



## HAXPES of Al<sub>2</sub>O<sub>3</sub> with Cr K $\alpha$ excitation

Pierre-Marie Deleuze, Kateryna Artyushkova, Eugénie Martinez, Olivier Renault

### ► To cite this version:

Pierre-Marie Deleuze, Kateryna Artyushkova, Eugénie Martinez, Olivier Renault. HAXPES of Al<sub>2</sub>O<sub>3</sub> with Cr K $\alpha$  excitation. Surface Science Spectra, 2022, 29 (1), pp.014003. 10.1116/6.0001509 . cea-03564243

**HAL Id: cea-03564243**

**<https://cea.hal.science/cea-03564243>**

Submitted on 10 Feb 2022

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

## HAXPES of Al<sub>2</sub>O<sub>3</sub> with Cr K $\alpha$ excitation

Pierre-Marie Deleuze<sup>1, a)</sup>, Kateryna Artyushkova<sup>2</sup>, Eugénie Martinez<sup>1</sup> and Olivier Renault<sup>1</sup>

<sup>1</sup> Univ. Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France

<sup>2</sup>Physical Electronics, 18725 Lake Drive East, Chanhassen, Minnesota 55317

(Received day Month year; accepted day Month year; published day Month year)

A thick alumina sample was analyzed by hard x-ray photoelectron spectroscopy (HAXPES) by means of a lab-scale spectrometer equipped with a Cr K $\alpha$  (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:** Al<sub>2</sub>O<sub>3</sub>, HAXPES, Cr K $\alpha$

### 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 Al<sub>2</sub>O<sub>3</sub> 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 K $\alpha$  (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:** Al<sub>2</sub>O<sub>3</sub>

**CAS Registry #:** 1344-28-1

**Host Material Characteristics:** Homogeneous; solid; polycrystalline; dielectric; inorganic compound; Ceramic

**Chemical Name:** Aluminum oxide

**Source:** Unknown

**Host Composition:** Al<sub>2</sub>O<sub>3</sub>

**Form:** Thick sheet

**Structure:** Trigonal

**History & Significance:** Air exposed polycrystalline solid

**As Received Condition:** 20x25mm alumina sheet

**Accession#:** 01710

**Technique:** XPS

**Host Material:** Al<sub>2</sub>O<sub>3</sub>

**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

**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<sup>+</sup> 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<sup>-7</sup> 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=E<sup>N</sup>):** The energy dependence can be modeled using the following equation:  $\frac{A}{E_p} = \left(\frac{a^2}{a^2 + R^2}\right)^b$ , where a and b are constants, E<sub>p</sub> is the pass energy, A is the peak area and R is the retard ratio equal to E/E<sub>p</sub>, 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 K $\alpha$  monochromatic

<sup>a)</sup>Electronic mail: pierre-marie.deleuze@cea.fr

**Source Energy:** 5414.8 eV

**Source Strength:** 50 W

**Source Beam Size:** 100  $\mu\text{m}$  x 100  $\mu\text{m}$

**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:**  $1.4 \times 10^{-5}$  mA

**Current Measurement Method:** Faraday cup

**Sputtering Species:** Ar

**Spot Size (unrastered):** 100  $\mu\text{m}$

**Raster Size:** N/A

**Incident Angle:** 45 °

**Polar Angle:** 45 °

**Azimuthal Angle:** 45 °

**Comment:** Differentially pumped ion gun used for pre-sputtering of the sample and to prevent reoxidation during analysis.

## DATA ANALYSIS METHOD

**Energy Scale Correction:** The decrease of photoionization cross-sections in HAXPES (Refs. 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 K $\alpha$  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 K $\alpha$  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 K $\alpha$  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 TANDEM collaboration program.*

## DATA AVAILABILITY STATEMENT

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

## REFERENCES

1. M.B. Trzhaskovskaya and V.G. Yarzhemsky, [At. Data Nucl. Data Tables](#) **119**, 99 (2018).
2. M.B. Trzhaskovskaya and V.G. Yarzhemsky, [At. Data Nucl. Data Tables](#) **129-130**, 101280 (2019).
3. International Organization for Standardization 2015, *Surface chemical analysis - Auger electron spectroscopy and X-ray photoelectron spectroscopy - Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials*, ISO 18118:2015.

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.  
PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509

SPECTRAL FEATURES TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01710-02	Al 1s	1562.0	2.13	14593	4.238	35.9	Al <sub>2</sub> O <sub>3</sub>
01710-03	Al 2s	120.1	2.49	1089	0.369	...	Al <sub>2</sub> O <sub>3</sub>
01710-04	Al 2p	75.1	1.96	292	0.075	...	Al <sub>2</sub> O <sub>3</sub>
01710-05	O 1s	531.9	2.13	3048	0.589	60.1	Al <sub>2</sub> O <sub>3</sub>
01710-06	C 1s	285.0	2.15	68	0.199	4.1	Contamination

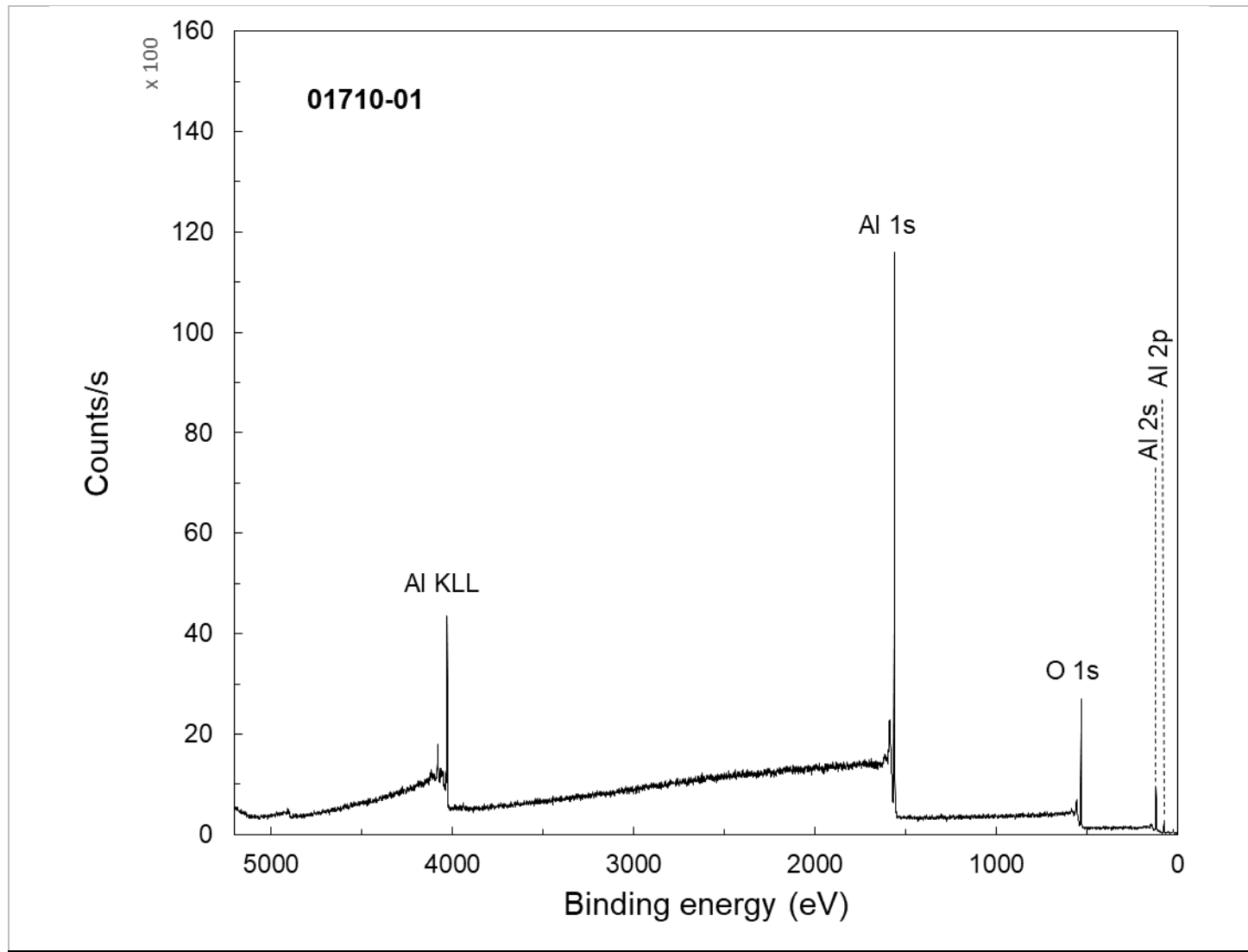
ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Ag 3d <sub>5/2</sub>	368.12	0.63	114999	...	...	...
...	Cu 2p <sub>3/2</sub>	932.61	0.96	40205	...	...	...
...	Au 4f <sub>7/2</sub>	83.89	0.78	100500	...	...	...

The spectra in the analyzer calibration table were recorded using Al K $\alpha$  photons.

GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01710-01	Survey	0	1	0	...
01710-02	Al 1s	-2.0	1	0	...
01710-03	Al 2s	-2.0	1	0	...
01710-04	Al 2p	-2.0	1	0	...
01710-05	O 1s	-2.0	1	0	...
01710-06	C 1s	-2.0	1	0	...

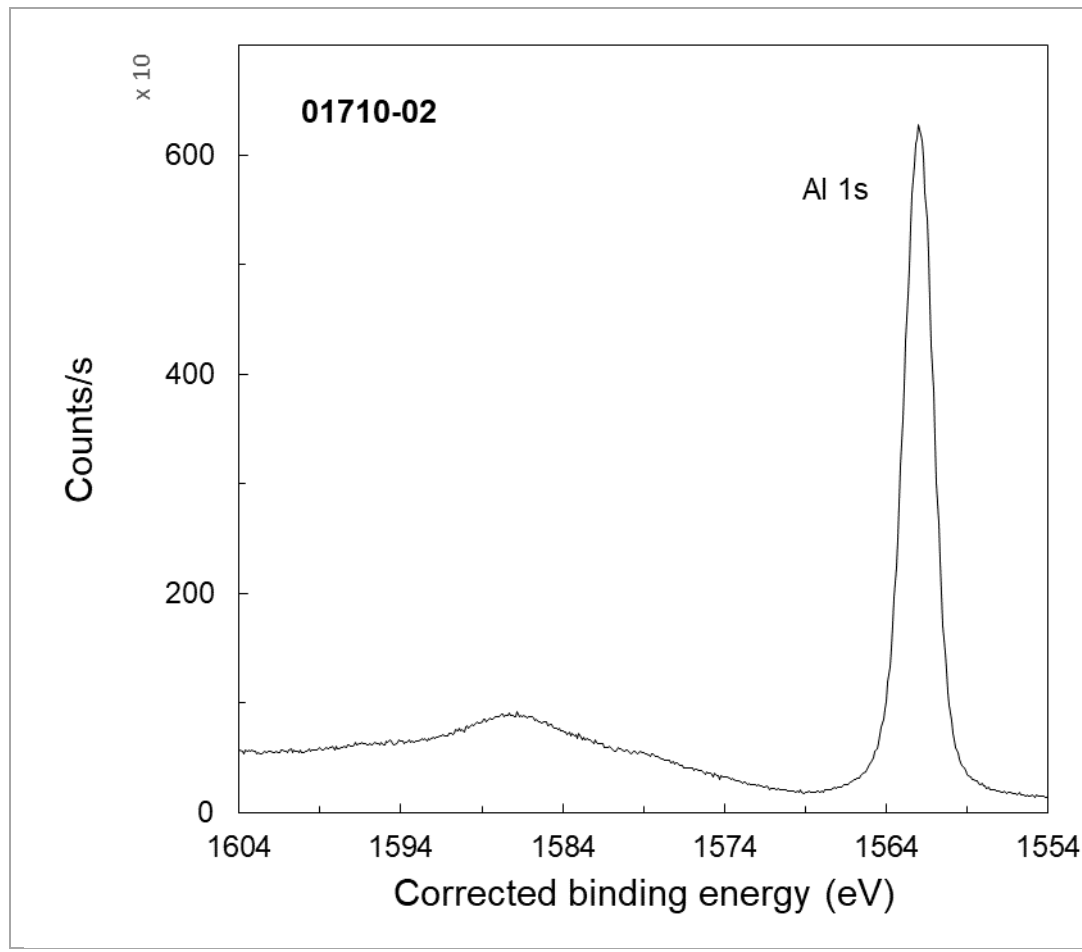
\*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.  
PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



Accession #	01710-01
Host Material	Al <sub>2</sub> O <sub>3</sub>
Technique	XPS
Spectral Region	survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K <sub>α</sub> 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

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.  
PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



■ **Accession #:** 01710-02

■ **Host Material:** Al<sub>2</sub>O<sub>3</sub>

■ **Technique:** XPS

■ **Spectral Region:** Al 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K<sub>α</sub> 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

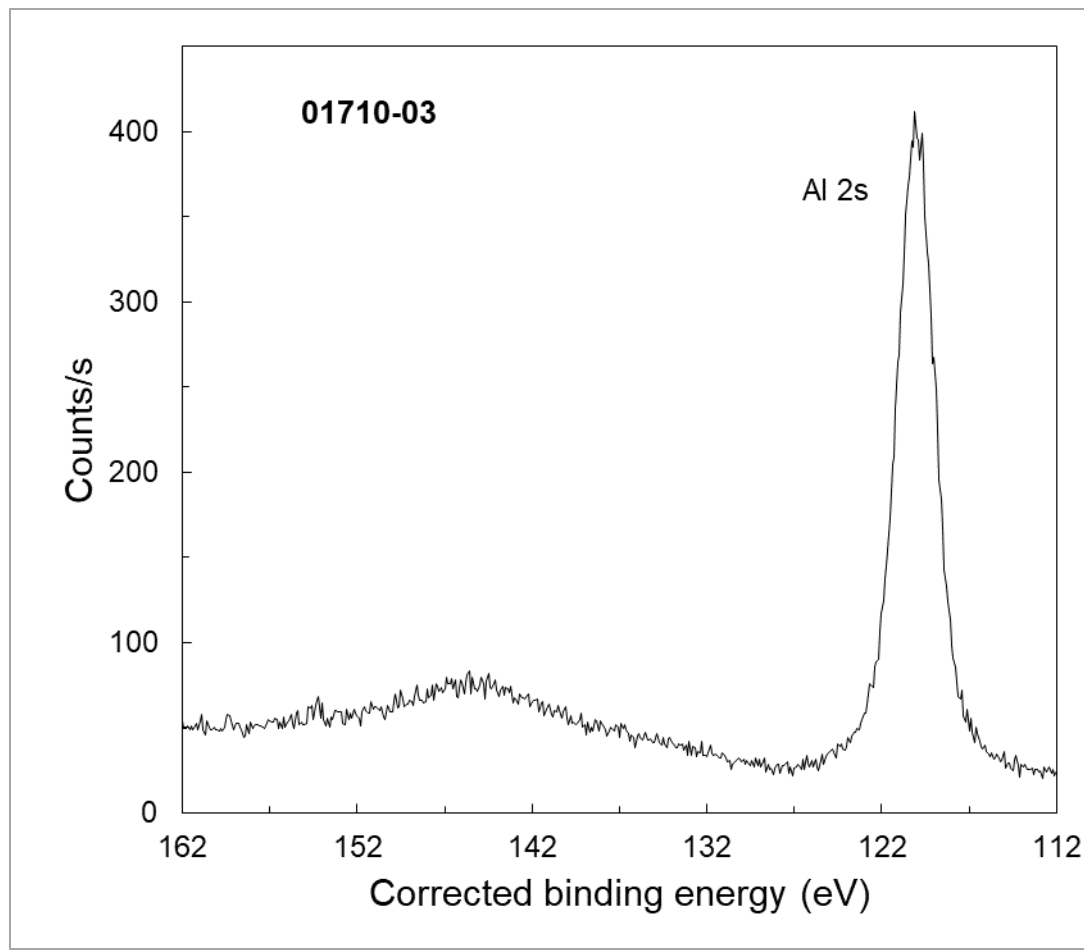
Total Elapsed Time: 4120 s

Number of Scans: 15

Effective Detector Width: 12.4 eV

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



■ **Accession #:** 01710-03

■ **Host Material:** Al<sub>2</sub>O<sub>3</sub>

■ **Technique:** XPS

■ **Spectral Region:** Al 2s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K<sub>α</sub> 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

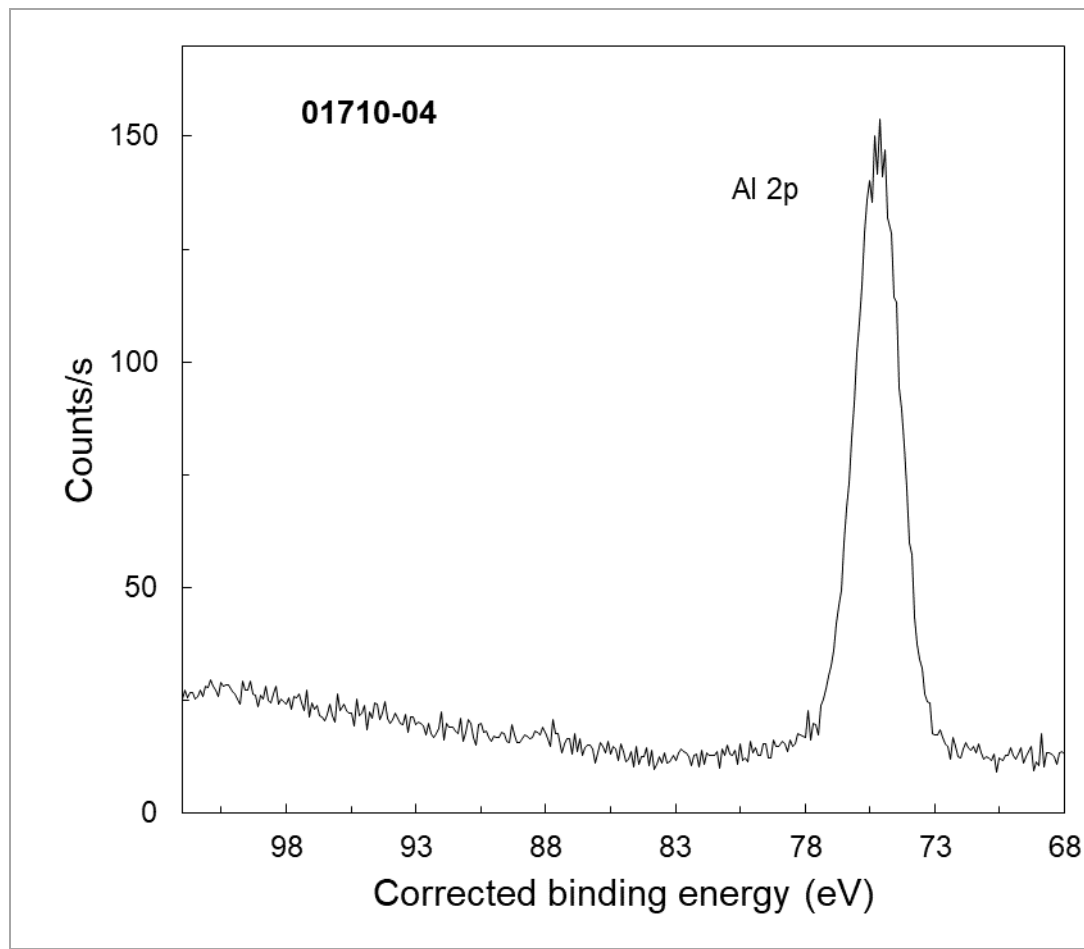
Total Elapsed Time: 4120 s

Number of Scans: 15

Effective Detector Width: 12.4 eV

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



■ **Accession #:** 01710-04

■ **Host Material:** Al<sub>2</sub>O<sub>3</sub>

■ **Technique:** XPS

■ **Spectral Region:** Al 2p

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K<sub>α</sub> 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

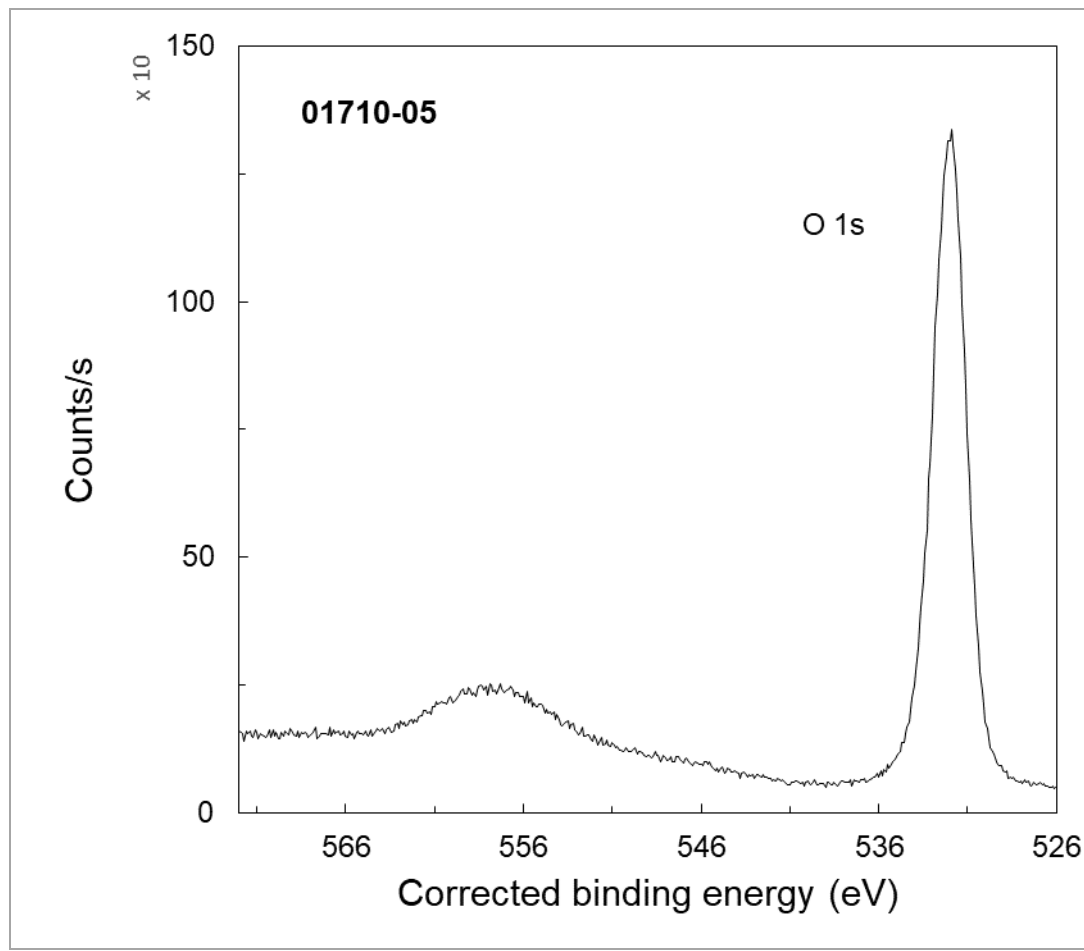
Number of Scans: 20

Effective Detector Width: 12.4 eV



This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



■ **Accession #:** 01710-05

■ **Host Material:**  $\text{Al}_2\text{O}_3$

■ **Technique:** XPS

■ **Spectral Region:** O 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

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

Incident Angle:  $22^\circ$

Emission Angle:  $45^\circ$

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

Time: 3970 s

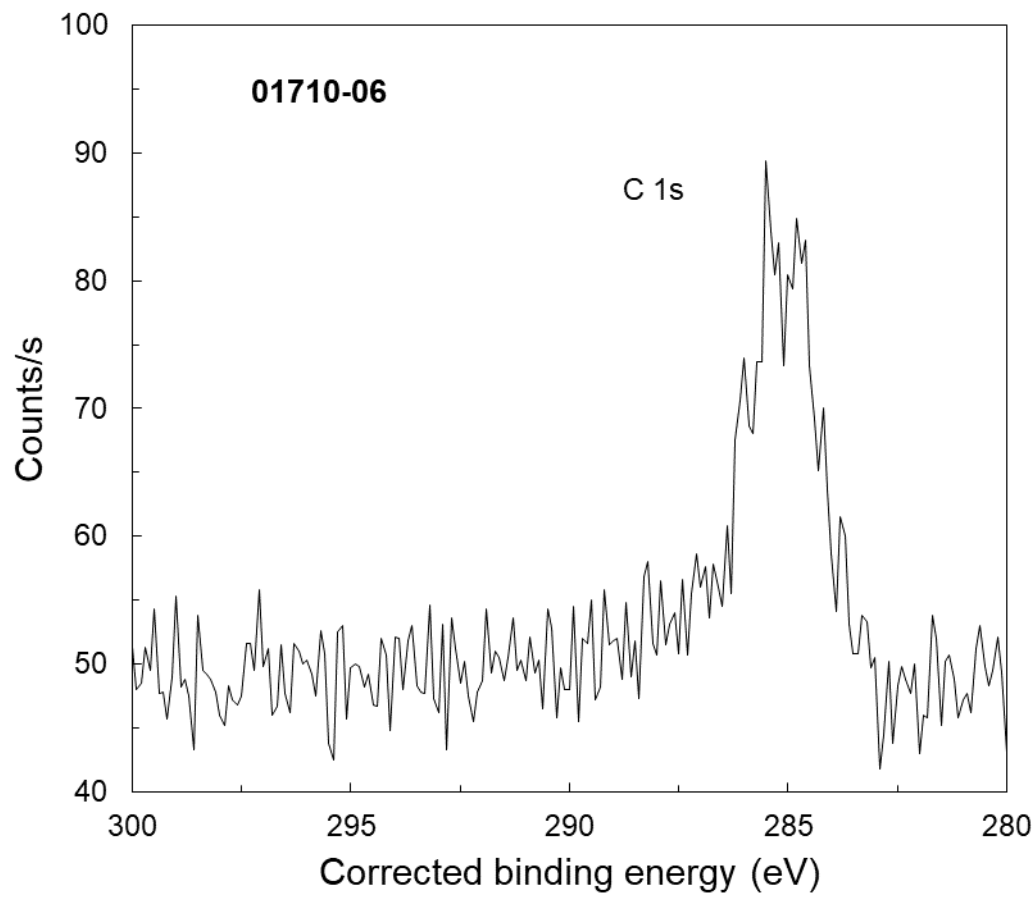
Total Elapsed Time: 4370 s

Number of Scans: 17

Effective Detector Width: 12.4 eV

This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



■ **Accession #:** 01710-06

■ **Host Material:** Al<sub>2</sub>O<sub>3</sub>

■ **Technique:** XPS

■ **Spectral Region:** C 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K<sub>α</sub> 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

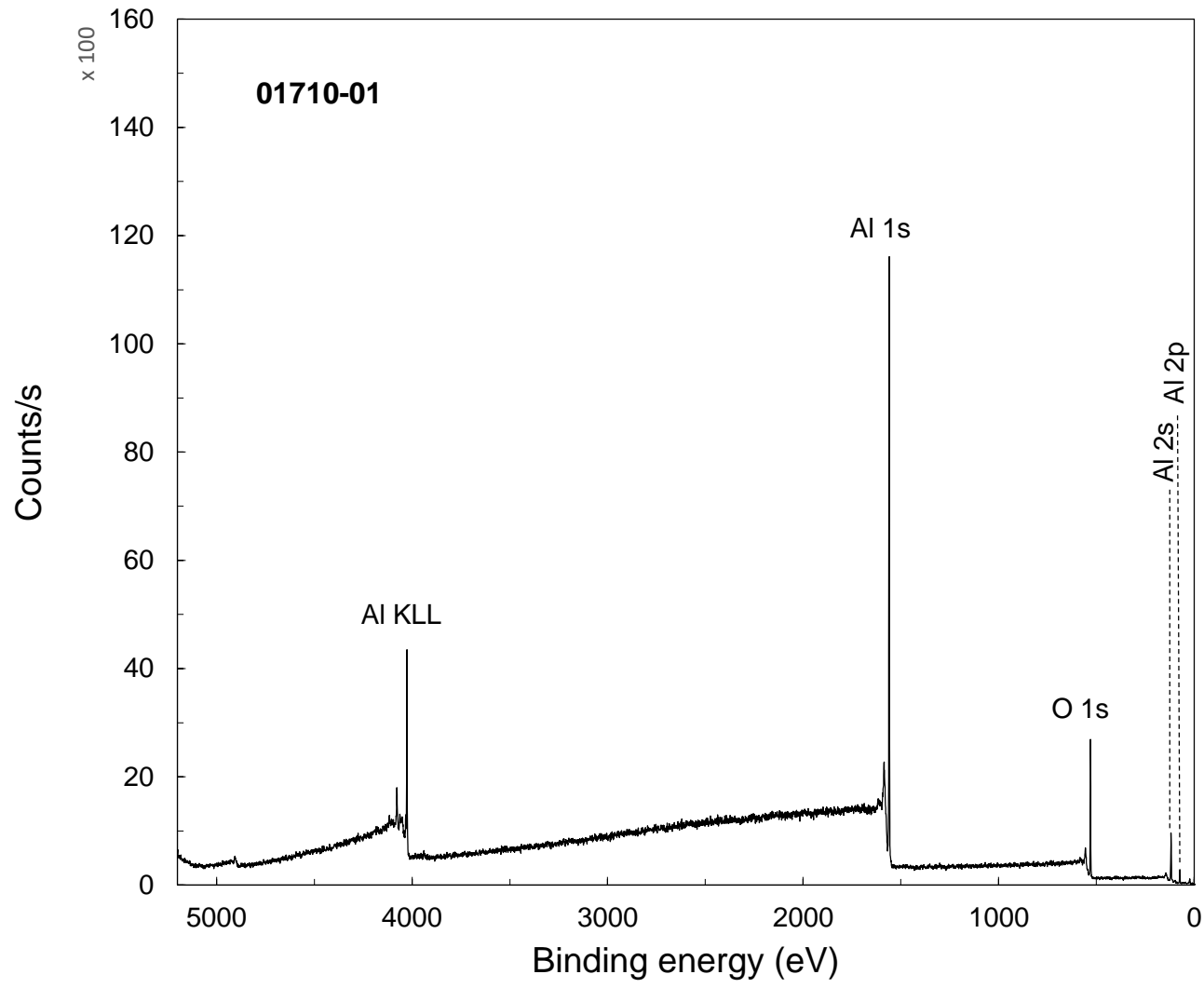
Total Elapsed Time: 2140 s

Number of Scans: 15

Effective Detector Width: 12.4 eV

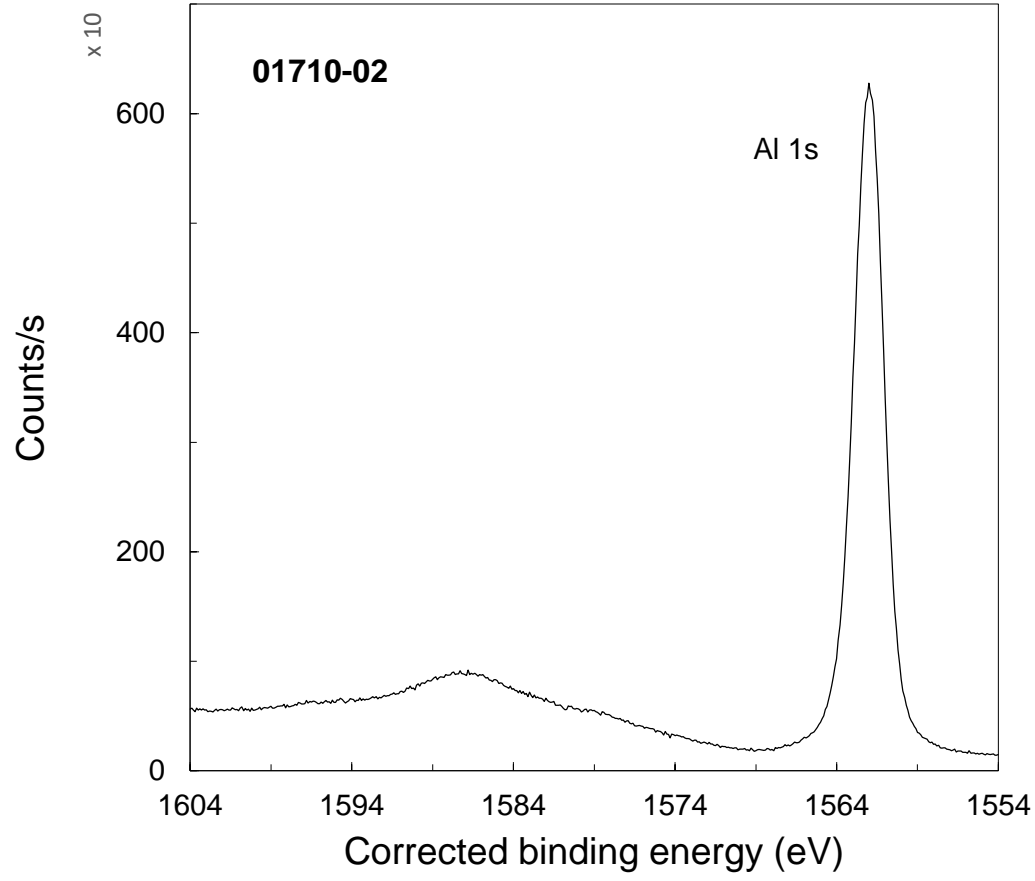
This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



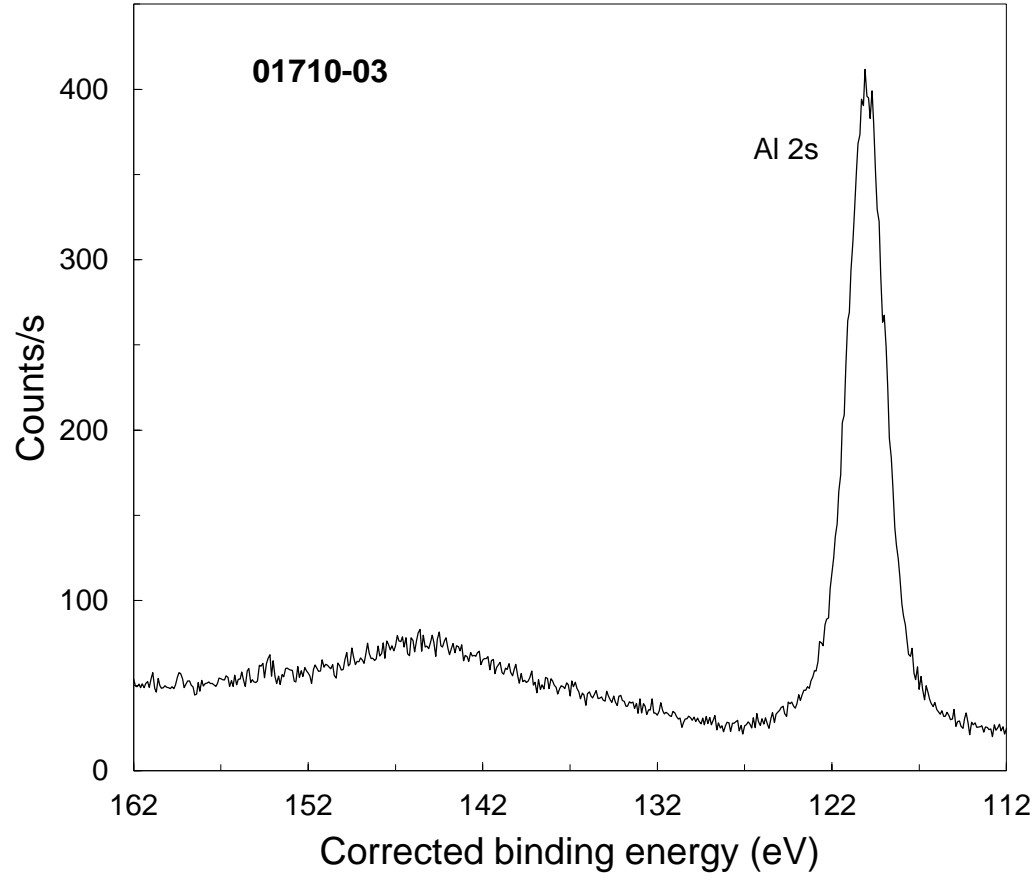
This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



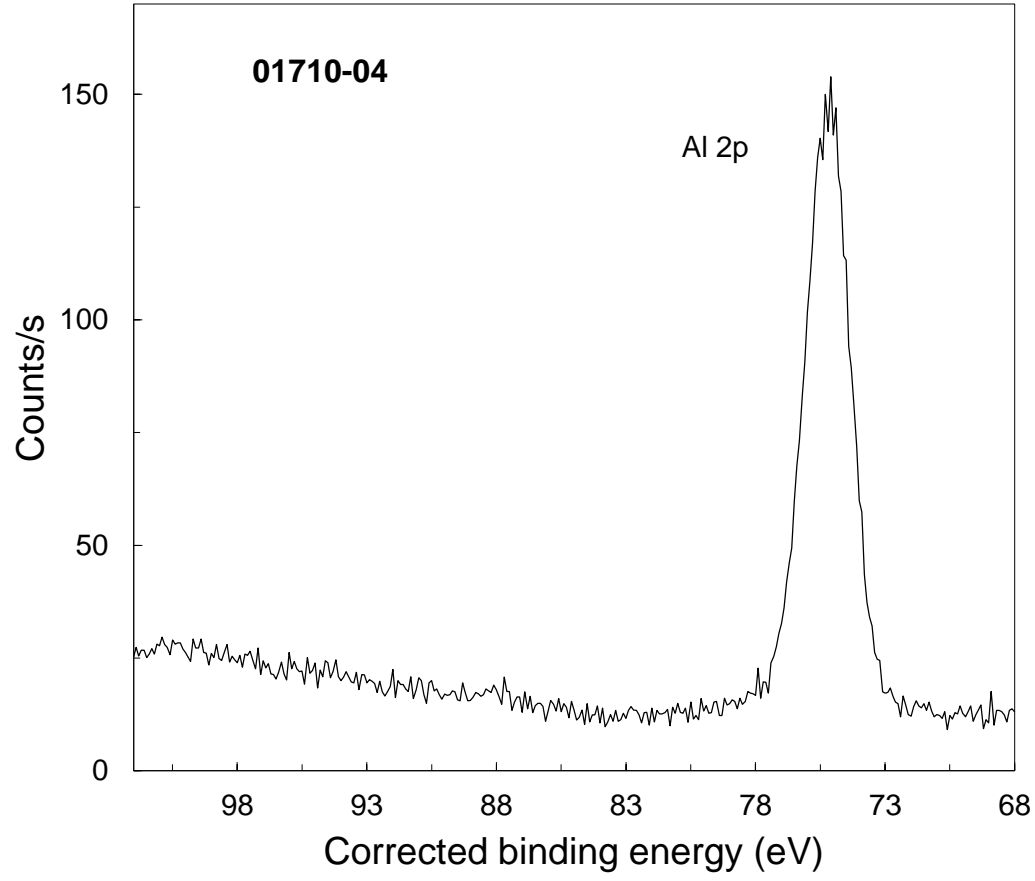
This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



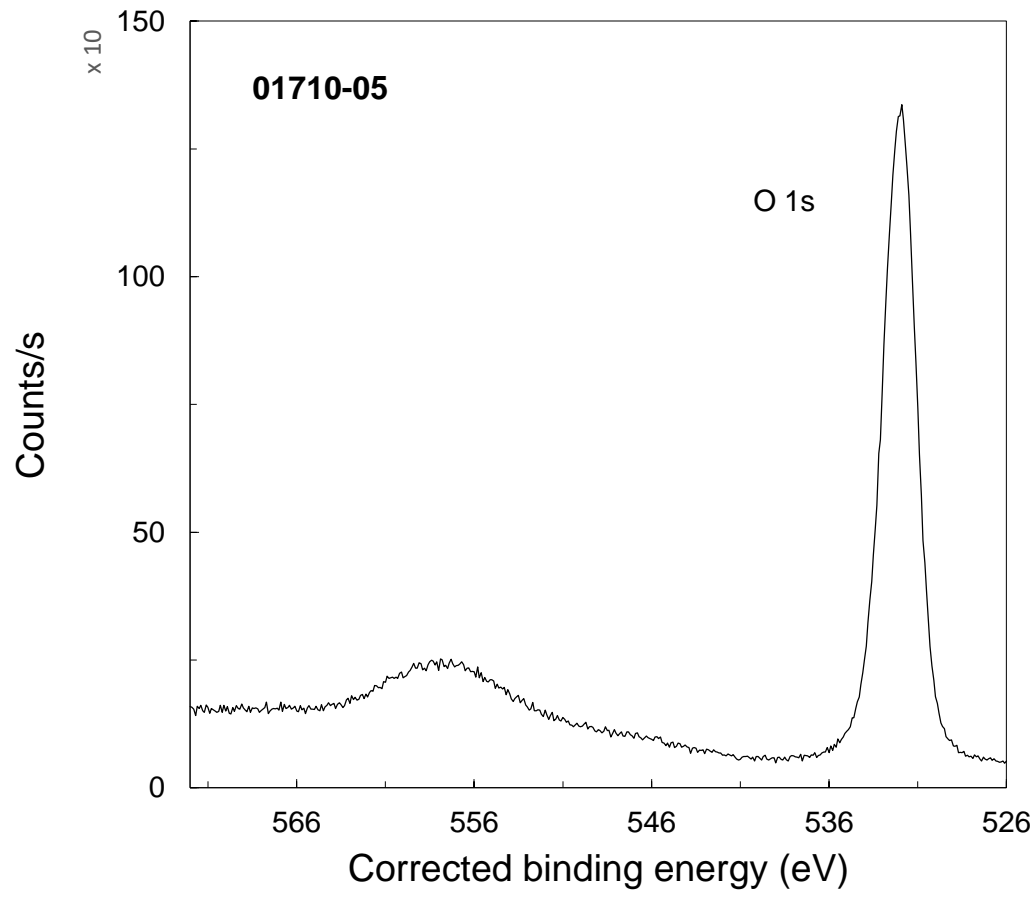
This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509



This is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset.

PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0001509

