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Gas Phase Deposition of Trichloro(1H,1H,2H,2H-perfluorooctyl)silane on Silicon Dioxide, by XPS

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Accession #: 01199

Technique: XPS

Host Material: poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on plasma-cleaned silicon

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Major Elements in Spectra: C, O, Si, F

Minor Elements in Spectra: P

Published Spectra: 5

Spectra in Electronic Record: 5

Spectral Category: technical

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Monolayers of trichloro(lH,1H,2H,2H-perfluorooctyl)silane, $Cl_3SiCH_2CH_2(CF_2)_5CF_3$, were deposited via chemical vapor deposition onto the native oxide layer on silicon after plasma-cleaning. The samples have high hydrophobicity, and provide a valuable comparison to perfluorinated alkyl silane layers obtained by liquid deposition. Gas-phase deposition of perfluorinated alkyl silanes is a useful means for reducing stiction in micro- and nano-electromechanical systems, which have narrow spaces that can trap bubbles and prevent liquid-based silane passivation. © *2012 American Vacuum Society*.
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Keywords: *perfluorinated silane; gas-phase; native silicon dioxide*

SPECIMEN DESCRIPTION (ACCESSION #01199)

Host Material: poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on plasma-cleaned silicon

CAS Registry #: 78560-45-9

Host Material Characteristics: homogeneous; solid; organic compound

Chemical Name: silicon oxide (SiO_2)

Source: Moctco Silicon Technologies, Inc.

Host Composition: monolayer on plasma-cleaned native oxide on silicon

Form: monolayer

Lot #: W9969

Structure: Si (1 0 0)

History & Significance: Silanes are often used to modify the surface free energy and/or add a functional group to silicon/silicon dioxide and/or glass surfaces. Perfluorinated alkyl silanes are desirable because of their hydrophobic natures and favorable tribological properties. The hydrophobicity of the resulting monolayers provides a protective coating that can effectively exclude water and oil. The reduction of static friction on silicon surfaces is especially important in the development of micro- and nano-electromechanical systems (MEMs and NEMs) since stiction has been a leading cause of device failure. Gas phase deposition provides a method for better coverage for critical areas of these devices.

Silicon wafers with a native oxide layer were cleaned with a 6-min exposure to a 500 W oxygen plasma in a YES-1224P (Yield Engineering Systems; Livermore, CA, USA) chemical vapor deposition system. After cleaning and without venting, trichloro (1H,1H,2H,2H-perfluorooctyl)silane was injected. The processing temperature was 100 °C. After cleaning, and without venting, the oven was heated to 100 °C and trichloro(1H,1H,2H, 2H-perfluorooctyl)silane was injected. After ten minutes of exposure the oven was evacuated, cooled to room temperature, and opened. The deposition was confirmed by measuring the static water contact angle of one sample, which was 110°.

As Received Condition: silicon wafer (DIA 125)

Analyzed Region: three different spots on each sample

***Ex Situ* Preparation/Mounting:** standard Kratos sample holder

***In Situ* Preparation:** none

Pre-Analysis Beam Exposure: total irradiation time per spot <22 min

Charge Control: none

Temp. During Analysis: 298 K

Pressure During Analysis: $<6.65 \times 10^{-7}$ Pa

INSTRUMENT DESCRIPTION

Manufacturer and Model: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Analyzer Type: Other

Detector: multichannel plates and delay line detector

Number of Detector Elements: 128

Analyzer Description: hemispherical mirror analyser, magnetic snorkel lens

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INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: constant pass energy

Throughput ($T = E^N$): N = 0

Excitation Source Window: none

Excitation Source: Al K_α, monochromatic

Source Energy: 1486.69 eV

Source Strength: 225 W

Source Beam Size: 800 μm × 800 μm

Signal Mode: multichannel direct

Geometry

Incident Angle: 45°

Source to Analyzer Angle: 45°

Emission Angle: 0°

Specimen Azimuthal Angle: 0°

Acceptance Angle from Analyzer Axis: 0°

Comments:

DATA ANALYSIS METHOD

Peak Shape and Background Method: Shirley background function; in the case where the Shirley function was undefined, a linear background was used.

Quantitation Method: Sensitivity factors were obtained from software supplied by Kratos. The areas are the areas above the background.

ACKNOWLEDGMENTS

The authors thank Loren Rieth for his assistance in operating the Kratos instrument and in accurately reporting the instrument's characteristics.

REFERENCES

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2. Z. Tao and B. Bhushan, Langmuir **21**, 2391 (2005).
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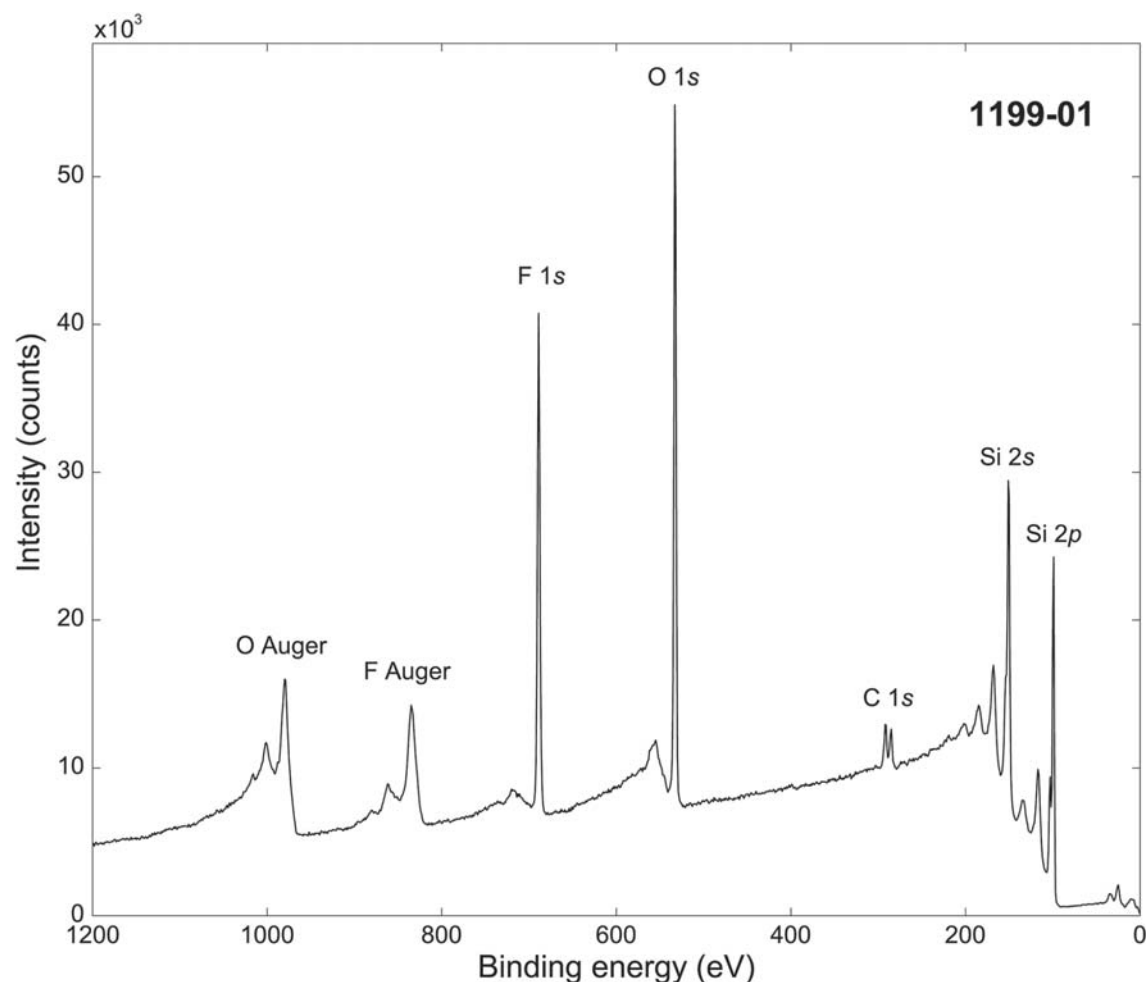
SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV-cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01199-02	C 1s	292.1	1.423	2451	0.278	6.2	$C\text{-}F_2$
01199-02	C 1s	285.4	1.223	1708	0.278	4.3	C-C, C-H
01199-02	C 1s	294.5	0.892	530	0.278	1.3	$C\text{-}F_3$
01199-02	C 1s	286.9	1.494	1069	0.278	2.7	C-O
01199-03	O 1s	533.2	1.506	42871	0.780	34.7	...
01199-04	Si 2p	99.5	0.402	4821	0.328	11.7	Si wafer
01199-04	Si 2p	100.1	0.563	3878	0.328	9.4	Si split
01199-04	Si 2p	103.7	1.663	3335	0.328	8.1	SiO_2
01199-04	Si 2p	101.9	1.575	1303	0.328	3.1	other Si
01199-05	F 1s	689.6	1.750	26982	1.000	15.8	F-C
01199-05	F 1s	687.4	2.373	4634	1.000	2.7	F-O

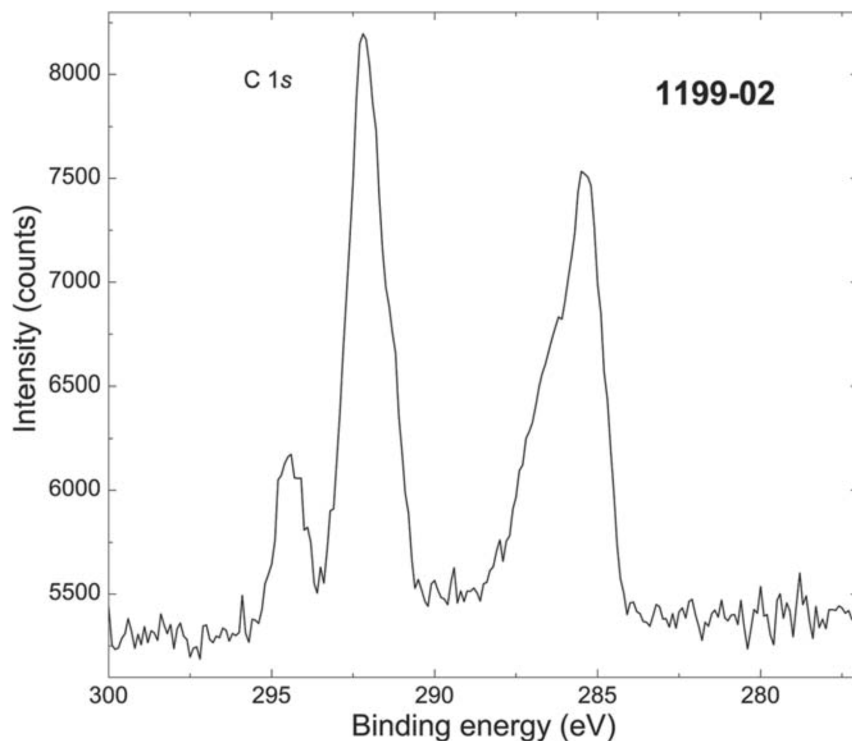
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
1	survey	0	1	0	
2	C 1s	0	1	0	
3	O 1s	0	1	0	
4	Si 2p	0	1	0	
5	F 1s	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.



Accession#	01199–01
Host Material	poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on plasma-cleaned silicon
Technique	XPS
Spectral Region	survey
Instrument	Kratos Analytical, Manchester, U.K. Axis UltraDLD
Excitation Source	Al K_α monochromatic
Source Energy	1486.69 eV
Source Strength	225 W
Source Size	0.8 mm $\times$ 0.8 mm
Analyzer Type	hemispherical mirror analyser, magnetic snorkel lens
Incident Angle	45°
Emission Angle	0°
Analyzer Pass Energy	150 eV
Analyzer Resolution	1.5 eV
Total Signal Accumulation Time	240 s
Total Elapsed Time	440 s
Number of Scans	1
Effective Detector Width	1200 eV



- **Accession #:** 01199–02
- **Host Material:** poly-(1H,1H,2H,2H-perfluorooctyl) siloxane monolayer on plasma-cleaned silicon
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Excitation Source: Al K_α monochromatic

Source Energy: 1486.69 eV

Source Strength: 225 W

Source Size: 0.8 mm × 0.8 mm

Analyzer Type: hemispherical mirror analyser, magnetic snorkel lens

Incident Angle: 45°

Emission Angle: 0°

Analyzer Pass Energy: 50 eV

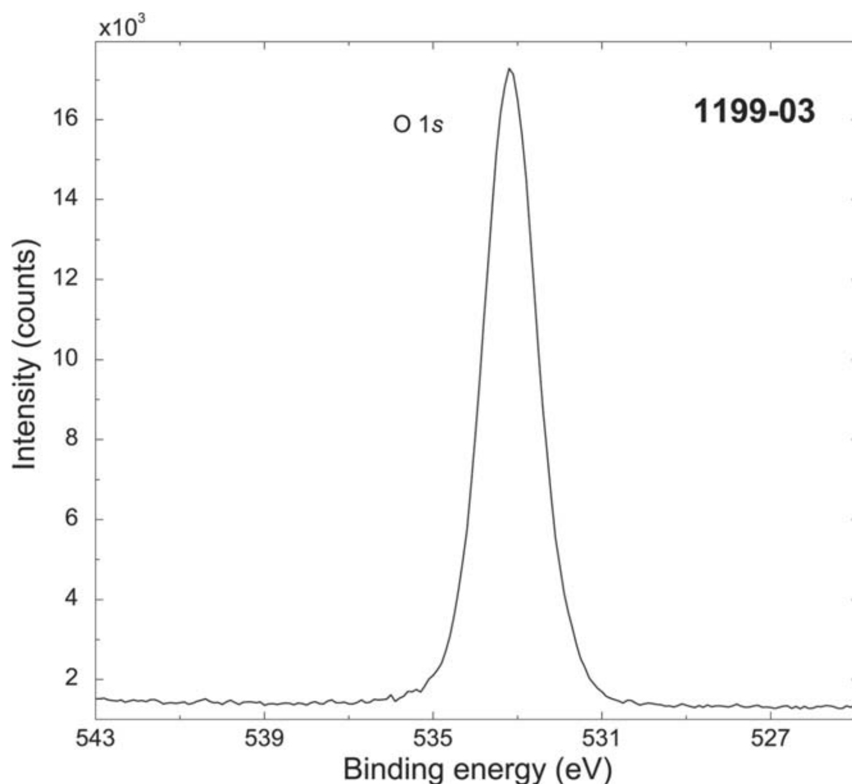
Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 139 s

Total Elapsed Time: 339 s

Number of Scans: 2

Effective Detector Width: 19.70 eV



- **Accession #:** 01199–03
- **Host Material:** poly-(1H,1H,2H,2H-perfluorooctyl) siloxane monolayer on plasma-cleaned silicon
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Excitation Source: Al K_α monochromatic

Source Energy: 1486.69 eV

Source Strength: 225 W

Source Size: 0.8 mm × 0.8 mm

Analyzer Type: hemispherical mirror analyser, magnetic snorkel lens

Incident Angle: 45°

Emission Angle: 0°

Analyzer Pass Energy: 50 eV

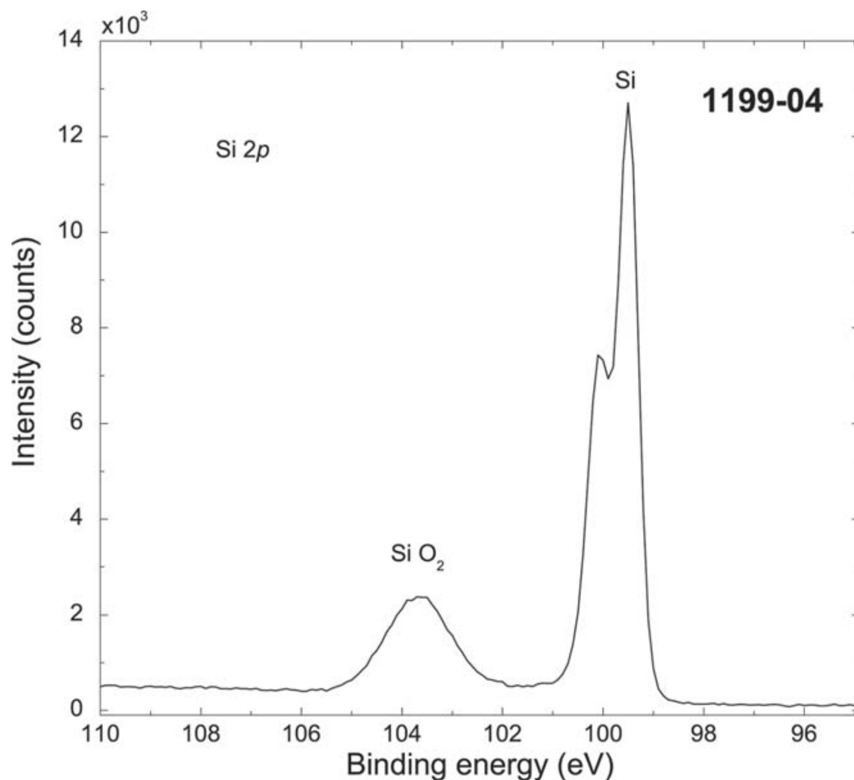
Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 109 s

Total Elapsed Time: 309 s

Number of Scans: 2

Effective Detector Width: 19.70 eV



- **Accession #:** 01199–04
- **Host Material:** poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on plasma-cleaned silicon
- **Technique:** XPS
- **Spectral Region:** Si 2*p*

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Excitation Source: Al K_α monochromatic

Source Energy: 1486.69 eV

Source Strength: 225 W

Source Size: 0.8 mm × 0.8 mm

Analyzer Type: hemispherical mirror analyser, magnetic snorkel lens

Incident Angle: 45°

Emission Angle: 0°

Analyzer Pass Energy: 50 eV

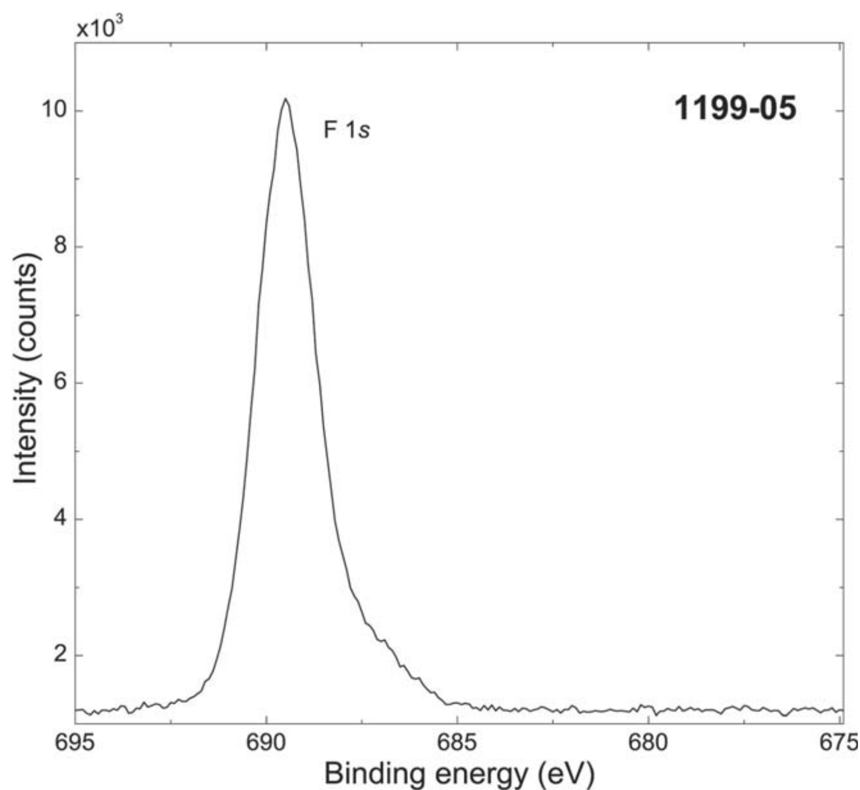
Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 91 s

Total Elapsed Time: 291 s

Number of Scans: 2

Effective Detector Width: 19.70 eV



- **Accession #:** 01199–05
- **Host Material:** poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on plasma-cleaned silicon
- **Technique:** XPS
- **Spectral Region:** F 1*s*

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD

Excitation Source: Al K_α monochromatic

Source Energy: 1486.69 eV

Source Strength: 225 W

Source Size: 0.8 mm × 0.8 mm

Analyzer Type: hemispherical mirror analyser, magnetic snorkel lens

Incident Angle: 45°

Emission Angle: 0°

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 121 s

Total Elapsed Time: 321 s

Number of Scans: 2

Effective Detector Width: 19.70 eV