

# SHORT RANGE ORDER IN Pd AND PdO NANOPARTICLES EMBEDDED IN CARBONACEOUS MATRIX STUDIED WITH THE XAFS SPECTROSCOPY

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Nanocrystalline films formed of nano-Pd grains embedded in carbon matrix show a highly developed surface. Films were obtained during the physical vapour deposition (PVD) process from two separated sources containing fullerene and palladium acetate. TEM observations revealed nano-Pd grain shapes. Significant fraction of constituent atoms was located in the grain boundary region. Those atoms occur in the environment different than that in the bulk crystal. The short-range order around Pd atoms was investigated by mean of Pd K- and L-edge X-ray absorption fine structure (XAFS) spectroscopy in order to find what kind and how far extended is a geometrical arrangement around those atoms.

XAFS measurements were performed at A1 beamline at DORIS III storage ring in Hasylab for the films prepared with various temperatures and Pd concentrations. Fourier Transform curves calculated for the measured fine structure oscillations showed that palladium atoms are involved in Pd–Pd and Pd–O bonding. For the samples containing palladium oxide, the range of order was found limited to the first and second coordination shells only. For those samples, the corresponding parameters describing contribution from Pd–O–Pd scattering path were found equal within their error ranges. The second shell, although, observed in FT, was strongly disordered. Corresponding Debye-Waller factor was found greater than 0.8. The electron wave backscattered on that shell contributes to the EXAFS in a very short  $k$  range only, thus precludes the reasonable FT analysis.

We performed FEFF calculations [1] of Pd K-edge EXAFS for Pd fcc and PdO tetragonal crystal structures and separated the contributions originating from particular scattering paths. The minor contribution coming from Pd–Pd'–Pd was observed as a hump disturbing the main oscillation at  $k = 4 \text{ \AA}^{-1}$ . The magnitude of this feature was evaluated and interpreted in terms of changes in the atomic order occurring in the range of second coordination shell.

The applied analysis permitted to conclude qualitatively on the highly disordered structure. We found that the creation of amorphous palladium oxide deteriorates the growth of metallic grains.

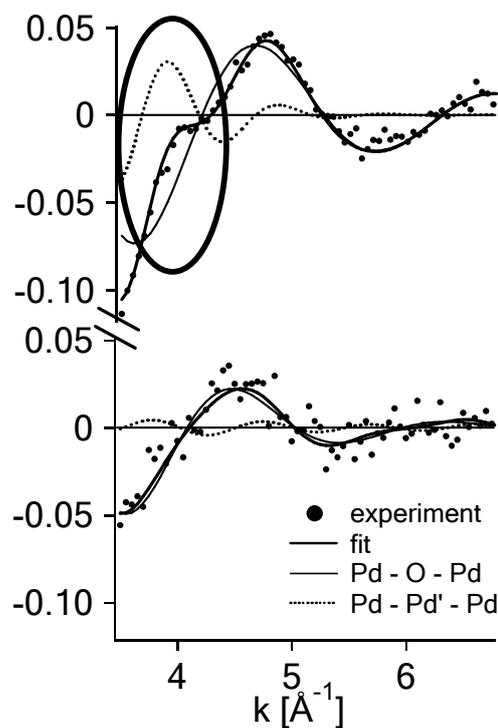


Figure 1. PdO Pd K-edge fine structure oscillations split into contributions. The second coordination shell is manifested by the feature in the oval.

## References

- [1] B. Ankudinov, J.J. Rehr, S.D. Conradson, "Real-space multiple-scattering calculation and interpretation of x-ray absorption near-edge structure", *Phys. Rev. B* **58** (1998) 7565-7576.