SII NanoTechnology Inc.

TGA AS PROBLEM-SOLVING TOOL: COMPOSITIONAL ANALYSIS OF COMPOSITES

Problem

A chemist working for a company which produces glass filled composite materials wishes to have an easy to use means of determining the compositional analysis of the samples. It is important to have a technique which is precise, accurate and provides a high degree of resolution.

Solution

Thermogravimetric analysis (TGA) provides a means of accurately determining the compositional analysis of materials, including composites. TGA measures the mass loss of a sample as it is heated under controlled conditions and provides excellent quantitative compositional information.

The Seiko TG/DTA6000, in particular, offers outstanding features for the measurement of the composition of a sample, including:

- high sensitivity for detection of small weight losses
- stable baseline performance across entire temperature range
- horizontal purge gas flow
- simultaneous measurement of DTA signal
- conversion of DTA signal to DSC output (mW)
- direct temperature calibration
- auto stepwise isothermal mode of operation for the highest resolution
- automatic 20-point temperature calibration
- automatic gas switching available
- state-of-the-art robotic accessory for auto-sampling

For the determination of the composition analysis of glass filled composite materials, the following procedure is recommended:

Instrument:	Seiko TG/DTA6200
Heating rate:	20°C/min
Initial temperature:	25°C
Purge gases:	nitrogen from 25 to 600°C
Purge gases:	air from 600 to 1000°C
Flow rate:	200 ml/min
Sample container:	open Pt pan

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The Seiko TG/DTA can be calibrated for temperature response using high purity metal standards, such as indium and tin. The simultaneous DTA signal permits direct temperature calibration yielding the highest possible accuracy of temperature response, since the thermocouple is in direct contact with the sample platform. The software featured with the SSC5300 data station permits the DTA signal to be automatically converted to user-friendly DSC output (mW) based on the heats of melting of the metal standards.

Displayed in Figure 1 are the TGA results obtained on a nylon-glass composite sample. The plot shows the percent mass and the rate of mass loss as a function of the sample temperature. The sample yields a small mass

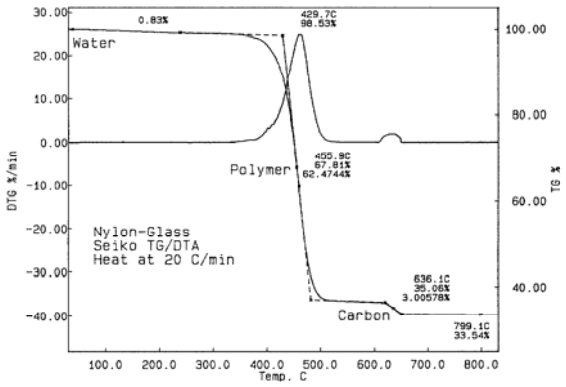

Figure 1

loss between room temperature and 250°C due to the evolution of absorbed moisture (0.83%). The thermal degradation of the nylon polymer is observed as a large weight loss (62.47%) beginning at 429°C. At 600°C, the purge gas was automatically switched from nitrogen to air to burn off the carbon residue. The carbon weight loss, due to oxidation, occurs at 636°C with a mass change of 3.01%. The residue left behind is that of the inert glass and this has a mass of 33.54%.

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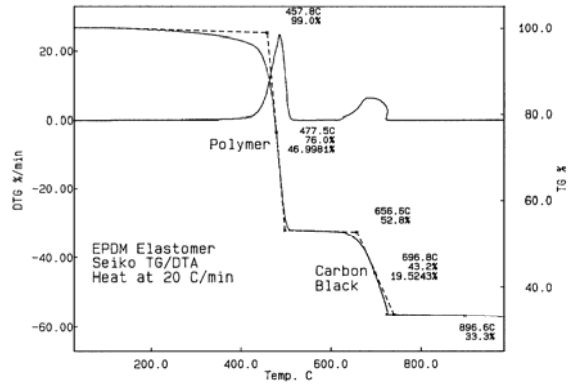

Figure 2

The TGA compositional results obtained on an EPDM glass filled elastomeric sample are shown in Figure 2. This sample yields 47.00% polymer, 19.53% carbon black and 33.3% glass filler.

Shown in Figure 3 are the TGA results generated for a phenolic-glass composite

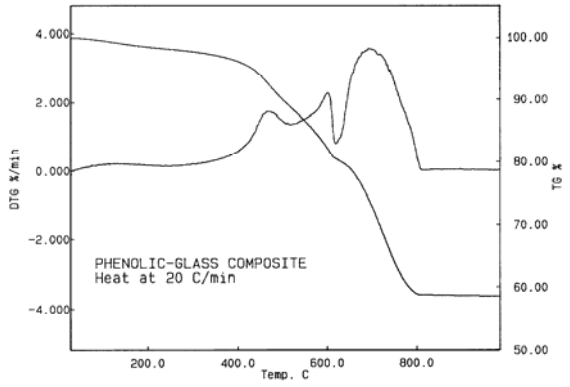

Figure 3

sample when heated at a rate of 20°C/min. The degradation of the phenolic resin takes place at 450°C. A two-stage degradation event takes place for the phenolic resin, as the derivative trace shows by the occurrence of two peaks. However, the two steps severely overlap preventing good resolution. After the purge gas is switched to air at 600°C, the carbon burns off, leaving behind the fiberglass residue.

Better, more highly resolved degradation information can be obtained using the auto stepwise isothermal mode, featured with the Seiko TG/DTA instruments. With this approach, the sample is heated at a constant rate until the rate of mass loss exceeds the

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user selected entrance threshold value (usually 300µg/min). The TG/DTA holds the sample under isothermal conditions and permits degradation to take place until the rate of weight loss becomes insignificant or less than the user selected exit threshold value (15µg/min). The instrument automatically resumes heating until the next significant mass loss event is encountered. In this manner, the auto stepwise mode yields excellent separation or resolution between overlapping weight loss events.

Shown in Figure 4 is a comparison of the TGA results obtained on the phenolic composite sample using both the standard heating mode and the auto stepwise mode. Much better resolution is obtained using the auto stepwise approach and the two-step thermal degradation

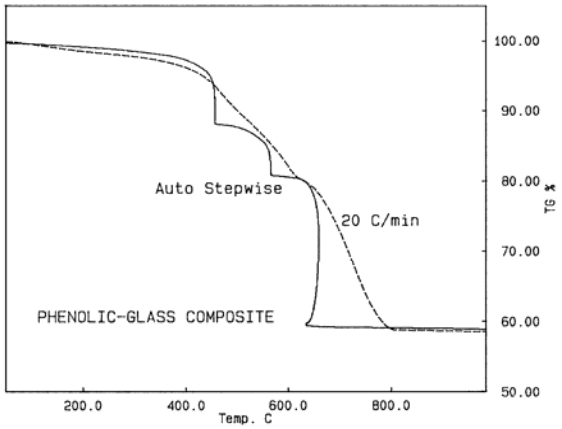

Figure 4

between 400 and 600°C is cleanly separated. The TGA mass loss results, as obtained via the auto stepwise approach, are displayed in Figure 5 for the phenolic composite specimen. The first weight loss occurs at 455°C with a mass loss of 11.65% followed by the second thermal degradation event at 560°C with a weight loss of 7.37%. The weight loss due to the oxidation of carbon is 21.53%. The inert glass residue comprises 59.26%.

Summary

The Seiko TG/DTA provides excellent compositional analysis information on

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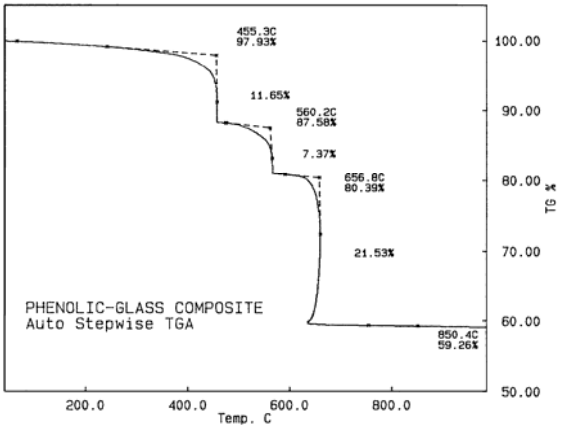

Figure 5

composite materials. The auto stepwise mode, featured with the instrument, provides the highest possible degree of resolution between successive weight loss events.

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