

TA NO.67 MAR.1995

Thermal Decomposition Measurement of ABS resin II

- TG/FT-IR Measurements in air -

1. Introduction

A previous brief¹⁾ presented a TG analysis of ABS resin. Quasi-isothermal thermogravimetry and thermogravimetry / Fourier transform infrared spectroscopy (TG/FT-IR) were combined to analyze thermal decomposition of ABS resin in N₂.

When TG is used to evaluate the heat resistance of materials such as polymers, it is important that atmospheres be near actual conditions. Therefore, air must be considered along with inert gases such as nitrogen and argon when selecting atmospheres.

With this in mind, this brief presents an example of TG/FT-IR analysis of the oxidative decomposition of ABS resin in air²⁾.

2. Measurements

Commercially available boards (moldings) of ABS resin were used as samples.

A TG/DTA220 Thermogravimetry / Differential Thermal Analyzer was connected to a SSC5200H Disk station. A gas transfer system was connected to the TG/DTA220 to send the formed gases to a FT-IR gas cell.

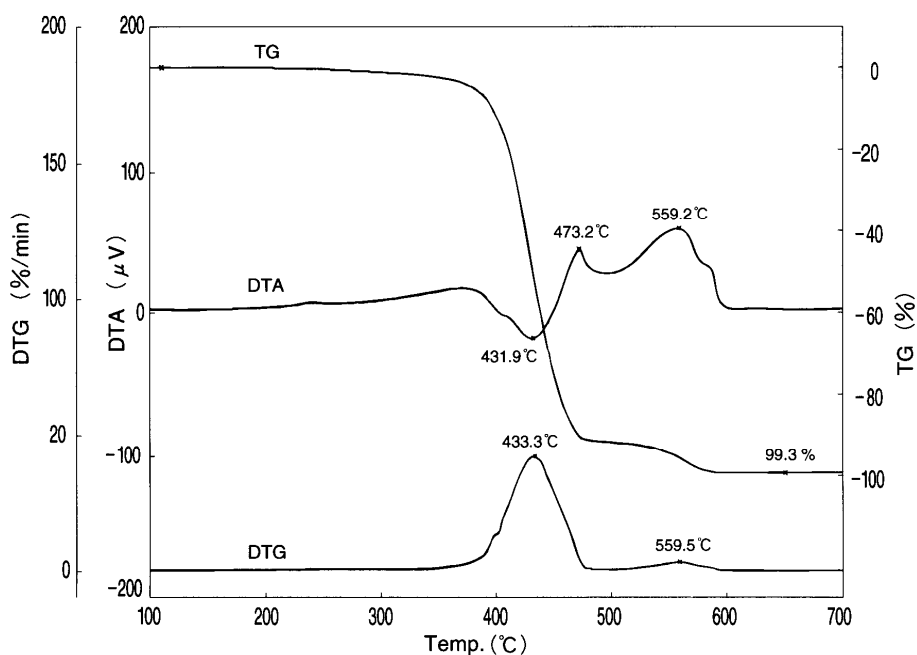


Figure 1 TG/DTA Measurement Results for ABS resin in air

For the TG/DTA measurements, the temperature range was room temperature to 700°C and the heating rate was 20°C/ min. The sample weight was 10mg and a platinum sample container was used. The atmosphere was air and the flow rate was 200ml/min.

For the FT-IR measurements, the wave number resolution was 8cm⁻¹, the resolution time was 1.7seconds per spectrum and a DTGS detector was used. Furthermore, the gas transfer line and gas cell temperature was set to 280°C.

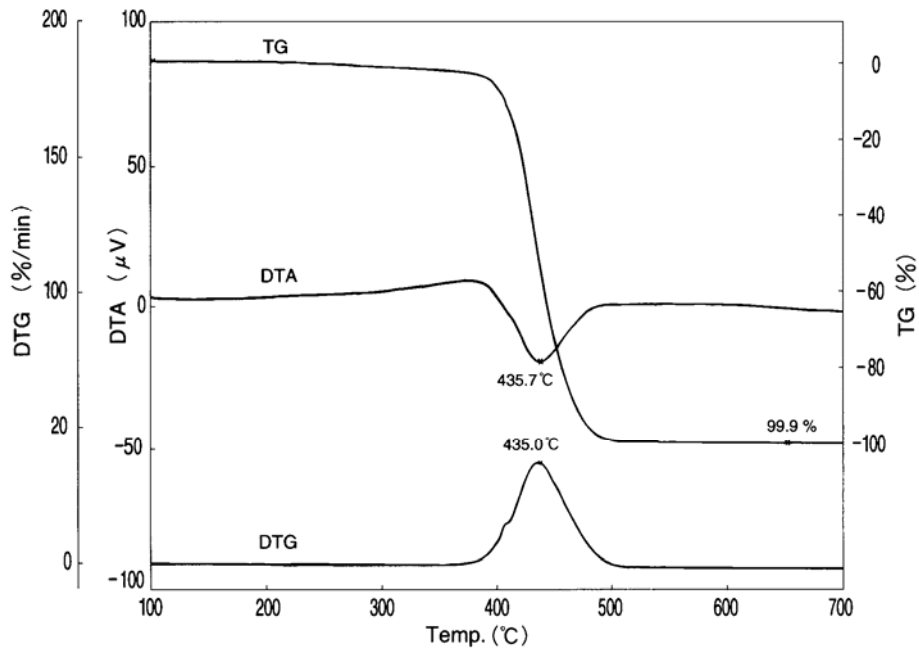


Figure 2 TG/DTA Measurement Results for ABS resin in N₂¹⁾

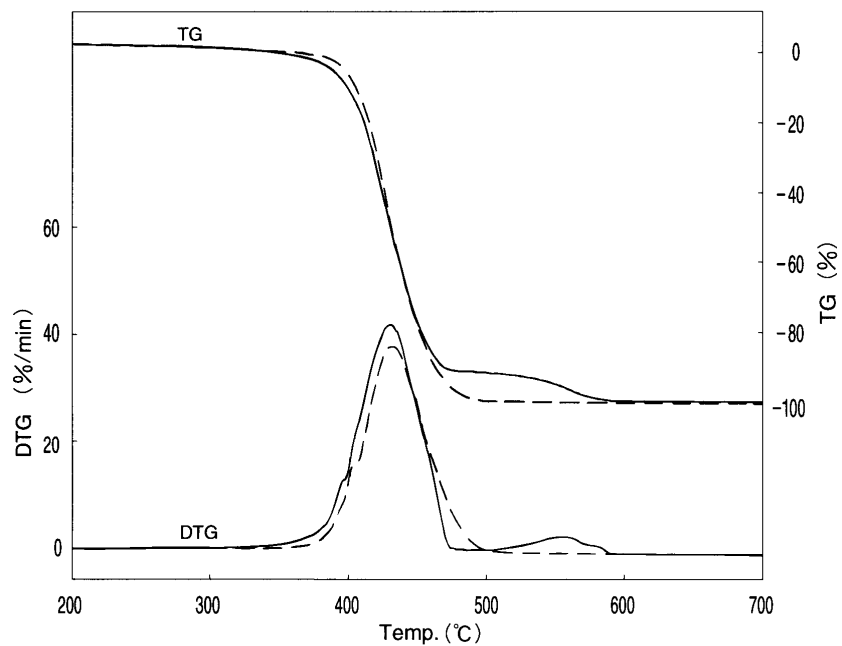


Figure 3 Comparisons of TG and DTG curves in air and N₂

———— : air
 - - - - - : N₂

3. Measurement Results

3-1 TG/DTA Measurement Results

Figure 1 shows the TG/DTA results for ABS resin in air. The TG curve has a two-stage weight decrease between 350°C and 600°C. The DTA curve has both endothermic and exothermic peaks during decomposition. In general, thermal decomposition causes endothermic peaks and oxidative decomposition causes exothermic peaks. During the first weight decrease, it is likely that the endothermic and exothermic peaks overlap and thermal and oxidative decomposition occur back to back. Conversely, the second weight decrease has only an exothermic peak. While the weight decrease was low at about 7%, the peak was a relatively large, which suggests that only oxidative decomposition occurred in this temperature range.

Figure 2 shows the results for N₂¹⁾. The TG curve has a one-stage weight decrease between 350°C and 500°C and the DTA curve has only an endothermic peak. This suggests that only thermal decomposition occurs in N₂.

Figure 3 shows the TG and DTG curves for air (Figure 1) and N₂ (Figure 2). The data shows that decomposition during the first stage of decomposition occurs earlier in air (around 400°C).

3-1 FT-IR Measurement Results

Figure 4 shows the IR absorption spectrums of decomposition gases formed during measurements in air (Figure 1) and N₂ (Figure 2). The numbers in the figure indicate the spectrum conditions.

- ① 400°C in N₂
- ② 400°C in air
- ③ 560°C in air

In other words, Spectrums ① and ② are initial decomposition stages and Spectrum ③ is the second weight decrease in air (Figure 1).

Spectrum ① is the first stage in N₂¹⁾. It shows an absorption peak near 3000cm⁻¹ for hydrocarbons formed by decomposition. Conversely, Spectrum ②, the first stage in air, shows a CO₂ peak between 2300cm⁻¹ and 2400cm⁻¹, as well as the peak near 3000cm⁻¹. This difference indicates that ABS resin decomposition in air forms hydrocarbons and CO₂ during the first stage.

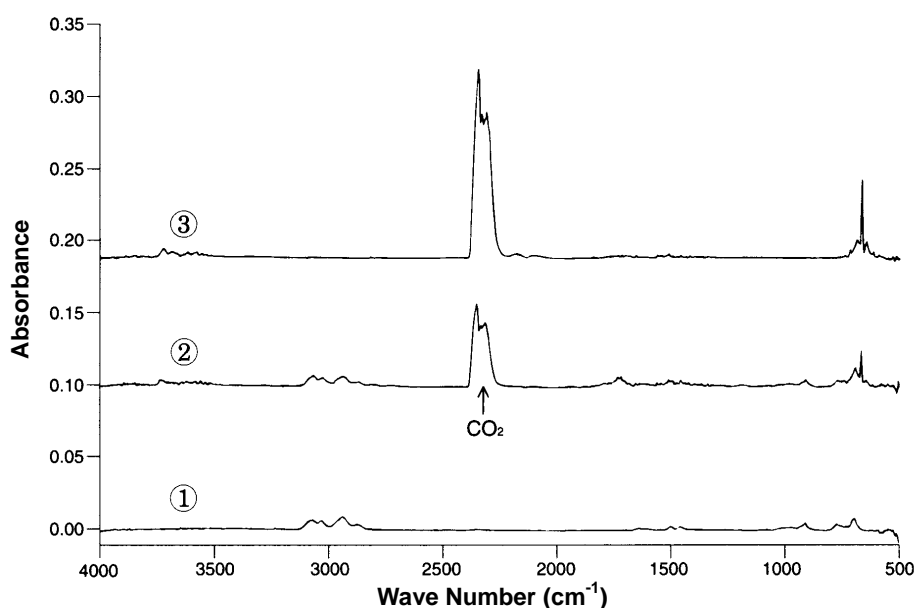


Figure 4 IR absorption spectrums of decomposition gas in air and N₂
 ① 400°C in N₂ ② 400°C in air ③ 560 °C in air

In Spectrum ③, the second stage in air, the only significant absorption peak is between 2300cm^{-1} and 2400cm^{-1} due to CO_2 . This indicates that the decomposition gas in this temperature range is mostly CO_2 . In addition, the DTA curve for this period has a large exothermic peak. It is likely that the second weight decrease occurred because the carbide that formed during the first stage of decomposition went through further oxidization decomposition in this temperature range.

4. Summary

To follow up on a previous application brief, TG/FT-IR was used to analyze the decomposition of ABS resin. TG measurements of ABS resin in air found weight decreases in two stages of decomposition. FT-IR was then used to analyze the decomposition gases formed. During the first weight decrease, carbide and CO_2 were detected during ABS resin decomposition. During the second weight decrease, CO_2 was detected from the oxidative decomposition of carbide formed during the first weight decrease.

Reference

- 1) Application Brief TA No.66, Hitachi High-Tech Science Corporation (1995)
- 2) R. Kinoshita, T. Nakamura, Y. Ichimura and Y. Teramoto, The 28th Japanese Conference on Calorimetry and Thermal Analysis, 1116A (1992)