Chemical State Information from Insulating Materials using PHI-5802

Improved Charge Compensation by using Mesh Screen with Flood Gun

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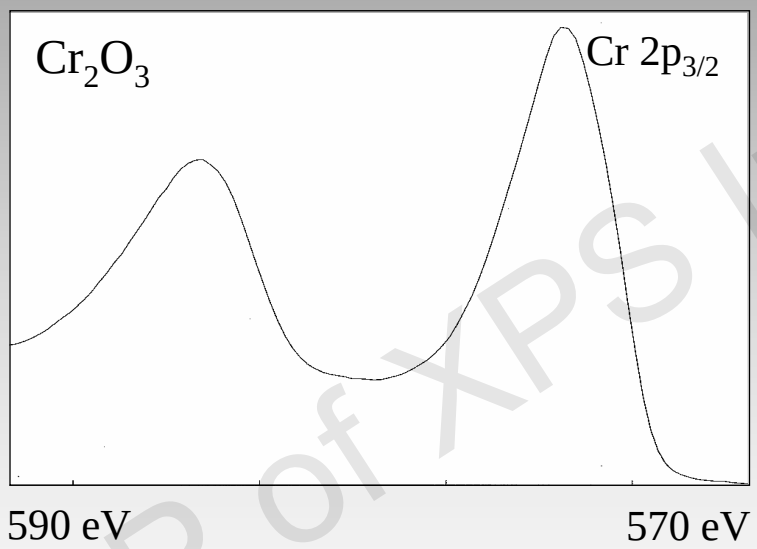
Summary

- High quality high energy resolution spectra from any insulating material are now routinely available by using a “Mesh-Screen Flood-Gun” combination on a PHI 5800 XPS.
- From these high quality spectra we can now resolve many chemical states not previously observed by using the previous flood-gun conditions.
- The flood gun conditions that provide near-optimum charge compensation conditions are:
  - Maximum current setting: 1.2A
  - Bias voltage: 6-8 V
  - Much closer proximity of the flood gun to sample
- Optimum conditions require the use of the Mesh-Screen.
- With the mesh-screen flood-gun combination the FWHM of the Si 2p is now in the range of 1.2-1.4 eV which is 100% smaller than the usual 2.0-2.8eV range obtained without the mesh-screen and the optimized conditions.
- As a measure of the quality of charge compensation, the makers look at the C 1s FWHM of the ester peak of PET. VG guarantees <0.95 eV on their XL system for this peak. By using the mesh-screen flood-gun combination we obtained a C 1s FWHM = 0.85 eV with a PE = 23 eV. If we were to decrease our PE more, then the FWHM will decrease by another 5-10%.

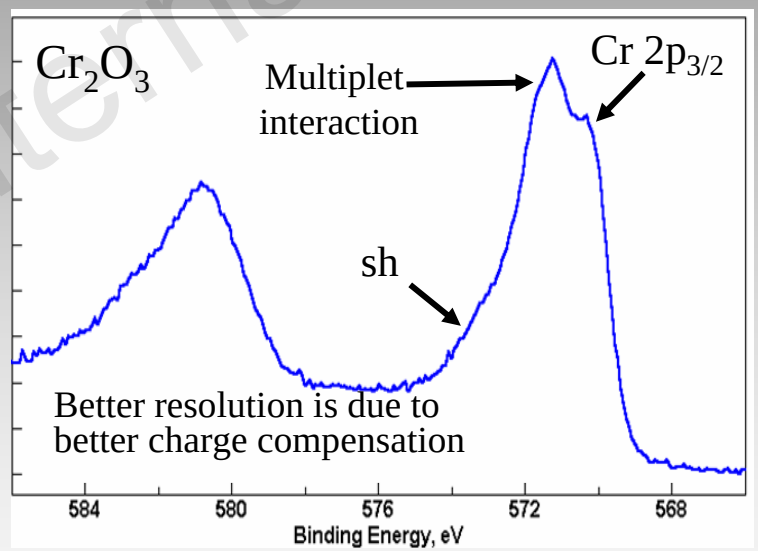
Charge-Compensation Quality versus Information

Both samples are insulators. Both require charge compensation (charge control).
The spectrum on the left has symmetrical peaks and is from a popular XPS handbook.
The spectrum on the right has more features. Why? The mesh-screen was used.
Reference spectrum suffers from charge broadening (vertical / horizontal)
Charge compensation for spectrum on right is better.

Reference spectrum from PHI's 2nd Book



True Spectrum of Cr_2O_3



Reference: The lowest BE peak at 575.7 eV is the true Cr 2p3 peak in pure Cr_2O_3, not 576.6 eV, as is commonly reported. The most intense peak at 576.9 eV is due to the presence of "multiplet interactions" (ref. A.R.Pratt and N.S.McIntyre, *Surface and Interface Analysis*, **24**, 529 (1996)).

C (1s) FWHM as an Indicator of Charge Compensation Quality

These FWHM are derived from Vol. 2 of the *Handbooks of Monochromatic XPS Spectra*. The mesh-screen flood-gun combination was used to obtain these values using everyday high energy resolution settings. The FWHM shown here are 100-200% smaller than those previously obtained with the PHI-5802 XPS.

Metal Oxide	Metal FWHM	Oxygen FWHM	Hydrocarbon FWHM
Ag_2O	1.04 eV	0.96 eV	1.10 eV
Cr_2O_3	1.33	1.24	1.45
Fe_2O_3	1.32	1.05	1.29
Ga_2O_3	1.37	1.51	1.48
HfO_2	1.40	1.68	1.55
HgO	1.06	0.96	1.16
MnO_2	1.12	1.02	1.21
MoO_3	1.01	1.20	1.48
Nb_2O_5	1.14	1.36	1.35
Sc_2O_3	1.27	1.33	1.45
SiO_2	1.25	1.40	1.27
SnO_2	1.28	1.29	1.36
Ta_2O_5	1.12	1.46	1.56
TiO_2	1.02	1.18	1.34
Y_2O_3	1.25	1.30	1.51

Experimental Observations

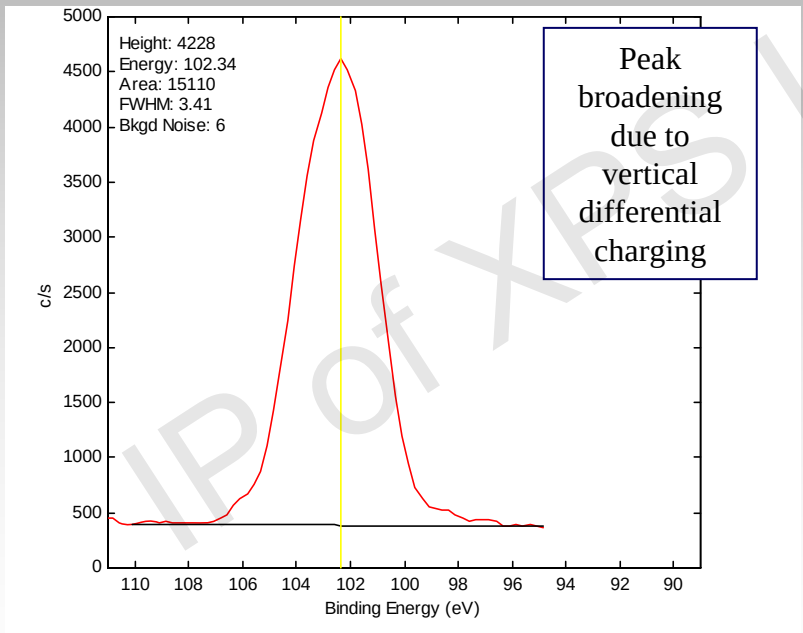
1. Metal FWHM is similar to O 1s FWHM and also C 1s.
2. Smaller FWHM for C 1s correlates with smaller FWHM of metal FWHM and O 1s FWHM.
3. **C 1s FWHM serves as a useful guide of the quality or goodness of charge compensation.**

High Energy Resolution Scans on PHI-5802

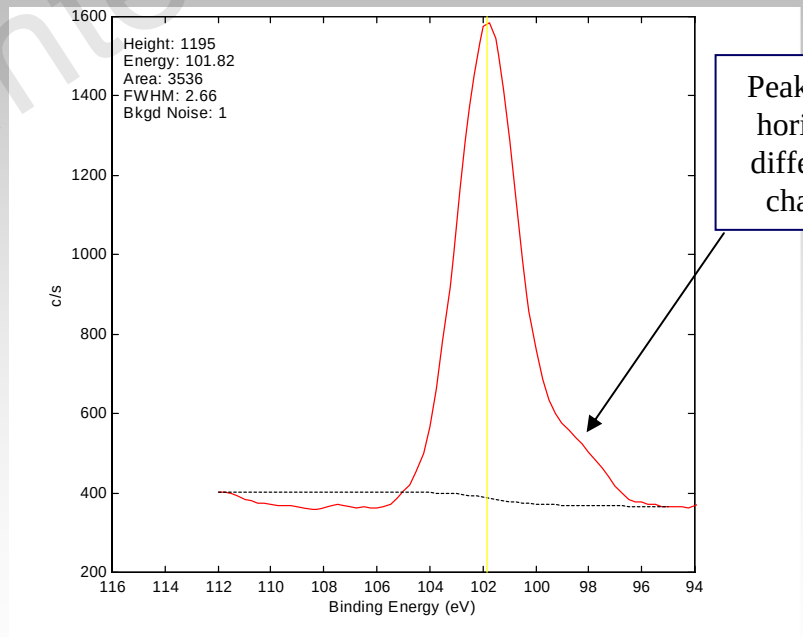
Both spectra suffer from differential charging. The spectrum on the left suffers from vertical differential charging which broadens the peak uniformly. The spectrum on the right suffers from horizontal differential charging which produced the shoulder.

A user with 3 years experience, operated the flood gun in the usual style, trying to achieve C 1s = 284.8 eV, with less than the max current and without beam alignment. Here are the results using the standard aperture traps and without the mesh-screen.

Si 2p FWHM = 3.41 eV

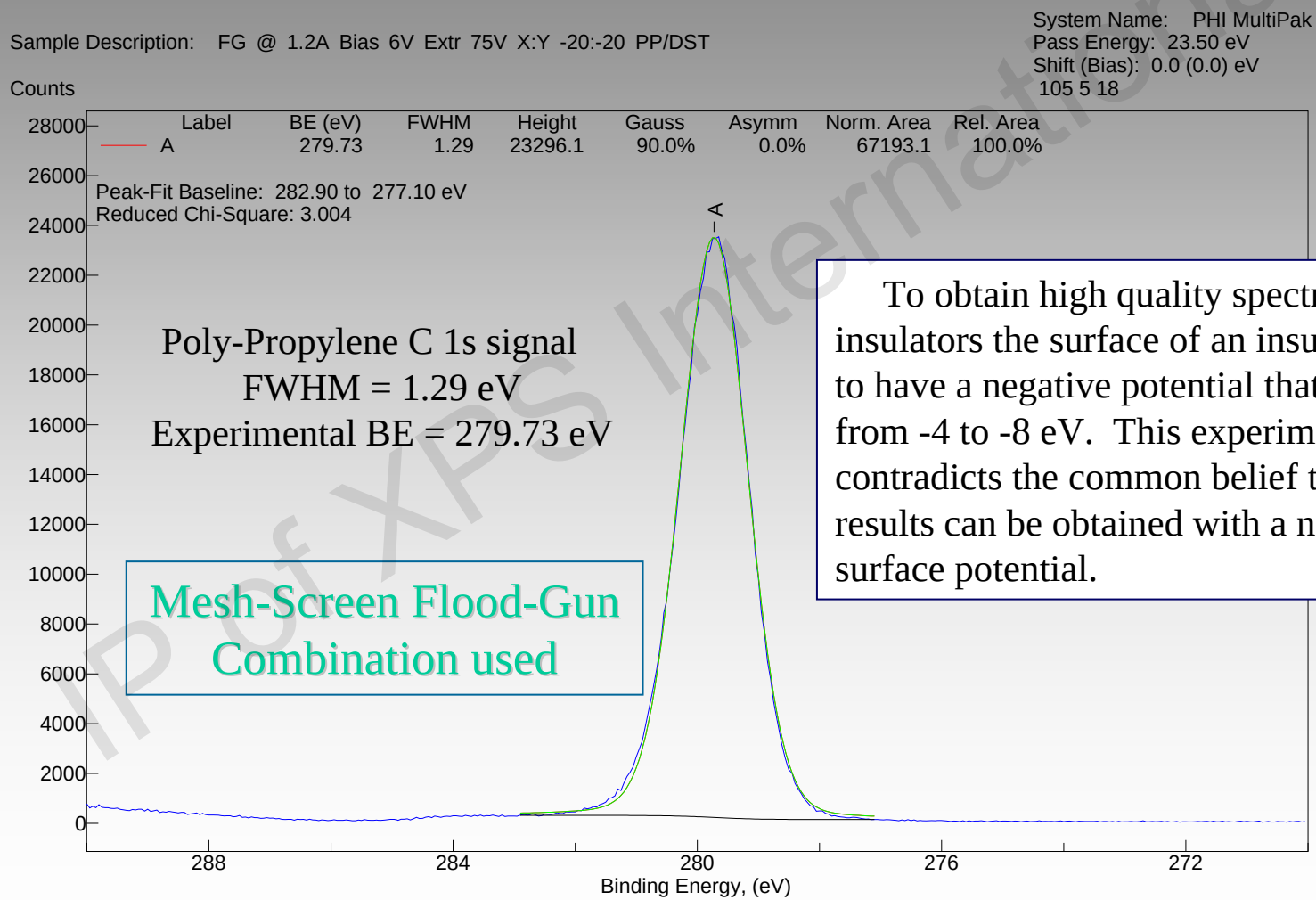


Si 2p FWHM = 2.66 eV



Charge Compensation on PHI-5802 using Mesh-Screen Flood-Gun Combination

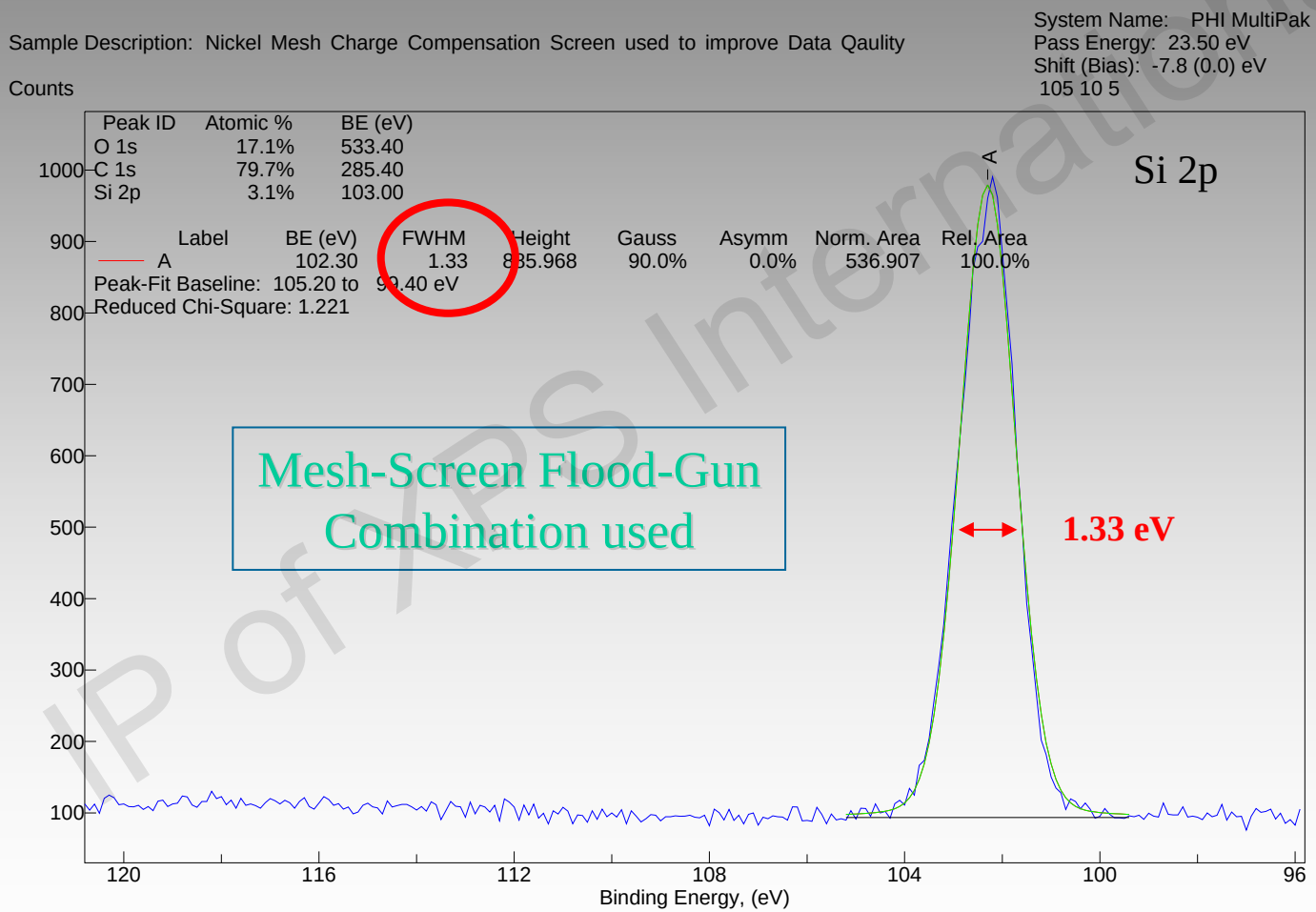
After experimenting with various flood gun setting with the Mesh-Screen standing over a piece of Poly-Propylene, we found the flood gun needs to set to its' maximum current setting with a 6-8 volt bias to produce high quality spectra from insulating materials.



To obtain high quality spectra from insulators the surface of an insulator needs to have a negative potential that ranges from -4 to -8 eV. This experimental fact contradicts the common belief that good results can be obtained with a near zero surface potential.

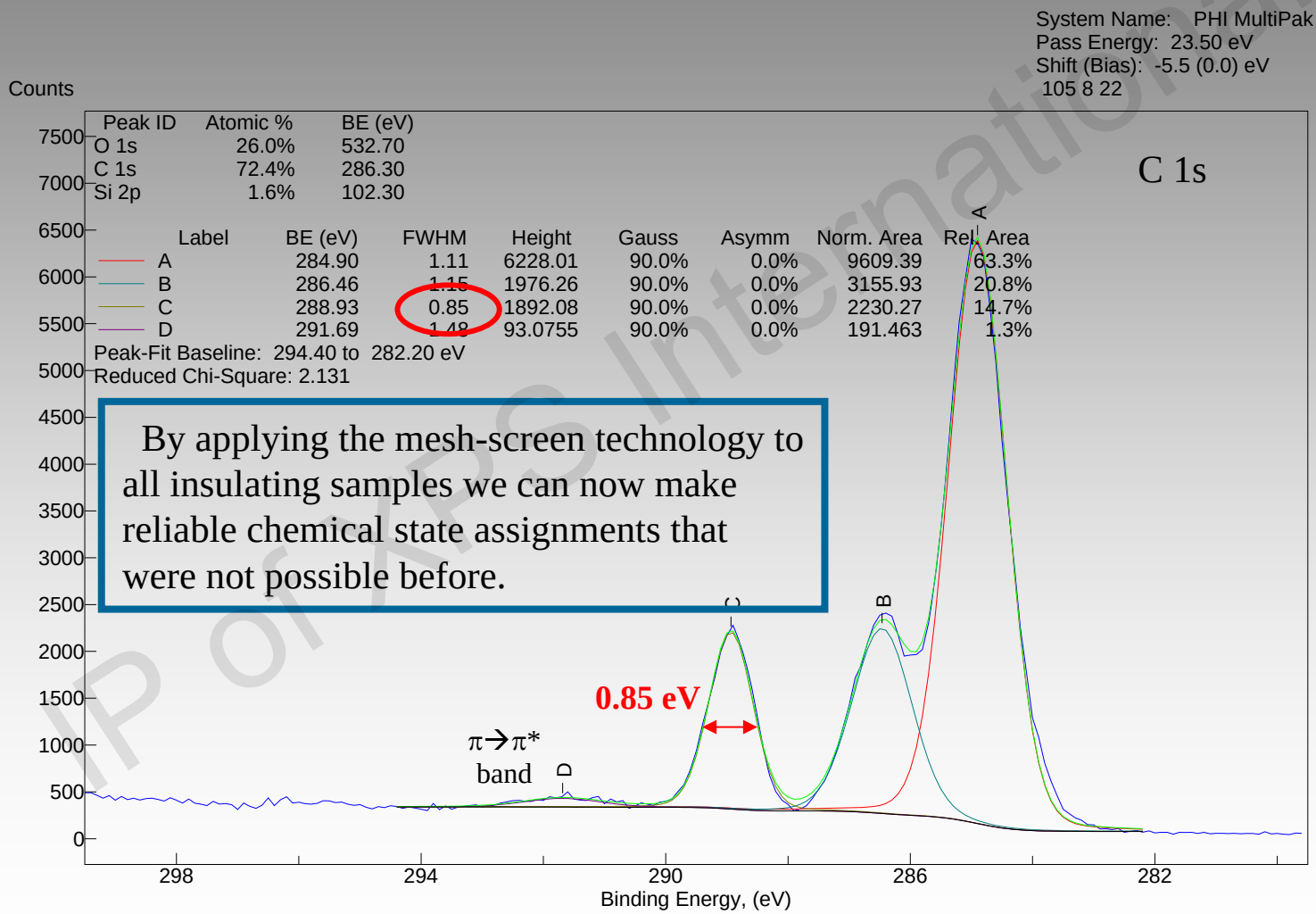
Si 2p FWHM = 1.33 eV A Recent Result from the Adhesive side of a Label containing Silicone Oil using Mesh-Screen Flood-Gun Combination

The experimental conditions needed to collect this high quality spectrum were obtained within the normal amount of time (1-2 min.) used for "Set-up Align".



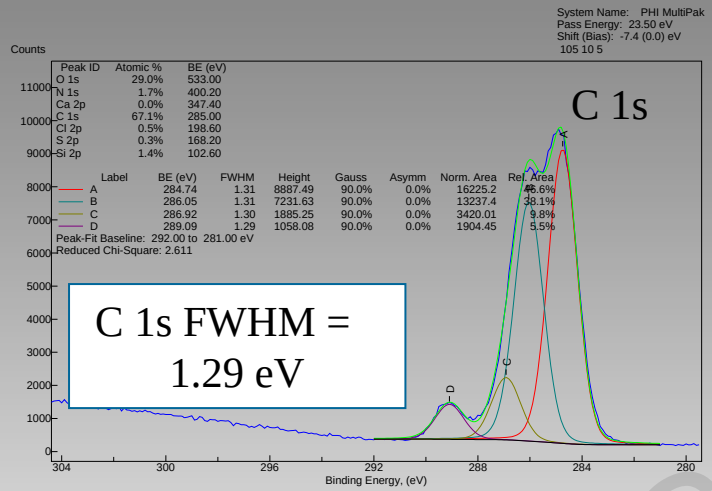
C 1s FWHM = 0.85 eV A Recent Result from the Support Film of a Label Tested for the presence of Silicone Oil using Mesh-Screen Flood-Gun Combination

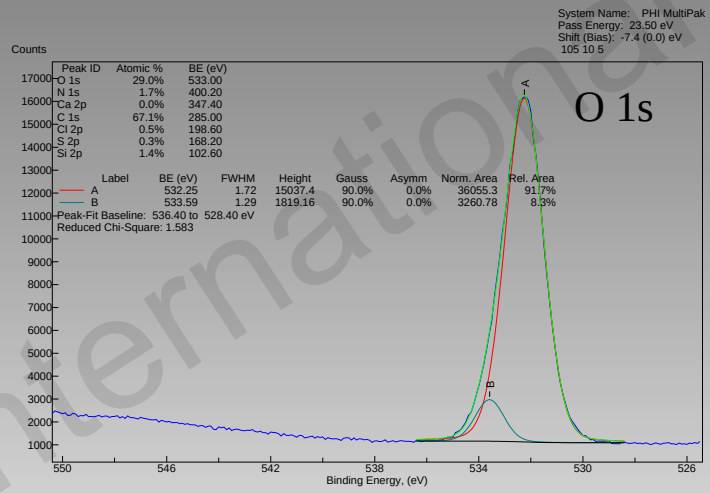
The peak area ratios, chemical shifts and π→π* band clearly indicate that the sample is made of PET (Mylar).

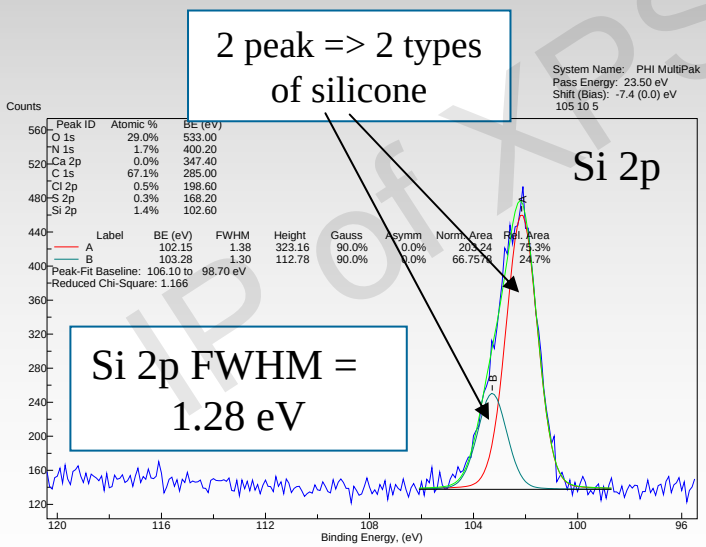


Example 1: Chemical States are Now Obvious & Reliable

Paper Side of Label







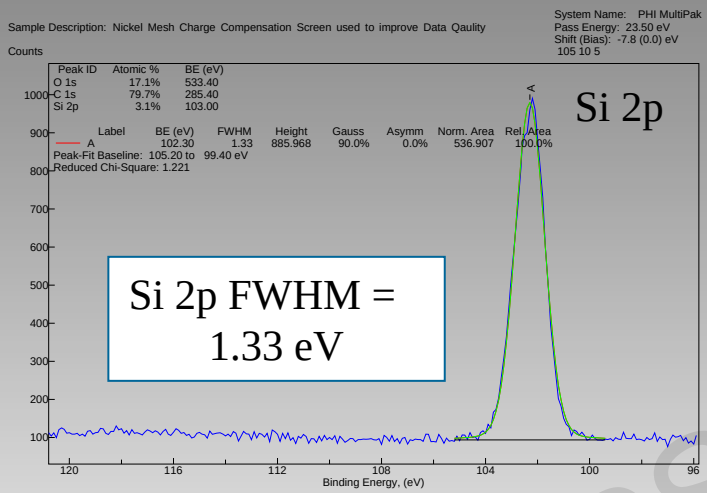
Mesh-Screen Flood-Gun Combination used

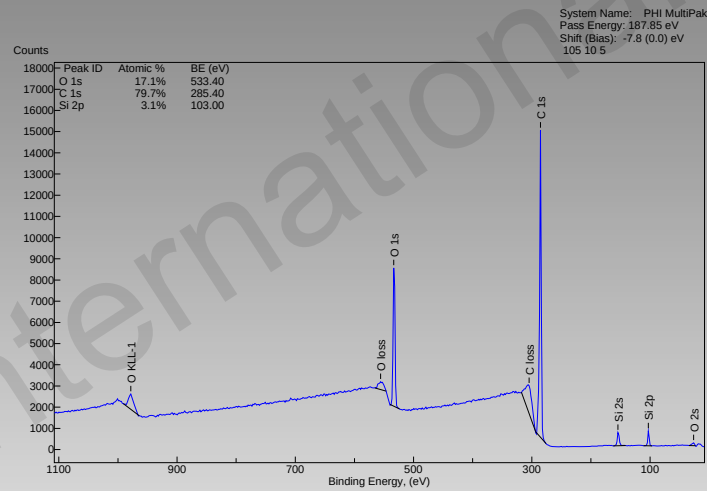
The FWHM in the C 1s spectrum are less than 1.5 eV which implies good quality charge compensation. With this condition, we can indeed trust that there are two different types of silicon chemical as indicated by the two peaks seen in the peak-fit of the Si 2p spectrum.

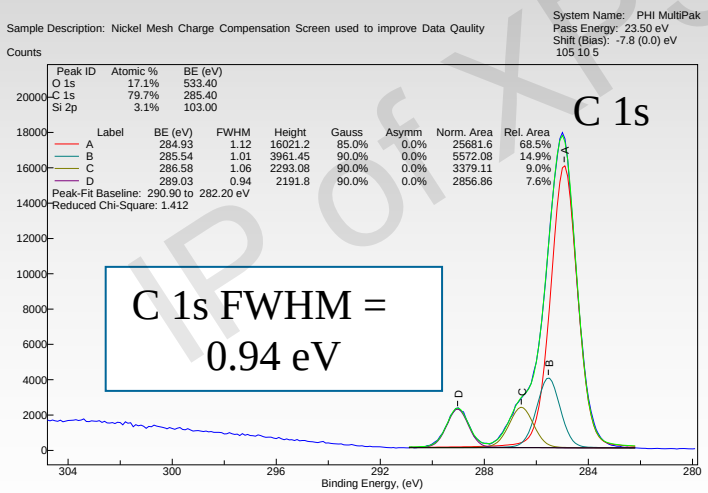
Example 2: Chemical States are Now Obvious & Reliable

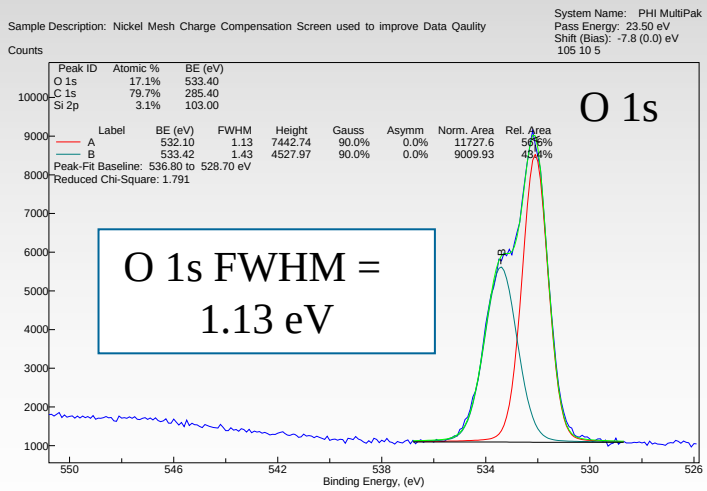
Adhesive Side of Label

Mesh-Screen Flood-Gun Combination used









Example 3: Chemical States are Now Obvious & Reliable

Bottom Side of Support Film

Mesh-Screen Flood-Gun Combination used