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

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Introduction

The main aim of this work is to investigate in situ the structural and microstructural evolution of Pd phases on a Pd/C catalyst as a function of thermal treatment, in a wide temperature range, by using the TR-XRD technique.
In fact, although several studies have been published on these catalysts, only little is known about structural evolution of catalyst under annealing and the effects produced by the interaction between the metal and the support.

Experiment

TR-XRD patterns have been collected at GILDA beamline using a translating IP chamber [1] and a reaction chamber specifically designed to control the gas flowing through the sample during heating.
The IP support linearly translates behind two vertical slits selecting narrow portion of the Debye rings and a continuous series of diffractograms are collected as a function of time and sample treatment. The data stored in the latent image are recovered and digitalized using a Fuji BAS-2500 laser scanner.
Pd/C samples are contained in a pure SiO_2 capillary mounted on a modified Swagelok T-piece. A needle allows to flux the gas through the sample during the thermal treatment. The T-piece was mounted on a Huber goniometer head allowing an accurate alignment on the beam. The capillary was kept rotating around its axis during the data acquisition to improve the grain orientation statistics.
Sample heating was achieved by a gas flow Heather (Cyberstar Grenoble, France) whose temperature was previously calibrated measuring, in the same experimental condition, the thermal lattice expansion of annealed Ag powder sample (325 mesh) (Aldrich).

Results

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.

Reference

[1] Meneghini C. et al. J. Synchrotron Rad. 8, 1162 (2001).

Publication

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