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Instrumentation of SAXS for the Analysis of Heterogeneous Structure in Polymer

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Polymer materials have hierarchal structure in the very wide range of scale. It is well known that the property is dependent on the hierarchical structure. In order to improve the performance of the materials, clarifying the hierarchical structure in a wide range and the feed back to the manufacturing process are important. However, it is difficult to clarify the hierarchical heterogeneous structure of polymer materials using only single method. Therefore the combination of microbeam small- and wide- angle X-ray scattering (SAXS/WAXS) is useful for evaluation of the hierarchical heterogeneous structural of polymer materials. The BL03XU, in alias, FSBL, in SPring-8 was constructed by consortium of industrial and academic groups and has been used from 20101),2). Structure characterization of advanced materials in the industrial field has been carried out using microbeam SAXS/WAXS method. In addition to the description of the SAXS/WAXS measurement system at BL03XU, we will report on the local structural evaluation of carbon fiber (CF). A hierarchal heterogeneous structure of CFs was visualized in the space resolution of 1 µm using a microbeam and an X-ray imaging technique. The image contrasts were identified by the difference in peak positions corresponding to the void size, the peak width corresponding to the crystallite size, and intensities corresponding to the amount of crystallites and voids. The X-ray scattering images of high-modulus CF are shown in a figure. Nanometer-size voids estimated by SAXS are abundant in the center of a fiber, on the other hand, the crystallite is abundant in the vicinity of a surface was revealed. It is suggested that the voids were generated near the center of the fiber to relax the strain during the crystallization process from the surface during the graphitization of fibers. We succeeded in visualizing the distribution of voids and crystallite of a few nanometers, which cannot be observed by an X-ray transmission imaging method.



[1] H.Masunaga, H. Ogawa, et al, Polymer journal, 2011, 43, 471-477, [2] H. Ogawa, H. Masunaga, et al, Polymer Journal, 2013, 45, 109–116

Figure A mapping image of CF according to the SAXS intensity;(a) and the 002 diffraction intensity of a graphite;(b), respectively. Red and blue parts indicate high and low scattering intensity.

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