Mobile Fourier Transform Infrared Spectroscopy for Composites analysis

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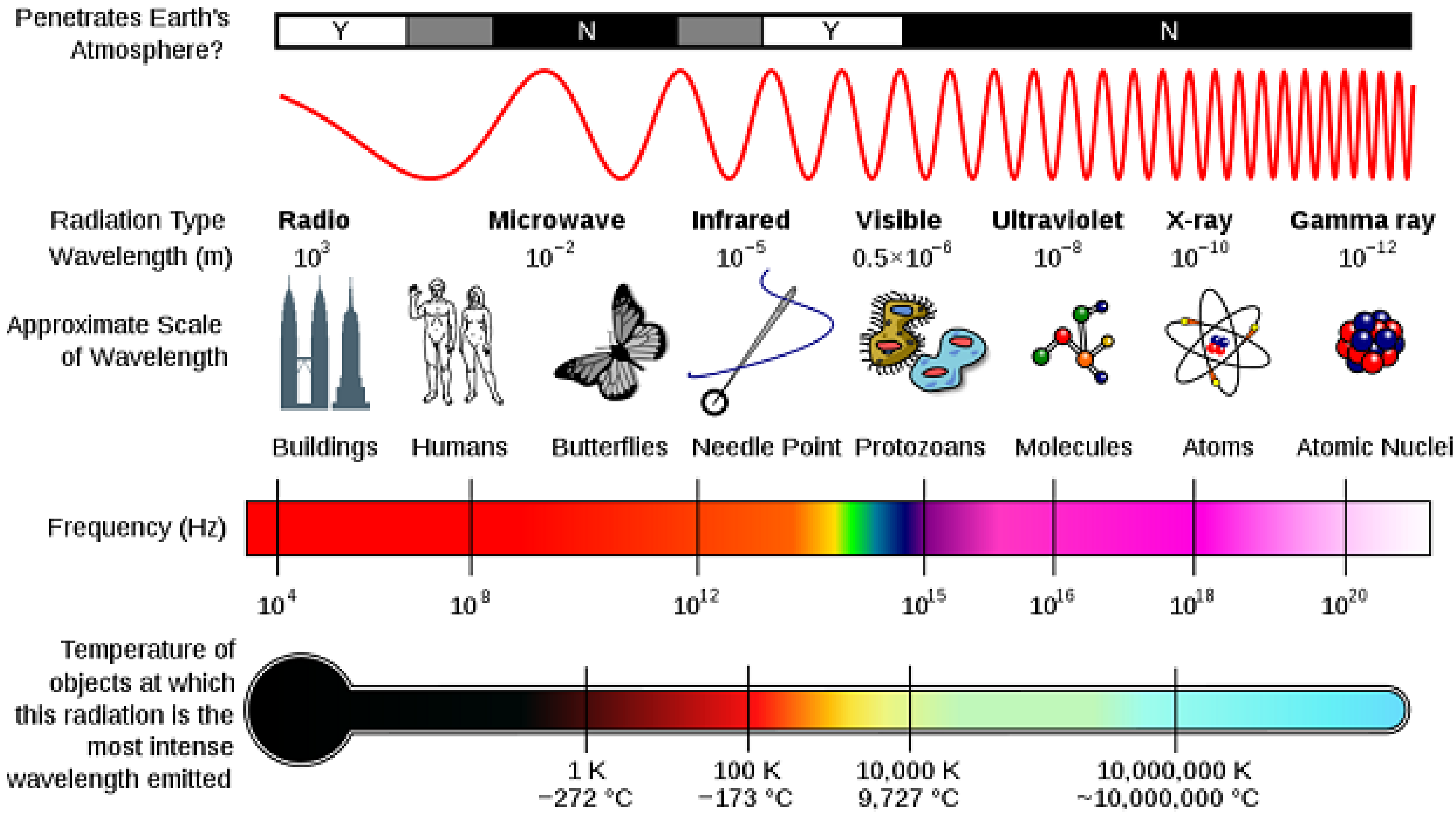
The Electromagnetic Spectrum

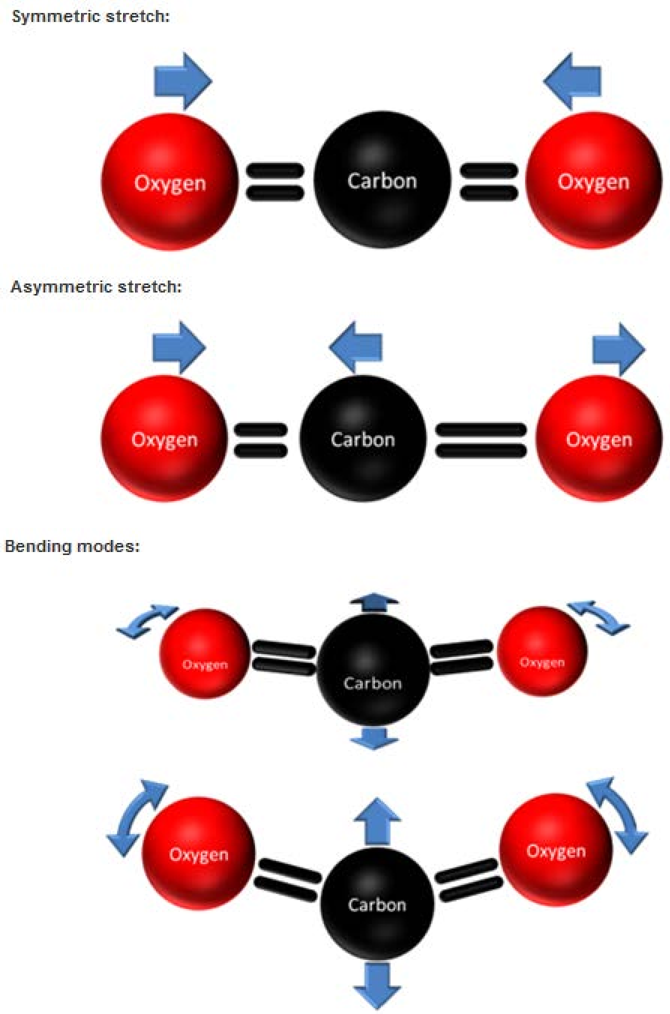


What is Infrared Spectroscopy (FTIR)?

Infrared light is shone through a thin slice of the sample you are investigating, and then the light is detected after it has emerged on the other side. **Comparing light before and after it passes through the sample shows which frequencies within the light have been absorbed**.

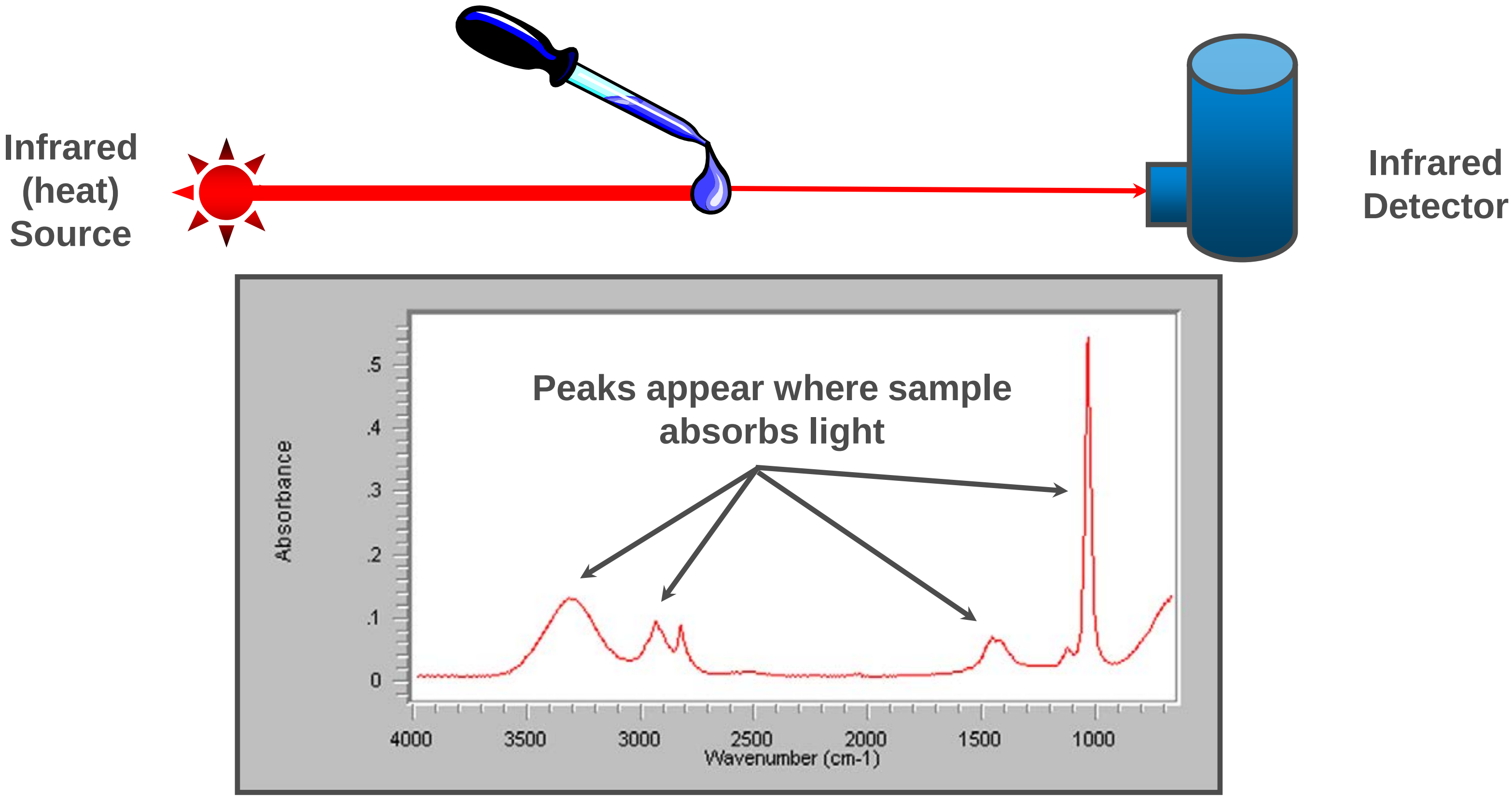
The material making up the sample will be full of chemical bonds. Chemical bonds vibrate in different ways (twisting, bending and stretching), depending on the type of bond present (e.g. CO_2, N_2).

Different types of vibrations absorb different frequencies of infrared radiation. By looking at which frequencies of the infrared light are absorbed by the sample, scientists can tell which chemical bonds are present within it

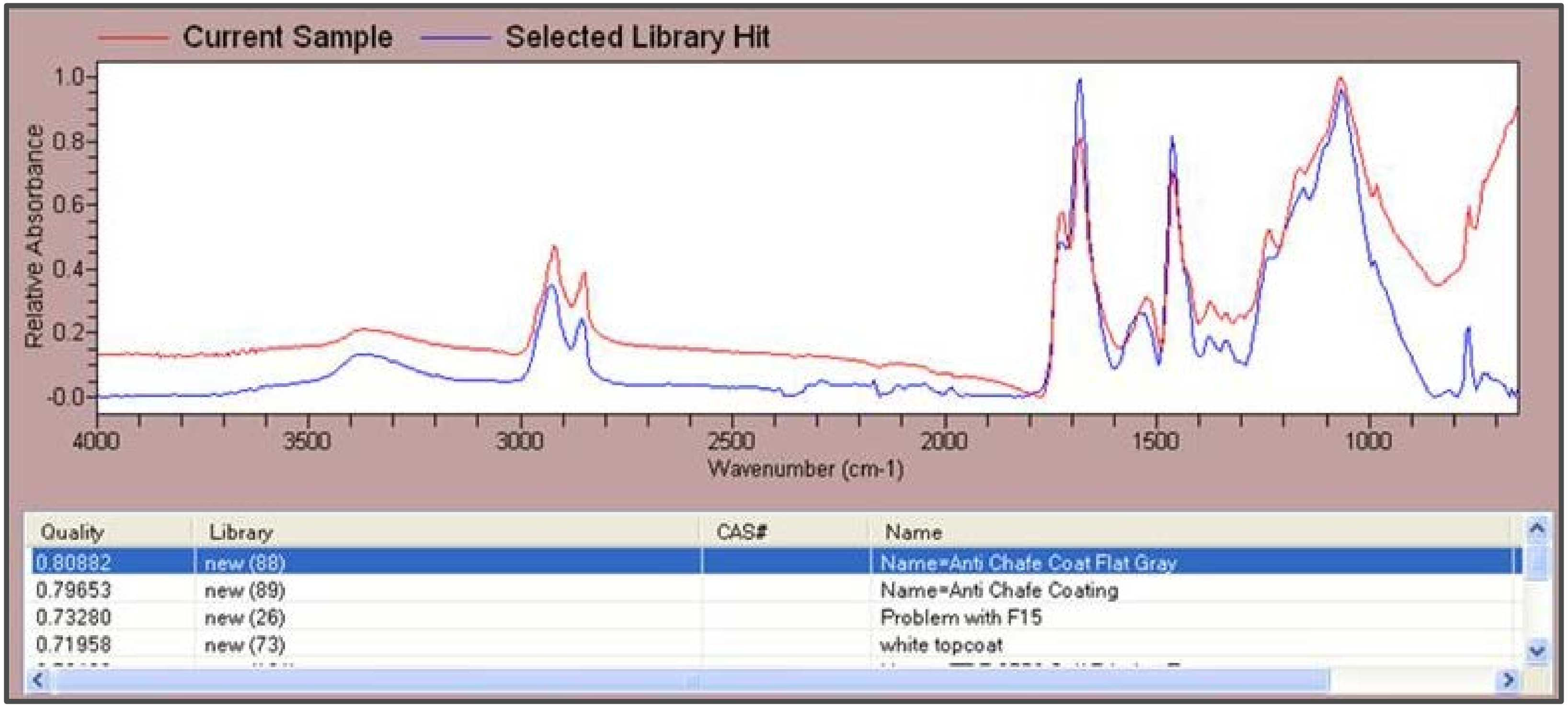


Infrared Spectrum

A spectrum is a graph of how much infrared light is *absorbed* by molecules at each *wavenumber* of infrared light

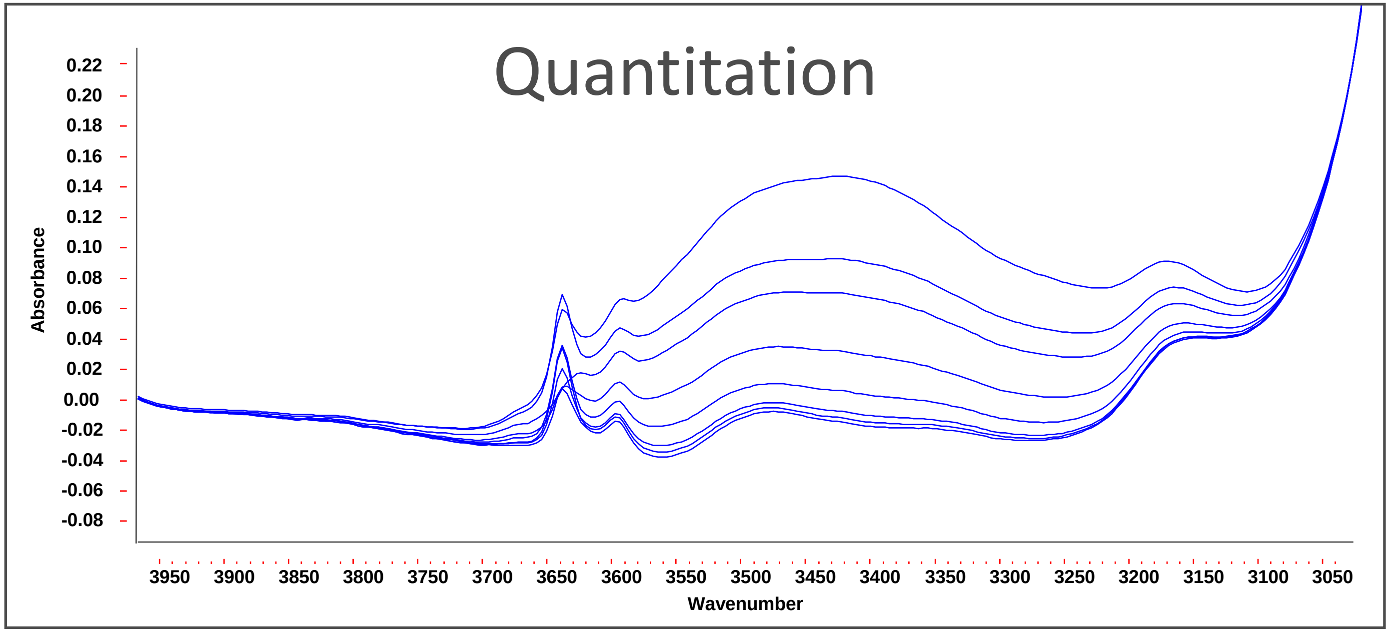


What is FTIR Good for?



Spectral Identification of Unknowns

What is FTIR Good For? (cont.)



IR spectral overlay of turbine oil 5-4300ppm

Calibration allows prediction of the concentration from the IR spectrum.

Calibration can be made to any quantity which tracks the change in sample chemistry.

Portable and Dedicated FTIRs

Exoscan 4100 Handheld FTIR

- 7 lbs
- 7” x 4.7” x 9” inches
- Single Enclosure (optics & electronics)
- PDA Controlled
- Interchangeable sample interfaces

Flexscan 4200 Handheld FTIR

- 4 lbs optical head
- Optical Enclosure wire connected to Electronics
- PDA Controlled
- Dedicated sample interfaces

Agilent 4300 Handheld FTIR

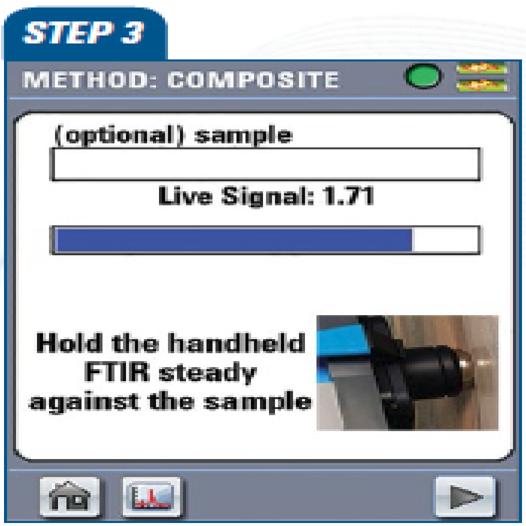
- 4 lbs
- Single Enclosure (optics & electronics)
- Integrated Computer
- Interchangeable sample interfaces
- MCT or DTGS detectors

Visual, intuitive user interface and software enable rapid system implementation

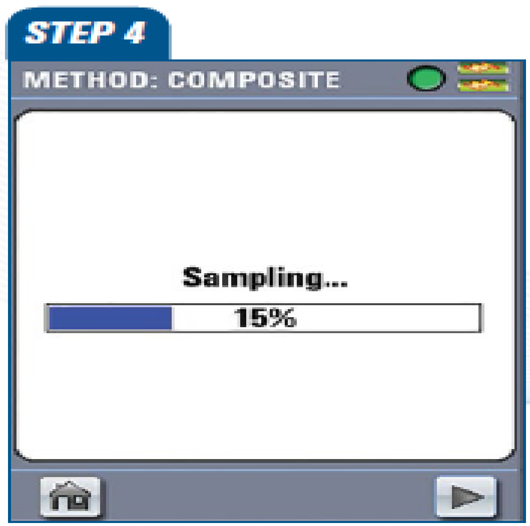
The highly acclaimed Agilent MicroLab Mobile software enables users of varied experience to get great results from the 4300 with a minimal training. The software guides the user through the measurement, and the RFID-equipped sample interfaces ensure that the method and measurement parameters are correctly matched. These innovations mean the 4300 will rapidly become an important part of your company's workflow.

From the home screen you can quickly launch analysis, choose a method, and create a new reference method.

MicroLab Mobile Software will instruct when to position the spectrometer's sample interface on the object to be analyzed.



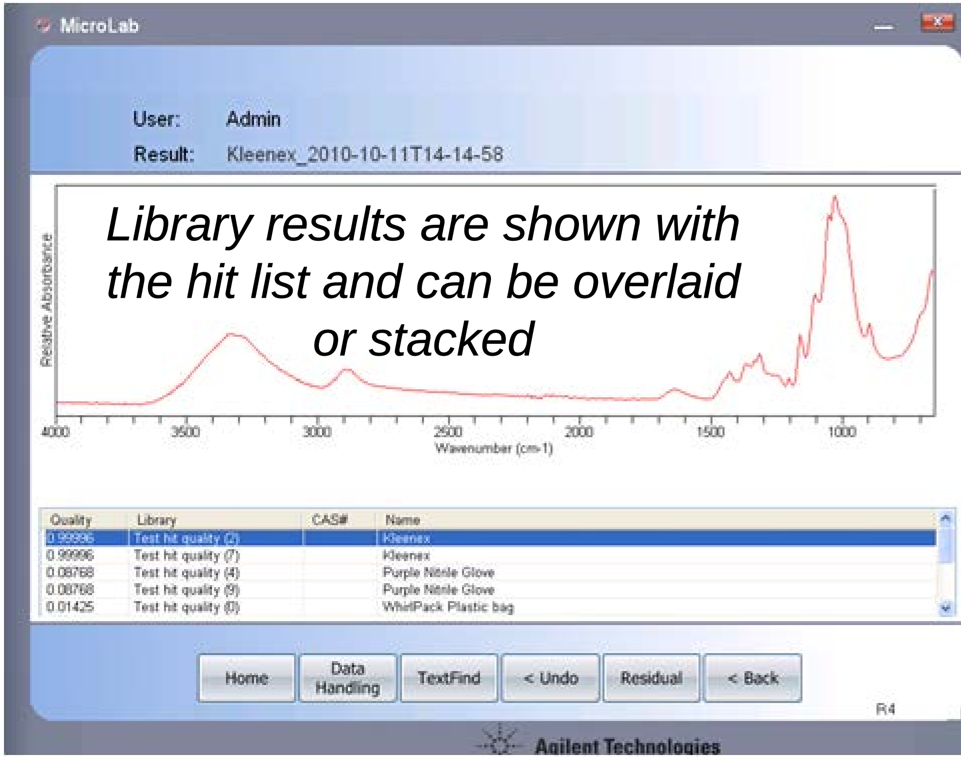
During sampling, the progress bar shows the advancement of the data collection.



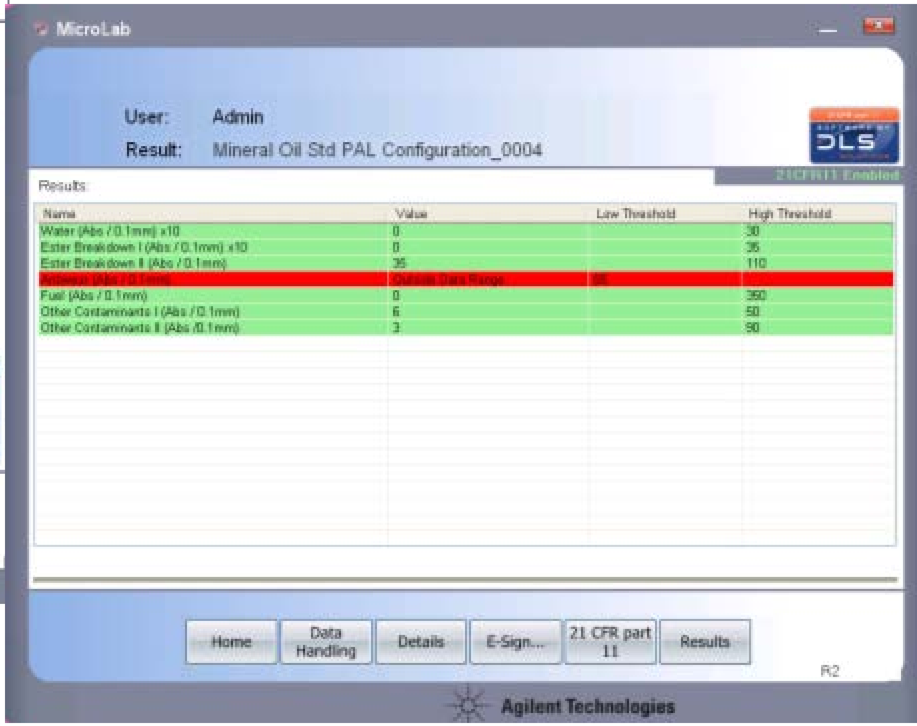
When the progress bar reaches 100%, the prompt will change to Transferring Data. You can then remove the instrument from the sample.

Results screen: The results screen will display the calculated component values relative to their critical limits. Components within the acceptable range are

Highly Visual results depending on applications



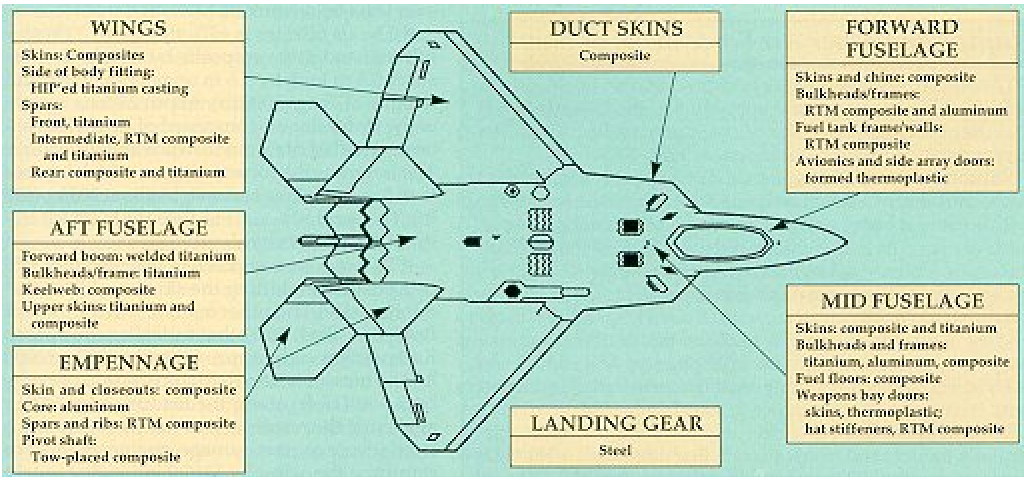
Library results are shown with the hit list and can be overlaid or stacked



Quant results can be programmed for critical action levels with color coded queues (red, green, yellow)

Today's Overview of Applications

Composites Material Manufacturing and Engineering



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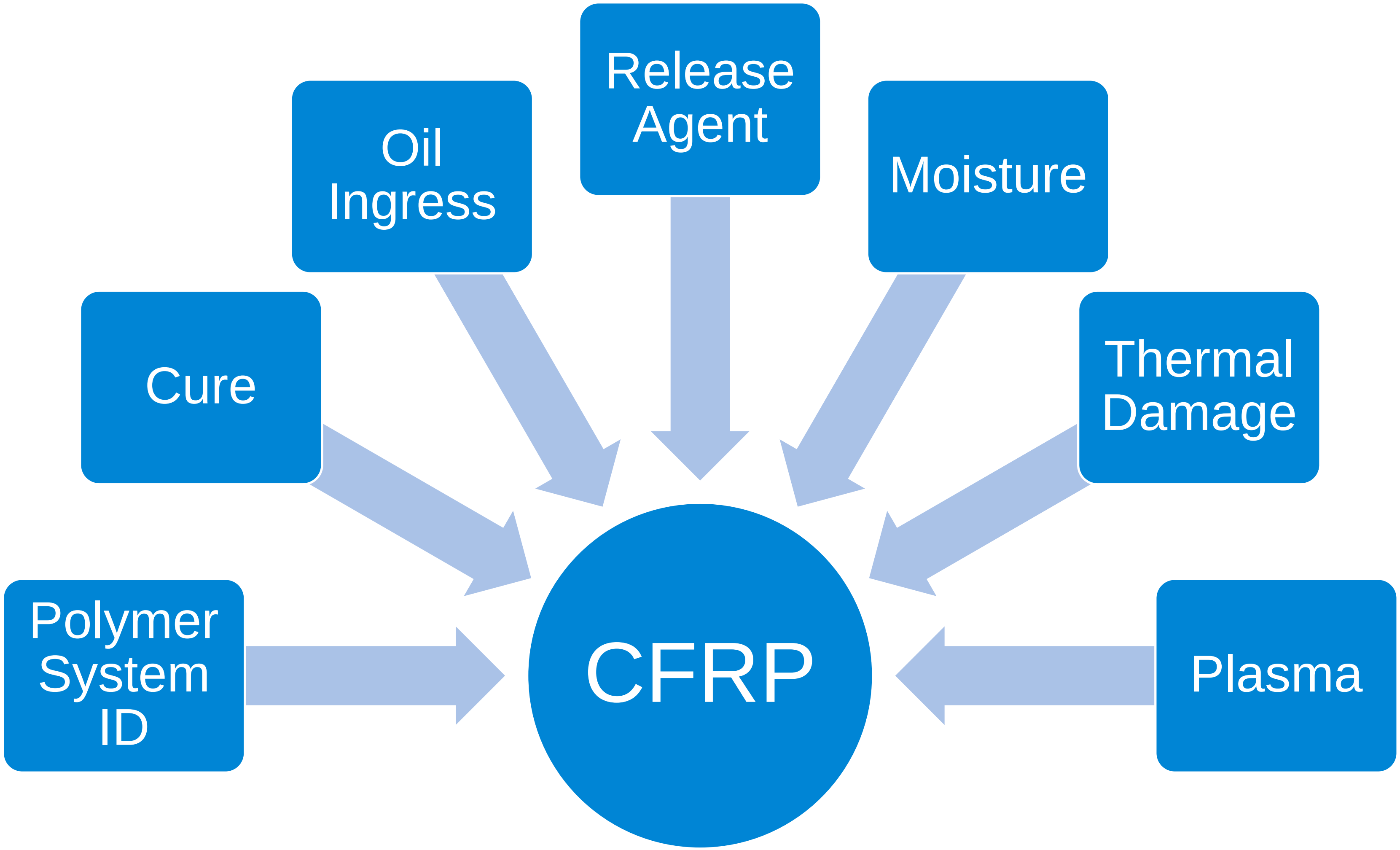
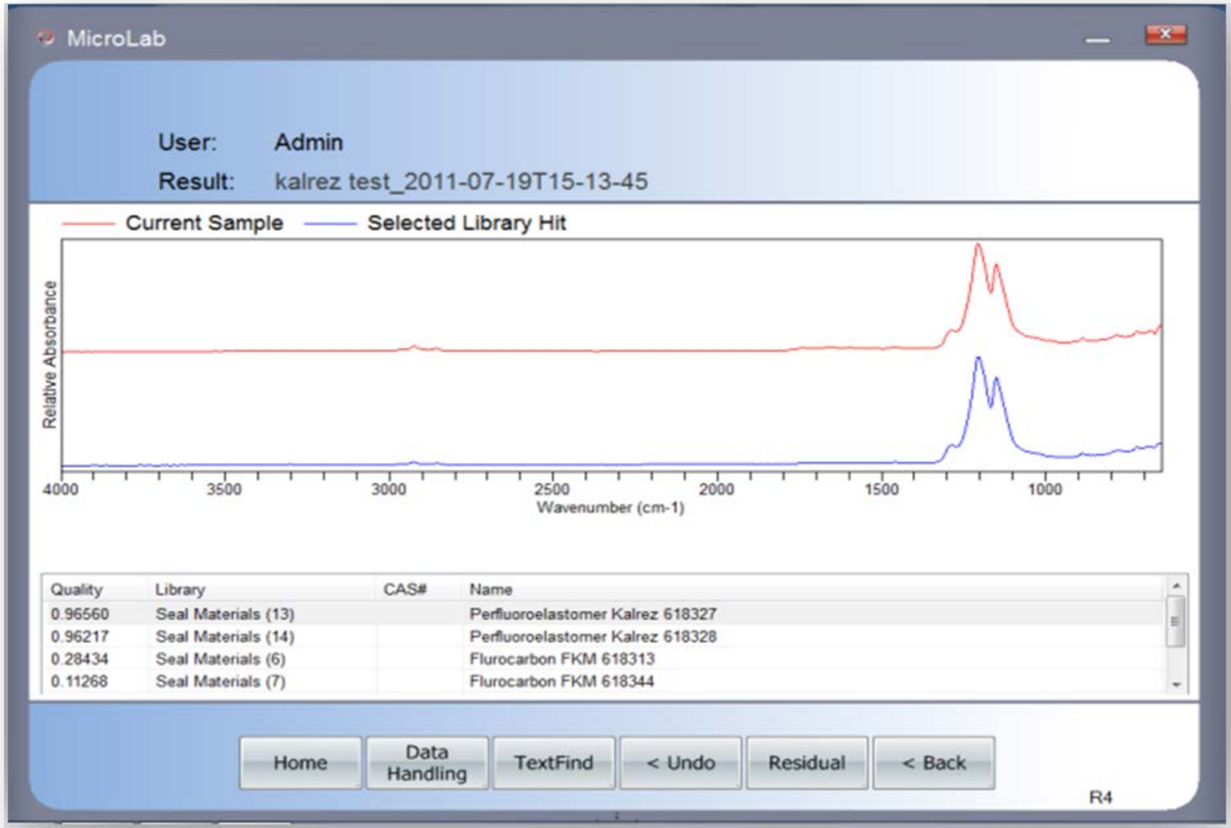
Important Measurements for Carbon Fibre Re-Enforced Polymers: All Possible with FTIR



Positive Material Analysis by FTIR

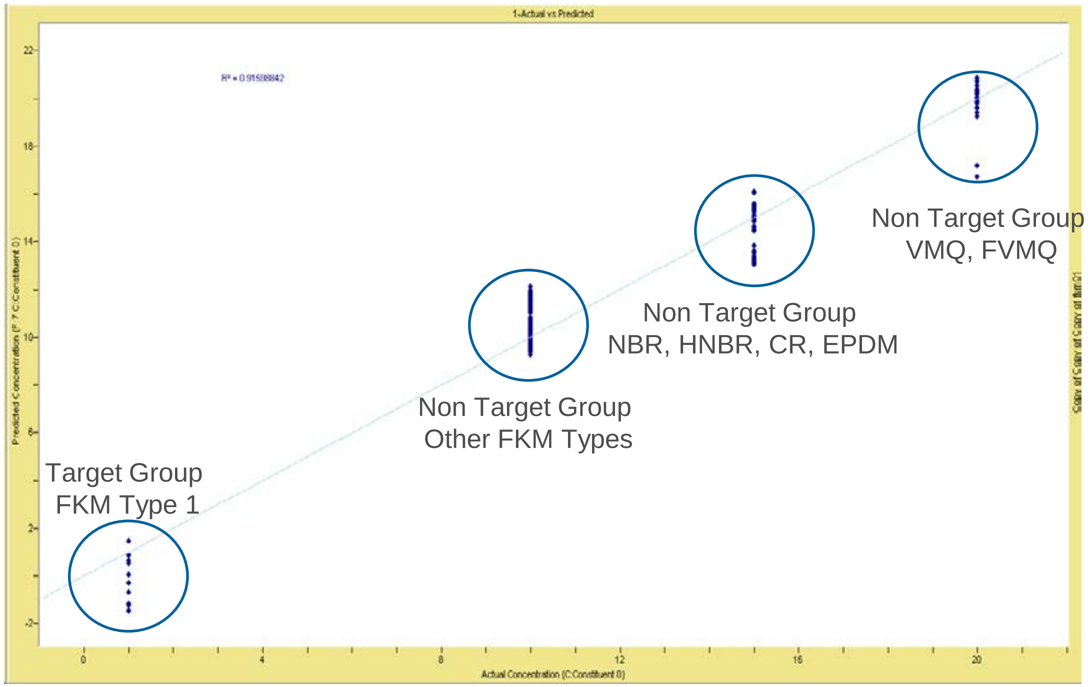


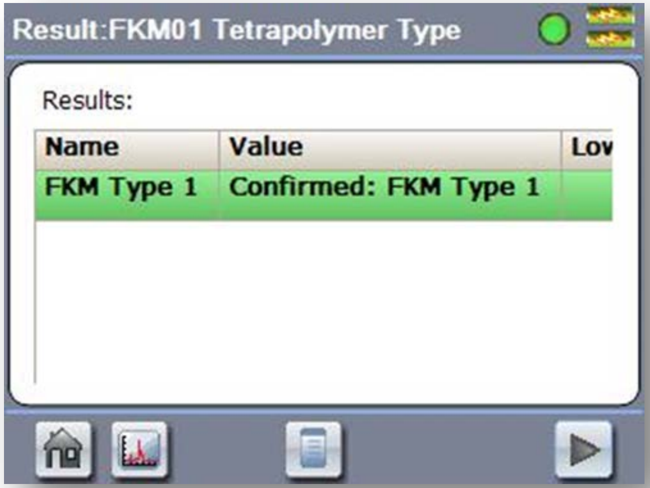
Verification of Material through Library Searching and Database Matching is easily accomplished.

Commercial libraries can be purchased or customers can conveniently create their own. This is important when proprietary materials may be involved.

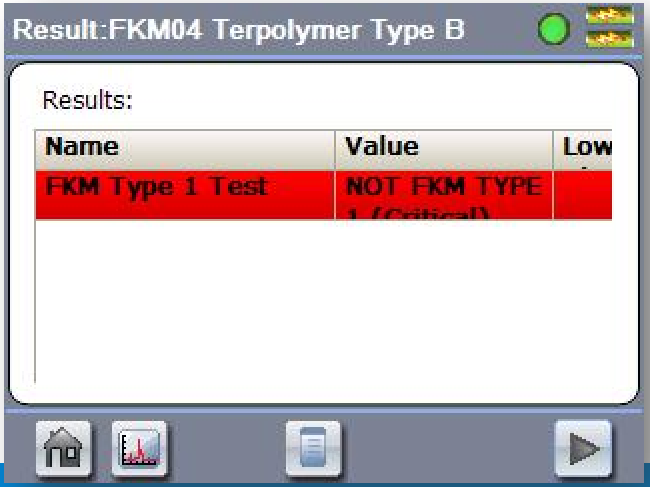
QC Verification of Incoming Raw Material, or in Process Materials(pure and blend, compounded)

Is this the material of Interest?





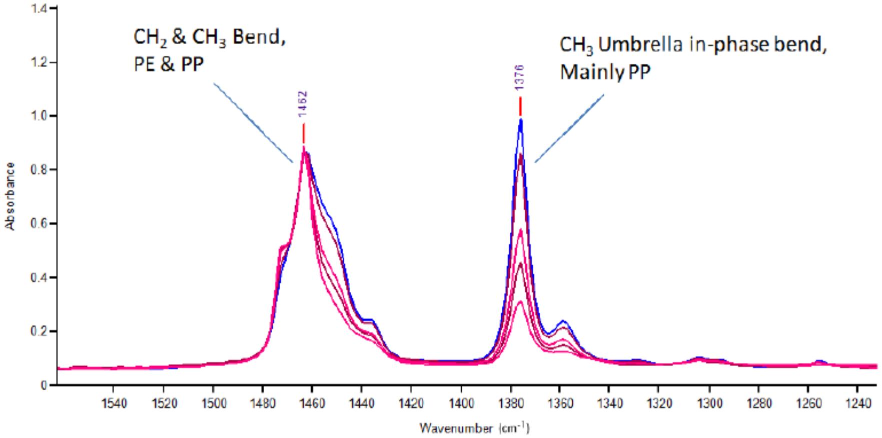
OR



For determining PASS/FAIL ID qualifications, PLS-DA can be used to clearly target a specific classification of material.

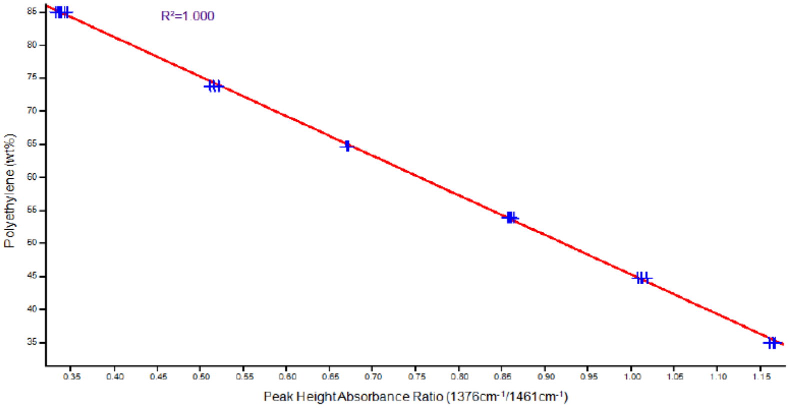
QC Verification of Thermoplastic Polymer Blend Process by FTIR: check your dispensing machines!

Example of PE/PP blend

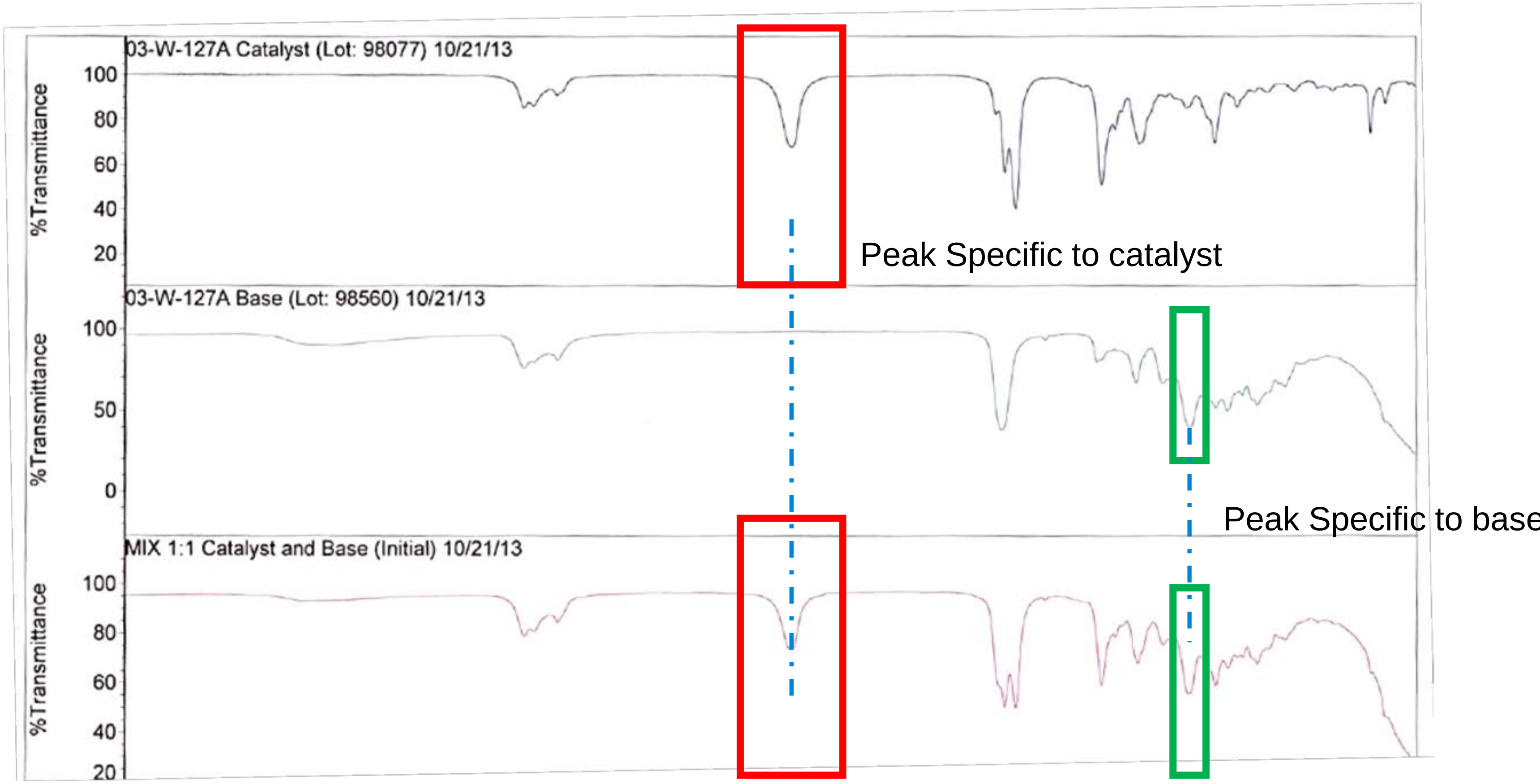


Overlaid aliphatic bend region of the FTIR PE/PP blend calibration spectra.
Quantitative method for %PE uses a ratio of the methyl 1376 cm-1 (PP) to the 1462cm-1(methyl and methylene bend) band.

Calibration plot of PE/PP blend: Peak Height versus Concentration level of PE in PP



Urethane Blend Measurement : Verification of 1:1 Ratio Catalyst/ Base for Urethane Topcoat Deft (03W127A)



Ratio of Abs Red Peak/ Abs of Green Peak must be 1 to PASS Blend test in FTRI

Surface Qualification – Resin Cure

- **Resin or coating cure – essential for performance**
  - Under-cured resin structurally weaker
  - Under-cured coatings have poor weather resistance
  - Visually identical to fully cured resin

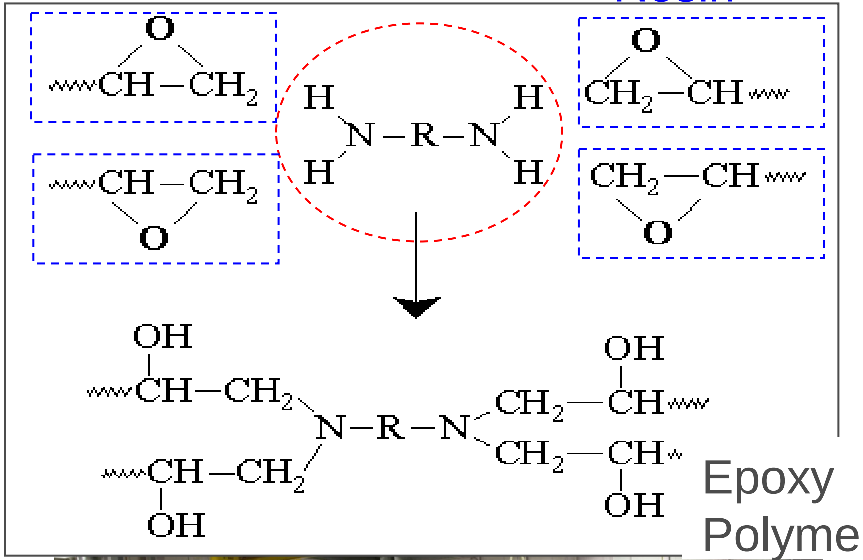
- **Conventional Method**

- Recipe: Time/Temperature/Vacuum profile

- Witness samples
  => PROBLEMS….
  Variations between autoclaves or across a large part.
  Witness samples may not catch the problem.

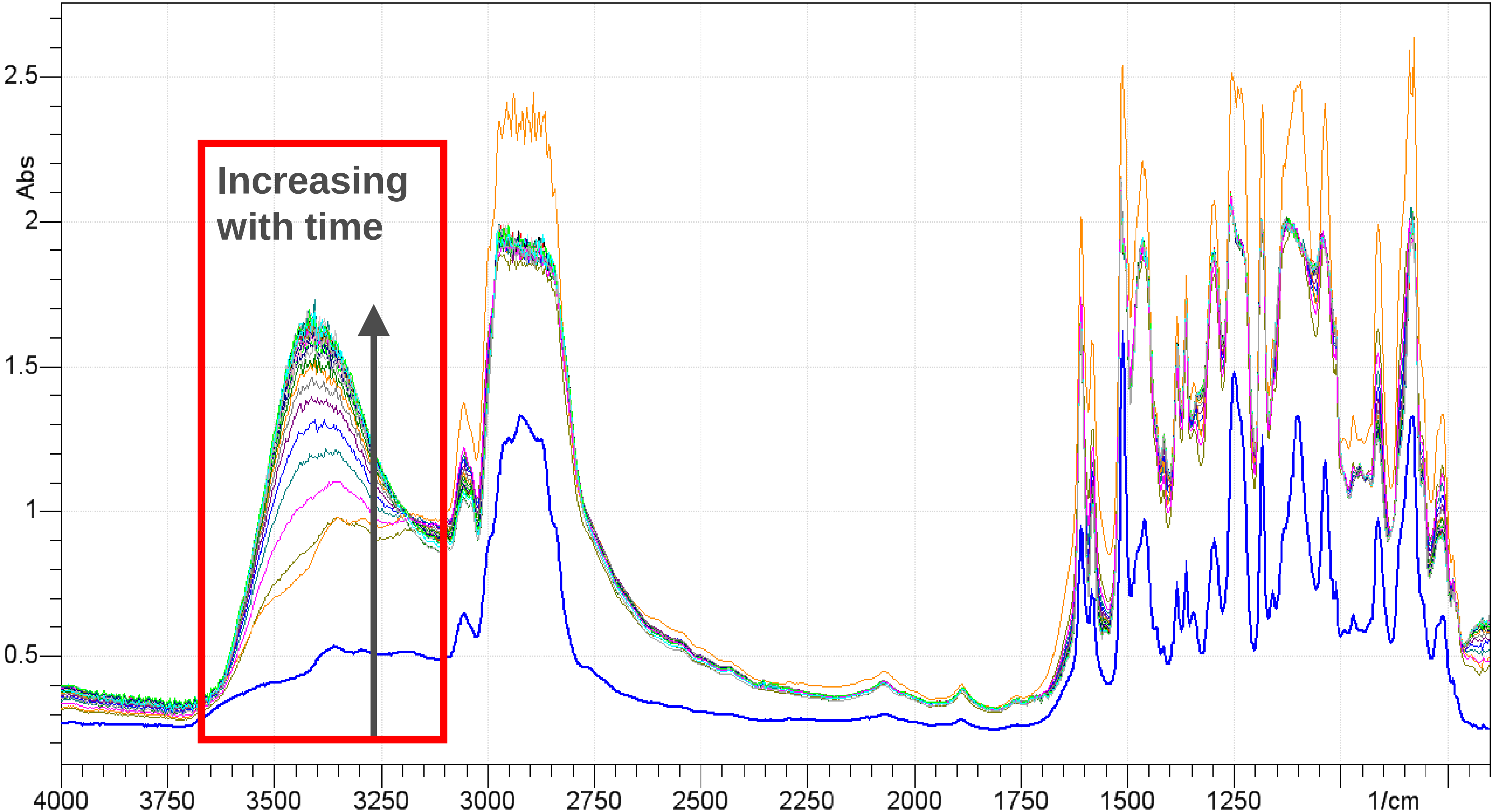
Hardener

Epoxy Resin



Epoxy Polymer

End Cure Detection/ Unfinished Cure

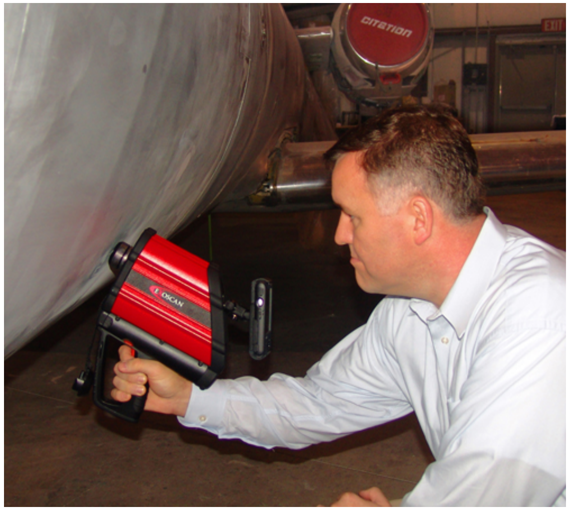


30°C cure, spectra collected at 30 min intervals

Hand Held FTIR Surface Contamination

Diffuse Reflectance and Grazing Angle

- FTIR well suited to surface contamination
  - Hydrocarbon and Silicone are strong absorbers
  - Unique signal = positive ID of contaminant
- Different Sample Interfaces for Carbon and Aluminum
  - Aluminum highly reflective
    -> Grazing angle interface
  - Carbon fiber low reflectivity
    -> Diffuse Reflectance



Surface Cleanliness – Silicone on Carbon Fiber

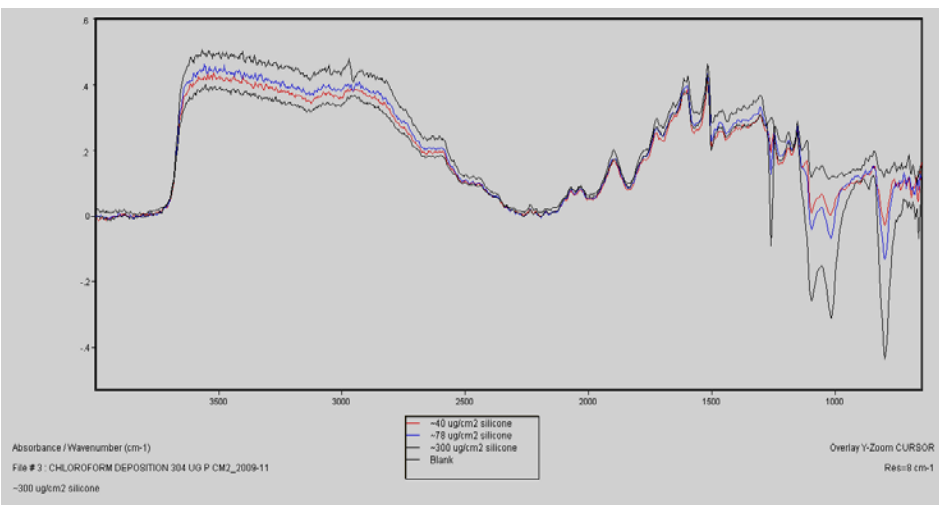
Silicone grease on carbon fiber composite

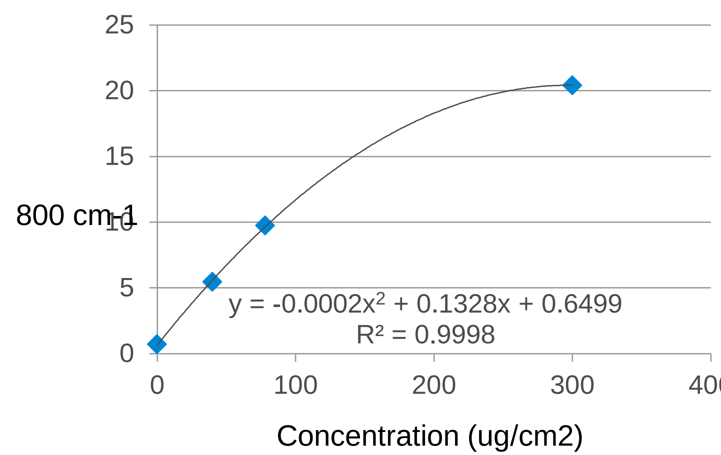
- 40 – 300 µg/cm2

Diffuse Reflectance Sample Interface

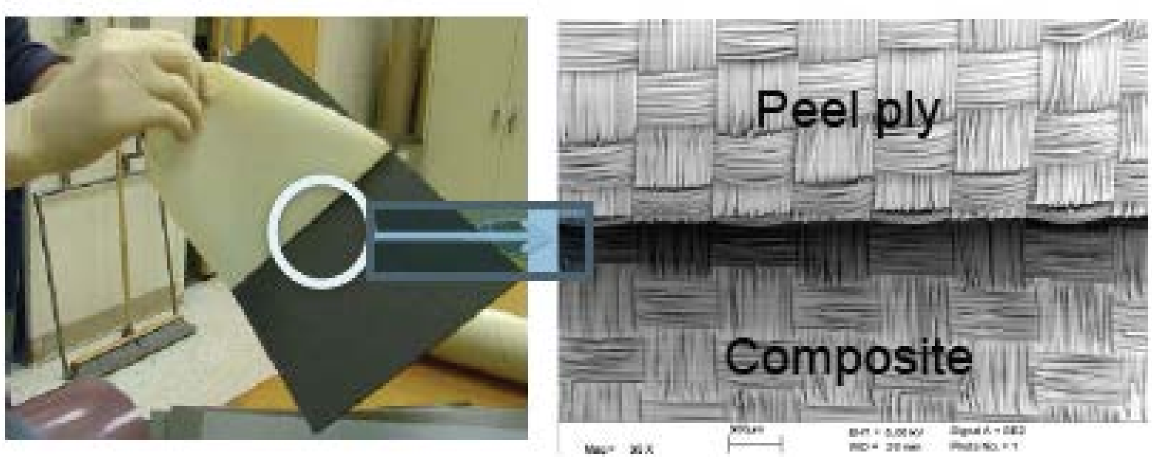
- Negative bands due to Restahlen effect -> still correlate to concentration

Limit of Detection (~ 3x p-p noise) ~= 10 µg/cm2





QA for Composite Bonding



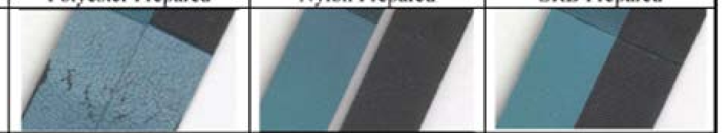
Composite bonds are materials system specific (prepreg/ surface prep/adhesive).

Bond quality can be altered by:

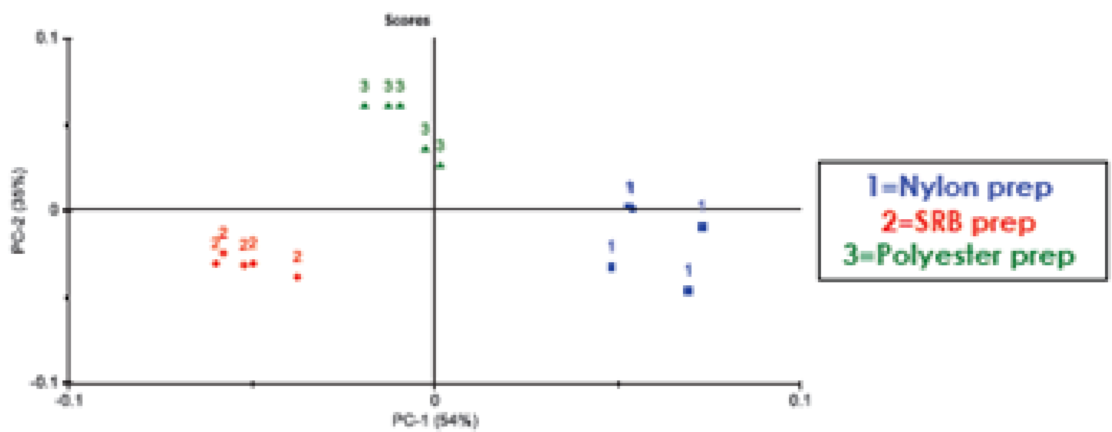
- Incorrect peel ply
- Contamination
- Improper abrasion

ID of Peel Ply Prep. with Portable DRIFT

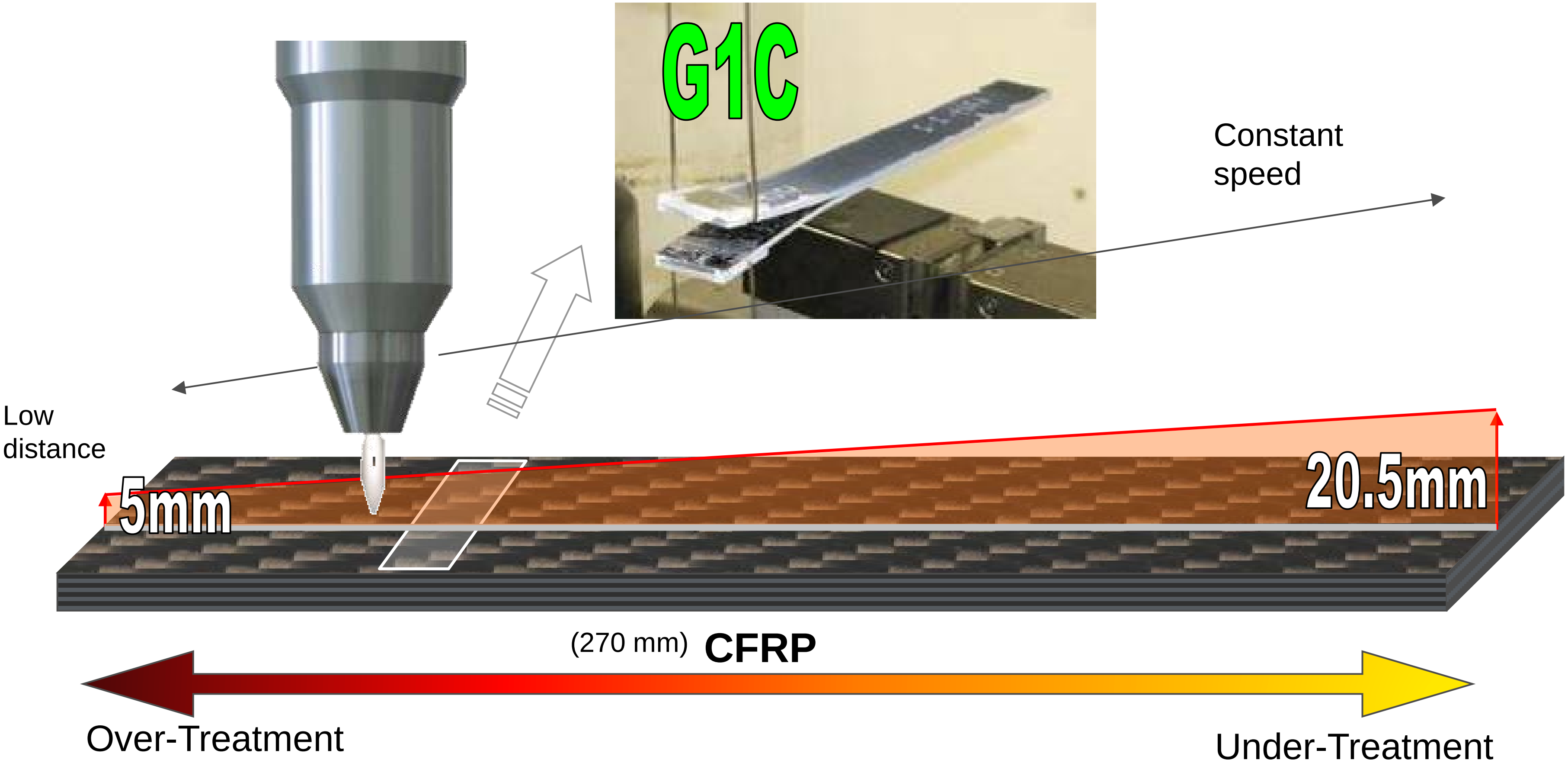
- Peel ply surface preparation for adhesive bonding is materials system specific

	Polyester Prepared	Nylon Prepared	SRB Prepared
			
Failure Mode	Cohesive	Adhesion	Adhesion
G_{IC}	812±35 J/m²	G_{IC} = 122±15 J/m²	G_{IC} < 94 J/m²

- DRIFT FTIR can differentiate CFRP prepped with different peel plies
  - Multivariate Analysis: PCA of two PCs
    - Preprocessing: Savitsky-Golay 1st derivative with 5 smoothing points fit to a 2nd order polynomial

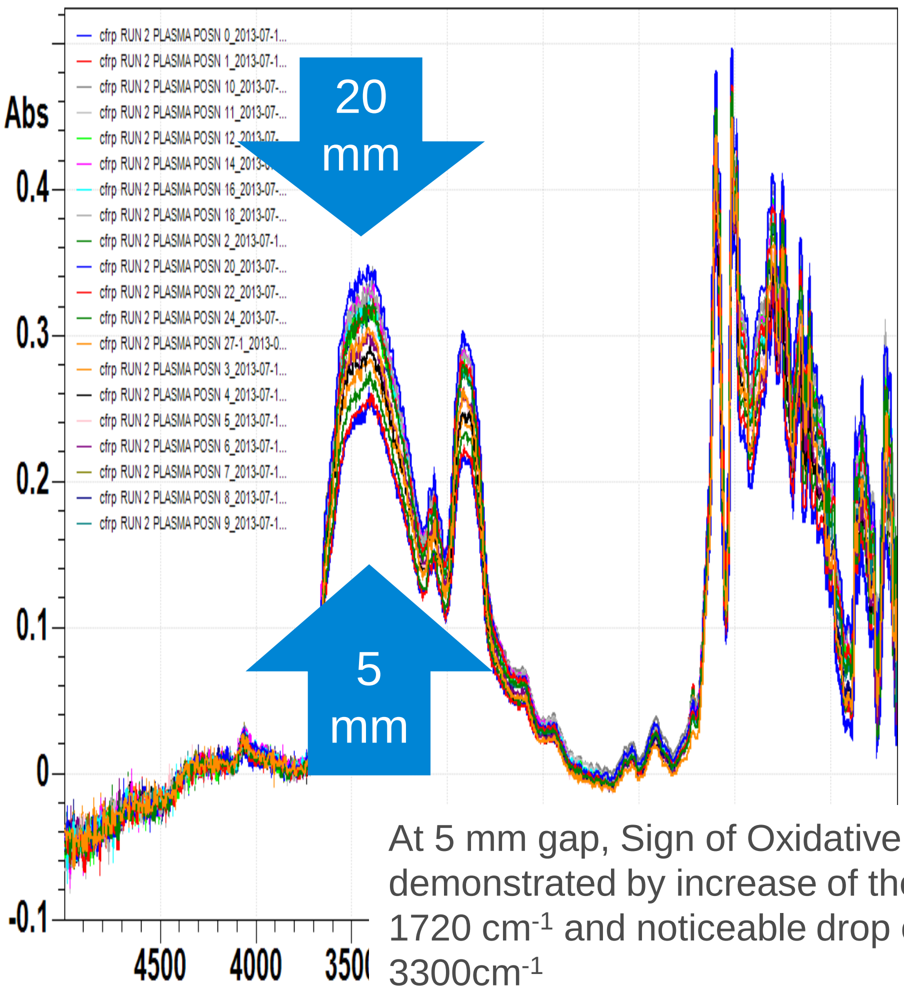


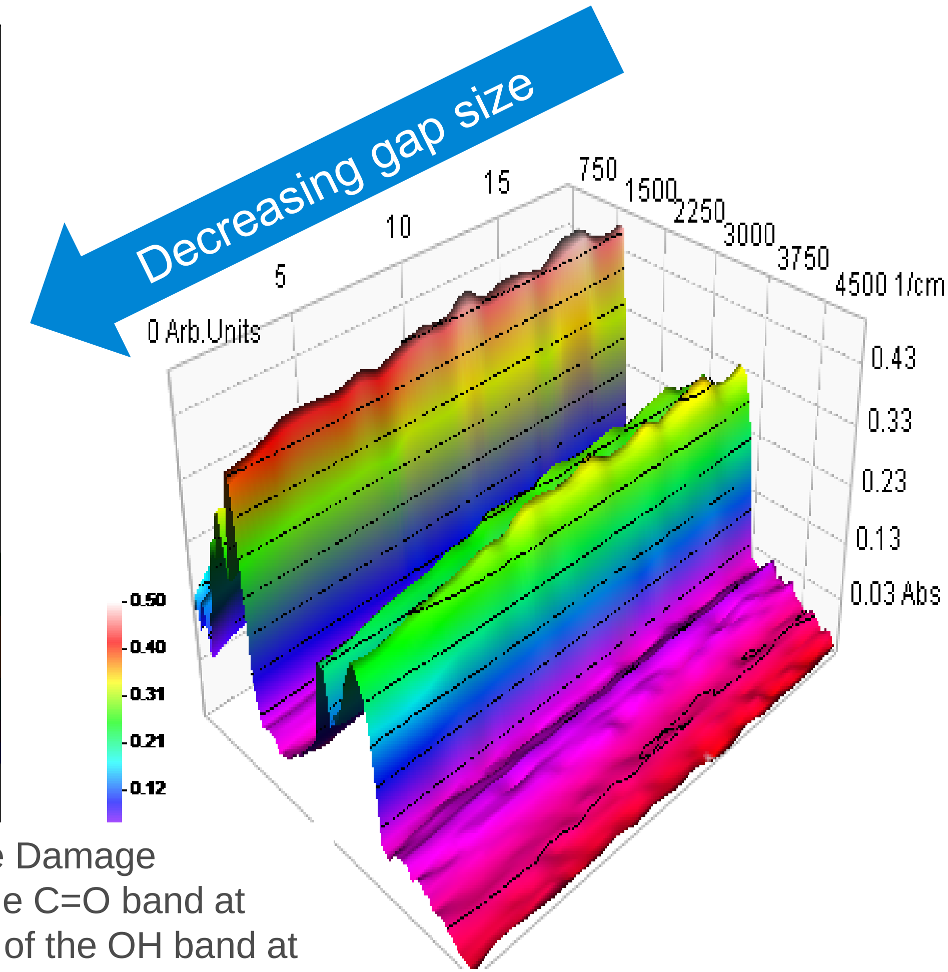
Surface Treatment and Activation via RF Plasma: Under, Over and Optimal Treatment Determination



Gap size affect the temperature at which the CFRP surface is submitted to: at the 5 mm gap, surface is treated at 260˚C; at 20 mm, surface is treated at 100˚C

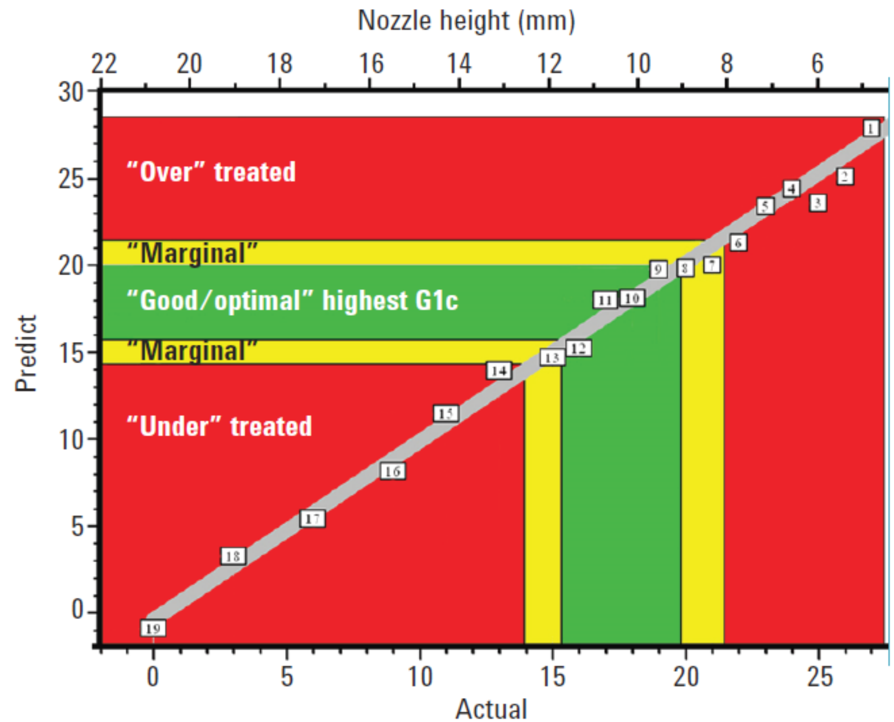
Wax based release agent residues on CFRP spectra, stacked view and 3d view at different Plasma Gaps





At 5 mm gap, Sign of Oxidative Damage demonstrated by increase of the C=O band at 1720 cm^{-1} and noticeable drop of the OH band at 3300cm^{-1}

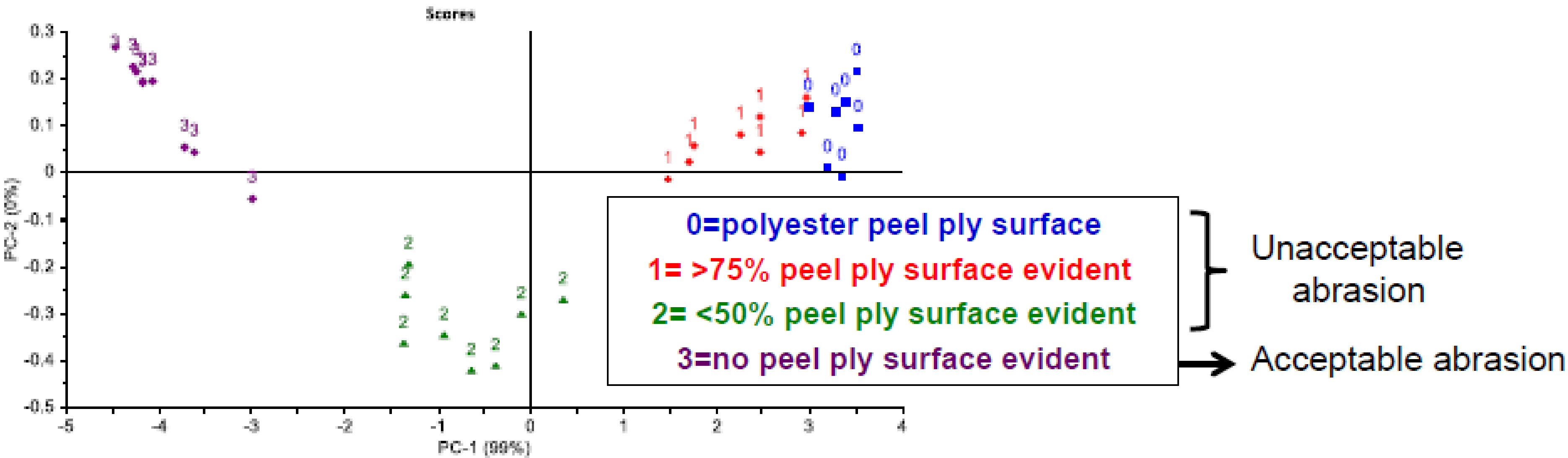
FTIR for Rapid Determination of Treatment Level of the CFRP Surface in the surface



- A PLS Algorithm was developed to correlate gap and hence temperature against changes in the FTIR spectrum for the rapid determination of treatment level
  - Adhesive Peel Strength combined with % Silicon values from XPS analysis were used to determined under, optimal and over treated region and used to predict values generated from the PLS model

DRIFT FTIR Sensitive to Abrasion Level of Peel Ply Texture on CFRP

- Polyester peel ply prep of 177°C cure composites fail in adhesion when bonded with paste adhesives
- Surface abrasion to remove peel ply texture/residue from composite (cohesive failure when bonded with paste adhesives after abrasion)



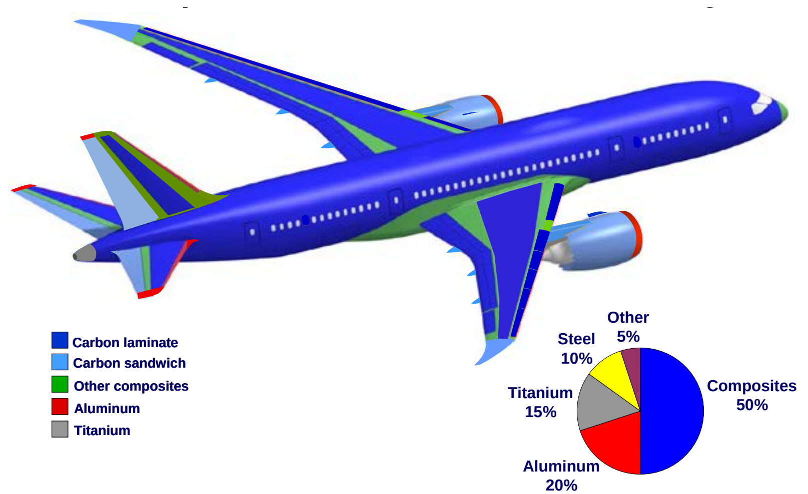
PCA of Diffuse Reflectance Spectra

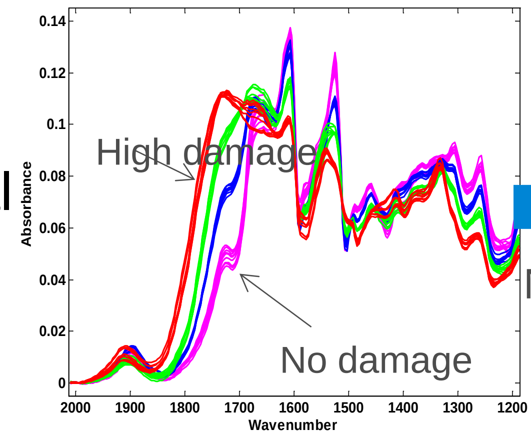
- DRIFT FTIR shows difference between acceptable and unacceptable abrasion levels to remove polyester peel ply texture/residue

Surface Damage

Oxidation of Carbon Composite

- Aircraft and other structures will increasingly be constructed from composite material
- Composites are susceptible to heat and oxidative damage Chemical damage of the epoxy resin
- Exoscan provides new type of non destructive testing to detect heat damage
- Exoscan specified in Boeing 787 NDT Manual





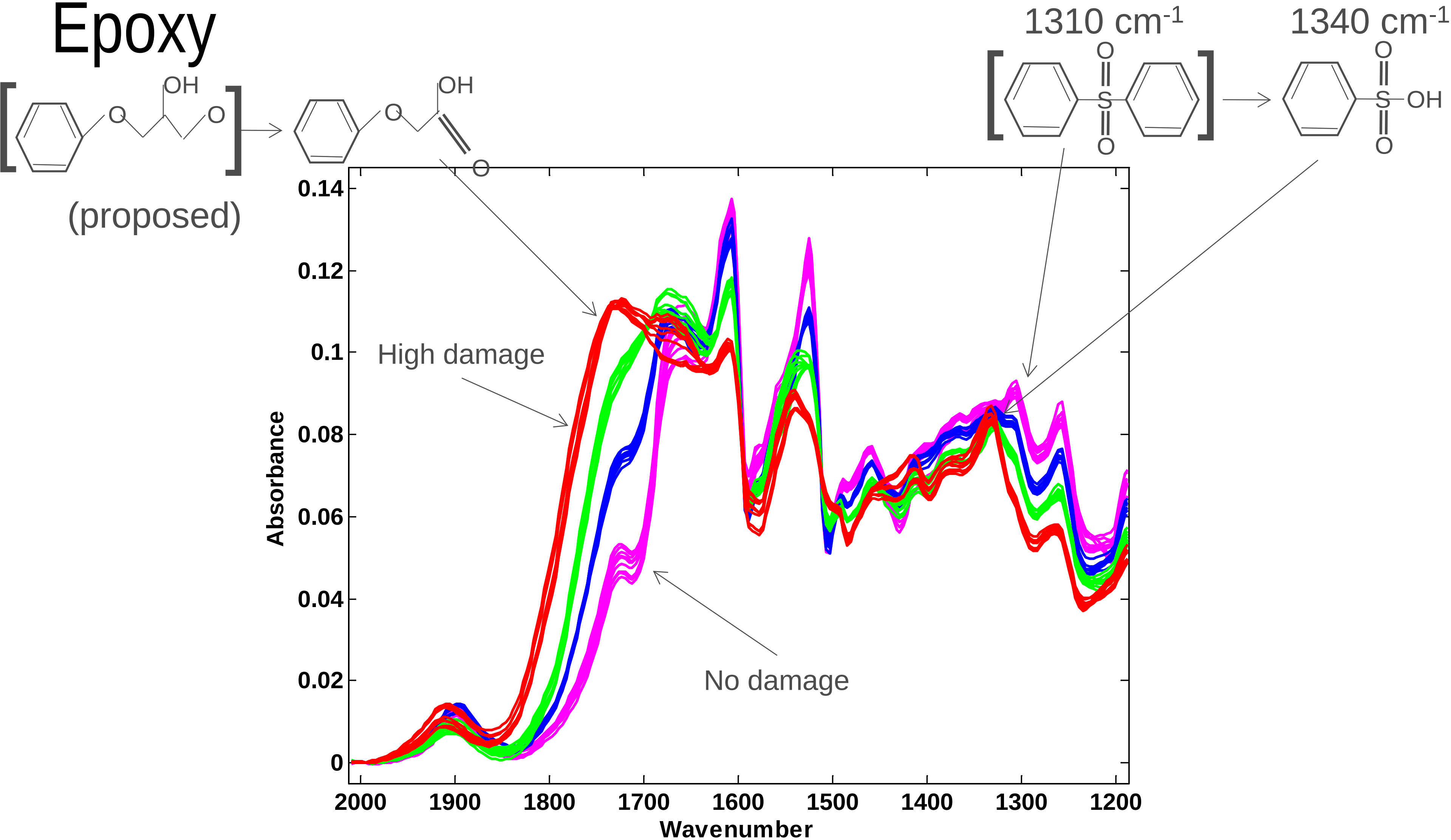
Neural Network

Output Class	1	2	3	4	
1	43 24.7%	0 0.0%	0 0.0%	0 0.0%	100% 0.0%
2	0 0.0%	44 25.3%	0 0.0%	0 0.0%	100% 0.0%
3	0 0.0%	0 0.0%	36 20.7%	0 0.0%	100% 0.0%
4	0 0.0%	0 0.0%	0 0.0%	51 29.3%	100% 0.0%
	100% 0.0%	100% 0.0%	100% 0.0%	100% 0.0%	100% 0.0%

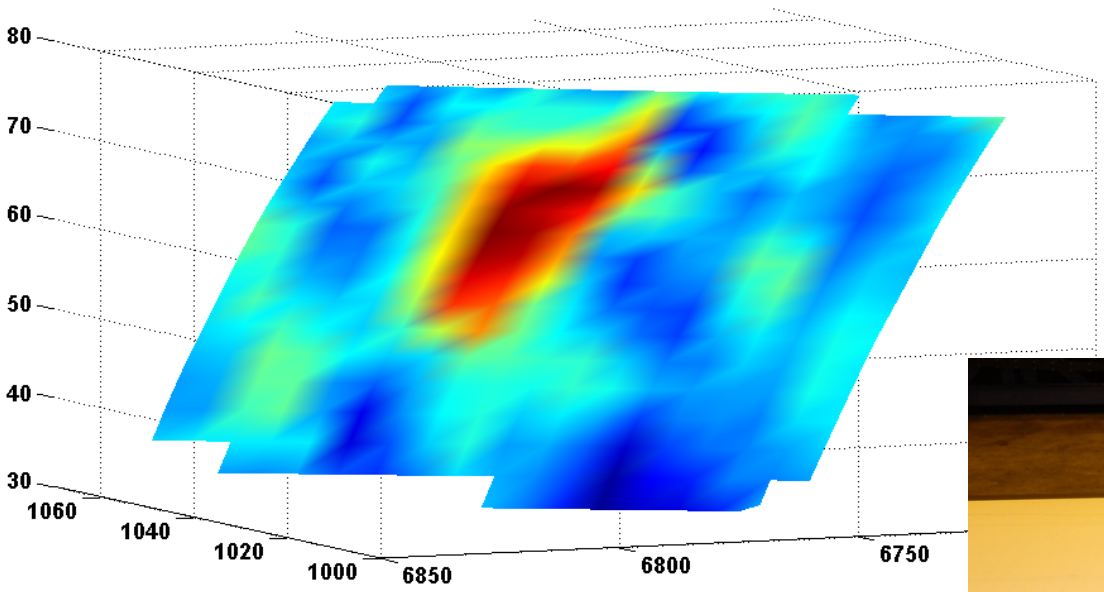
Target Class

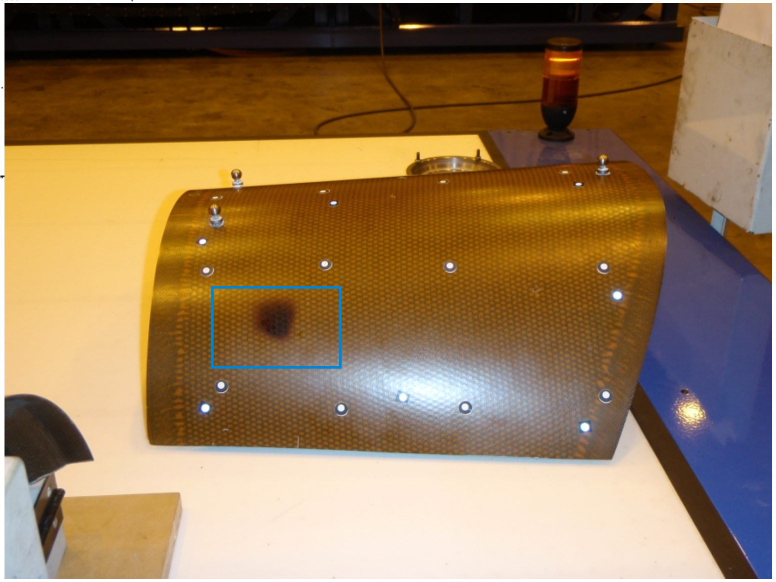
Low High

Thermal Damage Study Changes in FTIR Spectra - Epoxy



Heat Damage Mapping by Handheld FTIR





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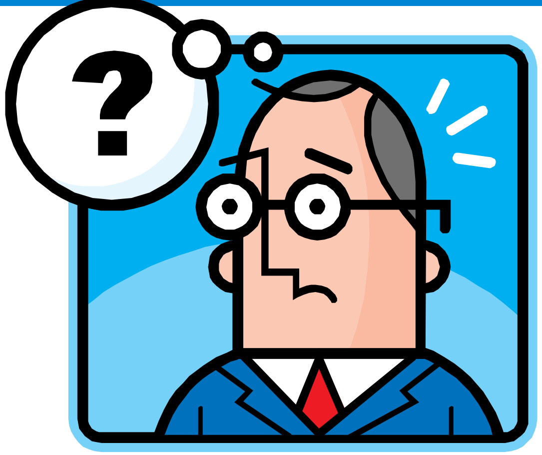
Questions?



Coatings, Films and Surfaces – Applications by Handhed FTIR (other Surfaces)

- **Evaluation of Curing Process**
  - Effectiveness of curing agent
  - Solvent residue after curing process
  - Detect hardener in curing process
  - Evaluate polyurethane curing on metal
- **Primers**
  - Effectiveness of adhesive bonding primer as a function of ambient conditions
  - Measure primer thickness on aluminum
  - Primer adherence on damaged composite
  - Thermal and UV degradation of composites
  - PVC analysis on steel
- **QA/QC of materials and surfaces**
  - Pass/fail analysis of epoxy on steel
  - Identify special coatings on aluminum
  - Detect and confirm anodization thickness on aluminum
- **Contamination analysis**
  - Effect of silicone and hydrocarbon oil on bonding