Application Note: 31022

Method for Charge Compensation on Sigma Probe, Theta Probe and Theta 300

Key Words

- Surface Analysis
- Digital Control
- Operation of the Electron/Ion Gun
- XPS Spectra from Insulators

When X-ray photons strike a surface, they cause the emission of electrons. This, of course, is the basis of the XPS technique. If the surface is electrically insulating then the emission of electrons causes a positive charge to accumulate at the surface. When a non-monochromated source is used for XPS analysis there are usually a sufficient number of electrons in the region of the sample to limit the effect of the charging. However, when using a monochromated X-ray source for XPS measurements, there are too few stray electrons to control the charging and the resulting positive charge severely affects the XPS spectrum. It causes the peaks in the spectrum to shift to high binding energies and become distorted. For this reason, it is necessary to use a low energy electron beam to control the charging process.

The energy of the electron beam is usually a few electron volts (typically < 6 eV). This causes the peaks to shift by a controlled amount to lower binding energy, a shift that can be removed by spectrum processing in the Thermo Scientific *Avantage* data system.

In practice, a number of factors need to be considered for effective charge compensation, these factors will be discussed here.

The Electron Source

It is important that the electron beam is of low energy to avoid unintended sample degradation caused by the electrons.

Equally important is that the electron energy spread is as small as possible. The surface of an insulating sample will charge to the potential of the most energetic electrons arriving at its surface, while all those at lower energies will be reflected away to the walls of the chamber. When X-rays impinge on part of the sample surface, there would be insufficient flux of these high energy electrons to maintain the surface potential in this region. This would lead to differential charging and peak broadening.

On the Thermo Fisher Scientific products Sigma Probe, Theta Probe and Theta 300, the energy spread has been minimized by using a LaB_6 electron emitter in the electron gun. This source is beneficial in two ways:

- It is indirectly heated, eliminating the voltage drop which occurs along a conventional hot-filament source.
- It has a low work function which means that, for a given emission current, the temperature is lower than it would be for a filament source. Hence, thermal broadening is reduced.

Use of the LaB_6 emitter in the electron gun minimizes the problems associated with a large energy spread in the electron beam.

Static Charge

A difficulty that can arise when analyzing insulating samples is that a negative static charge can occur at the surface of the sample, illustrated in Figure 1.

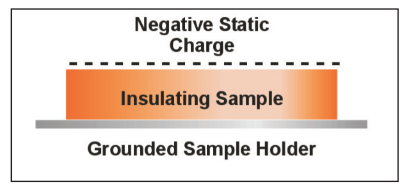

Figure 1: A negative static charge may occur at the surface of an insulating sample

When the focused X-ray beam is directed to the sample, the negative charge is replaced by a positive charge in the area that is exposed to the X-rays (the analysis area) while the surrounding area remains negative, Figure 2.

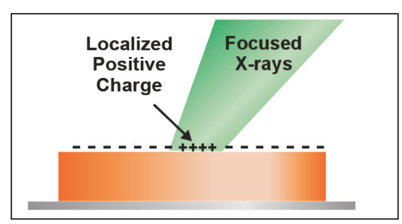

Figure 2: A localized positive charge is formed in the area exposed to the X-rays

When the low energy electrons are directed to the analysis position, they experience the field due to the large area of negative charge and are prevented from reaching the analysis position, Figure 3. Under these conditions there is no effective charge compensation.

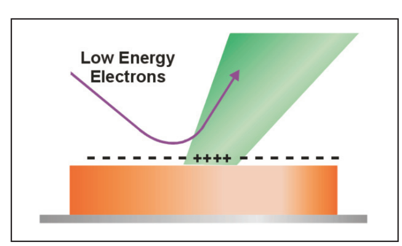

Figure 3: Low energy electrons are prevented from reaching the analysis position by the field due to the static charge on the sample

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The problem can be overcome by removing the static charge using a low energy ion beam. With the negative charge removed, the low energy electrons can now reach the analysis position, Figure 4.

By this means, good charge compensation can be achieved.

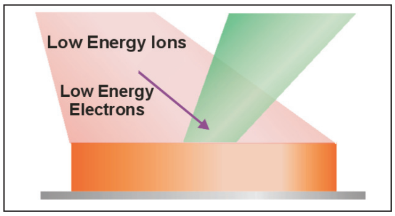

Figure 4: By flooding the surface with low energy ions, the negative charge is removed and electrons can now reach the analysis position

Combination Ion Gun

The use of a single gun to produce both the focused low energy electron beam and the large area ion flux has a number of distinct advantages over the use of independent sources. A new, combined ion/electron gun has been designed which incorporates all the above advantages.

This novel design uses a LaB_6 emitter to produce a bright source of low energy electrons, with an energy spread of only about 0.3 eV. These are focused to a small spot on the sample. Precise alignment to the X-ray beam is provided using integral X-Y deflectors. Focusing and alignment of this low energy beam is enhanced by the extremely low residual magnetic fields present in the mu-metal analysis chambers.

Figure 5 shows a schematic diagram of the combined gun.

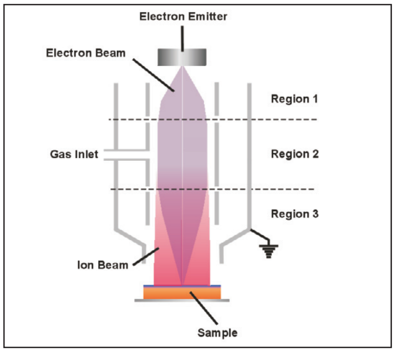

Figure 5: Schematic representation of the combined electron/ion gun used in Sigma Probe and Theta Probe. (For improved clarity, the steering electrodes have been omitted from the diagram).

Region	Electrons	Ions
1	Accelerated	N/A
2	Cause ionization	Formed
3	Retarded	Accelerated

Table 1: Processes occurring in each region of the combined ion/electron gun

Within the electron gun, the electrons are accelerated by a small amount to ensure efficient ionization of the noble gas (usually argon). This gas is injected directly into the gun and a sufficiently high flux density of ions is generated at a low chamber pressure. The ion beam is well collimated but still covers an area several times larger than the electron beam. The electron beam is retarded to its original energy of a few eV before leaving the gun.

In this way, charge neutralization is rapid and easy, on even the largest samples.

Digital Power Supply

The combination ion/electron gun is controlled using a digital power supply, operated from the *Avantage* data system. All of the operating parameters are controlled this way and their values can be stored for future use on all similar samples.

Conclusion

This new design is now available on all Sigma Probe and Theta Probe instruments and represents a major improvement in small spot XPS performance.

The combination ion/electron gun is suitable for a wide range of samples and can be used without the need for masks or grids. This type of charge compensation system does not put any restriction upon the size of the sample.

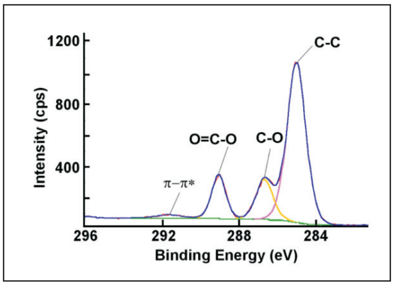

Figure 6: PET spectrum from Theta Probe

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