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HAXPES of AlN with Cr K α excitation

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Aluminum nitride was analyzed by high-energy photoelectron spectroscopy (HAXPES) using monochromatic Cr K α (5414.8 eV) radiation. The reported data include a survey spectrum and high-resolution spectra of Al 1s, Al 2s, Al 2p, N 1s, O 1s and C 1s core-levels.

Keywords: AlN, HAXPES, Cr K α

INTRODUCTION

Aluminum nitride is a material of interest for packaging applications thanks to its high thermal conductivity and low thermal expansion coefficient. Its wide band gap (6.2 eV) allows it to be used as an insulating film in electronic devices.

In this work, we analyzed a thick AlN sample using a lab-based HAXPES spectrometer equipped with a monochromatic Cr K α (5414.8 eV) source. We provide a survey scan and high-resolution spectra of Al 1s, Al 2s, Al 2p, N 1s, O 1s and C 1s regions which can serve as comparison.

SPECIMEN DESCRIPTION (ACCESSION # 01711)

Host Material: AlN

CAS Registry #: 24304-00-5

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

Chemical Name: Aluminum nitride

Source: Goodfellow

Host Composition: AlN

Form: Polycrystalline solid

Structure: Hexagonal

History & Significance: Air exposed AlN sheet

As Received Condition: 1 mm thick AlN 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 one hour prior to measurements to remove carbon and oxygen contamination.

Charge Control: Low-energy electrons (1 eV, filament 1.1 A) and low-energy Ar⁺ ions (10 eV)

Temp. During Analysis: 300 K

Accession#: 01711

Technique: XPS

Host Material: AlN

Instrument: ULVAC-PHI Quantes

Major Elements in Spectra: Al, N

Minor Elements in Spectra: O, C

Published Spectra: 7

Spectra in Electronic Record: 7

Spectral Category: comparison

Pressure During Analysis: < 3.10⁻⁷ 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=E^N): 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_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 K α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Beam Size: 100 μ m x 100 μ m

Signal Mode: multichannel direct

■ Geometry

Incident Angle: 22 °

Source-to-Analyzer Angle: 46 °

Emission Angle: 45 °

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Specimen Azimuthal Angle: 0 °

Acceptance Angle from Analyzer Axis: 0 °

Analyzer Angular Acceptance Width: 20 ° × 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 μ 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 the XPS analysis.

DATA ANALYSIS METHOD

Energy Scale Correction: The decrease of photoionization cross sections in HAXPES 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 α radiation after shifting the C 1s peak to 284.8 eV. Doing so, the Al 2p binding energy was 73.4 eV. The spectra recorded with the Cr K α source were then rescaled by shifting the Al 2p to 73.4 eV.

Recommended Energy Scale Shift: 1.6 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 which were measured on pure element samples using a Cr K α 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 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

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.

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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
01711-02	Al 1s	1560.4	2.19	14788	4.238	42.6	AlN
01711-03	Al 2s	118.5	2.50	1072	0.369	...	AlN
01711-04	Al 2p	73.4	1.96	295	0.075	...	AlN
01711-05	N 1s	396.7	2.00	997	0.331	41.7	AlN
01711-06	O 1s	531.4	2.94	556	0.589	12.8	Contamination
01711-07	C 1s	284.8	1.33	41	0.199	2.9	Contamination

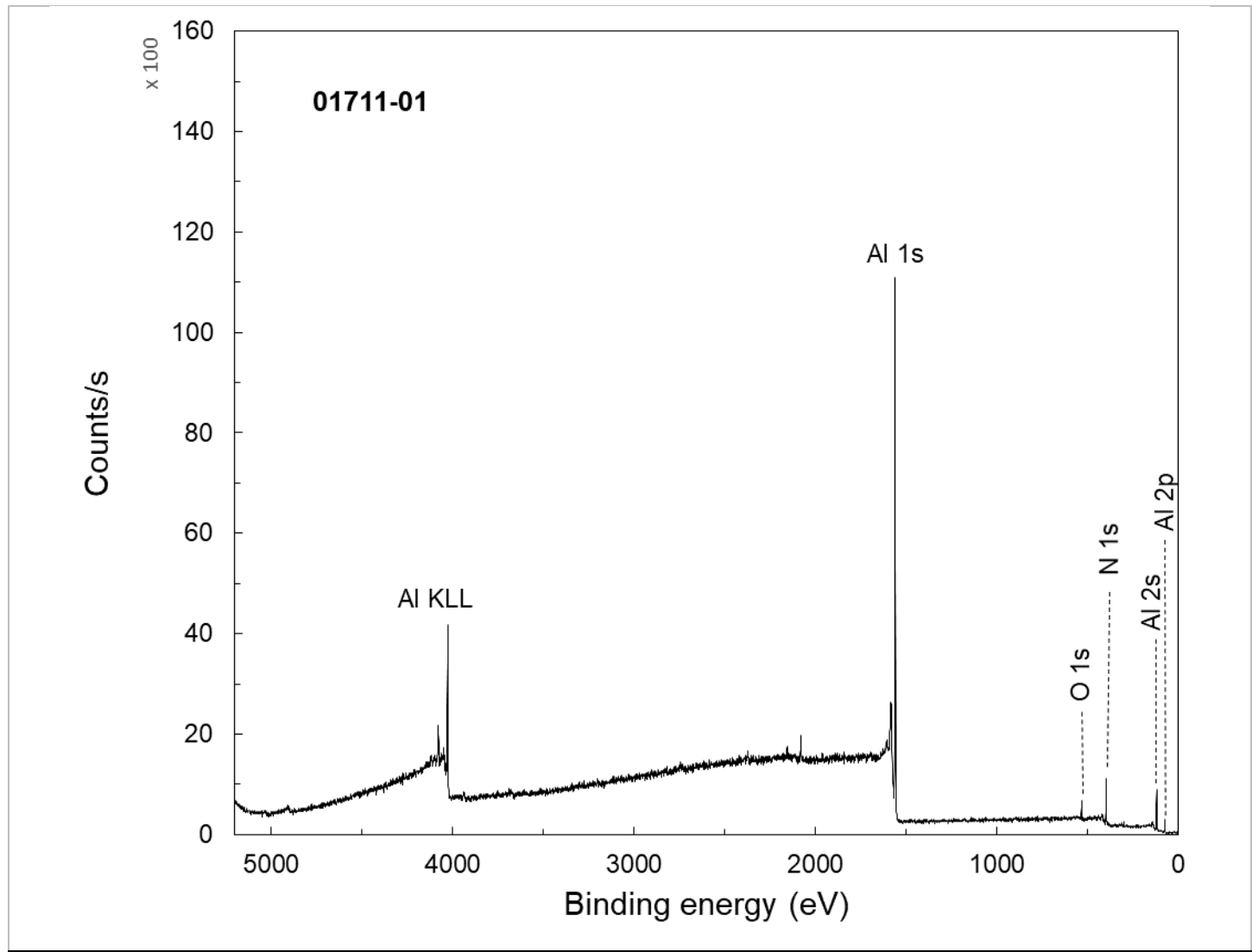
ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Ag3d _{5/2}	368.12	0.63	114999
...	Cu2p _{3/2}	932.61	0.96	40205
...	Au4f _{7/2}	83.89	0.78	100500

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

GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01711-01	Survey	0	1	0	...
01711-02	Al 1s	-1.6	1	0	...
01711-03	Al 2s	-1.6	1	0	...
01711-04	Al 2p	-1.6	1	0	...
01711-05	N 1s	-1.6	1	0	...
01711-06	O 1s	-1.6	1	0	...
01711-07	C 1s	-1.6	1	0	...

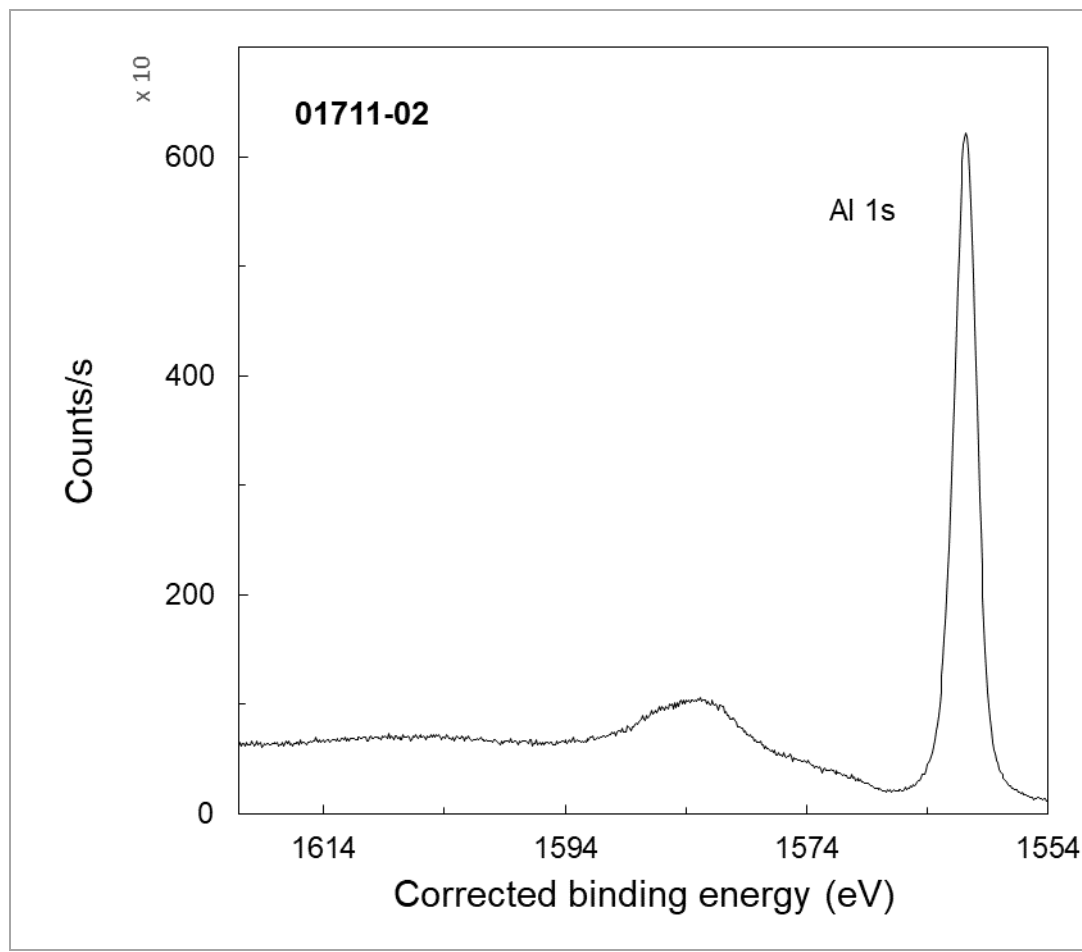
*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

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Accession #	01711-01
Host Material	AlN
Technique	XPS
Spectral Region	survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K α 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

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■ **Accession #:** 01711-02

■ **Host Material:** AlN

■ **Technique:** XPS

■ **Spectral Region:** Al 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K_{α} 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

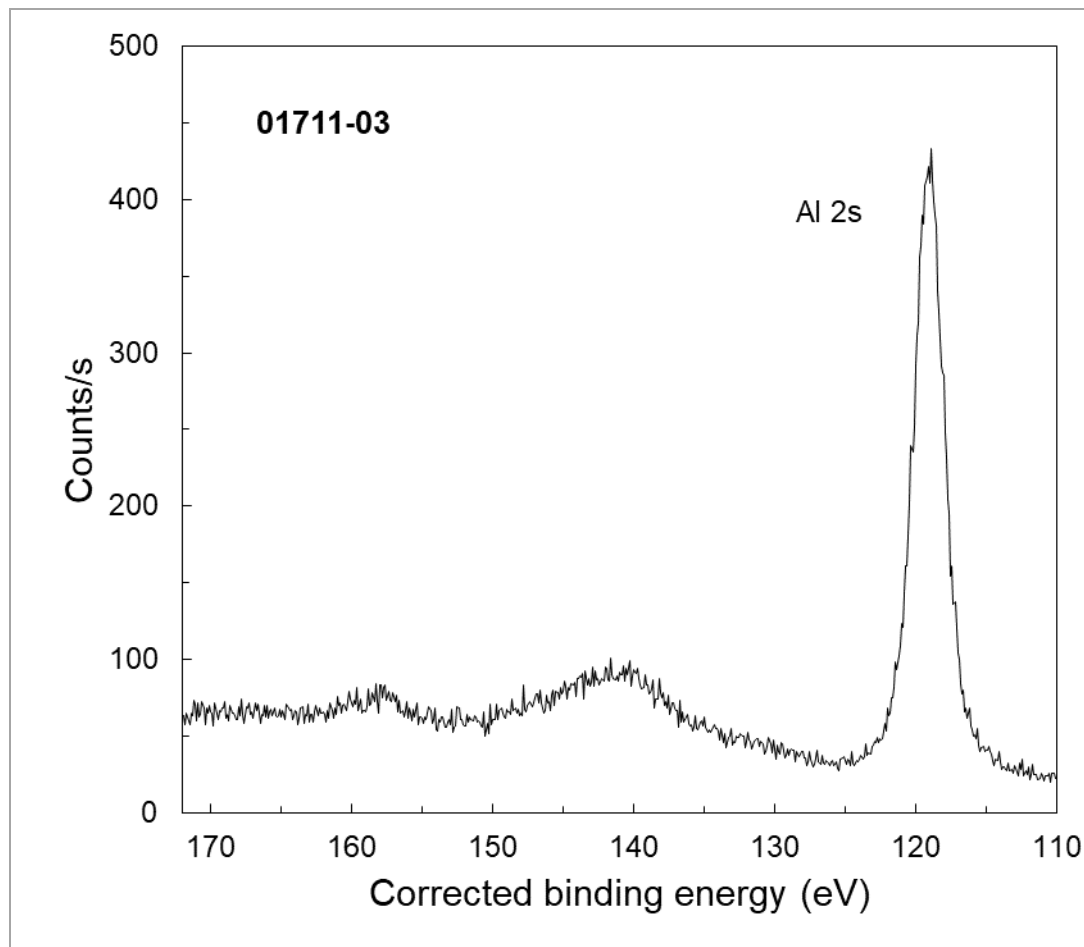
Time: 3810 s

Total Elapsed Time: 4194 s

Number of Scans: 12

Effective Detector Width: 12.4 eV

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■ **Accession #:** 01711-03

■ **Host Material:** AlN

■ **Technique:** XPS

■ **Spectral Region:** Al 2s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K α 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: 3570 s

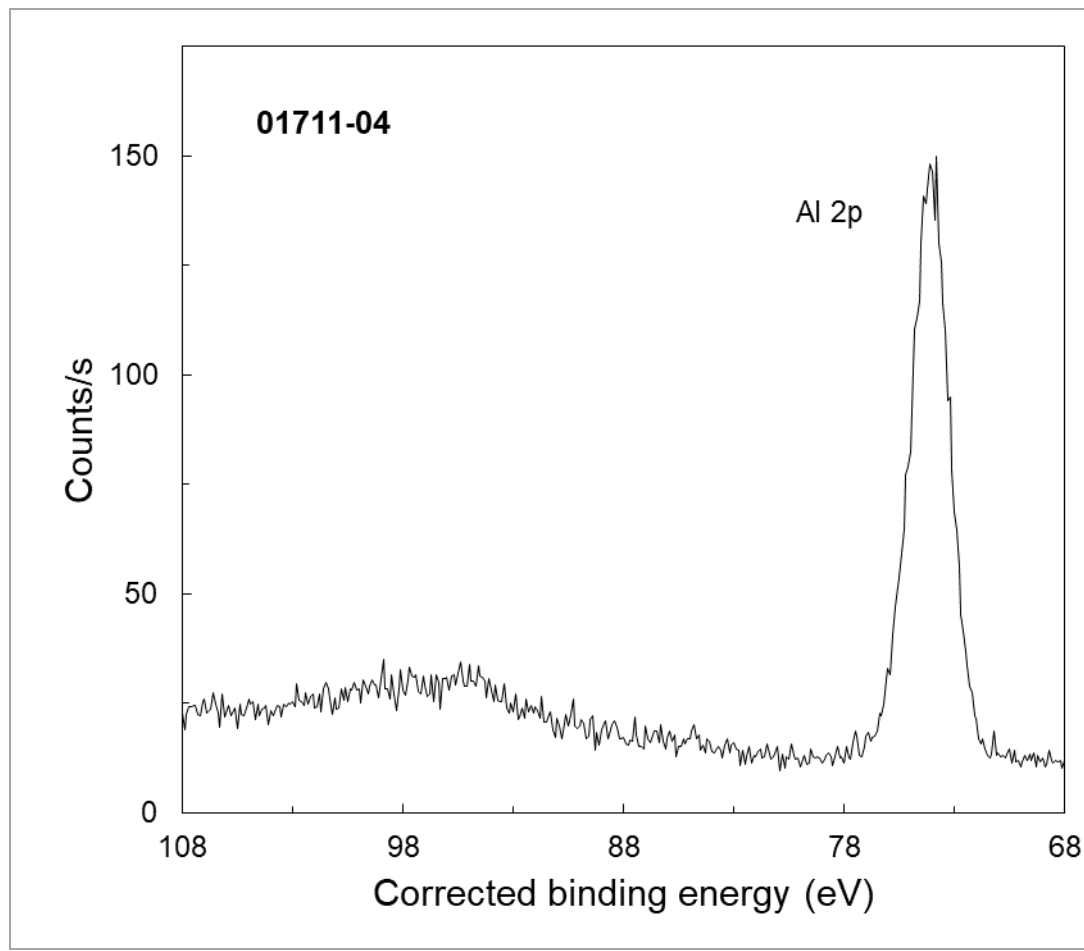
Total Elapsed Time: 3930 s

Number of Scans: 12

Effective Detector Width: 12.4 eV

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■ **Accession #:** 01711-04
 ■ **Host Material:** AlN
 ■ **Technique:** XPS
 ■ **Spectral Region:** Al 2p

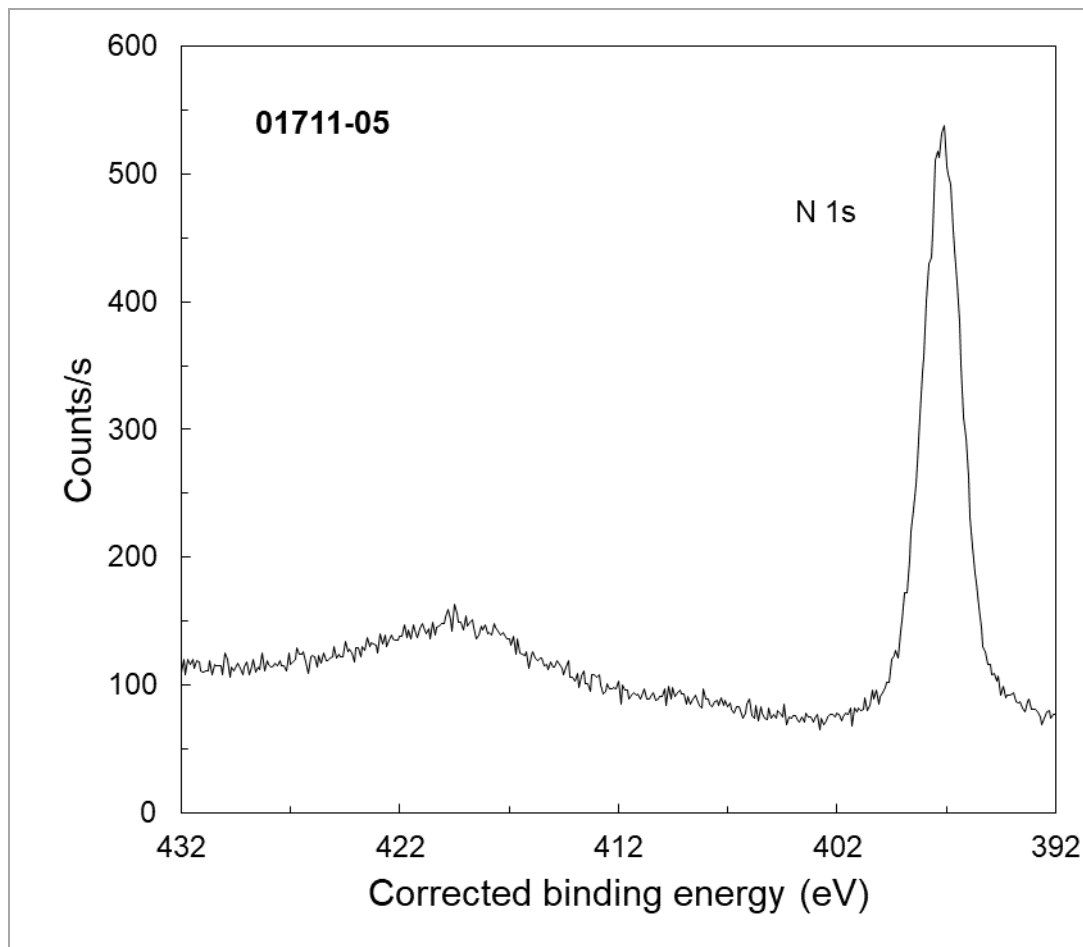
Instrument: ULVAC-PHI
 Quantes
 Excitation Source:
 Cr K α monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 50 W
 Source Size: 0.1 mm x 0.1 mm
 Analyzer Type: spherical sector
 Incident Angle: 22 °
 Emission Angle: 45 °
 Analyzer Pass Energy 112 eV
 Analyzer Resolution: 1.17 eV
 Total Signal Accumulation
 Time: 3774 s
 Total Elapsed Time: 4158 s

Number of Scans: 18

Effective Detector Width: 12.4 eV

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■ **Accession #:** 01711-05

■ **Host Material:** AlN

■ **Technique:** XPS

■ **Spectral Region:** N 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K α 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: 3774 s

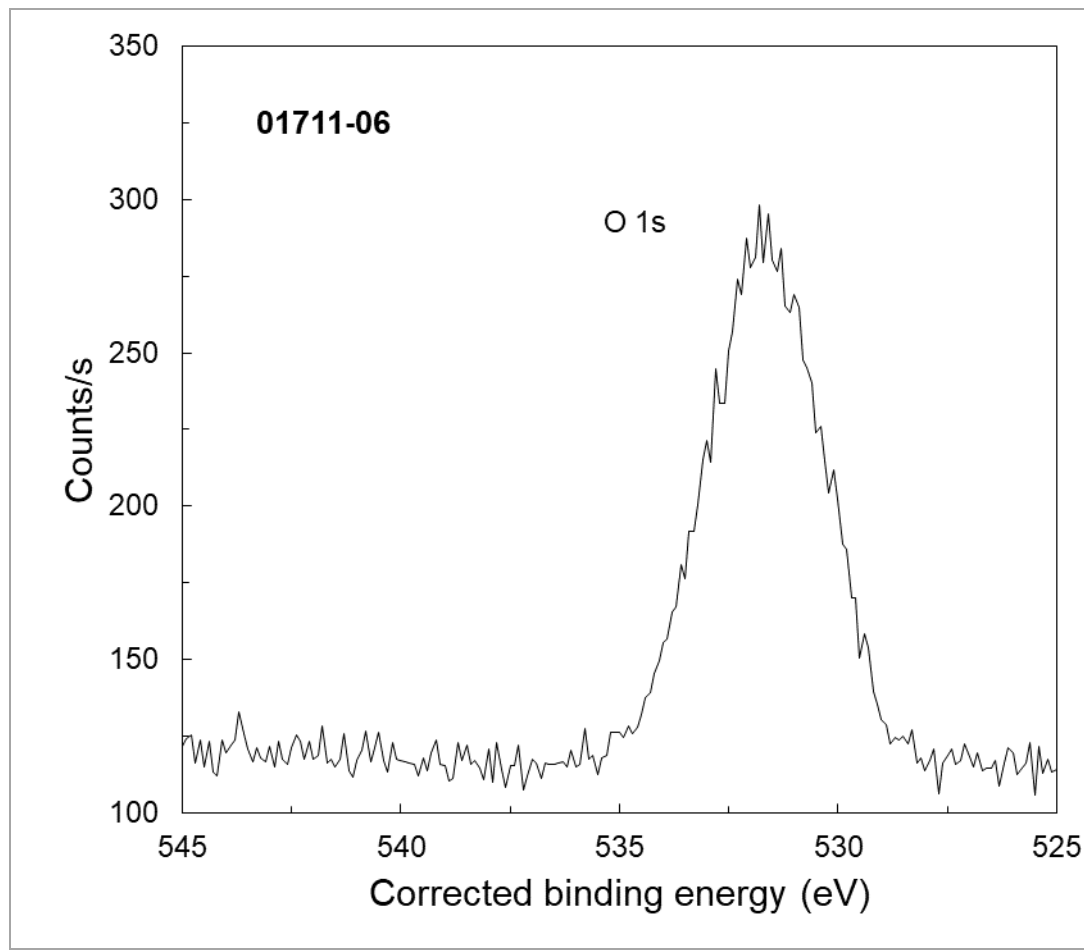
Total Elapsed Time: 4158 s

Number of Scans: 18

Effective Detector Width: 12.4 eV

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■ **Accession #:** 01711-06
 ■ **Host Material:** AlN
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

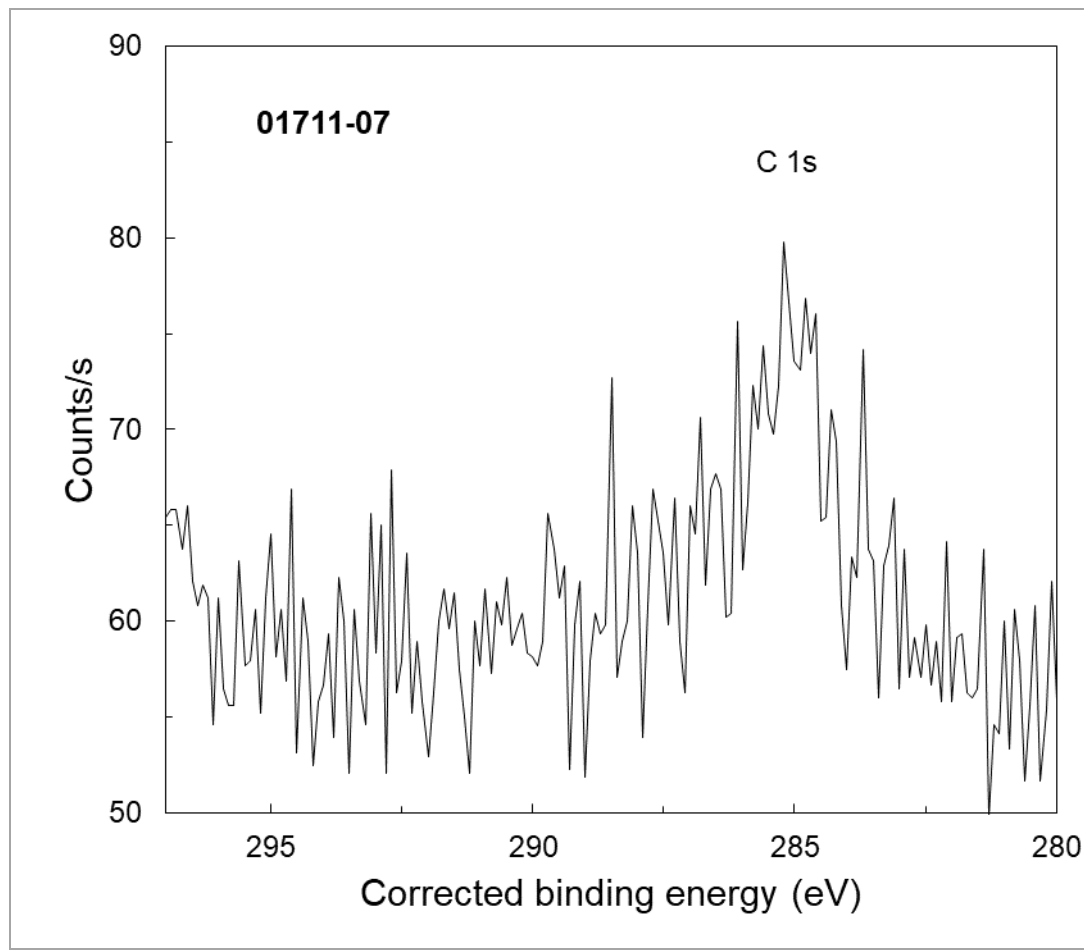
Instrument: ULVAC-PHI
 Quantes
 Excitation Source:
 Cr K α monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 50 W
 Source Size: 0.1 mm x 0.1 mm
 Analyzer Type: spherical sector
 Incident Angle: 22 °
 Emission Angle: 45 °
 Analyzer Pass Energy 112 eV
 Analyzer Resolution: 1.17 eV
 Total Signal Accumulation
 Time: 2334 s
 Total Elapsed Time: 2574 s

Number of Scans: 18

Effective Detector Width: 12.4 eV

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■ Accession #: 01711-07

■ Host Material: AlN

■ Technique: XPS

■ Spectral Region: C 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K α 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: 1554 s

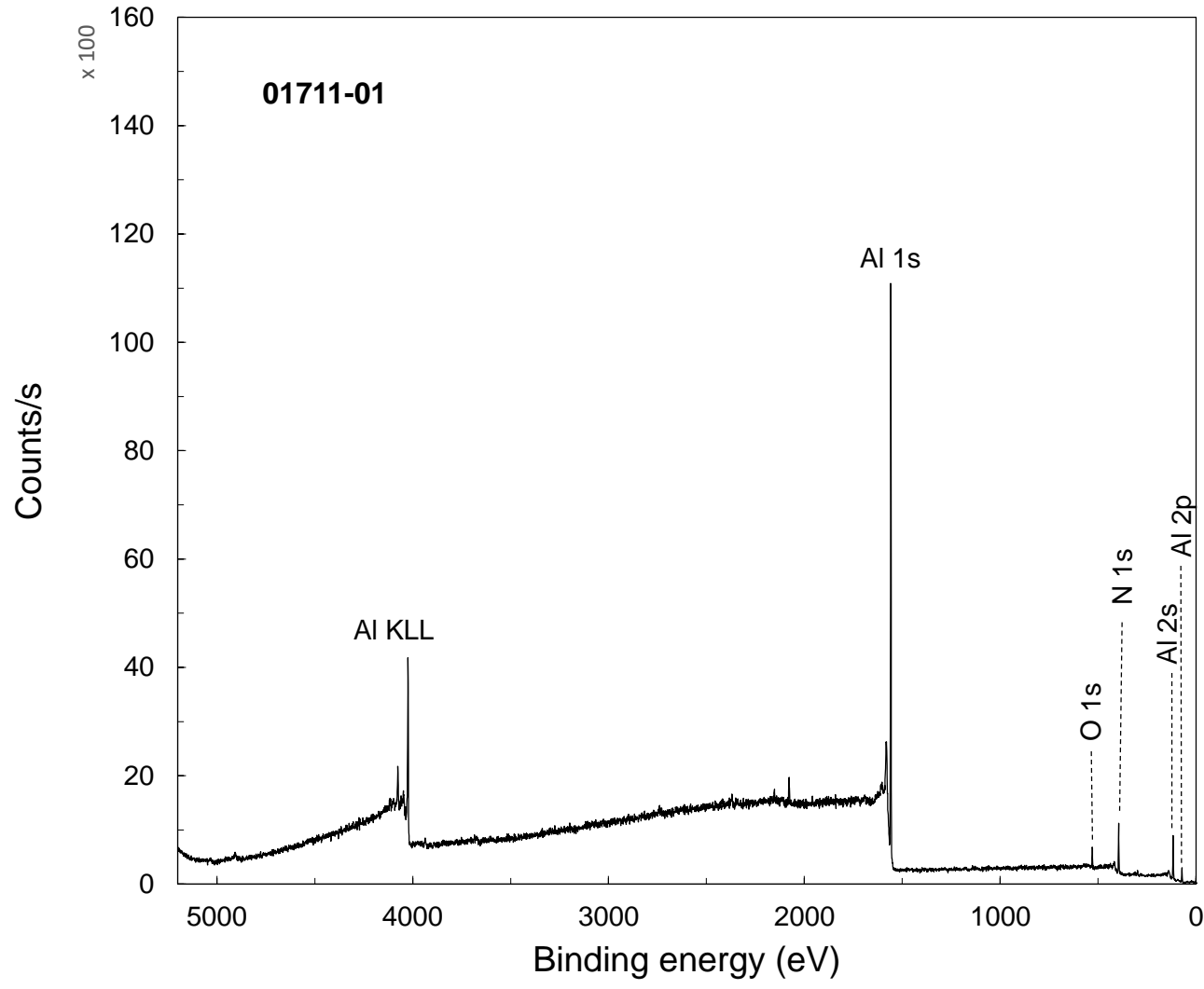
Total Elapsed Time: 1716 s

Number of Scans: 12

Effective Detector Width: 12.4 eV

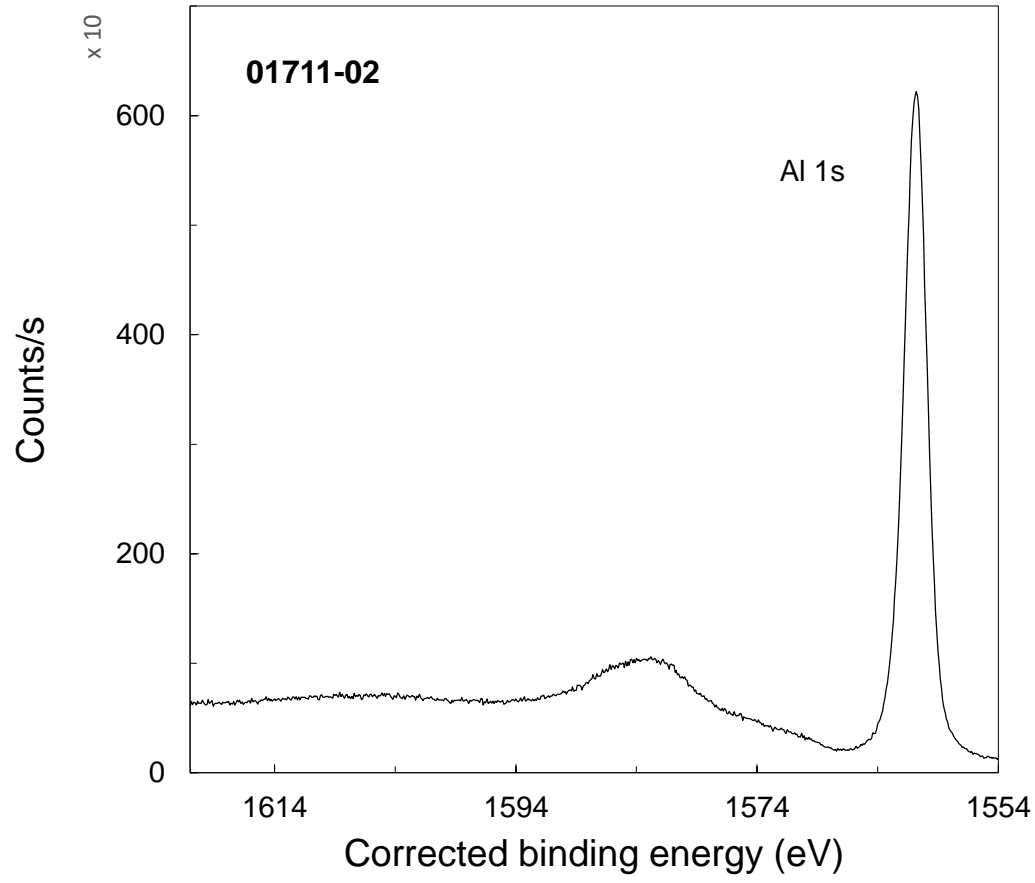
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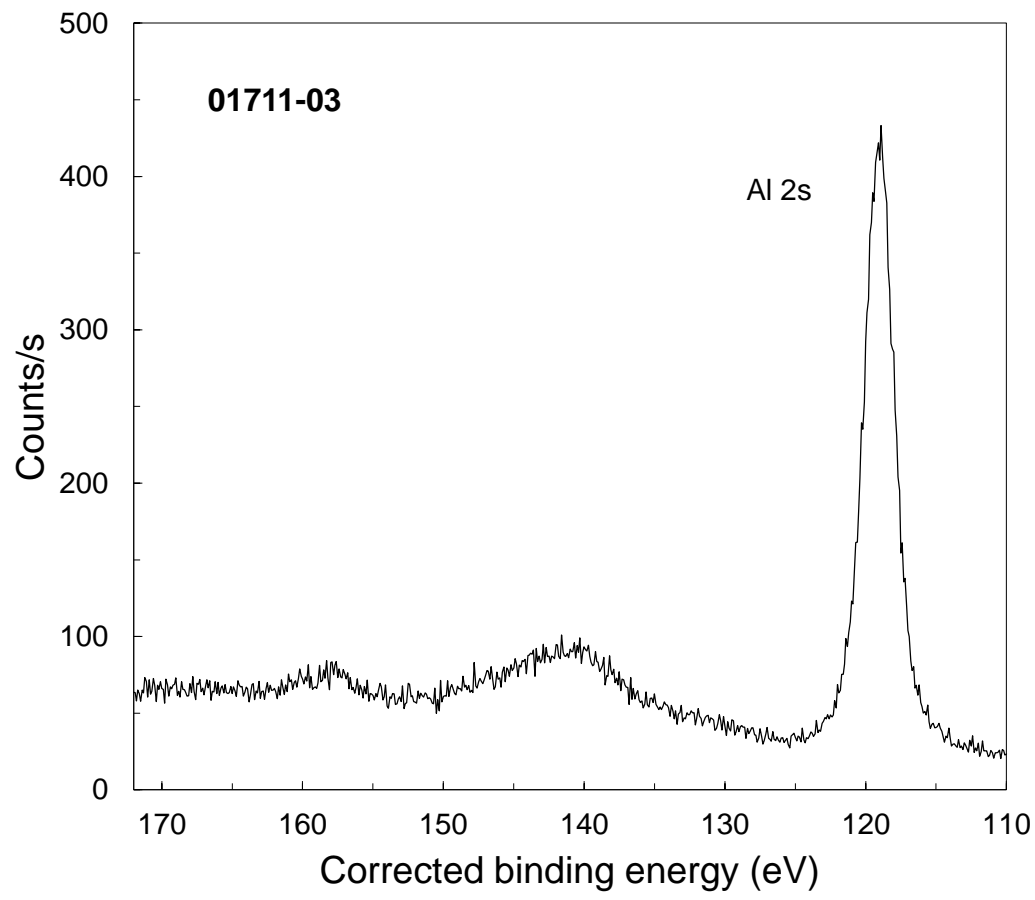
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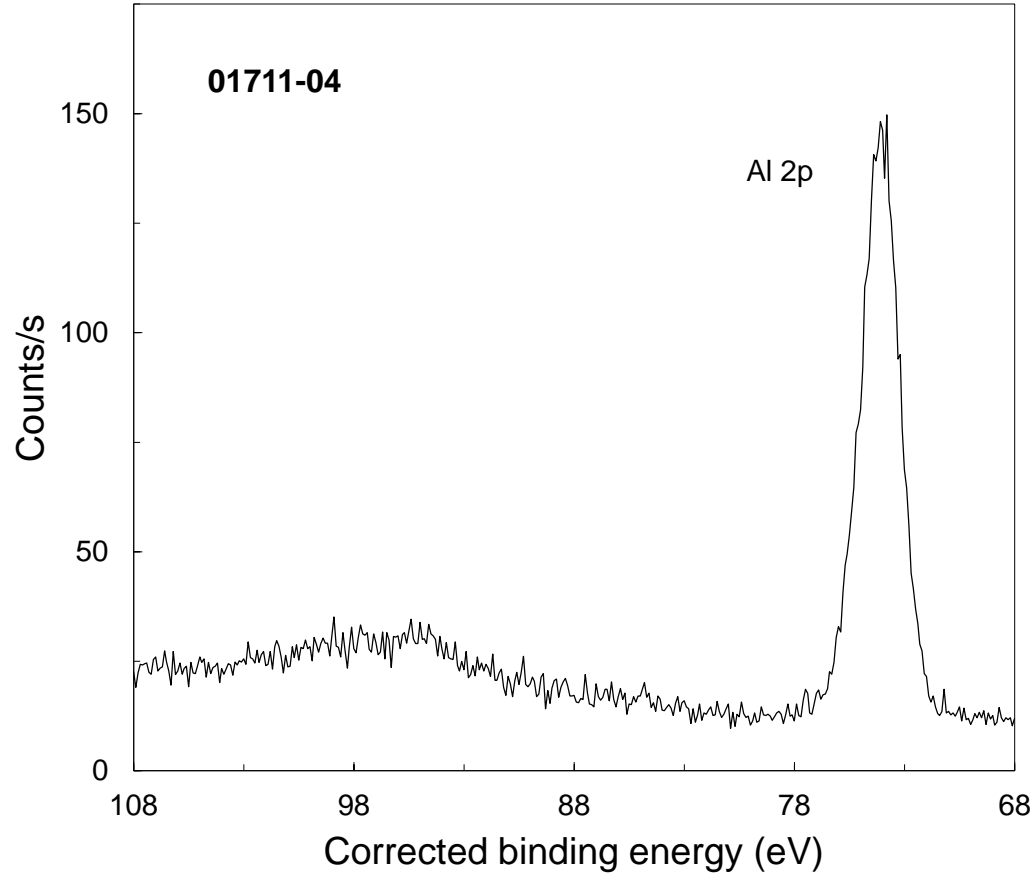
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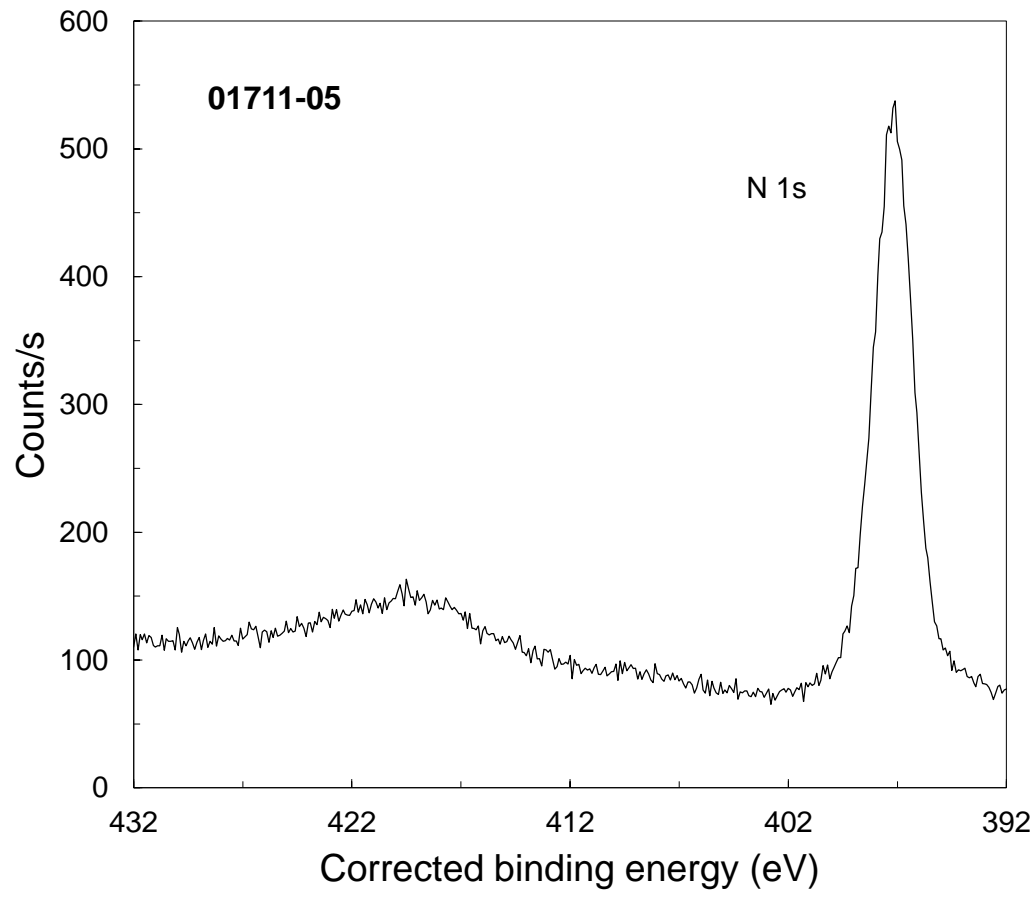
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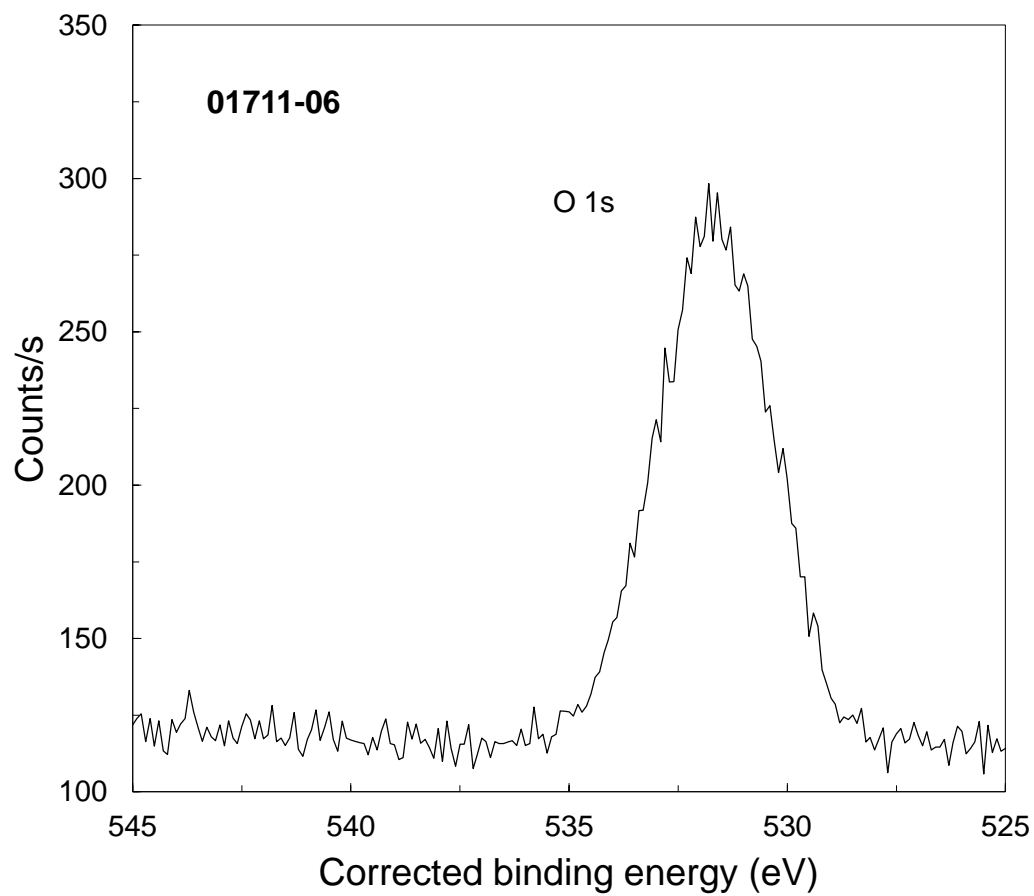
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