SAND2013-5643C

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Fire Science & Technology

JULY 14–19 2013 Minneapolis, MN

ASME 2013 SUMMER HEAT TRANSFER CONFERENCE

Discriminating Composite Panels by Use of a Spectral Reflectometer

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SAND2013-????C
HT2013-17614

U.S. DEPARTMENT OF ENERGY | NNSA

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Introduction

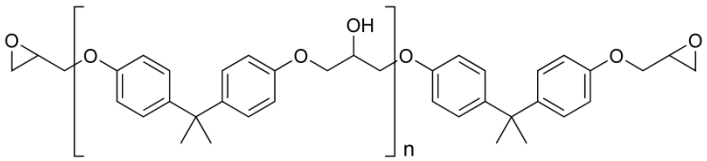
- Carbon fiber composites are being increasingly used in engineering applications; they are costly, but light-weight
- Aviation and other applications are becoming prevalent
- We are motivated by an interest in the fire environment due to composites increasing prevalence
- We have obtained some materials, and don't know their type
- We would like to have a simple analytical method to identify the unknown panels
- The spectral reflectometer may provide such a solution

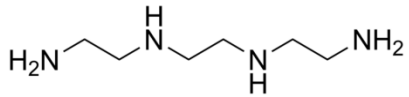
Carbon Fiber Epoxy Aircraft Composites

- Around ~35% epoxy, ~65% carbon fiber
- Fabric (woven) or uni-tape sheets, usually multiple layers thick
- Possibly sandwich material with high void fraction material between two composite sheets
- Pressed and cured in an autoclave
- Fibers around 5 μm diameter, 95% carbon

Epoxy and TETA hardener (From wikipedia):

$[C_{18}H_{20}O_3]_n$

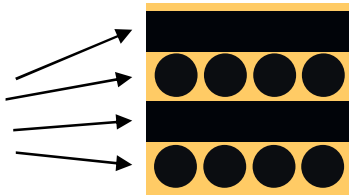




$C_6H_{18}N_4$

A four layer cross-section illustration:

Fibers in varying orientation



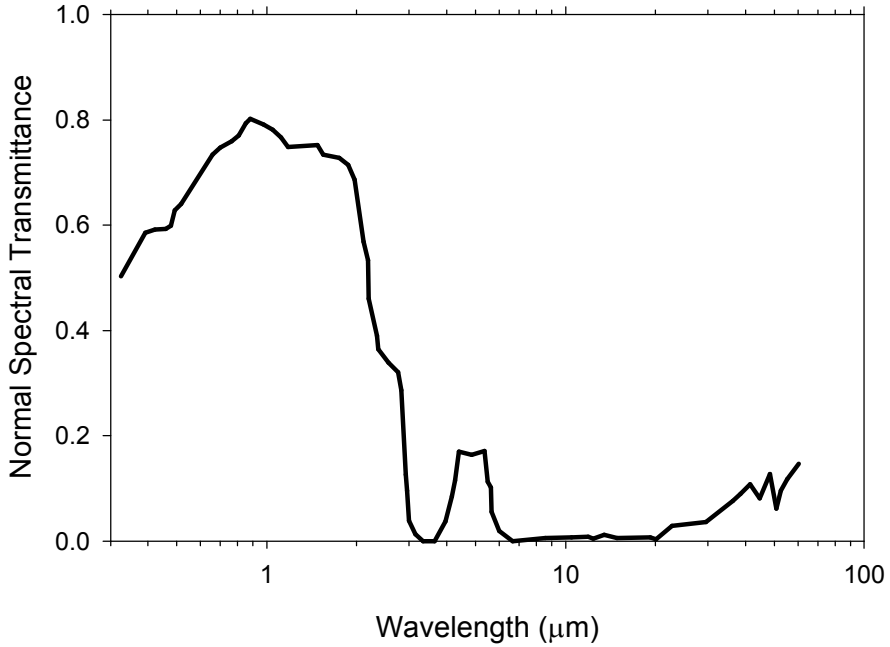
Key:

Carbon Fibers

Epoxy Resin

Background

- Reference materials suggest low transmittance at high wavelength
- ‘Cat-A-Lac clear’ epoxy resin 250 microns thick on a 20 micron thick polyethylene substrate:
- Different epoxies use different formulations
- Formulae are proprietary; one only knows that materials fit a manufacturer specification
- There might be enough detail in the spectral content of the epoxy to discriminate materials of different manufacturers



Methods

- Used a Surface Optics Corporation model ET100 reflectometer
- Integrates six spectral bands in the IR
- Tests at two incidence angles, 20 and 60°
- Calibrated shots to a specular gold coupon with reference measurements
- Testing occurred in several installments over the course of several months
- Leveraged data from an ongoing test series
- Bands:

1-1.5; 2-3.5; 3-4; 4-5; 5-10.5; 10.5-21 μm

Samples

- Three categories of samples: Epoxy, BMI, and other
- Many samples with multiple spectra (count)

Name	Designation	Fiber	Thermoset	Count
E1F	Cytec 977-3	Fabric	Yes	1
E1T	Cytec 977-3	Tape	Yes	3
E2F	Hexcel 3501-6	Fabric	Yes	2
E3F	McMaster-Carr 1	Fabric	No	1
E4F	McMaster-Carr 2	Fabric	No	1
E5F	Cytec 5208	Fabric	Yes	4
E5T	Cytec 5208	Tape	Yes	4
E6F	Hercules 3501-6	Fabric	Yes	1
E7F	ACG MTM 45-1	Fabric	Yes	1
E8T	Hercules 8551-7A	Tape	Yes	5

Epoxies

Others

Name	Designation	Description	Count
Aramid	Kevlar Epoxy	A yellow non-carbon composite	3
Desk	Black Paint	A wood textured black painted desk-top	2
Monitor	Black Plastic	The black plastic on an NEC 1880SX monitor	2
Paper	White Paper	A white piece of copy paper	1

Name	Designation	Fiber	Thermoset	Count
B1F	Cytec 5250-4	Fabric	Yes	1
B1T	Cytec 5250-4	Tape	Yes	3

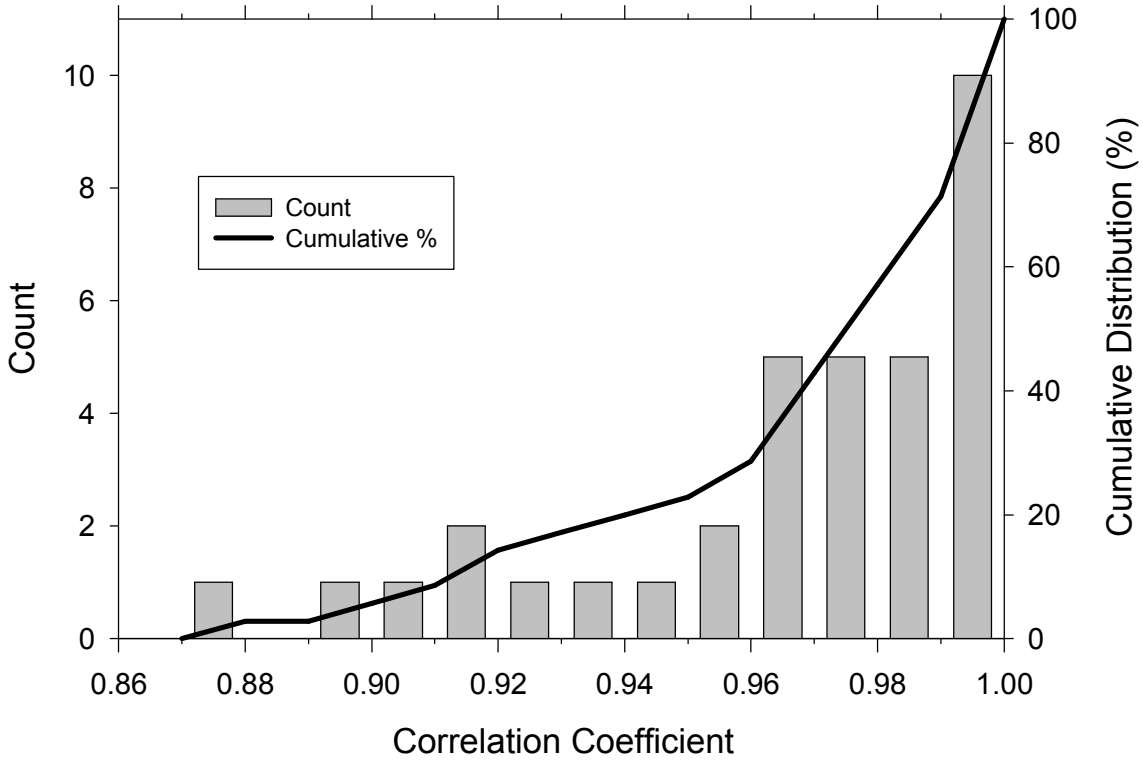
Bismaleimides

Approach

- Correlation analysis performed between spectral response of all the samples
- Correlation analysis was performed for all spectral data
- Concept:
  - The correlation coefficient might be used to screen out obviously dissimilar materials
  - Once the initial screening by correlation analysis was done, a second more detailed analysis might be made
- Looking to define a rule set whereby samples can be assessed for similarity

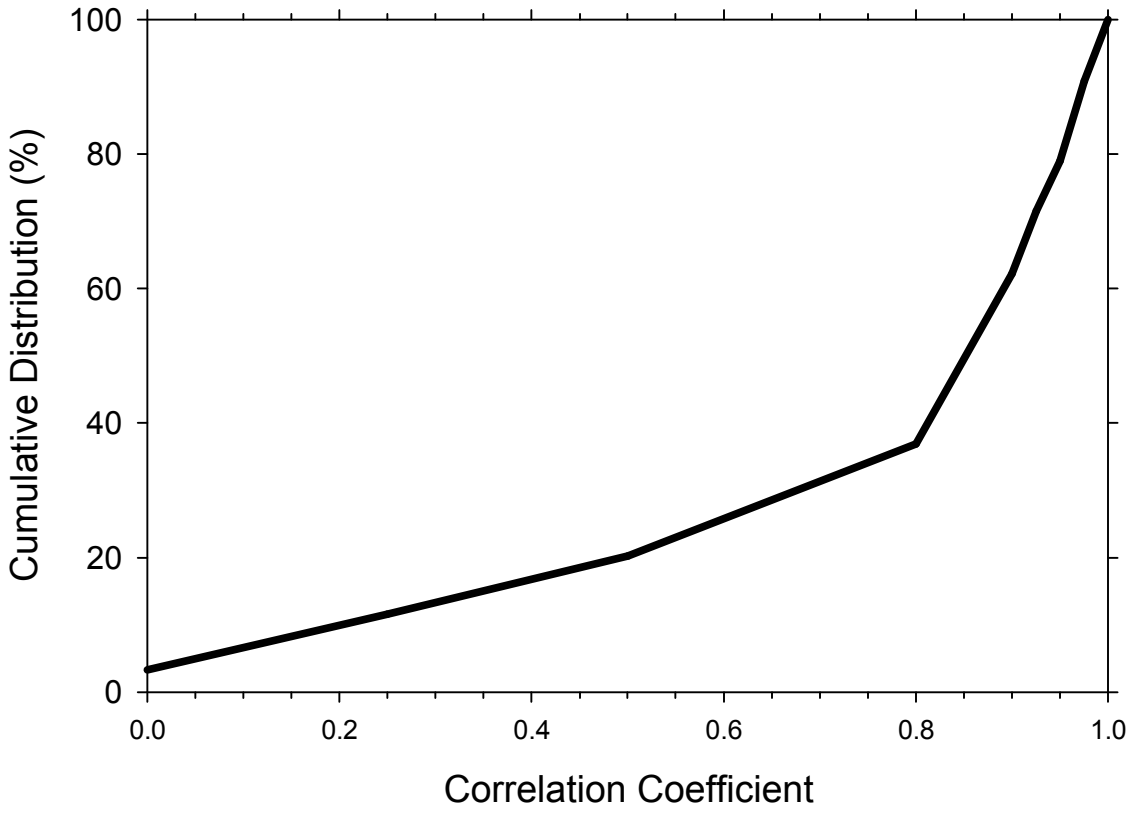
First Screening

- Looking for cut-off lower threshold correlation coefficient
- Cumulative distribution of correlations coefficients between identical pairs:



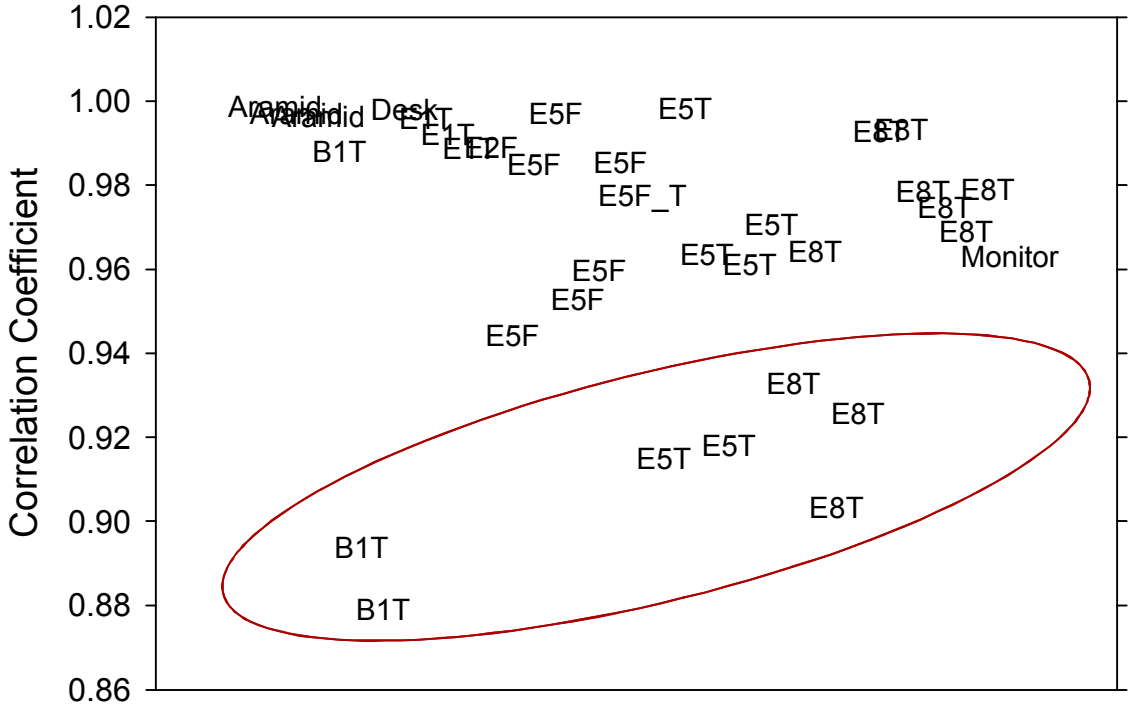
Cumulative Distribution of All Samples

- Threshold of 0.85 includes approximately 50% of all samples
- Not exclusive enough (0.85 includes 50% of the pairs)



Details by Sample Pair Type

- Lower correlation coefficients for many of the ‘T’ape samples
- Warranted further investigation



Re-analysis by Directionality

- Re-analyzed samples paying attention to the orientation of the fibers with respect to the instrument
- High correlation for similarly oriented samples, much lower for different orientations:

	Parallel	Perpendicular	Parallel	Perpendicular
Parallel	1			
Perpendicular	0.899	1		
Parallel	**0.989**	0.906	1	
Perpendicular	0.935	**0.993**	0.933	1

- **Finding**: the orientation of the fibers is significant to the readings
- **Finding**: a correlation coefficient of 0.95 can be used to exclude if the orientation is consistent

Inclusive detailed analysis

- A correlation coefficient of 0.95 can adequately screen dissimilar many samples
- Many correlations exist for correlation coefficients about 0.95
- The subsequent graphs compare one sample to all correlated samples
- Error bars are one standard deviation
- The hope is to find a rule-set to adequately distinguish different materials with high correlation coefficients.

B1F and correlated samples

- The bismaleimide samples should be different from the epoxies
- Tape samples (B1T) were significantly higher
- Best correlations with E5T samples; very different materials
- These analyses would result in false positives

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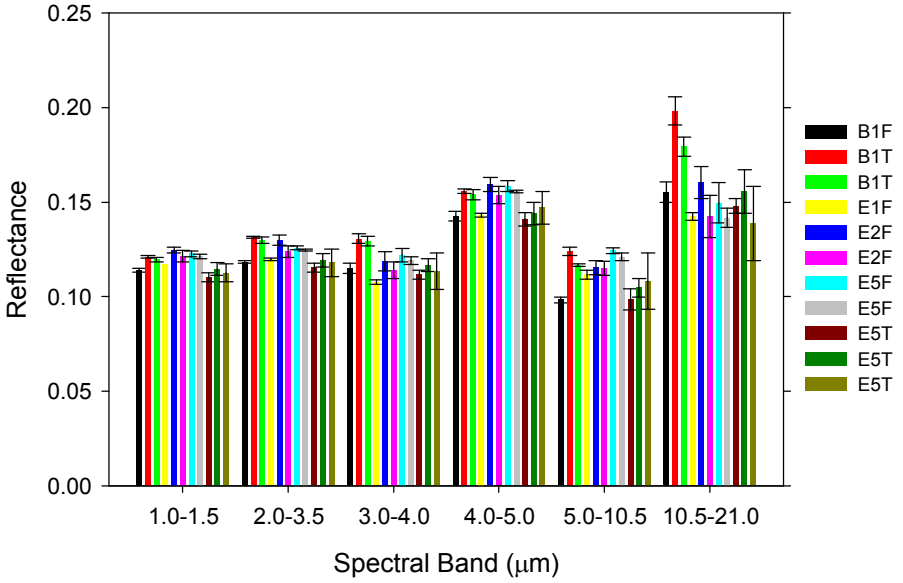

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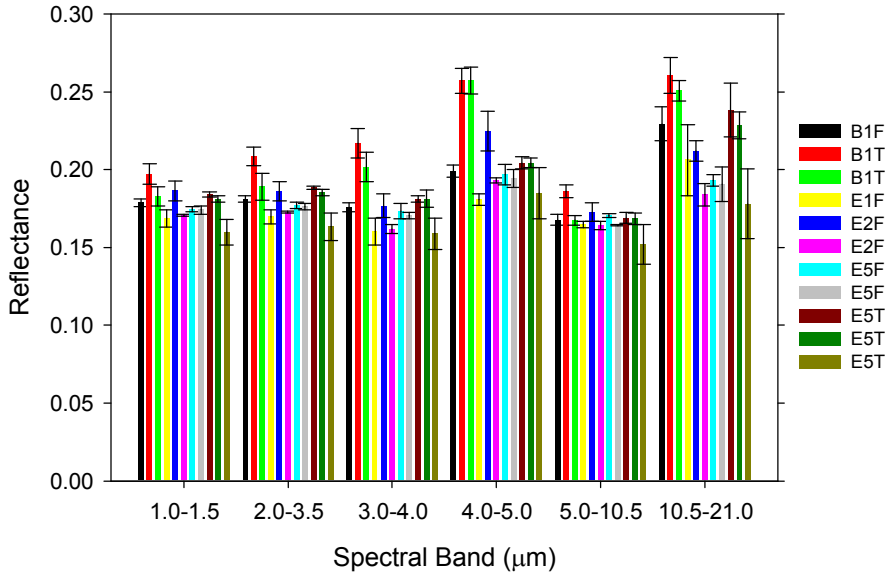

E3F and correlated samples

- E3F was a non-thermoset McMaster-Carr sample
- E2F and E5F samples were correlated, but have higher reflectance signals
- E5T and E8T samples cannot be discriminated based on the high similarity, even though the samples are visibly different

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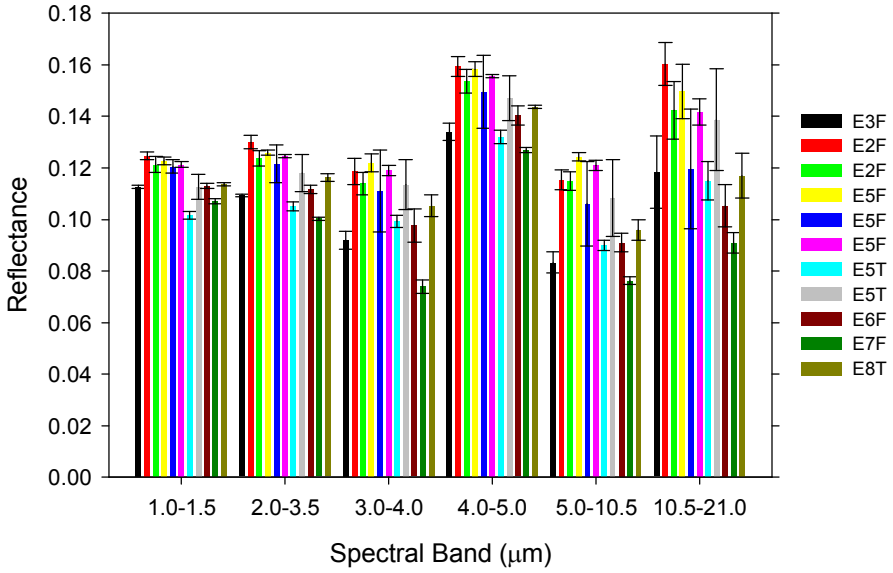

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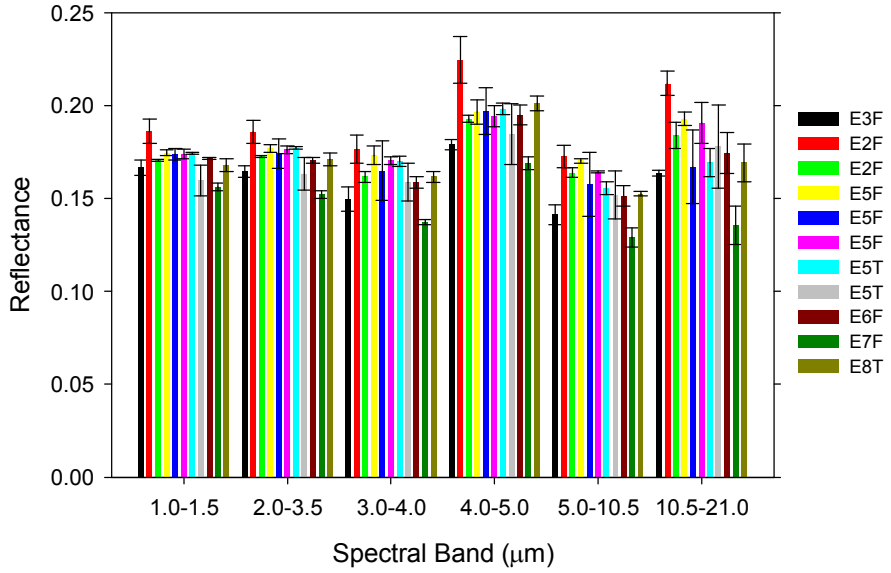

E4F and correlated samples

- E4F was visibly different from other epoxy samples, a non-thermoset McMaster-Carr sample
- Correlated with many of the 'other' samples
- Even with correlation coefficients above 0.95 threshold, differences at detailed bands allow discernment

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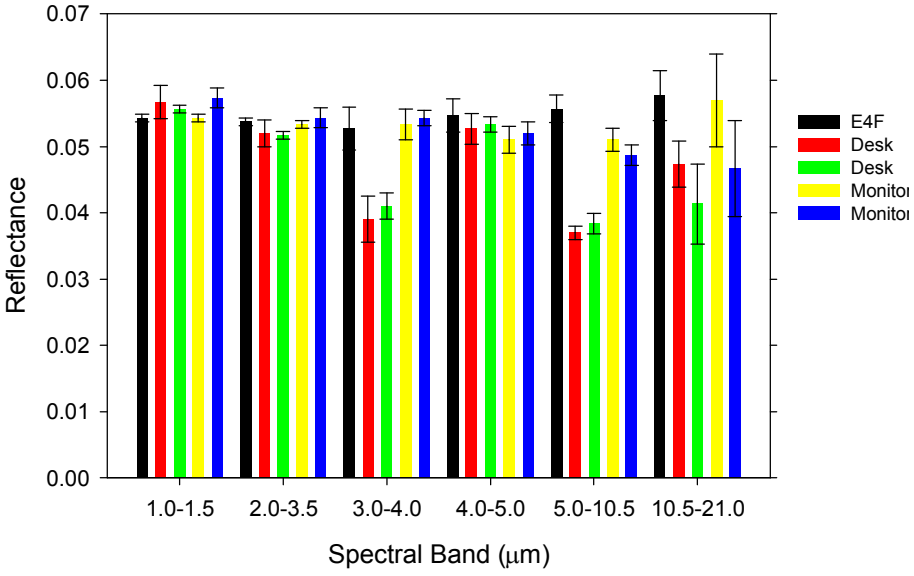

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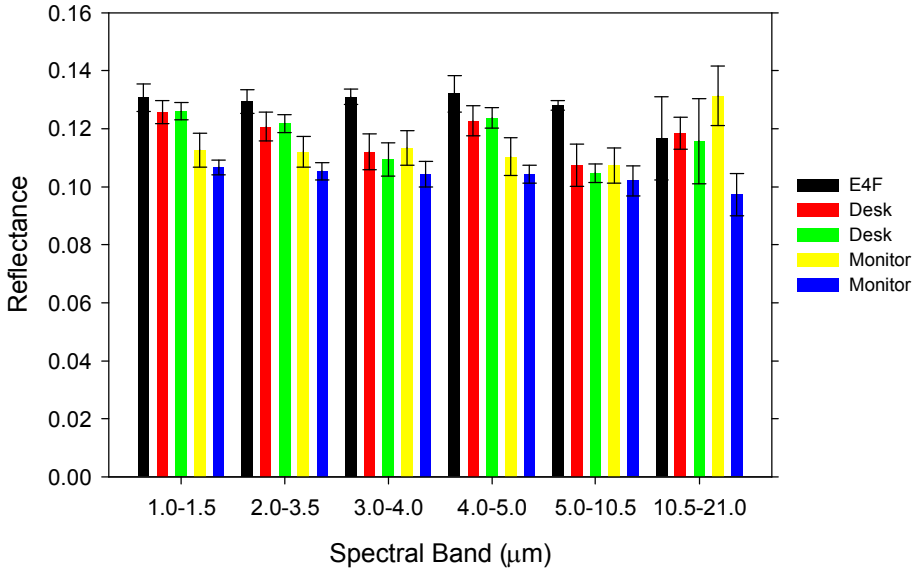

Textured E5F and correlated samples

- Both E5F_T samples were correlated
- One had very small uncertainty range, can be distinguished from other samples
- The other had large uncertainty range, difficult to distinguish from other samples

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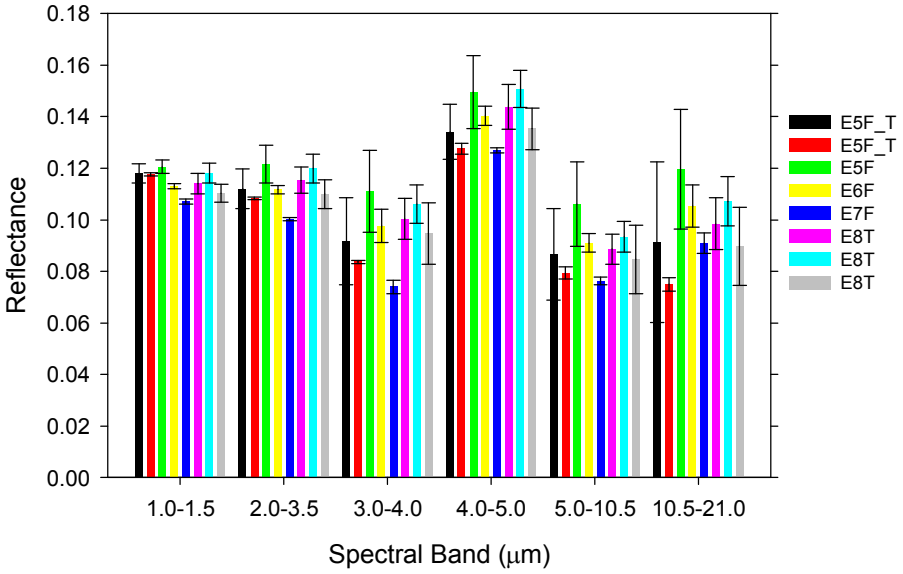

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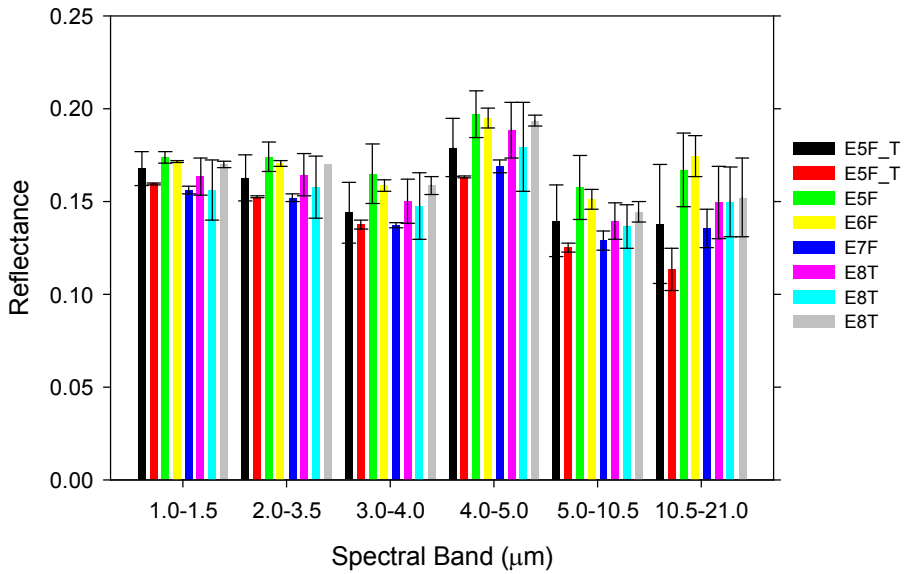

Integrated Reflectivity

- Room temperature (300 K) gray reflectivities were calculated for all samples
- Epoxies had reflectivities between 0.078-0.178
- BMI samples were between 0.132-0.158
- Paper reflectivity was surprising, but consistent with literature values

Sample	Reflectivity	Sample	Reflectivity
E1F	0.130	B1F	0.132
E1T	0.178	B1T	0.158
E2F	0.137		
E3F	0.105	Aramid	0.062
E4F	0.057	Desk	0.043
E5F	0.137	Monitor	0.054
E5F Textured	0.078	Paper	0.111
E5T	0.123		
E6F	0.100		
E7F	0.086		
E8T	0.114		

Summary

Proposed methods were generally unsuccessful for three apparent reasons:

- Composite binders tested in this effort appear to all be spectrally very similar
- Surface roughness and fiber morphology were found to be at least as important as the binder to the reflectometer signal
- The data uncertainty was too high to draw substantial conclusions in many cases
- Spectral resolution from the instrument insufficiently fine

- For unidirectional tape samples, the orientation of the instrument relative to the fibers may be important, and should be more carefully observed in the future.
- Reflectivity data for several different types of composites were measured

Acknowledgements

Randy Foster helped with data collection.

Support from the WSEATprogram is appreciated.

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