



Analysis of Ceramic Composite Materials with Double-Shot Pyrolyzer and Peripheral Devices  
Part 3 : Analysis by Heart cutting EGA-GC/MS Technique

In cases where multiple peaks are observed on an evolved gas analysis (EGA) curve, EGA-GC/MS technique is most useful to determine the origin of each peak. In this technique, a Selective Sampler and Microjet CryoTrap (MJT-1030E) are used. Species generated in each temperature region are selectively introduced into a separation column, and trapped. This is followed by GC/MS analysis. Since four peaks (A through D) were observed on the EGA curve of the ceramic composite material described in *Double-Shot Pyrolyzer® Technical Note PYA1-010E*, evolved gases produced in each of four temperature regions were introduced into a GC in programmed heating mode. This gave chromatograms shown in Fig. 2. It was found that peak A was due to dibutyl phthalate (DBP); peak B, butyl methacrylates (iso-BMA, n-BMA), i.e. a thermal decomposition product of polybutylmethacrylate and C<sub>25</sub>-C<sub>40</sub> saturated hydrocarbon; peak C, iso-BMA and n-BMA; peak D, styrene monomer (S), dimer (SS), and trimer (SSS), i.e. thermal decomposition products of polystyrene. As shown in this example, further analysis can be made by GC/MS analysis of each temperature region of EGA curve.

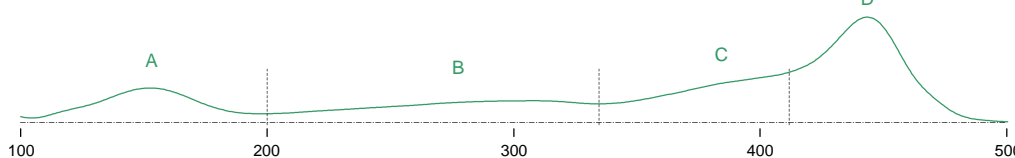


Fig. 1 EGA Curve of a Material Used for Injection Molding

(See *Double-Shot Pyrolyzer® Technical Note PYA1-010E* for analytical conditions.)

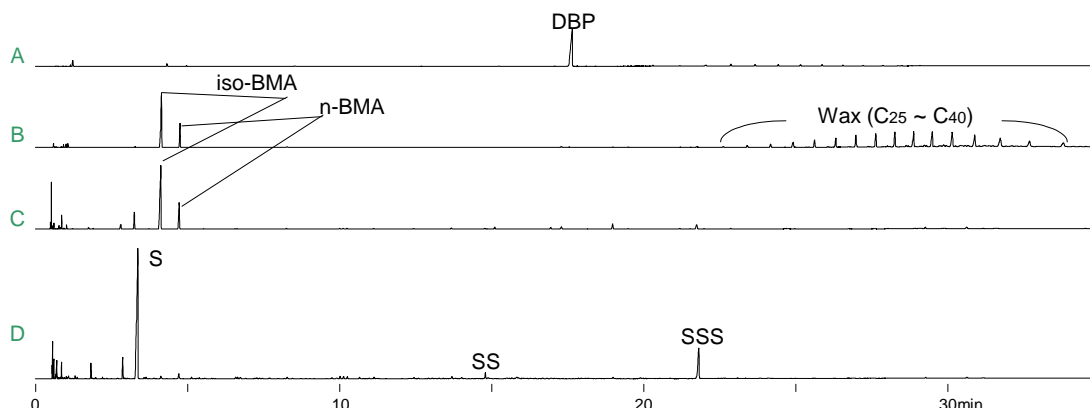


Fig. 2 Chromatograms of Evolved Gas Composition from Each Temperature Region

Pyrolysis furnace temp : 100°C-500°C (20°C/min), Column head pressure : 140kPa, Split ratio : 1/50  
Separation column : Ultra ALLOY\*-5 (5% diphenylpolysiloxane), Length : 30m, 0.25mm id, Film thickness : 0.25µm  
GC oven temp : 40°C-320°C ( 10°C/min), Sample : 300µg, Detector : MS (m/z=29-400, 2 scans/sec)

Excerpt from A. Hosaka, K. Sato, C. Watanabe, H. Ohtani, S. Tsuge, *J. Mass Spectrom. Soc. Jpn.*, 46, 332 (1998)

Keyword : Selective Sampler, Microjet Cryo-Trap, Ceramic Composite Material, EGA, EGA-GC/MS

Application : General Polymer Analysis

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