TA Instruments

Thermal Analysis & Rheology

Modulated Thermogravimetric Analysis: A new approach for obtaining kinetic parameters

Introduction

Thermal analysis is the generic name used to describe a series of analytical techniques which measure physical and chemical changes in materials as a function of temperature and time. Thermogravimetric analysis (TGA) is one of the most widely used thermal analysis techniques and specifically measures the weight changes (gains and losses) in materials. Such measurements provide information about the material's thermal stability as well as the material's composition.

One application of TGA is the estimation of product life time. TGA decomposition kinetics can provide useful aging stability information and lifetime predictions. The TGA decomposition kinetics method (ASTM Standard E1640 [1]) uses the data from experiments run at several heating rates to calculate kinetic parameters including activation energy and the specific rate constant. Limitations of this traditional kinetics method include the amount of time required for several experiments, and the fact that kinetic parameters are calculated as a single value, based on the assumption that a single decomposition mechanism controls the entire decomposition range of the material.

TA Instruments has recently introduced a new approach, Modulated TGA™ (MTGA™), which delivers superior kinetic analysis without the time and single decomposition mechanism limitations of the traditional technique. This patent pending* approach is the result of TA Instrument's continuing commitment to technological innovation and leadership. MTGA™ shares its roots with the now widely accepted Modulated DSC™ (MDSC®) technology. Like High Resolution TGA™, MTGA is an optional capability that can be added to the TA Instruments TGA 2950.

Principle of Operation

MTGA™, like its predecessor MDSC®, superimposes a sinusoidal temperature modulation on the traditional underlying heating profile. Just as it does in MDSC, the application of a sinusoidal temperature program produces a change in the response of the test specimen. In MTGA it is the rate of weight loss that responds to the temperature modulations. Evaluation of this rate of weight loss provides an experimental tool to study the kinetics of decomposition or volatile reactions. The sinusoidal temperature program used in MTGA is based on an approach first described in 1968 by Flynn [2].

The MTGA approach can be combined with a linear or HiRes™ underlying heating rate to scan from one weight loss region to another. It can also be used under quasi-isothermal conditions to analyze a single weight loss. Figure 1 shows the results of a Modulated TGA experiment performed on 60% EVA (Ethylene vinyl acetate) where a linear underlying heating rate was used. The plot shows the temperature modulation and the effect of that modulation on the rate of weight loss.

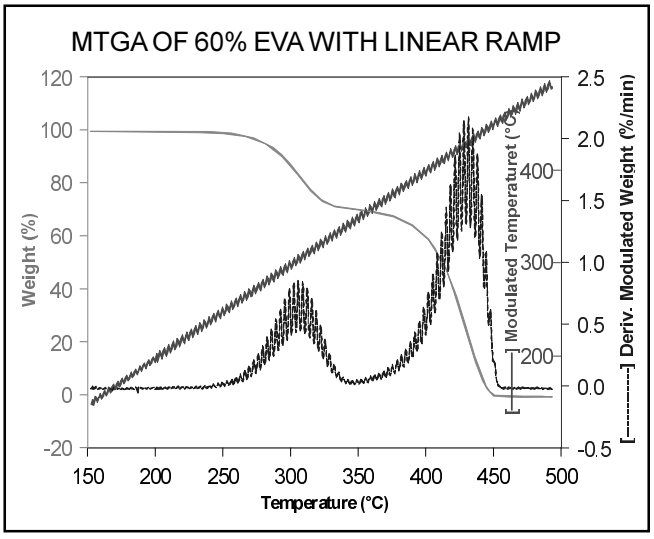

Figure 1

The use of discrete Fourier transformation allows kinetic parameters, such as activation energy and pre-exponential factor, to be calculated on a continuous basis. This permits them to be studied as a function of other experimental parameters such as time, temperature, and conversion. Figure 2 shows the resulting Activation Energy vs Temperature plot from the MTGA experiment shown in Figure 1. Note that the activation energy changes throughout the decomposition process and from step 1 to step 2.

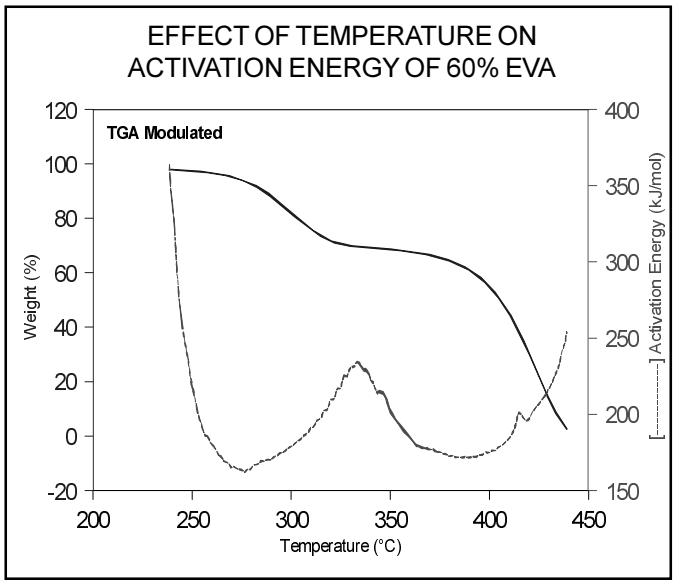

Figure 2

Applications

MTGA applications include :

- Polymer characterization and lifetime studies
- Hazard potential of materials
- Complex reaction studies such as coal combustion
- Drying processes

MTGA Specifications

Temperature Range:	Room Temp to 1000° C
Temperature Modulation Amplitude:	0 to 10° C
Period Modulation (Frequency)	100 to 1000 seconds
Deconvolution:	Discrete Fourier Transformation

Specifications are subject to change.

* TA Instruments has filed patent applications for this technology, and trademark applications for the terms Modulated TGA™ and MTGA™.

References

1. E 1641, Test Method for Decomposition Kinetics by Thermogravimetry, American Society for Testing and Materials.

2. Flynn, J.H., Thermal Analysis, Vol. 2, R.F. Schwenker, Jr. and P.D. Garn, Eds, Academic Press, pp 1111 (1969).

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