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We present the XPS survey and detailed element spectra of a copper-carbon nanotube composite synthesized in our laboratories. We compare the copper spectrum to a reference spectrum of copper(II) acetate measured under the same conditions to ascertain the chemical composition of the copper in the composite.

**Keywords:** Copper, XPS, Multi-walled carbon nanotubes

**INTRODUCTION**

The development of copper-carbon nanotube composites is of interest for application in ultra-conductive copper wire\(^1\). Ultra-conductive copper wire consists of a copper species and a nanocarbon, such as graphene or carbon nanotubes\(^2\). The copper-carbon composites are also of interest as electrocatalysts for the electrocatalytic reduction of carbon dioxide to higher value products such as methane and ethylene\(^3\). A number of synthetic procedures are available and have been used for manufacturing such composites\(^4\)-\(^7\). Depending on the procedure the copper can be chemically bound to the carbon nanotubes or just associated with the carbon nanotubes. The composites can be characterized using solid-state techniques such as scanning electron microscopy, x-ray diffraction and x-ray photoelectron spectroscopy (XPS). For the analysis of copper-carbon nanotube composites it is particularly important to determine the copper oxidation state. In addition, the modelling of the carbon 1s signal is non-trivial as it consists of sp\(^2\), sp\(^3\), adventitious and oxidized carbon, along with plasmon signals. We have characterized a commercial sample of copper(II) acetate as a reference sample.\(^8\) We have also measured the XPS spectra of a synthesized copper-carbon composite\(^9\). We have ascertained the chemical composition and oxidation state of the copper in the composite using our reference spectra.\(^9\) Following literature precedent we have modelled the carbon 1s signal using a mixed Donjiach-Sunjik Sum Gaussian Lorentzian (30) fitting\(^10\). We have found this model to be particularly effective. In this study, particular importance is given to the analysis of the C 1s and Cu 2p signals.

**Host Material:** CuAc

- **CAS Registry #:** 142-71-2
- **Host Material Characteristics:** homogeneous; solid; polycrystalline; conductor; composite; Other
- **Chemical Name:** Copper(II) acetate
- **Source:** Sigma-Aldrich
- **Host Composition:** C, Cu, H, O
- **Form:** Polycrystalline composite
- **Structure:** Cu(CH\(_3\)OO)\(_2\)
- **History & Significance:** Cu(Ac)\(_2\) powder was used as received.
- **As Received Condition:** The as-received sample was a blue crystalline solid.
- **Ex Situ Preparation/Mounting:** The composite was pressed onto carbon tape.
- **In Situ Preparation:** none
- **Charge Control:** Charge neutralizer was used.
- **Temp. During Analysis:** 300K
- **Pressure During Analysis:** 4 x 10\(^{-6}\) Pa
- **Pre-analysis Beam Exposure:** 0 s

**SPECIMEN DESCRIPTION (ACCESSION #00000)**

**Host Material:** CuO/MWCNT

- **CAS Registry #:** unknown
- **Host Material Characteristics:** homogeneous; solid; polycrystalline; conductor; composite; Other
- **Chemical Name:** Copper(II) oxide on multi-walled carbon nanotubes
Source: Cu(II)Ac purchased from Sigma Aldrich and used as received. MWCNTs synthesized at Rice University, Texas, USA and purified before use11.

Host Composition: CuO on MWCNTs

Form: Amorphous composite

Structure: CuOCx

History & Significance: 6 mg of powdered CuAc were added to a vial containing 10 mg of MWCNTs. 6 ml of distilled water was added and the mixture sonicated for 15 minutes in order to disperse the copper particles amongst the MWCNTs. The water was evaporated by drying the sample in the oven at 80 °C for 4 hours. The sample was then heated in the microwave for 3 x 1 minutes at 1000 W to decompose the copper(II) acetate to copper(II) oxide. The product obtained was a black powder.

As Received Condition: The as-prepared sample was a black solid.

Analyzed Region: Same as host material.

Ex Situ Preparation/Mounting: The material was pressed onto indium foil.

In Situ Preparation: none

Charge Control: Charge neutralizer was used

Temp. During Analysis: 300 K

Pressure During Analysis: 4 x 10^-6 Pa

Pre-analysis Beam Exposure: 0 s.

As Received Condition: Pre-analysis Beam Exposure:

INSTRUMENT DESCRIPTION

Manufacturer and Model: Kratos Axis Supra

Analyzer Type: spherical sector

Detector: multichannel resistive plate

Number of Detector Elements: 3 MCP, 128 channel DLD

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput (T=EN): N=0

Excitation Source Window: not specified

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 225 W

Source Beam Size: 700 µm x 300 µm

Signal Mode: multichannel direct

■ Geometry

Incident Angle: 54.7°

Source-to-Analyzer Angle: 54.7°

Emission Angle: 0°

Specimen Azimuthal Angle: N/A°

Acceptance Angle from Analyzer Axis: 0°

Analyzer Angular Acceptance Width: 30° x 30°

■ Ion Gun

Manufacturer and Model: Kratos GCIS Minibeam 6

Energy: 10 k eV

Current: 23 mA

Current Measurement Method: biased stage

Sputtering Species: Argon 1000+ ion clusters

Spot Size (unrastered): 200 µm

Raster Size: 2000 µm x 2000 µm

Incident Angle: 40°

Polar Angle: 0°

Azimuthal Angle: 0°

Comment: Sputtering was carried out on reference samples only.

DATA ANALYSIS METHOD

Energy Scale Correction: The binding energy scale was referenced to C 1s = 248.8 eV

Recommended Energy Scale Shift: Spectra 5-9 have been shifted by 3.146 eV.

Peak Shape and Background Method: A Shirley background was used12. Peak shape was Gaussian Lorentzian product formula GL(30) for all components except the dominant C (1s) peak which was fitted as a mixture of Doniach-Sunjik DS (0.03, 0) and Sum Gaussian Lorentzian SGL (30) following the protocol set by Kallbac et. al.10

Quantitation Method: Quantification was done using component definitions with CasaXPS version 2.3.15. Sensitivity factors supplied by Kratos Analytical.

ACKNOWLEDGMENTS

Dr. James McGettrick is thanked for training JAR on the XPS instrument and for helpful discussions regarding the modelling of peaks. We would also like to thank reviewer 2 for their thorough comments on the paper, which has undoubtedly improved it. JAR and EA acknowledge funding from EPSRC project reference EP/N009525/1, DRJ and CWD acknowledge funding from the Flexis project, which is part-funded by the European Regional Development Fund (ERDF) through the Welsh Government. We would like to thank EPSRC for funding Swansea University AIM Facilities (EP/M028267/1) and the European Regional Development Fund (80708) via Welsh Government.

REFERENCES

A hundred fold increase in current carrying capacity in a carbon nanotube–copper composite. *Nature Communications* 2013, 4, 2202.


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<tr>
<th>Spectrum ID #</th>
<th>Element/Transition</th>
<th>Peak Energy (eV)</th>
<th>Peak Width FWHM (eV)</th>
<th>Peak Area (eV x cts/s)</th>
<th>Sensitivity Factor</th>
<th>Concentration (at. %)</th>
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**ANALYZER CALIBRATION TABLE**

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<th>Concentration (at. %)</th>
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**GUIDE TO FIGURES**

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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.

1. CuAc.
2. CuO on MWCNTs.
3. Ag calibration.
4. Au calibration.
5. Cu calibration.
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Spectrum 2a

Spectrum 2b

Residual standard deviation = 1.15
**Spectrum 3a**

- **Host Material**: CuAc
- **Technique**: XPS
- **Spectral Region**: O 1s
- **Instrument**: Kratos Axis Supra
- **Excitation Source**: Al Ka monochromatic
- **Source Energy**: 1486.6 eV
- **Source Strength**: 225 W
- **Source Size**: 0.7 mm x 0.3 mm
- **Analyzer Type**: spherical sector
- **Incident Angle**: 54.7°
- **Emission Angle**: 0°
- **Analyzer Pass Energy**: 20 eV
- **Analyzer Resolution**: 0.616 eV
- **Total Signal Accumulation Time**: 347 s
- **Total Elapsed Time**: Not specified s
- **Number of Scans**: 1
- **Effective Detector Width**: 2 eV

**Spectrum 3b**

- **Host Material**: CuAc
- **Technique**: XPS
- **Spectral Region**: O 1s
- **Instrument**: Kratos Axis Supra
- **Excitation Source**: Al Ka monochromatic
- **Source Energy**: 1486.6 eV
- **Source Strength**: 225 W
- **Source Size**: 0.7 mm x 0.3 mm
- **Analyzer Type**: spherical sector
- **Incident Angle**: 54.7°
- **Emission Angle**: 0°
- **Analyzer Pass Energy**: 20 eV
- **Analyzer Resolution**: 0.616 eV
- **Total Signal Accumulation Time**: 347 s
- **Total Elapsed Time**: Not specified s
- **Number of Scans**: 1
**Accession #:**

**Host Material:** CuAc

**Technique:** XPS

**Spectral Region:** Cu 2p

Instrument: Kratos Axis Supra

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 225 W

Source Size: 0.7 mm x 0.3 mm

Analyzer Type: spherical sector

Incident Angle: 54.7°

Emission Angle: 0°

Analyzer Pass Energy: 20 eV

Analyzer Resolution: 0.616 eV

Total Signal Accumulation Time: 3758 s

Total Elapsed Time: Not Specified s

Number of Scans: 1

Effective Detector Width: 2 eV
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.616 eV
Total Signal Accumulation Time: 3758 s
Total Elapsed Time: Not specified s
Number of Scans: 1
Effective Detector Width: 2 eV
Spectrum 5

- **Host Material**: CuO on MWCNTs
- **Technique**: XPS
- **Spectral Region**: survey
- **Instrument**: Kratos Axis Supra
- **Excitation Source**: Al Ka monochromatic
- **Source Energy**: 1486.6 eV
- **Source Strength**: 225 W
- **Source Size**: 0.7 mm x 0.3 mm
- **Analyzer Type**: spherical sector analyzer
- **Incident Angle**: 54.7°
- **Emission Angle**: 0°
- **Analyzer Pass Energy**: 160 eV
- **Analyzer Resolution**: 1 eV
- **Total Signal Accumulation Time**: 120 s
- **Total Elapsed Time**: 120 s
- **Number of Scans**: 1
- **Effective Detector Width**: 16 eV
**Spectrum 6a**

- Counts/second vs. Corrected binding energy (eV)

- Peak at approximately 284 eV

**Spectrum 6b**

- Counts/second vs. Corrected binding energy (eV)

- Various peaks indicating C=O, C-OH, C-C/C-H, etc.

- Peak at approximately 284 eV

**Host Material:** CuO on MWCNTs

**Technique:** XPS

**Spectral Region:** C 1s

**Instrument:** Kratos Axis Supra

**Excitation Source:** Al Ka, monochromatic

**Source Energy:** 1486.6 eV

**Source Strength:** 225 W

**Source Size:** 0.7 mm x 0.3 mm

**Analyzer Type:** spherical sector

**Incident Angle:** 54.7˚

**Emission Angle:** 0˚

**Analyzer Pass Energy:** 20 eV

**Analyzer Resolution:** 0.616 eV

**Total Signal Accumulation Time:** 332 s

**Total Elapsed Time:** Not Specified s

**Number of Scans:** 1

**Effective Detector Width:** 2 eV
Accession #:  
Host Material: CuO on MWCNTs  
Technique: XPS  
Spectral Region: O 1s  
Instrument: Kratos Axis Supra  
Excitation Source: Al Ka monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 225 W  
Source Size: 0.7 mm x 0.3 mm  
Analyzer Type: spherical sector  
Incident Angle: 54.7 °  
Emission Angle: 0 °  
Analyzer Pass Energy: 20 eV  
Analyzer Resolution: 0.616 eV  
Total Signal Accumulation Time: 347 s  
Total Elapsed Time: Not Specified s  
Number of Scans: 1  
Effective Detector Width: 2 eV

Residual standard deviation = 0.86

Accession #:  
Host Material: CuO on MWCNTs  
Technique: XPS  
Spectral Region: O 1s  
Instrument: Kratos Axis Supra  
Excitation Source: Al Ka monochromatic  
Source Energy: 1486.6 eV  
Source Strength: 225 W  
Source Size: 0.7 mm x 0.3 mm  
Analyzer Type: spherical sector  
Incident Angle: 54.7 °  
Emission Angle: 0 °  
Analyzer Pass Energy: 20 eV  
Analyzer Resolution: 0.616 eV  
Total Signal Accumulation Time: 347 s  
Total Elapsed Time: Not Specified s  
Number of Scans: 1  
Effective Detector Width: 2 eV
Spectrum 8a

Counts/second

Corrected binding energy (eV)

Spectrum 8b

Counts/second

Corrected binding energy (eV)

Residual standard deviation = 1.03

Satellite

CuO

Satellites

CuO

Publish in SSS: Yes ☒ No ☐

Accession #:

Host Material: CuO on MWCNTs

Technique: XPS

Spectral Region: Cu 2p

Instrument: Kratos Axis Supra

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 225 W

Source Size: 0.7 mm x 0.3 mm

Analyzer Type: spherical sector

Incident Angle: 54.7°

Emission Angle: 0°

Analyzer Pass Energy: 20 eV

Analyzer Resolution: 0.616 eV

Total Signal Accumulation Time: 3758 s

Total Elapsed Time: Not Specified s

Number of Scans: 1

Effective Detector Width: 2 eV
| Accession #: |  |
| Host Material: | CuO on MWCNTs |
| Technique: | XPS |
| Spectral Region: | In 3d |

Instrument: Kratos Axis Supra
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 225 W
Source Size: 0.7 mm x 0.3 mm
Analyzer Type: spherical sector
Incident Angle: 54.7 °
Emission Angle: 0 °
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.616 eV
Total Signal Accumulation Time: 116 s
Total Elapsed Time: Not Specified s
Number of Scans: 1
Effective Detector Width: 2 eV

---

| Accession #: |  |
| Host Material: | CuO on MWCNTs |
| Technique: | XPS |
| Spectral Region: | In 3d |

Instrument: Kratos Axis Supra
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 225 W
Source Size: 0.7 mm x 0.3 mm
Analyzer Type: spherical sector
Incident Angle: 54.7 °
Emission Angle: 0 °
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.616 eV
Total Signal Accumulation Time: 116 s
Total Elapsed Time: Not Specified s
Number of Scans: 1
Effective Detector Width: 2 eV
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<thead>
<tr>
<th>Accession #</th>
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<tbody>
<tr>
<td><strong>Host Material</strong></td>
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<td><strong>Technique</strong></td>
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<td><strong>Spectral Region</strong></td>
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<td><strong>Excitation Source</strong></td>
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<td><strong>Source Strength</strong></td>
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</tr>
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<td><strong>Effective Detector Width</strong></td>
<td>16 eV</td>
</tr>
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</table>
Spectrum 11

Counts/second

Corrected binding energy (eV)

\[ \times 10^4 \]

[Image of a spectrum graph showing a peak between 367 and 368 eV with a y-axis ranging from 0 to 9 counts/second.]

- **Host Material**: Ag
- **Technique**: XPS
- **Spectral Region**: Ag 3d5/2

**Instrument**: Kratos Axis Supra
**Excitation Source**: Al Ka monochromatic
**Source Energy**: 1486.6 eV
**Source Strength**: 60 W
**Source Size**: 0.7 mm x 0.3 mm
**Analyzer Type**: spherical sector
**Incident Angle**: 54.7 °
**Emission Angle**: 0 °
**Analyzer Pass Energy**: 10 eV
**Analyzer Resolution**: 0.498 eV
**Total Signal Accumulation Time**: 84 s
**Total Elapsed Time**: Not specified
**Number of Scans**: 1
**Effective Detector Width**: 1 eV
Accession #

Host Material  
Technique  
Spectral Region  
Instrument  
Excitation Source  
Source Energy  
Source Strength  
Source Size  
Analyzer Type  
Incident Angle  
Emission Angle  
Analyzer Pass Energy  
Analyzer Resolution  
Total Signal Accumulation Time  
Total Elapsed Time  
Number of Scans  
Effective Detector Width  

Au calibration  
XPS  
survey  
Kratos Axis Supra  
Al Ka monochromatic  
1486.6 eV  
15 W  
0.7 mm x 0.3 mm  
spherical sector analyzer  
54.7˚  
0˚  
160 eV  
1 eV  
120 s  
120 s  
1  
16 eV
Spectrum 13

Counts/second

Corrected binding energy (eV)

×10^5

0

1

2

3

83

84

85
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<tr>
<td>Number of Scans</td>
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<tr>
<td>Effective Detector Width</td>
</tr>
</tbody>
</table>
Spectrum 15

Counts/second

Corrected binding energy (eV)

10^5
Accession #:  
Host Material: Au 
calibration 
Technique: XPS 
Spectral Region: Cu LMM 
Instrument: Kratos Axis Supra 
Excitation Source: Al Ka monochromatic 
Source Energy: 1486.6 eV 
Source Strength: 300 W 
Source Size: 0.7 mm x 0.3 mm 
Analyzer Type: spherical sector 
Incident Angle: 54.7 ° 
Emission Angle: 0 ° 
Analyzer Pass Energy 20 eV 
Analyzer Resolution: 0.616 eV 
Total Signal Accumulation Time: 118 s 
Total Elapsed Time: Not specified 
Number of Scans: 1 
Effective Detector Width: 1 eV