Application Note

PHYSICAL ELECTRONICS

Thin Film Analysis of POLYMER ADDITIVE MIGRATION Using the PHI Model 06-C60 Sputter Ion Gun

INTRODUCTION

In this application note we will show how a PHI *Quantera* XPS (x-ray photoelectron spectroscopy) microprobe, equipped with a unique C_{60} sputtering capability, was used to obtain a sputter depth profile from the surface region of a polyurethane plastic part and detect a thin layer formed by a wax additive that migrated to the surface of the part.

Plastic parts often contain additives to enhance desired physical properties. For example PTFE, graphite, or wax may be added to reduce wear or to limit the penetration of liquids. Migration of additives to a plastic part's surface may further enhance a desired property or create unforeseen problems. The ability to detect and quantify the migration of an additive to the surface is essential when developing new materials or processes for the manufacture of high performance plastic parts.

EXPERIMENTAL

A polyurethane plastic part that contained a wax additive was manufactured using a standard process that included a bake prior to its final use. A C_{60} sputter depth profile was obtained using a PHI *Quantera* XPS Microprobe and a PHI Model 06-C60 sputter ion gun (figure 5). The 06-C60 produces 10 keV C_{60}^{+} ions for sputter etching. The *Quantera's* system geometry provides a low angle of incidence which has been shown to minimize

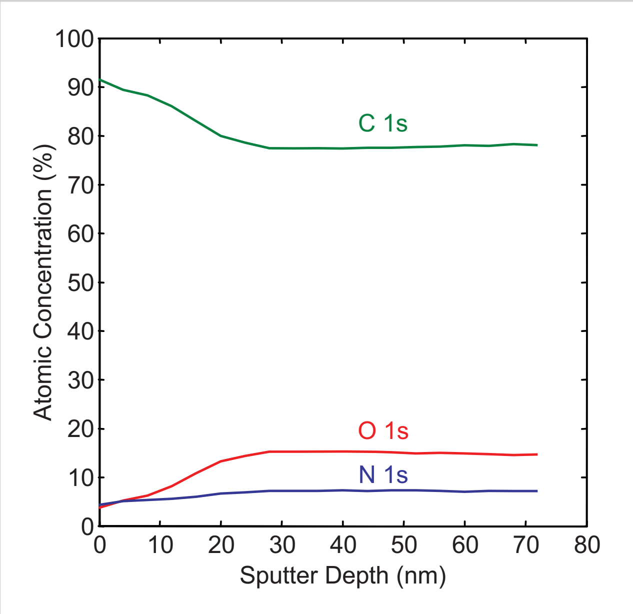

Fig. 1: C_{60} depth profile of a polyurethane plastic part that shows carbon enrichment at the surface of the part.

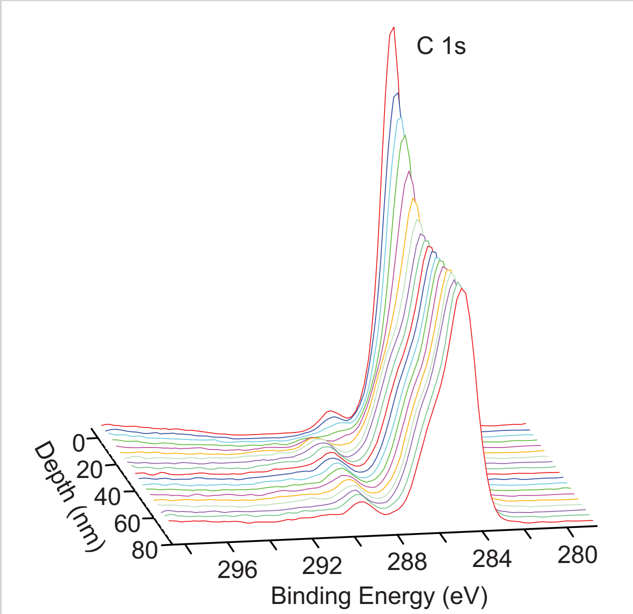

Fig. 2: XPS spectra from the C_{60} sputter depth profile show the presence of a thin wax layer on the surface of the polyurethane plastic part.

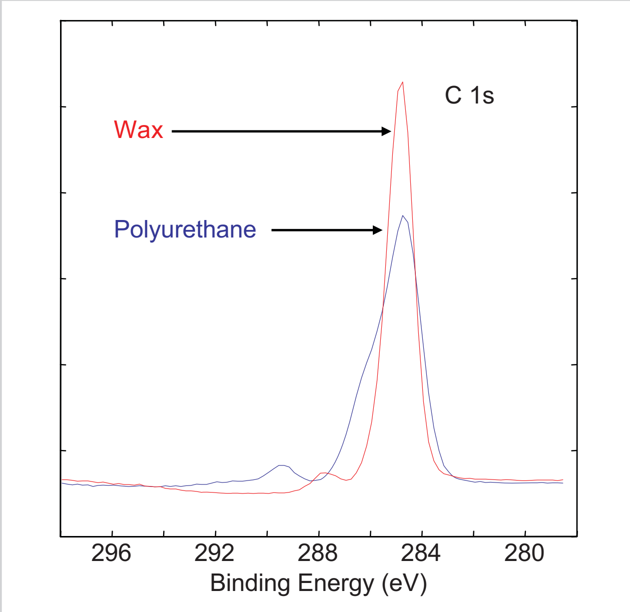

Fig. 3: Carbon 1s reference spectra extracted from the depth profile data set shown in figure 1 are used by the Linear Least Squares fitting algorithm in PHI *MultiPak* to create a chemical state sputter depth profile.

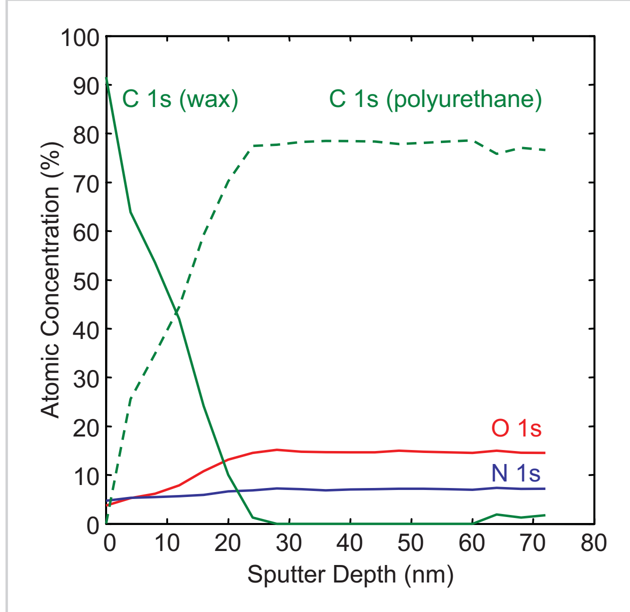

Fig. 4: Chemical state depth profile, showing the presence of a thin wax layer on the surface of the polyurethane plastic part.

chemical damage. The estimated etch rate for the wax was approximately 8 nm per minute for the conditions used. Charge neutralization was accomplished using PHI's patented dual beam charge neutralization method.

RESULTS

The elemental sputter depth profile displayed in figure 1 shows the presence of a 10-15 nm thick surface layer that is carbon rich and depleted in N and O. A careful examination of the carbon 1s data (figure 2) shows the spectra in the surface layer have a different signature than the spectra from the polyurethane substrate. Using the Linear Least Squares (LLS) fitting module in PHI *MultiPak*, the reference spectra shown in figure 3 were extracted from the depth profile data set. The results of the LLS fitting process using these reference spectra, shown in figure 4, transform the elemental profile for carbon into a chemical state depth profile for wax and polyurethane.

C_{60} VERSUS ARGON SPUTTERING

Until recently, it has not been possible to sputter depth profile through an organic layer and obtain useful chemical information due to the significant chemical damage caused by sputtering organics and polymers with commonly used ion sources such as Ar^+. To illustrate the benefits of C_{60} sputtering for this application, 10 keV C_{60} and 500 eV Ar sputter depth profiles were obtained from a control sample of the material used to manufacture the polyurethane plastic part. The C_{60} sputter depth profile data in figures 6 and 7 show relatively uniform composition with depth and stable chemistry (peak shape) with depth. In contrast, the Ar sputter depth profile (figures 8 & 9) shows an immediate loss of O and N after sputtering and a dramatic change in observed chemistry after sputtering with Ar.

Fig. 5: PHI Model 06-C60 sputter ion gun

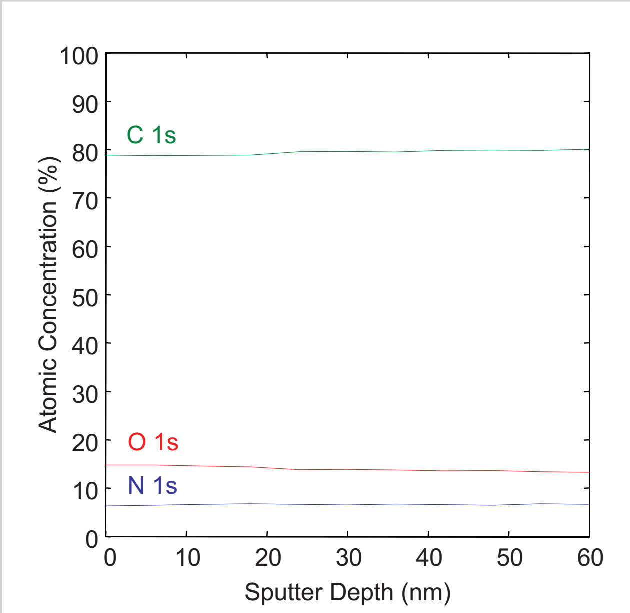

Fig. 6: 10 keV C_{60} sputter depth profile of a polyurethane control sample showing stable composition.

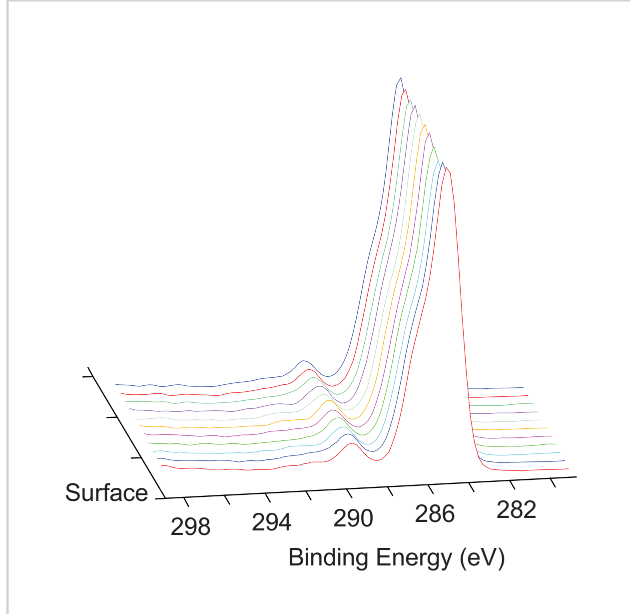

Fig. 7: Carbon 1s spectra from the C_{60} depth profile of the control sample showing minimal chemical damage.

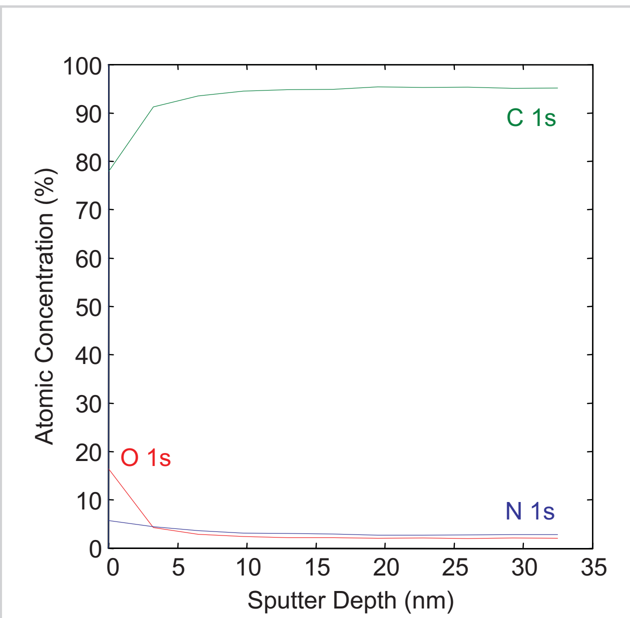

Fig. 8: 500 eV Ar^+ sputter depth profile of a polyurethane control sample showing a loss of oxygen and nitrogen after sputtering.

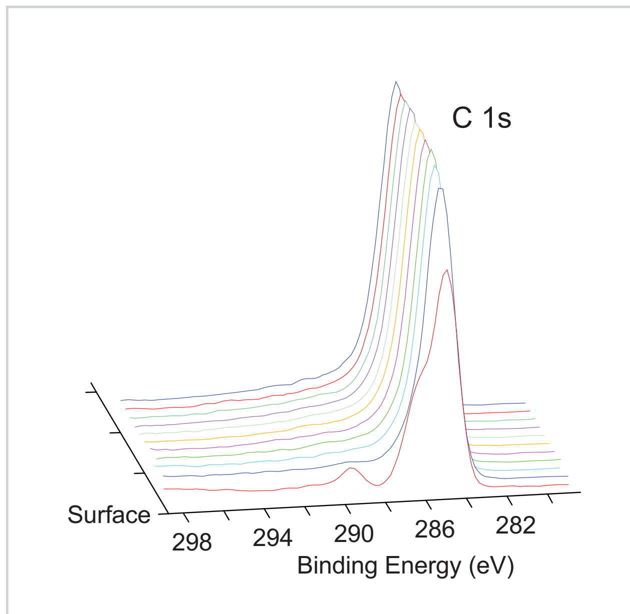

Fig. 9: Carbon 1s spectra from the Ar^+ depth profile showing significant chemical damage after the first sputter cycle.

SUMMARY

We have shown that a PHI *Quantera* XPS Microprobe equipped with a PHI 06-C60 sputter ion gun can be used to perform thin film analysis of organic and polymer layers with minimal chemical damage. In this example, a thin wax layer was removed and the polyurethane substrate exposed, providing a thickness measurement of the wax layer and quantitative chemical state analysis of both the wax layer and the substrate below.

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