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# Conduction band-edge d-states in high-k dielectrics due to Jahn–Teller term splittings

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#### Abstract

X-ray absorption spectroscopy (XAS) is used to study conduction band edge electronic structure of high-k transition metal (TM) and trivalent lanthanide series rare earth (RE) oxide dielectrics. Empty TM/RE d-states are studied by intra-atomic transitions originating in core level spin-orbit split p-states, and conduction band states are studied in inter-atomic transitions which originate in the oxygen atom 1s core level state. In non-crystalline Zr and Hf silicate alloys, the local bonding symmetry, or *crystal field* splits these d-states into doubly and triply degenerate features. In nano-crystalline oxides, there are additional d-state splittings due to contributions of more distant neighbors that completely remove d-state degeneracies via the Jahn–Teller effect mechanism. This gives rise to highly localized band edge states that are electronically active in photoconductivity, internal photoemission, and act as bulk traps in metal oxide semiconductor (MOS) devices.

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### 1. Introduction

TM/RE oxides, silicate and aluminate alloys, and other oxide-based dielectrics have been proposed as alternative gate dielectrics to replace  $SiO_2$  in advanced silicon devices [1]. The proposal is made on the basis of their high relative dielectric constants, k, and the high values of capacitance that can be obtained in films that are physically thicker films than  $SiO_2$  at the same capacitance level.

The lowest conduction band states in transition metal (TM) and trivalent lanthanide rare earth (RE) oxides and silicate alloys are associated, respectively with TM and RE anti-bonding d\*-states that are mixed with oxygen

atom (O) 2p\* anti-bonding states [1]. X-ray absorption spectroscopy (XAS) has been used to study empty TM/ RE d-states via intra-atomic transitions from core level TM/RE atom spin orbit split p-states. It has also been used to study anti-bonding TM/RE d\*-states via interatomic transition from O-atom core level 1s states, the O K<sub>1</sub> edge, that terminate in anti-bonding "molecularorbital" states comprised of O-states 2p\*-states mixed with TM/RE d\*-states. The final states for these O K<sub>1</sub> transitions are qualitatively and quantitatively similar to the conduction band states that are of importance in Si metal oxide semiconductor (MOS) device performance and reliability [1].

This article extends XAS studies from TM/RE oxides and their silicate and aluminate alloys to complex oxides comprised of TM oxides that combined with (i) a different TM oxide, as in  $(HfO_2)_{0.5}$ (TiO<sub>2</sub>)<sub>0.5</sub>, or (ii) a RE oxide as in

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 $(Sc_2O_3)_{0.5}(Dy(La)_2O_3)_{0.5}$ . These studies have identified a new aspect of the d-state structure, term splittings of the triply degenerate  $d_{5/2}$ , and double degenerate  $d_{3/2}$  empty and mixed d-states that result from the Jahn–Teller effect [2]. These band edge states are electronically active and may limit the application of TM/RE high-k dielectrics in advanced Si devices.

#### 2. X-ray absorption spectroscopy studies

Fig. 1 presents XAS spectra for a Zr silicate alloy prepared by remote plasma CVD [3]. The figure includes the O K<sub>1</sub> edge spectrum, as well as the numerical differentiation of that spectrum. Two Zr d\*-state features are evident; the lowest of these is shifted in energy by about 0.5 eV with respect to the dominant feature in the O K<sub>1</sub> edge of a nano-crystalline ZrO<sub>2</sub> film, and second is overlapped in energy by the Si 3s\* feature of the Si-O bonded component of the silicate film. Zr M<sub>2,3</sub> spectra for these silicate have been obtained as well. A devolution of these spectra empty Zr 4d-state and 5s-state features indicates the following spectral splittings: (a) an empty d-state splitting,  $\Delta(4d_1, 4d_2)$ , of 2.2 $\pm$ 0.2 eV, and (b) a 4d<sub>1</sub>-5s,  $\Delta$ (4d<sub>1</sub>,5s), splitting of  $11.8\pm0.3$  eV. The difference between the 4d-state spittings in the O K<sub>1</sub> edge,  $3.2\pm0.2$  eV and in M<sub>2.3</sub> 3 edge,  $2.2\pm0.2$ eV, is a demonstration of the qualitative differences between the 4d-states features in the molecular orbitals of the  $O K_1$ edge final states and  $M_{2,3}$  spectrum empty 4d final states.

Fig. 2 is the  $L_{2,3}$  edge for a nano-crystalline thin film of TiO<sub>2</sub> prepared by step-wise plasma-assisted oxidation of



Fig. 1. Absorption and differentiated absorption spectra for the O K<sub>1</sub> edge of a Zr silicate alloy with a  $ZrO_2$  fraction of  $0.3\pm0.05$ . The dashed lines indicate the energies of d\*-state features of as-deposited  $ZrO_2$  with a nanocrystalline morphology.



Fig. 2. The Ti  $L_{2,3}$  spectrum for a thin film of nano-crystalline TiO<sub>2</sub>. The spectral features corresponding to empty Ti  $3d_{5/2}$  and  $3d_{3/2}$  states are indicated, as well as the two groups of these d-states associated with the Ti 2p spin-orbit splitting with transitions originating in  $2p_{3/2}$  and  $2p_{1/2}$  states.

ultra-thin Ti films prepared by e-beam evaporation in a UHVsystem [4]. The L<sub>3</sub> transitions originate in the spin-orbit split Ti  $2p_{3/2}$  state, and terminate in empty Ti  $3d_{5/2}$ ,  $3d_{3/2}$  and 4pstates. The degeneracies of the Ti  $3d_{5/2}$  and  $3d_{3/2}$  are completely removed with the 3d states being split into three states and two features, respectively. The L<sub>2</sub> transitions originate in the Ti  $2p_{1/2}$  state, and terminate in the same empty d-states with the L<sub>2</sub> spectra shifted in energy by the  $L_{2.3}$  spin-orbit splitting of ~5.5 eV. A determination of the multiplicity of features in the L<sub>2</sub> edge by numerical differentiation also indicates that the  $3d_{5/2}$  feature is split into three states, and the  $3d_{3/2}$  into 2 states thereby completely removing the degeneracies of these states as well. The degeneracy removal or term splitting is a direct result from the Jahn-Teller (J-T) effect [2]. The J-T effect is present in the spectra of all the complex oxides of this study, and has important implications for states at the conduction band edge that are can be populated by optical absorption, internal photoemission and electron trapping in MOS devices [5,6].

Absorption, and numerically differentiated absorption spectra as presented in Fig. 3 for the O K<sub>1</sub> edge of nanocrystalline HfO<sub>2</sub>. The positions of the spectral features and their relative splittings have been estimating by considering the second derivative of the absorption spectrum as well. These spectra display a doublet at lower photon energies, and symmetrically split triplet at higher photon energies, consistent with the lower d-state having  $5d_{3/2}$  symmetry and the upper d-state,  $5d_{5/2}$  symmetry. The relative strengths and widths of the 4d\* features in the O K<sub>1</sub> edge of nano-crystalline ZrO<sub>2</sub> is consistent with a similar ordering of the Zr 4d\* states as well.



Fig. 3. Absorption and differentiated absorption spectra for the O  $K_1$  edge of nano-crystalline HfO<sub>2</sub>. The energy markers at the top of figure indicate the energies of the 5 d\*-state features.

Complex oxides comprised of TiO<sub>2</sub>, and either ZrO<sub>2</sub> or HfO<sub>2</sub>, have been prepared by reactive evaporation in an MBE system using e-beam evaporation [7], and complex oxides comprised of Sc<sub>2</sub>O<sub>3</sub> and RE oxides, e.g., La<sub>2</sub>O<sub>3</sub>, have been prepared in a similar system, but using effusion cells [8]. The Ti L<sub>2,3</sub> spectra of the group IVB complex oxides, e.g.,  $(Hf(Zr)O_2)_x(TiO_2)$ , with *x*~0.33, 0.5 and 0.67 display a complex removal d-state degeneracy, and the O K<sub>1</sub> edges display d\*-state features indicative degeneracy removal as well. As an example, Fig. 4 displays the



Fig. 4. Absorption and differentiated absorption spectra for the O K<sub>1</sub> edge of nano-crystalline  $(TiO_2)_{0.33}(ZrO_2)_{0.67}$ . The markers indicate the energies of the 5 d\*-state features.

absorption and numerically differentiated absorption spectra for the O K<sub>1</sub> edge 0f  $(ZrO_2)_{0.67}(TiO_2)_{0.33}$ . The energy markers for the five d\*-features have been estimated to  $\pm 0.2$  eV from using second derivative information as well. Consistent with degeneracy removal for the Ti L<sub>2,3</sub> edge, triplet d<sub>5/2</sub> state is the lower energy feature, the doublet d<sub>3/2</sub> state is the higher energy feature. Fig. 5 and displays the Ti L<sub>3</sub> edge spectra for two different Zr titanate complex crystals: 5(a)  $(ZrO_2)_{0.67}(TiO_2)_{0.33}$ , and 5(b)  $(ZrO_2)_{0.33}$ (TiO<sub>2</sub>)<sub>0.67</sub>. The Ti 3d<sub>5/2</sub> triplet state splittings associated with  $\sigma$ -bonded MO states are the same for each of these oxides, while the doublet Ti 3d<sub>3/2</sub> splittings associated with  $\sigma$ -bonded MO states are significantly different.

Sc  $L_{2,3}$  spectra have been obtained in thin film LaScO<sub>3</sub> prepared by reactive evaporation, and subjected to annealing at temperatures up to 1000 °C. The spin-orbit splitting of the Sc 2p state is approximately 4 eV. The Sc  $L_3$  edges for LaScO<sub>3</sub> are shown in Fig. 6(a) for a film deposited at ~20–25 °C, and in Fig. 6(b) for a film annealed at 1000 °C. Each



Fig. 5. Ti  $L_3$  edge spectra for (a)  $(TiO_2)_{0.33}(ZrO_2)_{0.67}$  and (b)  $(TiO_2)_{0.33}(HfO_2)_{0.67}.$ 



Fig. 6. Sc L3 spectra for nano-crystalline LaScO\_3: (a) as-deposited, and (b) after a 1000  $\,^\circ C$  anneal in an inert ambient.

of these spectra demonstrates a complete removal of d-state degeneracy, with three features evident in the  $3d_{5/2}^*$  portion of the spectrum at lower photon energies, and two features in the  $3d_{3/2}^*$  portion at higher photon energy. The major difference between the as-deposited and annealed samples is a decrease in the line-width of each of the features in the annealed film.

The behavior the La  $M_{2,3}$  edge is qualitatively different, and spectra are displayed in Fig. 7(a) for an as-deposited sample, and in Fig. 7(b) for a film annealed at 1000 °C. The two sharp features are completely degenerate 5d-states; this observation is supported by the symmetry of the derivate spectra. The separation between the two relatively sharp features is 80 eV and is the spin-orbit splitting of the La 3p state. The line-widths are these samples are large, >5 eV and are due to the short core hole life-times in these deep atomic states.

These results for LaScO<sub>3</sub> films provide a more sensitive measure of nano-crystallinity than can be obtained by conventional X-ray diffraction (XRD). The difference between as-deposited and annealed LaScO<sub>3</sub> thin films is in the crystallite or grain size. Crystallinity has been detected by XRD after an 800 °C anneal, whereas the asdeposited films are nano-crystalline by the combination of an XAS detected term-split for Sc, and the absence of a term splitting for La. A similar situation prevails for thin film



Fig. 7. O K<sub>1</sub> spectra for nano-crystalline LaScO<sub>3</sub>: (a) as-deposited, and (b) after a 1000  $^\circ C$  anneal in an inert ambient.

LaAlO<sub>3</sub>, i.e., these films are nano-crystalline on deposition as well.

Finally, J–T term-splittings have also been observed in the O K<sub>1</sub> edge in thin film LaAlO<sub>3</sub>. These are accompanied by degeneracy removal in the La  $M_{2,3}$  edge as well. This means the La atom in this perovskite structured material has moved out of its center of symmetry with respect to 12 O atom neighbors, and that this distortion is not restricted to short range order in amorphous material, where a crystal field effect would be completely suppressed by 12-fold coordination in a spherically symmetric local bonding environment.

## 3. Discussion

## 3.1. Jahn–Teller term splittings

Three different energy level splittings have been identified in the spectra presented above. One of these is atomic in origin, and has been presented in the core level p-spectra in Fig. 2 for the Ti 2p core level transitions in TiO<sub>2</sub>, and Fig. 6 for the La 3p core level transitions in LaScO<sub>3</sub>. This is an initial state effect in which the triply degenerate core level atomic core p-state is split into a  $p_{3/2}$  doublet and a  $p_{1/2}$ singlet state by the interaction between the orbital angular momentum L, and the electron spin S, the so-called  $L \cdot S$ coupling mechanism [9]. The nominal spin-orbit splittings observed in the XAS spectra are 6 eV for the Ti 2p state, 5 eV for the Sc 2p state, and 81 eV for the La 3p state [10]. Spin-orbit splittings have also been found in XAS spectra for Zr, 12 eV, and Hf, 58 eV; however, these spectra have been shown in this article. For a given principal quantum number, these splittings scale with increasing atomic number of the atomic species. Within a given atomic species, the spin orbit splittings decrease as the principal quantum number increases [9]. These splittings are small for the valence states, and are generally not observed in the highest energy valence band, the conduction band and the empty d-states of TM/RE atoms in the oxide dielectrics studied in this article.

The d-state spittings in the valence bands of the TM/RE elementary and binary oxides are dominated by two solid state effects, and these are illustrated in Fig. 8 for spherically symmetric and octahedral atomic bonding arrangements. In a spherically symmetric solid state environment, the five d-states are degenerate. An example of this is the 12-fold coordination of La in a perovskite structure. In this case the La atom is the center of a cube, and its nearest neighbors are 12 O atoms situated at the centers of the cube edges. For atoms at the center of an octahedron of six nearest neighbors, the d-states are split into a triply degenerate  $t_{2g}$  state, and a doubly degenerate  $e_g$ . This splitting has been designated as a *crystal field splitting*, and will be distinguished in this article from distortions in bonding which result in partially or completely removing the 2- and



Fig. 8. Crystal field and Jahn–Teller d-state splittings for symmetric bonding, ideal octahedral bonding, partially distorted octahedral bonding with a *z*-axis distortion, and completely distorted octahedral bonding with distortions in the x, y and z directions.

3-fold degeneracies of the tri-valent and quartra-velent TM/ RE atoms of this article that are bonded in 6-fold, 8-fold and 12-fold coordination. These bonding distortions can be described as Jahn–Teller (J–T) distortions that arise because of the low occupancy of the valence bonding d-states, one for Sc, Y, La, and the trivalent lanthanide RE atoms, and two for Ti, Zr and Hf.

The symmetric bonding arrangements can occur in a crystalline oxide, or in an amorphous oxide where there is only short range atom order. In non-crystalline Zr and Hf silicates, the Zr and Hf atoms are bonded in a cubic arrangement with eight O atom nearest neighbors. This gives a crystal field splitting that is reversed in order compared to a 6-fold octahedral arrangement; i.e., the lower state is a 2-fold degenerate  $e_g$  state, and the higher state is the 3-fold degenerate  $t_{2g}$  state.

In contrast, the spectroscopic term splittings observed in the L<sub>2,3</sub> spectra for Sc and Ti are consistent with the Jahn-Teller effect for Sc and Ti atoms with 6-fold coordinated in a completely distorted octahedral bonding environment [2]. Three bonding distortions are required for complete removal of the t<sub>2g</sub> and e<sub>g</sub> degeneracies. These are represented symbolically in Fig. 9 as elongations along the z-axis, and a distortion in the x,y bonding plane. As noted above, these L<sub>2,3</sub> transitions, as well as the La M<sub>2,3</sub> transitions, terminate in empty d-states which reflect a complete d-state degeneracy removal. The final d-state splittings, and the transition matrix elements which determine their relative strength of the features in the spectra are determined by symmetry of the p-states, as well as the nearest, and next nearest neighbors in a crystalline environment. This is demonstrated by the differences if the Ti L<sub>2</sub> features in the Zr titanate materials. The  $t_{2g}$  states are reflect the symmetry of  $\pi$ bonded states, where the  $e_{\rm g}$  states reflect the symmetry of  $\sigma\text{-}$ bonded states are more sensitive to second neighbor effects.



Fig. 9. Photoconductive response of  $HfO_2$  indicating the onset of strong photoconductivity at ~5.85 eV, and a lower energy threshold for substantially weaker photoconductivity at ~5.4 eV.

The term splittings for the conduction band states of complex oxides are better described by "molecular orbital states" the mix the d-states of different TM and different TM and RE atoms through  $\sigma$ - and  $\pi$ -bonding overlap with their immediate O-atoms. The bonding states contribute to the uppermost valence band, and the anti-bonding states to the lowest conduction band. The states at the top of the valence band are  $\pi$ -bonded, and those deeper in the valence band are  $\sigma$ -bonded. The ordering of d-state features in the conduction band is a reflection of the valence band states, with the  $\pi$ -bonded states at the band edge, and the  $\sigma$ -states at higher energies.

### 3.2. Jahn-Teller term split states at conduction band edges

Additional evidence for Jahn-Teller term splittings of other wise degenerate anti-bonding d\*-states comes from studies of photoconductivity reported in Ref. [6]. Fig. 9 indicates a blow-up and replot of a previously published photoconductivity (PC) edge for  $HfO_2$  [5]. The spectrum shows two photoconductivity edges, at ~5.85 eV, and 5.4 eV, each with an experimental uncertainty of about  $\pm 0.1$  eV. These two edges are attributed to a Jahn-Teller term splitting of a doubly degenerate  $E_{\rm g}$  state at conduction band edge of HfO<sub>2</sub>. An analysis of the PC edge of ZrO<sub>2</sub> using the data of Ref. [9], also indicates at band edge state separated from the strongest edge feature by about 0.5 eV. Similar band edge "tail" states have been reported from LaAlO<sub>3</sub>, and the thin film complex oxides, LaScO<sub>3</sub>, DyScO<sub>3</sub> and GdScO<sub>3</sub> [11]. The PC edge for Al<sub>2</sub>O<sub>3</sub> shows a significantly smaller "bandtail". It extends approximately 0.15 eV below the primary photoconductivity edge, and the values of photoconductivity are significantly lower than those shown for HfO<sub>2</sub> in Fig. 9.

Studies of electron injection into, and transport through Si-SiO<sub>2</sub>-HfO<sub>2</sub> gate stacks with mid-gap TiN electrodes exhibit asymmetries for gate and substrate injection [6]. This study has proposed that localized band edge traps are present approximately 0.5 to 0.8 eV below the conduction edge of the HfO<sub>2</sub> component of the stacked SiO<sub>2</sub>-HfO<sub>2</sub> dielectric. Due to the factor of approximately four difference in the dielectric constants of  $HfO_2$  (~20), and  $SiO_2$  (~3.9), the potential distributions for substrate injection (negative bias to the Si) and gate injection (positive bias to the Si) are qualitatively different. The localized band edge trapping states have been shown to be active only for substrate electron injection. At low bias levels they give rise to current increases with respect to direct tunneling through a trap-assisted tunneling mechanism, and higher bias levels electrons are injected directly into these states, and the transport mechanism is via a Frenkel-Poole hopping process [12]. For gate injection, the field profile is qualitatively different and direct injection into these states is not possible. Trap assisted tunneling has been proposed at low bias levels, and Frenkel tunneling at higher bias levels.

These band edge states also provide an explanation for a bias temperature instability (BTI) in Si devices with HfO<sub>2</sub> dielectrics, and a thin interfacial layer of nitrided SiO<sub>2</sub> [13]. At low bias levels, positive shifts of the transistor threshold voltage are consistent with very high trapping densities of electrons, in excess of  $10^{12}$  cm<sup>-2</sup>; however as the negative substrate bias is increases, the threshold voltage shifts back to less positive values. An explanation that this due to hole trapping at the Si–SiO<sub>2</sub> interface is ruled out by the significant difference between the negative trapped charge,  $>10^{12}$  cm<sup>-2</sup>, and the significantly smaller changes in interface traps (D<sub>it</sub>),  $\sim 10^{10}$  cm<sup>-2</sup>, at which the threshold voltage shifts are reversed. In this article it is proposed that the mechanism for trapping and trap release is essentially the same as that reported in Ref. [6], and discussed above.

## 4. Summary

Spectroscopic studies by XAS of empty and molecular orbital conduction band d-states in TM/RE complex oxides, supported by similar studies on TM oxides and TM/RE silicate and aluminate alloys have unambiguously identified d-state multiplicities associated with degeneracy removal due to local bonding distortions. These distortions have been addressed in terms of the Jahn–Teller, or J–T mechanism. This explanation is consistent with the low occupancy of d-electrons in the group IIIB and IVB TM, and trivalent lanthanide RE oxides, silicates and complex oxides of this study; similar considerations also apply to the group V TM oxide dielectrics as well, e.g.,  $Ta_2O_5$ , where Frenkel–Poole conduction has been observed for substrate injection of electrons as well [14].

The J-T term-splittings are much easier to understand for this low occupancy d-state TM/RE oxide dielectric constituent atoms than in the high occupancy d-state TM oxides which display more complicated splittings due to magnetic effects associated with d-state occupancies greater than five.

In addition to be observable in XAS spectra, the band edge d-states in HfO<sub>2</sub>, and other dielectrics account for (i) large band edge "tail-states" observed in photoconductivity [5], (ii) trap assisted tunneling and Frenkel–Poole transport in Si–SiO<sub>2</sub>–HfO<sub>2</sub> gate stacks with mid-band TiN gate electrodes [6], and (iii) bias temperature instabilities in aggressively scaled advanced Si devices with HfO<sub>2</sub> dielectrics [13].

Additional research is clearly in order to investigate the J–T states, and in particular to determine their effects on the proposed integration of TM/RE gate dielectrics in advanced bulk complementary MOS (CMOS) devices.

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