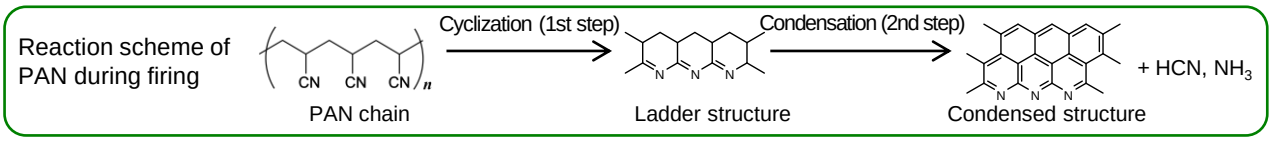
Analysis of the stabilization mechanism of heat-treated polyacrylonitrile-based carbon fiber precursors

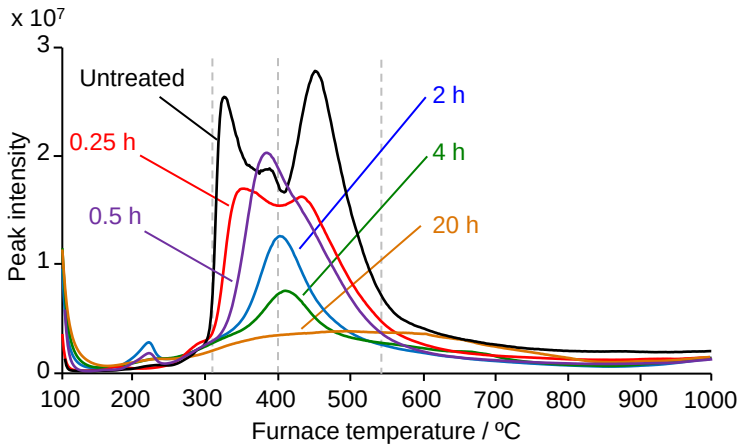
Part 1 Evolved gas analysis of precursors with varied processing times

[Background] Polyacrylonitrile (PAN)-based carbon fiber has excellent physical properties such as high rigidity, low linear expansion coefficient, high heat resistance, and high electrical and thermal conductivities. It is used as a reinforcing fiber for structural materials in a variety of applications from commodity products to aerospace vehicles. This study was designed to provide information on the mechanism of calcination stabilization of PAN-based carbon fiber precursors. All data were acquired using evolved gas analysis (EGA)-MS.

[Experimental] The PAN-based carbon fiber precursors containing less than a few % 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. The stabilized fibers and an untreated fiber were used as samples. A GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC injector was used for EGA-MS analysis and EGA thermograms were obtained.

[Results] The untreated fiber and the fiber processed for 15 minutes showed two peaks (Fig. 1). The peak on the low-temperature side (<250 ºC) of the thermogram may be due to the cleavage of the PAN chain remaining in the sample, the dehydrogenation, and the HCN elimination reaction; the peak on the high-temperature side may be due to the dehydrogenation reaction between the ladder structures and the HCN elimination. On the other hand, the samples processed for 2 hours or more showed a thermogram with a single peak. The apex of the peak decreases with increasing processing time. This may be because the thermal stability of the samples improves as the proportion of the ladder structure in the sample. For the 20-hour treated sample, the peak intensity is lower, and the peak profile extends to ca. 900 ºC. This is presumably because the proportion of the condensed ladder structure increases, and the thermal stability is enhanced.





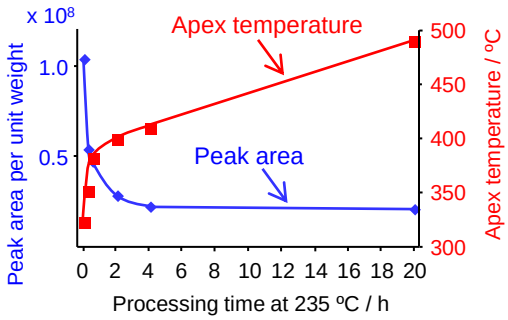


Fig. 2 Processing time at 235 ºC versus EGA peak area and apex temperature

Fig. 1 EGA thermograms of the precursors with varied processing times at 235 ºC

Furnace temp.: 100 – 1000 ºC (20 ºC/min),
EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm),
Column flow rate: 1 mL/min (He), Split ratio: 1/10, GC oven: 300 ºC, Sample amount: ca. 2 mg.

Ref. : T. Usami *et al.*, *Macromolecules*, 1990, 23, 9, 2460-2465

Keywords : Carbon fiber, Polyacrylonitrile, Carbon nanotube

Product used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, EGA tube, Vent-free GC/MS adapter

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

Related technical notes : PYA1-108E (Part 2)

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
http://www.frontier-lab.com/