively studied and widely used in light-emitting diodes, liquid crystal displays, and photovoltaic applications, for example$^9,10$. ITO exhibits the desirable combination of a high electrical conductivity and high optical transparency. High-quality epitaxial ITO thin films possess a low resistivity of 2.3 × 10$^{-4}$ Ω cm with a carrier concentration of 4.8 × 10$^{20}$ cm$^{-3}$ and mobility of 56 cm$^2$ V$^{-1}$ s$^{-1}$$^{11}$. However, the characteristics of ITO films are still not acceptable enough to fulfill the requirements for advanced optoelectronics, especially thin-film transistors, thin-film solar cells and high-frequency devices, due to their requirements of a super high charge mobility for improving their performance. For instance, in the study of...
optically transparent patch antennas, a large enhancement in the microstrip efficiency with resonant frequencies between 100 MHz and 10 GHz was obtained with an increase in the electron mobility from 50 cm² V⁻¹ s⁻¹ to 100 cm² V⁻¹ s⁻¹. Moreover, even with a high crystalline quality, the mobility of ITO is reduced for films fabricated on flexible substrates. Therefore, it is productive to search for new candidates that break this barrier.

The large-room-temperature mobility of molybdenum-doped indium oxide (IMO) films coupled with a low resistivity, high carrier concentration and optical transparency makes them a candidate for transparent flexible conducting elements to replace ITO. The IMO is 3, TCO can have a high carrier concentration, as shown by these defect chemical equations:

\[
\text{In}_n + \text{Mo}_3\text{O}_8 \rightarrow \text{Mo}_n\text{In}_3 + 1/2 \text{In}_2\text{O}_3 + 3/4 \text{O}_2 + 3e^- \\
\text{Mo}_n\text{In}_3\text{O}_{2-} \rightarrow \text{Mo}_n\text{In}_3 + 1/2 \text{O}_2 + 2e^- \\
\text{O}_2 \rightarrow 1/2 \text{O}_2 + V_0 + 2e^- 
\]

Both a high-mobility and high carrier concentration make IMO an extraordinary TCO with excellent performance. Therefore, it is worth considering the challenges and fabricating transparent flexible conducting IMO thin films for advanced transparent flexible applications.

To produce a high-mobility conducting element featuring optical transparency as well as mechanical flexibility, two necessary components must be combined. (1) There must be a high-quality epitaxial IMO thin film with low resistivity, high-mobility and high carrier concentration. (2) There must be an ideal substrate with a crystalline structure, high optical transparency and high carrier concentration. (2) There must be an ideal substrate with a crystalline structure, high optical transparency and high carrier concentration.

Growth of the IMO/AZO/mica Heterostructure

The epitaxial IMO/AZO/mica heterostructure was deposited via RF magnetron sputtering using commercial IMO (96% In₂O₃ and 4% MoO₃) and AZO (98% ZnO and 2% Al₂O₃) targets with a diameter of 3 inches. An exfoliated artificial muscovite mica (1 × 1 cm) without treatment was used in this study. The vacuum chamber was pumped to a base pressure of 10⁻⁶ Torr prior to deposition. During growth of the IMO and AZO, the chamber was maintained at 3 mTorr with an Ar flow of 20 sccm and an oxygen flow of 0.1 sccm. The deposition processes for both IMO and AZO were conducted while the substrate was heated to 550 °C, and the RF power was 100 W. After deposition, the heterostructures were annealed in situ at 750 °C for 1 h in the same atmosphere as during growth to improve the electrical conductivity.

Structural analysis

X-ray diffraction (XRD) θ–2θ scans along the normal direction and Φ scans were performed to obtain structural information in a Bruker D8 high-resolution X-ray diffractometer using a monochromatic Cu Kα1 radiation source. Cross-sectional TEM was used to study the microstructure of the heterostructure. The TEM specimens were prepared by a focused ion beam system along the normal direction. X-ray photoelectron spectroscopy spectra were obtained in a ULVAC-PHI PHI 5000 Versaprobe II.

Conductivity and transparency properties

The resistivity and Hall measurements were conducted in a physical property measurement system. The optical transmittance spectra were gathered in a PerkinElmer Lambda-900 spectrometer.

Bending tests

A homebuilt computer-controlled bending system was used to conduct all bending tests. While one end was fixed, the other end could be moved by a stepping motor. The heterostructure was bent by the bending system and observed by an additional microscope.

Results and discussion

In this study, epitaxial IMO thin films were grown on muscovite by RF sputtering. The crystal structure and phase identification of the heteroepitaxial films were then characterized by XRD. Moreover, as shown in Fig. 1a, an Al-doped ZnO (AZO) layer was inserted between the IMO and mica as a seed layer to enable the epitaxial growth of a high-quality IMO film. It is difficult to deposit
an epitaxial IMO thin film on bare muscovite without a seed layer since the surface energies of IMO are similar for different orientations. Figure 1b shows an out-of-plane $\theta$$-2\theta$ scan of the IMO/AZO/mica heteroepitaxial system. Only IMO (111), AZO (00l) and muscovite (00l) diffraction peaks can be observed, indicating epitaxy of the heterostructure without undesirable secondary phases. The out-of-plane d-spacing of the IMO (222) planes was calculated from the XRD peak position and was 2.912 Å, suggesting a slight compressive strain of 0.3%. This measured strain was caused by the defects created during the deposition process and not the interaction between the IMO and muscovite substrate attributed to the van der Waals epitaxy. Moreover, $\Phi$ scans of IMO, AZO and muscovite reflections were applied to identify the in-plane epitaxial relationships, as shown in Fig. 1c. A primary set and two secondary sets of muscovite peaks at 120° intervals with different intensities suggest that different stacking sequences existed in the muscovite. Cubic bixbyite ITO {400} and hexagonal AZO {101} exhibited six peaks at 60° intervals, which indicates the existence of multdomain IMO and single crystalline AZO films on the muscovite substrate. Based on the XRD results, the epitaxial relationship was determined to be (111)$_{\text{IMO}}$/[001]$_{\text{AZO}}$/[001]$_{\text{mica}}$ and (01−1)$_{\text{IMO}}$/[010]$_{\text{AZO}}$/[001]$_{\text{mica}}$ for the IMO/AZO/mica heterostructure. To gather critical information about the crystallinity, rocking curve measurements were carried out, and the full width at half maximum values of ~1.28° and ~0.85° were determined for the IMO (222) and AZO (002) peaks, respectively, as shown in Fig. 1d. Furthermore, to study the microstructure of the IMO/AZO/mica heterostructure as well as explore the heteroepitaxy, interfaces between the thin films and substrate were investigated by transmission electron microscopy (TEM). Figure 1e exhibits high-resolution cross-sectional TEM images captured along the [010]$_{\text{mica}}$ direction, showing clear and defect-free IMO/AZO/mica interfaces. The reciprocal lattices of the IMO, AZO and muscovite, obtained from fast Fourier transforms, were clearly indexed to distinguish the structural information regarding the IMO/AZO/mica heteroepitaxy. The consistency of the epitaxial relationships between XRD and TEM results was carefully confirmed. The clear interfaces without observable defects indicate the presence of good heteroepitaxy. From the XRD and TEM results, high-quality IMO/AZO/mica heteroepitaxy was fabricated, which is key to attaining excellent properties. To verify the valence state of the In and Mo ions in the heterostructure and confirm the chemical composition, X-ray photoelectron spectroscopy (XPS) measurements were carried out. As shown in Supplementary Fig. 1, a typical XPS spectrum was obtained from the surface of the IMO/AZO/mica heterostructure. The high-resolution spectra of the In 3d and Mo 3d core levels indicate that the valence state of In was +3 and that for Mo was +6 in the heterostructure. By analyzing the XPS spectrum, the ratio of Mo dopant can be confirmed as ~4 wt % in the IMO films. It has been reported that IMO thin films exhibit a relatively high electron mobility as well as low resistivity at this doping level$^{17,20-22}$. After the production of van der Waals epitaxy in the IMO/AZO/mica heterostructure, it is crucial to build a comprehensive and quantitative knowledge of the characteristics of this heteroepitaxy for advanced soft
Comparison was made between the IMO/AZO/mica heterostructure and showed increased transport behavior. A crystallinity was obtained, which reduced the scattering of the carrier and showed increased transport behavior. The IMO/AZO/mica heterostructure exhibited excellent conductivity ($\mu_109 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$) at room temperature. The resistivity decreased with temperature across the whole temperature range, exhibiting nearly metallic behavior. In addition, this behavior can be simply controlled by annealing the samples in different oxygen atmospheres. The IMO heteroepitaxial structure, annealed in a high oxygen pressure, exhibited insulating behavior and its resistivity increased as the temperature increased, as shown in Supplementary Fig. S2. The Hall measurement of the heterostructure confirmed the n-type conducting nature. The carrier concentration, $n$, was $1.27 \times 10^{21} \text{ cm}^{-3}$ and showed almost no temperature dependence. The Hall electron mobility reached a value of $109 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ at 300 K and $140 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ at 10 K. The thickness-dependent resistivity, mobility and carrier concentration of the IMO heteroepitaxial structure are shown in Fig. 2b. The conductivity of the IMO/AZO/mica heterostructure showed an average value of $81–83\%$ with a thickness from 100 to 400 nm. The observed oscillation in the spectra was caused by interference effects. The transmittance of the heteroepitaxial structure in the visible range slightly decreased with increasing thickness. A figure of merit ($\Phi_{TC}$), which is an important parameter, was used to identify and compare the performance of all transparent conducting elements. The value of $\Phi_{TC}$ was defined to be $T^{10}/R_s (\Omega^{-1})$, where $R_s$ is the sheet resistance and $T$ is the average optical transmittance. Typically, a higher $\Phi_{TC}$ indicates an increased performance for transparent conducting films. The $\Phi_{TC}$ for the IMO/AZO/mica structure with various thicknesses, IMO films, and numerous TCO films on flexible substrates are shown in Fig. 3b. The ultrahigh figure of merit values for the IMO/AZO/mica heteroepitaxial structure can be observed due to its excellent characteristics. A very high...
ΦTC value of 0.0976 Ω⁻¹ was achieved for the best IMO/AZO/mica heterostructure, which exhibited high mobility (109 cm² V⁻¹ s⁻¹), low sheet resistance (1.37 Ω °⁻¹) and high optical transmittance (81.8%). As a result, the transparent conducting IMO/AZO/mica heterostructure exhibited remarkable performance beyond that for all reported flexible TCO films.

Furthermore, to extend the practical application of this IMO/AZO/mica heterostructure with mechanical flexibility, a range of cycling tests were carried out. The operational stability and mechanical durability of the flexible IMO/AZO/mica heteroepitaxial structures were tested under both compressive (flex-in) and tensile (flex-out) bending states, as shown in the insets in Fig. 4a, b. Figure 4a, b shows that the resistance of the 300-nm thick heterostructure remained nearly constant with a bending radius down to 3.5 mm under both compressive and tensile deformations, respectively. Heterostructures with thicknesses of 100 and 200 nm also exhibited a steady conductivity with bending radii down to 5 mm. Thus, IMO heteroepitaxial structures were robust against mechanical constraints for flexible device applications. Due to only weak van der Waals interactions between the IMO/AZO and muscovite and small film-to-substrate

### Table 1  An overview of recent works on IMO thin films with their properties.

| Technique          | Tprocess (°C) | Φ (10⁻³ Ω⁻¹) | R, (Ω/□) | T (%) | ρ (Ω cm) | μ (cm² V⁻¹ s⁻¹) | n (cm⁻³) | Mo doping | Thickness (nm) | Substrate      |
|--------------------|---------------|--------------|----------|-------|----------|----------------|----------|-----------|----------------|----------------|
| RF sputteringa     | 550           | 97.6         | 1.37     | 81.8  | 4.5 x 10⁻⁵ | 109            | 1.3 x 10⁻²¹ | 4 wt%     | 300            | Mica           |
| PLD23              | 700           | 39.18        | 8.9      | 90    | 8.9 x 10⁻⁵ | 138            | 4.7 x 10⁻²⁰ | 2 at%     | 100            | Quartz         |
| AA-CVD15           | 450           | 31.29        | 1.8      | 75    | 1.2 x 10⁻⁴ | 119            | 4.4 x 10⁻²⁰ | –         | 650            | Glass          |
| RTE17              | 350           | 21.47        | 5        | 80.0  | 1.8 x 10⁻⁴ | 130            | 2.6 x 10⁻²⁰ | 4 wt%     | 370            | Glass          |
| ARE26              | 300           | 18           | 26       | 93    | 6.5 x 10⁻⁴ | 24             | 4.0 x 10⁻²⁰ | 3 at%     | 250            | Glass          |
| AACVD36            | 450           | 13.3         | 2.1      | 69.9  | 1.4 x 10⁻⁴ | 123            | 3.7 x 10⁻²⁰ | 3.1 at%   | 670            | Soda lime glass |
| Spray pyrolysis24   | 550           | 10.2         | 1.33     | 83    | 4.0 x 10⁻⁴ | 148            | 1.0 x 10⁻²⁰ | 0.5 at%   | 300            | Glass          |
| Mist-CVD21         | 600           | 7.04         | 8        | 75    | 4.4 x 10⁻⁴ | 93             | 1.5 x 10⁻²⁰ | 0.8 at%   | 550            | Glass          |
| Co-sputtering27     | 600           | 5.24         | 24.57    | 81.6  | 4.9 x 10⁻⁴ | 40             | 2.5 x 10⁻²⁰ | –         | 200            | Glass          |
| Spray pyrolysis28   | 400           | 2.5          | 17.2     | 73    | 6.8 x 10⁻⁴ | 90             | 1.0 x 10⁻²⁰ | 0.5 at%   | 525            | Glass          |
| RF sputtering30     | 300           | 1.51         | 71       | 80    | 3.5 x 10⁻⁴ | 41.56          | 4.2 x 10⁻²⁰ | 2.36 wt%  | 50             | Glass          |
| RF sputtering22     | 550           | 1.14         | 49.48    | 75.0  | 4.95 x 10⁻⁴ | 27             | 4.7 x 10⁻²⁰ | 2.0 wt%   | 100            | Soda lime glass |

*aThis work.

Fig. 3 Transparency of the IMO/AZO/mica heterostructure. a Optical transmittance spectra of the IMO heteroepitaxial structure with various thicknesses. b The figure of merit as a function of thickness of IMO/AZO/mica (red), IMO films17,20,21,23–34 (black), and different TCO films on a flexible substrate14,35–44 (blue).
thickness ratio, it was clear that the strain applied by bending was not enough to affect the behaviors of the IMO. In addition, the fluctuation of the resistance for the IMO heterostructures with the same thickness in Fig. 4 was caused by the difference in the contact resistance and displacement of the contact, since the contact was remade when the sample switched between different bending states. The stability of the heterostructure was investigated by measuring the conducting behavior as a function of bending cycles and time in flex-in mode. The resistance of the IMO/AZO/mica structure was steady and increased ~7% after 10,000 bending cycles under a bending radius of 3.5 mm, as shown in Fig. 4c. The thickness-dependent conduction stability of the IMO heterostructures is shown in the inset of Fig. 4c, which showed little change in the resistance of the heteroepitaxial structure, especially at a thickness of 300 nm after 10,000 bending cycles under a bending radius of 3.5 mm. Figure 4d shows that the conduction behavior of the IMO heterostructure was stable under compressive bending for a long duration under room-temperature environmental conditions. Moreover, the durability of the IMO/AZO/mica structure was studied under both mechanical bending and electrical bias. To simulate a practical condition, we applied a steady voltage of 5 V to the heterostructure with a bending radius of 3.5 mm, and an additional resistor was added into the circuit to control the current (0.2 A) flow through the IMO/AZO/mica heterostructure. A negligible change (<3%) was observed after one month. This indicates long-term conducting stability of the heterostructure under mechanical bending and electrical bias. Table 2 exhibits the conducting features of the flexible IMO/AZO/mica heteroepitaxial structure and recently reported TCOs on flexible substrates. Obviously, the heterostructure in this work had the best conducting performance among all flexible TCOs with a mobility over 100 cm² V⁻¹ s⁻¹. Therefore, it is evident that the transparent flexible conducting IMO/AZO/mica heterostructure exhibited a performance superior to that of other flexible TCOs and an excellent stability, which are highly beneficial for advanced flexible optoelectronic applications.

**Fig. 4 Electrical properties under mechanical bending.** a, b Resistance of the IMO heterostructure as a function of bending radius under a flex-in and b flex-out conditions with various thicknesses. c, d The stability and durability of the heteroepitaxial structure as a function of c repeated bending cycles and d continued bending time. The insets show the resistance as a function of bending cycles for various thicknesses.
Table 2  Summary of the flexible TCOs reported.

| Technique | Φ (10⁻³ Ω⁻¹) | Rᵣ (Ω/√) | T (%) | ρ (Ω cm) | μ (cm² V⁻¹ s⁻¹) | n (cm⁻³) | Thickness (nm) | Substrate |
|-----------|-------------|-----------|-------|----------|----------------|---------|----------------|----------|
| IMO²³     | RF sputter  | 97.6      | 1.37  | 81.8     | 4.5 x 10⁻⁵     | 109     | 1.3 x 10⁻¹     | 300      | Mica      |
| ITO¹⁴     | PLD         | 10        | 10.74 | 80       | 5.0 x 10⁻⁵     | 50      | 2.5 x 10⁻¹     | 150      | Mica      |
| Ti-ZnO³⁵  | ALD         | 0.3       | 500   | 82.5     | 2.0 x 10⁻³     | 19.5    | 1.5 x 10⁻¹⁰   | 40       | Willow glass |
| AZO³⁹     | RF sputter  | 10.5      | 7     | 77       | 3.5 x 10⁻⁴     | 22      | 8.0 x 10⁻³⁰   | 500      | Willow glass |
| IZO³⁷     | RF sputter  | 0.6       | 223   | 82       | 2.9 x 10⁻³     | 27.7    | 1.3 x 10⁻²⁰   | 130      | PEN       |
| GZO³⁸     | RF sputter  | 1.73      | 108.24| 89.1     | 3.0 x 10⁻³     | 8.5     | 6.0 x 10⁻¹⁰   | 200      | PEN       |
| AZO³⁹     | RF sputter  | 10.5      | 7     | 77       | 3.5 x 10⁻⁴     | 22      | 8.0 x 10⁻³⁰   | 500      | PEN       |
| ATO/Ag⁴⁰  | Co-sputtering | 21.7   | 6.11  | 81.7     | 2.4 x 10⁻⁵     | 16      | 1.5 x 10⁻²³   | 40       | PEN       |
| ZnO⁴¹     | CAPD        | 2.0       | 96.4  | 85       | 5.2 x 10⁻³     | 18      | 1.3 x 10⁻³⁰   | 550      | PET       |
| ZnO⁴²     | MOCVD       | 8.0       | 13.44 | 80       | 1.8 x 10⁻³     | 22.2    | 2.1 x 10⁻¹⁶   | 1350     | PET       |

*This work.

Conclusion
In conclusion, a high-quality transparent flexible conducting IMO/AZO/mica heteroepitaxial structure was successfully fabricated with a mobility larger than 100 cm² V⁻¹ s⁻¹, very high figure of merit (0.0976 Ω⁻¹), relatively low resistivity (~4.5 x 10⁻⁵ Ω cm), high carrier concentration (1.3 x 10²¹ cm⁻³), and high optical transmittance (81.8%) at room temperature. In addition, this heterostructure retained the excellent performance with good flexibility. This study demonstrated an extraordinary achievement and can accelerate next-generation transparent flexible optoelectronics.

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Conflict of interest
The authors declare that they have no conflict of interest.

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