

TA no.78

Evolved Gas Analysis of Polystyrene using Simultaneous TG/DTA-MS or FTIR

2007.3

1. Introduction

Polymer materials are widely used in various fields, including electronic and electric parts and medical applications. As the applicability of these materials spreads, research and development is underway to increase heat resistance, durability and to add new functions.

Thermogravimetry (TG) is widely used due to its ability to evaluate heat resistance and determine the quantity of components. Moreover, in recent years, multi-purpose thermal analysis coupled with evolved gas analyzers has become very popular, as it can be used to carry out further analysis of evolved gases during TG measurement, producing details of thermal decomposition processes, which in turn facilitates estimation of sample structure and composition.

Here we introduce the results of an experiment to determine heat decomposition behavior in polystyrene, carried out by analyzing TG/FTIR and TG/MS measurements.

2. Measurement

This experiment was carried out using a thermogravimetry / differential thermal analyzer (TG /DTA 6200) connected to both a Fourier transform infrared spectrometer (FTIR) and a quadrupole mass spectrometer (MS).

Figure 1 shows a block diagram of the TG-MS system.

Measurement conditions were as follows:

TG-FTIR - sample weight 10mg, 40°C/min, in nitrogen 200 ml/min

TG-MS - sample weight 1mg, 40°C/min, in helium 200ml/min

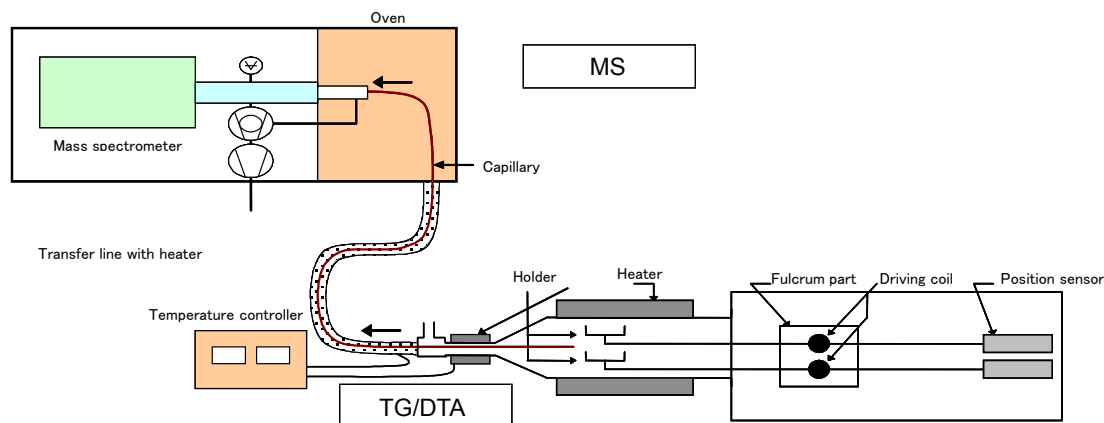


Figure 1 Block Diagram of TG-MS System

3. Results

Figure 2 shows the results for TG / DTA when measurement was carried out using TG-FTIR. Weight loss due to thermal decomposition was seen in the vicinity of 460 °C.

Figure 3 shows the FTIR spectrum. FTIR spectrum detected increases and decreases in gas that match the peak of differential TG (DTG) line in Figure 2. The IR spectrum for 469°C has the largest amount of IR absorbance. This line shows a large between 3200 cm⁻¹ to 3000 cm⁻¹. This is considered the peak that indicates the C-H bonding of the aromatic hydrocarbon and shows the C-H of the benzene ring in the composition of the polystyrene.

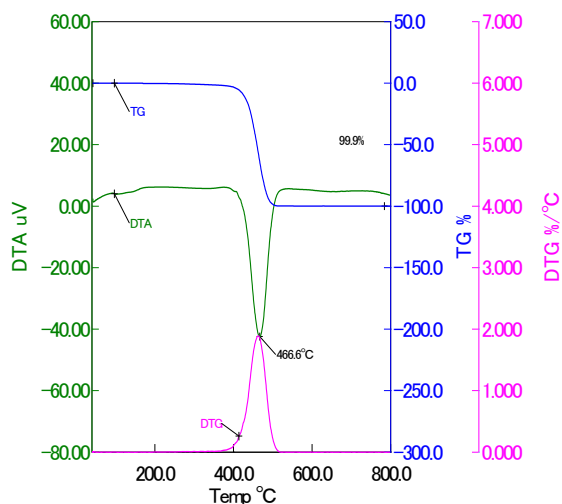


Figure 2 TG/DTA measurement results

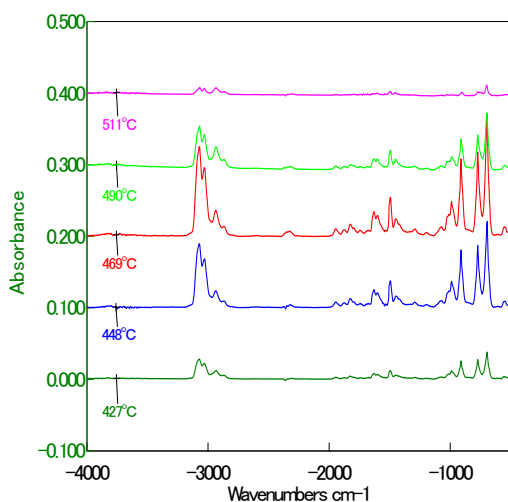


Figure 3 TG/FT-IR measurement results

Figure 4 shows mass spectra detected and TG/DTA curves using simultaneous TG/DTA-MS. The mass spectra correspond fairly well to the DTG curve shown in Figure 2. Mass m/z 104 is assigned to the molecular peak of styrene. This result of MS measurement indicates that styrene evolves from the polystyrene during thermal decomposition.

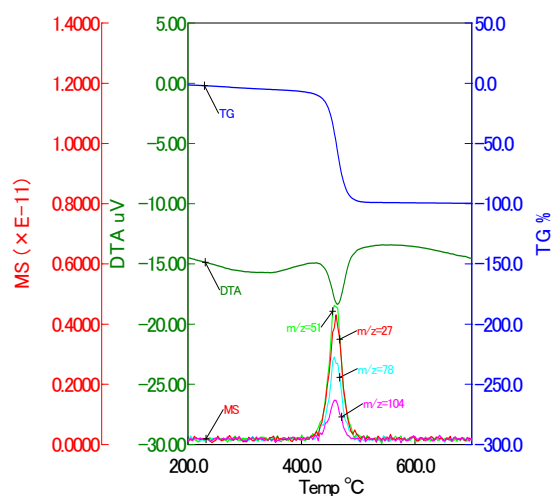


Figure 4 TG/DTA-MS curves of polystyrene

4. Summary

Evolved gas analysis of polystyrene was carried out using TG/DTA-MS or TG/DTA-FTIR. FTIR measurement makes it possible to identify the structure of gases evolved because the characteristic absorption bands of the material are detected. Furthermore, MS measurement is highly sensitive, and can even detect minute gas evolution. It is possible to identify evolved gases.

By using simultaneous TG/DTA-MS or TG/DTA-FTIR, we can investigate the thermal decomposition mechanisms or gas species of samples during TG measurement. These methods can also be used to detect residual volatile components during molding and to inspect for hazardous gases during decomposition.

Reference

Application Brief TA No. 66, Toshihiko Nakamura, Yutaka Ichimura (1995)