

TOTAL SOLUTIONS FOR THE PHOTOGRAPHIC INDUSTRY

CSMA



The surface chemistry of photographic film materials is complex and subject to a wide range of manufacturing problems. The identification of their cause generally requires sensitive, molecularly-specific analytical tools with high spatial resolution. Modern surface analysis techniques such as XPS (X-ray Photoelectron Spectroscopy), ToFSIMS (Time-of-Flight Secondary Ion Mass Spectrometry) and DSIMS (Dynamic Secondary Ion Mass Spectrometry) are particularly suited to provide such types of information. The case studies below show how CSMA has been involved in helping manufacturers to troubleshoot existing processes and augment research and development efforts.

BENEFITS TO CUSTOMER

- Cost effective - one day of analysis can often identify the cause of product failure
- Rapid information - clear decisions can be made to take remedial action to improve processes
- Validation of new processes by comparison to predicted models
- Assessment of competitors' products - reverse engineering



CASE STUDY ONE

Uniformity of Gelatine-Coated Photographic Paper

The analysis was undertaken to investigate the distribution of gelatine on the polyethylene layer of laminated photographic paper. Four gelatine coated matt paper samples with decreasing surface roughness (graded in the order A to D) were analysed using SIMS imaging. The distributions of the species were assessed by monitoring the CN^- and CH^- signals characteristic of gelatine (protein-based) and polyethylene respectively.



Sample A



Sample B



Sample C



Sample D

Overlay SIMS images for the four matt laminate paper samples showing the relative distribution of gelatine (CN^- , red) and polyethylene (CH^- , cyan). Field of view $\sim 300 \mu m \times 300 \mu m$.

The results show that gelatine coverage of the paper increases as the surface roughness decreases. There is evidence of 1-10 μm size exposed areas of polyethylene on samples B, C and D with some significantly larger exposed areas on A. This result is entirely consistent with a gelatine "coatability" model proposed by the photographic paper manufacturer whereby the roughest surfaces have extreme height areas uncoated by gelatine.



Discolouration of Steel Sheets used in Photo Film Canisters

The purpose of this analysis was to identify the cause of brown spots and a yellow discolouration on a defective steel sheet. Initial SEM-EDX analyses, conducted by the customer, identified the brown spots as iron oxide with an additional indication of a deficiency in the 10nm thick, electro-coated chromium overlayer used to prevent corrosion. Surface analysis was commissioned at CSMA to further characterise the stains, identify the yellow colouration and a possible cause for the lower level of chromium on defective sheets.

SURFACE CHEMICAL COMPOSITIONS FROM XPS (ATOMIC %)

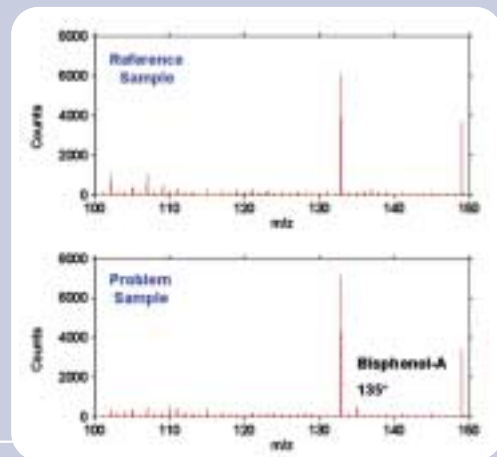
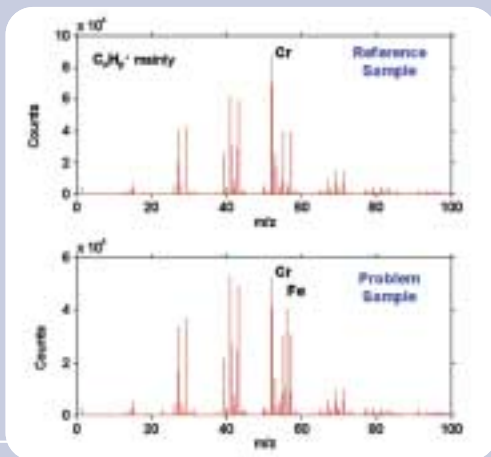
Sample	Fe	Cr	O	C	F
Reference sample	-	8.3	41.5	46.1	4.0
Problem sample (yellow area)	4.5	3.6	38.1	50.0	3.8
Problem sample (brown stain)	10.1	trace	44.1	45.8	-

The XPS data show that iron is not detected on the reference sample, indicating a chromium (and carbonaceous) overlayer of thickness greater than 5 nm. By contrast, iron oxides are detected in both yellow coloured and brown stain areas, indicating oxidation of the whole surface of the steel sheet.

HIGH RESOLUTION XPS - CHROMIUM AND IRON (2p) OXIDATION STATES

Sample	Cr (metal)	Cr (oxides)	Fe (metal)	FeO	Fe ₂ O ₃	Fe ₃ O ₄
Reference sample	25%	75%	-	-	-	-
Problem sample	0%	100%	0%	40%	37%	23%

High resolution chromium XPS spectra show only oxidised chromium on the defect sample whereas a metallic state is present on the reference sample, indicating an intact, protective chromium overlayer. ToF-SIMS confirms high levels of iron (and low levels of chromium) on the defect sample (see spectra). In addition, for the defect sample only, a bisphenol-A containing species is detected. This contaminant is related to, or indeed the cause of, poor chromium deposition.



CSMA provides a complete surface analysis service to industry to accommodate every level of demand:

- rapid turnaround analysis (24 hours)
- problem solving and failure analysis
- litigation and expert witnesses
- training courses
- reverse engineering and competitor analysis
- materials and product development
- patent registration / infringement

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