Composite thermal damage – correlation of short beam shear data with FTIR spectroscopy

Portable, non-destructive analysis

Application Note

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Abstract

Agilent is a member of the Center for Composite Materials Industrial Consortium with a different product focus than most other Consortium members. Agilent is in the business of producing portable, handheld non-destructive testing analyzers based on Fourier transform infrared spectroscopy (FTIR). Along with aircraft manufacturers, Agilent has been extensively analyzing advanced composites with this technology.

A specific area Agilent has been asked to explore is degradation processes in composites caused by a variety of external physical and chemical stresses. The following example describes work Agilent has carried out at CCM, correlating the physical effects of thermal exposure to composites with molecular analysis via FTIR.

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4300 Handheld FTIR

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Introduction

The use of medium modulus carbon fiber resin composite in aircraft manufacturing is expanding. There is a corresponding increasing need for non-destructive testing tools that can assess the affect of environmental stresses on composite. Environmental stresses can include moderate to high temperatures, ultraviolet (UV) light, or chemicals such as paint stripper, hydraulic fluid, jet fuel or de-icing solutions. Many of these environmental stresses may weaken the resin component of the composite, reducing the overall strength of the composite before the composite part cracks or de-laminates.

One of the tools that is used for assessing the condition of composite resin is Fourier transform infrared spectroscopy (FTIR). Traditionally, samples are removed and brought to a laboratory for FTIR molecular analysis. At the suggestion of airline manufacturers, Agilent developed the 4100 ExoScan FTIR, a high performance, handheld FTIR spectrometer. The rationale for this approach is to enable the analyzer to be brought directly to the site of the sample, enabling the condition of the composite resin to be assessed non-destructively.

Analyzing thermal damage in composites by FTIR

In collaboration with the University of Delaware Center for Composite Materials, the 4100 ExoScan system was used to detect changes in an epoxy carbon composite that result from exposure to thermal stresses. The specific goal was to determine if the handheld FTIR can detect changes in the molecular structure of the composite induced by the increasing exposure temperatures, and then to determine if the spectral changes can be correlated to changes in physical strength of the composite. One of the challenges was to determine if the FTIR system, which analyzes the surface of the composite, can provide information that is indicative of physical affects in the bulk composite.

Cytec 977-3/IM7 epoxy/carbon composite coupons were made by the CCM staff and then were individually exposed for 15 minutes to temperatures ranging from 350 to 550 F. Using the 4100 ExoScan FTIR, spectra of different spots on these coupons were measured at 8 cm^{-1} resolution; each analysis took approximately 30 seconds. The interlaminar shear strength was measured by a short beam shear (SBS) test on seven replicate samples at each temperature. The infrared (IR) measurements were correlated to the relative strength measured by SBS.

As the exposure temperature increases, increasingly strong carbonyl bands at 1680 and 1720 cm^{-1} appear, indicating oxidation of the epoxy resin. Additionally, other bands in the fingerprint region indicate degradation of the epoxy backbone.

Epoxy carbon composites decrease in strength due to temperature exposure before disbanding and de-lamination can be observed.

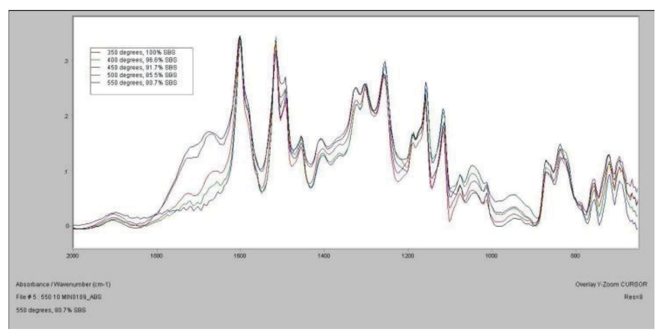

Figure 1. Changes that occur in the spectra of 977-3 composite as a function of temperature. The carbon fiber component of the composite strongly scatters the IR light; however, the resin component can still be easily measured

This decrease can be observed by a decreasing SBS strength with increasing temperatures as is shown in Figure 2. This decrease is due to degradation of the resin, which can be measured by FTIR as shown above. Therefore, a correlation should exist between the FTIR measurement and the SBS strength.

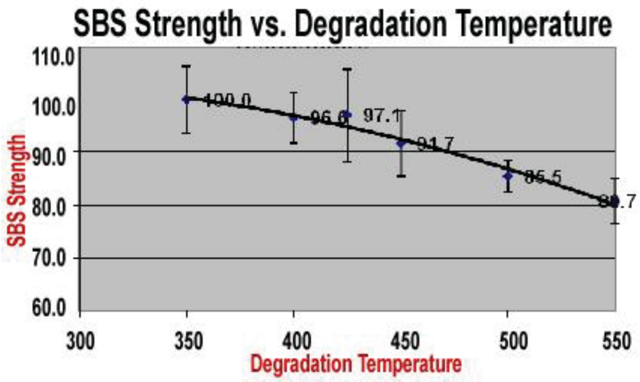

Figure 2. Decreasing SBS strength with increasing temperature

The relationship between the relative SBS strength and the FTIR spectra measured by the 4100 ExoScan was determined using a partial least squares correlation. The data was pre-processed using a Savitsky-Golay first derivative and mean centering. The correlation of actual to predicted values using a cross-validation was 0.95.

Using the above correlation, predictions were made on a separate set of samples. The results of the separate validation set are listed in Table 1. An average error of 1.89% was obtained, which is well within the standard deviation of the short beam shear data itself of 3 to 8%. This shows that FTIR can be used to accurately predict the reduction in strength of an epoxy carbon composite due to high temperature exposure.

Table 1. Separate validation set results

Temperature (°F)	Actual relative SBS	Predicted SBS	% Error
350	100	103	3.0
350	100	99.9	0.1
400	96.6	96.8	0.2
400	96.6	95.4	1.2
450	91.7	92.8	1.1
450	91.7	94.3	2.6
500	85.5	82.3	3.2
500	85.5	86.3	0.8
550	80.7	80.6	0.1
550	80.7	87.3	6.6
		Average error	**1.89%**

Conclusion and further work

In this brief project, the ability of a handheld FTIR to both detect thermal damage in a composite material and to correlate the damage to the physical change in strength as indicated by SBS measurements is demonstrated. Though the FTIR method is fundamentally a non-destructive surface measurement, in this study the results do effectively correlate to changes in the strength of the bulk composite.

Agilent plans far more extensive work with CCM in the use of the 4100 ExoScan FTIR for detecting damage in composites as well as other projects associated with the importance of understanding sur faces in composites, metals and ceramic materials. In other on-going projects, the 4100 ExoScan system has already provided benefit in detecting surface contamination that can affect bonding and coating processes, as well as in assuring that coatings, primers and paints are of correct thickness, uniformity and physical configuration. Agilent is interested to determine if other CCM Industrial Consortium members have problems or projects, for which this technology could provide.

In addition to the 4100 ExoScan FTIR, Agilent offers the 4200 FlexScan FTIR. The 4100 ExoScan and 4200 FlexScan both provide easy, handheld FTIR analysis, but with slightly different form factors. The 4200 FlexScan has the same optical components as the 4100 ExoScan, but the optics and electronics are separated by a cable. This makes the handheld component smaller, while still providing the spectroscopic performance needed for a variety of applications. The 4200 FlexScan has a 3 pound optical head attached to a 4 pound battery and electronics pack. Although the form factor is different, use of the two systems, including the software, is identical. While the 4100 ExoScan provides an integrated, compact package, the 4200 FlexScan has a smaller size to fit into spaces with tight clearances

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