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Argon Implanted into Graphite, by XPS

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Argon Implanted into Graphite, by XPS

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Argon ions (Ar^+) were implanted into a substrate of natural graphite (crystal) by 4 kV acceleration for a period of 5 min using a VG EX05 ion gun. (The surface plane of the substrate was perpendicular to the flight path of the argon ions.) No attempt was made to maximize the argon concentration within the graphite. The resulting concentration of argon within the graphite surface was approximately 4.0 at. %. The BE for Ar $2p_3$ is 241.7 eV which is similar to the 241.9 eV in silicon (Si) obtained by Perkin Elmer [see J. F. Moulder, W. F. Stickle, P. E. Sobol, and K. O. Bomben, *Handbook of X-ray Photoelectron Spectroscopy*, 2nd ed. (Perkin-Elmer Corporation, Eden Prairie, MN, 1992), p. 65].

Keywords: argon; Ar; XPS; ion implant; graphite

PACS: 79.60.−i, 82.80.Pv, 61.70.Vn

Accession #: 00063
Technique: XPS
Host Material: natural graphite crystal
Instrument: Fisons Instruments S-Probe E-2703
Major Elements in Spectrum: C, Ar
Minor Elements in Spectrum: N, O
Printed Spectra: 5
Spectra in Electronic Record: 8
Spectral Category: reference

SPECIMEN DESCRIPTION

Host Material: natural graphite crystal

CAS Registry #: 7440-37-1

Host Material Characteristics: inhomogeneous; solid; polycrystalline; conductor; inorganic compound

Chemical Name: graphite

Source: Onozato Sanko Co. supplied 99.999+% argon gas

Host Composition: C, H

Form: natural crystal with bubblelike surface

Structure: argon/graphite

History & Significance: The as received graphite sample had a trace of sulfur which was removed by ion etching. Subsequent to the etching, trace amounts of oxygen and nitrogen were found. The argon gas was of 99.999+% purity.

As Received Condition: An irregular sized piece of natural graphite crystal was implanted with argon ions obtained by accelerating the ions to 4 kV.

Analyzed Region: The analyzed region was at the center of the 4 × 4 mm area that was implanted with argon ions.

***Ex Situ* Preparation/Mounting:** The graphite substrate, held down by a screw and a washer, was mounted and implanted as received, without any cleaning or other *ex situ* treatment.

***In Situ* Preparation:** While the graphite substrate was in the analysis chamber, the argon ions were implanted during a 5 min period into a 4 × 4 mm area at a 90° angle of incidence by using a 4 kV acceleration potential, and 1.5×10^{-7} Torr total pressure within the analysis chamber.

Charge Control: none

Temp. During Analysis: 300 K

Pressure During Analysis: $<1\times10^{-6}$ Pa

SPECTROMETER DESCRIPTION

Manufacturer and Model: Fisons Instruments S-Probe E-2703

Analyzer Type: spherical sector

Detector: 128 channel

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput ($T=E^N$): The instrument throughput function (ITF) depends on pass energy and the lens voltages. For a 150 eV pass energy the ITF is $E^{1.4}$ while the 50 eV pass energy has an ITF of $E^{0.8}$.

Excitation Source: Al K_α monochromatic

Excitation Source Window: 0.2 μm Al

Source Energy: 1486.7 eV

Source Strength: 200 W

Source Beam Size: 250 μm × 700 μm

Analyzer Width at 1000 eV: 2000 μm × 2000 μm

Signal Mode: multichannel direct

Simultaneous Channels: 128

■ Geometry

Incident Angle: 20°

Source to Analyzer Angle: 71°

Emission Angle: 0°

Specimen Azimuthal Angle: 90°

Acceptance Angle from Analyzer Axis: 0°

Analyzer Angular Acceptance Width: 30° × 30°

Comments: Sampling depth was at its maximum which provides subsurface bulk information.

■ Ion Gun

Manufacturer and Model: Vacuum Generators EX-05

Energy: 4000 eV

Current: 5 μA

Current Measurement Method: biased stage

Sputtering Species: Ar^+

Spot Size (unrastered): 500 μm

Raster Size: 4000 μm × 4000 μm

Incident Angle: 0°

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Polar Angle: 71°

Azimuthal Angle: 180°

DATA ANALYSIS METHOD

Peak Shape and Background Method: Background counts were removed from each channel by subtracting the counts found in the lowest intensity channel. A Shirley-type background-baseline function and a Voight peak shape function were used to fit the high resolution data.

Quantitation Method: Relative sensitivity factors, which were used to estimate the atomic percentage of each element, were calculated by modifying the photoionization cross sections calculated by J. H. Scofield (Ref. 1) in accordance with "standard" algorithms supplied by the instrument manufacturer.

REFERENCES

1. J. H. Scofield, J. Electron. Spectrosc. Relat. Phenom. **8**, 129 (1976).
2. R. L. Chaney, Surf. Interface Anal. **10**, 36 (1987).

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
1	Ar $2p_{3/2,1/2}$	242 ± 1	...	31861	3.19	4.1	...
1	C $1s$	285 ± 1	...	233917	1.00	95.9	...
1	Ar $2s$	320 ± 1	...	14344	1.89	...	...
2	Ar $2p_{3/2}$	241.79 ± 0.05	0.89	...	...	...	...
2	Ar $2p_{1/2}$	243.93 ± 0.05	0.87	...	...	...	...
3	Ar $2s$	319.56 ± 0.05	2.4	...	...	...	...
4	C $1s$	284.53 ± 0.05	1.6	...	...	...	...

Footnote to Spectrum 00063-04: The asymmetry of this peak is attributed to the damage caused by the ion etching. Plasmons originating from the bulk were found at ~312.7 eV and 346.3 eV.

Footnote to Spectrum 00063-05: The $3p_{3/2,1/2}$ and $3s$ signals for argon are located at 9 and 23 eV on top of the valence band structure of ion etched graphite, which has a maximum at approximately 19 eV.

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
6[a]	Cu $2p_{3/2}$	932.70 ± 0.05	1.02	1175500	...	...	...
7[b]	Au $4f_{7/2}$	84.01 ± 0.05	0.82	1200555	...	...	...
8[c]	Ag $3d_{5/2}$	368.31 ± 0.05	0.70	1241190	...	...	...

[a]Experimental results gave 932.73 eV for Cu $2p_{3/2}$.
[b]Experimental results gave 84.04 eV for Au $4f_{7/2}$.
[c]Experimental results gave 368.35 eV for Ag $3d_{5/2}$.

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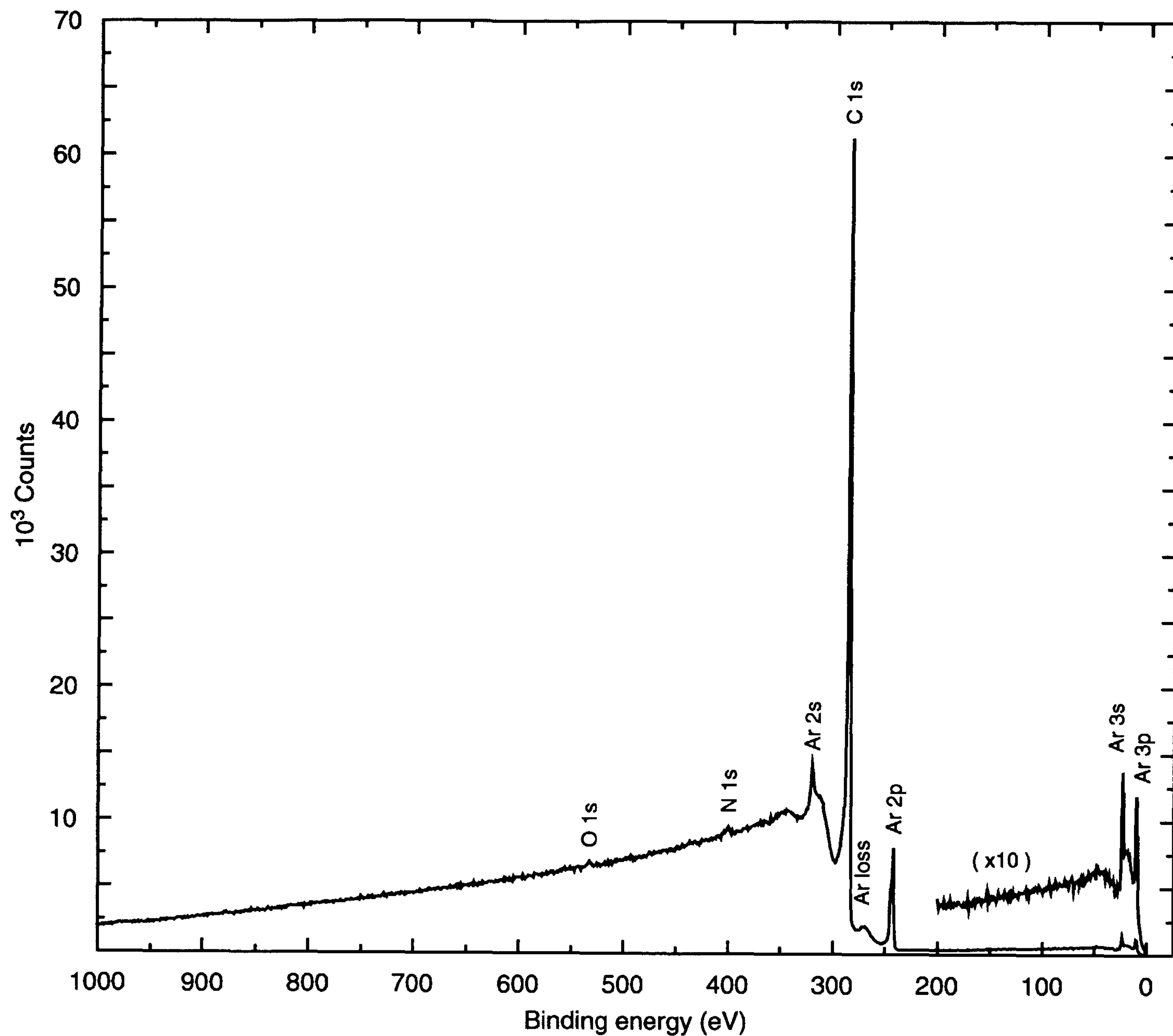

- **Accession #:** 00063-01
- **Host Material:** natural graphite crystal
- **Technique:** XPS
- **Spectral Region:** survey

Instrument: Fisons Instruments S-Probe E-2703

Excitation Source: Al K_α monochromatic

Source Energy: 1486.7 eV

Source Strength: 200 W

Source Size: 250 μm $\times$ 700 μm

Incident Angle: 20°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

Analyzer Resolution: 1.5 eV

Emission Angle: 0°

Data Acquisition Time: 800 s

Dead Time Correction: not specified

Number of Scans: 4

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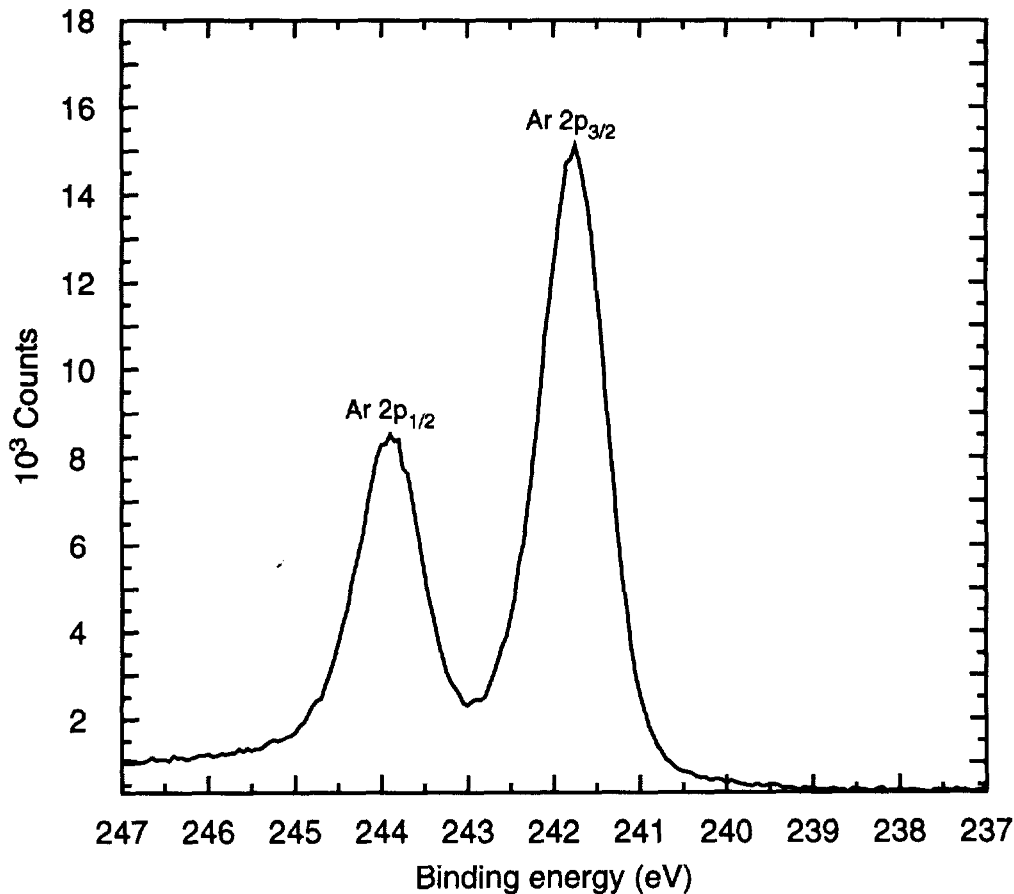
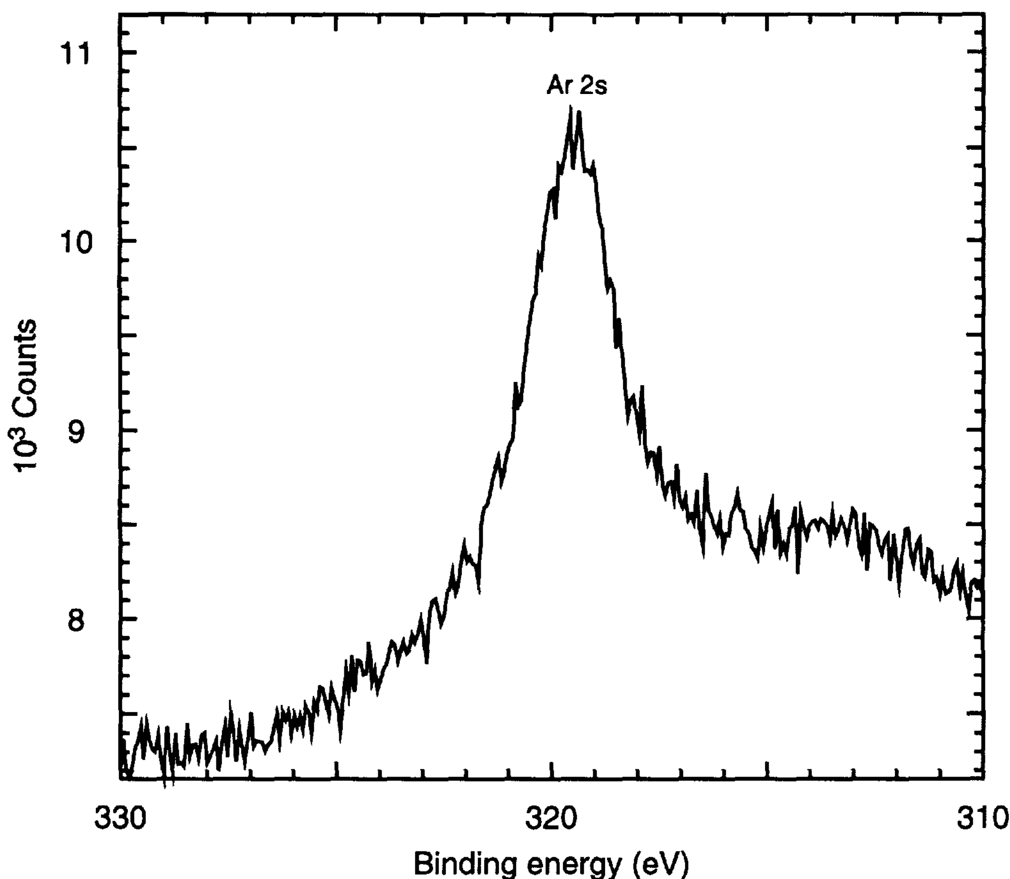


- **Accession #:** 00063-02
- **Host Material:** natural graphite crystal
- **Technique:** XPS
- **Spectral Region:** Ar $2p_{3/2}$; Ar $2p_{1/2}$

Instrument: Fisons Instruments S-Probe E-2703
Excitation Source: Al K_α monochromatic
Source Energy: 1486.7 eV
Source Strength: 200 W
Source Size: 250 μm $\times$ 700 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.50 eV
Emission Angle: 0°
Data Acquisition Time: 800 s
Dead Time Correction: none
Number of Scans: 20



- **Accession #:** 00063-03
- **Host Material:** natural graphite crystal
- **Technique:** XPS
- **Spectral Region:** Ar 2*s*

Instrument: Fisons Instruments S-Probe E-2703
Excitation Source: Al K_α monochromatic
Source Energy: 1486.7 eV
Source Strength: 200 W
Source Size: 250 μm $\times$ 700 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.50 eV
Emission Angle: 0°
Data Acquisition Time: 840 s
Dead Time Correction: none
Number of Scans: 14
Comment: The curved background is due to the overlap of the Ar 2*s* signal with the energy loss envelope of the C 1*s* signal.

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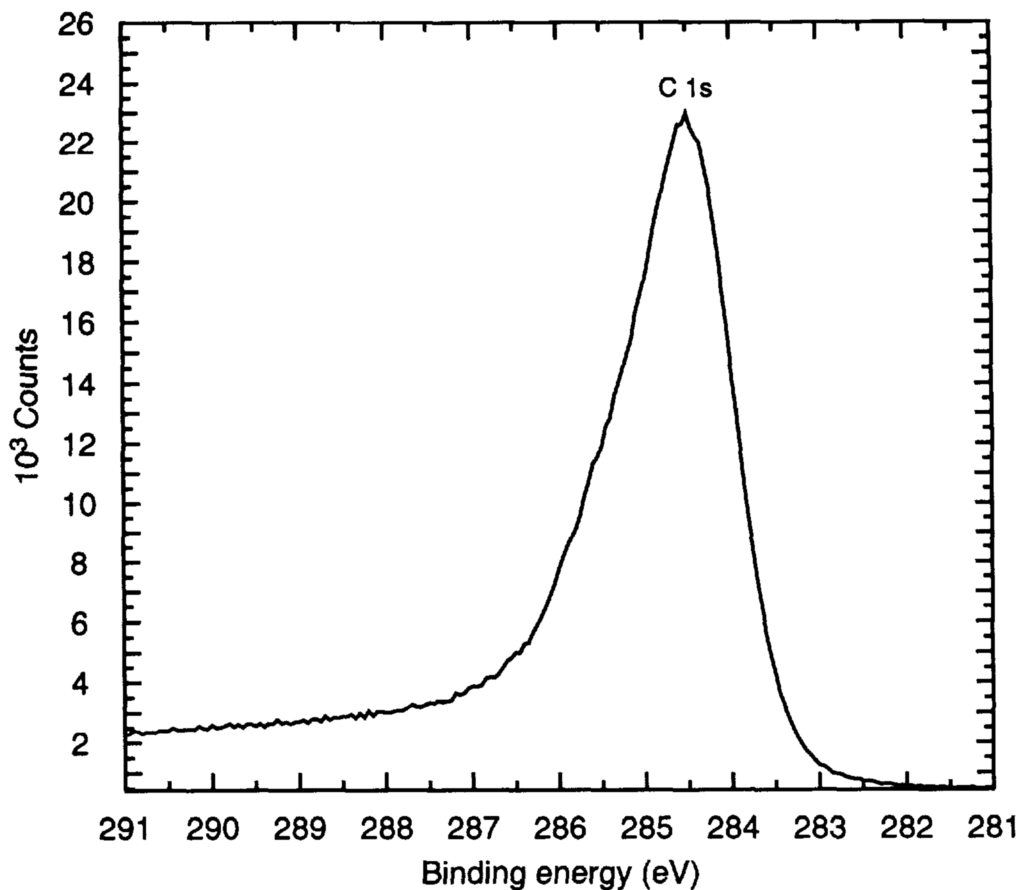
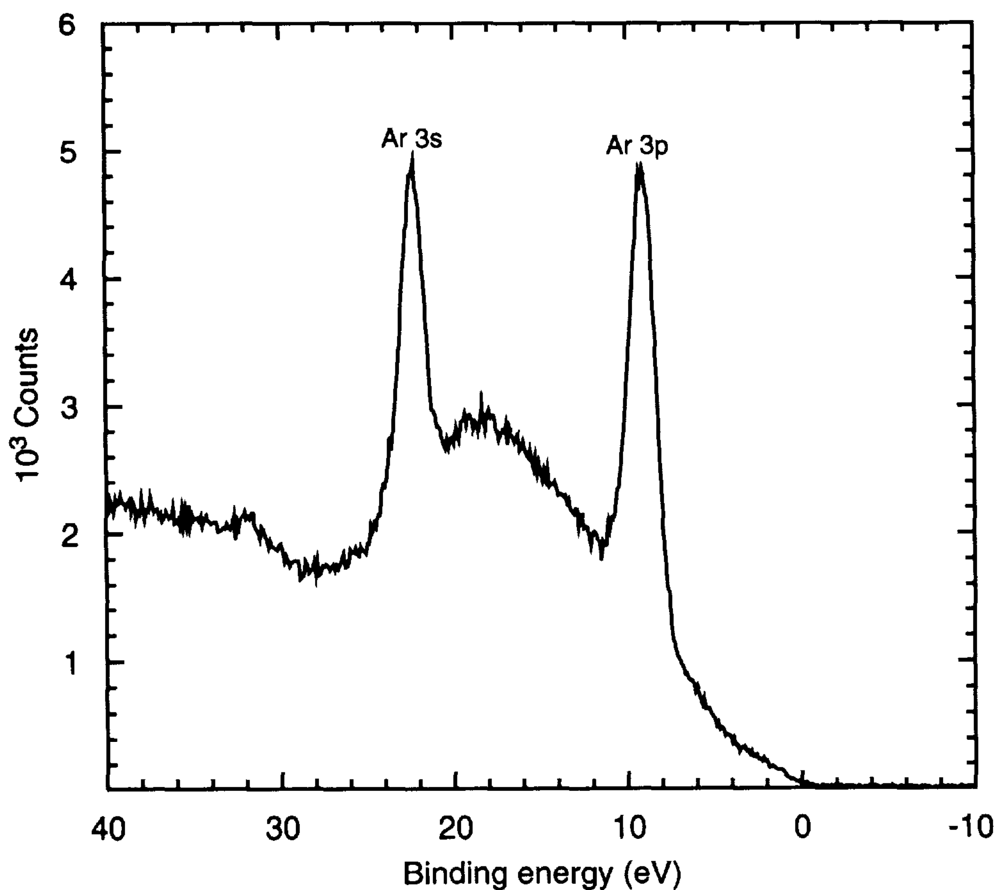


- **Accession #:** 00063-04
- **Host Material:** natural graphite crystal
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Fisons Instruments S-Probe E-2703
Excitation Source: Al K_α monochromatic
Source Energy: 1486.7 eV
Source Strength: 200 W
Source Size: 250 μm $\times$ 700 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.50 eV
Emission Angle: 0°
Data Acquisition Time: 100 s
Dead Time Correction: none
Number of Scans: 5
Comment: See footnote below the Spectral Features Table.



- **Accession #:** 00063-05
- **Host Material:** natural graphite crystal
- **Technique:** XPS
- **Spectral Region:** valence band

Instrument: Fisons Instruments S-Probe E-2703
Excitation Source: Al K_α monochromatic
Source Energy: 1486.7 eV
Source Strength: 200 W
Source Size: 250 μm $\times$ 700 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 150 eV
Analyzer Resolution: 1.5 eV
Emission Angle: 0°
Data Acquisition Time: 1500 s
Dead Time Correction: none
Number of Scans: 15
Comment: See footnote below the Spectral Features Table.

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