Composite heat damage measurement using the handheld Agilent 4100 ExoScan FTIR

Easy, non-destructive analysis of large parts

Application Note

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Abstract

The Agilent 4100 ExoScan FTIR enables large parts to be easily, non-destructively measured to determine the amount of heat damage present. Thermal damage causes oxidation of the epoxy resin component of composite materials, which can lead to a loss of strength. Fourier transform infrared (FTIR) spectroscopy is uniquely suited for identifying and quantifying oxidation products, and so it is an effective method for identifying heat damaged areas. However, even though FTIR is a non-destructive technique, it is traditionally carried out in a laboratory, where large samples must be destructively disassembled so that they can be accommodated by a spectrometer. The 4100 ExoScan FTIR is a portable handheld FTIR, which can measure heat damaged composites on large parts in situ. The 4100 ExoScan enables even large parts to be measured in any orientation. Customized optics are designed to obtain an optimum focus when the 4100 ExoScan is placed in contact with the sample, enabling easy sample measurement.

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4300 Handheld FTIR

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Introduction

Composite materials represent one of the most significant advances in materials for high performance applications. Carbon based composites can produce structures of great strength that are less than half the weight of comparable aluminum structures. These materials can be of great benefit to aviation, as lighter materials translate into lower fuel consumption and higher performance aircraft. Composites have been used since the mid 1990s in military aircraft and are now being developed into commercial aircraft as well.

Composites also hold the promise of reduced maintenance due to the fact that they do not atmospherically oxidize; atmospheric oxidation is the prime reason for maintenance of metal parts. Reduced maintenance was a key reason for the US Navy developing composite aircraft parts. Durability and maintenance concerns are not eliminated by composite parts but the concerns do change. Composites are far more susceptible to damage by heat and ultraviolet light than metal parts. Both heat and ultraviolet light can degrade the resin part of the composite by initiating chemical reactions such as oxidation. Degradation of the resin component can severely reduce the overall strength of the composites, often leading to premature failures. Heat stress due to lightning strikes, engine overheating or engine fires have been noted to cause loss in mechanical strength, enbrittlement and eventually cracking.

There are a few techniques that monitor heat or ultraviolet damage of the resin component. Carbon/epoxy composites exposed to greater than 550 °F show cracks, disbands, delaminating and surface blistering. These structural problems can be detected by many different non-destructive inspection (NDI) techniques. However, current NDI techniques cannot observe a diminishment of physical properties resulting from lower, incipient heat damage. This incipient heat or UV damage causes chemical changes in the resin component before cracks or other physical problems are present. The oxidation due to heat or UV exposure is recognized as a concern by both composite manufactures and regulatory agencies.

There are many examples in the literature of mid-infrared (mid-IR) spectroscopy employed to detect incipient heat damage. In 1990, Oak Ridge National Laboratory published the report 'Composite Heat Damage Spectroscopic Analysis'. This study evaluated several spectroscopic techniques for the determination of the extent of heat damage on composite panels. The study found that diffuse reflectance mid-IR spectroscopy and laser induced fluorescence had the highest degree of success; however, both techniques required further development in construction of a field unit that could be used for NDI. A second study, published in 1994 by the Navy Manufacturing Program through The Great Lakes Composites Consortium and the Navy Center of Excellence for Composites Manufacturing Technology, again found that diffuse reflectance mid-IR spectroscopy produced favorable results for the measurement of incipient heat damage; however, work on a field ready unit was still needed.

Composite measurement using the Agilent 4100 ExoScan FTIR

The small size and portability of the Agilent 4100 ExoScan FTIR enables measurement of the sample directly in the field. The 4100 ExoScan FTIR has two available sample interfaces. The internal reflectance interface (ATR) is used for highly absorbing or non-reflective samples. For composite samples, the external reflectance sample interface is used. The infrared (IR) light from the 4100 ExoScan is reflected off the sample at an angle of 45 degrees. The 4100 ExoScan collects the diffusely scattered light from the sample surface. This provides a high signal-to-noise spectrum of the composite resin, enabling quantitative determination of the sample degradation. Samples can be measured over the full mid-IR range from 4000 to 650 cm^{-1} at a maximum resolution of 4 cm^{-1}. The measurement of heat damage on aircraft composites was made at 8 cm^{-1} resolution; measurements took approximately 20 seconds.

The 4100 ExoScan FTIR software provides multiple levels of user interaction. The Administrator level allows full use of the system to develop methods, while the Technician level allows untrained users to collect data and view results of established methods. This allows the system to be used by experienced personnel to conduct the measurements needed to develop an analysis; then the same system can be put into the hands of manufacturing or maintenance personnel to routinely check for damaged parts.

Epoxy resin heat damage measurement

Several aircraft composites, which had been exposed to high temperatures, were measured with the 4100 ExoScan FTIR. Characteristic spectra showing the effect of heat damage on 977-3 and 5250-4 composites are shown in Figures 1a and 1b respectively. Similar changes are seen in both resin systems. The most obvious changes are an increase in the ester and perester bands near 1700 cm^{-1}. Other changes in the fingerprint region of the spectrum, also corresponding to oxidation of the epoxy backbone, can also be observed.

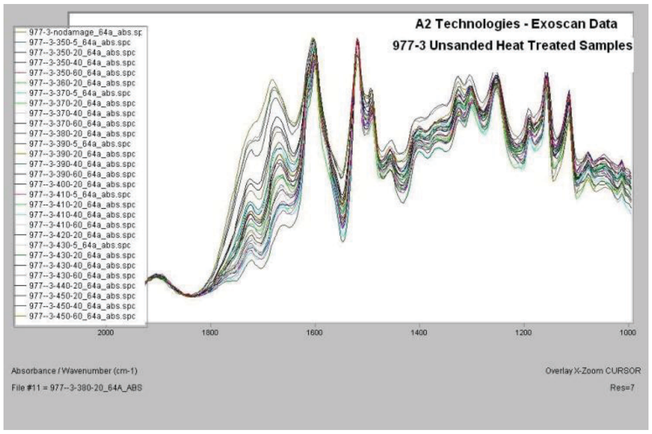


Figure 1a. Spectra of heat damaged samples of 977-3 composite

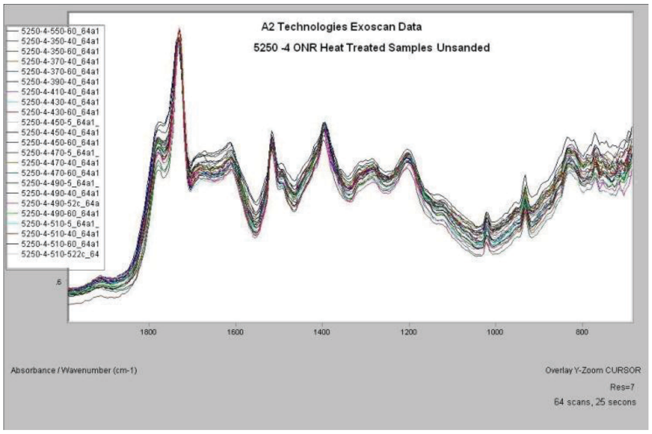


Figure 1b. Spectra of heat damaged samples of 3250-4 composite

Oxidation of the epoxy resin due to heat damage has been shown to reduce the physical properties and mechanical strength of a composite. If a measurement can predict the amount of heat to which a sample has been exposed, the physical properties can also be predicted. Six samples of BMS 8-212 composite were heat treated for twenty minutes. Treatment temperatures increased by 25 degrees Fahrenheit) from (350 to 500 °F). Spectra of the samples were measured on the 4100 ExoScan FTIR at 8 cm^{-1} resolution with a scan time of approximately 25 seconds. Spectra of these samples are shown in Figure 2. The sample spectra were correlated to the treatment temperature using a partial least squares (PLS) algorithm. The data was pre-processed by mean centering; first derivative spectra using a Savitsky-Golay algorithm with nine point spacing were used for the correlation. The statistics for the calibration are shown in Figure 3. Only one loading vector was required for the calibration, producing a standard error of cross validation of approximately 10 degrees as is shown in Figure 3a. An excellent correlation of 0.93 was obtained for the plot of actual versus predicted concentration using a cross validation routine. The first loading vector is shown in Figure 3b; the loading vector displays the areas of the spectrum that correlate to the temperature differences. The loading vector indicates that changes occur throughout the fingerprint region of the spectrum, which can be correlated to the exposure temperature.

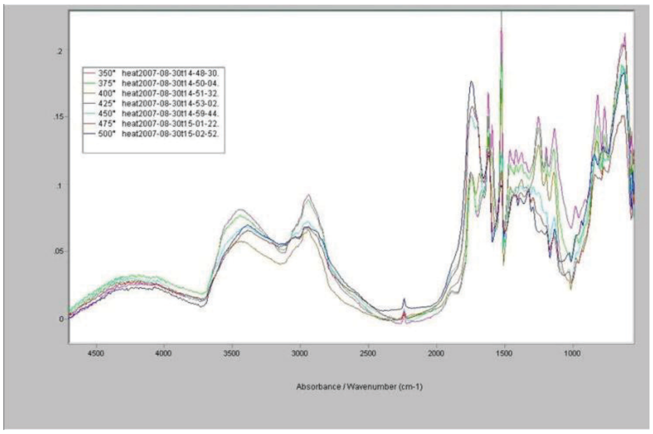


Figure 2. Spectra of BMS 8-212 composite after heat treatment at the listed temperatures for twenty minutes

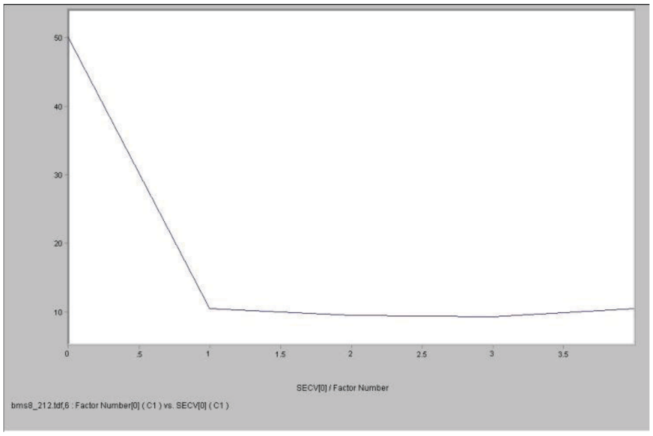


Figure 3a. Plot of standard error of cross validation versus factor number for 4100 ExoScan FTIR spectra of heat treated BMS 8-212 composite samples. This plot shows that with only one loading vector, the standard error is approximately 10 °F.

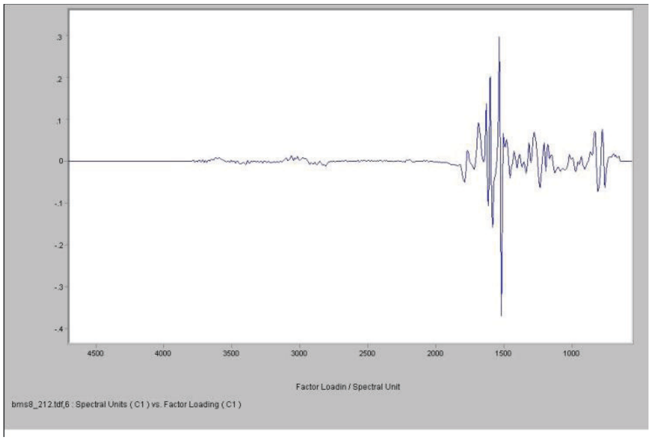
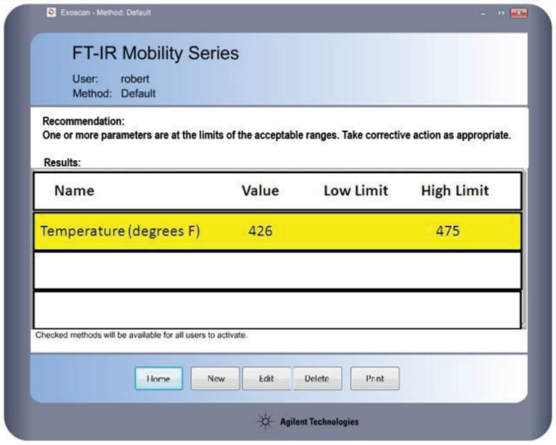


Figure 3b. First loading vector of PLS calibration correlating 4100 ExoScan FTIR spectra of heat treated BMS 8-212 composite samples to the treatment temperature. This loading vector shows that changes throughout the IR fingerprint region can be correlated to the temperature change.

Using the above PLS calibration, a method was created and then stored in the ExoScan 4100 FTIR software. The spectrum of a composite sample that had been heated to 425 °F was measured with the 4100 ExoScan and then the spectral data was processed using the aforementioned method. The results of the analysis, shown in Figure 4, display the predicted exposure temperature.



User: robert
Method: Default

Recommendation:
One or more parameters are at the limits of the acceptable ranges. Take corrective action as appropriate.

Results:

Name	Value	Low Limit	High Limit
Temperature (degrees F)	426		475

Checked methods will be available for all users to activate.

Figure 4. 4100 ExoScan FTIR software screen capture showing the results from a sample measured with the 4100 ExoScan using the composite exposure temperature method. The result is presented in yellow because it is between the marginal and critical limits.

4100 ExoScan FTIR quantitative methods can be programmed with marginal and critical limits. The software will warn the operator by presenting the result in yellow if it is above the marginal value or red if it is above the critical value; values within the acceptable range are presented in green. The predicted exposure temperature of 426 °F is presented in yellow because the critical value was set at 400 °F.

Composite aircraft part measurement

The 4100 ExoScan FTIR system has been used to measure aircraft parts that have been heat damaged. Figure 5 shows a 4100 ExoScan system being used to measure a part damaged in an engine fire. Due to heat transfer of the supporting structures, the damage due to the fire was not uniform across this part. In cases such as this, there is a need to measure the extent of damage at various points on the part in order to determine which areas of the composite can be repaired.

Figure 5. A 4100 ExoScan FTIR being used to measure heat damage on a composite aircraft part, which had been damaged by an engine fire

Composite parts are often sanded to remove paint or remove damaged portions before a repair. The sanding process exposes areas that are carbon- fiber rich and thus tend to absorb a large amount of the IR radiaton. For this reason, alignment of the IR beam with the fibers is required to maximize signal from these sanded surfaces. The 4100 ExoScan FTIR enables the user to view a display of spectral intensity while rotating the instrument and thus sample is measured when the highest intensity is obtained.

Spectra measured from four areas of the damaged part are shown in Figure 6. Differences in the carbonyl region are observed based on the measurement location. As previously stated, an increase in the absorbance in the carbonyl region is characteristic of heat damage. The heat damage was found to be highest in areas closest to metal support structures, most likely due to a large heat transfer between the metal and the composite. This information enables maintenance technicians to determine which areas of the part need to be completely replaced or whether the areas can be repaired via a patch.

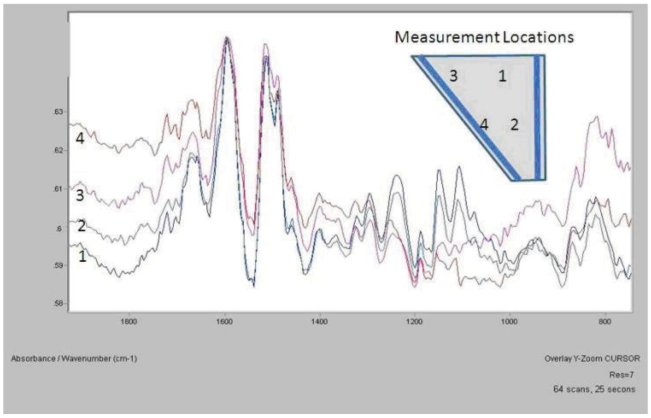


Figure 6. Measurements of a heat damaged composite aircraft part measured with the 4100 ExoScan FTIR. The locations of the measurements are shown on the inset diagram. The areas showing the greatest amount of heat damage are adjacent to the heat conducting metal support structures.

Conclusion

The Agilent 4100 ExoScan FTIR is shown to be an effective, non-destructive means of monitoring heat damage in composite materials. In addition to measuring the spectra of composites, a calibration method was developed using the 4100 ExoScan FTIR software that predicts the temperature to which the composite part had been exposed.

Since the 4100 ExoScan FTIR is a field-deployable system, even large composite parts and components can be readily analyzed by FTIR without the need for disassembly for laboratory measurement.

In addition to the 4100 ExoScan FTIR, Agilent also 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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