	Experiment title: **Structural evolution of gold nanoparticles under continuous exposure to synchrotron X-ray beam**	**Experiment number**: HC - 5301
Beamline: ID10	**Date of experiment**: from: 07 March 2023 to: 13 March 2023	**Date of report**: 20.10.2023
Shifts: 18	**Local contact(s)**: Dr. Oleg Konovalov	*Received at ESRF:*

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Report:

The study aimed to analyze the structural evolution of Au NPs exposed to the intense synchrotron X-ray beam. The EIGER 500K detector acquired SAXS and WAXS spectra in a q-value range from 0.057 to 7 Å-1.
The structural evolution of NPs under X-ray beams is sensitive to many factors; therefore, multiple sets of experiments were arranged. During the allocated 18 shifts, we successfully explored the dependence of X-ray damage on the following:
- Various-size Au@polyvinylpyrrolidone (PVP) nanoparticles: ~2,~ 3,~ 5, ~8 and ~23 nm;
- Concentration of metal: two samples had different metals to support mass ratios equal to 7, 3.5, and 1.5% of gold;
- Energy of the beam: 11918.7 and 11900 eV;
- Flux of the beam: 100 and 15% of the full intensity;
- Environmental conditions: He, Ar, Air, and water.

Figure 1 shows a summary WAXS and SAXS data analysis of one (out of 32) experiment: ~5.3 nm Au@PVP nanoparticles (μMe=7%) exposed to the high flux X-ray beam in He atmosphere with the energy equal 11900 eV.
The preliminary analysis of Figure *1* reveals that while X-ray beam interactions with nanoparticles primarily involve two competitive processes. The first one is a dissipation of gold atoms from the solid state into a single particle "gas-like" state. In other words, X-ray beams "ionize" gold atoms in clusters, causing their expulsion

and forming a “gas-like” metal cloud around them. The second process entails nanoparticles growth similar to the Ostwald ripening mechanism.
However, the precise mechanisms governing the interaction between X-rays and nanoparticles demand further in-depth exploration. The current understanding of this complex interplay will be refined through the analysis of the experiments conducted under the proposal.

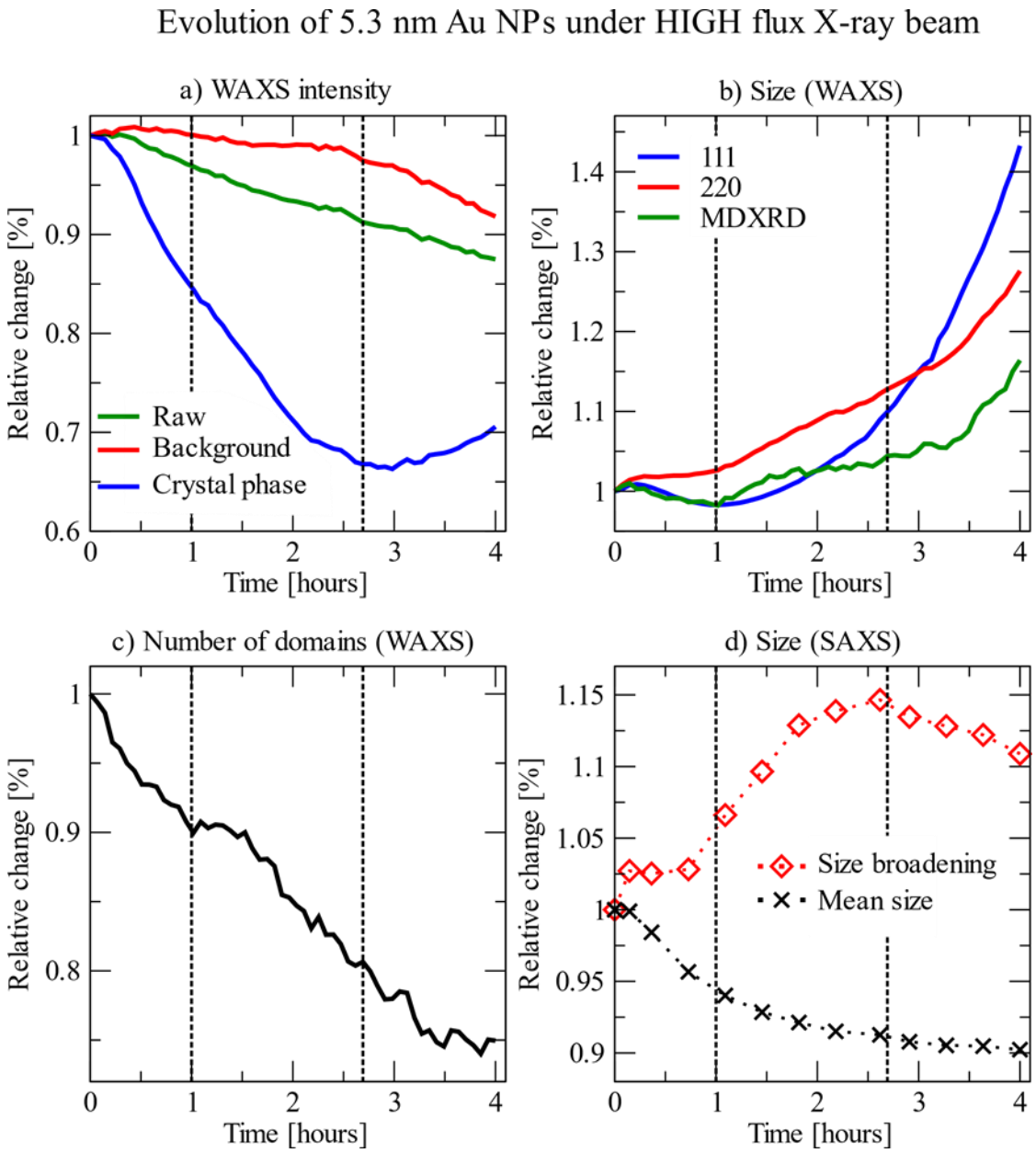

Figure 1 Summary of WAXS and SAXS data analysis on 5.3 nm Au NPs exposed to high flux X-ray beam for 4 hours. a) Relative change in the intensity of WAXS patterns calculated by integration of point intensities in the q-range from ~2.3 to 7 $Å^{-1}$: the green line shows the total intensity of raw patterns, the red line represents the intensity of the background line; the blue line represents the total intensity of a crystalline phase; b) Relative size estimated based on WAXS range: blue and red lines represent Scherrer equation estimations based on 111 and 220 peaks, respectively, the blue line shows the size analysis by MDXRD method; c) Analysis of a relative number of domains evolution by MDXRD approach; d) Relative size analysis estimated on SAXS patterns in the q-range from ~0.058 to 0.5 $Å^{-1}$: black crosses shows a change in the mean size, red diamonds show change in the size distribution broadening. Calculations were performed assuming NPs of spherical shape with lognormal size distribution.