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Band offsets of metal oxide contacts on TlBr radiation detectors

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ABSTRACT

Metal oxides are investigated as an alternative to metal contacts on thallium bromide (TlBr) radiation detectors. X-ray photoelectron spectroscopy studies of SnO2/TlBr and ITO/TlBr devices indicate that a type-II staggered heterojunction forms between TlBr and metal oxides upon contacting. By using the Kraut method of valence band offset (VBO) determination, the VBOs of SnO2/TlBr and ITO/TlBr heterojunctions are determined to be $1.05 \pm 0.17$ and $0.70 \pm 0.17$ eV, respectively. The corresponding conduction band offsets are then found to be $0.13 \pm 0.17$ and $0.45 \pm 0.17$ eV, respectively. The I–V response of symmetric In/SnO2/TlBr and In/ITO/TlBr planar devices is almost Ohmic with a leakage current of less than 2.5 nA at 100 V.

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I. INTRODUCTION

Thallium bromide (TlBr) is a promising material for the handheld detection of gamma rays, owing to its large bandgap (2.68 eV), high resistivity, and stopping power.1 At room temperature, however, TlBr undergoes ionic polarization when subject to an external electric field.2–4 It has been suggested that the primary effect of polarization is the migration of the Br$^-$ ions toward the anode through the mechanism of vacancy hopping and their subsequent reaction with the metallic anode material. The reaction of the metal anode with the migrating Br$^-$ ions can produce metal bromide reaction products under the electrode.3 If the device is under a prolonged bias, the metal electrode can be corroded until it is virtually impossible to apply any electric field, leading to total device failure.4,5

Reducing the reaction of the Br$^-$ ions with the electrode material has been the focus of a large body of TlBr research. Approaches include chemically treating the TlBr crystal to reduce defects at the surface,6–8 altering the electronic structure at the interface,9,10 and determining the optimal metallic electrode material.11 The use of thallium contacts has shown promising results, with extended lifetimes of over 10,000 h being reported.11,12 However, this often has to be used in tandem with bias switching techniques, which are not readily deployed for an in situ radioactive assay.

A recent study13 has reported that the use of metal oxide electrodes results in stable and low noise detection of gamma rays for more than 4000 h when under a unidirectional bias. This paper explores the use of metal oxide electrodes further by using x-ray photoelectron spectroscopy (XPS) and the Kraut method to determine the valence band offset and the resulting heterojunction at the contact/TlBr interface.14

II. METHODS

Four TlBr crystals were used in this study. They were grown at CapeSym, Inc. using the traveling molten zone technique as reported in Ref. 5. The crystals were cut to a size of $6.5 \times 6.5 \times 1.5 \text{mm}^3$ using a diamond wire saw. The samples were...
then cleaned using acetone in an ultrasonic bath for 20 min to remove any debris left from the cutting procedure. One crystal, labeled "Uncontacted TlBr" in Table I, underwent no further processing after this stage. The remaining three samples were then chemo-mechanically polished. This was achieved by grinding them on an SiC paper while simultaneously etching with a HBr:H$_2$O$_2$:H$_2$O (1:1:5) solution. The samples subsequently underwent fine polishing and smooth etching in a fresh batch of the HBr:H$_2$O$_2$:H$_2$O solution. Standard tabulated reduction potentials$^{15}$ indicate that H$_2$O$_2$ would be expected to oxidize HBr, and therefore, the mixture is thermodynamically unstable. However, at room temperature, the solution is kinetically stable. The polished surfaces were rinsed in methanol and dried in air.

The In/TlBr, SnO$_2$/TlBr, and ITO/TlBr samples had thermally evaporated indium, thermally evaporated SnO$_2$, or sputtered ITO onto its surface after processing. The SnO$_2$/TlBr and ITO/TlBr samples had $\approx 2$ nm of thermally evaporated tin, thermally evaporated SnO$_2$, or sputtered ITO onto its surface after processing. The SnO$_2$/TlBr and

\begin{table}[h]
\centering
\begin{tabular}{lccc}
\hline
Sample & Deposition type & Thickness (nm) \\
\hline
Uncontacted TlBr & … & … \\
In/TlBr & Thermal evaporation & 2 ± 0.1 \\
SnO$_2$/TlBr & Thermal evaporation & 2 ± 0.1 \\
ITO/TlBr & Sputtering & 2 ± 0.1 \\
Thick ITO$^a$ & Sputtering & 220 ± 30 \\
Thick SnO$_2$$^b$ & CVD & 350 ± 20 \\
\hline
\end{tabular}
\caption{Details of the selected samples used in the XPS study, including contact type, deposition method, and thickness.}
\end{table}

$^a$A thick ITO film was deposited on glass by Zhuhai Kaivo Optoelectronic Technology Co., Ltd.

$^b$A thick fluorine-doped SnO$_2$ film was grown on soda lime glass by chemical vapor deposition (CVD) at atmospheric pressure by the NSG group.

FIG. 1. (a) Br 3p core level peaks, (b) Tl 4f core level peaks, and (c) the valence band photoemission spectrum for the uncontacted TlBr sample.
ITO/TlBr samples also had $\approx 2$ nm of an indium metal deposited on top of the metal oxide layer. This is a typical device structure for radiation devices as the indium overlayer provides a good connection between the bonding wire and electrode. The contact area for all three samples was $5 \times 5 \text{ mm}^2$. Thicker films of SnO$_2$ and ITO were also used in the Kraut method valence band offset determination.

XPS measurements were performed on as-received samples at HarwellXPS using a Thermo Fisher Scientific NEXSA spectrometer with a micro-focused monochromatic Al K$_\alpha$ source ($h\nu = 1486.6$ eV) and an x-ray source power of 150 W. Spectra were collected with a pass energy of 40 eV and a resolution of 0.47 eV. The resolution was determined by measuring the width of the Fermi edge of a polycrystalline gold reference sample at room temperature. A low-energy electron flood gun was used for charge neutralization of the low-conductivity TlBr sample.

Absolute energy calibration is not needed for the determination of a VBO using the Kraut method. However, for the purpose of reporting core level positions, the binding energies were calibrated with reference to the Au Fermi level. The uncontacted TlBr sample had charged, and therefore, data relating to this sample were shifted based on the average C 1$s$ values of the uncharged samples. The Tl 4$f$, Br 3$p$, In 3$d$, and Sn 3$d$ core level (CL) peaks were fitted using Shirley background and Voigt (60%–70%/40%–30% mixed Lorentzian–Gaussian) line shapes. The position of the valence band maximum (VBM) was determined by extrapolating a linear fit to the leading edge of the valence band spectrum to the background level.

### Table II

| Sample          | Region | Component | Binding energy (eV) | FWHM (eV) |
|-----------------|--------|-----------|--------------------|-----------|
| TlBr            | Tl 4$f$| TlBr      | 118.94 ± 0.05      | 0.83      |
|                 | Br 3$p$| TlBr      | 181.69 ± 0.05      | 1.87      |
|                 | VBM    |           | 1.6 ± 0.1          | ...       |
| In/TlBr         | Tl 4$f$| TlBr      | 119.68 ± 0.05      | 1.01      |
|                 | Tl$_2$O$_3$ |           | 117.90 ± 0.05      | 0.54      |
|                 | In 4$s$|           | 123.80 ± 0.05      | 3.8       |
|                 | Br 3$p$| TlBr      | 182.33 ± 0.05      | 1.89      |
| SnO$_2$/TlBr    | Tl 4$f$| TlBr      | 119.51 ± 0.05      | 1.30      |
|                 | In 4$s$|           | 123.67 ± 0.05      | 4.28      |
|                 | Sn 3$d$| SnO$_2$   | 487.13 ± 0.05      | 1.14      |
|                 |        | CBP       | 487.65 ± 0.05      | 1.26      |
| ITO/TlBr        | Tl 4$f$| TlBr      | 119.64 ± 0.05      | 1.53      |
|                 | In 4$s$|           | 123.46 ± 0.05      | 3.67      |
|                 | In$_2$O$_3$ |         | 444.41 ± 0.05      | 1.03      |
|                 | CBP    |           | 445.01 ± 0.05      | 1.44      |
| Thick SnO$_2$   | Sn 3$d$| SnO$_2$   | 487.61 ± 0.05      | 1.56      |
|                 | CBP    |           | 487.03 ± 0.05      | 0.85      |
|                 | VBM    |           | 3.7 ± 0.1          | ...       |
| Thick ITO       | In 3$d$| In$_2$O$_3$ | 444.81 ± 0.05  | 0.85      |
|                 | CBP    |           | 445.60 ± 0.05      | 1.65      |
|                 | VBM    |           | 3.4 ± 0.1          | ...       |
III. RESULTS

XPS measurements of the Br 3p doublet, Tl 4f doublet, and the VBM region for the uncontacted TlBr sample are shown in Figs. 1(a), 1(b), and 1(c), respectively. The Tl 4f region was fitted using one doublet, with a spin–orbit separation of $\Delta E_{SO} = 4.43$ eV and an area ratio of 4:3. The Tl 4f$_{7/2}$ peak component was found to have a binding energy of 118.94 $\pm$ 0.05 eV. The Br 3p region was fitted using one doublet, with a spin–orbit separation of $\Delta E_{SO} = 6.7$ eV and an area ratio of 2:1. The Br 3p$_{1/2}$ peak component was found to have a binding energy of 181.69 $\pm$ 0.05 eV. The valence band maximum of the uncontacted TlBr sample was determined to be 1.6 $\pm$ 0.1 eV. The parameters of all fitted regions are displayed in Table II. The Tl 4f$_{7/2}$ component is consistent with literature reported values for the Tl–Br bond.\footnote{10} As both the Tl 4f and Br 3p regions could only be sensibly fitted using one component each, it was inferred that they both arose from the Tl–Br bond. The absence of any further components suggests that under atmospheric conditions, the surface of the TlBr crystal has not oxidized. Spectra for the Tl 4f and Br 3d peaks were also collected, and no evidence was found from them to contradict this interpretation. The binding energy values presented here for the TlBr components of the Tl 4f and Br 3p peaks were used in the analysis of the XPS data from the metal and metal oxide-contacted TlBr samples.

Figures 2(a) and 2(b) display the Br 3p and Tl 4f regions for the In/TlBr sample, respectively. The Tl 4f region was fitted using two doublets associated with the Tl 4f$_{7/2}$ peak in addition to an In 4s component at 123.80 $\pm$ 0.05 eV. It is likely that there were multiple indium components present; however, the broad nature of the In 4s component meant that they were unresolvable. The Br 3p component was found to have a binding energy of 182.33 $\pm$ 0.05 eV, and the two Tl 4f$_{7/2}$ components were found to have binding energies of 119.68 $\pm$ 0.05 eV and 117.90 $\pm$ 0.05 eV. The Br 3p component and the higher energy Tl 4f component were associated with the Tl–Br bond. Both components are slightly higher than that
found for the Tl–Br bond in the TlBr sample, but this could be due to a change in the electronic environment of the crystal atoms upon application of the contact material at the interface.

The lower binding energy Tl 4f component was associated with the presence of Tl₂O₃, possibly produced during the etching process when TlBr reacts with H₂O₂. The presence of a small amount of Tl₂O₃ on the surface of the etched and indium-contacted TlBr and its absence from the unetched TlBr surface suggests that Tl₂O₃ results from the strong oxidizing effect of H₂O₂. While according to tabulated standard reduction potentials the reaction 2TlBr + 3H₂O₂ → Tl₂O₃ + Br₂ + 3H₂O is indeed thermodynamically allowed, the concentration is small according to the low relative intensity of the Tl₂O₃-related Tl 4f peaks. This represents an unusual situation where the higher oxidation state component (Tl³⁺) is at a lower binding energy than the Tl⁺ component resulting from the Tl–Br bond but has previously been reported. The binding energy separation between the two Tl 4f½ components was calculated to be 1.78 ± 0.07 eV. This is consistent with the separation between TlBr and Tl₂O₃ reported in Ref. 16. The binding energy of the Tl 4f½, Tl₂O₃ component is also consistent with values reported by Glans et al. Here, strong satellites are observed in Tl₂O₃ due to the presence of a conduction band plasmon (CBP) loss feature, typically present in metal oxides with high doping levels, as reported by Egdell et al. These are not observed in the Tl 4f region presented in this paper. Their presence cannot be discounted, however, as the Tl₂O₃ plasmons may not be observable due to differences between the likely amorphous Tl₂O₃ formed at the surface of the TlBr crystal and the crystalline Tl₂O₃ studied by Glans et al. The plasmon lifetime may be very short and result in a broad and flat CBP component. Furthermore, the Tl₂O₃ components reported here provide only a small contribution to the intensity of the peaks (TlBr: 98.14 %, Tl₂O₃: 1.86 %), which could make the core level and CBP peaks difficult to resolve.

Figure 3(b) shows the Tl 4f doublet for the SnO₂/TlBr sample. This was fitted by a single doublet associated with the Tl 4f core level in addition to a broad In 4s component. The In 4s had a
binding energy of 123.67 eV, which was consistent with the In 4s component in the In/TlBr sample. The Tl 4f$_{7/2}$ component was found to have a binding energy of 119.51 eV, which was consistent with the Tl–Br bond presented in the In/TlBr sample. There are no Tl$_2$O$_3$ peaks evident in this region; however, this could be due to the large tail of the broad In 4s peak masking the low intensity peaks. It is, therefore, still possible that Tl$_2$O$_3$ is present to a small extent on the SnO$_2$/TlBr surface due to the chemical etch, in the same way as the In/TlBr sample.

A comparison of the Sn 3d regions from the SnO$_2$/TlBr and thick SnO$_2$ samples can be seen in Figs. 3(a) and 3(c), respectively. The Sn 3d core level peaks were fitted using two doublets for both samples. Each doublet had a spin–orbit splitting of $\Delta E_{SO} = 8.41$ eV$^{20}$ and an area ratio of 3:2.21 The lower binding energy component is associated with the SnO$_2$ bonding, while the higher energy component is thought to correspond to a CBP loss feature. Assignment of peaks in this way, for fluorine doped SnO$_2$ films, has previously been performed by Swallow et al.$^{22}$ with results consistent to those presented here. The VBM region for the thick SnO$_2$ sample is presented in Fig. 3(d). The VBM position was found to be 3.7 ± 0.1 eV with respect to the Fermi level.

Figure 4(b) shows the Tl 4f doublet for the ITO/TlBr sample. In a similar way to the SnO$_2$/TlBr sample, this region was fitted using a single doublet associated with the Tl 4f core level well as a broad In 4s component at 123.46 ± 0.05 eV. The Tl 4f component at 119.64 ± 0.05 eV is consistent with the binding energies for the Tl–Br bonding identified in both the In/TlBr and SnO$_2$-contacted samples. Again, there were no Tl$_2$O$_3$ components measured, but this could be due to the large intensity of the In 4s component.

The In 3d regions for the ITO/TlBr and thick ITO samples are presented in Figs. 4(a) and 4(c), respectively. The In 3d core level peaks were fitted using two doublets for both samples, with a spin orbit splitting of $\Delta E_{SO} = 7.52$ eV and an area ratio of 3:2 for each doublet. Here, the lower binding energy component was identified as In$_2$O$_3$, and the higher binding energy component is due to CBP losses. The VBM region for the thick ITO sample is presented in Fig. 4(d). The VBM was found to be 3.4 ± 0.1 eV below the Fermi level.

In order to assess the efficacy of metal oxide contacts on TlBr detectors, it is important to determine the band line up at the interface. The valence band offsets were determined using the Kraut$^{14}$ method,

$$\Delta E_v = \Delta E_{CL} + (E^\text{V}_{CL} - E^\text{F}_{CL}) - (E^\text{F}_{CL} - E^\text{V}_{CL}),$$  

where $E^\text{V}_{CL}$ and $E^\text{F}_{CL}$ refer to the VBM and core level energies in a bulk TlBr sample and $E^\text{V}_{CL}$ and $E^\text{F}_{CL}$ refer to the VBM and core level energies in either the thick SnO$_2$ or ITO sample. $\Delta E_{CL}$ is the separation between the Ti 4f and either an Sn 3d or In 3d component in the SnO$_2$/TlBr or ITO/TlBr, respectively. By using the TlBr component in the Ti 4f core level peak and the SnO$_2$ component of the Sn 3d peak, the valence band offset for the sample SnO$_2$/TlBr was calculated to be 1.05 ± 0.17 eV. By using the In$_2$O$_3$ component of the In 3d peak, the valence band offset for the sample ITO/TlBr was calculated to be 0.70 ± 0.17 eV. These calculated offsets, in addition to room temperature bandgaps for TlBr (2.68 eV$^3$), In$_2$O$_3$ (2.93 eV$^5$), and SnO$_2$ (3.60 eV$^{21}$) allow the heterojunction band structure to be determined, as shown in Fig. 5. Both the ITO and SnO$_2$ metal oxide contacts form a type-II staggered heterojunction when deposited on TlBr.

The I–V characteristics of 6.5 × 6.5 × 1.5 mm$^3$ symmetric In/TlBr, In/SnO$_2$/TlBr, and In/ITO/TlBr planar devices with a 5 × 5 mm$^2$ contact area are presented in Fig. 6. All three devices exhibit an Ohmic response and leakage current <2.5 nA at 100 V. The bulk resistivity for the In/SnO$_2$ and In/ITO contacted devices was found to be slightly lower than for In-only contacting: In/SnO$_2$/TlBr, (9.84 ± 0.10) × 10$^{10}$ $\Omega$ cm; In/ITO/TlBr, (7.25 ± 0.12) × 10$^{10}$ $\Omega$ cm; and In/TlBr, (1.06 ± 0.01) × 10$^{11}$ $\Omega$ cm.

IV. DISCUSSION

Metal electrodes are typically used to contact TlBr in radiation devices.$^{29}$ It has been suggested, however, that reactions of the anode material with migrating Br$^-$ ions lead to the formation of
non-conducting metal bromides, which ultimately degrades the performance of the device. Metal oxides are an alternative to metal contacts owing to their low reactivity. Device lifetime tests presented in Ref. 13 have shown that ITO-contacted devices have stable operation over long periods of time under unidirectional bias, suggesting that the Br\(^{-}\) ions do not readily react with this contact material.

Investigation into any new electrical contact must address the band alignment at the contact/TlBr interface and resulting electronic behavior of the device. Ohmic contacts are typically used in high resistivity devices such as TlBr due to their low contact resistance and linear and symmetric I–V relationships. In order to achieve an Ohmic contact, the potential barrier at the contact/TlBr interface must be as small as possible.

Here, the Kraut method was used to determine the conduction band offset at SnO\(_2\)/TlBr and ITO/TlBr interfaces. This can be used as a measure of the potential barrier formed when contacting, which was found to be 0.13 ± 0.17 and 0.45 ± 0.17 eV, respectively. Both values are smaller than the 0.80 eV barrier formed when TlBr is contacted with an In metal, calculated by aligning the indium work function and the electron affinity of TlBr, as shown in Fig. 7.25,26

The I–V relationships for symmetric In/SnO\(_2\), In/ITO, and In contacted devices are shown in Fig. 6. The resistivity values for each device satisfy the requirement for room temperature semiconductor detectors. All three devices show near Ohmic responses, with very little non-linearity and a leakage current of less than 2.5 nA in the range of −100 V to 100 V.

It is hypothesized that the use of metal oxide contacts, such as ITO or SnO\(_2\), will result in a stable Ohmic device with increased lifetime due to their low reactivity with Br\(^{-}\) ions. Moreover, implementation of a symmetrical device structure, as shown in Fig. 8, could allow for the use of bias switching to further increase the lifetime of the detector. This would need to be performed at a lower rate than when only metal contacts are used due to the low reactivity of the metal oxides.

V. CONCLUSION

In summary, planar TlBr devices with SnO\(_2\) and ITO electrodes were investigated using XPS. It was found that both the SnO\(_2\)/TlBr and ITO/TlBr interfaces form a type-II staggered heterojunction upon contact. By using the Kraut method of valence
band offset determination, the valence band offset of SnO$_2$/TlBr and ITO/TlBr heterojunctions was determined to be 1.05 ± 0.17 and 0.70 ± 0.17 eV, respectively. The corresponding conduction band offsets were then found to be 0.13 ± 0.17 and 0.45 ± 0.17 eV, respectively. The potential formed by the conduction band offset was found to be lower than that of an In/TlBr junction. The I–V relationship of symmetric In/SnO$_2$/TlBr and In/ITO/TlBr planar devices is Ohmic and similar to In/TlBr devices in the range of –100 V to 100 V. Metal oxides are typically less reactive than pure metals. This, combined with a similar electronic response compared to indium contacts shown here, suggests that metal oxides have the potential to replace metal electrodes in the fabrication of TlBr radiation devices.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors S.M. and A.D. are employed by CapeSym Inc. who make and sell TlBr crystals and detectors.

DATA AVAILABILITY

The data that support the findings of this study are available within the article.

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