ESRF	**Experiment title:** Differential AWAXS experiments for the structural characterization of pumice-supported Palladium catalysts.	**Experiment number:** CH-428
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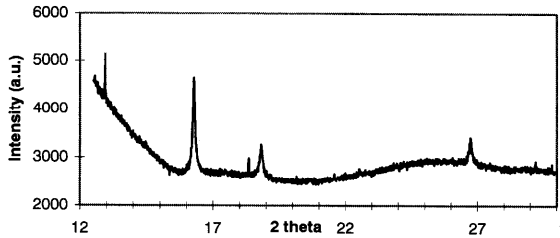
Report:

WAXS and AWAXS experiments on different Pd/Pumice catalysts with a metal content of 0.5% in weight and on one Pd-Ag/Pumice catalyst 0.25Pd-0.08Ag %wt, have been carried out. The investigated Pd/pumice samples have been obtained from the starting one by annealing in hydrogen atmosphere at 200 °C for 30', lh, 4h, and at 400 °C for 30', lh, 4h, 8h, 17h. The WAXS pattern of the not annealed Pd catalyst and of a pumice sample were recorded as well. All the measurements have been performed on spinning capillaries of 1.5 mm of thickness with a beam size of 1x5 mm. In order to obtain a suitable signal to noise ratio, each experiment took, on the average, a whole shift to be carried out. The experiments on the Pd samples and on the pumice one were performed at a wavelenght of 0.6358 A, off the Pd edge, whereas the WAXS patterns on the Pd/Ag sample have been recorded also at the Pd (λ=0.50909 A) and Ag (λ=0.48600 A) edge. It was possible to make only preliminary tests on the Pd samples at the Pd edge, that, according to the initial project, would allow to perform a detailed profile study by extracting the support scattering

by differential AWAXS.

The main results of the experiments can be summarized as follows:

a) The Pd/pumice samples have been analyzed by fitting to the whole WAXS pattern the calculated powder profile based on a model of size distribution and structural disorder 1). The support scattering has been taken into account by properly scaling the experimental pumice pattern. The Pd scattering is mostly due to fcc clusters that are size distributed and disordered by growth stacking faults. In particular, see the figure, the lorentzian shape of the diffraction peaks is indicative of an exponential distribution of sizes, and the peak intensity ratio between the 200 (2θ=18.77°) and the 111 (2θ=16.24°) peaks is the most evident token of structural disorder due to stacking faults 1). Either the average size of the clusters or the amount of disorder changes during the annealing. A sensible decrease in the fraction of stacking faults (ranging from 0.11 for the not annealed sample, to 0.0014 for the 17h, 400 °C annealed one) and an increase in the volume of the clusters is clearly observed.



b) The presence of a second phase can be inferred from the sharp peaks observed in fig. 1 at 12.94" and 18.34". These peaks have been indexed as the 110 and 200 peaks of a bcc structure with a lattice constant 2% larger than the fcc Pd one. Very faint 211, 220 and 310 peaks, hardly distinguishble from the noise, have been also detected.

c) The AWAXS experiments on the Pd-Ag/pumice catalysts have been carried out to support the EXAFS analysis performed at BM08 on bimetallic catalysts. By AWAXS, in particular, it was possible to validate the EXAFS result that there is no evidence of Pd-Ag alloying and that the Ag clusters are very small, under the limit of sensitivity of WAXS 2).

l)A. Martorana, G.Deganello, D.Duca, A.Benedetti, G.Fagherazzi, *J.Appl.Cryst. 25(* 1992)31

2) A. Balerna et al. (1998) (manuscript in preparation).