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**Title:**

Fibrillar structure development of polyacrylonitrile fibers treated by ultrasonic etching in oxidative stabilization

**Abstract:**

Polyacrylonitrile fibers were oxidatively stabilized through ten gradient-elevated temperature zones in sequence. The ultrasonic etching method was used for fibril separation of fibers heated at different temperatures, and the fibrillar structure development was studied by scanning electron microscopy. The voids among fibrils are the weak combination points. Under ultrasonic etching, the voids are enlarged. Subsequently, the solvent enters and spreads among fibrils, which results in the separation of fibrils. Separated fibrils with diameters of 100-400 nm appear in fibers heated at less than 235 ° C. Fibrils in fibers heated from 195 ° C to 235 ° C tend to adhere to each other, and the observed macrofibrils are composed of several to dozens of fibrils. For fibers heated from 195 ° C to 245 ° C, only a few fibril bundles emerge on the skin near the fiber end and the fibrils manifest themselves as numerous protuberances on the cross section. In the ranges of 255-275 ° C, fibrils compactly combine with each other, which suggests insolubility and infusibility, and no separated fibrils appear.