High Resolution XPS Study of a Thin CoO(111) Film Grown on Co(0001)

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Transition metal oxides are often used as the active components in heterogenous catalysis. Therefore the investigation of single crystal oxides as model systems is important to understand the reaction mechanisms on a microscopic level. The reactivity of a ~111 surface of ionic rocksalt type structures seems to be rather high as has been established for NiO~111%. The ideal ~111 surface is polar, and thus unstable, which means that stabilization mechanisms must exist. Here, high resolution XPS measurements of a thin epitaxial CoO~111 film grown on Co~0001% by exposing the surface to ~10000 L O₂ are reported. © 1998 American Vacuum Society.

Keywords: x-ray photoelectron spectroscopy; oxidation; cobalt oxide

PACS: 79.60.Dp, 82.65.Jv, 82.80.Pv

SPECIMEN DESCRIPTION (Accession #00298)

Host Material: Co(0001) metal
Host Material Characteristics: homogeneous; solid; single crystal; conductor; metal
Chemical Name: cobalt
Host Composition: Co
Form: single crystal
Structure: (0001) hexagonal close packed
As Received Condition: single crystal as grown
Analyzed Region: (0001) surface
Ex Situ Preparation/Mounting: single crystal cut in (0001) direction and polished, then spot-welded on the sample holder
In Situ Preparation: ion sputter cleaning and annealing
Charge Control: no charge control necessary as host material is metallic
Temp. During Analysis: 300 K
Pressure During Analysis: <1x10⁻⁷ Pa

SPECIMEN DESCRIPTION (Accession #00299)

Host Material: CoO(111) on Co
Host Material Characteristics: homogeneous; solid; single crystal; dielectric; inorganic compound; thin film
Chemical Name: cobalt oxide
Host Composition: Co, O
Form: thin film
Structure: CoO(111)

History & Significance: The analyzed sample was grown by oxidation of Co(0001) for 3 h with a background pressure of 10⁻⁶ mbar. During this time the sample was slowly heated up to 450 K. After this it was annealed for 1 h without oxygen background pressure (Ref. 1).

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Instrument: Leybold-Heraeus EA 11
Analyzer Type: spherical sector
Detector: 2 multichannel plates

Spectrometer
Analyzer Mode: constant pass energy
Throughput ($T=E^N$): $N=-1$
Excitation Source Window: 1.5 μm Al window
Excitation Source: Al Kα monochromatic
Source Energy: 1486.6 eV
Source Strength: 450 W
Analyzer Width: 3000 μm x 3000 μm
Signal Mode: multichannel direct

Geometry
Incident Angle: 45°
Source to Analyzer Angle: 45°
Emission Angle: 0°
Azimuthal Angle: 0°
Acceptance Angle from Analyzer Axis: 1.5°
Analyzer Angular Acceptance Width: 3° × 3°

**Ion Gun**
Manufacturer and Model: Leybold-Heraeus IQE 12/38
Energy: 500 eV
Current: 10 μA
Current Measurement Method: biased stage
Sputtering Species: Ne
Raster Size: 10000 μm × 10000 μm
Incident Angle: 45°
Polar Angle: 45°

**DATA ANALYSIS METHOD**

Energy Scale Correction: energy scale calibration to Fermi level of Co metal

**ACKNOWLEDGMENTS**

Several agencies have supported our work: Deutsche Forschungsgemeinschaft, Ministerium für Wissenschaft und Forschung des Landes NRW, and Fonds der Chemischen Industrie.

**REFERENCES**


### SPECTRAL FEATURES TABLE

<table>
<thead>
<tr>
<th>Spectrum ID #</th>
<th>Element/Transition</th>
<th>Peak Energy (eV)</th>
<th>Peak Width (FWHM) (eV)</th>
<th>Peak Area</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
<th>Peak Assignment</th>
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</thead>
<tbody>
<tr>
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<td>O 1s</td>
<td>530.0</td>
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<td>00299-04</td>
<td>Co 3s</td>
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### ANALYZER CALIBRATION TABLE

<table>
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<th>Peak Energy (eV)</th>
<th>Peak Width (FWHM) (eV)</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
<th>Peak Assignment</th>
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Comment to Analyzer Calibration Table: The energy scale was calibrated by determining the Fermi edge of the Co(0001) substrate.

### GUIDE TO FIGURES

<table>
<thead>
<tr>
<th>Spectrum (Accession) #</th>
<th>Sample Voltage*</th>
<th>Multiplier</th>
<th>Baseline</th>
<th>Comment #</th>
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* Sample voltage due to charging unless otherwise noted.
1. Peak energies were referenced to Fermi level of Co metal substrate which could be measured through thin oxide (see 299-5).
<table>
<thead>
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<th>Accession #</th>
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</thead>
<tbody>
<tr>
<td>Host Material</td>
<td>CoO(111) on Co</td>
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<tr>
<td>Technique</td>
<td>XPS survey</td>
</tr>
<tr>
<td>Spectral Region</td>
<td>Leybold-Heraeus EA 11</td>
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<tr>
<td>Instrument</td>
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<tr>
<td>Source Energy</td>
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<td>Emission Angle</td>
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<td>Analyzer Retard Ratio</td>
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<tr>
<td>Analyzer Resolution</td>
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<td>Comment</td>
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**Accession #: 00299-02**  
**Host Material:** CoO(111) on Co  
**Technique:** XPS  
**Spectral Region:** O 1s  
Instrument: Leybold-Heraeus EA 11  
Excitation Source: Al Kα monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 450 W  
Source Size: not specified  
Incident Angle: 45°  
Analyzer Type: spherical sector  
Analyzer Pass Energy: 25.2 eV  
Analyzer Resolution: 0.3 eV  
Emission Angle: 0°  
Total Signal Accumulation Time: not specified  
Total Elapsed Time: 900 s  
Number of Scans: 50  
Comment: oxidized Co single crystal

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**Accession #: 00299-03**  
**Host Material:** CoO(111) on Co  
**Technique:** XPS  
**Spectral Region:** Co 2p 1/2; Co 2p 3/2  
Instrument: Leybold-Heraeus EA 11  
Excitation Source: Al Kα monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 450 W  
Source Size: not specified  
Incident Angle: 45°  
Analyzer Type: spherical sector  
Analyzer Pass Energy: 25.2 eV  
Analyzer Resolution: 0.3 eV  
Emission Angle: 0°  
Total Signal Accumulation Time: not specified  
Total Elapsed Time: 2150 s  
Number of Scans: 50  
Comment: oxidized Co single crystal
Accession #: 00299-04
Host Material: CoO(111) on Co
Technique: XPS
Spectral Region: survey
Instrument: Leybold-Heraeus EA 11
Excitation Source: Al K\(_\alpha\) monochromatic
Source Energy: 1486.6 eV
Source Strength: 450 W
Source Size: not specified
Analyzer type: spherical sector
incident Angle: 45°
Emission Angle: 0°
Analyzer Retard Ratio: 4
Analyzer Resolution: 0.125 eV
Total Signal Accumulation Time: not specified
Total Elapsed Time: 330 s
Number of Scans: 5
Comment: survey of the oxidized Co single crystal

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Accession #: 00299-05
Host Material: CoO(111) on Co
Technique: XPS
Spectral Region: Co valence band; O valence band
Instrument: Leybold-Heraeus EA 11
Excitation Source: Al K\(_\alpha\) monochromatic
Source Energy: 1486.6 eV
Source Strength: 450 W
Source Size: not specified
Incident Angle: 45°
Analyzer Type: spherical sector
Analyzer Pass Energy: 25.2 eV
Analyzer Resolution: 0.3 eV
Emission Angle: 0°
Total Signal Accumulation Time: not specified
Total Elapsed Time: 4400 s
Number of Scans: 200
Comment: oxidized Co single crystal