Analytical Tools for investigating pressure sensitive adhesive-silicone interaction

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Adhesives Technology Session

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Agenda

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2. Overview Analytical Techniques
3. Case Study #1: Low Tack on Specialty Label
4. Case Study #2: Low Tack in Foam Tape
5. Case Study #3: Low Peel Medical Self-wound Tape
6. Conclusions

Overview

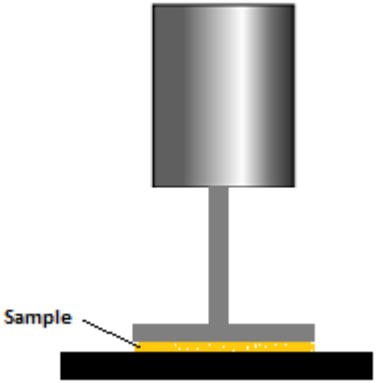
Pressure Sensitive Adhesives

- Pressure sensitive adhesives (PSA) are viscoelastic materials that flow and deform based on the temperature and stress level
- PSA's have a wide range of chemistries which affect the way they are coated
- Commonly PSA's are coated to a release liner (with surface treatment) and then laminated to the application substrate
- Adhesive-silicone release interactions are critical for end-use performance
- Analytical techniques help understand critical quality and performance issues

Rheology

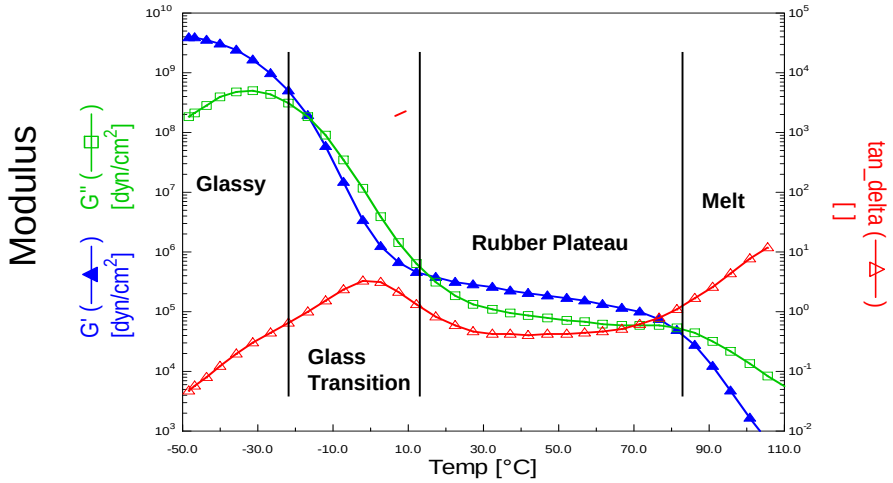
Temperature Sweep Dynamic Mechanical Analysis (DMA)

- Science of flow and deformation of matter
- Relationship between stress and deformation of the material
- $Viscosity = \frac{Stress}{Shear\ Rate}$
- $Modulus = \frac{Stress}{Strain}$
- Measure modulus at constant frequency and vary the temperature



Rheology

Temperature Sweep Dynamic Mechanical Analysis (DMA)



- Glass transition temperature – Tg - temperature at which polymers change from glassy to rubbery state
- Storage Modulus - G’ - energy storage (memory)
- Loss Modulus - G” - energy dissipation (loss)
- tan δ = G” / G’ - relaxation or deformation

G’ (Storage Modulus, Elastic modulus) → Hardness (Hard vs. Soft)

G” (Loss Modulus, viscous component) → Viscosity (Thick vs. Thin)

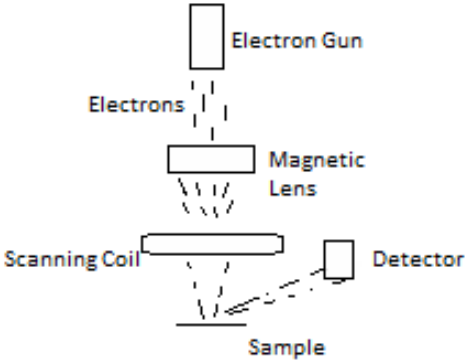
Tan δ → Balance of cohesive strength vs. flow

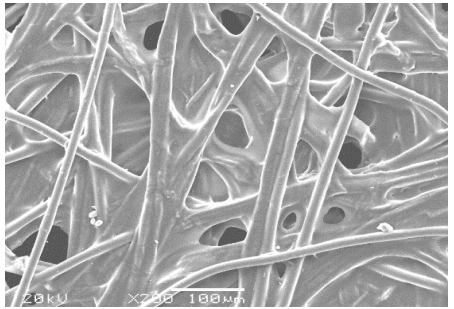
Tan δ peak height → Potential to wet out, tack

Tan δ peak temperature → Tg

Scanning Electron Microscope (SEM)

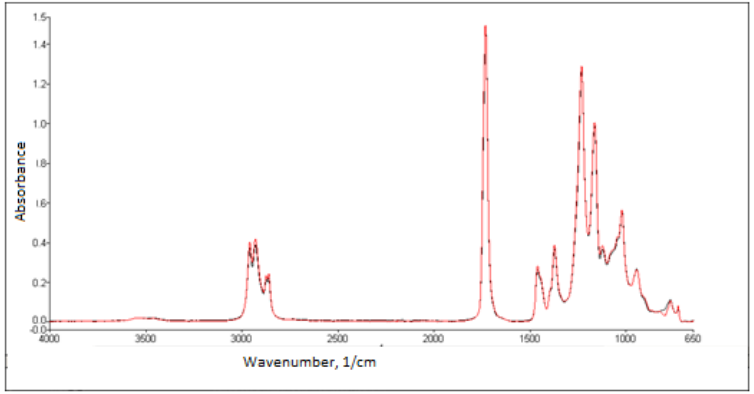
- Morphological Analysis
- Focused beam of high-energy electrons
- Generate a variety of signals at the surface of solid state materials.
- High resolution and good control in the degree of magnification





Fourier Transform Infrared Spectroscopy (FTIR)

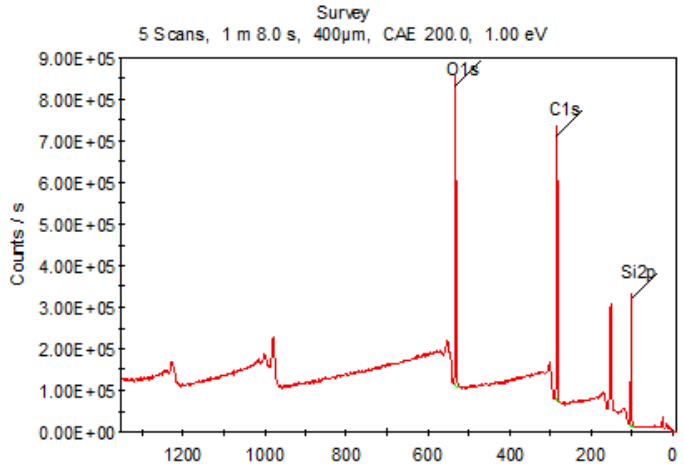
- Composition Analysis
- Vibrational spectroscopic technique
- Molecular stretching, vibration, and rotation of chemical bonds as they are exposed to designated wavelengths of light.



Verify composition of the construction, identify any changes in raw materials, or possible contamination.

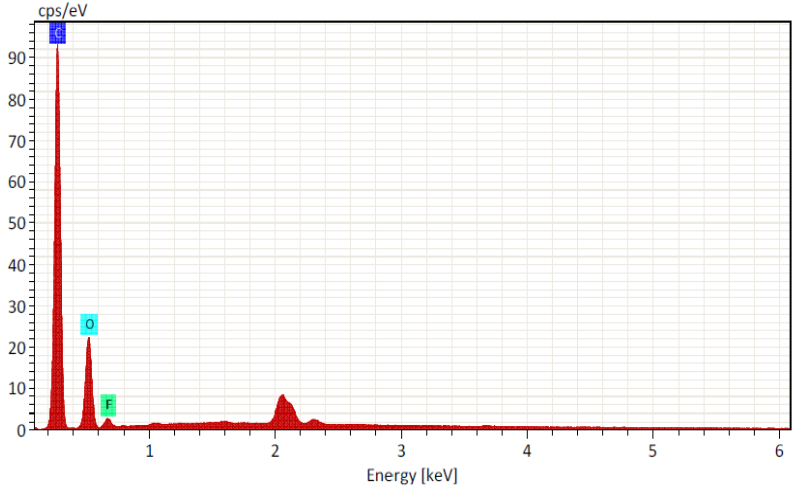
X-ray photoelectron spectroscopy (XPS):

- Quantitative spectroscopy technique
- Composition of sample surface
- Irradiate the sample surface, hitting the core electrons (e^-) of the atoms.
- The X-Rays penetrate the sample to a depth 1-12nm.
- Identify the chemical composition of the substrate
- Residual migration that can occur between the adhesive and substrates.



Energy-dispersive X-ray spectroscopy (EDS)

- Chemical microanalysis technique
- Elemental analysis
- Detects x-rays emitted from the to characterize the elemental composition
  - >500nm
  - Higher accuracy

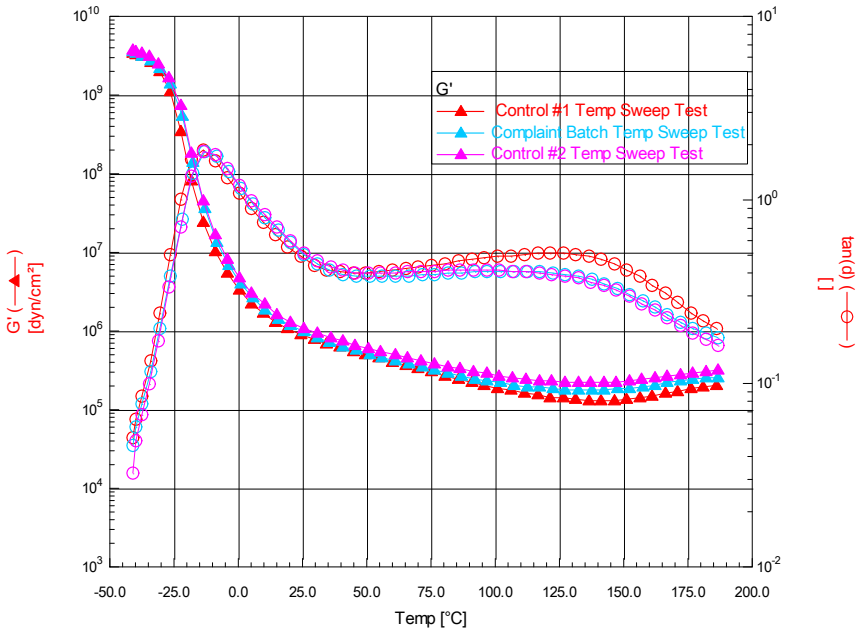


Case Study #1
Low Tack on Specialty Label

- Issue: Low tack on finished labels
- Application: High temperature label
- Adhesive: Solution Acrylic PSA
- Samples Analyzed: Two positive controls and one complaint sample of coated finished product
- Testing:
  - Performance
  - FTIR
  - Rheology
  - XPS

Case Study #1 Low Tack on Specialty Label

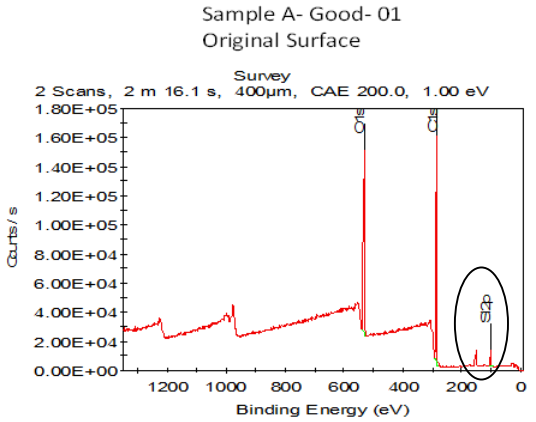
Analytical Technique #1 - Rheology



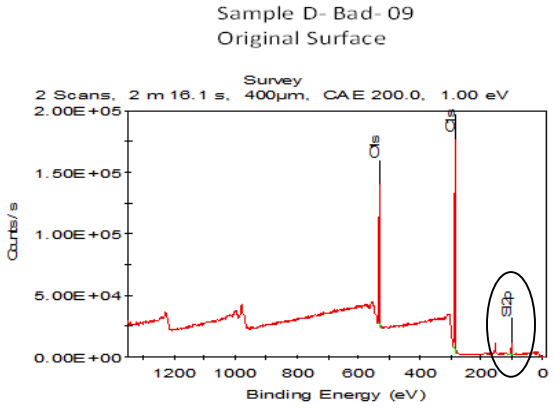
- Rheology curve for complaint lot fall between the curves of the control batches. FTIR was also tested and showed the same composition
- No major compositional differences from the control.

Case Study #1 Low Tack on Specialty Label

Analytical Technique #2 - XPS



Element	C	O	Si
Concentration(at%)	72.51	22.35	5.13

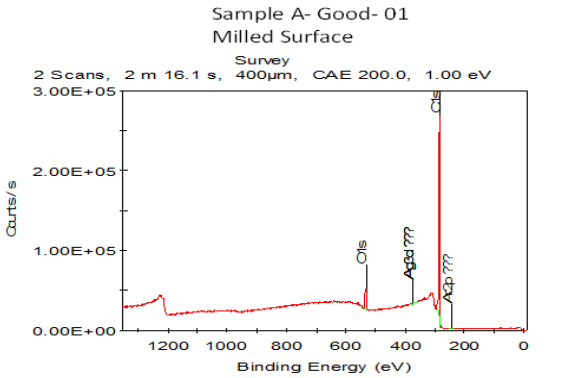


Element	C	O	Si
Concentration(at%)	75.2	25.37	4.43

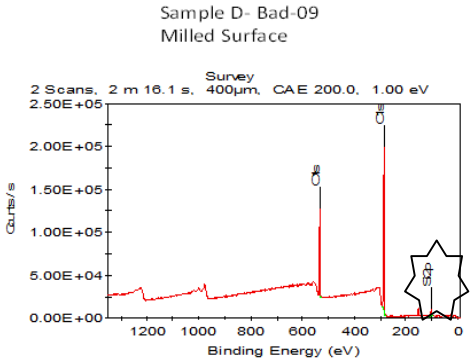
- Submitted for XPS for surface silicone identification
- All samples showed relatively the same amount of silicone residue on the initial adhesive surface

Case Study #1 Low Tack on Specialty Label

Analytical Technique #2 - XPS



Element	C	O	Si
Concentration(at%)	65.42	4.42	N/A



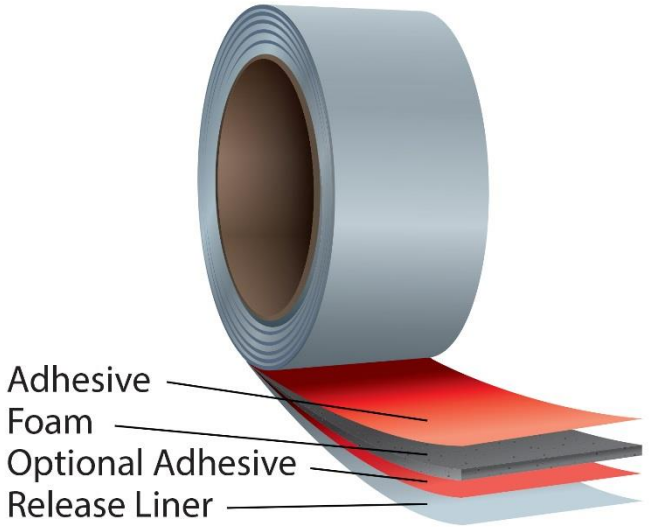
Element	C	O	Si
Concentration(at%)	77.37	18.17	4.46

- Further milled with argon ions for 5 minutes and then scanned by XPS again to see if any silicone remained on the surface.
- After ion milling complaint sample still had silicone on the milled adhesive surface
- Silicone penetrated deeper into the adhesive layer. Potentially some level of uncured silicone

Case Study #2

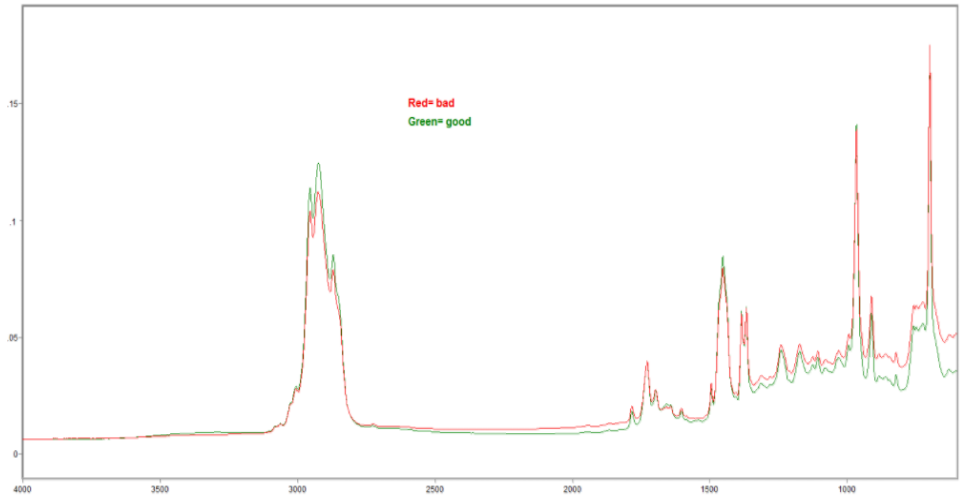
Low Tack in double-side Foam Tape

- Issue: Low Tack on the inside coating of the tape
- Application: Foam Tape
- Adhesive: Rubber-based PSA
- Samples Analyzed: One control, one complaint (multiple coated samples)
- Testing:
  - Performance
  - FTIR
  - SEM
  - XPS



Case Study #2 Low Tack in Foam Tape

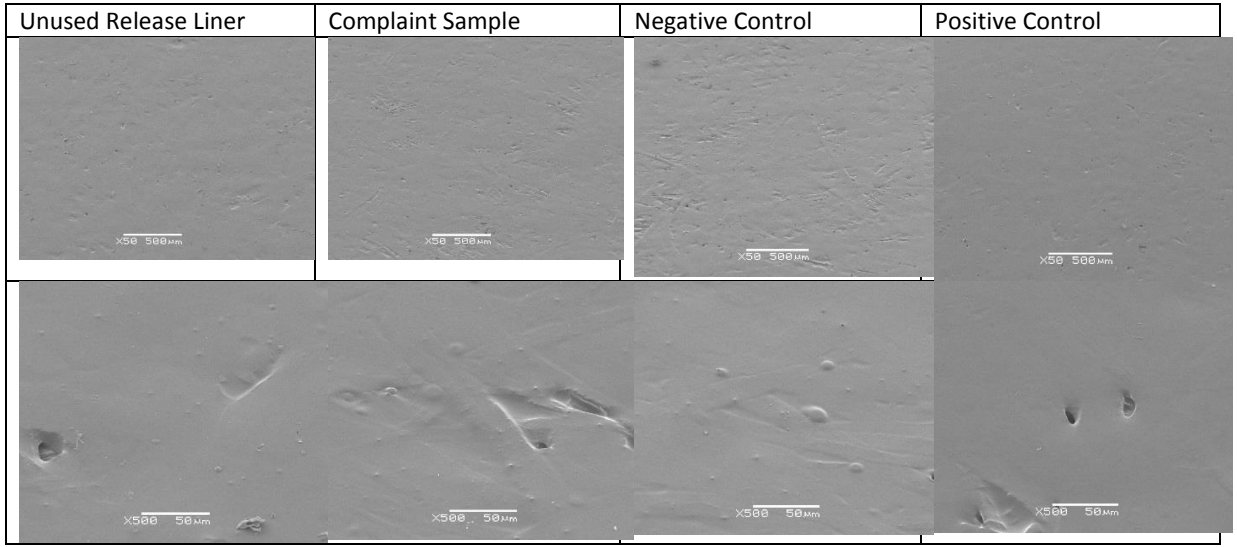
Analytical Technique #1 - FTIR



- IR spectra of the complaint adhesive was very similar to the adhesive on the control
- No notable differences in the composition of the adhesives.

Case Study #2 Low Tack in Foam Tape

Analytical Technique #2 - SEM



- Surface morphology is relatively consistent between the samples
- No evidence that liner morphology is the cause of the low tack issue.

Case Study #2

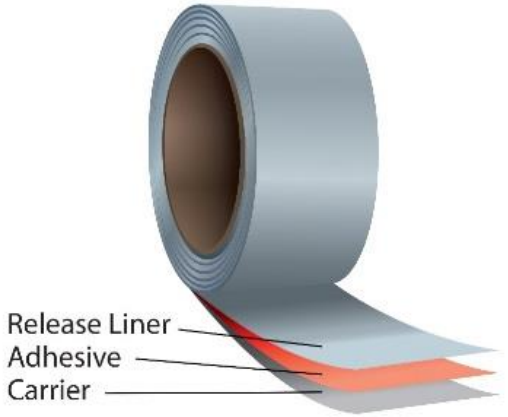
Further Direction

- Additional Tests:
  - X-ray Photoelectron Spectrometer (XPS): Constant silicon levels as a function of sample (i.e. control vs. complaint)
- Consistent silicone transfer from both sides of the liner
- The liner does not appear to be the root cause of the issue.
- Root cause did not lie with adhesive-silicone interface
- Results indicate more adhesive testing need to determine root cause
  - Analytical techniques helped investigation in the right path.

Case Study #3

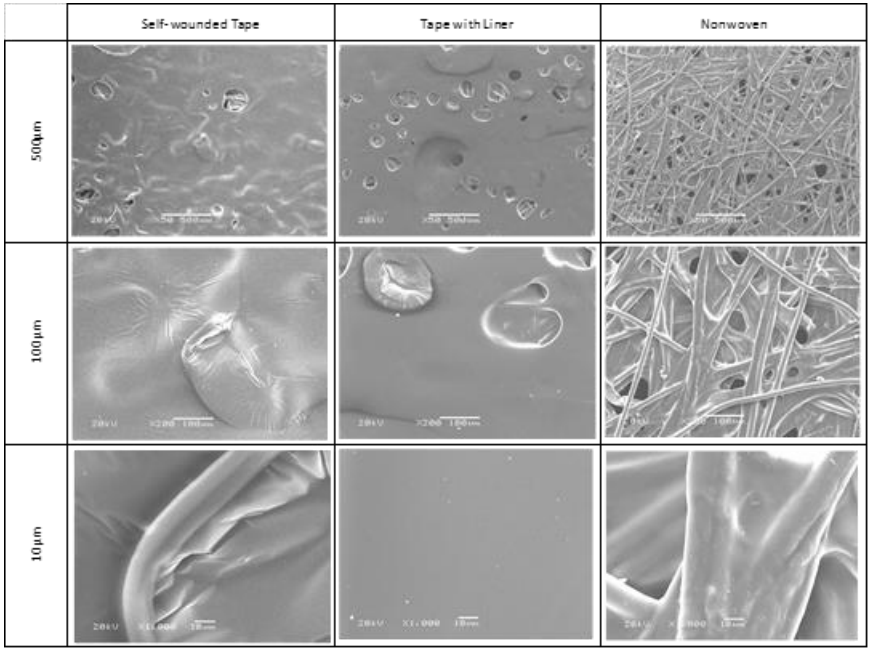
Low Peel Medical Self-wound Tape

- Issue: Low peel values
- Application: Self-wound medical non-woven tape
- Adhesive: Solution Acrylic PSA
- Samples Analyzed: self-wound tape, adhesive laminated to release liner, non-woven, liner
- Testing:
  - Performance
  - SEM
  - XPS
  - EDS



Case Study #3 Low Peel Medical Self-wound Tape

Analytical Technique #1 - SEM



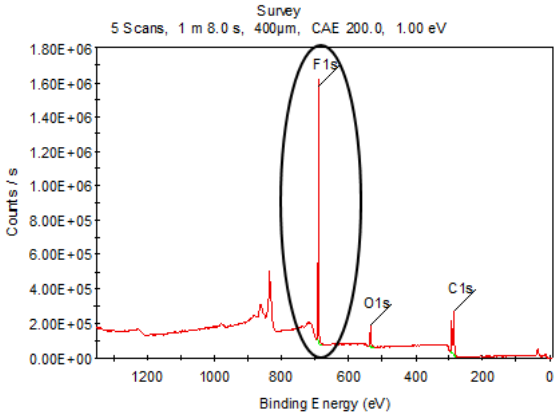
- Identify any surface differences between the finished self-wound tape, the tape backed with silicone liner and the un-coated non-woven substrate.

- No major differences between coated samples and non-woven

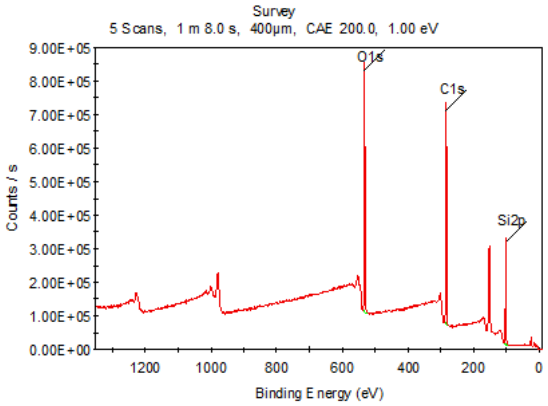
- Eliminate the idea that there could have been any issues with adhesive coating

Case Study #3 Low Peel Medical Self-wound Tape

Analytical Technique #2 - XPS



Surface of uncoated nonwoven



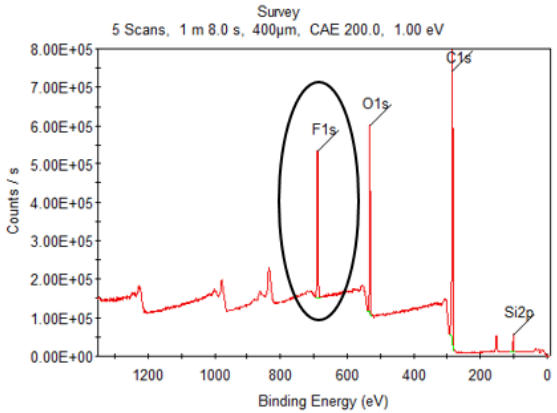
Surface of release liner

- Analyze interaction that may occur between the adhesive surface, the self-wound non-woven, and the release liner.

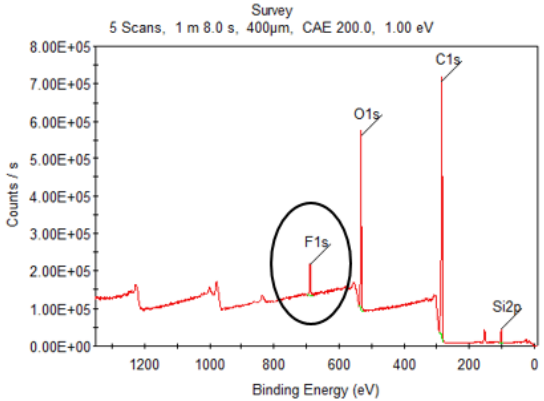
- High fluorine concentration in the surface of the non-woven

Case Study #3 Low Peel Medical Self-wound Tape

Analytical Technique #2 - XPS



Surface of the self-wound roll

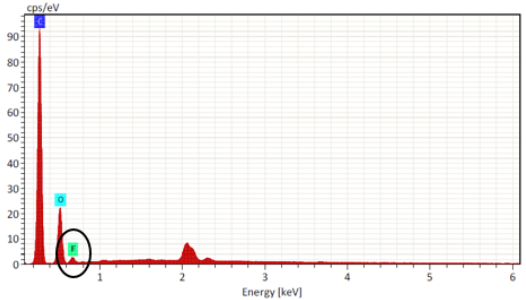


Surface of adhesive laminated with release liner

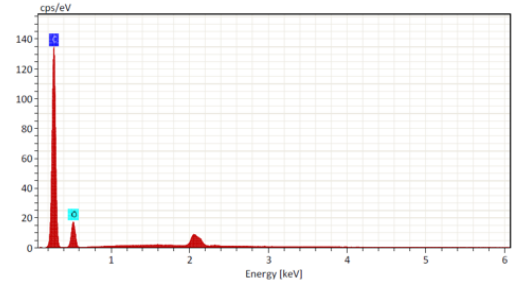
- Self-wound roll has higher fluorine content than sample laminated to the release liner.
- Adhesive could be removing the fluorocarbon treatment from the surface of the uncoated non-woven.

Case Study #3 Low Peel Medical Self-wound Tape

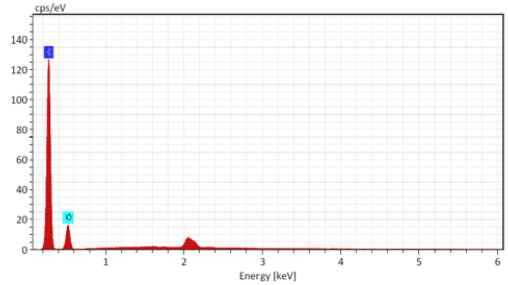
Analytical Technique #3- EDS



Nonwoven spectra



Self-wounded roll spectra



Sample laminated to release liner spectra

- Deeper analysis of the substrates and identify possible diffusion of fluorocarboned treatment from the non-woven to the adhesive surface.
- Presence of fluorine in the surface and inside the non-woven
- No traces of fluorine inside the adhesive in either of the two coated samples

No diffusion of the fluorocarboned release treatment from the non-woven to the adhesive surface.

Case Study #3 Low Peel Medical Self-wound Tape

Results

- Confirmed that the fluorine (fluorocarbon) source is the non-woven, release liner has no presence of this element.
- Higher concentration of fluorine on the self-wound roll, indicating that it can be removing the fluorocarbon treatment from the uncoated side of the non-woven.
- EDS showed no diffusion of the fluorine from the non-woven through the adhesive towards the surface.
- Fluorocarbon compounds are used as release agents, the presence of fluorine in the self-wound finished medical tape explain the low adhesion issue.

Summary

- Analytical techniques have proven to help troubleshoot issues that may occur during the adhesive coating process on siliconized liner at the silicone-adhesive interface.
- Importance of knowing when and which techniques to use
- First identify change in composition in the adhesive or raw material associated with the application (FTIR, DMAIII).
- Morphological differences between the good and bad samples (SEM)
- Further analysis of the elemental composition at the silicone-adhesive interface (XPS) and into deeper layers in the sample (EDS).

Acknowledgements:

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Thank you!

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