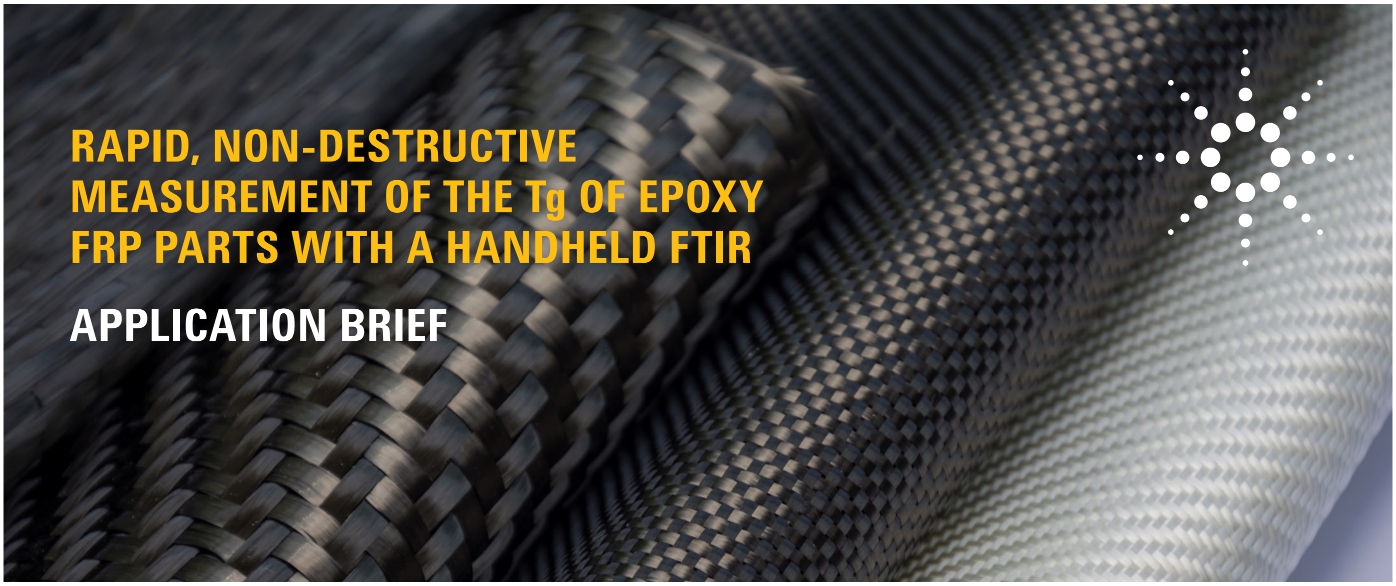

RAPID, NON-DESTRUCTIVE MEASUREMENT OF THE Tg OF EPOXY FRP PARTS WITH A HANDHELD FTIR

APPLICATION BRIEF

The Agilent 4300 Handheld FTIR fitted with the specular 45° external reflectance interface used in this study

Major benefits of determining T_g with a handheld 4300 FTIR

- Sample measurement in seconds
- Non-destructive multipoint mapping of a sample
- No consumables required
- Portable, easy-to-use instrument
- Method-driven software guides the user in each step of the analysis
- On-board analysis for immediate, color-coded results
- Results are independent of the orientation of data collection

Introduction

Assessing the degree of cure of a 2-part epoxy-based fiber-reinforced polymer (FRP) composite is typically performed with Differential Scanning Calorimetry (DSC). The glass transition temperature (T_g) determined using DSC directly relates to the degree of cure of the polymer in the composite material. To ensure optimal performance of the final product, the curing must be held until the T_g reaches a minimum specified level for that material. If that T_g value is not achieved before the material is demolded, the part could be under-cured and may deform or be otherwise unfit for purpose.

Fourier Transform Infrared (FTIR) spectroscopy has long been used measure complex matrices, with the data being analyzed using chemometric methodologies. In this study, FTIR spectra were collected during the chemical changes related to various temperature time combinations through a real part's curing regime (Figure 1). These changes to the spectra were correlated with the T_g values of the samples, as determined by DSC, to develop a multivariate calibration model.

The Agilent MicroLab Expert software was used to develop the model to correlate the spectra to the T_g values. Replicate spectra from samples with a range of known T_g were collected. These spectra were then separated into calibration and validation sets and used to optimize and test the model. The model was then integrated into an analysis method. In a production environment such a method could be incorporated into a Standard Operating Procedure (SOP).

Agilent Technologies

Figure 1 shows FTIR reflectance spectra of the curing resin at different T_g temperatures.

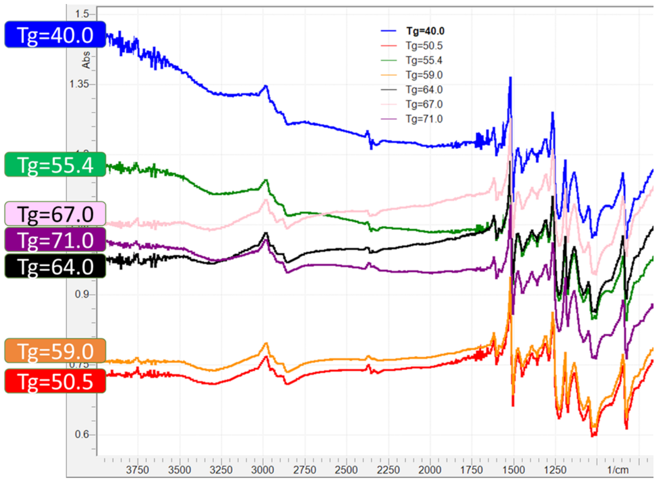

Figure 1. Specular reflectance spectra of the polymer at different Tg values.

Figure 2 shows the calibration set of known values (circles) and the predicted values of the validation set (diamonds). The model is based on the partial least squares (PLS1) algorithm and shows excellent linearity with R^2=0.9894. The T_g values were determined by the DSC reference technique and those values correlated to the specular reflectance FTIR spectra.

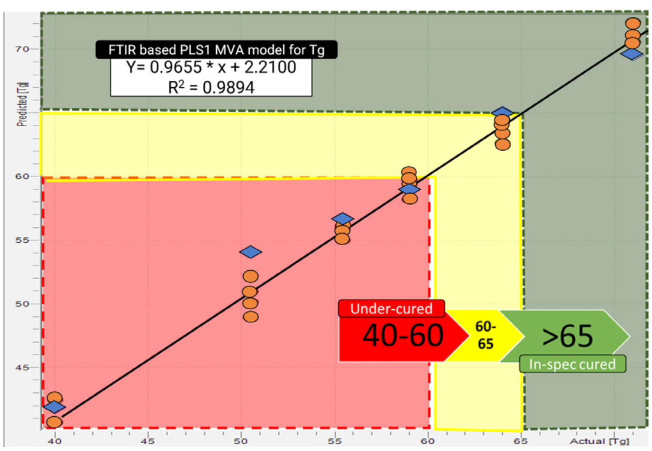

Figure 2. Calibration set model known values (orange circles) and the validation set predicted values (blue diamonds) used with the correlation model show excellent linearity.

One of the advantages of the 4300 FTIR MicroLab Expert software is the ability to visually display the results in an easy to understand format. In this case, color-coding the results red, yellow or green allowed the user to quickly determine whether the required T_g temperature had been reached, as shown in Figure 3.

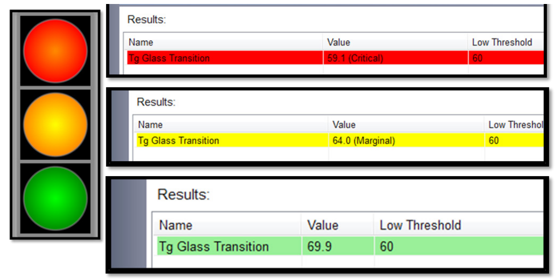

Figure 3. The Microlab Expert interface can color-code the results, allowing the user to quickly determine if the correct cure level has been reached.

The results show that adding the multivariate PLS1 algorithm to the onboard Microlab software on the 4300 FTIR allows it to accurately determine the T_g of an epoxy based composite polymer. All results were collected on the hand-held instrument.

The 4300 FTIR has a full complement of sampling interfaces, allowing the best choice to suit the purpose. In this case, 45° specular reflectance provided a clear advantage over the more commonly considered ATR. The non-contact 45° specular reflectance interface provides better spectral information while leaving the sample undamaged.

The FTIR can achieve accuracy equivalent to DSC with measurements taking less than 30 seconds; in contrast to 30-40 minutes for DSC. With its portability and fast measurements, the 4300 FTIR can non-destructively create a multi-point spatial map across large or high value objects. This allows the user to identify areas of differing cure levels.

The 4300 FTIR is an ideal high throughput instrument for measuring T_g and thus cure level. High selectivity, speed and sensitivity reduce the need for DSC measurements while enhancing quality assurance.

For more information:

Contact your local Agilent representative or visit:

www.agilent.com/chem

This information is subject to change without notice.

Published March 1, 2018
Publication number: 5991-9075EN

Agilent Technologies