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HAXPES on SiO_2 with Ga Kα photons

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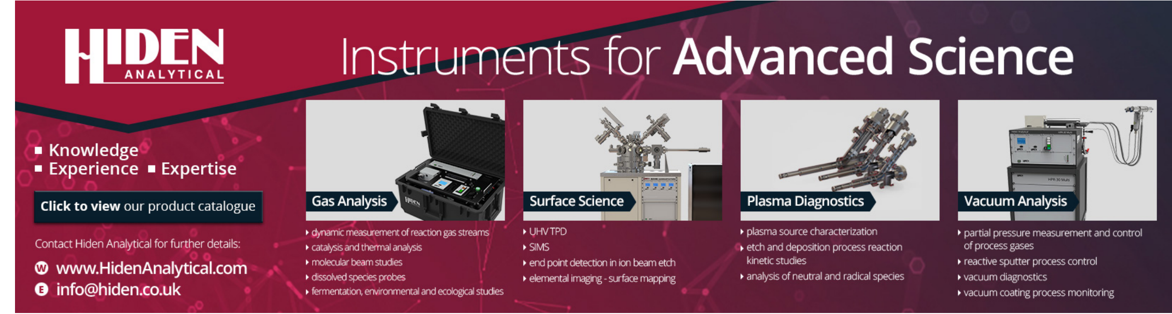

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ARTICLE

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Note: This paper is part of the 2022 Special Topic Collection on Higher Energy X-ray Photoelectron Spectroscopy.

ABSTRACT

Silicon oxide (SiO_2) grown by rapid thermal oxidation (RTO) was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of SiO_2 obtained using monochromatic Ga Kα radiation at 9252.13 eV include a survey scan and high-resolution spectra of Si 1s, Si 2p, O 1s, and C 1s.

Key words: SiO_2, HAXPES, Ga Kα

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Accession#: 01733 and 01734
Technique: XPS
Host Material: SiO_2
Instrument: Scienta Omicron, HAXPES Lab
Major Elements in Spectra: Si and O
Minor Elements in Spectra: C
Published Spectra: 6
Spectra in Electronic Record: 6
Spectral Category: Comparison

INTRODUCTION

While high-energy photoemission has been in use for decades, very few reference spectra are available, even for common materials. The recent availability of performant lab-scale photoemission spectrometers (Refs. 1–3) requires reliable reference data.

In this work, silicon oxide (SiO_2) grown by Rapid Thermal Oxidation (RTO) was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of SiO_2 obtained using monochromatic Ga Kα radiation at 9252.13 eV include high-resolution spectra of Si 1s, Si 2p, O 1s, and C 1s. This work is submitted in parallel to HAXPES spectra of identical SiO_2 samples obtained at imec using monochromatic Cr Kα radiation at 5414.8 eV.

SPECIMEN DESCRIPTION (ACCESSION # 01734)

Host Material: SiO_2
CAS Registry #: 7631-86-9
Host Material Characteristics: Homogeneous; solid; amorphous; dielectric; inorganic compound; thin film
Chemical Name: Silicon dioxide
Source: RTO-grown SiO_2
Host Composition: Si
Form: Film
Structure: Amorphous film
History and Significance: Air exposed RTO SiO_2
As Received Condition: As grown-air exposed, piece of a 200 mm wafer
Analyzed Region: Same as the host material
***Ex Situ* Preparation/Mounting:** Mounted on a double-sided conducting tape.
***In Situ* Preparation:** None
Charge Control: No electron nor ion flood guns are used
Temp. During Analysis: 300 K
Pressure During Analysis: $<2\times10^{-7}$ Pa
Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01733)

Host Material: Au
CAS Registry #: 7440-57-5
Host Material Characteristics: Homogeneous; solid; amorphous; conductor; metal; other

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Chemical Name: Gold
Source: Sputtered thin film Au (500 nm)/Cr (50 nm) on silicon
Host Composition: Au
Form: Film
Structure: Amorphous
History and Significance: Long-time in-UHV reference sample (sputtered)
As Received Condition: Piece of a Si wafer
Analyzed Region: Same as host materials
***Ex Situ* Preparation/Mounting:** The sample was taped on the sample holder using an insulating removable 3 M double-sided tape.
***In Situ* Preparation:** None
Charge Control: None
Temp. During Analysis: 300 K
Pressure During Analysis: $<2 \times 10^{-7}$ Pa
Pre-analysis Beam Exposure: 0s

INSTRUMENT DESCRIPTION

Manufacturer and Model: Scienta Omicron, HAXPES Lab
Analyzer Type: Spherical sector
Detector: Two multichannel plates with a phosphor screen and a digital camera detection system
Number of Detector Elements: A camera detector with 512 pixels in the dispersive direction

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy
Throughput ($T = E^N$): $N = -2$
Excitation Source Window: The exit window of the Ga-source is Be. The HAX9-5 entrance and exit windows are Capton (Polyamid) windows.
Excitation Source: Ga K_α monochromatic
Source Energy: 9252.13 eV
Source Strength: 250 W
Source Beam Size: $80 \times 20\,\mu m^2$
Signal Mode: Multichannel direct

Geometry

Incident Angle: 3°
Source-to-Analyzer Angle: 90°
Emission Angle: 87°
Specimen Azimuthal Angle: 0°
Specimen Polar Angle: −3°
Acceptance Angle from Analyzer Axis: 0° ± 30° from the lens axis in one angular dispersive direction
Analyzer Angular Acceptance Width: 60° × 60°

DATA ANALYSIS METHOD

Energy Scale Correction: Due to the too low intensity of the C 1s peak with Ga Kα, the binding energy was referenced by determining the binding energy position of the Si 2p peak recorded by the Al Kα XPS measurement and the subsequent correction shift of the C 1s peak to 284.8 eV. The determined binding energy position of the Si 2p peak is 103.4 eV. The binding energy shift of the Ga Kα data was then performed by shifting the Si 2p peak to 103.4 eV.

Recommended Energy Scale Shift: −1.9 eV for binding energy (i.e., the Si 2p peak is shifted from 105.3 to 103.4 eV)

Peak Shape and Background Method: Data treatment was performed using a Shirley background with parameters Av Width 5, St Offset 0, and End Offset 0.

Quantitation Method: Data treatment was performed using CasaXPS software version 2.3.17PR1.1. Theoretical sensitivity factors have been used, more in detail 3.45, 30, and 0.343 for O 1s, Si 1s, and Si 2p, respectively.

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ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material (Ref. 4).

REFERENCES

[1]See https://www.ulvac-phi.com/en/products/xps/quantes/
[2]See https://www.kratos.com/products/axis-supra-xps-surface-analysis-instrument
[3]See https://scientaomicron.com/en/system-solutions/electron-spectroscopy/HAXPES-Lab
[4]See supplementary material at https://www.scitation.org/doi/suppl/10.1116/6.0001523 for the figures.

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SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01734-02	Si 1s	1843.9	1.76	4586	30	38.05	SiO_2
01734-03	Si 2p	103.4	1.63	45	0.343	…	SiO_2
01734-04	O 1s	532.9	1.57	827.1	3.45	60.74	SiO_2
01734-05	C 1s	284.8	1.49	4.71	1	1.22	Contamination

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
…	Au $4f_{7/2}$	83.34[a]	0.94	184.5	…	…	…

[a]No binding energy correction shift applied.

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift[a]	Multiplier	Baseline	Comment #
01734-01	Survey	0	1	0	…
01734-02	Si 1s	1.9	1	0	…
01734-03	Si 2p	1.9	1	0	…
01734-04	O 1s	1.9	1	0	…
01734-05	C 1s	1.9	1	0	…
01733-01	Survey	0	1	0	Au reference[b]

[a]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.
[b]The reference spectrum included in this article is the same as those included with other articles in this collection that use this x-ray source. This was intentional and not an error as submissions were requested to be accompanied by a wide-scan spectrum of sputter-cleaned gold taken on the same instrument and using the same settings as the wide-scan spectrum of the material or materials in the submission.

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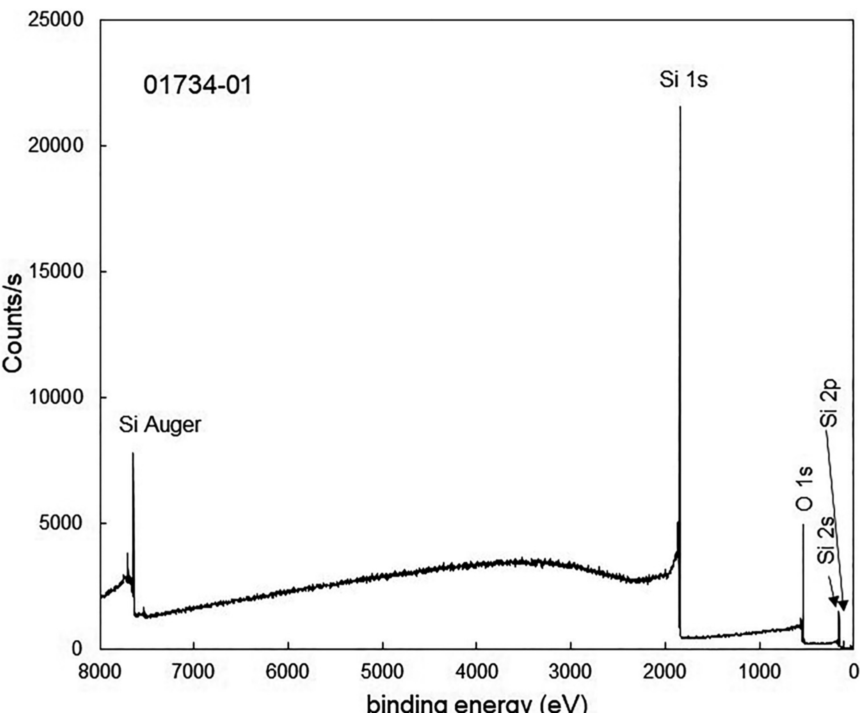

Accession #	01734-01
Host Material:	SiO_2
Technique:	XPS
Spectral Region:	Survey
Instrument:	Scienta Omicron, HAXPES Lab
Excitation Source:	Ga K_α monochromatic
Source Energy:	9252.13 eV
Source Strength:	250 W
Source Size:	0.08 × 0.02 mm^2
Analyzer Type:	Spherical sector analyzer
Incident Angle:	3°
Emission Angle:	87°
Analyzer Pass Energy:	500 eV
Analyzer Resolution:	1.25 eV
Total Signal Accumulation Time:	5274 s
Total Elapsed Time:	5275 s
Number of Scans:	10
Effective Detector Width:	40 eV

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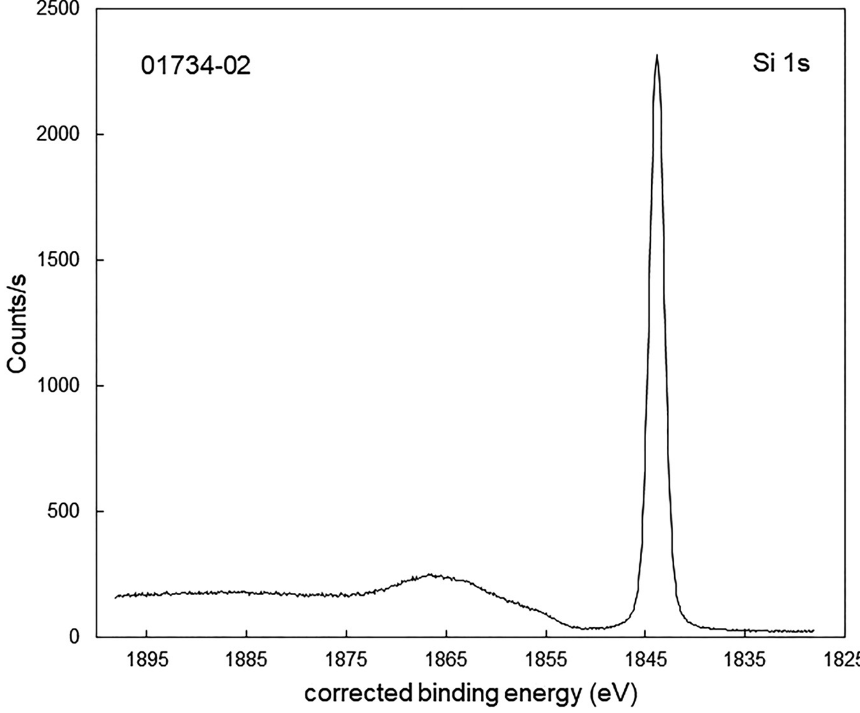
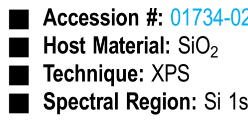
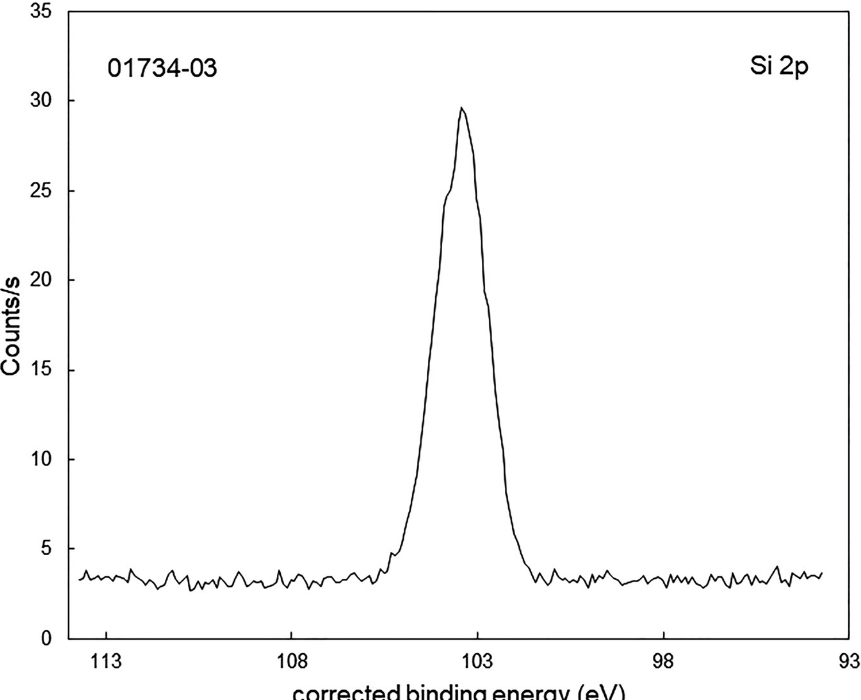


- **Accession #:** 01734-02
- **Host Material:** SiO_2
- **Technique:** XPS
- **Spectral Region:** Si 1s

Instrument: Scienta Omicron, HAXPES Lab
Excitation Source: Ga K_α monochromatic
Source Energy: 9252.13 eV
Source Strength: 250 W
Source Size: 0.08 × 0.02 mm^2
Analyzer Type: Spherical sector
Incident Angle: 3°
Emission Angle: 87°
Analyzer Pass Energy 100 eV
Analyzer Resolution: 0.25 eV
Total Signal Accumulation Time: 10 786 s
Total Elapsed Time: 10 800 s
Number of Scans: 70
Effective Detector Width: 8 eV





- **Accession #:** 01734-03
- **Host Material:** SiO_2
- **Technique:** XPS
- **Spectral Region:** Si 2p

Instrument: Scienta Omicron, HAXPES Lab
Excitation Source: Ga K_α monochromatic
Source Energy: 9252.13 eV
Source Strength: 250 W
Source Size: 0.08 × 0.02 mm^2
Analyzer Type: Spherical sector
Incident Angle: 3°
Emission Angle: 87°
Analyzer Pass Energy 100 eV
Analyzer Resolution: 0.25 eV
Total Signal Accumulation Time: 16 143 s
Total Elapsed Time: 16 200 s
Number of Scans: 285
Effective Detector Width: 8 eV

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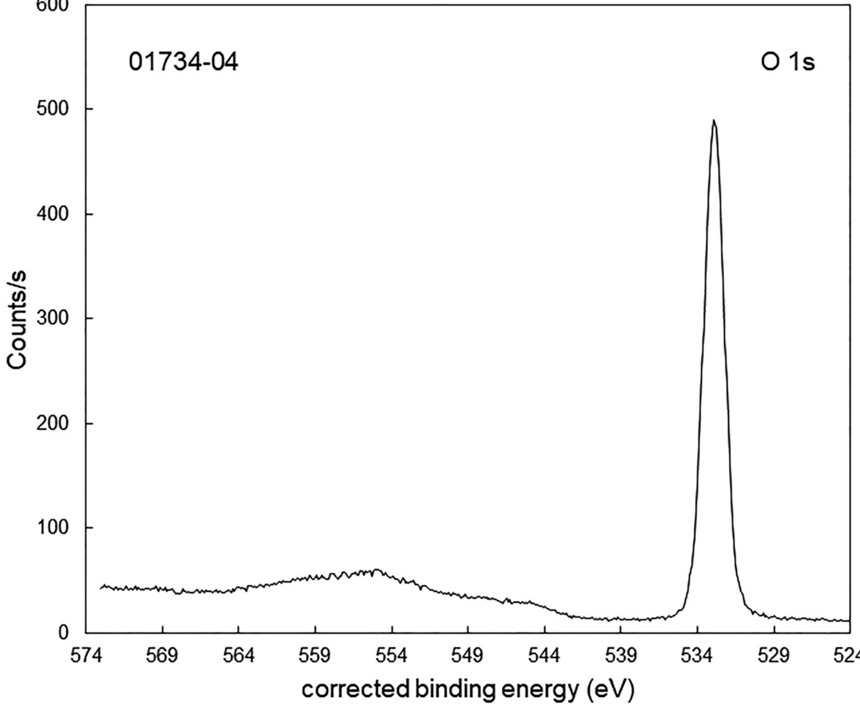
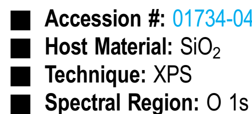
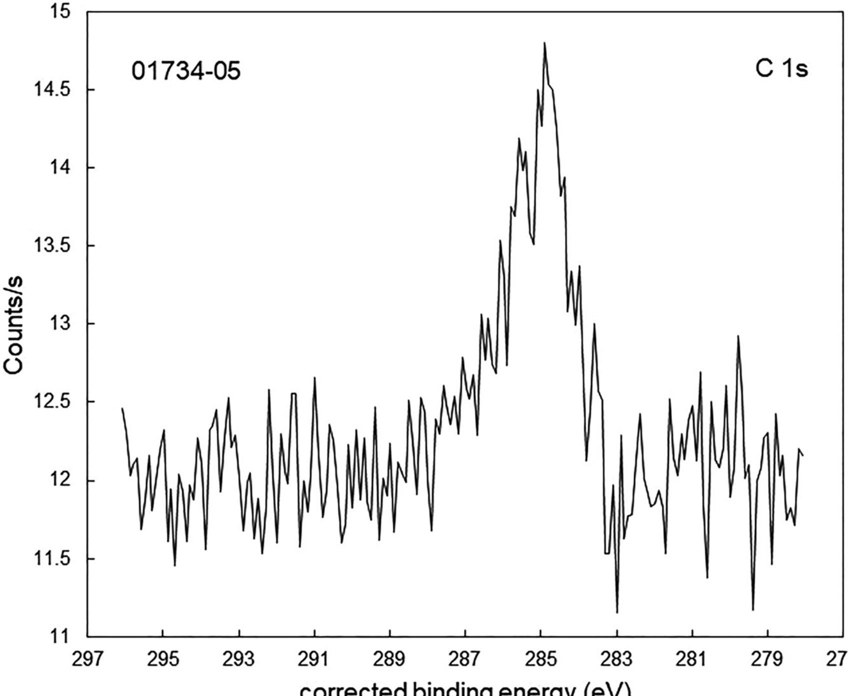




■ **Accession #:** 01734-04
■ **Host Material:** SiO_2
■ **Technique:** XPS
■ **Spectral Region:** O 1s

Instrument: Scienta Omicron, HAXPES Lab
Excitation Source: Ga K_α monochromatic
Source Energy: 9252.13 eV
Source Strength: 250 W
Source Size: 0.08 × 0.02 mm^2
Analyzer Type: Spherical sector
Incident Angle: 3°
Emission Angle: 87°
Analyzer Pass Energy 100 eV
Analyzer Resolution: 0.25 eV
Total Signal Accumulation Time: 12 579 s
Total Elapsed Time: 12 600 s
Number of Scans: 106
Effective Detector Width: 8 eV



■ **Accession #:** 01734-05
■ **Host Material:** SiO_2
■ **Technique:** XPS
■ **Spectral Region:** C 1s

Instrument: Scienta Omicron, HAXPES Lab
Excitation Source: Ga K_α monochromatic
Source Energy: 9252.13 eV
Source Strength: 250 W
Source Size: 0.08 × 0.02 mm^2
Analyzer Type: Spherical sector
Incident Angle: 3°
Emission Angle: 87°
Analyzer Pass Energy 100 eV
Analyzer Resolution: 0.25 eV
Total Signal Accumulation Time: 30 621 s
Total Elapsed Time: 30 725 s
Number of Scans: 522
Effective Detector Width: 8 eV

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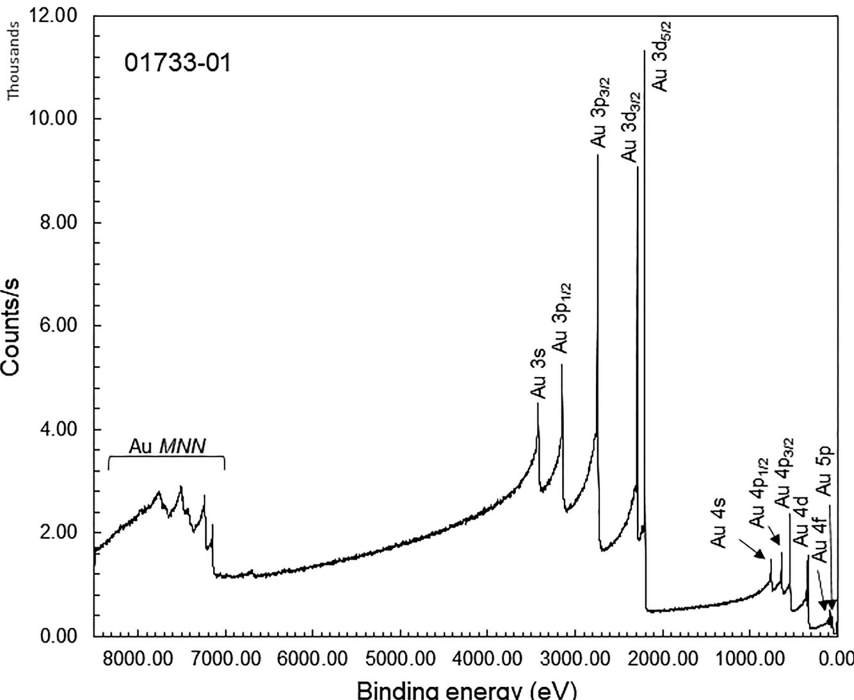

Accession #	01733-01
Host Material:	Au
Technique:	XPS
Spectral Region:	Survey
Instrument:	Scienta Omicron, HAXPES Lab
Excitation Source:	Ga K_{α} monochromatic
Source Energy:	9252.13 eV
Source Strength:	250 W
Source Size:	0.08 × 0.02 mm^2
Analyzer Type:	Spherical sector analyzer
Incident Angle:	3°
Emission Angle:	87°
Analyzer Pass Energy:	500 eV
Analyzer Resolution:	1.25 eV
Total Signal Accumulation Time:	5274 s
Total Elapsed Time:	5275 s
Number of Scans:	10
Effective Detector Width:	40 eV

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