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HAXPES of Al₂O₃ with Cr Kα excitation

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A thick alumina sample was analyzed by hard x-ray photoelectron spectroscopy (HAXPES) by means of a lab-scale spectrometer equipped with a Cr Kα (5414.8 eV) excitation source. The reported spectra include a survey scan as well as Al 1s, Al 2s, Al 2p, O 1s and C 1s core-level spectra.

Keywords: Al₂O₃, HAXPES, Cr Kα

INTRODUCTION

Alumina has attracted a lot of attention for its use as gate insulator material in high electron mobility transistor (HEMT) applications. X-ray photoelectron spectroscopy (XPS) is a powerful method to investigate the interface between Al₂O₃ and the underlying semiconductor. However, the low probing depth of XPS prevents from studying realistic devices. Thanks to the increase of the inelastic mean free path of electrons with increasing photon energy, hard x-ray photoelectron spectroscopy (HAXPES) has become a useful tool to probe deeper into materials and, therefore, study more realistic stacks. Interestingly, the recent advent of novel lab-based instruments brings this technique to the expected access level compared to synchrotron facilities.

In this work, we analyzed a thick alumina sample by HAXPES with a monochromatic Cr Kα (5414.8 eV) source. We provide data that can be used for comparison which include a survey scan as well as high-resolution spectra of Al 1s, Al 2s, Al 2p, O 1s and C 1s.

SPECIMEN DESCRIPTION (ACCESSION # 01710)

Host Material: Al₂O₃
CAS Registry #: 1344-28-1
Host Material Characteristics: Homogeneous; solid; polycrystalline; dielectric; inorganic compound; Ceramic
Chemical Name: Aluminum oxide
Source: Unknown
Host Composition: Al₂O₃
Form: Thick sheet
Structure: Trigonal
History & Significance: Air exposed polycrystalline solid
As Received Condition: 20x25mm alumina sheet

Analyzed Region: Same as host material
Ex Situ Preparation/Mounting: The sample was mounted on the sample holder using double sided conductive tape.
In Situ Preparation: The sample was sputter cleaned by low energy Ar⁺ ions (100 eV) for 1h prior to measurements to remove carbon contamination.
Charge Control: Low-energy electrons (1 eV, filament 1.1 A) and low-energy ions (10 eV)
Temp. During Analysis: 300 K
Pressure During Analysis: < 6.10⁻⁷ Pa
Pre-analysis Beam Exposure: 0 s.

INSTRUMENT DESCRIPTION

Manufacturer and Model: ULVAC-PHI Quantes
Analyzer Type: spherical sector
Detector: multichannel resistive plate
Number of Detector Elements: 32

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer
Analyzer Mode: constant pass energy
Throughput (T=E⁰): The energy dependence can be modeled using the following equation: \( \frac{A}{E_p} = \left( \frac{a}{E_p + b} \right)^k \), where a and b are constants, \( E_p \) is the pass energy, A is the peak area and R is the retard ratio equal to \( E/E_p \), where E is the kinetic energy. Three spectral regions are recorded on a sputter cleaned sample at different pass energies. The values of a and b are then determined by a linear least square fit of the data applying the equation described above.
Excitation Source Window: Al
Excitation Source: Cr Kα monochromatic

Accession#: 01710
Technique: XPS
Host Material: Al₂O₃
Instrument: ULVAC-PHI Quantes
Major Elements in Spectra: Al, O
Minor Elements in Spectra: C
Published Spectra: 6
Spectra in Electronic Record: 6
Spectral Category: comparison

aElectronic mail: pierre-marie.deleuze@cea.fr
Source Energy: 5414.8 eV
Source Strength: 50 W
Source Beam Size: 100 μm x 100 μm
Signal Mode: multichannel direct

Geometry
Incident Angle: 22˚
Source-to-Analyzer Angle: 46˚
Emission Angle: 45˚
Specimen Azimuthal Angle: 0˚
Acceptance Angle from Analyzer Axis: 0˚
Analyzer Angular Acceptance Width: 20˚ x 20˚

Ion Gun
Manufacturer and Model: ULVAC-PHI Quantes
Energy: 10 and 100 eV
Current: 1.4 × 10⁻⁵ mA
Current Measurement Method: Faraday cup
Sputtering Species: Ar
Spot Size (unrastered): 100 μm
Raster Size: N/A
Incident Angle: 45˚
Polar Angle: 45˚
Azimuthal Angle: 45˚
Comment: Differentially pumped ion gun used for presputtering of the sample and to prevent reoxidation during analysis.

DATA ANALYSIS METHOD
Energy Scale Correction: The decrease of photoionization cross-sections in HAXPES (Ref. 1 and 2) leads to a very low C 1s intensity. Therefore, the binding energy was referenced to the Al 2p binding energy position measured with Al Kα radiation after shifting the C 1s peak to 284.8 eV. Doing so, the Al 2p binding energy was 75.1 eV. The spectra recorded with the Cr Kα source were then rescaled by shifting the Al 2p to 75.1 eV.
Recommended Energy Scale Shift: 2.0 eV for binding energy
Peak Shape and Background Method: Shirley background was employed for peak area determination. No curve fitting was performed on the spectra.
Quantitation Method: PHI Multipak software Version 9.9.0.8 was used to perform quantification. Empirically determined sensitivity factors (RSFs) were provided by the software. The RSFs were derived from the pure-element relative sensitivity factor as defined in ISO 18118:2015 (Ref 3) which were measured on pure element samples using a Cr Kα source. They therefore account for the decrease of cross-section and different escape depth of photoelectrons using higher energy photons. RSFs are reported proportional to the RSF of F 1s equal to 1. The reported concentrations were calculated using these RSFs corrected to include the transmission function and the asymmetry parameter.

ACKNOWLEDGMENTS
This work was performed at the Platform For NanoCharacterization (PFNC) of CEA-Leti with support from the Recherche Technologique de Base (RTB) program of the french ministry of research. The authors acknowledge the support of the PHI-Leti TANDEMS collaboration program.

DATA AVAILABILITY STATEMENT
The data that supports the findings of this study are available within the article and its supplementary material.

REFERENCES
### SPECTRAL FEATURES TABLE

<table>
<thead>
<tr>
<th>Spectrum ID #</th>
<th>Element/Transition</th>
<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>
<th>Peak Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>01710-02</td>
<td>Al 1s</td>
<td>1562.0</td>
<td>2.13</td>
<td>14593</td>
<td>4.238</td>
<td>35.9</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>01710-03</td>
<td>Al 2s</td>
<td>120.1</td>
<td>2.49</td>
<td>1089</td>
<td>0.369</td>
<td>...</td>
<td>Al₂O₃</td>
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<tr>
<td>01710-04</td>
<td>Al 2p</td>
<td>75.1</td>
<td>1.96</td>
<td>292</td>
<td>0.075</td>
<td>...</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>01710-05</td>
<td>O 1s</td>
<td>531.9</td>
<td>2.13</td>
<td>3048</td>
<td>0.589</td>
<td>60.1</td>
<td>Al₂O₃</td>
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<tr>
<td>01710-06</td>
<td>C 1s</td>
<td>285.0</td>
<td>2.15</td>
<td>68</td>
<td>0.199</td>
<td>4.1</td>
<td>Contamination</td>
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### ANALYZER CALIBRATION TABLE

<table>
<thead>
<tr>
<th>Spectrum ID #</th>
<th>Element/Transition</th>
<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>
<th>Peak Assignment</th>
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<tbody>
<tr>
<td>...</td>
<td>Ag 3d₅/₂</td>
<td>368.12</td>
<td>0.63</td>
<td>114999</td>
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<td>...</td>
<td>...</td>
</tr>
<tr>
<td>...</td>
<td>Cu 2p₃/₂</td>
<td>932.61</td>
<td>0.96</td>
<td>40205</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>...</td>
<td>Au 4f₇/₂</td>
<td>83.89</td>
<td>0.78</td>
<td>100500</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>

The spectra in the analyzer calibration table were recorded using Al Kα photons.

### GUIDE TO FIGURES

<table>
<thead>
<tr>
<th>Spectrum (Accession) #</th>
<th>Spectral Region</th>
<th>Voltage Shift*</th>
<th>Multiplier</th>
<th>Baseline</th>
<th>Comment #</th>
</tr>
</thead>
<tbody>
<tr>
<td>01710-01</td>
<td>Survey</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>...</td>
</tr>
<tr>
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<td>Al 1s</td>
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<td>1</td>
<td>0</td>
<td>...</td>
</tr>
<tr>
<td>01710-03</td>
<td>Al 2s</td>
<td>-2.0</td>
<td>1</td>
<td>0</td>
<td>...</td>
</tr>
<tr>
<td>01710-04</td>
<td>Al 2p</td>
<td>-2.0</td>
<td>1</td>
<td>0</td>
<td>...</td>
</tr>
<tr>
<td>01710-05</td>
<td>O 1s</td>
<td>-2.0</td>
<td>1</td>
<td>0</td>
<td>...</td>
</tr>
<tr>
<td>01710-06</td>
<td>C 1s</td>
<td>-2.0</td>
<td>1</td>
<td>0</td>
<td>...</td>
</tr>
</tbody>
</table>

*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.
**Accession #**

01710-01

**Host Material**

$\text{Al}_2\text{O}_3$

**Technique**

XPS

**Spectral Region**

survey

**Instrument**

ULVAC-PHI Quantes

**Excitation Source**

$\text{Cr K}_\alpha$ monochromatic

**Source Energy**

5414.8 eV

**Source Strength**

50 W

**Source Size**

0.1 mm x 0.1 mm

**Analyzer Type**

spherical sector analyzer

**Incident Angle**

22°

**Emission Angle**

45°

**Analyzer Pass Energy**

280 eV

**Analyzer Resolution**

2.33 eV

**Total Signal Accumulation Time**

5240 s

**Total Elapsed Time**

5760 s

**Number of Scans**

10

**Effective Detector Width**

31 eV
Accession #: 01710-03
Host Material: Al₂O₃
Technique: XPS
Spectral Region: Al 2s
Instrument: ULVAC-PHI Quantes
Excitation Source: Cr Kα monochromatic
Source Energy: 5414.8 eV
Source Strength: 50 W
Source Size: 0.1 mm x 0.1 mm
Analyzer Type: spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 1.17 eV
Total Signal Accumulation Time: 3740 s
Total Elapsed Time: 4120 s
Number of Scans: 15
Effective Detector Width: 12.4 eV
Accession #: 01710-04
Host Material: Al₂O₃
Technique: XPS
Spectral Region: Al 2p
Instrument: ULVAC-PHI Quantes
Excitation Source: Cr Kα monochromatic
Source Energy: 5414.8 eV
Source Strength: 50 W
Source Size: 0.1 mm x 0.1 mm
Analyzer Type: spherical sector
Incident Angle: 22 °
Emission Angle: 45 °
Analyzer Pass Energy 112 eV
Analyzer Resolution: 1.17 eV
Total Signal Accumulation Time: 3710 s
Total Elapsed Time: 4090 s
Number of Scans: 20
Effective Detector Width: 12.4 eV
Accession #: 01710-06
Host Material: Al₂O₃
Technique: XPS
Spectral Region: C 1s
Instrument: ULVAC-PHI Quantes
Excitation Source: Cr Kα monochromatic
Source Energy: 5414.8 eV
Source Strength: 50 W
Source Size: 0.1 mm x 0.1 mm
Analyzer Type: spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 1.17 eV
Total Signal Accumulation Time: 1940 s
Total Elapsed Time: 2140 s
Number of Scans: 15
Effective Detector Width: 12.4 eV