Characterizing a high resolution, scanning fluorescence X-ray microscope with the attoFPSensor

B. Sipos
attocube systems AG, Munich, Germany

X-ray microscopy has been established as a standard tool in many areas of science. The development of novel focusing techniques and ultrabrilliant synchrotron sources pushed the resolution below 1 micron. However, the ultimate goal of sub10 nm spatial resolution is still an open challenge. The difficulty is twofold: On one hand, the spot size has to be reduced, but on the other hand, also the mechanical and consequently the thermal stability of the microscope assembly have to be improved. Whereas the first one can already be achieved using multilayer Laue lenses (MLL), the latter remains a difficult task. Thermal fluctuations due to the environment or heat generated by the components of a microscope can cause large mechanical drifts. In the case of scanning probe microscopes (SPM), where even sub-nm resolution is possible, the desired stability is commonly achieved by compact design. This not only increases the resonance frequency but the reduced linear size also limits the effect of thermal drifts. Therefore, compact form and good thermalization are the right principles to follow in any instrument design where nanometer resolution is desired.

An X-ray microscope is a more complex instrument then an average SPM. The target distance limits the possibilities in size reduction. Furthermore, the resolution not only depends on the stability of a sample, but also on the stability of the beamline and the pieces delivering it to the sample. This stability has to be maintained over much longer period of time when compared to SPM measurements.

When developing an X-ray microscope capable of nm resolution, careful design is a must. Thermal and mechanical stability of the components and assemblies has to be followed throughout the process. When E. Nazaretski *et al*. set the goal to build such an instrument [1], they needed a tool that can monitor mechanical stability at sub-nm scale over several hours. They chose attocube's award winning attoFPS sensor [2,3]. The attoFPS sensor is specifically designed to measure sub-nm displacements while having an outstanding stability. To ensure both, measurement stability and resolution, the group first designed two experiments to characterize them.

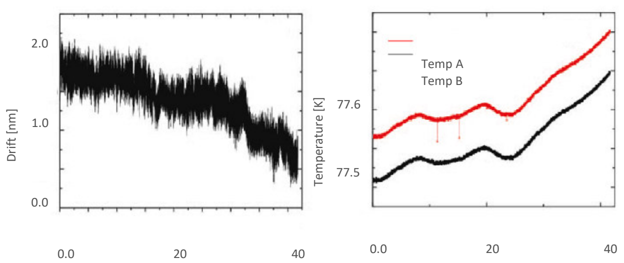

Figure 1: Long term stability measurement at liquid nitrogen temperature. Left: interferometer readings as a function of time. Right: temperature readings on the top and bottom flanges of the cavity, the observed drift of the distance measurements correlates well with the temperature drift due to slow boiling off of LN_2. [4]

Long term stability measurement

To measure the long term drifts of the attoFPS, they constructed a 50 mm long cavity, where on one end of the cavity they placed a reflector and on the other end the attoFPS collimator. To limit the temperature drifts and thermal extension coefficient of the cavity, it was placed in a vacuum chamber and cooled down to liquid nitrogen temperature. This was possible thanks to the special fiber based design of the attoFPS and the available low temperature compatible collimator. To monitor the temperature of the setup a calibrated temperature sensor was mounted on the cavity.

Figure 1 shows the data collected over 40 hours. The total measured drift was 1.25 nm. However, this can be mainly attributed to the slow change in the temperature of the setup due to the boil off of the liquid nitrogen. The correlation between the temperature and the measured cavity length can be seen by comparing panel a and b of Figure 1.

Resolution measurement

To characterize the inherent noise and resolution of the attoFPS, the group constructed a special setup where a small reflector was placed on top of a high performance scanner equipped with a capacitive sensor. Around the reflector in three directions, three collimators of the attoFPS were mounted. The distances between the reflector and collimators were 25 mm (Figure 2). To limit the acoustic and mechanical noise as well as the effect of temperature and pressure fluctuation, the setup was enclosed into a double walled insulating box that was further covered by sound absorbing foam. Once the stability was ensured, several 1000 pm, 500 pm, and 300 pm steps were performed. The step sizes were adjusted using a capacitive sensor and measured by the interferometer. The results of the measurements in one channel can be seen in Figure 3. The results in two other channels were similar. The interferometer

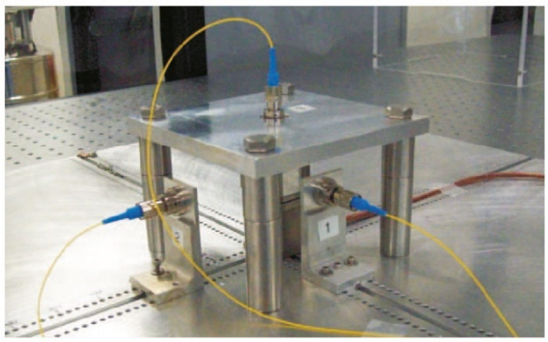

Figure 2: Setup for interferometer resolution test. Photograph of the setup showing interferometer heads (1) and scanner (2). [4]

was able to resolve even the smallest step with 100 Hz bandwidth. They also observed that at ambient conditions (without the enclosure) the background noise, due to thermal and pressure fluctuations, increases to 1 nm.

Characterizing the microscope

Once the outstanding performance of the attoFPS had been proven, the group used it to characterize the stability of the MLL optics. A special frame was constructed around the microscope that can hold three collimators and measure the drifts of the horizontal or vertical MLL optics in the three spatial directions. An environmental shielding box, similar to the one used during the resolution measurements, was mounted around the setup. On top of the mechanical drifts, the temperature of the MLL optics and the base plate, the temperature and pressure of the room were monitored during the measurement. The total drift in the most sensitive y direction did not exceed 10 nm. In the x direction the drift was 25 nm, and in the *z* (along the Xray beam), they observed 11 nm. This was measured while the temperature of the microscope stayed within a 5 mK range. The small temperature drift together with the slight change in the room pressure can be attributed to some of displacement sensed by the interferometer due to the change in the refractive index of the air. In the case of the horizontal MLL optics the results were similar.

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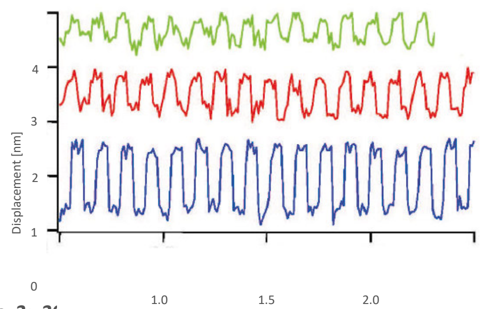

Figure 3: 300 pm, 300 pm, an[illegible] pm steps performed by the scanner in the z-direction and recorded by the fiber-optic interferometer. Interferometer head-reflector separation was set to 25 mm. [4]

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Characterization using X-ray

Finally the setup was placed in the I13 beamline of the Diamond Light Source. To characterize the system, they used an Au test sample created with electron beam lithography. The measured pattern was an array of 1 um wide Au crosses. Figure 4 shows results of the measurement in two scan directions. The data was collected at the L line of Au, with 100 nm pixel size and the total acquisition time of 6 hours. Using this result, they determined the stability and resolution of the microscope. They found that the resolution is in the order of 40 nm in both directions, and the stability is below 45 nm over the entire time needed for data collection.

Conclusion

E. Nazaretski et al. built a new ultra stable multilayer Laue optics based scanning hard X-ray fluorescent microscope capable of resolution on the order of 40 nm. To achieve this they needed to characterize the mechanical drifts of their setup, and choose the attoFPS sensor because of its outstanding resolution and stability. According to their measurements, the senor has a better than 1.25 nm stability over 40 hours, and a better then 300 pm resolution at 100 Hz bandwidth in a controlled environment. Using the attoFPS they were able to determine that the stability of their microscope optics is better than 10 nm in the most sensitive direction over 3 hour time period in the laboratory conditions, ensuring an outstanding spatial resolution. Finally, the microscope was placed into an X-ray beam. By measuring a sample of a pattered 1 um wide Au crosses they demonstrated the excellent stability (45 nm over 6 hours) and resolution (on the order of 40 nm) of the system under real measurement conditions.

References

[1] E. Nazaretski, Jungdae Kim, H. Yan, K. Lauer, D. Eom, D. Shu, J. Maser, Z. Pešić, U. Wagner, C. Rau, and Y. S. Chu, Performance and characterization of the prototype nm-scale spatial resolution scanning multilayer Laue lenses microscope, Rev. Sci. Instrum. 84, 033701 (2013).

[2] http://www.attocube.com/attoMETROLOGY/introduction.html

[3] http://www.rdmag.com/award-winners/2012/08/picoscale-sensing-telecom-parts

[4] Reprinted with permission from E. Nazaretski, et al., Rev. Sci. Instrum. 84, 033701 (2013). Copyright 2013, AIP Publishing LLC.

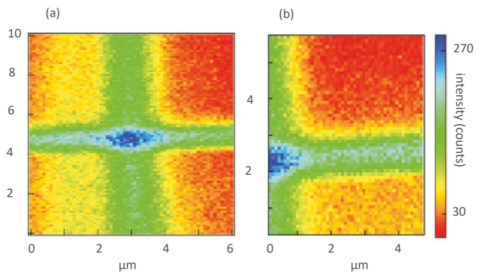

Figure 4: Fluorescence images of the Au test pattern (scan parameters: step size – 100 nm, dwell time – 1 s; note the difference in scan ranges for panels (a) and (b), respectively). (a) Horizontal axis is the fast scanning direction, (b) vertical axis is the fast scanning direction. [4]