Single crystal timelapse measurement using ultrasonic acoustic levitation

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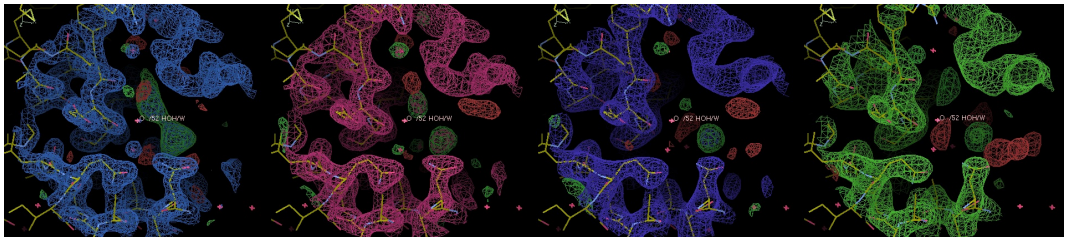
A new diffractometer has been developed and installed at the Swiss Light Source, Paul Scherrer Institut. The diffractometer enables us to complete a dataset in few hundred milliseconds in a combination with EIGER $\times$ 1M detector. Recently, we have demonstrated the successful acquisition of complete datasets with such a diffractometer using EIGER $\times$ 16M detector within a few seconds [1]. The crystal structure solved from the datasets indicated that no damage was induced by the ultrasonic pressure. We consider that our new diffractometer, delivering the sample at room temperature in container-less liquid droplet, offers a unique capability to measure the time-evolution of single crystal upon changing the sample environment. In this presentation, as an example of such a timelapse measurement using the acoustic levitation diffractometer, we present the ligand soaking of a single crystal and the following confirmation change due to the ligand binding. In contrast to the time-resolved studies [2][3] requiring a large number of crystals and the limited maximum time scale in order of nano- microsecond seconds, here presented time-lapse measurement realized by the acoustic levitation diffractometer is advantageous for performing single-crystal experiments with the time scale of a few seconds or longer.

The figure shows the partial electron density maps of a lysozyme crystal at 30, 120, 210 and 300 seconds after initiation of the ligand (p-toluenesulfonic acid solution) soaking. The experiment was conducted on X06SA at SLS with 10 keV X-ray beam with a beam size of 100 $\times$ 50 (H$\times$V, FWHM). The sample was delivered in a 4 μl droplet, levitated in ambient air of an acoustic levitator operating at 39 kHz ultrasonic frequency. We collected 10 datasets from a single lysozyme crystal in total, processed those by CrystFEL program suite, and solved the crystal structure by the molecular replacement method with the CCP4 program suite. Radiation damage is clearly seen at the side chains exposed to the protein surface in the electron density maps. At 30 s (left most panel), the binding of the ligand to the crystal is apparent. With the increase of the measurement time, the electron density of the ligand (in green) gradually becomes weaker at the binding site. At 300 s, the radiation damage at the side chains is observed as the ambiguous density map.



Electron density maps of a lysozyme crystal at 30, 120, 210 and 300 seconds after the ligand (p-toluenesulfonic acid solution) soaking.

References

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