Thermal Evolution of Carbon Supported Pd Nanoparticles Studied by Time-resolved X-ray Diffraction

Time-resolved X-ray diffraction measurements were performed in situ on a Pd/C catalyst during two successive thermal treatments from 300 to 873 K.
Analysis of the diffraction patterns, as a function of thermal treatment, reveals anomalous features in the evolution of Pd particles. An intermediate $Pd_{1-x}C_x$ phase (x~0.1) is observed and dissolved into pure Pd at about 700K.
Moreover, the size of metal particles, remaining almost constant (~10 nm) during the annealing, abruptly increases to 30 nm in close connection with the dissolution of the $Pd_{1-x}C_x$ phase.
These effects clearly point out an interaction between the metal and the support and suggest that the formation of the $Pd_{1-x}C_x$ phase would prevent the metal particle sintering, thus maintaining the catalyst dispersion at high level. There is no evidence of the formation of a $Pd_{1-x}C_x$ phase during the second temperature treatment since only metallic Pd was detected. As for the growth in particle size, a small increase to 32 nm was observed.
Time resolved XRD patterns were recorded using a translating IP and a reaction chamber to control the gas flow through the sample.