

K-Alpha: XPS Analysis of a Dry Film Photo-Resist

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Key Words

- K-Alpha
- Photo-Resist
- Surface Analysis
- XPS

The Thermo Scientific K-Alpha was used to analyze the interface between a dry film photo-resist layer and its protective polypropylene layer. When the protective layer was peeled away, it was found that some photo-resist material was also removed.

Introduction

Dry film photo-resists are increasingly used in photolithography applications, such as the manufacture of integrated circuit boards.¹ The photo-resist often forms part of a laminated stack, with a polymer substrate (such as PET) and a protective layer (such as polypropylene). The protective layer is peeled away during use, but the efficacy of this process will depend on the properties of the dry film-protective layer interface. These properties in turn can be investigated by analyzing the surfaces of the dry film and protective layers after peeling. X-ray photoelectron spectroscopy (XPS) is the ideal analytical technique for this purpose, combining surface sensitivity with chemical selectivity.

To fully characterize the polymer surfaces after peeling, it may be necessary to detect and distinguish subtle differences in carbon and oxygen bonding states. Additionally, since the polymers are insulators, it is necessary to neutralize the electrical charge which builds as a result of X-ray analysis. This requires an XPS tool which combines turn-key charge neutralization with high sensitivity and excellent energy resolution.

The Thermo Scientific K-Alpha was used to investigate the surfaces formed by peeling apart the polypropylene protective layer from a dry film. The dry film was mounted on a PET substrate, as shown in Figure 1. The chemistry of the dry film was also investigated with K-Alpha.

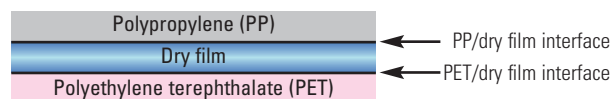


Figure 1: Schematic of dry film photo-resist with protective polypropylene layer and PET substrate



Experimental

The photosensitivity of the dry film means that the XPS analysis must be carefully controlled if accurate chemical information is to be obtained. Once the protective layer has been removed, and the dry film exposed, the sample must be transferred to the analysis chamber as quickly as possible. This will minimize any possibility of chemical degradation in the surface by exposure to ambient UV light. The Thermo Scientific K-Alpha enables rapid transfer of samples from atmosphere to the analysis chamber. Most polymer samples can be transferred within ten minutes, which maximizes throughput and productivity, as well as protecting the sample.

Results

Dry Film Photo-Resist Surface

XPS analysis of the dry film surface after the polypropylene layer had been peeled away showed that it was composed of carbon and oxygen; no other elements were detected. A high energy resolution XPS spectrum of carbon (Figure 3) reveals the chemical bonding states in the surface.

Peak fitting of the raw data in the Avantage datasystem (the Thermo Scientific integrated software solution for all of its XPS systems) indicates that the dry film surface could be a mixture of partially esterified cellulose (giving rise to the C-C*=O, C-O and C*-C=O components) and aliphatic carbon (C-C).

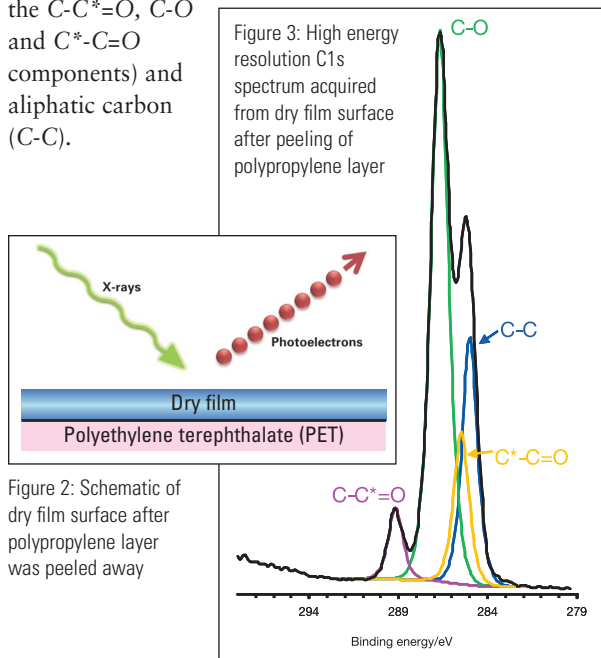


Figure 2: Schematic of dry film surface after polypropylene layer was peeled away

Figure 3: High energy resolution C1s spectrum acquired from dry film surface after peeling of polypropylene layer

Protective Layer (polypropylene) Surface

The carbon spectrum of a pure polypropylene surface should have a single asymmetric peak, which can be deconvoluted into contributions from CH₂ and CH₃ groups.² The surface of the protective layer, which was formerly in contact with the dry film, was analyzed (Figure 4) and it was found that the carbon spectrum was not that of pure polypropylene.

Components due to the dry film were observed in the carbon spectrum acquired from the peeled polypropylene surface (Figure 5).

The Avantage datasystem was used to quantify the relative proportions of dry film residue and polypropylene.

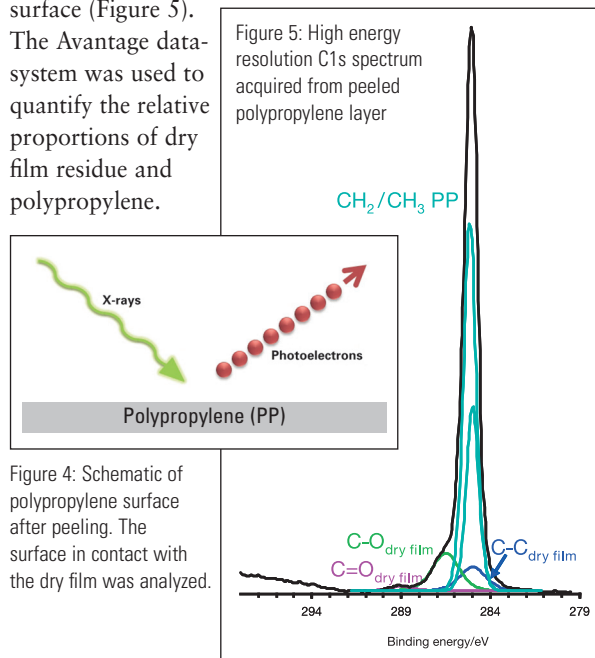


Figure 4: Schematic of polypropylene surface after peeling. The surface in contact with the dry film was analyzed.

Figure 5: High energy resolution C1s spectrum acquired from peeled polypropylene layer

The peak fitting protocol for the dry film was applied to the carbon spectrum from the peeled propylene surface. Two further components were added, with relative binding energies, peak widths and peak intensities appropriate for pure polypropylene.² The Avantage datasystem then allowed the XPS spectrum to be fitted in terms of the total area for pure polypropylene and the total area for the dry film. The composition of the surface was found to be four parts polypropylene to one part dry film.

Summary

Thermo Scientific K-Alpha was used to investigate the surfaces formed by peeling apart the polypropylene protective layer from a dry film. Material transfer from the dry film to the polypropylene layer was observed. XPS analysis of the dry film indicated it was similar to partially esterified cellulose.

References

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2. High Resolution XPS of Organic Polymers, The Scienta ESCA300 Database, G Beamson and D Briggs, 1992, Wiley, Chichester

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