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Nanocrystalline peak profile analysis revisited

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In our paper [1] we have shown that Warren-Averbach peak profile analysis in the case of metal nanocrystals meets substantial problems and results in not reliable crystallite size estimation. The principal problem lays in peak asymmetry caused by the lattice parameter being dependent on the crystallite size. The effects of surface relaxation (or reconstruction) are becoming increasingly important with lowering the crystal size.

As a solution, we propose now a different approach to the analysis enabling estimation of both: lattice parameter size dependence and the crystal size distribution. The method consists in fitting the peak profile to a number of Gaussian profiles of different widths. A stable fit has been obtained for 4 Gaussian components. Accurate data then reveal peak position-width (corresponding to lattice parameter- crystal size) dependency (Fig.1) for a sample of nanocrystalline Au supported on amorphous carbon measured in situ while exposed to three gaseous environments: H_2, He, O_2. These values are compared to the lattice parameter-crystal size dependency obtained from atomistic simulations results for a range of sizes of Au cubooctahedra. The model clusters were energy relaxed using Sutton-Chen type potentials and their diffraction patterns calculated via Debye formula determining the diffraction measured lattice parameter.

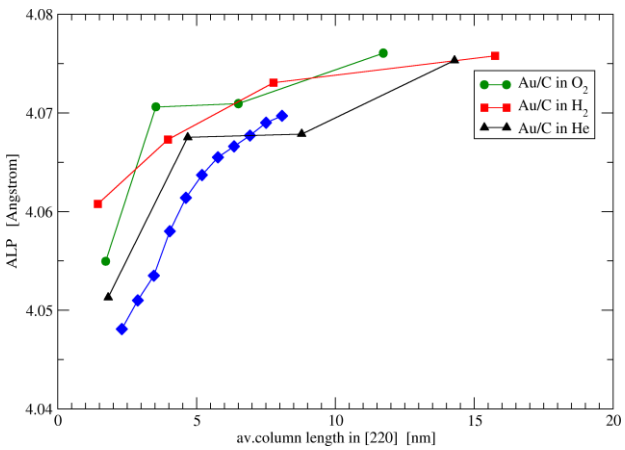

Figure 1. Experimental lattice parameter-crystal size dependency from 220 Au peak profile compared to values simulated theoretically (blue diamonds).

We present results of this peak profile analysis for 220 reflection of gold. The difference between peak profiles in different atmosphere is detectable and analyzed in terms of variation of the average crystal shape leading to a noticeable reshaping of the column length distribution.

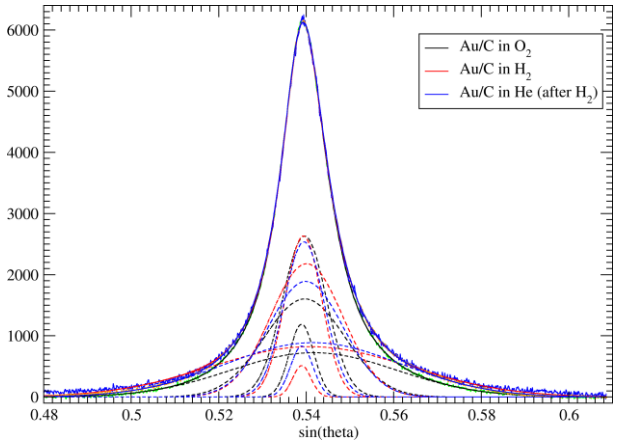

Figure 2. 220 peak profiles of nanocrystalline gold exposed to three gaseous atmospheres H_2, He, O_2. The component Gaussian profiles are given as dashed lines.

The resulting column length distribution calculated assuming no strain, is compared to the distributions from FW1/5M and FW4/5M method [2] differing from the latter.

Gold, similarly like other 5d metals, is susceptible to surface reconstruction occurring on free surfaces as well as on the chemisorbed or physisorbed crystal faces. Our in situ diffraction results provide evidence of bonding states of various gases on nanocrystalline gold corresponding e.g. to bonding energy of approx. 0.2 eV per atom for oxygen adsorption. The diffraction peak shifts on exposition to reactive gases are detectable for crystallites up to 20 nm size.

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[2] R. Pielaszek, *J.Alloy Compd.* **382** (2004) 128.