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

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Monolayers of trichloro(1H,1H,2H,2H-perfluorooctyl)silane, Cl₃SiCH₂CH₂(CF₂)₅CF₃, 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. [DOI: 10.1116/11.20071103]

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: homogenous; solid; organic compound

Chemical Name: silicon oxide (SiO₂)

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)

Ex Situ Preparation/Mounting: standard Kratos sample holder

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

Temp. During Analysis: 298 K

Pressure During Analysis: < 6.65 × 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

a) Author to whom correspondence should be addressed.
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 \( K_a \), monochromatic
Source Energy: 1486.69 eV
Source Strength: 225 W
Source Beam Size: 800 \( \mu \)m \( \times \) 800 \( \mu \)m
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

### SPECTRAL FEATURES TABLE

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<th>Element/Transition</th>
<th>Peak Energy (eV)</th>
<th>Peak Width FWHM (eV)</th>
<th>Peak Area (eV-cts/s)</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
<th>Peak Assignment</th>
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* 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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<td>poly-(1H,1H,2H,2H-perfluorooctyl) siloxane monolayer on plasma-cleaned silicon</td>
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<td>Technique</td>
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<td>Spectral Region</td>
<td>survey</td>
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<td>Instrument</td>
<td>Kratos Analytical, Manchester, U.K. Axis UltraDLD</td>
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<td>Al Kα monochromatic</td>
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<td>Source Energy</td>
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<td>Source Strength</td>
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<td>Source Size</td>
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<td>Analyzer Type</td>
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<tr>
<td>Incident Angle</td>
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<tr>
<td>Emission Angle</td>
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<tr>
<td>Analyzer Pass Energy</td>
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<td>Analyzer Resolution</td>
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Host Material: poly-(1H,1H,2H,2H-perfluorooctyl) siloxane monolayer on plasma-cleaned silicon
Technique: XPS
Spectral Region: C 1s

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD
Excitation Source: Al Kα 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 1s

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD
Excitation Source: Al Kα 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 2p

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD
Excitation Source: Al Kα, monochromatic
Source Energy: 1486.69 eV
Source Strength: 225 W
Source Size: 0.8 mm x 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 1s

Instrument: Kratos Analytical, Manchester, U.K. Axis UltraDLD
Excitation Source: Al Kα, monochromatic
Source Energy: 1486.69 eV
Source Strength: 225 W
Source Size: 0.8 mm x 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

92 Surface Science Spectra, Vol. 17, 2010 Poly-(1H,1H,2H,2H-perfluorooctyl)siloxane monolayer on SiO₂, by XPS