Proposal Code: MA-3959
Proposal Title: Asymmetry of diffraction peaks from nanocrystalline materials: origin and analysis.
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The study aimed at analysis of intrinsic asymmetry of powder diffraction peaks from metal nanocrystals having certain size distribution. The asymmetry stems from different degree of relaxation of crystals of different size. The relaxation causes peak shift and its range depends on the size. Correct description of structure and analysis depends on evaluation of strain effects and this can be done by comparing peak shape for few orders of reflection (corresponding to the same crystallographic direction). For this reason we required detection of high Miller indices peaks and use of a reasonable small wavelength.
During allocated 6 shifts we succeeded in powder pattern collection of samples of nanocrystalline metal supported on amorphous silica: Pt/SiO_2, Au/SiO_2 and Pd/SiO_2 exposed to several atmospheres: air, oxygen, helium and hydrogen. The calibrated wavelength was 0.40025588 Å and the measurement covered scattering angle range up to 50 degrees. The use of 2D detector allowed for excellent statistical accuracy at high angle that makes possible analysis of high indices peaks of measured fcc nanocrystals of size 5-10 nm (see example for Au - figure).

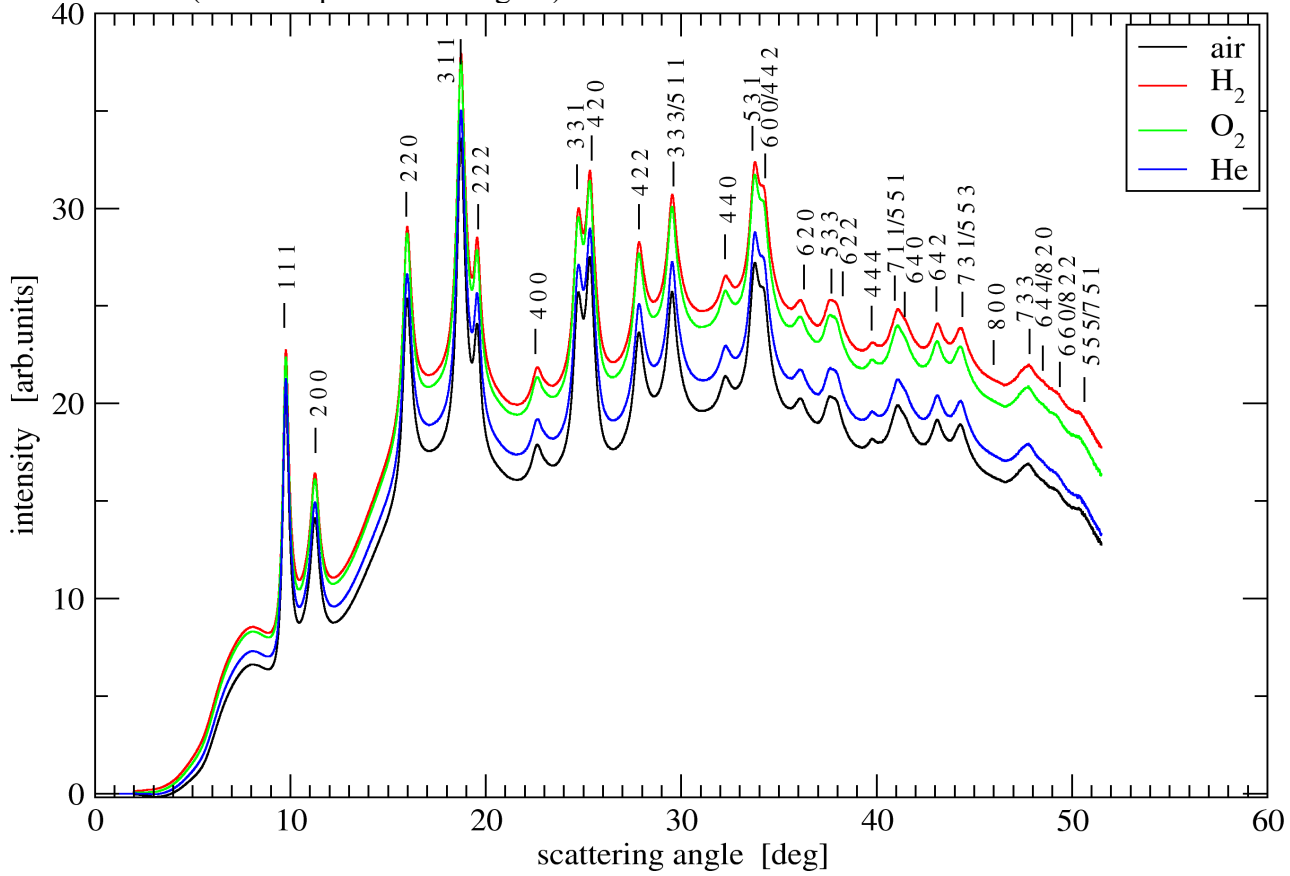

Fig. Powder diffraction pattern (capillary Debye-Scherrer geometry) for nanocrystalline gold sample exposed to several atmospheres. For better visibility of the high angle peaks the pattern of silica was subtracted and the difference was multiplied by $\sin^2(\theta)$.
The data are being analyzed using developed by us technique (Kaszkur et al., J. Appl. Cryst. (2017). 50, 585–593) and Radial Distribution Function analysis. Careful inspection of the measured profiles in various atmospheres reveals their slight shift and change of shape. It is connected with surface evolution of the metal nanocrystals triggered by interaction with gases (amorphization, relaxation and reconstruction triggered by chemisorption).