Evolution of Carbon Fiber Microstructure During Carbonization and High-Temperature Graphitization Measured In Situ Using Synchrotron Wide-Angle X-ray Diffraction

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Carbon fiber exhibits a unique combination of material properties, including high tensile strength and modulus, low weight, high temperature resistance, and low thermal expansion, that has made it an ideal choice as a structural component in light-weight composites for a wide range of applications from aerospace to sports equipment. The unique properties of this material are a direct consequence of its constituent highly-oriented graphitic microstructure, which is typically obtained commercially through controlled pyrolysis of either polyacrylonitrile (PAN) or mesophase pitch-based precursor fiber, but can also be produced from non-traditional relatively inexpensive precursors, including polyethylene (PE). Measurement of the characteristics of the graphitic microstructure, such as orientation, domain size, and interlayer spacing as a function of processing conditions is thus critical to understand structure-property-process relationships for these very different starting materials.

This presentation will describe the design and operation of a custom high-temperature tensile device that, when combined with synchrotron wide-angle x-ray diffraction (WAXD), enables us to observe in situ and in real time the microstructural transformation from carbon fiber precursor to high-modulus carbon fiber. Specifically, this tensile device heats fiber bundles at a variable rate from 25 ºC to greater than ~2300 ºC, while simultaneously applying tensile stress, and monitoring the resulting fiber strain. Synchrotron WAXD patterns obtained as a function of temperature reveal the conversion to graphitic microstructure, and provide key insights into the physical processes that occur during carbonization and high-temperature graphitization. Experiments conducted using PAN-, pitch-, and PE-derived fiber precursors reveal stark differences in the carbonization and high-temperature graphitization behavior among these three precursor types.