Effects of the Top-Up Operation Mode on X-ray Photon Correlation Spectroscopy Measurements

P. Falus, M. A. Borthwick, L. B. Lurio, A. Rühm, S. G. J. Mochrie*

Center for Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA, U.S.A.

**Present Address: Departments of Physics and Applied Physics, Yale University, New Haven, CT, U.S.A.*

Introduction

In order to carry out an effective x-ray photon correlation spectroscopy (XPCS) measurement, one has to make sure that dynamics detected in the measurement are coming from the sample being studied not from the experimental setup. Previous experiments warranted that the topup mode of operation at the APS may cause fluctuations in the beam size and the beam position thus introducing unwanted coherence and intensity changes in our XPCS setup. To test the feasibility of XPCS measurements at time scales longer than the interval between two top-up fills, a systematic study was carried out testing different methods to exclude data taken during a fill and trying to optimize slit settings for the top-up mode. To our pleasant surprise the beam stability was much better in March 2001 than in December 2000. The beam stability in top-up mode in March 2001 was comparable to previous non-top-up mode results.

Methods and Materials

The layout details of the 8-ID-E SAXS-XPCS beamline can be found in previous publications.[1,2] Here we would only repeat that the white beam from the undulator source passes through a set of cooled slits at 29 m from the source, then it is monochromatized by a germanium monochromator, and the beam is finally collimated by a pair of precision crossed slits 55 m from the source, 40 cm upstream of the sample. In this setup, by closing down the white beam slits, one can reduce the overall intensity and can also limit the source size thus reducing the dependence of coherence on the beam size. By closing the white beam slits, one can also reduce the intensity fluctuations caused by beam movement. The December 2000 measurements were carried out at 200 μm x 400 μm white beam slit sizes, while in March, measurements at several smaller slit sizes were carried out. During all of these measurements the collimating slits were set to 20 μm x 20 μm openings. The detailed effects and the optimization of these slit sizes were described in a previous work.[1]

While optimizing the slit sizes may reduce the effects of beam movement, another approach is to detect the data frames that are affected by a fill and exclude them from the analysis. We detected these "damaged" frames by monitoring the appropriate EPICS variables. We modified our previous data reduction system to allow for exclusion of frames.[2]

The sample used to produce the scattering is one of our "benchmark" silica gels; its preparation has been published earlier.[3] Aerogels are very useful to characterize coherent x-ray scattering beamlines as they are very strong scatterers and they also generate strong speckle due to their random structure. Aerogels are practically static. In the time range accessible to XPCS (up to 10000 s), they do not exhibit movement; thus, if a time constant is detected, it should be due to some other instability not sample dynamics. Our regular beamline test includes taking data on our aerogel sample. The flatter the correlation function, the longer the characteristic time constant, the better the beamline stability. We compared the beamline stability at different settings using this aerogel standard.

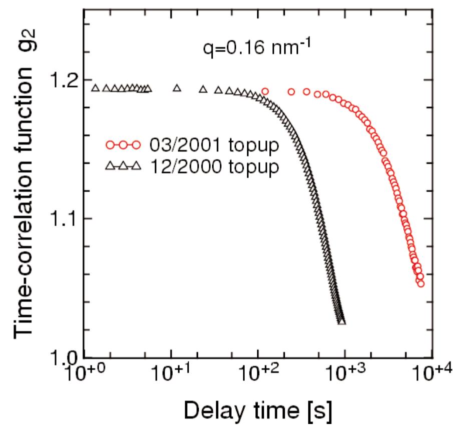

FIG. 1. Time correlation function measured on our aerogel sample in top-up mode in 3/2001 and 12/2000. Note that the much faster decay in the December data effectively limits measurements to at most 200 s, while the March results allow the detecting of time constants up to 10000 s.

Results

Looking at Fig. 1, it is apparent that, in the March top-up mode, correlation functions are flat for a longer time than the December results. It is clear that the decay of the December correlations begins at 200 s—approximately the top-up cycle time—while the March results extend much beyond the refill period.

For a more qualitative evaluation, we fitted each of the correlation functions with a single exponential decay and plotted the decay time versus wave number in Fig. 2. The decay times in top-up mode have improved by a factor of 15 between December

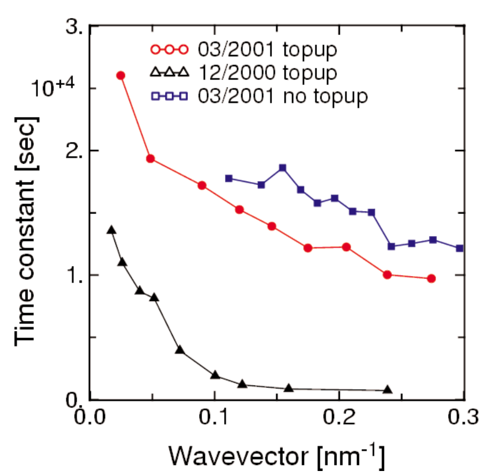

FIG. 2. Fitted time constants of XPCS correlation functions on our aerogel sample versus wave number. The higher time constants signal a more stable beamline. Note the marked improvement from December to March.

March. Even though the March top-up results do not reach the non-top-up mode results, they are only 20% worse, thus comparable.

The time constants with smaller white beam slits and excluded frames improved less then 4% from the uncorrected top-up mode results, thus we were unable to show if these methods are effective in increasing stability.

Discussion

A marked increase in beam stability occurred between December 2000 and March 2001. As our beamline was not changed between the two runs, it is clear that the improvements were made on the APS side. While we do not have control over the fill parameters, we have developed a quick test to determine if XPCS measurements are feasible. We hope that, in the future, top-up APS runs will be as good as the March 2001 one, allowing XPCS measurements to take advantage of the top-up mode.

Acknowledgments

8-ID was developed with support from the NSF Instrumentation for Materials Research Program (DMR 9312543), from the DOE Facilities Initiative Program (DE-FG02-96ER45593), and from NSERC. Work at MIT was also supported by the NSF MRSEC Program (DMR 9808941). Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38. We thank Harold Gibson for his invaluable assistance.

References

[1] A.R. Sandy L.B. Lurio S.G.J. Mochrie, A. Malik, G.B. Stephenson, J.F. Pelletier, and M. Sutton J. Synchrotron Rad. **6**, 1174 (1999).

[2] D. Lumma, L.B. Lurio, S.G.J. Mochrie, and M. Sutton, Rev. Sci. Instrum. **71**, 3274 (2000).

[3] O.K.C. Tsui, S.G.J. Mochrie, and L.E. Berman, J. Synchrotron Rad. **5**, 30 (1998).