Sample cleaning using Ar-GCIS

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Ar-GCIS, Sample Cleaning, Surface Chemical Restoration

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Ar-GCIS as an effective tool for surface cleaning

Typical samples are often presented for analysis following transportation in a less than ideal environment. This results in an analysis and measured surface composition that is not representative of the true surface of the original material. There are a limited number of methodologies available that can effectively clean the sample and restore the original surface without inducing some additional chemical changes and thereby changing the very surface that is being investigated. The development of Ar-gas cluster ion source has changed this.

Ar-Gas Cluster Ion Source Novel design enhances capability

The Kratos Ar-Gas Cluster Ion Source provides a unique capability in that it can generate both monoatomic Ar^+ ions and Ar_n^+ ions (n = 100 - 2500) over a wide range of ion beam energies (200eV – 20KeV depending upon the mode). This broad capability makes it ideal for sample surface cleaning applications (amongst others), where material removal with no loss of chemical information is desired. The ion source, shown in Fig. 1, can be easily installed on the AXIS instruments, and is fully controlled by the ESCApe software.

The analysis of a typical, commercially supplied Polyethylene Terephthalate (PET) sample usually exhibits a low level of surface contamination as demonstrated in the XPS survey scan shown in Figure 2. The spectrum shows, in addition to the expected Carbon and Oxygen, low levels of Nitrogen ~(2.5% atomic) and 0.35% of Calcium.

Figure 1: Ar-GCIS source for the AXS Supra.

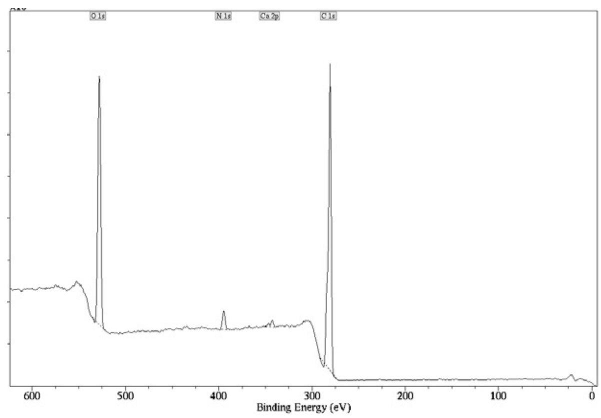

Figure 2: As received XPS survey spectrum from PET surface.

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The high resolution C 1s spectrum from this surface also reveals that there's an increased level of hydrocarbon on the surface, as the expected peak shape/composition isn't achieved, (Figure 3).

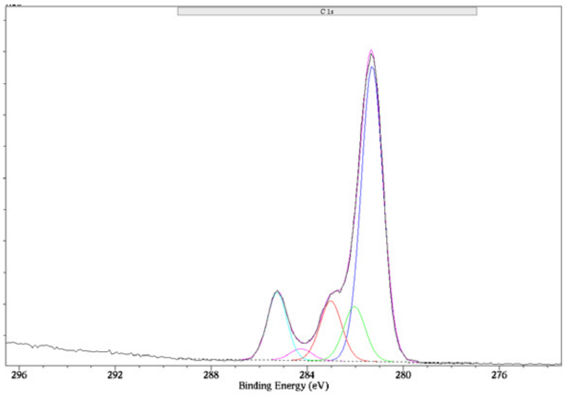

Figure 3: High resolution C 1s 'as received' surface.

Following the acquisition of the spectra, the sample surface was cleaned using Ar_n^+ clusters (n = 1000, beam energy = 2.5KeV, sputter time = 10 minutes). The effect of this cleaning process is shown in Figure 4.

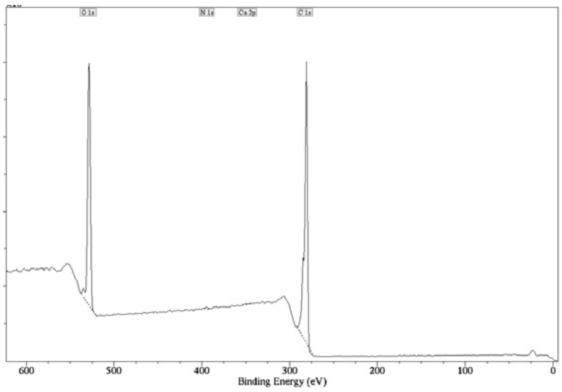

Figure 4: Survey spectrum following surface cleaning using Ar-GCIB.

It is clear from the survey spectrum that the surface composition has been changed and that the previous contamination has been completely removed. The question remains however as to whether any surface chemical damage has been done during the sputtering process. Figure 5 shows the high resolution C 1s spectrum following the surface cleaning process.

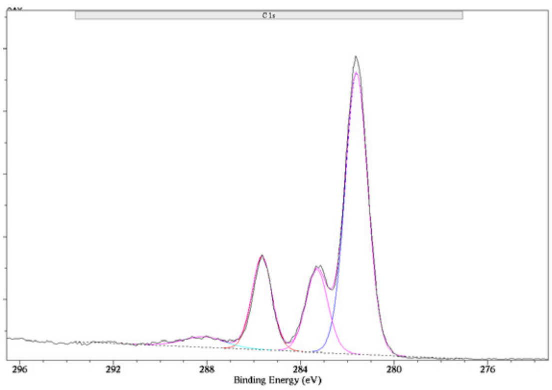

Figure 5: High resolution C 1s spectrum following surface cleaning by Ar-GCIS

It is clear from the spectrum that no chemical damage has been done to the surface of the PET. The expected peak shape has been measured, with good resolution further indicating that no damage has been induced.

In order to further demonstrate that the Ar-GCIB sputtering process does not induce any chemical damage, a fresh area of the PET sample was irradiated with low energy monoatomic Ar^+ ions (500eV beam energy for 3 minutes). The resulting spectrum is shown in Figure 6. It is clear from this spectrum that significant chemical damage has been done to the surface, with a significant increase in the C-C (hydrocarbon) peak and the evolution of additional C-O species.

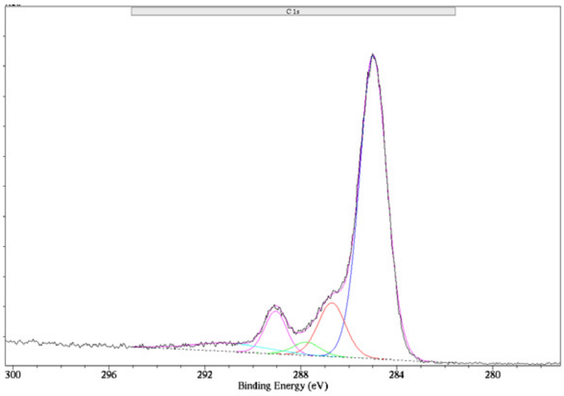

Figure 6: High resolution C 1s spectrum following low energy Ar ion sputtering.

This chemically modified surface was then re-sputtered using the same Ar-GCIB beam as earlier (n = 1000, 2.5KeV beam energy). A series of spectra were recorded to monitor the progress of the sputtering process.

After sputtering the sample for several minutes, the high resolution C 1s spectrum shown in Figure 7 was obtained.

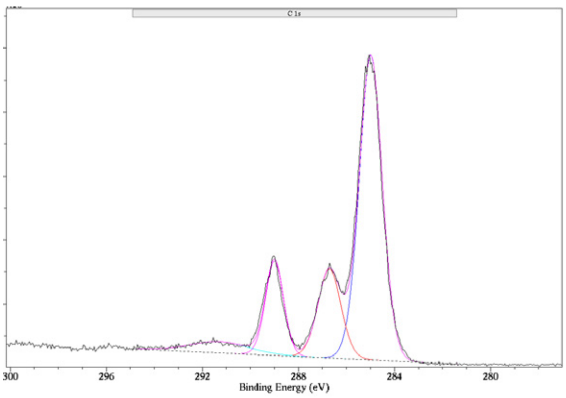

Figure 7: Final C 1s spectrum following Ar-GCIB sputtering.

It is clear from the spectrum that the original C 1s spectrum typical of PET has been completely restored indicating that the cluster beam sputtering process can be used to clean and restore sample surfaces with no chemical degradation.

Conclusion

The Kratos Ar-GCIS sputtering source is ideally suited to sample cleaning and surface chemical restoration. Even after significant chemical damage has been induced into the sample surface, sputtering with the cluster source can effectively eliminate it.

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