# Anomalous Small Angle X-ray Scattering for the Distribution and Aggregation of the Gold Nanoparticles in a PS-b-P4VP Diblock Copolymer

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Using anomalous small angle X-ray scattering (ASAXS), we have studied the ordered structure formed by 2-phenylethanethiol-coated gold nanoparticles (NPs) embedded in a spherical poly(styrene-*b*-4-vinylpyridine) diblock copolymer (PS-*b*-P4VP). The SAXS image taken for the copolymer/NPs composite illustrates a powder ring in conjunction with diffraction spots distributed along the powder ring. From the diffraction peak positions and profiles, an ordering spacing of 3.5 nm and an ordering size up to 100 nm are estimated. With the X-ray energy tuned closed to the $L_{III}$-absorption edge of gold (11.919 keV), the diffraction peaks decreases obviously when the photon energy is changed from 11.200 keV to 11.910 keV. This result indicates clearly that the 2 nm gold nanoparticles are responsible for the highly ordered structure observed. The dissociation of the ordered gold clusters in an isothermal annealing at 170 °C is monitored by in-situ small angle X-ray scattering. The four-hour isothermal annealing disrupts the ordered gold clusters completely, while P4VP spheres develop better ordering inside the composite. With ASAXS and TEM, further, we have found that the dissociated gold NPs have relocated themselves to the surfaces of the P4VP spheres, forming NPs shells outside the P4VP cores. We discuss the mechanism of the structural evolution of the gold NPs in the copolymer/NPs composite.

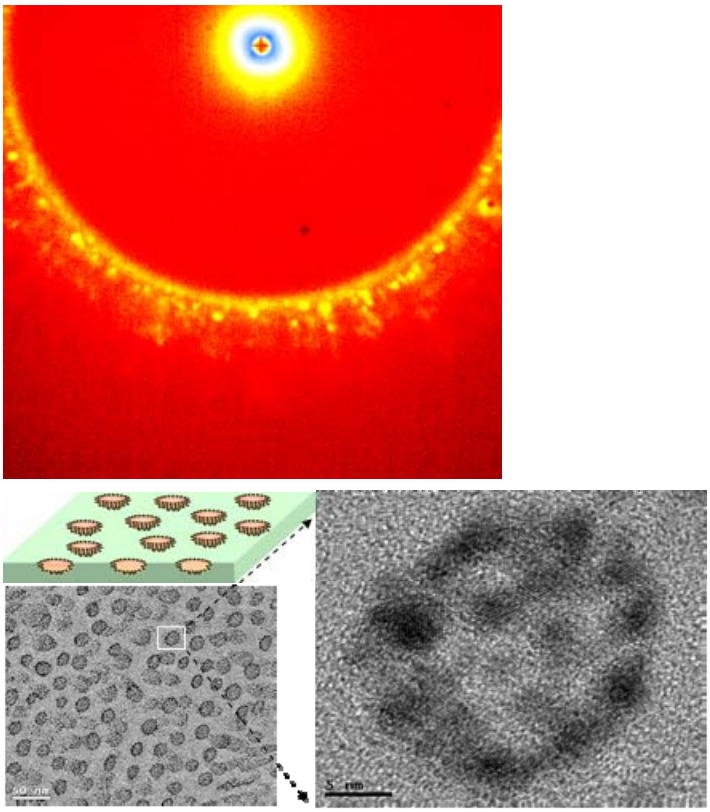

**Figure 1.** SAXS (before sample annealing) and TEM images (after sample annealing) for the PS-b-P4VP/gold NPs composite.