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## EXAFS STUDY OF SPUTTERED Pd CLUSTERS

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The structural properties of small metal clusters have been extensively investigated with several experimental techniques, to ascertain possible difference from the corresponding bulk structure.

Generally, a contraction in the lattice parameter is experimentally found<sup>(1)</sup>, for metal clusters non interacting with substrate, while, up to day, no clear evidence of a structural transition from the bulk crystallographic structure to icosahedral one has been observed, in contrast with thermodynamical calculations.

An anomalous behaviour has been found for Pd clusters. By transmission electron microscopy, Helnemann and Poppa<sup>(2)</sup> found an expansion of the lattice parameter of  $2.9 \pm 0.2\%$ , for in situ evaporated clusters of 15 Å diameter. This has been confirmed by an EXAFS measurement of De Crescenzi et al.<sup>(3)</sup> on 14 Å sputtered Pd cluster. Both authors attributed such an expansion to structural transition to icosahedra. A different interpretation has been propounded by Garmon<sup>(4)</sup> who attributed the expansion to a lattice strain induced by the substrate.

Considering such different interpretations, we began an extensive EXAFS study of sputtered Pd/C clusters as a function of the cluster size and temperature. Samples were prepared in vacuum, by cosputtering Pd and graphite and depositing them on 10 μm Mylar thick films. Samples were characterized by electron microscopy, to determine the clusters mean diameter (23 Å, 17 Å, 11 Å, 5 Å). An evaporated high purity Pd film and a sputtered thick Pd film, were used as reference. Pd L-edges were measured, in absorption, on the PULS X-ray beam line. Data analysis on the L<sub>I</sub> edge shows a great difference between EXAFS spectra of the sputtered clusters and the evaporated film. The difference is already present between the two reference samples. It consists not only in an expansion of the interatomic distance of 2.5%, but also in an anomalous behaviour of the back-

scattering amplitude function.

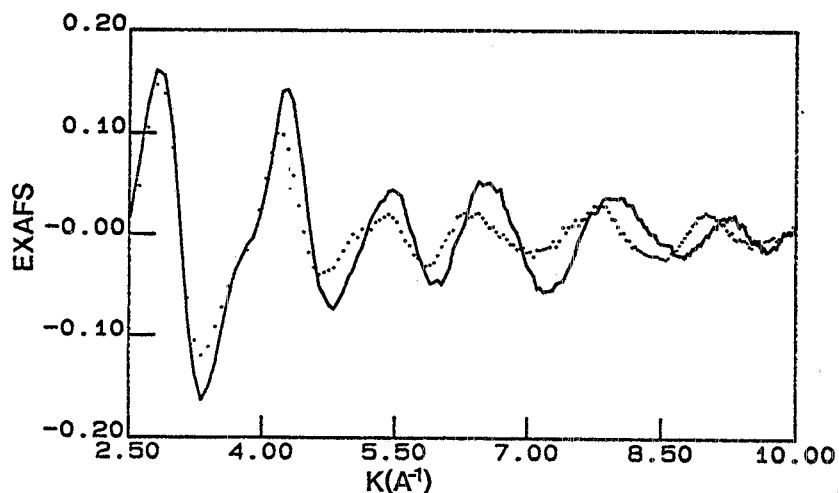


Fig. 1 - EXAFS spectrum of evaporated (—) and sputtered (···) Pd thick films.

To explain such results different mechanisms have been considered: A) Hydrogenation; B) Lattice expansion due to an unusual cluster effect; C) Structural transition; D) Metal-support interaction. Hypothesis A can be discarded, since our  $L_{III}$  edge spectra don't show the characteristic peak due to  $Pd(4d)-H(1s)$  antibonding level, at 6 eV above the  $L_{III}$  edge. Hypothesis B seems unrealistic for the previous considerations on the backscattering amplitude. To evaluate the role of point C and D more information from the EXAFS data, should be obtained using fitting procedures.

#### References:

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