Supporting Information for

Observation of Flower-Like Patterns in Syndiotactic Polystyrene/Carbon Nanotube Nanocomposite Films

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1. EXPERIMENTAL

1.1. Materials. The sPS, XAREC SP130, was kindly provided by Japan Idemitsu Kosan Co. Ltd. Its syndiotacticity, \( r_r \), is greater than 97%. The MWCNTs used here are commercial Baytubes C150 P from Bayer, MaterialScience AG, which were produced in a high-yield catalytic process based on chemical vapor deposition (CVD). The C-purity measured by elemental analysis is higher than 95%, provided by Bayer. And its average diameter is about 40 nm according to our transmission electron microscopic (TEM) measurement.

1.2. SPS/MWCNT nanocomposite preparation by solution mixing. The preparation procedure of the sPS/MWCNT nanocomposites has been reported in Ref. 18. Typically, MWCNT dispersion in NMP (0.1 mg mL\(^{-1}\)) was first homogenized at rotor speed of 10000 rpm by a high-shear homogenizer for 2 min, then ultra-sonicated for 1 h. During the ultrasonication, the system was shaken intermittently. The sPS/MWCNT nanocomposite with 1 wt.% of MWCNTs was obtained by adding the homogeneous dispersion of MWCNT/NMP (0.1 mg mL\(^{-1}\)) into sPS/NMP (50 mg mL\(^{-1}\)) solution at 190 °C, and then refluxed for 30 min. Subsequently, the resultant mixture was added into water to precipitate. Finally, the product was filtrated, washed, and dried under vacuum overnight.

1.3. Preparation of sPS/MWCNT composite by melt mixing. The sPS/MWCNT composite sample containing of no solvents was prepared by simple melt mixing of sPS with MWCNT at 260 °C with a CSI-183 MMX Mini Max Molder (CSI Custom Scientific Instrument, Inc.).
1.4. **Film preparation.** Between iron laminates, two polyimide films or aluminum foils were inserted as substrates. And the sPS/MWCNT nanocomposite was sandwiched by the two substrates. The dried sPS/MWCNT nanocomposites sample was hot-pressed between two iron laminators at 300 °C under a pressure of 10 MPa for 10 min. Subsequently, thin films with thickness about 80 μm were obtained by rapidly quenching the nanocomposite into ice-water mixture or slowly cooled down in air. The composite films containing of no solvents were prepared in the same way.

1.5. **Characterization.** The photographic pictures were taken by a digital camera. SEM images were collected using a HITACHI S-4300 field-emission scanning electron microscope measuring at an accelerating voltage of 15.0 kV. The etched film sample was obtained by being immersed in a mixture of 0.60 g KMnO₄ / 20 mL H₂SO₄ / 10 mL H₃PO₄. Raman spectra were recorded with a RENISHAW invia Raman microscope, with an excitation laser at 633 nm. XRD results measurements were conducted on a Rigaku D/max 2400 diffractometer with CuKα radiation (λ = 0.154 nm) at a scanning rate of 4°/min in the 2θ range of 5-30°.
Figure S1. Image for the sPS/MWCNT composite (1 wt% MWCNTs) film obtained at 300 °C under 10 MPa for 10 min, wherein the sPS/MWCNT composite was prepared by melt mixing of sPS with MWCNTs at 260 °C.
Figure S2. XRD patterns of region (a) without or (b) with flower-like patterns for the sPS/MWCNT nanocomposite films being (A) slowly cooled or (B) rapidly quenched.