

Au/SiO₂ Nanosystems by XPS

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Au/SiO₂ nanocomposites were prepared by rf-sputtering of gold on amorphous silica substrates. Deposition experiments were carried out in Ar plasmas at temperatures as low as 60 °C. Particular attention was devoted to the combined influence of the applied rf power and total pressure on the chemico-physical properties of the final nanosystems. In particular, low pressures (<0.2 mbar) and high rf powers (>20 W) resulted in a higher sputtering yield, allowing the deposition of continuous gold films on silica. Conversely, decreased sputtering yields (at higher pressures and lower powers) enabled the preparation of discontinuous Au/SiO₂ nanosystems. A thorough investigation of the structure-properties relationships was attained by means of a multi-technique characterization. In particular, laser reflection interferometry (LRI) was employed for an *in situ* monitoring of growth processes, while glancing-incidence x-ray diffraction (GIXRD) and transmission electron microscopy (TEM) provided valuable information on the system nanostructure. Moreover, x-ray photoelectron spectroscopy (XPS), UV-Vis spectroscopy, and atomic force microscopy (AFM) were used to investigate the chemical composition, optical properties, and surface morphology, respectively. This study is dedicated to an XPS investigation of the principal core levels (Au, Si, O) of Au/SiO₂ nanosystems. In particular, detailed scans for the Au 4*f*, Si 2*s*, O 1*s*, and C 1*s* regions and related data for a gold film on silica and a discontinuous Au/SiO₂ specimen are presented and discussed. © 2004 American Vacuum Society. [DOI: 10.1116/11.20040101]

Keywords: Au/SiO₂; thin films; island-like systems; rf-sputtering; x-ray photoelectron spectroscopy

PACS: 81.07.-b, 82.50.Kx, 82.50.Hp

INTRODUCTION

Gold nanoparticles dispersed on insulating matrices like SiO₂ are considered promising materials for several technological applications, such as catalysis (Refs. 1 and 2) and non-linear optics (Refs. 3 and 4). As a general rule, a careful control of gold nanoparticle size, shape and composition in Au/SiO₂ nanosystems might result in systems with well tailored functional properties. Specifically, two major categories of gold-silica nanocomposites have been recognized as a function of morphology and overall metal content, namely discontinuous (cluster- or island-like) systems (Refs. 5 and 6) and continuous thin films (Ref. 7). The possibility of obtaining prescribed material features by tailoring Au/SiO₂ nanostructure and morphology has motivated the utilization of various preparation methodologies for these nanosystems. Among them, rf-sputtering is one of the most versatile thanks to its flexibility that allows one to control nanocluster size and distribution under relatively mild conditions.

This work is part of a research project aimed at investigating the nucleation and growth of gold nanoparticles deposited by rf-sputtering on different substrates. In fact, it is generally recognized that the substrate nature plays a prominent role in determining the chemico-physical properties of supported Au nanosystems, that, in turn, strongly influence their functional performances. In the present case, our attention was specifically devoted to the investigation of Au/SiO₂ nanosystems. As a matter of fact, the weak Au-SiO₂ chemical interactions allow investigation of the morphology of gold nanoparticles as a function of the synthesis conditions. To this regard, previous literature reports have not been

completely exhaustive.

Gold nanosystems were prepared on commercial silica substrates (Au/SiO₂) at temperatures as low as 60 °C from Ar plasmas. A proper choice of the preparation conditions enabled the switching from discontinuous systems to Au thin films with a fine modulation of the system characteristics. In this article, XPS spectra of two selected specimens belonging to the two categories are reported.

SPECIMEN DESCRIPTION (Accession #00774)

Host Material: Au thin film on silica

CAS Registry #: 7440-57-5

Host Material Characteristics: homogeneous; solid; polycrystalline; conductor; metal; thin film

Chemical Name: gold

Source: sample obtained by rf-sputtering of gold on silica

Host Composition: Au

Form: thin film

Lot #: AuSi13

Structure: GIXRD analysis revealed the presence of fcc metal gold ($\Phi \approx 9$ nm), with no appreciable preferential orientation.

History & Significance: Gold depositions were performed in Ar plasmas (purity 5.0) by means of a custom-built radio frequency (rf) sputtering apparatus ($\nu = 13.56$ MHz) equipped with two vertical electrodes (Ref. 7). A gold target (BAL-TEC AG, 99.99%) was fixed on the powered electrode, while the

Accession #s 00774, 00778

Technique: XPS

Host Material: #00774: Au thin film on silica; #00778: Au/SiO₂ nanocomposite thin film

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci

Major Elements in Spectrum: Au, Si, O

Minor Elements in Spectrum: C

Printed Spectra: 9

Spectra in Electronic Record: 11

Spectral Category: comparison

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substrates were placed on a grounded electrode maintained at 60 °C throughout each experiment. The electrode-to-electrode distance and Ar flow were fixed at 50 mm and 10 sccm, respectively.

The Au film analyzed in the present work was obtained at a total pressure of 0.080 mbar and a rf power of 35 W (deposition time = 10 min). The sample displayed the typical yellow color of bulk gold. The optical spectrum revealed the presence of the so-called *transmission window* centered at $\lambda \approx 500$ nm, characteristic of continuous Au films on SiO₂ (Ref. 7).

Before film preparation, HeraSil silica slides (Heraeus, Quarzschmelze, Hanau, Germany) were cleaned by a well-established procedure (Ref. 8) aimed at removing undesired contamination of the growth surface.

As Received Condition: as grown

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: sample mounted as-received by a fast entry lock system

In Situ Preparation: none

Charge Control: none

Temp. During Analysis: 298 K

Pressure During Analysis: $<1 \times 10^{-6}$ Pa

SPECIMEN DESCRIPTION (Accession #00778) ---

Host Material: Au/SiO₂ nanocomposite thin film

Host Material Characteristics: homogeneous; solid; polycrystalline; dielectric; thin film

Chemical Name: gold/silicon dioxide

Source: sample obtained by rf-sputtering of gold on silica

Host Composition: Au, Si, O

Form: nanocomposite thin film

Lot #: AuSi14

Structure: GIXRD analysis revealed the presence of fcc metal gold ($\Phi \approx 6$ nm), with no appreciable preferential orientation.

History & Significance: The sample was deposited using the previously described rf-sputtering instrumentation (Accession #00774, History & Significance), at the same substrate temperature, deposition time, electrode-to-electrode distance, and Ar flow rate. The differences with respect to the previous case were the total pressure (0.100 mbar) and rf power (5 W). The present specimen was blue, in agreement with the appearance of a broadened absorption extending towards the IR region. A similar phenomenon, further supported by other characterization techniques (Ref. 7), suggested the presence of a discontinuous layer, i.e., an incomplete surface coverage of the silica substrate. In particular, the obtained sample was characterized by an island-like morphology, also indicated by TEM investigation (Ref. 7).

As Received Condition: as-grown

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: sample mounted as-received by a fast entry lock system

In Situ Preparation: none

Charge Control: none

Temp. During Analysis: 298 K

Pressure During Analysis: $<1 \times 10^{-6}$ Pa

INSTRUMENT DESCRIPTION ---

Manufacturer and Model: Perkin-Elmer Physical Electronics, Inc. 5600ci

Analyzer Type: spherical sector

Detector: multichannel detector, part no. 619103

Number of Detector Elements: 16

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA ---

■ **Spectrometer**

Analyzer Mode: constant pass energy

Throughput ($T = E^N$): $N = -1$

Excitation Source Window: 1.5 μm Al window

Excitation Source: Mg K_{α}

Source Energy: 1253.6 eV

Source Strength: 400 W

Source Beam Size: $>25000 \mu\text{m} \times >25000 \mu\text{m}$

Analyzer Width: $800 \mu\text{m} \times 800 \mu\text{m}$

Signal Mode: multichannel direct

■ **Geometry**

Incident Angle: 9°

Source to Analyzer Angle: 53.8°

Emission Angle: 45°

Specimen Azimuthal Angle: 0°

Acceptance Angle from Analyzer Axis: 0°

Analyzer Angular Acceptance Width: $14^{\circ} \times 14^{\circ}$

■ **Ion Gun**

Manufacturer and Model: PHI 04-303A

Energy: 3000 eV

Current: 0.4 mA/cm²

Current Measurement Method: Faraday cup

Sputtering Species: Ar⁺

Spot Size (unrastered): 250 μm

Raster Size: $2000 \mu\text{m} \times 2000 \mu\text{m}$

Incident Angle: 40°

Polar Angle: 45°

Azimuthal Angle: 111°

Comment: differentially pumped ion gun

DATA ANALYSIS METHOD ---

Energy Scale Correction: The adventitious hydrocarbon signal at 284.8 eV was used as the energy reference to correct for charging (Ref. 9).

Recommended Energy-Scale Shift: none for Accession #00774-01 to -04; -13.58 eV for Accession #00778-01 to -05

Peak Shape and Background Method: After a Shirley-type background subtraction (Ref. 10), the raw spectra were fitted by a least-squares procedure adopting Gaussian-Lorentzian

functions for C 1s, O 1s, and Si 2s peaks, while an asymmetric peak fitting was performed on the Au 4f signal. Peak positions and widths were determined from fitting using the standard PHI V5.4A software.

Quantitation Method: Sensitivity factors were taken from the standard PHI V5.4A software. The peak areas were measured above an integrated background. The atomic compositions were evaluated using the standard PHI V5.4A software.

ACKNOWLEDGMENTS

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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
00774-02	C 1s	284.8	2.0	30772	17.059	43.5	adventitious carbon
00774-03 ^a	Au 4f _{7/2}	84.4	1.5	403001	359.657	47.2	metallic Au
00774-03	Au 4f _{5/2}	88.1	1.5	302250	metallic Au
00774-04	O 1s	532.4	2.9	15836	41.068	9.3	adsorbed oxygen
00778-02	C 1s	284.8	2.1	32375	17.059	32.7	adventitious carbon
00778-03 ^a	Au 4f _{7/2}	84.3	1.7	311271	359.657	26.1	metallic Au
00778-03	Au 4f _{5/2}	88.0	1.7	233453	metallic Au
00778-04	O 1s	533.0	2.4	65682	41.068	27.6	O in SiO ₂
00778-05	Si 2s	154.9	3.3	14608	18.515	13.6	Si in SiO ₂

^a The sensitivity factor and concentration refer to the entire Au 4f signal.

Footnote to Spectrum 00774-02: After recording the core level spectra, 1 min Ar⁺ sputtering (3 keV, 2 × 2 mm² raster size) led to the disappearance of the C signal, indicating that it arose from atmospheric contamination.

Footnote to Spectrum 00774-03: The Au 4f_{7/2} component was located at BE ≈ 84.4 eV (FWHM = 1.5 eV), in agreement with literature data for metallic gold (Refs. 11–15). The shift of ≈ +0.4 eV with respect to values reported for bulk metal might suggest the presence of core-level shifts (Refs. 16–18), caused by an incomplete screening of the core-hole (Ref. 19) typical for nanostructured gold on poorly conducting substrates, such as SiO₂. This shift could be related to the relatively low Au nanocrystal size ($\phi \approx 9$ nm).

Footnote to Spectrum 00774-04: The O 1s signal (BE = 532.4 eV; FWHM = 2.9 eV) was completely removed after 1 min Ar⁺ erosion (3 keV, 2 × 2 mm² raster size), indicating it arose from atmospheric contamination (adsorbed oxygen).

Footnote to Spectrum 00778-02: After recording the core-level spectra, 1 min Ar⁺ sputtering (3 keV, 2 × 2 mm² raster size) led to the disappearance of the C signal, indicating that it arose from atmospheric contamination.

Footnote to Spectrum 00778-03: The Au 4f_{7/2} component was located at BE ≈ 84.3 eV (FWHM = 1.7 eV). The shift of +0.3 eV with respect to values reported for bulk metal might suggest the presence of core-level shifts (Refs. 16–19; see comment to Accession #00774-03). Despite a higher BE shift would be expected for the present specimen, due to the incomplete silica coverage and the lower crystallite size ($\phi \approx 6$ nm) with respect to the nanostructured Au layer on silica, more detailed conclusions are prevented due to the analyzer resolution employed (0.6 eV in both cases). A comparison with data for the previous sample indicates an increase in the Au 4f peak FWHM of +0.2 eV, in agreement with previous reports (Ref. 19).

Footnote to Spectrum 00778-05: The Si 2s signal was recorded instead of the Si 2p one since the latter was located on the high BE side of the intense Au 4f peak. The Si 2s BE (154.9 eV; FWHM = 3.3 eV) was in agreement with SiO₂ presence (Refs. 11, 20–25).

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
00840-01	Au 4f _{7/2}	84.0	1.4	186403
00841-01	Cu 2p _{3/2}	932.7	1.6	86973

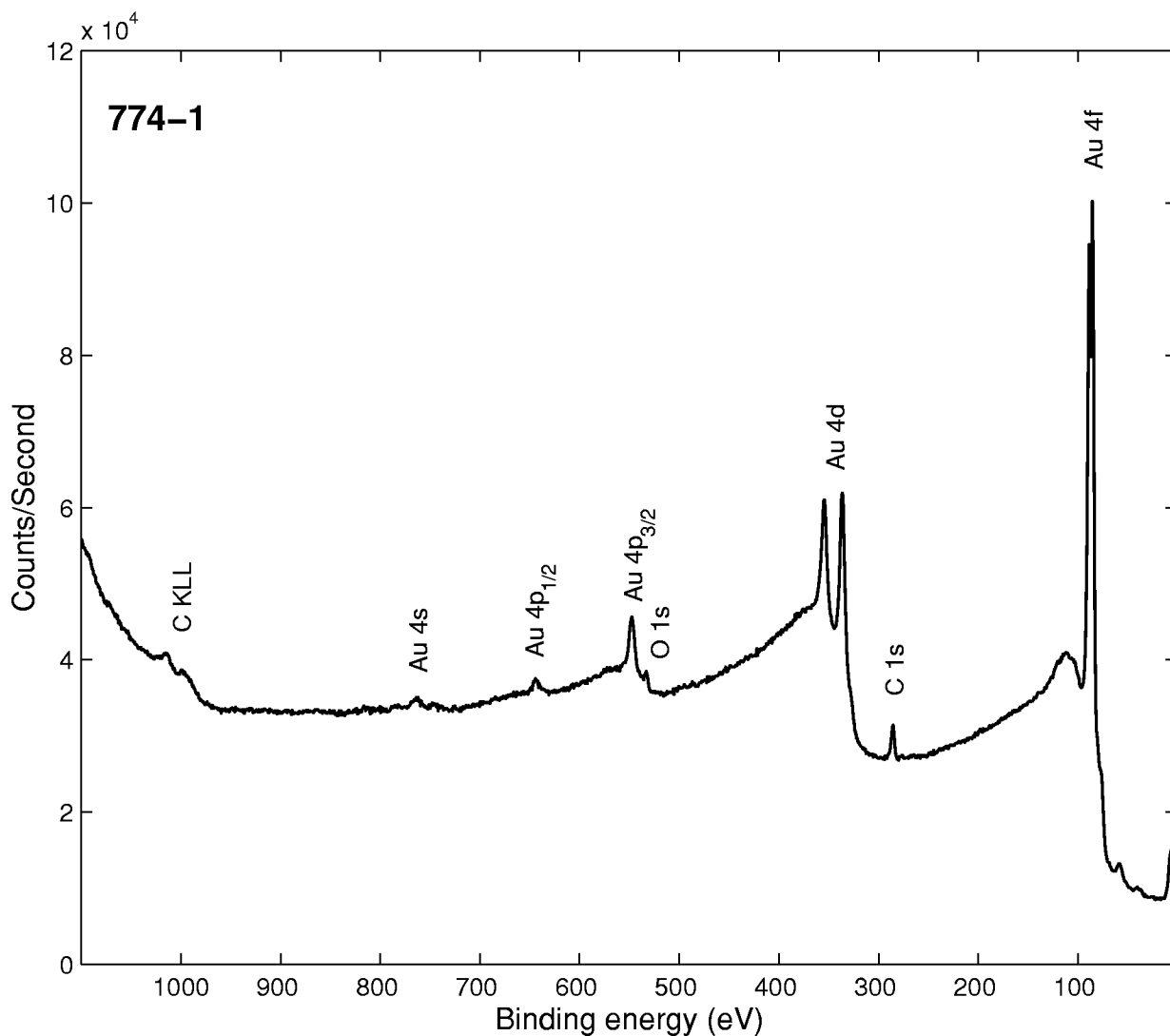
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
774-1	Survey	0	1	0	1
774-2	C 1s	0	1	0	1
774-3	Au 4f	0	1	0	1
774-4	O 1s	0	1	0	1
778-1	Survey	+13.58	1	0	2
778-2	C 1s	+13.58	1	0	2
778-3	Au 4f	+13.58	1	0	2
778-4	O 1s	+13.58	1	0	2
778-5	Si 2s	+13.58	1	0	2
840-1 [NP]**	Au 4f _{7/2}	0	1	0	3
841-1 [NP]	Cu 2p _{3/2}	0	1	0	3

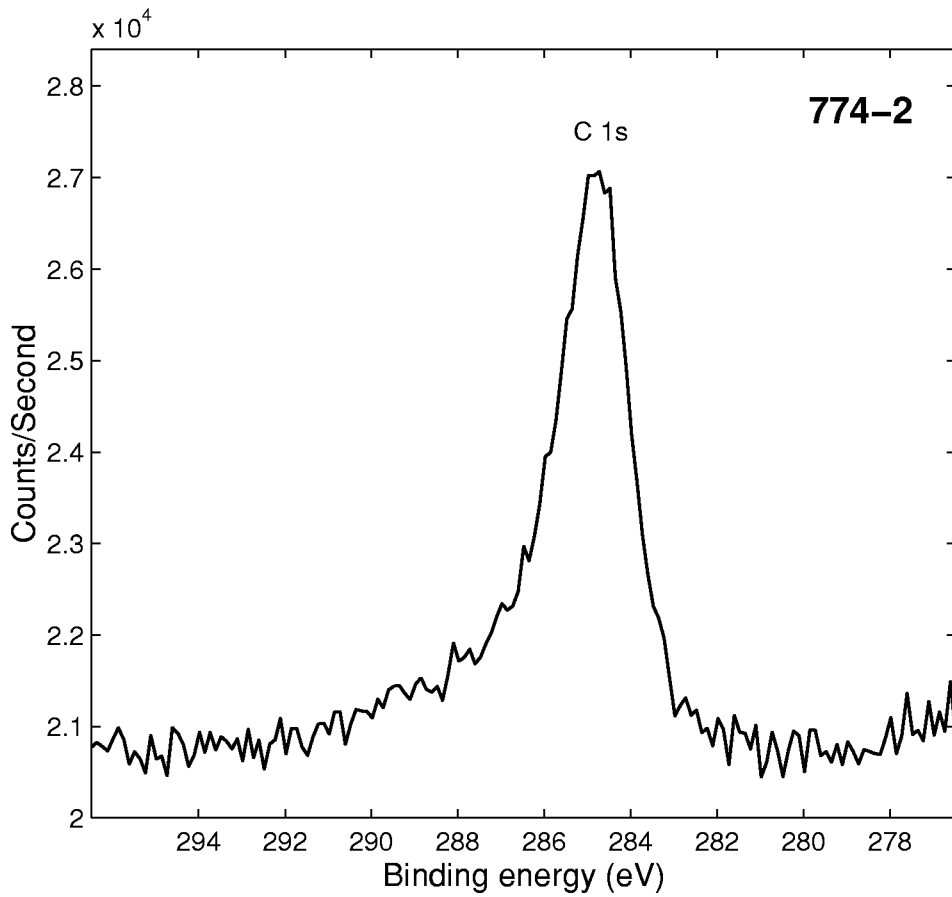
* Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

** [NP] signifies not published; digital spectra are archived in SSS database but not reproduced in the printed journal.

1. Au thin film on silica substrate
2. Au/SiO₂ nanocomposite thin film
3. Calibration spectrum

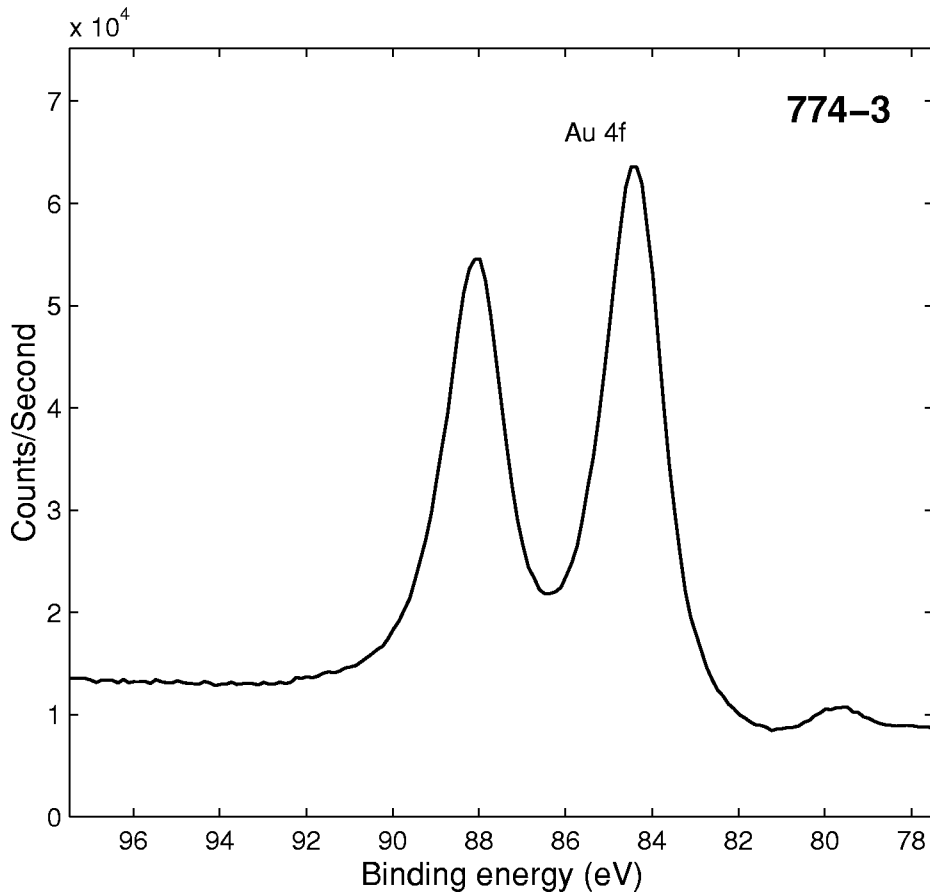


Accession #	00774-01
Host Material	Au thin film on silica
Technique	XPS
Spectral Region	survey
Instrument	Perkin-Elmer Physical Electronics, Inc. 5600ci
Excitation Source	Mg K_{α}
Source Energy	1253.6 eV
Source Strength	400 W
Source Size	>25 mm \times >25 mm
Analyzer Type	spherical sector
Incident Angle	9°
Emission Angle	45°
Analyzer Pass Energy	187.85 eV
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	247.5 s
Total Elapsed Time	272.2 s
Number of Scans	9
Effective Detector Width	1.9 eV
Comment	The absence of silicon signals indicates a homogeneous coverage of the substrate surface.



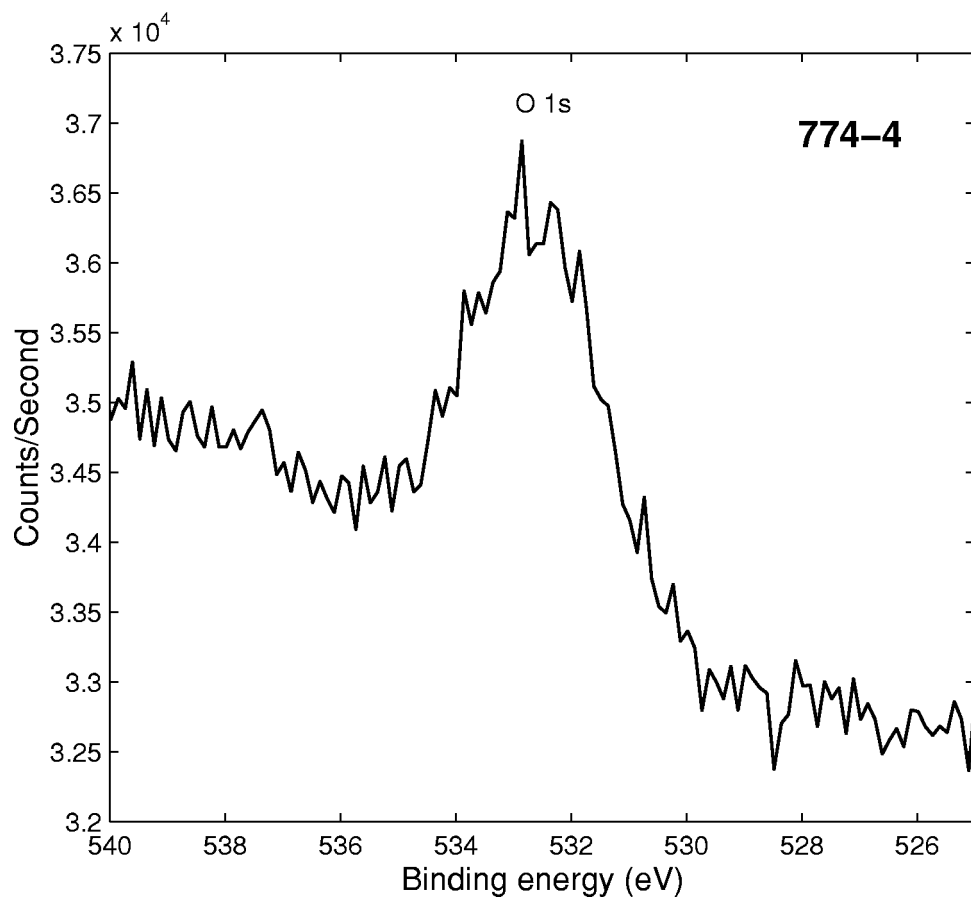
■ **Accession #:** 00774-02
 ■ **Host Material:** Au thin film on silica
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci
 Excitation Source: Mg K_{α}
 Source Energy: 1253.6 eV
 Source Strength: 400 W
 Source Size: >25 mm \times >25 mm
 Incident Angle: 9°
 Analyzer Type: spherical sector
 Analyzer Pass Energy: 58.70 eV
 Analyzer Resolution: 0.6 eV
 Emission Angle: 45°
 Total Signal Accumulation Time: 86.4 s
 Total Elapsed Time: 95.0 s
 Number of Scans: 18
 Effective Detector Width: 0.6 eV
 Comment: See footnote below the Spectral Features Table.



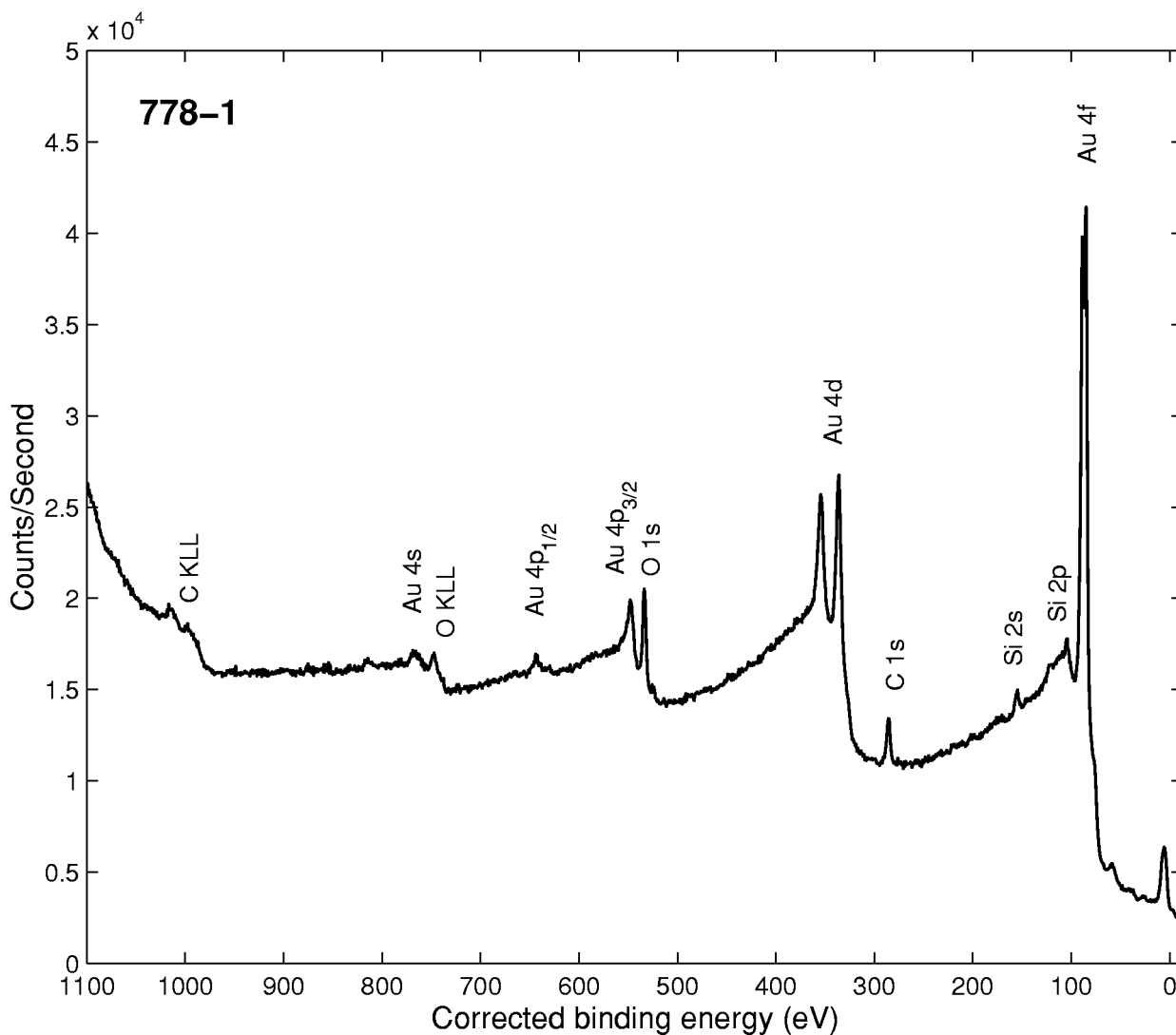
■ **Accession #:** 00774-03
 ■ **Host Material:** Au thin film on silica
 ■ **Technique:** XPS
 ■ **Spectral Region:** Au 4f

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci
 Excitation Source: Mg K_{α}
 Source Energy: 1253.6 eV
 Source Strength: 400 W
 Source Size: >25 mm \times >25 mm
 Incident Angle: 9°
 Analyzer Type: spherical sector
 Analyzer Pass Energy: 58.70 eV
 Analyzer Resolution: 0.6 eV
 Emission Angle: 45°
 Total Signal Accumulation Time: 43.2 s
 Total Elapsed Time: 47.5 s
 Number of Scans: 9
 Effective Detector Width: 0.6 eV
 Comment: See footnote below the Spectral Features Table.



■ **Accession #:** 00774-04
■ **Host Material:** Au thin film on silica
■ **Technique:** XPS
■ **Spectral Region:** O 1s

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci
Excitation Source: Mg K_{α}
Source Energy: 1253.6 eV
Source Strength: 400 W
Source Size: >25 mm \times >25 mm
Incident Angle: 9°
Analyzer Type: spherical sector
Analyzer Pass Energy: 58.70 eV
Analyzer Resolution: 0.6 eV
Emission Angle: 45°
Total Signal Accumulation Time: 100.8 s
Total Elapsed Time: 110.9 s
Number of Scans: 21
Effective Detector Width: 0.6 eV
Comment: See footnote below the Spectral Features Table.



Accession #

00778-01

Host Material

Au/SiO₂ nanocomposite thin film

Technique

XPS

Spectral Region

survey

Instrument

Perkin-Elmer Physical Electronics, Inc. 5600ci

Excitation Source

Mg K_α

Source Energy

1253.6 eV

Source Strength

400 W

Source Size

>25 mm × >25 mm

Analyzer Type

spherical sector

Incident Angle

9°

Emission Angle

45°

Analyzer Pass Energy

187.85 eV

Analyzer Resolution

1.9 eV

Total Signal Accumulation Time

150 s

Total Elapsed Time

165 s

Number of Scans

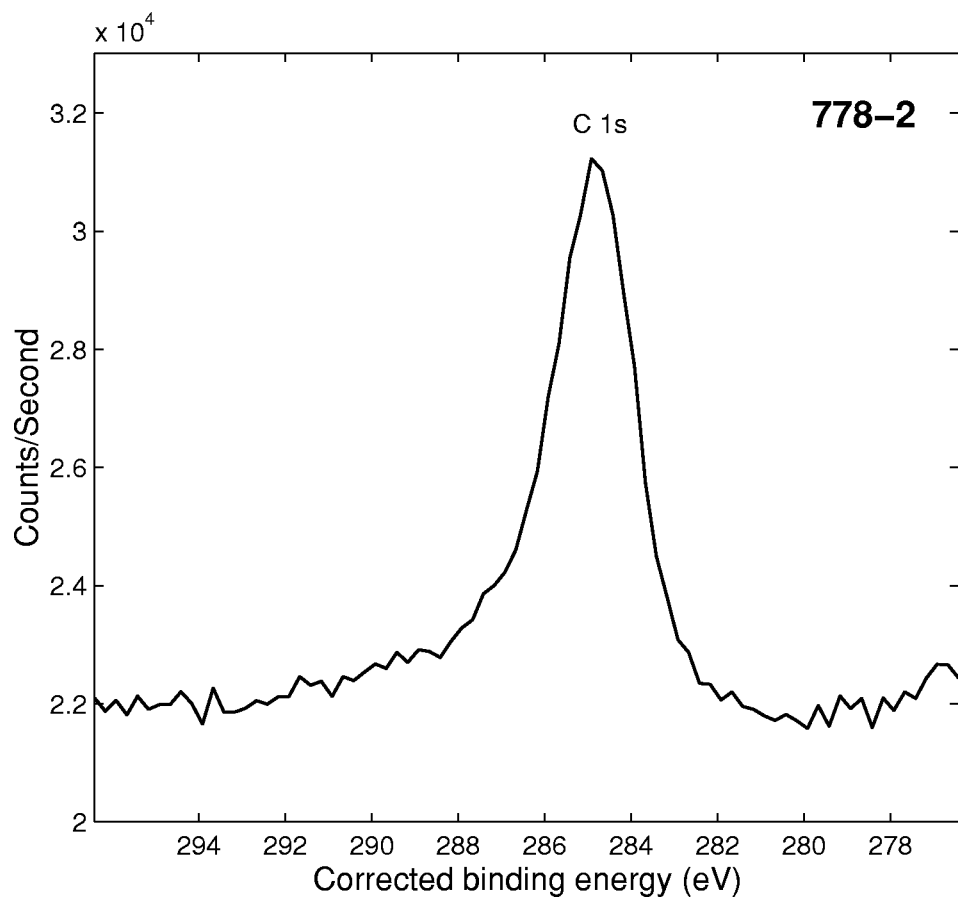
5

Effective Detector Width

1.9 eV

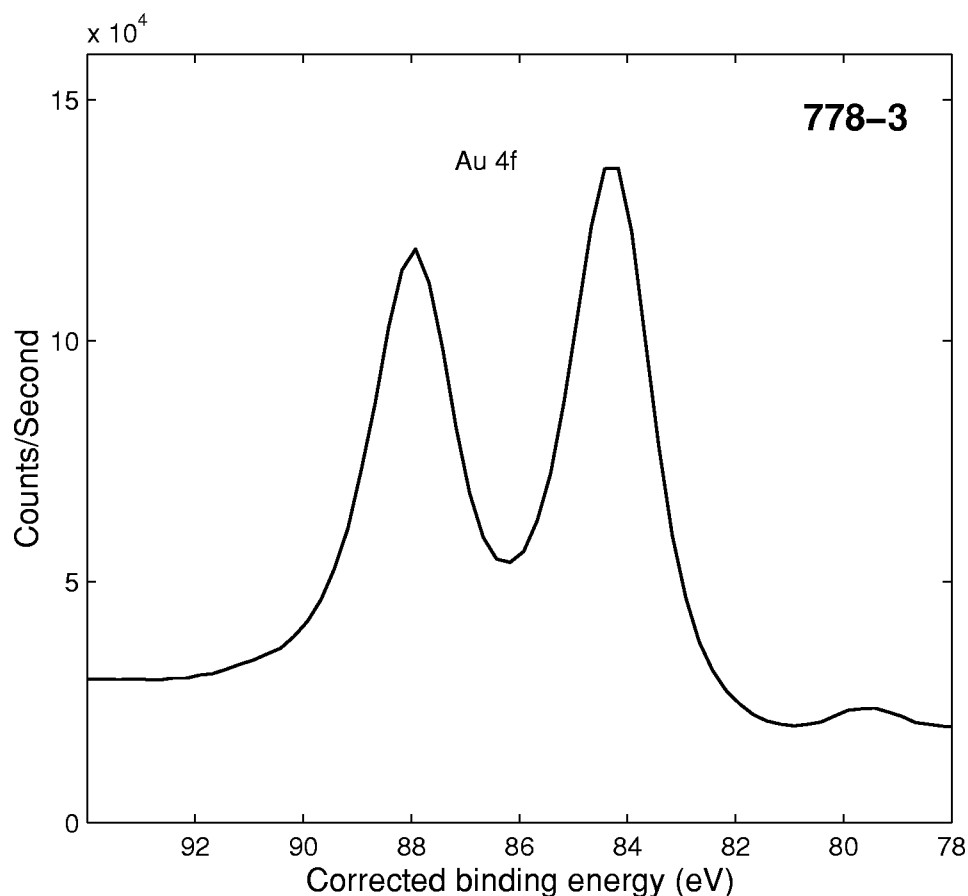
Comment

The presence of Si signals is in agreement with the formation of a discontinuous system.



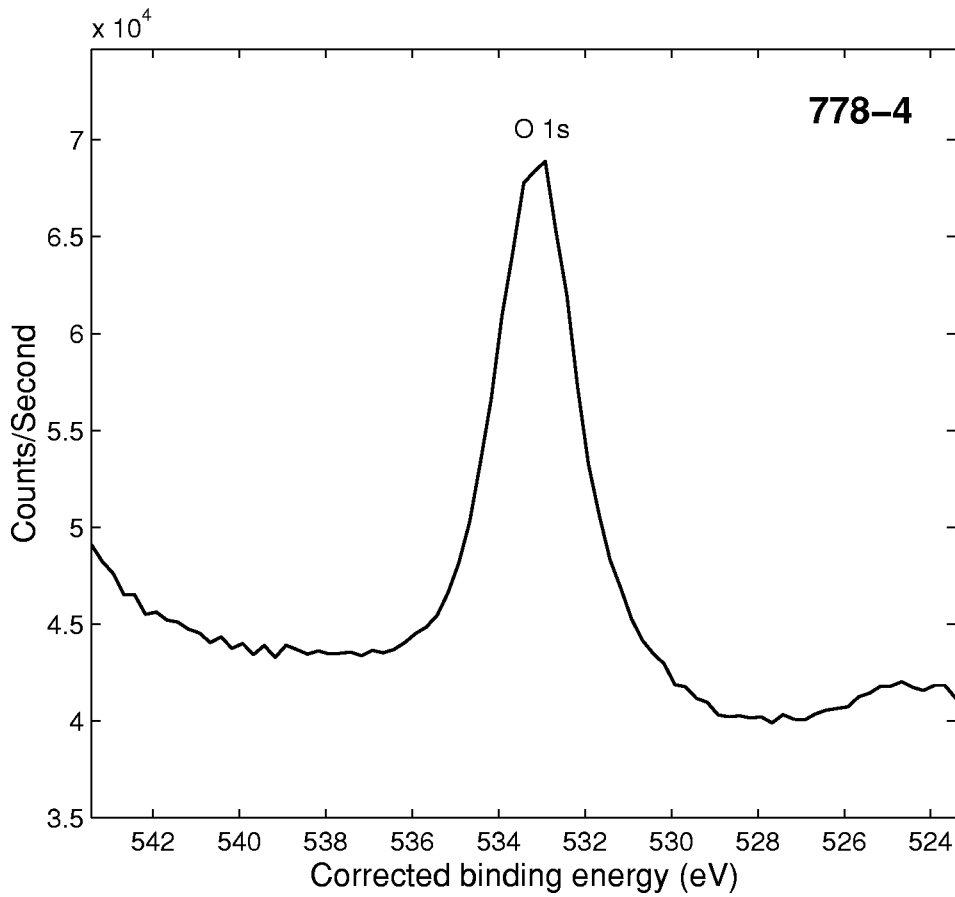
- **Accession #:** 00778-02
- **Host Material:** Au/SiO₂ nanocomposite thin film
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci
 Excitation Source: Mg K_α
 Source Energy: 1253.6 eV
 Source Strength: 400 W
 Source Size: >25 mm × >25 mm
 Incident Angle: 9°
 Analyzer Type: spherical sector
 Analyzer Pass Energy: 58.70 eV
 Analyzer Resolution: 0.6 eV
 Emission Angle: 45°
 Total Signal Accumulation Time: 64.8 s
 Total Elapsed Time: 71.3 s
 Number of Scans: 27
 Effective Detector Width: 0.6 eV
 Comment: See footnote below the Spectral Features Table.



- **Accession #:** 00778-03
- **Host Material:** Au/SiO₂ nanocomposite thin film
- **Technique:** XPS
- **Spectral Region:** Au 4f

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci
 Excitation Source: Mg K_α
 Source Energy: 1253.6 eV
 Source Strength: 400 W
 Source Size: >25 mm × >25 mm
 Incident Angle: 9°
 Analyzer Type: spherical sector
 Analyzer Pass Energy: 58.70 eV
 Analyzer Resolution: 0.6 eV
 Emission Angle: 45°
 Total Signal Accumulation Time: 64.8 s
 Total Elapsed Time: 71.3 s
 Number of Scans: 27
 Effective Detector Width: 0.6 eV
 Comment: See footnote below the Spectral Features Table.



■ **Accession #:** 00778-04

■ **Host Material:** Au/SiO₂ nanocomposite thin film

■ **Technique:** XPS

■ **Spectral Region:** O 1s

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 400 W

Source Size: >25 mm × >25 mm

Incident Angle: 9°

Analyzer Type: spherical sector

Analyzer Pass Energy: 58.70 eV

Analyzer Resolution: 0.6 eV

Emission Angle: 45°

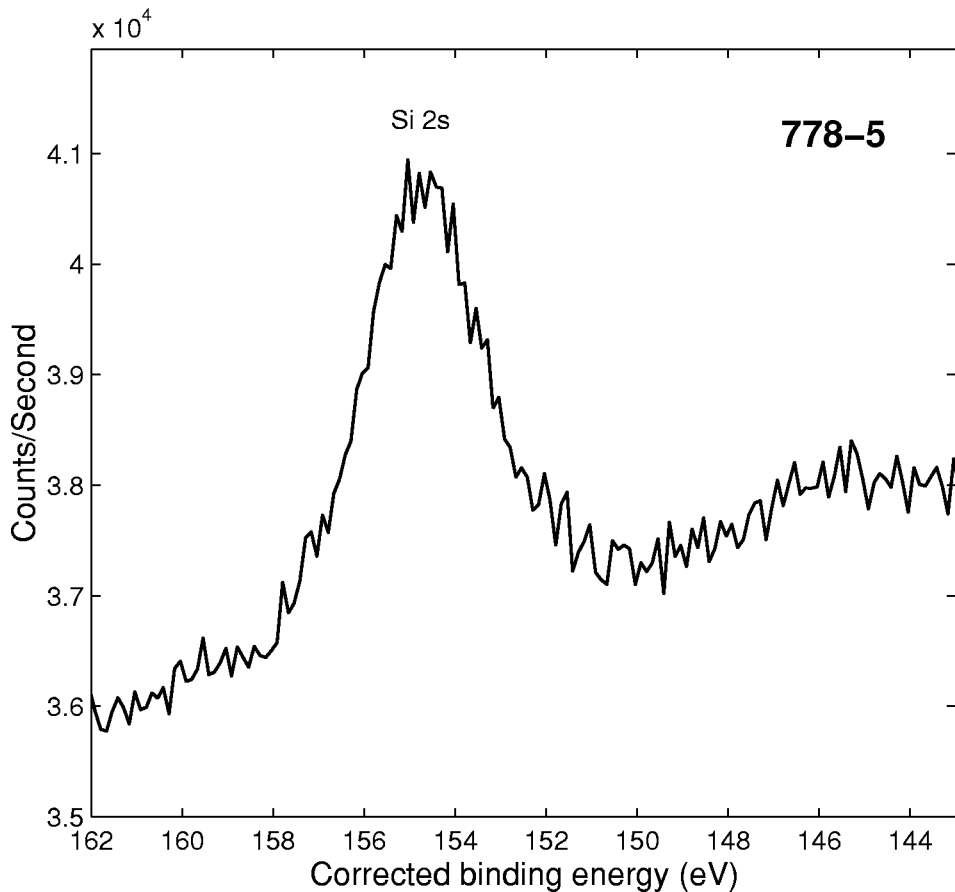
Total Signal Accumulation Time: 86.4 s

Total Elapsed Time: 95.0 s

Number of Scans: 36

Effective Detector Width: 0.6 eV

Comment: The O 1s signal was centered at BE ≈ 533.0 eV (FWHM = 2.4 eV), as expected for SiO₂ (Refs. 9, 18–23).



■ **Accession #:** 00778-05

■ **Host Material:** Au/SiO₂ nanocomposite thin film

■ **Technique:** XPS

■ **Spectral Region:** Si 2s

Instrument: Perkin-Elmer Physical Electronics, Inc. 5600ci

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 400 W

Source Size: >25 mm × >25 mm

Incident Angle: 9°

Analyzer Type: spherical sector

Analyzer Pass Energy: 58.70 eV

Analyzer Resolution: 0.6 eV

Emission Angle: 45°

Total Signal Accumulation Time: 259.2 s

Total Elapsed Time: 285.1 s

Number of Scans: 36

Effective Detector Width: 0.6 eV

Comment: See footnote below the Spectral Features Table.