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**Title:** X-Ray Photoelectron and Auger Electron Spectroscopic Study of the CdTe Surface Resulting from Various Surface Pretreatments: Correlation of Photoelectrochemical and Capacitance-Potential Behavior

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"X-RAY PHOTOELECTRON AND AUGER ELECTRON SPECTROSCOPIC STUDY OF THE CdTe SURFACE RESULTING FROM VARIOUS SURFACE PRETREATMENTS: CORRELATION OF PHOTOELECTROCHEMICAL AND CAPACITANCE-POTENTIAL BEHAVIOR WITH SURFACE CHEMICAL COMPOSITION"

by

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X-Ray Photoelectron and Auger Electron Spectroscopic Study of the CdTe Surface Resulting from Various Surface Pretreatments: Correlation of Photoelectrochemical and Capacitance-Potential Behavior with Surface Chemical Composition

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Abstract:

The surface chemistry and stoichiometry of p- and n-type CdTe photoelectrodes treated with oxidizing and reducing etches have been characterized by X-ray photoelectron and Auger electron spectroscopies. The results of surface analysis have been correlated with the photoelectrochemical and capacitance-potential behavior of the photoelectrodes. "Oxidized" surfaces are covered by a thin Te⁶⁻/TeO₂ layer (or a thicker Te⁶⁻ layer, if the etching procedure is slightly altered), resulting in Fermi level pinning; a constant photovoltage is found for a wide range of redox potentials and potential-independent space charge layer capacitance obtains. "Reduced" surfaces closely resemble ion sputtered CdTe in chemical state and stoichiometry, resulting in more nearly "ideal" behavior: the semiconductor/electrolyte interface is rectifying in the dark; capacitance-potential behavior follows the Mott-Schottky equation near flat band conditions; and photovoltage varies with redox potential, from 0 to 0.7 V for p-CdTe.

Introduction

Several recent reports have revealed a relationship between CdTe surface composition and the photovoltage (or barrier height) obtained from CdTe/metal and CdTe/electrolyte junctions. Vacuum cleavage, cleavage in air and polishing followed by chemical etching are the three commonly used methods of crystal surface preparation. The two latter methods generally result in non-stoichiometric, oxidized surfaces that are contaminated with Te and/or TeO2 prior to semiconductor/metal or electrolyte junction formation. An only recently explored exception to the general rule of oxidized CdTe surfaces is obtained by treating CdTe with a chemical reducing agent, such as dithionite, S2O42-, or hydrazine, N2H4. Oxidized surfaces exhibit interfacial electronic properties characteristic of Fermi level pinning, barrier heights, VB, and photovoltages, EV, are nearly independent of the work function, #m, of the contacting metal or the redox potential, Eredox, of the electrolytic solution. In contrast, VB's obtained from vacuum cleaved or reductively etched CdTe depend strongly on #m or Eredox and are regarded as more nearly ideal.

In this work we detail experiments using XPS and Auger spectroscopy that give a more complete understanding of the relationship between surface composition and the characteristics of the semiconductor/electrolyte junction. We correlate surface composition and the potential dependence of the interfacial capacitance of oxidatively and reductively etched p- and n-CdTe in contact with CH3CN/electrolyte. Further, we correlate
photovoltage vs. redox potential\textsuperscript{1,2} with surface composition of n- and p-CdTe. Measurements made previously on \textit{S}_2\textit{O}_4^{2-} reduced CdTe have now been extended to \textit{N}_2\textit{H}_4 reduced CdTe.

**Experimental**

**Crystal Preparation and Etching Procedures.** Single crystals of p- and n-CdTe (Cleveland Crystals, Cleveland, OH) were polished to a mirror finish with 0.3 \textmu m alumina. Crystals were then cleaned for 1 min in boiling 5 M KOH. The dichromate oxidizing etch was a 30 s immersion in 4 g \textit{K}_2\textit{Cr}_2\textit{O}_7/10 ml \textit{HNO}_3/20 ml \textit{H}_2\textit{O}. The 15\% \textit{HNO}_3 etch lasted 30 min. Prior to the reducing etches, the crystal surface was etched in \textit{HNO}_3/\textit{Cr}_2\textit{O}_7^{2-}. The dithionite etch was a 3 min immersion in boiling 0.6 M \textit{Na}_2\textit{S}_2\textit{O}_4/2.5 M \textit{NaOH}. The hydrazine etch was either 5 min in 30\% \textit{N}_2\textit{H}_4, pH 14, at \textdegree 50°C or -15 min in 95\% \textit{N}_2\textit{H}_4 at 25°C. After thorough rinsing, crystals were stored in an evacuated tube until surface analysis.

**Surface Spectroscopy and Photoelectrochemical Methods.** XPS and AES data were recorded on PDP 11/04-controlled Physical Electronics (Perkin Elmer) Model 548 and Model 590 spectrometers, respectively. Full details of instrumentation, spectrometer calibration, and data manipulation are given elsewhere\textsuperscript{2,26,27} Samples were grounded to the spectrometer. A full description of the preparation of p- and n-CdTe electrodes and details of the electrochemical and capacitance-voltage measurement techniques are published elsewhere\textsuperscript{1,2}

**Results and Discussion**

1. **X-Ray Photoelectron Spectroscopy.** To identify the chemical state of the CdTe surface, precise core electron binding energies, \pm 0.1 eV, were measured for the Cd 3d, Te 3d, and C 1s levels.
Samples examined included oxidized CdTe etched with either HNO₃/Cr₂O₇²⁻ or 15% HNO₃; reduced CdTe etched with either OH⁻/S₂O₄²⁻ or N₂H₄; and Ar ion sputtered CdTe. Because Cd and Te have similar atomic weights preferential sputtering effects are expected to be minimal;¹⁸,²⁹ this has been confirmed experimentally.¹³ For a given surface treatment, no significant or systematic differences between the spectra of n- and p-type CdTe were observed. To identify surface impurities and determine overall stoichiometry, a low resolution survey scan was obtained for each sample. In the following discussion, only the 3d⁵/₂ binding energies of the Cd and Te 3d doublets are cited because the spin-orbit splitting is independent of oxidation state, see Table I.

a. Chemical State. Figure 1 and Table I summarize the XPS results for oxidized, reduced, and ion sputtered CdTe. There is little difference in the Cd 3d binding energies for the various surface treatments, consistent with the absence of significant changes in the Cd oxidation state.⁸ In contrast, the Te 3d binding energies show large differences. Ion sputtered CdTe has a single Te 3d⁵/₂ peak at 572.5 eV, characteristic of Te²⁻. Dithionite or hydrazine reduced CdTe has a very similar Te 3d region, with the addition in some cases of a weak line at ~3.5 eV higher binding energy than the Te²⁻ peak. This high energy peak, with an average intensity only 4% of that of the Te²⁻ line, is a result of limited air oxidation of the CdTe surface, and the Te 3d⁵/₂ binding energy, ~576 eV, is characteristic of TeO₂.³¹ A few of the dithionite reduced CdTe surfaces showed slight asymmetry on the high binding energy side of the Te²⁻ peak, due to the presence
of a small amount of Te*. Thus, both reducing etches yield XPS binding energies which closely resemble bulk (ion sputtered) CdTe.

The Te 3d spectra of oxidized CdTe are quite different from those of reduced and ion sputtered samples. The dichromate etch results in the spectrum shown in Figure 1. The high energy Te 3d5/2 line, at 576.2 eV, is due to TeO2 formed during the oxidizing etch and/or air oxidation of the surface following the etch. Allowing the dichromate oxidized samples to sit in air for ~2 weeks noticeably enhances the TeO2 signals. The lower energy Te 3d5/2 peak, at 573.0 eV, is midway between the binding energies measured for Te2- and Te*. This peak is quite broad (FWHM ~1.8 eV) compared to the width of the 3d lines for sputtered CdTe (1.56 eV) and sputtered Te* (1.3 eV). Together, these data suggest that both Te* and Te2- are present on the dichromate oxidized surface, with the Te* being part of a thin layer of oxidation products left by the etch and the Te2- belonging to bulk CdTe. Auger data support this assignment of Te2- and Te*, vide infra. CdTe oxidized in 15% HNO3 yields yet a different Te 3d spectrum. There is only a single Te 3d5/2 peak, but the binding energy, 573.3 eV, is characteristic of Te*. In fact, there is no Cd signal visible at all for such samples which, according to Auger depth profile analysis, are covered by a fairly thick Te* layer. 12, 33, 34

The two Te 3d5/2 lines found for air oxidized elemental Te, Table I, indicate the presence of both Te* and an overlayer of TeO2. Sputtered elemental Te has only a single Te 3d5/2 line, giving the binding energy for Te* of 573.5 eV.

b. Stoichiometry. The Te:Cd ratio obtained from sputtered CdTe using integrated 3d peak areas and correcting for atomic
sensitivity factors is 1.0 ± 0.1 (Table II) supporting the conclusion (vide supra) that preferential sputtering is not a problem. For dithionite or hydrazine reduced CdTe, the ratio is 1.0 ± 0.3, with the rather large standard deviation an indication that the surface is not always precisely stoichiometric. The overall Te:Cd ratio of 1.6 for dichromate oxidized CdTe indicates a substantial enrichment of the surface in Te relative to the bulk, consistent with the hypothesis that the dichromate etch leaves oxidiation products of Te on the surface. This Te:Cd ratio, though greater than unity, is far less than that obtained by other workers using the same etch. They found that the dichromate etch left a contaminant layer so thick that the Cd signal was entirely obscured. A possible explanation for this behavior is presented in the next section.

2. Auger Spectroscopy and Depth Profile Analysis. AES gives results for oxidized and reduced samples which are in qualitative agreement with the results from XPS. Figure 2a shows the Auger spectrum of dichromate oxidized CdTe. The Te/Cd ratios determined from AES for oxidized samples are significantly larger than those found by XPS (Table II), consistent with the difference in electron escape depths. For the Cd MNN Auger line, the kinetic energy (KE) of electrons is -380 eV, giving an escape depth of ~7 Å; the Cd 3d5/2 line, excited by Mg Ka irradiation, gives electrons with KE ~850 eV, for an escape depth of ~15 Å. Thus, Auger data substantiate the hypothesis of bulk CdTe covered by a thin (<30 Å) Te*/TeO2 layer. A depth profile of dichromate etched CdTe, Figure 2b, shows that O extends nearly into the bulk, though the relative amount of O decreases with sputtering time, suggesting the
Te°/TeO₂ layer observed by XPS becomes richer in Te° as bulk CdTe is approached. This profile also shows that the surface contaminant layer is relatively thin. The Cd and Te signals reach their bulk values in <1 min at a sputtering current density of ~25 μA/cm² at 2 kV.

If a short time, ~30 s, elapses between the Cr₂O₇²⁻/HNO₃ etch and the H₂O rinse, the CdTe surface becomes covered with Te°, Figure 2a. An Auger depth profile of such a sample, Figure 2c, reveals that little O is present in this layer and that it is fairly thick relative to the Te°/TeO₂ layer formed on "immediately rinsed" dichromate etched CdTe, Figure 2b. Presumably, this is the explanation for the difference in thickness of the Te°/TeO₂ layer found in the present study compared to that found by others.¹,¹¹ The 15% HNO₃ etch gives a thick, dull-looking Te° layer, even if the crystal is rinsed immediately after etching.

With the exception of S and C impurity peaks, the Auger spectrum of dithionite reduced CdTe closely resembles that of Ar ion sputtered CdTe, Figure 2a. The S peak is presumably a result of impurities left behind by the dithionite etch. The sulfur does not, however, play an important role in the interfacial energetics of the reduced CdTe surfaces; an oxidized CdTe electrode may be electrochemically reduced to give the same differential capacitance vs. potential curve as obtained for a sample reduced with the dithionite etch. In addition, Auger spectroscopy reveals that N₂H₄ reduction of CdTe yields a nearly sulfur-free surface closely resembling that of ion sputtered CdTe. Importantly, the photo-electrochemical and capacitance-voltage behavior of hydrazine
reduced \( p^- \) and \( n^-\)CdTe is quite similar to that obtained as a result of dithionite reduction.

3. Photoelectrochemical and Capacitance-Voltage Measurements. Scheme I summarizes the situation for oxidatively etched \( p^- \) and \( n^-\)CdTe. Oxidized \( n^-\)CdTe yields photovoltages of 500-600 mV for redox potentials between +0.7 and -1.7 V vs. SCE, while \( p^-\)-CdTe yields little or no photovoltage (\( E_V < 100 \) mV) for \( E_{\text{redox}} \) between +0.2 and -1.7 V vs. SCE. This suggests that the Fermi level of \( n^-\)CdTe is pinned to a value at least -0.6 V below the conduction band. For oxidized \( p^-\)-CdTe, the Fermi level is apparently pinned quite near the valence band edge. Capacitance-voltage measurements in \( \text{CH}_3\text{CN/electrolyte} \) (no redox couple) for oxidized CdTe reveal nearly potential-independent space charge layer capacitance.
(-130 nF/cm² for n-type; -50 nF/cm² for p-type), implying that the band bending is potential-independent and thus supporting the case made above for Fermi level pinning.²

When CdTe is reductively etched, either with dithionite or hydrazine, the interfacial energetics represented by Scheme II apply. In the case of n-CdTe, when \( E_{\text{redox}} \) is negative of -1.2 V vs. SCE, the approximate location of the conduction band edge, ohmic contact occurs between solution species and the semiconductor. But for \( E_{\text{redox}} \) between -1.1 and +0.1 V vs. SCE, an approximately linear relationship between \( E_V \) and \( E_{\text{redox}} \) holds, with \( \Delta E_V/\Delta E_{\text{redox}} \approx 0.6 \). For reduced p-CdTe, ohmic contact exists for redox potentials positive of the valence band edge, -0.2 V vs. SCE, while \( E_V \) varies from 0 to -0.65 V for \( E_{\text{redox}} \) between -0.3 and -1.8 V vs. SCE, with a maximum rate of change, \( \Delta E_V/\Delta E_{\text{redox}} \), of 0.6. Capacitance-voltage measurements also suggest a more ideal interface for reduced CdTe, and linear Mott-Schottky plots are obtained. From the Mott-Schottky plots, values of the flat band potential and the donor (n-type, \( 2 \times 10^{17} \) cm\(^{-3} \)) and acceptor (p-type, \( 2.5 \times 10^{15} \) cm\(^{-3} \)) densities are calculated. The \( E_{\text{FB}} \) values and donor/acceptor densities allow calculation of the location of the valence and conduction band edges, yielding values of -0.2 and -1.5 V vs. SCE, respectively.

**Conclusions**

Surface spectroscopy reveals that the Fermi level pinned behavior characteristic of oxidized p- and n-CdTe is a result of oxidation products left on the surface after etching. Specifically, Te\(^+\), a small band gap semiconductor, \( E_g = 0.35 \) eV,\(^{33,36} \) is found on the surface of oxidized samples. The Te\(^+\)
Scheme II. Interfacial energetics for reductively etched, stoichiometric p- and n-CdTe. Note that the extent of band bending (and thus the barrier height and photovoltage) changes with redox potential. For very positive redox potentials at n-CdTe or very negative redox potentials at p-CdTe, the amount of band bending reaches its maximum, hence additional potential drop is across the Helmholtz layer, cf. Scheme I.

overlayer has a large work function and would be expected to give a larger barrier on n- than on p-CdTe. This is entirely consistent with the reasonably large photovoltage obtained for surface oxidized n-CdTe and the near-zero photovoltage obtained for surface-oxidized p-CdTe in electrolyte/redox couple solutions. That Fermi level pinning is a result of the surface treatment is illustrated by the results from the reducing etches, namely the nearly ideal behavior of reduced p- and n-CdTe coupled with the close resemblance of the surface composition to clean CdTe.

Because most solid state measurements have been made on air cleaved or oxidatively etched CdTe crystals, it is reasonable to
conclude that the nearly constant barrier heights found in these studies result from the oxidation of the CdTe surface. Results from metal deposition on vacuum cleaved CdTe and from electrochemical studies on reduced CdTe support this conclusion.

Acknowledgements. This research was supported in part by the Office of Naval Research and in part by the National Science Foundation Materials Research Laboratory Program at the M.I.T. Center for Materials Science and Engineering.

References

26. The software for computer control of the XPS spectrometer, as well as data acquisition and curve fitting, is part of the MACS (Version VI) software package: Physical Electronics Division, Perkin-Elmer Corp., Eden Prairie, MN.

Figure Captions

Figure 1. X-ray photoelectron spectra of the Te 3d region showing, from top: Ar ion sputtered CdTe; S2O42-/OH- reduced CdTe; NH42+ reduced CdTe; Cr2O72-/HNO3 oxidized CdTe; and 15% HNO3 oxidized CdTe.

Figure 2. (a) Auger spectra of CdTe showing, from top: Cr2O72-/HNO3 oxidized CdTe, rinsed immediately after etching; Cr2O72-/HNO3 oxidized CdTe, rinsed 30 s after etching; S2O42-/OH- reduced CdTe; and Ar ion sputtered CdTe. (b) Auger depth profile of Cr2O72-/HNO3 oxidized CdTe, rinsed immediately after etching. (c) Auger depth profile of Cr2O72-/HNO3 oxidized CdTe, rinsed 30 s after etching. The Ar ion current densities are the same for (b) and (c).
Table I: Summary of XPS Multiplier Data for Chemical State of Oxidized, Reduced, and Ion Sputtered QTe.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Te 3d5/2 Core Level</th>
<th>OI 3d5/2 Core Level</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Assignment</td>
<td>Center</td>
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<tr>
<td>Ion Sputtered QTe</td>
<td>2(^-)</td>
<td>572.47(5)</td>
</tr>
<tr>
<td>S(_2)O(_4)2(^-)/NaOH Reduced QTe</td>
<td>2(^-)</td>
<td>572.50(7)</td>
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<tr>
<td></td>
<td>Te(_2)</td>
<td>575.96(11)</td>
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<tr>
<td></td>
<td>N(_2)H(_4) Reduced QTe</td>
<td>Te(_2)</td>
</tr>
<tr>
<td>Cr(_2)O(_7)2(^-)/HNO(_3) Oxidized QTe</td>
<td>&quot;Mixed&quot;</td>
<td>572.98(11)</td>
</tr>
<tr>
<td></td>
<td>Te(_2)/Te(_6)</td>
<td>Te(_2)</td>
</tr>
<tr>
<td>15% HNO(_3) Oxidized QTe</td>
<td>Te(^+)</td>
<td>573.24(10)</td>
</tr>
<tr>
<td>Oxidized Elemental Te</td>
<td>Te(^+)</td>
<td>573.5(10)</td>
</tr>
<tr>
<td></td>
<td>Te(_2)</td>
<td>576.4(2)</td>
</tr>
<tr>
<td>Ion Sputtered elemental Te</td>
<td>Te(^+)</td>
<td>573.54(10)</td>
</tr>
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</table>

\(a\) Each tabulated value for QTe is the average of 4–6 runs. Uncertainty in the last digit of each entry, given parenthetically, is estimated as twice the standard deviation of the average.

\(b\) Data obtained at 25 eV pass energy, 0.2 eV/point.

\(c\) Center of the 3d5/2 line from curve fit data, in eV.

\(d\) Separation of the 3d3/2 and 3d5/2 lines, in eV.

\(e\) Average full width at half-maximum of the 3d3/2 and 3d5/2 lines, in eV.

\(f\) Relative intensities are calculated from integrated peak areas of the curve fit data and are corrected for atomic sensitivity factors as given in ref. 32.
<table>
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<tr>
<th>Sample</th>
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<th>Te</th>
<th>O</th>
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</thead>
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<td>Ion Sputtered CdTe</td>
<td>1.0</td>
<td>1.0(1)</td>
<td>1.0</td>
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<td>NaOH/S₂O²⁻ Reduced CdTe</td>
<td>1.0</td>
<td>1.0(2)</td>
<td>0.8(3)</td>
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<td>N₂H₄ Reduced CdTe</td>
<td>1.0</td>
<td>1.0(3)</td>
<td>1.1(2)</td>
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<tr>
<td>HNO₃/Cr₂O₇²⁻ Oxidized CdTe</td>
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<td>1.6(3)</td>
<td>3.5(6)</td>
</tr>
<tr>
<td>15% HNO₃ Oxidized CdTe</td>
<td>0.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Oxidized Elemental Te</td>
<td>---</td>
<td>1.0</td>
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**Relative Intensity**

**Table II. Comparison of Stoichiometry Determined by XPS and AES for Reduced, Oxidized, and Ion Sputtered CdTe.**

- **Intensity** for XPS data is taken from survey scans, 100 eV pass energy, by integrating the Cd 3d5/2, Te 3d5/2, and O 1s lines and correcting for the respective sensitivity factors. The XPS intensities include all oxidation states of each element, i.e. Te²⁻, Te⁴⁺, and TeO₂ are all included in the Te intensity.

- **For AES data, relative intensity of Te to Cd** was determined experimentally for ion sputtered CdTe using the MNN Auger lines. (Preferential sputtering effects are unimportant, see ref. 8 and text).
XPS of CdTe, Te 3d Region

3d₃/₂

Te²⁻

Ion Sputtered CdTe

Dithionite Reduced CdTe

Hydrazine Reduced CdTe

Dichromate Oxidized CdTe

15% HNO₃ Oxidized CdTe

Te⁰

N(E)/E

BINDING ENERGY, eV

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