



Experiment title: EXAFS experiment for the structural analysis of pumice-supported Pd-Ag bimetallic catalysts.	Experiment number: CH-463	
Beamline: BM08	Date of experiment: from: July 4 th 1998 to: July 11 th 1998	Date of report: July 30 rd 1998
Shifts: 18	Local contact(s): Stefano Colonna	<i>Received at ESRF:</i> 866L 100V 0 1

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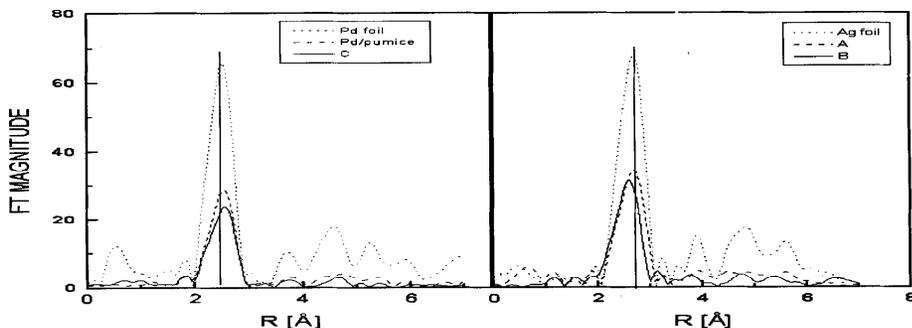
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Report:

Silver-Palladium bimetallic catalysts were synthesised, in different Ag:Pd ratios [1], by decomposition of organometallic compounds that have been anchored on the pumice OH groups. The major advantage of this preparation route is that the precursors reduction is carried out at room temperature, thus limiting possible sintering effects. Preliminary tests on mono and bimetallic samples synthesised with the SMAD technique [2] were also performed. The reported XAFS experiments have been carried out to get more information about the structure of the investigated samples and, in particular, to check the effectiveness of the different preparations in alloy formation. The XAFS measurement at the Ag (25.514 keV) and Pd (24.350 keV) K-edges were performed on the Italian beam line GILDA. The photon energy range was 24.150 to 25.450 keV for the Pd K-edge and 25.460 to 27.000 keV for the Ag K-edge. Beam energies were defined using a Si(3 1 1) double crystal monochromator that gives a resolution of about 1.5 eV at these energies. The dynamical sagittal focusing of the GILDA monochromator [3] allowed to focus 4mrad of the horizontal

beam divergence to achieve a small and intense ($\sim 10^{11}$ ph/sec) spot at the sample. This configuration is particularly suited for fluorescence XAFS measurements on diluted **samples because high photon fluxes are required. The XAFS measurements were performed** in transmission geometry, with two Ar filled ionisation chambers, on three bulk reference samples (Ag, Pd and Ag_2O) and in fluorescence geometry, using a 7-elements Ge multidetector, on the pumice supported samples. All XAFS spectra were recorded at 77°K to reduce the thermal disorder. Some noteworthy Fourier transforms ($\text{C}=\text{Pd } 0.25\% \text{ Ag } 0.5\% \text{ wt}$, $\text{A}=\text{Pd } 0.5\% \text{ Ag } 0.5\% \text{ wt}$, $\text{B}=\text{Pd } 0.5\% \text{ Ag } 0.15\% \text{ wt}$) are shown in the figure below. The calculations were preliminarily carried out using the software package “EXAFS pour le mac” [3]; the shifting of the first Pd shell to larger R-values and the contraction of the Ag one suggests the possibility of alloy formation. More careful analysis is being performed with GNXAS. The SMAD catalysts, as it results from the first data analysis, do not show variation in the first shell distance of Pd and Ag; however, the fine details of the preparation route should be subjected to further investigation.



References :

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