JPS-9200 Application Data

Micro analysis

The JPS-9200 acquires wide photoelectron images from areas 10 mm x 10 mm or larger (maximum 18 mm x 50 mm).
The surface (10 mm dia.) of SUS303, polished and cleaned with an organic solvent, was analyzed in the JPS-9200.The polished surface of the sample lost its wetness to a degree.
Spot analysis at 50 um dia. and photoelectron imaging of an area of 10 mm x 10 mm were performed to identify the cause of the reduced wetness.
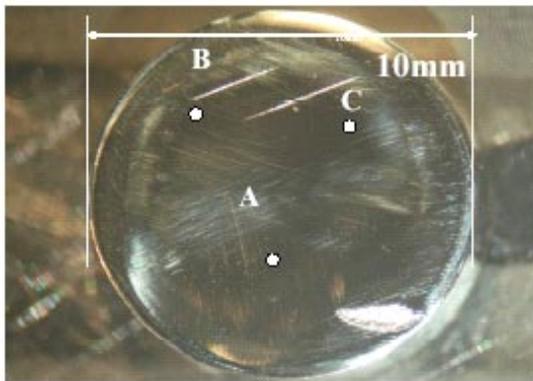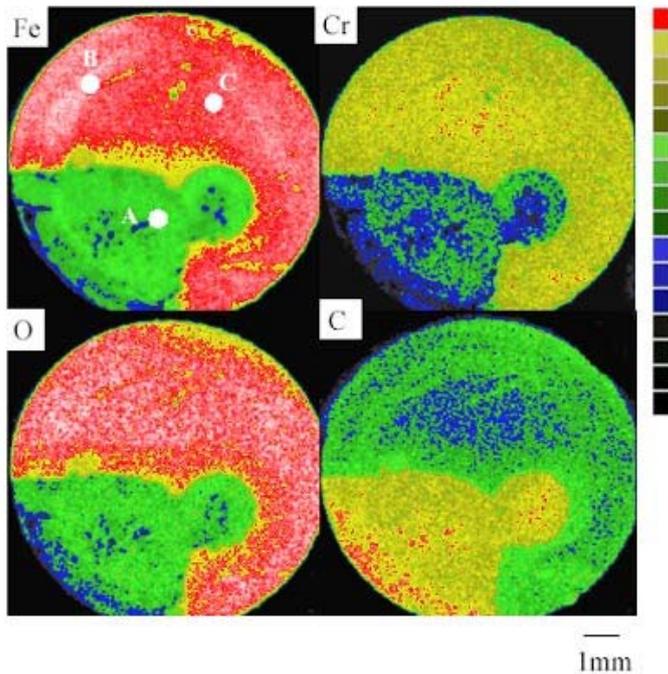

Optical micrograph

The photo only shows polishing traces.
Shown below are wide spectra acquired from points A, B, and C on the polished surface. At each point, the C1s peak intensity changed. No major difference was seen in the wide spectra of B and C, while the C1s peak intensity significantly increased at A.
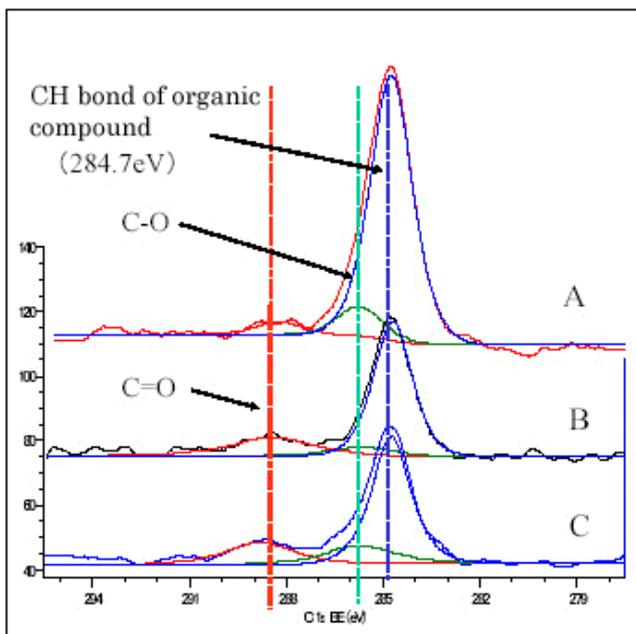The data suggest that organic compounds are distributed on the sample surface.
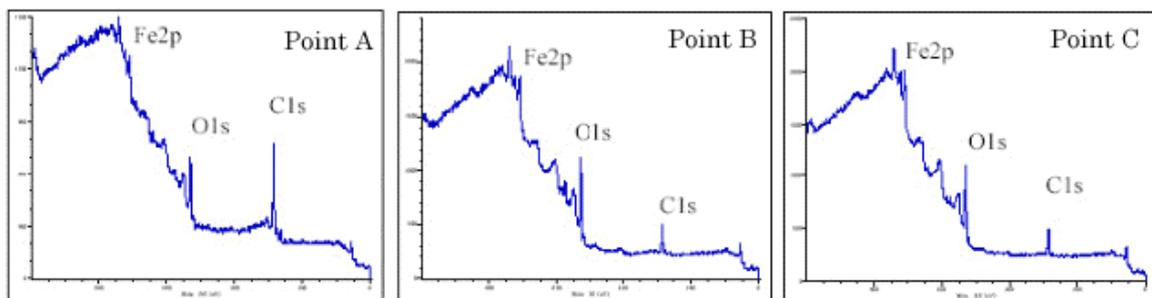
Photoelectron images of entire sample surface
Shown above are photoelectron images of Fe, Cr, O, and C.
They demonstrate different levels of contamination on the surface, not viewed in the optical micrograph.

C1s peak separation spectra at A, B, and C
The C1s spectra, after peaks were separated, show the same hydrocarbon compound at B and C. At A, however, the CH bond peak increased, indicating the presence of a fair number of organic compounds containing CH bonds.

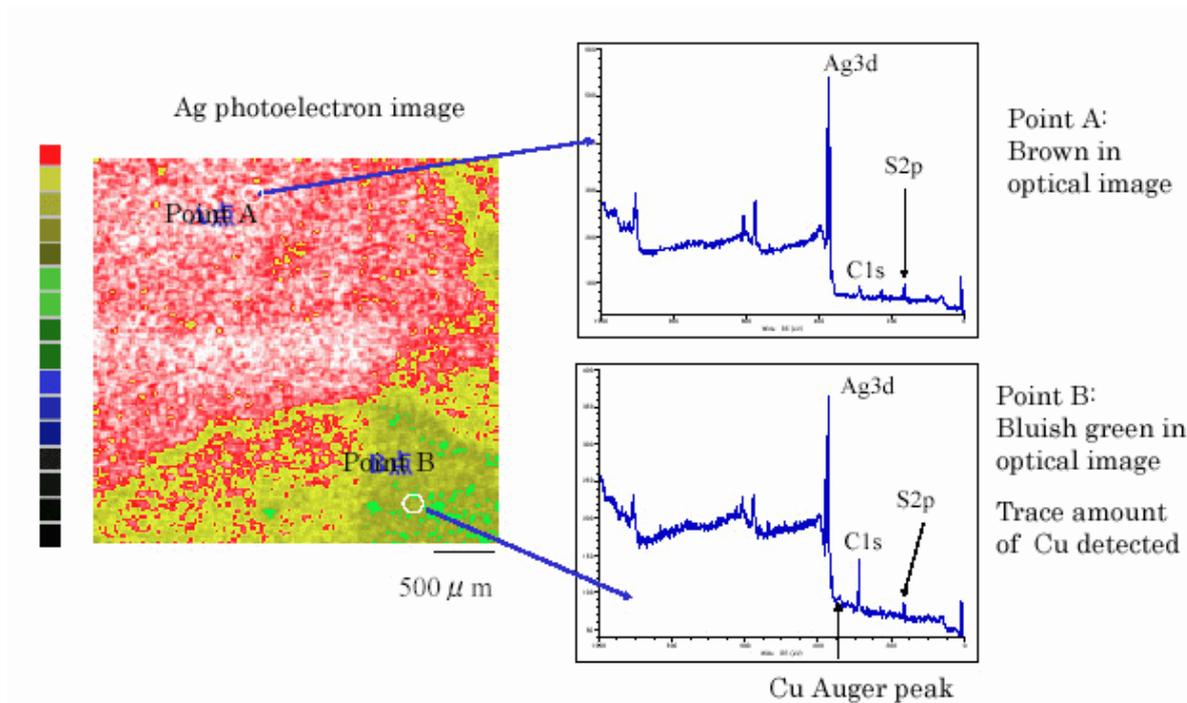| CH peak ratio A: 93%; B: 63%; C: 73% The CH intensity increased substantially at point A, suggesting the residual organic compound used for cleaning. C-O and C=O bonds are functional groups of the surface contamination, in good agreement with the ratio at each analytical spot.	Residual organic compound in cleaning ⬇ Organic compound: Paraffinic hydrocarbon (C_nH_{2n+2})

JPS-9200 Application Data (2)

Micro analysis 2

The JPS-9200 has a high performance ion gun to support depth profiling. With the X/Y fully motorized stage drive, the system acquires depth profile data from up to 20 specified micro areas.
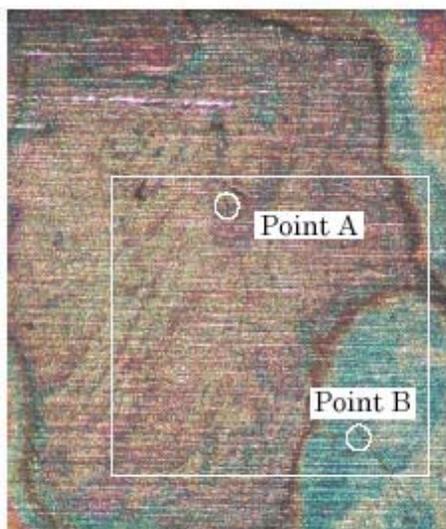
On the left is an optical micrograph showing the discolored area of a silver plated copper lead frame. Brown and bluish green portions represent different levels of discoloring. Shown below is a photoelectron image of Ag in the framed area of 2.8 mm x 2.8 mm. The image shows Ag distribution, not viewed in the optical image. The analytical spot was determined by the AP-Navi system.

Ag photoelectron image

Point A

Point B

500 μ m

Ag3d

S2p

C1s

Point A:
Brown in
optical image

Ag3d

S2p

C1s

Point B:
Bluish green in
optical image

Trace amount
of Cu detected

Cu Auger peak

The photoelectron data show that the Ag intensity decreased in the bluish green area in the optical micrograph. Also, the wide spectrum of point B in the bluish green area detected a trace amount of Cu.

Wide spectra data were acquired at points A and B where Ag was distributed differently. The area of analysis at each point was 50 um in diameter. At both areas, Ag and S were detected. The wide spectrum of point B detected a trace amount of Cu.
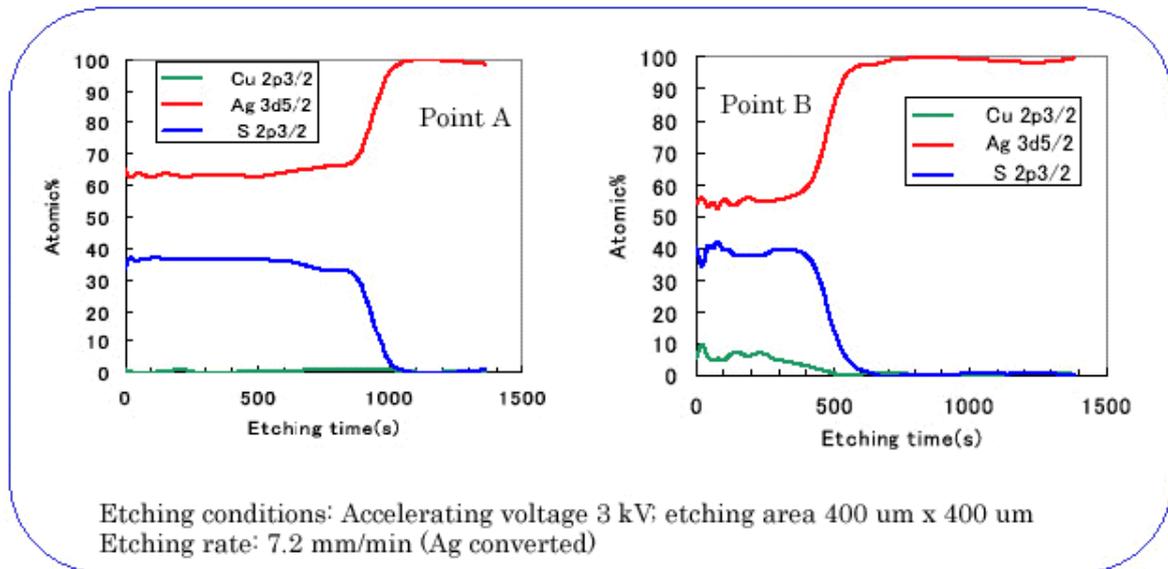

S2p1/2

S2p3/2

S2p3/2
peak position

160.9eV→Ag_2S
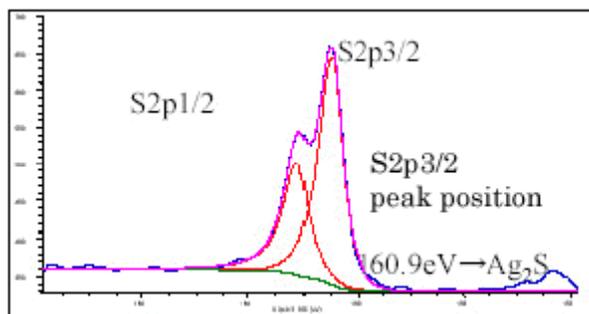
S2p spectrum at A (peaks separated)

Shown on the left is the S2p spectrum acquired at A. Ag_2S was identified from the S2p3/2 peak position.

At point B, a trace amount of Cu was detected. Shown below are depth profiles acquired at points A and B.
The depth profile data show that the Ag_2S layer at point A was approximately twice thicker than at point B, and that Cu was segregated on the surface at point B.

Etching conditions: Accelerating voltage 3 kV; etching area 400 um x 400 um
Etching rate: 7.2 mm/min (Ag converted)

The data indicates that the discoloration observed in the optical micrograph was caused by:
1) Thick layer of Ag_2S (up to 120 nm) at point A (brown)
2) Ag_2S layer up to 60 nm thick with Cu segregated at point B (bluish green)
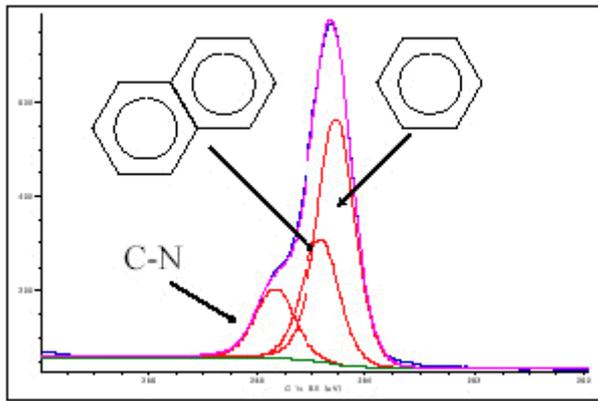
JPS-9200 Application Data (3)

High energy resolution analysis

Organic electro luminescence (organic EL) is the emission of light when voltage is applied to extremely thin organic materials composed mainly of carbon and hydrogen.

Organic EL uses low and high polymers as organic source materials. Low polymer films are developed by vacuum evaporation, while high polymer films by spin coating.

To demonstrate the effectiveness of XPS spectral data in studying these organic EL materials, organic EL materials were analyzed in XPS in the high energy resolution mode (monochromatic X-ray source used).

Low polymer EL material
Preparation:
Al evaporated on glass substrate →α-NPD evaporated (pressure 1×10^{-5} Torr, film thickness 50 nm)

α-NPD:4,4'-bis[phenyl(1-naphtyle)amino]-1,1'bihenyl
Light emitting band: 450 to 700 nm

Wide spectrum

C1s

N1s

No impurities were detected in XPS.

Cls peak separation spectrum

Two types of shake-up peaks representing π electrons emerged on the high bond energy range of the C1s spectrum.
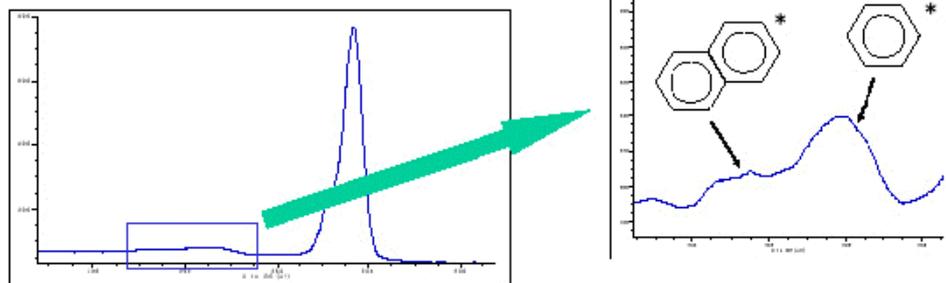

Cls narrow spectrum

Aromatic
rings have in-plane vibration in addition to CH expansion/contraction. This shift, although small at 0.2 eV or less, emerged adjacent to the main peak. An asymmetrical function (Sherwood function) was applied to the aromatic ring to separate peaks. The result was in good agreement with the α-NPD molecular structure.
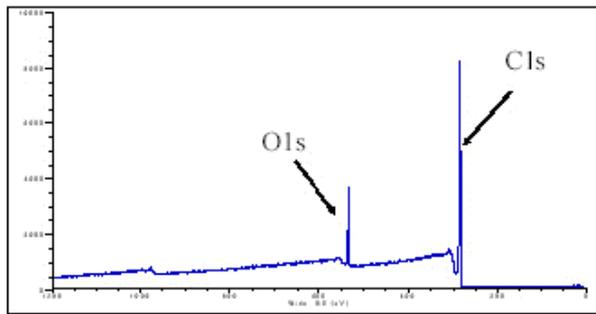
High polymer EL material

Preparation: THF (tetra-hydrofuran) film developed on ITO substrate in nitrogen atmosphere by spin coating
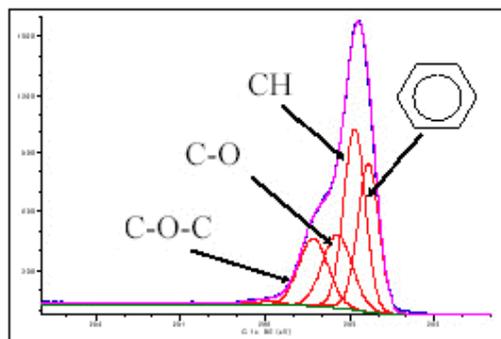Molecular weight: 700000

MEH-PPV : Light emitting band à 450 to 700 nm
[poly(2-methoxy,5-(2'-ethyl-hexyloxy)-p-phenylenevinylene)]
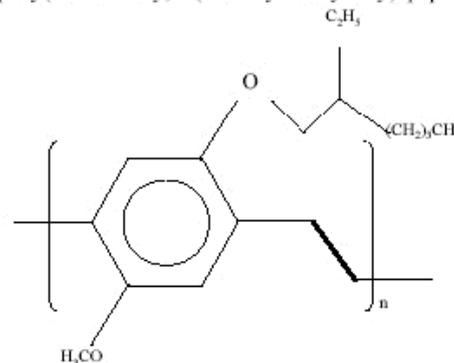
Bonding status: Aromatic ring, C=C, C-H, C-OH, and C-O-H bonds

Wide spectrum

Cls peak separation spectrum

The peak separation spectrum on the left did not detect shake-up peaks unlike the low polymer EL material. Lack of shake-up peaks is considered specific to this material. Thin film deposition may have changed the molecular structure, but details of this change need further investigation.

Samples courtesy of Dr. Masakazu Nakamura, Electronic Engineering, School of Engineering, Chiba University

JPS-9200 Application Data (4)
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TRXPS analysis

Total reflection x-ray photoelectron spectroscopy (TRXPS), with its shallow X-ray penetration depth, has advantages over conventional XPS, including higher surface sensitivity and low spectral background. It is effective for analysis of trace elements on the surface, chemical bonding, film thickness, and interface of surface layers of thin film. TRXPS was applied to study the chemical bonding on surface layers of a thin film sample.

The sample was a WS_2/C multiple layered film created by a multi target RF sputtering device. The photo on the right is a TEM image of the sample. The TEM photo shows that WS_2 and C layers 1.2 nm and 0.8 nm thick respectively are alternately developed. The surface layer is WS_2.


TEM image (bright field image of cross sectional layer structure)

←C
←WS$_2$

10.0nm
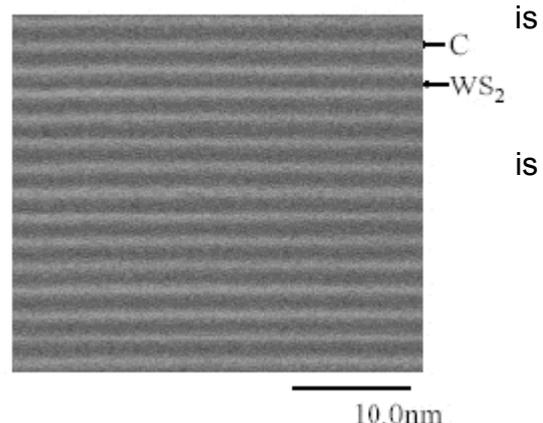
Shown below are the wide spectrum (red) acquired in TRXPS at an X-ray incident angle of 1.0 degree or less, and the wide spectrum (blue) acquired in regular XPS.
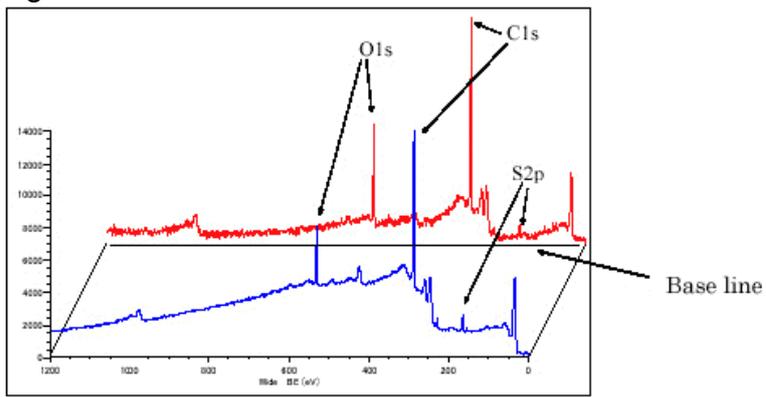
Compared to the regular XPS spectrum, the TRXPS spectrum has an extremely low background. It also shows that the O intensity increased while the S intensity decreased.
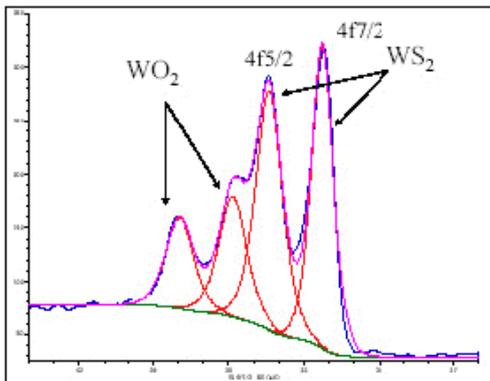

WS2:WO2＝68.7%:31.3%

W4f spectrum from conventional XPS
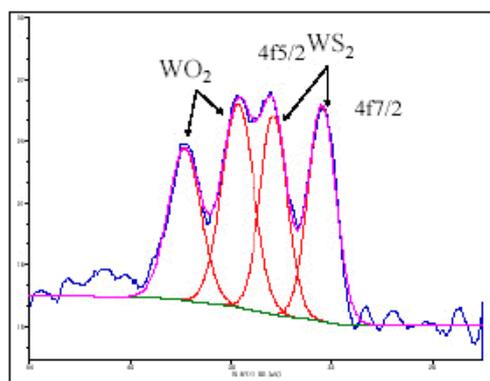
WS2:WO2＝49.6%:50.4%

W4f spectrum from TRXPS

The W4f peaks detected in TRXPS are quite different in shape from those detected in XPS. Peak separation was applied to each W4f. Results are shown above: XPS on the left and TRXPS on the right.

Both spectra detected WO_2 peaks other than WS2. The ratio between these two was approximately 7:3 in XPS and 1:1 in TRXPS, suggesting that the surface WS_2 layer was oxidized.

(1) The figure left compares C1s spectra acquired in XPS and TRXPS. They


Red: TRXPS
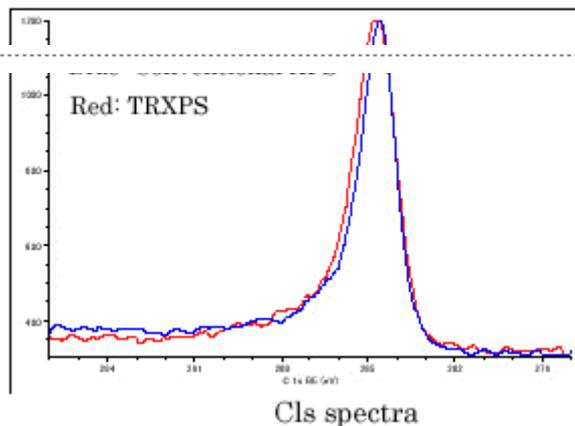
C1s spectra

are slightly different in shape. Shown below are results of peak separation applied to the C1s spectra in the following steps:The Sherwood function was applied to the C1s spectrum of XPS to determine the C1s spectral form.
(2) The resulting function parameters were applied to the C1s spectrum of TRXPS.

As a result of peak separation, the C1s spectrum of TRXPS shows two components (C-O, CH, and CS_2 bonds) that were not detected in the XPS spectrum. C-O and CH bonds are attributed to sample surface contaminants, while the CS_2 bond is inherent in the C layer.
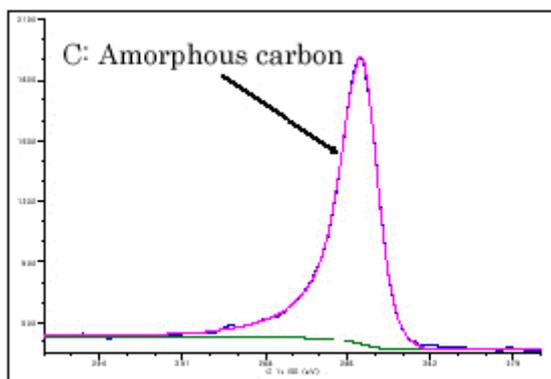
Cls spectrum from conventional XPS
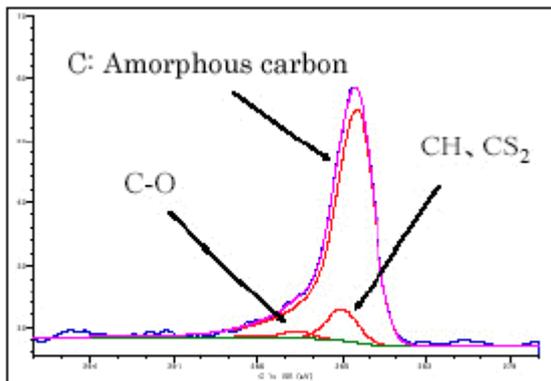
Cls spectrum from TRXPS

Samples courtesy of Dr. Watanabe, Department of Systems Engineering, Nippon Institute of Technology

[1 2 3 4]