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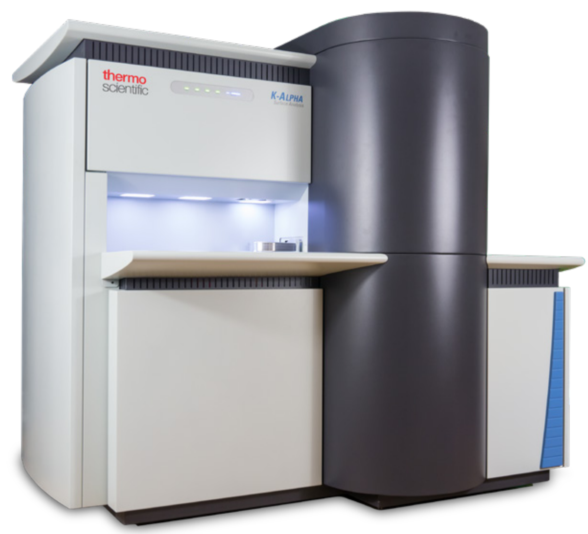
APPLICATION NOTE

Using X-ray photoelectron spectroscopy to investigate the surface treatment of fabrics

The Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) Surface Analysis System was used to investigate the surface treatment of a polyester fabric. Prior to the application of the fabric protector, the sample had been masked so that treated and untreated areas could be compared. The surface was imaged to determine the distribution of the chemistries.



Introduction

The modification of fabrics to add or enhance properties or performance is one of the most enduring areas of surface technology. Coatings have been applied to garments for many years to improve their waterproofing capabilities or to condition fibres so that they feel softer to the touch. In more recent times, methods have been used to apply nanomaterials to the surface of fabrics in order to improve such properties as UV protection factor, anti-bacterial capabilities, or stain resistance.[1]

X-ray photoelectron spectroscopy (XPS) is an analytical technique that is particularly well-suited to investigating these types of samples. XPS, also known as ESCA (electron spectroscopy for chemical analysis) delivers chemical composition information from the outer 10 nm of the surface of a material. It is capable of analyzing both conducting and insulating samples without special sample preparation, meaning that fabrics and fibers can be easily investigated using the technique (for another example, see Application Note 0145 for a description of the analysis of metal nanoparticles dispersed upon a fabric support).

Method

Half of a 25x50 mm piece of polyester lint-free cloth was sprayed with a soft furnishings protector, allowed to dry in air and then mounted on the standard 60x60 mm K-Alpha sample holder using four clips as shown in Figure 1. Once dry, the location of the fabric protector is not optically visible, so its location can only be determined analytically. This sample was designed to simulate a possible production failure, where a transparent coating had not applied consistently to a fabric.

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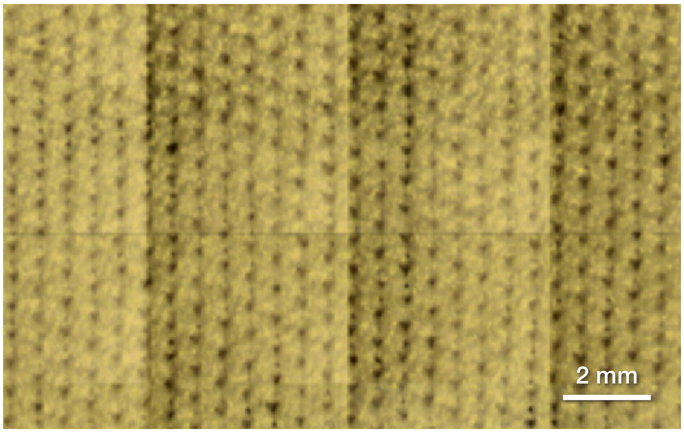

Figure 1: Mosaic view of polyester sample mounted on the K-Alpha sample holder.

A 400 µm X-ray spot was then used to analyze a point in each half of the sample to obtain quantified elemental data to compare the compositions of the untreated and treated areas of the sample. As the samples were insulating, the K-Alpha turnkey charge compensation system was used. This system replaces the electrons lost from the surface by the photoelectric effect, preventing a large surface charge from accumulating, which would distort the acquired data. It is not required for conducting samples but is essential for the analysis of insulators. The K-Alpha XPS System is extremely simple to use, while being fully capable of maintaining consistent analysis conditions across even the most challenging sample set.

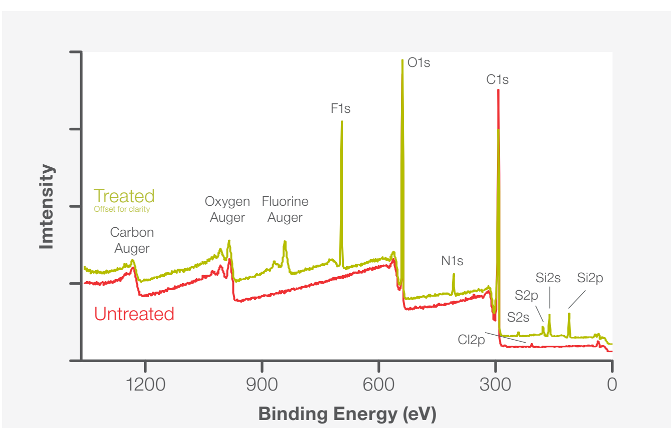

Figure 2: Overlay of survey scans on untreated and treated areas of the polyester sample.

A 16x12 mm area (shown in Figure 1) was then mapped using a 400 µm X-ray spot and a 400 µm step size to determine the distribution of the elements across the surface. The 128-channel detector was employed in snapshot acquisition mode to record the C1s, O1s, Cl2p, F1s, N1s, Si2p and S2p regions at every analysis position, minimizing the required total acquisition time, while retaining chemical information.

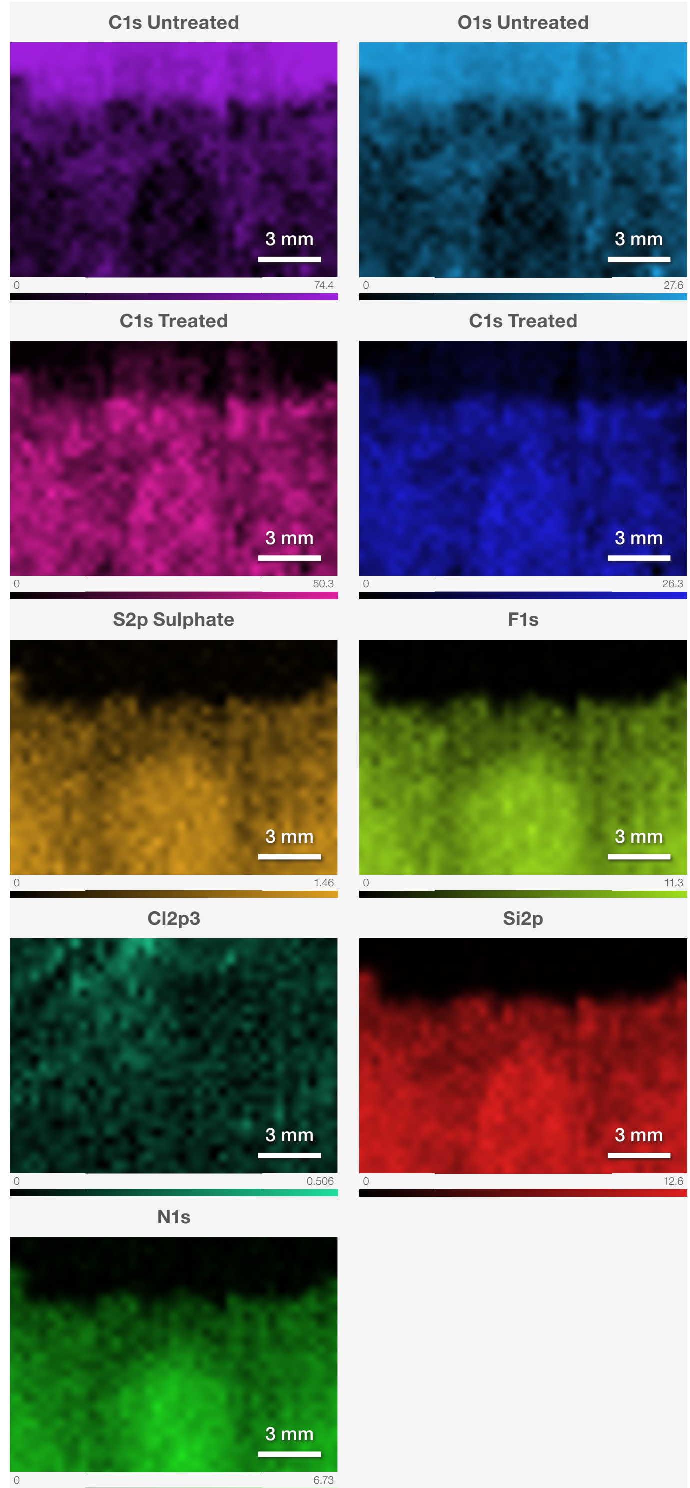

Figure 3: Atomic percent chemical maps of each chemical state over an interface area between treated and untreated parts of the sample.

Results

The survey scans giving elemental quantification from untreated and treated parts of the polyester sample are shown in Figure 2. The elemental composition of the untreated sample is consistent with cleaned polyester. The composition of the treated sample shows that the surface has changed dramatically. In particular, elements from compounds in the treatment can clearly be detected: fluorine and sulfur (from perfluorobutane sulfonic acid), silicon (from silicone) and nitrogen.

Atomic percent maps of the chemical states present in each of the elements detected in the survey scans are shown in Figure 3. The atomic percent chemical maps clearly show the untreated part of polyester in the top 3 mm and the treated part in the lower section.

Averaged C1s spectra from the treated and untreated side of the sample are shown in Figure 4. The untreated C1s spectrum shows the expected components for polyester. The treated spectrum shows a reduction in the amount of C-O and C=O, which are highly characteristic of the ester groups in the untreated part of the sample, and an increase in C-F bonding, which comes from the backbone of the perfluorobutane sulfonic acid in the fabric protector.

These chemical maps can then be overlaid on the optical view of the sample, to create the chemical map overlay in Figure 5.

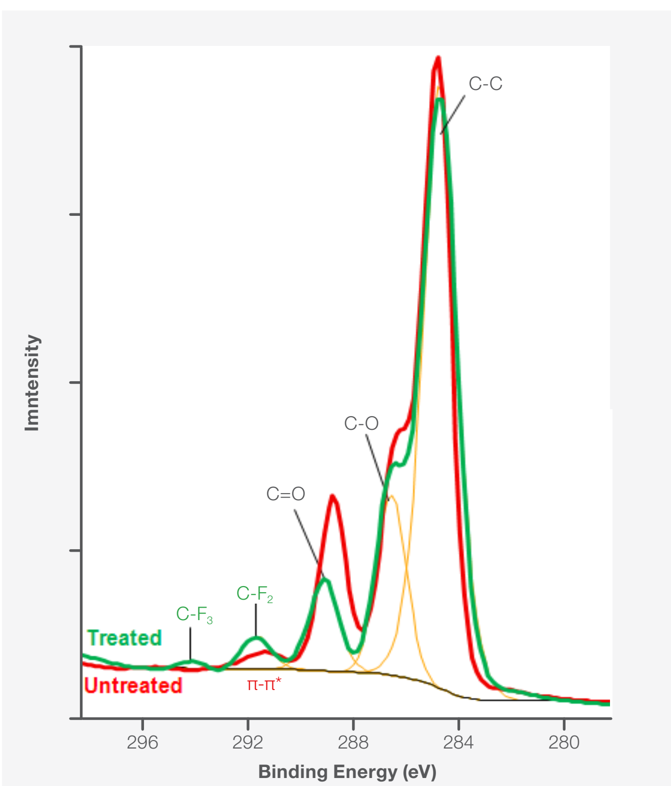

Figure 4: Averaged C1s spectra from the treated and untreated side of the sample.

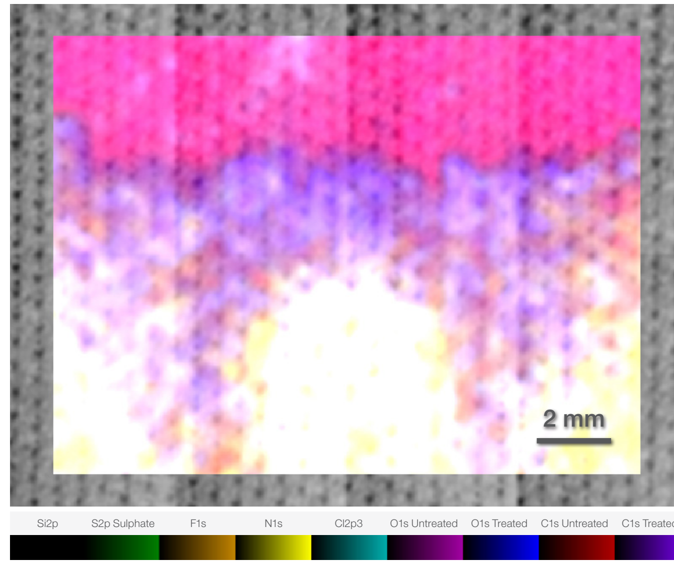

Figure 5: Atomic percent chemical maps overlaid on the optical image of the sample.

Summary

As XPS is a quantifiable, surface-sensitive, analytical technique, it is ideal for determining the areas where an invisible surface modification, such as a fabric protection coating, has been applied, and also for finding the amount of coating present.
It can also be used for quality control of invisible coatings on fabrics and other materials to determine whether they have been applied uniformly.

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Notes

Acknowledgements
We thank Tom Ross, a student at Oakwood School, Horley, UK, for his work in preparing and analyzing the fabric samples during his work experience placement with Thermo Fisher Scientific.

References

1. D. Mihailovic et al., Cellulose (2011), 18, 811-825

Keywords
K-Alpha, Coating, Fabric, Mapping, Surface Analysis, Surface Treatment, XPS

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