Generation and growth behavior of nanometric palladium species in SSZ-13 zeolite directly investigated by cryogenic transmission electron microscopy (TEM)

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Palladium-impregnated small-pore zeolites have gained a significant attention due to its high catalytic performances toward after-treatment and hydrogen generation. To design an efficient palladium-impregnated small-pore zeolite catalysts, achieving highly dispersed palladium species in small-pore zeolite with high phase homogeneity is crucial. However, the complicity of nanoparticle formation process during the thermal treatment and the electron-beam sensitive nature of the zeolite hinder the rational control of the dispersion and phase homogeneity of zeolite-encapsulated nanoparticles. Herein, we introduce cryogenic TEM coupled with ultramicrotomy to directly investigate the generation and growth behavior of nanometric palladium species in small-pore SSZ-13 zeolite, with preventing unwanted electron-beam induced damage. We elucidate that controlling the thermal treatment that induces rapid nucleation of isolated Pd²⁺ ions and suppressed nanoparticle growth allows successful preparation of uniformly-dispersed nano-sized PdO clusters in SSZ-13.