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(LaAlO3)0.3(Sr2TaAlO6)0.7(001)

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Richard T. Haasch, Eric Brekenfeld, and Lane W. Martin

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Single Crystal Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 4. (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇(001)

Richard T. Haaschᵃ

Department of Materials Science and Engineering and Materials Research Laboratory, University of Illinois, Urbana-Champaign

Eric Breckenfeld

Department of Materials Science and Engineering and Materials Research Laboratory, University of Illinois, Urbana-Champaign; and Naval Research Laboratory, Washington, DC

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Department of Materials Science and Engineering and Materials Research Laboratory, University of California, Berkeley; and Materials Science Division, Lawrence Berkeley National Laboratory

(Received 3 October 2014; accepted 4 December 2014; published 29 December 2014)

X-ray photoelectron spectroscopy (XPS) was used to analyze a commercially available (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇ (001) bulk single crystal. XP spectra were obtained using incident monochromatic Al Kα radiation at 0.83401 nm. A survey spectrum together with La 3d, O 1s, C 1s, Sr 3p, Ta 4d, La 4p, O 1s, Al 2s, La 4d, Al 2p, Ta 5p, La 5s, Ta 4f, O 2s, Sr 4f and La 5p core level spectra and the valence band are presented. The spectra indicate the principle core level photoelectron and Auger electron signals and show only minor carbon contamination. Making use of the O 1s, Sr 3d, La 4d, Al 2p Ta 4f lines and neglecting the components related to surface contaminants, XPS quantitative analysis reveals an altered stoichiometry of the air-exposed crystal surface of La₀.₃Sr₀.₈₈Al₀.₈₂Ta₀.₄₃O₃. © 2014 American Vacuum Society.

[http://dx.doi.org/10.1116/11.20140904]

Keywords: lanthanum strontium aluminum tantalum oxide; perovskite

INTRODUCTION

Transition metal oxides present an impressive variety of functionality which is not available in more traditional systems such as group IV and III-V semiconductors or elemental metals. Among the many possible functionalities are, for instance, ferroelectricity (Ref. 1) and magnetism (Ref. 2), colossal magnetoresistance (Ref. 3), and high temperature superconductivity (Ref. 4), with transport character ranging from insulating to semiconducting to metallic. Furthermore, these properties are extremely sensitive to perturbations from chemistry, structural defects, strain and many other effects and this, in turn, provides the materials engineer a number of routes by which to engineer new functionalities in this class of materials (Ref. 5). While even simple oxide systems, such as binary oxides, exhibit a broad diversity of properties, it is the complex oxide systems which have received the most attention in recent years. In particular, materials possessing the perovskite and substituted perovskite structure (with chemical formula ABO₃) have been observed to exhibit an incredible variety of functionality and phenomena. Advances in thin film epitaxy, particularly pulsed laser deposition, RF magnetron sputtering, and molecular beam epitaxy, have enabled researchers to carefully tune material properties using epitaxial strain. Such approaches have provided an opportunity to apply large biaxial strains (as much as several percent in some cases) to nanoscale films of various materials which would lead to cracks in bulk materials under similar values of hydrostatic strain (Ref. 6).

ᵃAuthor to whom correspondence should be addressed.

SPECIMEN DESCRIPTION (ACCESSION #01313)

Host Material: Single crystal (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇

CAS Registry #: 272780-20-8

Host Material Characteristics: homogeneous; solid; single crystal; dielectric; inorganic compound

Chemical Name: Lanthanum strontium aluminum tantalum oxide

Source: Crystec, GmbH. Grown by the Czochralski method.

Host Composition: (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇

Form: single crystal

Structure: cubic Pn₃(bar)m mixed perovskite, a = 0.₇₇₃₀ nm (Ref. 7)

History & Significance: (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇ has been widely used as a designed substrate for a number of important applications. It originally rose to prominence as a competitive substrate for high TC oxide superconductors due to its low dielectric response, exceptional crystallinity, cubic symmetry, and small lattice constant (Ref. 8). Since then, it has been used as an epitaxial substrate for ferroelectric materials (Ref. 9), high-quality optoelectronic semiconductors (Ref. 10), and colossal magnetoresistive materials (Ref. 11). In order to gain an increased understanding of the surfaces and hetero-interfaces of perovskite-based materials, (LaAlO₃)₀.₃(Sr₂TaAlO₆)₀.₇ bulk single crystal was analyzed using X-ray photoelectron spectroscopy.

As Received Condition: as grown
Recommended Energy Scale Shift: +1.956 eV for high-resolution spectra

**In Situ Preparation/Mounting:** Samples were cleaned ultrasonically for 5 min each in Formula 409®, methyl alcohol, and deionized water. Samples were mounted onto the sample holder using double-sided carbon tape (Pella product number 16074).

**Preparation/Mounting:** Samples were cleaned ultrasonically for 5 min each in Formula 409®, methyl alcohol, and deionized water. Samples were mounted onto the sample holder using double-sided carbon tape (Pella product number 16074). The binding energy scale was referenced to C 1s = 285.0 eV.

**Pre-Analysis Beam Exposure:** less than 2 min; no x-ray degradation effects observed

**Temperature During Analysis:** 300 K

**Pressure During Analysis:** <3 × 10⁻⁷ Pa

---

**REFERENCES**

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**Note:**
- a Result of exposure to air
- b O 2p and Al 3s (Refs. 14–16)
- c O 2p, Al 3p, and Ta 5d (Refs. 14–16)
- d The position of VBM was estimated by subtracting 1/2 of the full width at half maximum (FWHM) from the position of the maximum intensity at the VBM.

---

114 Surface Science Spectra, Vol. 21, 2014 Single Crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$ by XPS
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<th>Spectrum ID #</th>
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<th>Peak Energy (eV)</th>
<th>Peak Width FWHM (eV)</th>
<th>Peak Area (eV x cts/s)</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
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*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.
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<td>Incident Angle</td>
<td>54°</td>
</tr>
<tr>
<td>Emission Angle</td>
<td>0°</td>
</tr>
<tr>
<td>Analyzer Pass Energy:</td>
<td>160 eV</td>
</tr>
<tr>
<td>Analyzer Resolution</td>
<td>2.4 eV</td>
</tr>
<tr>
<td>Total Signal Accumulation Time</td>
<td>560 s</td>
</tr>
<tr>
<td>Total Elapsed Time</td>
<td>1120 s</td>
</tr>
<tr>
<td>Number of Scans</td>
<td>4</td>
</tr>
<tr>
<td>Effective Detector Width</td>
<td>33.6 eV</td>
</tr>
</tbody>
</table>
**Host Material:** Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$

**Technique:** XPS

**Spectral Region:** La 3d

**Instrument:** Kratos Axis Ultra

**Excitation Source:** Al K$_\alpha$ monochromatic

**Source Energy:** 1486.6 eV

**Source Strength:** 180 W

**Source Size:** 2 mm $\times$ 2 mm

**Analyzer Type:** spherical sector

**Incident Angle:** 54°

**Emission Angle:** 0°

**Analyzer Pass Energy:** 20 eV

**Analyzer Resolution:** 0.3 eV

**Total Signal Accumulation Time:** 3606 s

**Total Elapsed Time:** 9916.5 s

**Number of Scans:** 20

**Effective Detector Width:** 4.2 eV

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**Host Material:** Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$

**Technique:** XPS

**Spectral Region:** O 1s

**Instrument:** Kratos Axis Ultra

**Excitation Source:** Al K$_\alpha$ monochromatic

**Source Energy:** 1486.6 eV

**Source Strength:** 180 W

**Source Size:** 2 mm $\times$ 2 mm

**Analyzer Type:** spherical sector

**Incident Angle:** 54°

**Emission Angle:** 0°

**Analyzer Pass Energy:** 20 eV

**Analyzer Resolution:** 0.3 eV

**Total Signal Accumulation Time:** 1333 s

**Total Elapsed Time:** 3665.75 s

**Number of Scans:** 20

**Effective Detector Width:** 4.2 eV
**Accession #: 01313–04**

- **Host Material:** Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
- **Technique:** XPS
- **Spectral Region:** C 1s; Sr 3p

Instrument: Kratos Axis Ultra

Excitation Source: Al $K_α$

Source Energy: 1486.6 eV

Source Strength: 180 W

Source Size: 2 mm × 2 mm

Analyzer Type: spherical sector

Incident Angle: 54°

Emission Angle: 0°

Analyzer Pass Energy: 20 eV

Analyzer Resolution: 0.3 eV

Total Signal Accumulation Time: 3186 s

Total Elapsed Time: 8761.5 s

Number of Scans: 20

Effective Detector Width: 4.2 eV

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**Accession #: 01313–05**

- **Host Material:** Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Ta 4d

Instrument: Kratos Axis Ultra

Excitation Source: Al $K_α$

Source Energy: 1486.6 eV

Source Strength: 180 W

Source Size: 2 mm × 2 mm

Analyzer Type: spherical sector

Incident Angle: 54°

Emission Angle: 0°

Analyzer Pass Energy: 20 eV

Analyzer Resolution: 0.3 eV

Total Signal Accumulation Time: 2346 s

Total Elapsed Time: 6451.5 s

Number of Scans: 20

Effective Detector Width: 4.2 eV
Accession #: 01313–06
Host Material: Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
Technique: XPS
Spectral Region: La 4p
Instrument: Kratos Axis Ultra
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 180 W
Source Size: 2 mm × 2 mm
Analyzer Type: spherical sector
Incident Angle: 54°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.3 eV
Total Signal Accumulation Time: 2526 s
Total Elapsed Time: 6946.5 s
Number of Scans: 20
Effective Detector Width: 4.2 eV

Accession #: 01313–07
Host Material: Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
Technique: XPS
Spectral Region: Sr 3d
Instrument: Kratos Axis Ultra
Excitation Source: Al K$_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 180 W
Source Size: 2 mm × 2 mm
Analyzer Type: spherical sector
Incident Angle: 54°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.3 eV
Total Signal Accumulation Time: 1206 s
Total Elapsed Time: 3316.5 s
Number of Scans: 20
Effective Detector Width: 4.2 eV
**Accession #:** 01313–08  
**Host Material:** Single crystal \((LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}\)  
**Technique:** XPS  
**Spectral Region:** Al 2s

Instrument: Kratos Axis Ultra  
Excitation Source: Al Kα monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 180 W  
Source Size: 2 mm × 2 mm  
Analyzer Type: spherical sector  
Incident Angle: 54°  
Emission Angle: 0°  
Analyzer Pass Energy: 20 eV  
Analyzer Resolution: 0.3 eV  
Total Signal Accumulation Time: 1608 s  
Total Elapsed Time: 4422 s  
Number of Scans: 20  
Effective Detector Width: 4.2 eV

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**Accession #:** 01313–09  
**Host Material:** Single crystal \((LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}\)  
**Technique:** XPS  
**Spectral Region:** La 4d

Instrument: Kratos Axis Ultra  
Excitation Source: Al Kα monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 180 W  
Source Size: 2 mm × 2 mm  
Analyzer Type: spherical sector  
Incident Angle: 54°  
Emission Angle: 0°  
Analyzer Pass Energy: 20 eV  
Analyzer Resolution: 0.3 eV  
Total Signal Accumulation Time: 1446 s  
Total Elapsed Time: 4422 s  
Number of Scans: 20  
Effective Detector Width: 4.2 eV
Accession #: 01313–10
Host Material: Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
Technique: XPS
Spectral Region: Al 2p

Instrument: Kratos Axis Ultra
Excitation Source: Al K$_\alpha$, monochromatic
Source Energy: 1486.6 eV
Source Strength: 180 W
Source Size: 2 mm $\times$ 2 mm
Analyzer Type: spherical sector
Incident Angle: 54°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.3 eV
Total Signal Accumulation Time: 1608 s
Total Elapsed Time: 4422 s
Number of Scans: 20
Effective Detector Width: 4.2 eV

Accession #: 01313–11
Host Material: Single crystal (LaAlO$_3$)$_{0.3}$(Sr$_2$TaAlO$_6$)$_{0.7}$
Technique: XPS
Spectral Region: Ta 5p$_{3/2}$; La 5s; Ta 4f; O 2s; Sr 4p; La 5p; valence band

Instrument: Kratos Axis Ultra
Excitation Source: Al K$_\alpha$, monochromatic
Source Energy: 1486.6 eV
Source Strength: 180 W
Source Size: 2 mm $\times$ 2 mm
Analyzer Type: spherical sector
Incident Angle: 54°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.3 eV
Total Signal Accumulation Time: 4088 s
Total Elapsed Time: 11242 s
Number of Scans: 20
Effective Detector Width: 4.2 eV