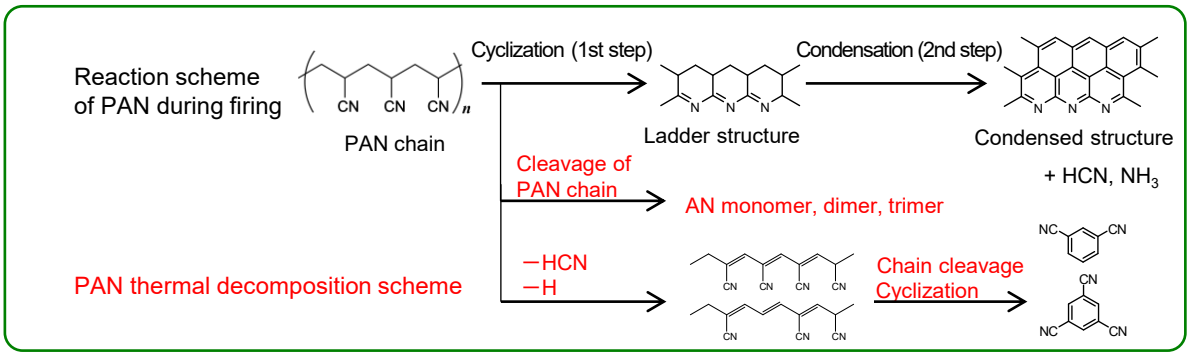
Analysis of the stabilization mechanism of heat-treated polyacrylonitrile-based carbon fiber precursors

Part 2 Analysis of evolved gases from precursors with varied heat-treatment times

[Background] In the previous report (PYA1-107E), evolved gas analysis (EGA)-MS was performed on PAN-based carbon fiber precursors with varied heat-treatment times. Information on the structural changes caused by the heat-treatment was obtained. In this report, the decomposition products from the fiber precursors produced by the heat-treatment with varied processing times were analyzed using EGA-GC/MS.

[Experimental] PAN-based carbon fiber precursors containing less than a few percent of methyl methacrylate and itaconic acid as comonomers were heat-treated in air at 215 ºC for 20 minutes. The fibers were subsequently heated to 235 ºC for ever-increasing periods of time (e.g., 0.25 - 20 h.) for the oxidative stabilization. Stabilized fibers and untreated fiber were used as samples. A GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC split/splitless injector was used. The GC/MS system was also equipped with a MicroJet Cryo-Trap (MJT-1035E). The temperature of the pyrolyzer furnace was programmed from 300 to 1000 ºC at 20 ºC/min. Volatile components emanating from the sample were temporarily trapped at the head of a separation column to refocus the band width. Once the furnace temperature reached 1000 ºC, the cooling by liquid nitrogen was turned off and the GC/MS analysis started.

[Results] In the untreated sample, large amounts of acrylonitrile (AN) monomer, dimer, and trimer formed by the cleavage of the PAN chain were observed, but the peak intensity decreased as the processing time increased. The chromatogram of the sample treated for 20 h showed no measurable response in the monomer to trimer region. This suggests that most of precursors are stabilized to the condensed structure by increased heating time.



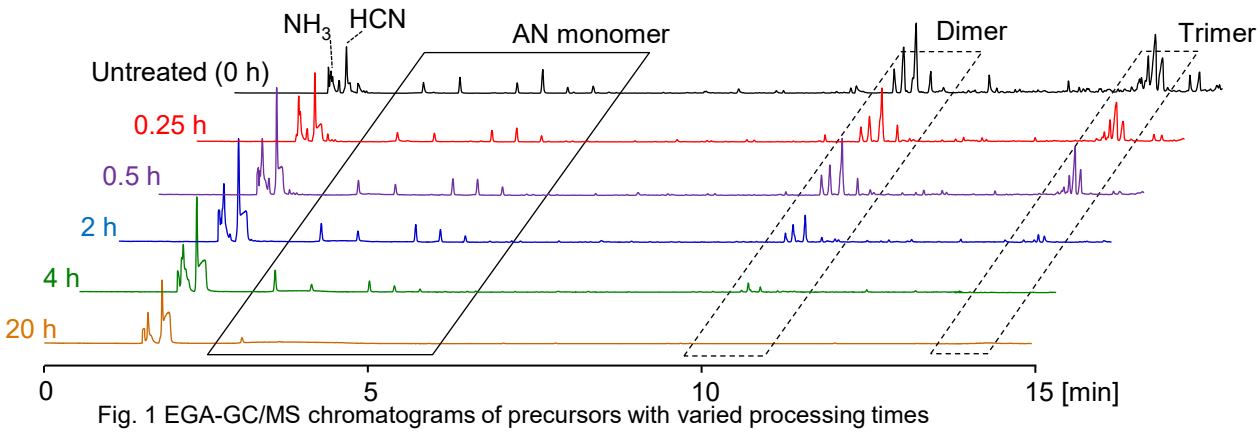


Fig. 1 EGA-GC/MS chromatograms of precursors with varied processing times

Furnace temp.: 100 - 1000 ºC (20 ºC/min, 2min hold), Separation column: UA5-30M-1.0F (*L*=30 m, i.d.=0.25 mm, df=1.0 µm), Column flow rate: 1 mL/min (He), Split ratio: 1/20, GC oven: 40 ºC (3 min hold) - 260 ºC (20 ºC/min, 5 min hold), Sample amount: ca. 0.2 mg.

Ref.: T. Usami, T. Itoh, H. Ohtani, and S. Tsuge, Macromolecules,1990, 23, 2460.

Keywords : Carbon fiber, Polyacrylonitrile

Product used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, MicroJet Cryo-Trap, UA+-5, Vent-free GC/MS adapter

Applications : General polymer analysis, Material analysis, Aircraft, Electronics, Structural materials

Related technical notes : PYA1-107E (Part 1)

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